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NATIONAL
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LOCKHEED MARTIN

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**Life Prediction Methodology for Ceramic
Components of Advanced Vehicular
Heat Engines**

Volume I - Final Report

**P. K. Khandelwal
N. J. Provenzano
W. E. Schneider**

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LOCKHEED MARTIN ENERGY RESEARCH CORPORATION
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LIFE PREDICTION METHODOLOGY FOR CERAMIC COMPONENTS OF
ADVANCED VEHICULAR HEAT ENGINES

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TABLE OF CONTENTS

<u>Section</u>	<u>Title</u>	<u>Page</u>
	Abstract.....	1
I	Introduction.....	1
II	Material and Specimens.....	2
	2.1 Material Selection.....	2
	2.2 Specimen Fabrication.....	2
III	Data Base Generation.....	6
	3.1 Fast Fracture.....	6
	3.1.1 Modulus-of-Rupture Flexural Strength.....	6
	3.1.2 Uniaxial Button-Head Tensile Strength.....	7
	3.2 Slow Crack Growth.....	21
	3.2.1 MORE Dynamic Fatigue Evaluation.....	21
	3.2.2 Tensile Dynamic Fatigue Evaluation.....	32
	3.3 Uniaxial Creep and Stress Rupture.....	55
	3.3.1 Creep Rupture Testing using Button-Head Specimens.....	56
	3.3.2 Creep Rupture Tensile Testing of Dogbone Specimens.....	88
	3.4 Oxidation.....	98
	3.5 Multiaxial Testing.....	103
	3.5.1 Biaxial Fast Fracture Strength.....	109
	3.5.2 Mixed Mode Fracture of Through-Cracks (Chevron Notch).....	118
	3.5.3 Mixed Mode Fracture of Surface Cracks (Vickers Indents).....	122
	3.5.4 Biaxial Creep.....	125
IV	Nondestructive Evaluation Development.....	133
	4.1 Fabrication of Reference Specimens.....	135
	4.1.1 Reference Standards with Surface Defects.....	135
	4.1.2 Reference Standards with Volume or Internal Defects.....	139
	4.1.3 Fabrication of Ultrasonic Imaging Resolution Standards.....	142
	4.2 Microfocus Radiography.....	142
	4.3 Acoustic Microscopy.....	146
	4.4 Computer Tomography (CT).....	158
	4.5 Reference Standard Evaluation by Aracor Tomoscope CT System.....	159
	4.6 Theoretical and Experimental CT Studies at Chalk River Laboratories, Canada.....	165
V	Analytical Life Prediction Models.....	177
	5.1 Fast Fracture Model.....	177
	5.1.1 Corner Failure Modes.....	177
	5.1.2 Consolidation of the Weibull Exponent.....	179
	5.1.3 Room Temperature Fracture Toughness and Shear Sensitivity.....	183
	5.2 Slow Crack Growth Methodology.....	189
	5.2.1 Uniaxial Slow Crack Growth Theory.....	189
	5.2.2 Application of Slow Crack Growth Methodology to Component Design.....	195

TABLE OF CONTENTS

<u>Section</u>	<u>Title</u>	<u>Page</u>
	5.2.3 Slow Crack Growth Methodology, Application to a General Mission.....	196
5.3	Creep Analysis Methodology.....	198
	5.3.1 Creep/Stress Rupture Model.....	198
	5.3.2 Statistical Stress Rupture Failure Model.....	201
5.4	Oxidation.....	204
	5.4.1 Oxidation Failure Model.....	204
	5.4.2 Probabilistic Oxidation Model.....	206
VI	Confirmatory Test Correlation	212
6.1	Fast Fracture Biaxial Confirmatory Tests.....	213
	6.1.1 Ball-on-Ring Confirmatory Testing	213
	6.1.2 Fast Fracture Analysis of NT154 Pressurized Flat Plate ...	216
	6.1.3 Room Temperature NT154 Spin Test Disks	224
6.2	Slow Crack Growth Biaxial Verification	232
	6.2.1 Ball-on-Ring Testing of PY-6 Material at 1200°C	232
	6.2.2 PY-6 Ball-on-Ring Testing at 1300°C.....	236
	6.2.3 Biaxial SCG Verification — NT154 Spin Disk Testing at 1204°C.....	237
	6.2.4 NT154 Spin Disk Testing at 1371°C (2500°F).....	245
VII	Conclusions.....	253
7.1	Multiaxial Fast Fracture.....	253
	7.1.1 High Strength Silicon Nitride is Shear Insensitive	253
	7.1.2 The Accurate Prediction of Biaxial Surface Fast Fracture Strength is Difficult.....	253
7.2	Biaxial Slow Crack Growth.....	253
7.3	Creep Deformation and Failure.....	253
7.4	Oxidation in the Absence of Stress.....	253
VIII	Recommendations	253
8.1	Multiaxial Fast Fracture.....	253
8.2	Biaxial Slow Crack Growth.....	254
8.3	Creep Deformation and Failure.....	254
8.4	Oxidation in the Absence of Stress.....	255
IX	Summary.....	255
9.1	Material Data Base.....	255
9.2	Determination of Time Dependent Material Properties.....	256
9.3	Probabilistic Time Dependent Models.....	256
9.4	Subcomponent Model Verification	257
	Acknowledgments.....	258
	References	259
	Nomenclature.....	263

LIST OF FIGURES

<u>Figure</u>	<u>Title</u>	<u>Page</u>
1	Types of specimens and tests used to generated the data base required for the development of the ceramic life prediction methodology	5
2	Flexural strength of PY-6 injection molded and HIPed silicon nitride material.....	6
3	Fractography of fast fracture tested MOR specimens tested at (a) room temperature and (b) 1400°C	8
4	(a) Schematic of the gas bearing tensile facility and (b) close-up view of pull rods with the button-head specimen and heating elements....	8
5	Setup for load train alignment in a gas bearing tensile machine.....	10
6	Preliminary button-head tensile specimen geometry	11
7	Composite plot of GTE PY-6 silicon nitride tensile stress-strain responses from three strain gages located 120 deg apart on specimen TN-AX-1.....	12
8	Schematic of button-head tensile specimen.....	13
9	Alignment of specimen/pull rod assembly	14
10	Uniaxial tensile strength of button-head PY-6 injection molded and HIPed silicon nitride specimens from room temperature to 1400°C.....	15
11	Stress-strain behavior of button-head specimen at room and elevated temperatures	16
12	Fast fracture uniaxial testing at 1400°C showing nonlinear stress-strain behavior and grain boundary cavitation.....	17
13	Distribution of gage length failures in button-head specimens	18
14	Fractography of button-head specimen failed at room temperature	19
15	SEM fractography of room temperature tested tensile specimen failed from internal iron inclusion containing nickel and chrome	20
16	Dynamic fatigue behavior of MOR specimens tested at Allison	22
17	MOR bar tested at a rate of 0.212 MPa/sec (0.00508 mm/min) at 1000°C shows softening and bubbling of the material.....	25
18	MOR specimen tested at 1200°C at a average stress rate of 1.55 MPa/sec (cross-head speed 0.0508 mm/sec). Specimen failed from an internal inclusion.....	26
19	Stress assisted oxidation of the MOR at 1200°C and 0.089 MPa/sec in air. Note the dark gray/black ring around a center light region. Surface pitting of the tensile surface is also visible.....	26
20	Optical micrographs of specimens tested at 1400°C at an average stressing rate of 0.035 MPa/sec. Surface pitting of the tensile surface is also visible.....	27
21	MOR specimen tested at 1400°C; 0.035 MPa/sec showing a large prominent gray ring around the light center region	29
22	Dynamic fatigue behavior of PY-6 MOR bars tested in air and argon at ORNL	29
23	Fractography of a specimen tested at 1400°C and 0.001 MPa/sec in air (a) fracture surface showing the damage shown, (b) polished cross-section with ring around the perimeter, and (c) side view of the specimen depicting parallel Mod I cracks on the tensile surface of the MOR bar.....	31

LIST OF FIGURES (cont)

<u>Figure</u>	<u>Title</u>	<u>Page</u>
24	Stress-strain behavior of button-head specimens tested at (a) 6.9 MPa/sec and (b) 0.115 MPa/sec.....	33
25	Fractography of specimens tested at 1300°C and 6.9 MPa/sec (a) failed at 3.307 MPa from surface flaw and (b) failed at 3378.9 MPa from internal inclusion.....	41
26	Fractography of specimen tested at 1400°C and 6.9 MPa/sec with strength of 268.8 MPa.....	42
27	Fractography of specimen tested at 1100°C and 0.115 MPa/sec. The specimen failed from an internal inclusion.....	43
28	Fractography of a specimen tested at 1200°C and 0.115 MPa and failed from an internal flaw at 228.9 MPa. The defect is very porous.....	44
29	Fractography of a specimen characterized at 1300°C and 0.115 MPa/sec. The strength was 336.5 MPa. Failure occurred via SCG and creep damage.....	45
30	Fractography of a specimen tested at 1300°C and 0.115 MPa/sec and failed from an internal inclusion at 254.01 MPa.....	46
31	Fractography of a specimen tested at 1400°C and 0.115 MPa/sec and failed from a surface flaw at 156 MPa.....	47
32	Schematic of dogbone tensile specimen tested at NIST.....	49
33	Optical bottom photograph of specimens after FPI inspection.....	50
34	Ultrasonic C-scan surface wave images of the back surface of dogbone specimens.....	51
35	Surface wave acoustic microscopy of the two side walls of the gage area of dogbone specimens machined from billet 65R.....	52
36	Acoustic microscopy characterization of the gage area of a dogbone specimen using surface wave.....	53
37	Dynamic fatigue behavior of PY-6 Si ₃ N ₄ dogbone specimens.....	55
38	Comparison of dynamic fatigue behavior of button-head and dogbone specimens of PY-6 Si ₃ N ₄ material.....	56
39	Creep rupture test set up at SORI.....	57
40	Typical creep strain versus time curves at 1200, 1260, and 1300°C.....	58
41	Typical creep strain versus time curves at 1370 and 1400°C.....	59
42	Minimum creep rate versus applied stress as a function of temperature.....	66
43	Monkman-Grant behavior showing time to failure as a function of minimum creep rate (some tests were stopped before failure was reached; however, those with $t_f > 200$ hours were used in the regressions and are included on the plot.....	66
44	Stress rupture behavior of PY-6 silicon nitride.....	67
45	Residual room temperature strength of unfailed button-head tensile specimen during creep rupture testing.....	68
46	Optical micrographs of specimens tested at 1149°C (a) 249.1 MPa, $t_f = 0.003$ hr, (b) 239.8 MPa, $t_f = 41.9$ hr.....	69
47	Optical micrographs of button-head creep rupture specimens which failed in less than 1 hr.....	70
48	Typical lower power SEM photographs of specimens tested at 1200°C.....	71

LIST OF FIGURES (cont)

<u>Figure</u>	<u>Title</u>	<u>Page</u>
49	Creep rupture of button-head tensile specimens tested at 1260°C ($\sigma_a = 175$ MPa). Failure time = 364 hr. Initiation of interfacial two-grain junction cavities	72
50	SEM micrograph of interfacial cavities in specimen 515A tested at 1260°C $\sigma_a = 175.2$ MPa, $t_f > 1154$ hr.....	73
51	SEM micrograph of interfacial cavities in specimens tested at 1300°C.....	73
52	Typical SEM photos of a specimen tested at 1370°C, specimen No. 84B, $\sigma_a = 73.9$ MPa, $t_f = 287$ hr	74
53	Excessive creep cavitation in uniaxial tensile specimens tested at 1400°C was detected by SEM. Specimens were prepared perpendicular to the loading axis.....	75
54	(a) $Y_2Si_2O_7$ standard spectra, 10 spectra collected; (b) as received PY-6 Specimen 3561A spectra, 25 spectra collected	78
55	(a) SEM fractography and (b) TEM thin-foil photographs of fracture surface Specimen 636A tested at 1200°C, 175 MPa, > 389 hr.....	79
56	EDS spectra of Specimen 636A in four spots showing identical composition	80
57	EDS spectra of Specimen 636A showing similar composition in two areas (c) and (d).....	81
58	(a) Spectrum of β - Si_3N_4 and (b) hole count in spectrum.....	82
59	TEM photograph of a region with a β - Si_3N_4 grain surrounded by grain boundary phase in location A, B, and C. Specimen 463A tested at 1260°C under 175 MPa and failed after 364 hr	83
60	Diffraction patterns of areas A and B indicating presence of identical material in Specimen 463A	84
61	Comparison of (a) EDS spectra of area A and (b) EDS spectra of $Y_2Si_2O_7$ standard.....	85
62	TEM micrograph of (a) two-grain junction with a crack, (b) magnification of crack region show beginning of lenticular cavities, and (c) EDS spectra of region (a) showing Si_3N_4 with traces of yttrium and oxygen.....	86
63	Fractography of specimen 192B showing interfacial cavitation and grain pull-out. Test temperature: 1400°C, $\tau_a = 75$ MPa, $t_f = 844$ hr	87
64	EDS spectra of grain boundary pocket in specimen 192B tested at 1400°C.....	87
65	Schematic of creep testing set-up at NIST.....	88
66	Typical creep data obtained on NIST specimens. Very long primary transient creep and the absence of tertiary creep before failure is characteristics of silicon nitride.....	90
67	Long-term creep curves showing a sharp break in the slope of the curves. This behavior was observed in about half of the specimens tested and may be due to devitrification at grain boundaries during testing. Test conditions: 86 MPa, 1350°F.....	91
68	Minimum creep rate versus applied stress	92
69	Applied stress versus time-to-failure	93

LIST OF FIGURES (cont)

<u>Figure</u>	<u>Title</u>	<u>Page</u>
70	Creep rupture data follow Monkman-Grant model both for button-head and dobone specimens	94
71	Interface separating silicon nitride grains in PY-6. Within central region (G) interphase is glassy and typically about 1 nm thick; within outer regions (X) interphase is thicker (>10 nm) and contains crystalline second phase.....	95
72	Crack-like interfacial cavity produced in PY-6 during creep at 1400°C. With reference to Figure 71, rapid cavity growth within wider portions of the interface results in crack-like morphology...	96
73	Pair of interfaces containing discrete, ellipsoidal shaped interfacial cavities typically produced at narrow glassy interfaces (1 nm) in PY-6 during tensile creep. Similar phenomenon is observed in NT154. Interfacial cavitation of this type was found in all samples tested at 1250 to 1400°C. Test conditions for this specimen were 1350°C, 86 MPa, test stopped after 1034 hr. Tensile stress axis is vertical to the figure, i.e., specimen was cut parallel to the tensile axis	97
74	Large-scale multigrain junction cavity in PY-6. Diffusive transport of second phase from triple and multigrain junctions during creep results in cavities of varied size and shape	97
75	Surface wave acoustic imaging of oxidized MOR bar specimens.....	99
76	Oxidation behavior of PY-6 Si ₃ N ₄ as a function of temperature after 100 and 500 hours exposure in air	101
77	Effect of time and temperature on oxide layer thickness in MOR bars. Data fitted to (a) logarithmic law and (b) power law	102
78	Room temperature MOR retained strength decreases after 100 hours oxidation in air as a function of temperature	103
79	Fractography of 1000°C oxidized MOR specimens tested at room temperature.....	104
80	Oxidation of PY-6 Si ₃ N ₄ at 1250°C after 100 hours in air.....	105
81	Oxidation of PY-6 at 1400°C after 100 hours and 500 hours in air (note surface pitting).....	106
82	X-ray diffraction of MOR specimens (a) as received and (b) after 100 hours exposure at 1000°C.....	107
83	X-ray diffraction of MOR surface after 100 hours at (a) 1300°C and (b) 1400°C. Both cristobalite and B-Y ₂ Si ₂ O ₇ were detected.....	108
84	X-ray diffraction shows increase in the amount of cristobalite after 100 hours of oxidation in air as a function of temperature (note decrease in B-Si ₃ N ₄ content).....	109
85	Biaxial strength measurement of a circular disk specimen using ball-on-ring test	110
86	Fast fracture biaxial testing of disks at room temperature (a) failed in six pieces and (b) failed in five pieces.....	112
87	Low magnification scanning electron micrographs of silicon nitride biaxial tested specimens.....	113
88	High magnification scanning electron micrographs of silicon nitride biaxially tested specimens.....	114

LIST OF FIGURES (cont)

<u>Figure</u>	<u>Title</u>	<u>Page</u>
89	Comparison of fast fracture strength of biaxial disks and transverse ground bars	117
90	The chevron notched, diametrically compressed crack disk specimen.....	118
91	Mixed-mode testing of chevron notch disk specimens in diametral compression at room temperature with through-cracks.....	119
92	Experimental setup to conduct high temperature mixed-mode fracture toughness testing.....	120
93	Photograph of experimental system to measure elevated temperature fracture toughness using chevron notch disk specimens.....	121
94	Vickers indentation and associated cracks on the surface of MOR specimens dissected from circular disks with a surface finish similar to disks tested for fast fracture biaxial strength. The MOR specimens were tested with varying angles of the cracks with respect to the loading axis	123
95	Mixed mode testing of MOR bars with surface cracks (Vickers indents) at room temperature.....	124
96	Shear-mode/opening-mode fracture toughness ratio.....	125
97	Test setup for biaxial creep experiments using ball-on-ring arrangement; heated in furnace with MoSi ₂ elements	126
98	Setup for the biaxial creep experiments.....	127
99	Representative load/displacement/time record for fast fracture tests at 1200 and 1300°C	130
100	Representative load/time/displacement/time record for creep tests at 1200 and 1300°C	130
101	Biaxial stress rupture (a) 1200°C and (b) 1300°C.....	131
102	Dark field optical photograph at the center of the tensile surface (a) Specimen 34.2563, (b) Specimen 34.2564, and (c) is the bright field photograph of Specimen 34.2564 showing grinding marks and creep cracks.....	132
103	Representative load/displacement curves for (a) short time to failure and (b) long time to failure at 1400°C.....	134
104	Biaxial stress rupture behavior at 1400°C.....	135
105	Layout of excimer laser-drilled holes on the surface of as-machined MOR specimen.....	136
106	Layout of surface-drilled holes in tensile specimens.....	137
107	Layout of surface-drilled holes in tensile specimens.....	138
108	Photograph of laser-drilled surface holes showing damage around the holes.....	140
109	Biaxial disk with simulated surface holes. Fifty (50) holes of each size were produced	141
110	Sapphire reference standard for ultrasonic image calibration.....	143
111	Real-time microfocus X-ray system.....	144
112	Schematic of ceramic component inspection technique by projection microradiography using Feinfocus X-ray system.....	145
113	Optimization of X-ray image contrast for the inspection of MOR bars and biaxial disks.....	145
114	Schematic of 100 MHz acoustic microscopy system.....	147

LIST OF FIGURES (cont)

<u>Figure</u>	<u>Title</u>	<u>Page</u>
115	A 100 MHz acoustic imaging system developed at Allison for the inspection of ceramic specimens	148
116	Detection of 25 μm x 25 μm surface holes in MOR bars	150
117	Detection of 25-100 μm diameter and depth surface holes in the gage area of the button-head tensile specimens.....	151
118	Detection of 10 μm diameter holes in the gage area of the button-head specimen by surface wave acoustic microscopy (AM).....	152
119	Acoustic microscopy detected 25 μm surface holes in the biaxial disks.....	153
120	Acoustic microscopy detected micron internal hole 2 mm below the surface.....	154
121	Ultrasonic detected 50 μm diameter holes 14 mm below the surface.....	155
122	Acoustic C-span in age of the surface etched sapphire water. No image processing was performed. On magnification, Group 3, Element 6 line-pair 70 μm apart was resolved.....	156
123	Acoustic microscopy detected 223 micron apart line-pair 2 mm below the surface in a sapphire standard	157
124	Acoustic microscopy resolved Group 1, Element 6 line-pair 4 mm below the surface. The line-pair was 280 micron apart	158
125	Schematic of a third generation CT system at ARACOR.....	160
126	Layout of reference standard with varying size and spaced holes	161
127	Computer tomography Aracor Tomosope. Detected and resolved 500 μm diameter holes	162
128	Layout and size of reference specimen after machining	162
129	Detection of 100 μm diameter holes by Tomoscope at Aracor	163
130	Detection of 50 μm diameter holes by Tomoscope at Aracor.....	164
131	Detection of 25 μm diameter holes by Tomoscope at Aracor.....	165
132	Photograph of a Chalk River Laboratory CT scanner	166
133	Minimal defect size calculated for a cylindrical defect detectable in a CT scan with rectangular voxels ($0.25 \times 0.25 \times 1 \text{ mm}^3$) as a function of the image noise. Defects are air cylinders (>1 mm long) in a silicon nitride matrix. Scan resolution is consistent with the pixel size. η defines the signal-to-noise ratio. As explained in the text (Section 5.2), black dots mark defects clearly observed in CT scans, crossed-detected with some uncertainty, and open dots - not detected.....	169
134	Same as Figure 133, but for a voxel of $0.125 \times 0.125 \times 1 \text{ mm}^3$	169
135	Depicted are: (a) CT scan of two ceramic blocks, with series of 250 μm and 100 μm holes (pixel size 125 μm , image noise 5%. Density profile along horizontal line crossing); (b) 250 μm holes, and (c) 100 μm holes. Horizontal scale is in pixels. All holes are clearly distinguished.....	171
136	Depicted are: (a) CT scan of two ceramic blocks with penes of 50 μm and 100 μm holes. (pizel size 125 μm , image noise 1.5%); (b) detectability of 50 μm holes (other holes appeared clustered); and (c) density profile along horizontal line crossing 50 μm holes Pixel 8 representing one hole. Pixel 12-16 show clustered holes	172

LIST OF FIGURES (cont)

<u>Figure</u>	<u>Title</u>	<u>Page</u>
137	Calculated maximal pixel size for defect detection of a 3-D defect in a CT image with rectangular voxels ($\Delta t \times \Delta t \times 1$ mm). Defects are spherical inclusions in a silicon nitride matrix. Defect material as specified (iron, air, iron oxides). σ is the image noise. The signal-to-noise ratio η between 2 and 4 was assumed to provide the useful range in a range of CT scanners.....	174
138	Calculated maximal pixel size for defect detection of a 3-D defect in a CT image with cubic voxels ($\Delta t \times \Delta t \times 1$ mm). Defects are spherical inclusions in a silicon nitride matrix. Defect material as specified (iron, air, iron oxides). σ is the image noise. The signal-to-noise ratio η between 2 and 4 was assumed to provide the useful range in a range of CT scanners.....	176
139	PY-6 silicon nitride MIL-B MOR bar fast fracture Weibull modulus based on surface failures.....	180
140	Comparison of predicted FF data fit versus experimental results with PY-6 Type-B MOR specimens with surface failures using the consolidated Weibull modulus of 11.97.....	182
141	Results of mixed mode room temperature fast fracture testing of PY-6 compressively loaded diametrical through-cracked disks used to determine both the Mode I and shear fracture toughness.....	184
142	Results of mixed mode room temperature fast fracture testing of a PY-6 Vicker's indented Type-B MOR bars used to determine both the Mode I and the shear fracture toughness for part through-cracks.....	189
143	Prediction of PY-6 button-head tensile specimen SCG static fatigue behavior at 1200°C using dynamic fatigue test material constants.....	193
144	Comparison of PY-6 button-head tensile specimen SCG static fatigue behavior at 1200°C with predictions made using the static fatigue model along with material constants obtained from both static and dynamic test.....	195
145	Predicted versus observed stress rupture failure time for PY-6 button-head tensile bars that failed at elevated temperature.....	201
146	Predicted versus observed creep rates fro PY-6 button-head tensile bars that failed from stress rupture at elevated temperatures.....	202
147	Comparison of predicted and observed creep results for PY-6 button-head tensile bars based on the application of the Monkman-Grant theory to elevated temperature rupture.....	202
148	Predicted versus observed room temperature fast fracture strength of PY-6 Type-B MOR bars after exposure to oxygen at 1400°C.....	207
149	Predicted versus measured PY-6 Type-B MOR bar oxide thickness after exposure to air at elevated temperatures.....	208
150	PY-6 biaxial fast fracture room temperature ball-on-ring confirmatory test prediction of surface failure probability versus experimental results.....	216
151	NT154 biaxial fast fracture room temperature pressurized flat circular plate confirmatory test.....	224

LIST OF FIGURES (cont)

<u>Figure</u>	<u>Title</u>	<u>Page</u>
152	NT154 biaxial fast fracture room temperature spin disk internal failure confirmatory test.....	231
153	PY-6 button head tensile specimen slow crack growth model prediction versus stress rupture tensile specimen data from 1200°C to 1300°C.....	234
154	Comparison of predicted and observed slow crack growth behavior of PY-6 ball-on-ring flat circular disks tested at 1200°C and 1300°C.....	236
155	NT154 biaxial SCG spin disk surface failure confirmatory test at 1204°C.....	245
156	NT154 button-head tensile specimen slow crack growth model prediction versus measured stress rupture tensile data from 1315 to 1400°C	249
157	Comparison of observed NT154 1371°C spin disk lives with both the mean life and the 5% failure life predicted from SCG methodology and the CARES program.....	251
158	Comparison of NT154 1371°C spin disk SCG time zero effective strength versus predicted fast fracture strength. Test results lie within the predicted 90% confidence band.....	252

LIST OF TABLES

<u>Table</u>	<u>Title</u>	<u>Page</u>
1	Chemical analysis of powders and blended compositions.....	3
2	Test specimens of PY-6 silicon nitride fabricated for materials characterization and data base generation	4
3	Analysis of failure origin of MOR bars.....	7
4	Analysis of failure origin of button-head monotonic tensile specimens.....	18
5	Average dynamic fatigue behavior of MOR specimens characterized in air at Allison.....	23
6	Average dynamic fatigue behavior of MOR specimens characterized at ORNL	28
7	Average dynamic fatigue behavior of button-head tensile specimens.....	38
8	Dynamic fatigue behavior of button-head tensile specimens.....	39
9	Dynamic fatigue behavior of dogbone tensile specimens characterized at NIST.....	54
10	Creep rupture characterization of button-head specimens at SoRI.....	60
11	Room temperature residual strength of uniaxial creep specimens that did not fail during testing.....	67
12	Summary of TEM and EDS results of PY-6 creep specimens	76
13	Creep rupture characterization of dogbone tensile specimens at NIST	89
14	Average oxidation behavior of PY-6 silicon nitride in air.....	100
15	Fast fracture biaxial strength of PY-6 silicon nitride circular disks at 25°C	111
16	Fast fracture strength of MOR specimens machined from circular disks	115
17	Fast fracture MOR strength of transverse ground Type B specimens at room temperature.....	116
18	Summary of diametral compression experiments at 25°C.....	119
19	Summary of diametral compression fast fracture testing at elevated temperatures	122
20	Strength of Vicker's indented surface cracked silicon nitride MOR bars at 25°C	123
21	Biaxial creep behavior of PY-6 silicon nitride at 1200°C	128
22	Biaxial creep rupture behavior of PY-6 silicon nitride at 1300°C.....	129
23	Biaxial creep rupture behavior of PY-6 silicon nitride at 1400°C.....	133
24	Dimension and layout of laser-drilled holes in MOR bars.....	139
25	Detection of surface holes by microfocus X-radiography	146
26	Detaching of surface holes by acoustic microscopy.....	149
27	The calculated minimal defect size for cylindrical voids in a silicon nitride matrix, detectable in a CT scan with rectangular voxels ($0.250 \times 0.250 \times 1 \text{ mm}^3$) and image noise σ . The cylinder length is more than 1 mm. Scan resolutions is consistent with the pixel size, η defines the signal-to-noise ratio.....	168
28	The calculated minimal defect size for cylindrical voids in a silicon nitride matrix, detectable in a CT scan ($0.125 \times 0.125 \times 1 \text{ mm}^3$) and image noise σ . The cylinder length is more than 1 mm. Scan resolution is consistent with the pixel size, η defines the signal-to-noise ratio	168

LIST OF TABLES (cont)

<u>Table</u>	<u>Title</u>	<u>Page</u>
29	Summary of the experimental data for CT scans performed on ceramic specimens with laser-drilled holes (cylindrical voids). The voxel size was $0.125 \times 0.125 \times 1 \text{ mm}^3$, image noise σ as specified. Three columns list defects clearly detected (1), detected with some uncertainty (2), and not detected (3) in a CT image. Scan 1275 was measured using a thinner slice (about 0.5 mm), and this measurement is not included in a graph verifying calculated defect detectability	173
30	Summary of the experimental data for CT scans performed on ceramic specimens with laser-drilled holes (cylindrical voids). The voxel size was $0.25 \times 0.25 \times 1 \text{ mm}^3$, image noise s as specified. Three columns list defects clearly detected (1), detected with some uncertainty (2), and not detected (3) in a CT image	173
31	The calculated minimal defect size for a 3-D defect detectable in a CT scan with cubic voxels ($0.25 \times 0.25 \times 0.25 \text{ mm}^3$) and image noise σ . Scan resolution is consistent with the pixel size. The 3-D defect is a void or inclusion in a silicon nitride matrix. Defect material as specified. Data are for the signal-to-noise ratio η equal to 3.....	175
32	The calculated minimal defect size for a 3-D defect detectable in a CT scan with cubic voxels ($0.125 \times 0.125 \times 0.125 \text{ mm}^3$) and image noise σ . Scan resolution is consistent with the pixel size. The 3-D defect is a void or inclusion in a silicon nitride matrix. Defect material as specified. Data are for the signal-to-noise ratio η equal to 3.....	175
33	PY-6 Type-B MOR bar fast fracture data summary	178
34	For PY-6 Type-B MOR bar, a comparison of the consolidated and individual temperature surface Weibull fast fracture material constants.....	183
35	Summary of PY-6 room temperature Brazilian cracked compressive disk test results.....	184
36	Tabulation of constants S_i and T_i used in computing fracture toughness of a notched disk.....	185
37	Equations to computer A_i and B_i used to determine the fracture toughness of a through-cracked disk; $S = \sin (\text{Ang})$ $C = \cos (\text{Ang})$	186
38	Fast fracture bend strength of indented room temperature PY-6 bars.....	188
39	A comparison of PY-6 dynamic fatigue SCG material constants obtained using differing specimens and test facilities.....	192
40	Initial strength of PY-6 specimens tested in dynamic fatigue	193
41	A comparison of PY-6 1200°C SCG material constants obtained from static and dynamic testing of button-head tensile specimens.....	194
42	Creep rupture of PY-6 button-head tensile bars tested by Southern Research Institute.....	200
43	A comparison of PY-6 Type-B MOR bar room temperature fast fracture strength after temperature exposure with the predicted strength .	207
44	A comparison of PY-6 Type-B MOR bar predicted oxide thickness with the measured value.....	208

LIST OF TABLES (cont)

<u>Table</u>	<u>Title</u>	<u>Page</u>
45	A comparison of PY-6 Type-B MOR bar room temperature fast fracture strength with the predicted strength after exposure to oxygen at elevated temperatures.....	209
46	A summary of the multiaxial slow crack growth and fast fracture verification tests for PY-6 and NT154 materials.....	212
47	A summary of the room temperature PY-6 circular flat plate ball-on-ring fast fracture surface test results.....	213
48	Comparison of the room temperature PY-6 ball-on-ring predicted surface characteristic strength with the measured value.....	214
49	Room temperature surface fast fracture strength of PY-6 MOR specimens cut from ball-on-ring flat circular plates.....	217
50	Room temperature NT154 fast fracture material constants obtained from E-bar, Type-B, and tensile button-head surface failures	218
51	Room temperature NT154 surface fast fracture strength from MOR testing of E-bars.....	219
52	Room temperature NT154 surface fast fracture strength from Type-B MOR testing	220
53	Room temperature NT154 surface fast fracture strength from button-head tensile specimens.....	221
54	Room temperature NT154 surface fast fracture test results obtained from flat circular plates loaded with an increasing uniform pressure until failure.....	222
55	Room temperature NT154 pressurized flat plate surface characteristic strength predicted from uniaxial Weibull test and the PIA methodology	223
56	Room temperature fast fracture strength of NT154 spin disks.....	224
57	Dimensions and load spans of two types of NT154 fast fracture MOR bars.....	225
58	Summary of NT154 room temperature E-bar MOR fast fracture strength....	226
59	Summary of NT154 room temperature 4-inch bar MOR fast fracture strength.....	228
60	NT154 room temperature fast fracture material properties for internal failures of MOR specimens	229
61	NT154 room temperature spin disk internal stresses and computer FF failure probability at 50,000 rpm.....	229
62	Comparison of NT154 room temperature spin disk internal fast fracture experimental and predicted failure probabilities	230
63	Comparison of NT154 room temperature spin disk surface fast fracture experimental and predicted failure probabilities	232
64	PY-6 1200°C SCG static surface strengths of flat circular plates loaded by a ball-on-ring fixture.....	232
65	PY-6 1200°C SCG static fatigue test results of button-head tensile specimens with surface failures.....	234
66	PY-6 1260 and 1300°C SCG static fatigue test results of button-head tensile specimens with surface failures.....	235
67	PY-6 1300°C SCG static surface strengths of flat circular plates loaded by a ball-on-ring fixture.....	237
68	NT154 button-head tensile fast fracture specimen strength at 1204°C.....	238

LIST OF TABLES (cont)

<u>Table</u>	<u>Title</u>	<u>Page</u>
69	NT154 1204°C spin disk fast fracture test results.....	239
70	Comparison of NT154 1204°C spin disk surface stresses obtained from NASTRAN and back predicted from Equation 99.....	240
71	NT154 button-head tensile SCG stress rupture internal failure results at 1149 and 1204°C	241
72	Predicted surface SCG probability of survival of NT154 1204°C spin disks using CARES PIA methodology.....	242
73	Predicted surface values of NT154 1204°C spin disk fast fracture characteristic strength and SCG coefficient from CARES PIA analysis	243
74	Applied load spectrum for four NT154 spin disks which failed on the surface in SCG at 1204°C.....	244
75	Predicted failure probability for four NT154 spin disks which failed on the surface in SCG at 1204°C.....	244
76	NT154 spin disk 1371°C surface static failure data used to verify SCG methodology	246
77	NT154 Kic values obtained from notched MOR bar tests at 1204°C and 1371°C.....	246
78	Fast fracture volume strength of NT154 button-head tensile specimens at elevated temperature	247
79	Stress rupture failure data for NT154 button-head tensile bars used to determine SCG constants at 1371°C.....	248
80	Predicted survival probability using CARES of 5 NT154 spin disks which failed on the surface at 1371°C from SCG.....	250
81	NT154 spin tested at 1371°C and failed on the surface have lives that are roughly bounded by the mean and 5% failure probability lives predicted by CARES using the SCG methodology.....	251
82	Effective time zero stresses, calculated from CARES results for NT154 spin disks at 1371°C, for comparison with fast fracture results	252
83	Confirmatory test correlation summary.....	257

LIFE PREDICTION METHODOLOGY FOR CERAMIC COMPONENTS OF ADVANCED VEHICULAR HEAT ENGINES – FINAL REPORT

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ABSTRACT

One of the major challenges involved in the use of ceramic materials is ensuring adequate strength and durability. This activity has developed methodology which can be used during the design phase to predict the structural behavior of ceramic components. The effort involved the characterization of injection molded and hot isostatic pressed (HIPed) PY-6 silicon nitride, the development of nondestructive evaluation (NDE) technology, and the development of analytical life prediction methodology. Four failure modes are addressed: fast fracture, slow crack growth, creep, and oxidation. The techniques deal with failures initiating at the surface as well as internal to the component. The life prediction methodology for fast fracture and slow crack growth have been verified using a variety of confirmatory tests. The verification tests were conducted at room and elevated temperatures up to a maximum of 1371°C. The tests involved 1) flat circular disks subjected to bending stresses and 2) high speed rotating spin disks. Reasonable correlation was achieved for a variety of test conditions and failure mechanisms. The predictions associated with surface failures proved to be optimistic, requiring reevaluation of the components' initial fast fracture strengths. Correlation was achieved for the spin disks which failed in fast fracture from internal flaws. Time dependent elevated temperature slow crack growth spin disk failures were also successfully predicted.

I. INTRODUCTION

The major goals associated with this effort are to develop and demonstrate the following:

- design methodology which can predict the useful life of ceramic components
- nondestructive examination (NDE) technology required to ensure material integrity
- ceramic material data base appropriate for use in the design of advanced vehicular heat engine components

In order to accomplish the goals, the following four major tasks are involved:

- material data base/material characterization
- development of NDE technology
- development of analytical life prediction models
- verification

The characterization of the PY-6 material involved room and elevated temperature tests using a variety of specimen types. The fast fracture testing utilized modulus of rupture (MOR), button-head tensile, and dog bone tensile specimens. Slow crack growth testing also utilized the three specimen types involved in the fast fracture testing. The creep/stress rupture testing employed the button-head and dogbone tensile specimens. Oxidation tests involved the use of MOR

bars exposed to elevated temperatures for an extended time period. A representative sample from each test series and specimen type was subjected to an intensive fractography investigation to ensure adequate knowledge of the failure modes and mechanisms.

NDE activity is centered on determining if a number of advanced inspection techniques can be used to ensure the integrity of ceramic components. As a result of the unique properties associated with ceramic materials, it is imperative that inspection techniques be developed to detect small defects. The techniques involve the use of ultrasonic imaging, microfocus radiography, and X-ray computed tomography. This effort involves the development of specimens (reference standards) containing premachined flaws of various sizes. The reference standards are used to optimize the inspection parameters for each inspection technique; this is accomplished by determining the smallest flaw that could be detected for a given set of inspection parameters.

The development of the analytical life prediction models builds upon the knowledge and information gained during the material characterization phase. A series of statistical analytical models has been utilized to address each of the four failure modes. Both uniaxial and biaxial models have been developed and correlated with the material test data. The fast fracture model is based on a Weibull distribution which is the current industry standard, except the proposed model utilizes a different distribution depending on specimen/component failure origin.

Separate durability models have been developed for slow crack growth and creep. Both models have been enhanced and time dependent probability of survival is predicted so the probability of failure increases with time. An improved oxidation model has also been developed. In addition to the life prediction models, a creep deformation model has been developed, coupling the creep rate to the stress rupture information. The life prediction models developed by this program have been incorporated into the NASA software, CARES.

The final phase involves additional tests to confirm the adequacy of the durability models and establishes suitable safety factors for use in component design. The verification tests utilized flat circular disks subjected to bending loads and high speed spin tests. The confirmatory tests were conducted at room and elevated temperatures up to a maximum of 1400°C. Pretest inspection of the PY-6 spin disks revealed that the material quality was unacceptable. The material supplier discontinued all efforts to develop and supply ceramic materials. Under these circumstances, an alternate method had to be located and involved the use of the material data base and confirmatory test results for the NT154 material. This information was obtained from the associate program conducted by the AlliedSignal Aerospace Company, Garrett Auxiliary Power Division. The methodology and correlations developed by this program have also been applied to the NT154 material.

II. MATERIAL AND SPECIMENS

2.1 MATERIAL SELECTION

The principal requirement of the Ceramic Life Prediction program was to select a material system which was a candidate material for the gasifier rotor application in the DOE funded ATTAP program at Allison. Allison selected the GTE Laboratories' PY-6 injection molded and HIPed silicon nitride, which was a leading candidate material and processing method when the program was first envisioned and initiated. The selection of the ceramic material was approved by Oak Ridge National Laboratory (ORNL) program management as required by the contract.

2.2 SPECIMEN FABRICATION

Five hundred kilograms of Ube silicon nitride powder, namely 300 kg of coarse E03 and 200 kg of fine E10, were acquired at the beginning of the program and reserved to eliminate lot-to-lot variation in the powder supply. Chemical analysis of the powders showed normal elemental content (Table 1). The surface area of the E10 powder was more than three times larger than

Table 1. Chemical analysis of powders and blended compositions

Characteristic	Ube E03	Ube E10	Powder ratios	
			80/20	60/40
O, wt %	0.93	1.31	1.006	1.082
C, wt %	<0.2	<0.2	<0.2	<0.2
Cl, ppm	<100	<100	<100	<100
Fe, ppm	<100	<100	<100	<100
Ca, ppm	<50	<50	<50	<50
Al, ppm	<50	<50	<50	<50
% beta, wt %	<5	<5	<5	<5
Surface area, m ² /gm	3.0	10.9	4.58	6.16

E03. Based on GTE's experience under the AGT 100 and the ATTAP program, the two powders were blended together in 80/20 (E03/E10) and 60/40 weight percentage ratio batches to assess the variation in the chemistry. Chemical analysis of two blends showed no change in the chemistry, including the iron content, when compared to the virgin powder chemistries (Table 1). The surface area of the 80/20 blend was measured to be 4.58 m²/gm and that of 60/40 was found to be 6.16 m²/gm. The majority of the test specimens were fabricated using the 80/20 powder blend. Some specimens were fabricated using the 60/40 ratio because of poor production yield of test specimens.

The blended powders were ball milled with 6 weight percentage yttria to a surface area of about 10 to 12 m²/gm. The milled powder was mixed with a binder, injection molded, and HIPed in an ASEA unit using GTE's optimized proprietary processing parameters to fabricate various type of test specimens. The Type-B modulus-of-rupture (MOR) bar was fabricated to near net shape and subsequently longitudinally machined and beveled by Bomas Specialty Machining of Massachusetts, using optimized machining parameters prior to shipment by GTE to Allison. The circular disk specimens were fabricated in near net shape. The flat surfaces were rough machined to a nominal thickness of 2.75 mm (0.110 in.) prior to shipment to Allison. Final surface preparation was done by Battelle Research Laboratory, Columbus, Ohio, prior to testing.

Extensive experimental efforts to fabricate near net shape button-head tensile specimen failed to provide a quality test specimen. The program switched to fabricating cylindrical rods from which tensile specimens of the desired geometry and surface finish were machined. Machining studies conducted at GTE indicated outside vendors were unable to control the quality of the surface finish and the cost of the machining was expensive. Therefore, GTE developed an in-house machining capability enabling GTE to control the quality of the machined specimen and its cost.

All the button-head tensile specimens were machined at GTE in this program. The rectangular billets were injection molded and HIPed to near net shape and delivered to Allison. Flat dogbone tensile specimens were fabricated from these billets by the National Institute of Science and Technology (NIST) to conduct creep rupture studies. The delivery of the four types of specimens was conducted in several lots, as shown in Table 2. Delivery of the MOR bars and the button-head tensile specimens was made in many lots which may have resulted in subtle variation in material and grain boundary composition, respectively. These variations can potentially cause large variability and scatter in the high temperature properties of the material. Figure 1 shows the four type of specimens and test conducted to generate the required data base for the development of the ceramic life prediction methodology.

Table 2. Test specimens of PY-6 silicon nitride fabricated for materials characterization and data base generation

Type of specimen	Lot number	Number of specimens	Powder composition	Total number of specimens
Type-B MOR bar, 50 x 4 x 3 mm	Q21-19	200	80/20	850
	Q21-90-34	110	80/20	
	Q21-90-42	240	80/20	
	Q21-90-64	250	80/20	
	80/20 ^a	25	80/20	
	60/40 ^a	25	60/40	
Circular disk, diameter = 31.75 mm thickness = 3.175 mm	Q21-90-120	70	80/20	140
	Q21-90-131	70	80/20	
Rectangular billets, 60 x 50 x 15 mm	One lot	20	80/20	20
Button-head tensile specimens ^b	Q21-16-91	35	80/20	650
	Q21-4-91	60	80/20	
	Q21-90-72	18	80/20	
	Q21-90-56	22	80/20	
	Q21-25-91	29	80/20	
	900326	5	80/20	
	900517	24	80/20	
	901210	10	80/20	
	910118	48	80/20	
	910121	48	80/20	
	910124	55	80/20	
	910125	51	80/20	
	910617	28	80/20	
	910211	22	80/20	
	900516	1	80/20	
	900725	7	80/20	
	900914	8	80/20	
	900919	5	80/20	
	910620	45	80/20	
	901009	12	80/20	
	901016	8	80/20	
	901017	5	80/20	
	910305	41	60/40	
	910307	37	60/40	
	910311	26	60/40	

a No lot number given by GTE Laboratories.

b Large number of tensile specimens delivered towards the closing time of GTE Laboratories structural ceramics business were distorted and unsuitable for testing.

- **MODULUS OF RUPTURE (TYPE-B)**
 - **FAST FRACTURE**
 - **FRACTURE TOUGHNESS**
 - **OXIDATION**
 - **SLOW CRACK GROWTH**
- **TENSILE BUTTON-HEAD SPECIMEN**
 - **FAST FRACTURE**
 - **SLOW CRACK GROWTH**
 - **CREEP RUPTURE**
- **TENSILE DOG-BONE**
 - **SLOW CRACK GROWTH**
 - **CREEP RUPTURE**
- **CIRCULAR DISK**
 - **THRU-CRACK CHEVRON NOTCH**
 - **BIAXIAL FAST FRACTURE STRENGTH**
 - **BIAXIAL CREEP RUPTURE**

Figure 1. Types of specimens and tests used to generate the data base required for the development of the ceramic life prediction methodology.

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III. DATA BASE GENERATION

3.1 FAST FRACTURE

3.1.1 Modulus-of-Rupture Flexural Strength

Fast fracture MOR strength of 320 specimens was measured from room temperature (RT) to 1400°C using the Type-B MOR bars, 3 mm x 4 mm x 50 mm, in 4-point bending at a cross-head speed of 0.508 mm/min. The inner loading span was 20 mm, while the outer loading span was 40 mm. The specimens were longitudinally machined by BOMAS Speciality, Waltham, Massachusetts, to a nominal surface finish of about 16 rms. All the MOR specimens were subjected to density and dimensional measurements and characterized by several NDE flaw detection methods, including visual (10x to 40x), fluorescent penetrant inspection (FPI), microfocus X-ray, and acoustic microscopy inspection prior to mechanical testing. The measured strength and number of specimens tested at each temperature is shown in Figure 2. Appendix I gives the details for all the specimens. The average RT strength of the material was 812.9 MPa with a standard deviation of 153.8 MPa. The average strength decreased to 744.5 MPa with a standard deviation of 73.6 MPa at 1000°C and to 603.7 MPa at 1100°C with a standard deviation of 90.9 MPa. The decrease in strength at 1100°C is believed to be due to the softening of the grain boundary intergranular glassy phase. The average measured strength at 1200°C was 605.7 MPa with a standard deviation of 94.2 MPa, whereas at 1300°C, the measured strength was 567.3 MPa with a standard deviation of 55.5 MPa. Apparently, the average strength from 1100°C to 1300°C did not change much indicating that the viscosity of the intergranular grain boundary phase did not vary in this temperature range. The average strength further reduced to 490.9 MPa at 1400°C with a standard deviation of 53.3 MPa due to increased viscous flow of the glassy grain boundary phase. The MOR strength data were analyzed using NASA/CARES data reduction and analysis algorithm. The two parameter Weibull modulus (m) and the characteristic strength (σ_0)

Figure 2. Flexural strength of PY-6 injection molded and HIPed silicon nitride material.

were estimated as a function of temperature using the maximum likelihood suspended item analysis method. Details of this work are discussed in the analytical methodology section of the report.

Failure analysis of all the specimens was conducted to ascertain the fracture origin, type of defect, and location. The data are summarized in Table 3. Fractographic analysis revealed that the failure at room temperature was predominantly controlled by chamfer (corner) and surface flaws, as shown in Figure 3. The bars that failed from the chamfer did not have strengths appreciably different from the bars that failed from surface defects. SEM examination of several chamfer failed specimens did not show any damage to the corner due to machining and the specimens appeared similar to the specimens that failed from surface flaws. Since the crack intensity factor (K_I) at the corner is higher than the flat tensile surface, subtle changes at the corner increase the probability of corner failures.

AlliedSignal under their Ceramic Life Prediction program observed similar problems with the cold isostatically pressed (CIPed) NT154 Type-B and Type-E longitudinally machined MOR specimens. AlliedSignal alleviated the problem by polishing the chamfer, which decreased the fracture controlling flaw size and increased the strength of the chamfer material. This allowed an increased number of failures on the surface which had a different surface finish, i.e. a differing flaw population. Allison decided early in the program not to follow this route so results were not biased, a problem plaguing the testing of new very high strength silicon nitride materials in flexure. Above 1000°C, the failure initiated both from surface and volume flaws (Figure 3[b] and 3[c]). Furthermore, X-ray energy dispersive analysis (XEDA) indicated that failure controlling inclusions contained iron. Erosion by hard silicon nitride powder of the steel components of the injection molding equipment may have contaminated the material with iron.

3.1.2 Uniaxial Button-Head Tensile Strength

Uniaxial (monotonic) tensile strength from RT to 1400°C was measured at Southern Research Institute (SoRI), Birmingham, Alabama. Fast fracture tensile evaluations were performed in a gas-bearing tensile system, a schematic of which is shown in Figure 4. The system used gas-bearing universals in the load linkage to prevent the introduction of unknown bending moments in the load train from cross-head motion or eccentricity and allowed monitoring of the straightness of the load train. Emphasis in the design of the load train was placed on large length-to-diameter ratios at each connection, close sliding fits of all the mating surfaces, elimination of threaded connections, and other factors essential to minimizing bending stresses. All the components were machined true and concentric to within 12.5 microns. The entire load train was regularly

Table 3. Analysis of failure origin of MOR bars.

Temperature—°C	No. of specimens	Surface		Volume			Average strength—MPa	Standard deviation—MPa
		SF	Inclusion	SFC	Pore	Inclusion		
RT	80	14	2	64	0	0	812.9	153.8
1000	20	7	0	9	1	3	744.5	73.6
1100	20	13	0	6	0	1	603.7	90.9
1200	88	45	7	21	3	12	605.7	94.2
1300	32	20	2	3	1	6	587.3	55.5
1400	80	53	1	21	4	1	465.6	53.3

Figure 3. Fractography of fast fracture tested MOR specimens tested at (a) room temperature and (b) 1400°C.

Figure 4. (a) Schematic of the gas bearing tensile facility and (b) close-up view of pull rods with the button-head specimen and heating elements.

checked by spinning a circular alignment rod specimen, machined to within 12.5 microns, between the lower and upper loading rods (Figure 5). The gas bearings were adjusted until the total indicated runout (TIR) was less than 75 micron at any position around the circumference of the rod. The precision machined tensile grip used a three-piece split collet assembly, and the deformation of the specimen gage length from RT to 1400°C was measured using two-clip-on extensometers 180 deg apart. The electrical outputs of the two extensometers were averaged. The clip-ons were mounted on special flags clamped to the specimen using a specially machined gage block with a specified initial separation. The clip-ons were calibrated over the expected range of deformation by means of a supermicrometer. The load and deformation data were recorded on a calibrated X-Y chart recorder.

The button-head specimen geometry was empirically designed and experimentally evaluated to ensure gage failures. Based on the SoRI experience of measuring the tensile strength of strong silicon nitride materials, three button-head specimens (Figure 6) were machined from injection molded and HIPed cylindrical blank rods of GTE PY-6. The specimen length was nominally 180 mm long with a 63.5 mm gage length and diameter of 5.588 mm. The specimen gage was longitudinally machined to a surface finish of 6 to 16 rms, while the grip area was machined to a surface finish of 16 to 32 rms. One specimen was strain gaged 120 deg apart with three strain gages to assess the bending stresses during testing. The results show the specimens were very well aligned and the bending was less than 5%. The average tensile strength of this material was measured to be 302 MPa (Figure 7). Two specimens failed in the head and one failed from a surface defect which was unacceptable. The head grip area-to-gage area ratio of this specimen was around 6.25, which appears to be low for the material and surface finish used. Failure analysis of the fracture surface area did not reveal any major problems with the machined surface. The extreme sensitivity of this material to minute and random surface variations necessitated further reducing the gage diameter and increasing the head-to-gage area ratio. The specimen design and dimensions were modified so the new ratio was around 7.69 (Figure 8). Five cylindrical rods of PY-6 material were CIPed, HIPed, and button-head specimens were machined to fabricate the new specimen geometry and assess its integrity. Four specimens failed in the gage with an average strength of 646.8 MPa (93.8 ksi), and one specimen was accidentally damaged while loading.

The results indicated that the new specimen design would obtain gage failure. It was also revealed that the PY-6 material is inherently strong if iron inclusions and other processing defects can be eliminated. The specimen design shown in Figure 8 was used to fabricate all the button-head tensile specimens characterized during this program to measure both the fast fracture and creep rupture behavior of the PY-6 material. The nominal dimensions of the specimens were 137.2 mm length with a gage section of 55.9 mm length and 4.76 mm diameter. The nominal surface finish was 6 to 9 rms.

Concentricity and runout of each tensile specimen were checked at SoRI using loading rods, collet, and specimen assembly in a specially designed rig. The total length of the assembly was around 406 mm. The bottom pull rod/specimen was first mounted horizontally with one end of the assembly being spun while the runout was measured on the other end by a dial indicator (Figure 9[a]). Thereafter, the top pull rod was attached to the second end of the specimen mounted vertically, spun, and top nut adjusted until the assembly TIR was less than 125 micron (0.005 in.) (Figure 9 [b]). At high temperatures, the gage section described earlier was resistively heated using silicon carbide elements developed at SoRI¹ and was successfully used up to 1650°C in air. The temperature gradient from the center of the gage section to its two ends, ± 27.95 mm, was within $\pm 5^\circ\text{C}$ in the worst case at 1400°C. The loading pull rods were also heated to about 850°C to minimize the temperature gradient across the gage length of the specimen.

Two hundred sixty-two button-head PY-6 HIPed silicon nitride specimens were characterized at a loading rate of 4137 MPa/min (600 ksi/min) from RT to 1400°C. The fast fracture tensile strength of an advanced ceramic is commonly measured in a stressing rate mode², and therefore this was used in the present work. The fast loading rate minimized any oxidation or other time

Figure 5. Setup for load train alignment in a gas bearing tensile machine.

TE94-2390

Figure 6. Preliminary button-head tensile specimen geometry.

dependent effect on the strength of the material. Density and dimensional inspection, as well as NDE (visual, FPI, ultrasonic velocity, microfocus radiography, and acoustic microscopy) inspection, of each specimen was conducted prior to testing.

The button-head diameter is 15.24 mm and the gage diameter is 4.76 mm. Microfocus radiographs were obtained at 2x for both areas with the following experimental parameters:

- Kv = 75
- mA = 0.3
- exposure in the gage = 200 seconds
- exposure in the button-head = 300 seconds

Each location was inspected at 0 and 90 deg, resulting in four radiographs per specimen, providing complete characterization of each specimen, and increasing the probability of detecting fracture controlling flaws which may have irregular characteristics and orientation. Analysis of the radiographs revealed that 63 micron defects were detected in the gage area. Specimens were also examined by surface wave acoustic microscopy at 50 MHz using F#0.8 transducer and scan plan developed using reference standard with laser-drilled surface holes.

The average density of the specimens via Archimedes method was measured to be 3.2596 gm/cc with a standard deviation of 0.0081 gm/cc. The average longitudinal ultrasonic velocity was measured to be 1.0655 ± 0.0158 cm/ μ sec using the time-of-flight method. No correction for acoustic diffraction was made. The surface finish ranged between 9 to 18 rms, compared to the specification of 6 to 9 rms per drawing. Machining chatter caused the surface to be rough on some specimens, but no specimen was deleted from the test matrix. The gage diameter of each of the 120 specimens was measured at three locations, namely in the center and near the two extreme ends of the gage length. In general, the measured diameter was $4.76 \text{ mm} \pm 0.025 \text{ mm}$. Some specimens did show taper toward the radius, indicating undercut. The concentricity of each specimen and loading rod assembly was measured to be within 75 microns, which was well within the 250

Figure 7. Composite plot of GTE PY-6 silicon nitride tensile stress-strain responses from three strain gages located 120 deg apart on specimen TN-AX-1.

microns allowable over the entire length of the assembly. In other words, the bending stresses in the loading system were very low.

The measured fast fracture strength from RT to 1400°C is depicted in Figure 10. Appendix II shows the compilation of the data for all the specimens tested. Stress-strain behavior of each specimen was recorded at all the temperatures, and Figure 11 shows the specimens tested at RT and 1400°C. The stress-strain behavior from RT to 1300°C was linear, whereas at 1400°C the material exhibited nonlinear plastic behavior. This is primarily due to the softening and void formation at the grain boundary phase (Figure 12). The average room temperature tensile strength was measured to be 414.4 MPa with a standard deviation of 60.04 MPa. The average strength at 1000°C and 1100°C degraded to 379.70 MPa with a standard deviation of 115 MPa and to 313.70 MPa with a standard deviation of 64.8 MPa, respectively. The average strength at 1200°C was found to be 311.20 MPa with a standard deviation of 61 MPa, and at 1400°C, the average strength degraded to 301.5 MPa with a standard deviation of 71.80 MPa. The average increase at 1300°C may be an aberration because of the small number of samples tested. The most probable strength between 1100°C to 1400°C is nominally about 315 MPa. The tensile strength data were analyzed using NASA/CARES data reduction and analysis algorithm. The two parameter Weibull modulus (m) and the characteristic strength (σ_0) were estimated as a function of temperature using the maximum likelihood suspended item analysis method. Details of this work are discussed in the analytical methodology portion of the report.

Notes:

1. Diameters true and concentric to ± 0.0127 mm
2. Do not undercut radius at tangent points
3. Both ends to be flat and perpendicular to ± 0.0127 mm
4. Axial grind gage and radius together with 152.4 mm ϕ wheel
5. All dimensions are in millimeters
6. Tolerances unless otherwise noted: decimals ± 0.0254 mm, on diameters ± 0.0508 mm, on lengths ± 0.0508 mm

TE92-3969

Figure 8. Schematic of button-head tensile specimen.

Figure 9. Alignment of specimen/pull rod assembly.

Figure 10. Uniaxial tensile strength of button-head PY-6 injection molded and HIPed silicon nitride specimens from room temperature to 1400°C.

Figure 11. Stress-strain behavior of button-head specimen at room and elevated temperatures.

TE92-3976A

Figure 12. Fast fracture uniaxial testing at 1400°C showing nonlinear stress-strain behavior and grain boundary cavitation.

Optical micrography and failure analysis (20x to 40x) of all the specimens was conducted. A limited number of specimens were examined by scanning electron microscopy (SEM) including XEDA. Table 4 summarizes the average strength and flaw characteristics (location, type, and number) of failure controlling defects at each temperature. Over 93% of the specimens failed within the 55 mm gage section of the specimen. The distribution along the gage between the top half and the bottom half is shown in Figure 13. The random distribution of the failures along the entire gage length clearly demonstrates that the tensile testing methodology is sound and provides a robust reliable data base reflecting true behavior of high strength silicon nitride ceramics. At room temperature 80% of the specimens failed from both the volume pores and inclusions of iron base compounds. The room temperature strength of the CIPed and HIPed PY-6 silicon nitride was 646.8 MPa, compared to 414.4 MPa for injection molded and HIPed material. This is a drop of 36% in strength due to the presence of iron inclusions. Singh et. al.³ have systematically shown that addition of 0.5 to 5 wt % Fe inclusions in Si_3N_4 with 6% yttria causes a 15 to 40% decrease in the strength of the material, which is similar to the drop observed in the present study. Failures were observed to originate from iron inclusions rich in silicon or from regions of Si_3N_4 matrix degraded as a result of reaction between Si_3N_4 and molten iron. Furthermore, Singh et. al. reported a nearly 50% drop in the fracture toughness of silicon nitride matrix at the failure site, which is consistent with the results of Baumgartner and Richerson⁴. Singh et. al.³ also reported the effective flaw size to be much larger than the measured defect size. It is apparent the presence of iron impurity is very detrimental to the performance of the ceramic material and components. At higher temperatures, the failure was mixed between surface and volume flaws except at 1200°C. About 70% of the specimens failed from surface defects and 30% from volume flaws at 1200°C. Failure at RT predominantly was initiated from internal inclusions of iron (Figure 14). Another specimen showed the presence of iron, nickel, and chrome indicating the abrasion of process equipment components by a hard silicon nitride powder (Figure 15), and similar failures were observed at other temperatures. In addition, the stress-strain behavior was nonlinear at 1400°C even though the specimen failed in less than 10 sec (Figure 12). Fractography revealed that grain boundary cavities were formed during the short duration of testing

Table 4. Analysis of failure origin of button-head monotonic tensile specimens

Temperature—°C	Average strength—MPa	Standard deviation—MPa	No. of specimens	Surface failures		Volume failures		
				Machining defect	Inclusion	Inclusion	Pore	Other
RT	414.40	60.04	70	5	2	45	6	2 II (O) 10 BHF
1000	379.40	115.00	20	9	1	7	3	
1100	313.70	64.80	20	12	2	2	4	
1200	311.20	61.00	75	50	3	16	6	
1300	383.90	53.70	20	4	0	10	6	
1400	301.50	71.80	57	18	7	20	9	3 SF (O)

II = internal inclusion

IP = internal pore

SF = surface failure from machining defects

SI = surface inclusion

(O) = outside gage in the transition region

BHF = button-head failures

TE94-2394

Figure 13. Distribution of gage length failures in button-head specimens.

VS91-1608
TE94-2395

Figure 14. Fractography of button-head specimen failed at room temperature.

VS91-1607
TE92-428

Figure 15. SEM fractography of room temperature tested tensile specimen failed from internal iron inclusion containing nickel and chrome.

(Figure 12). This clearly indicates that this material may be unsuitable for usage at 1400°C where the component undergoes severe thermomechanical loads during transient conditions in gas turbine engines.

3.2 SLOW CRACK GROWTH

Structural ceramic materials are susceptible to slow crack growth (SCG) at elevated temperatures due to preexisting flaws induced during the various stages of the processing cycle. Currently available high strength silicon nitride materials primarily fail from machining induced surface and near-surface damage causing cracks, porosity, and inclusions. Classically, these flaws grow as a function of time at a given temperature under a given applied stress intensity (K_I) and cause failure once a single flaw reaches the critical stress intensity (K_{IC}). However, the environmental effects such as oxidation can accelerate the SCG process and cause failure prior to reaching the K_{IC} value. The SCG failure process, therefore, is time, temperature, stress, and environmentally dependent and effects lifetime predictions of structural ceramic components.

The nature of the SCG behavior in ceramic materials can be described by the crack velocity (V) equation:

$$V = A(K_I)^N \quad (1)$$

where V is the crack velocity, K_I is the stress intensity factor, A is a constant, and N is the crack velocity exponent. The parameter N can be used as a measure of material sensitivity to SCG and can be established from dynamic and static fatigue experiments.

In the dynamic fatigue experiments, the fracture stress (σ_f) is measured as a function of stressing rate $\dot{\sigma}$ at a given temperature. The slope of the line deduced from the linear regression on a log-log plot of σ_f versus $\dot{\sigma}$ is used to determine the value of N from Equation 2:

$$\sigma_f = C \dot{\sigma}^{(1/(N+1))} \quad (2)$$

where $C = [B(N+1)\sigma_i^{N-2}]^{1/(N+1)}$, $B = 2/[(N-2)AY^2K_{IC}^{N-2}]$, A and Y are material and environment constants, K_{IC} is the critical stress intensity factor, and σ_i is the fracture strength in an inert environment.

In the static fatigue experiments, the time to failure (T_f) is measured as a function of the applied stress σ_a . From Equation 3-1 it can be derived that

$$T_f = C\sigma_a^{-N} \quad (3)$$

A detailed theoretical treatise behind the SCG failure mechanism, data reduction/analysis, and interpretation is described in Section 5.

3.2.1 MOR Dynamic Fatigue Evaluation

Flexural, type B, 3 x 4 x 50 mm rectangular, longitudinally machined specimens were characterized both at Allison and ORNL. The density and dimensions of all the specimens were measured, and the specimens were examined using several NDE methods including visual, dye penetrant, microfocus X-ray, and acoustic microscopy prior to testing.

3.2.1.1 Flexural Dynamic Fatigue Testing at Allison. Allison tested 120 MOR specimens from 1000°C to 1400°C at cross-head speed ranging from 0.508 mm/min to 0.00508 mm/min. The standard fast fracture strength of the MOR specimens was measured at 0.508 mm/min and was the baseline cross-head speed for all comparisons. Specimens were tested in 1/4 point loading with a 40 mm outer span and a 20 mm inner span. Testing was conducted using a two-post MTS servo-hydraulic mechanical testing system. The load train consisted of a load cell connected to a cooling jacket/mullite assembly loading ram, a silicon nitride four-point fixture firmly resting on a mullite tube mounted in a metal fixture and plate bolted to the bottom actuator. A thermocouple was mounted from the side of the box furnace to maintain the desired temperature. A load displacement curve for each specimen was recorded and analyzed to calculate the stressing rate. Fracture strength and the stressing rate were subsequently used to determine the value of the crack velocity exponent N . The data analysis and consolidation methodology are discussed in Section 5.

Table 5 summarizes the results of the dynamic fatigue specimens characterized at Allison including the average measured strength, the calculated stress rate at each of the three cross-head speeds, and the failure analysis. The details of the individual specimens are given in Appendix III. The average strength at 1000°C with a cross-head speed of 0.0508 mm/min was 731.6 MPa (SD = 91.3 MPa); this decreased to 605.2 MPa (SD = 71.4) with a cross-head speed of 0.00508 mm/min, a drop of about 17% in strength. At 1200°C, the average strength decreased from 612.7 MPa (SD = 72.0 MPa) to 529.5 MPa (SD = 54.2 MPa) at the above mentioned cross-head speeds, a change of about 14%. Furthermore, at 1400°C, the average strength decreased about 22% over the same test conditions. Figure 16 shows the power law curve fit using Equation 2 at each temperature. The strength decreased both as a function of temperature and stressing rates. The parallelism of the individual fitted lines indicate a similar failure mechanism over the test temperature range which readily allows for the combination of the data at the various temperatures to calculate an overall SCG exponent N . The deviation at 1200°C may be just due to the sample population.

Figure 16. Dynamic fatigue behavior of MOR specimens tested at Allison.

Table 5. Average dynamic fatigue behavior of MOR specimens characterized in air at Allison

Temperature—°C	No. of specimens	Stroke rate—mm/min	Stress rate—MPa/sec	<u>Failure strength</u>		<u>Standard deviation</u>		Failure time—sec	Fractography results
				ksi	MPa	ksi	MPa		
1000	11	0.508	14.51	106.08	731.6	13.26	91.3	50	SFC(7), SF(2), IP(1), II(1)
	10	0.0508	1.55	102.44	704.6	16.31	113.9	455	SFC(5), SF(5)
	11	0.00508	0.20	87.77	605.2	10.36	71.4	3117	SFC(7), SF(3), SI(1)
1200	18	0.508	14.03	89.03	612.7	10.43	72.0	44	SFC(6), SF(9), IP(1), II(2)
	9	0.0508	1.55	89.48	617.0	10.43	74.5	398	SFC(2), SF(4), II(2), UI(1)
	8	0.00508	0.089	76.80	529.5	7.85	54.2	6071	SFC(2), SF(3), SI(1), II(2)
1300	12	0.508	12.22	82.05	568.0	10.09	70.3	47	SFC(2), SF(5), SI(1), II(3), IP(1)
	6	0.00508	0.048	56.03	386.3	5.29	36.4	8640	SF(6)
1400	17	0.508	10.62	64.09	443.7	3.81	24.2	42	SF(15), II(1), IP(1)
	8	0.0508	0.963	54.56	375.8	3.09	21.5	415	SFC(6), SF(2)
	10	0.00508	0.035	45.73	347.4	4.34	102.8	10482	SFC(4), SF(6)

SFC = surface machining induced flaw in the corner

SF = surface machining flaw

SP = surface pore

SI = surface inclusion

II = internal inclusion

IP = internal pore

UI = unidentified or lost origin

Failure analysis of all the specimens was conducted using optical microscopy to determine the type, location, and coordinate of the failure controlling defect (Appendix III). Table 5 summarizes the results at each temperature and stressing rate. A large number of failures occurred from the surface flaws at the corner of the specimens for all temperatures and stressing rates with similar fracture surface features. SEM analysis of the limited number of specimens was conducted. Figure 17 is a SEM of a specimen tested at 1000°C and a 0.20 MPa/sec stressing rate depicting the SCG area and softening of the glassy phase responsible for the decrease in the strength of the material. No permanent deformation of the bars was observed at these conditions. At 1200°C, failure at a nominal stress rate of 1.55 MPa/sec occurred from internal and surface flaws with no clear sign of a SCG region (Figure 18). At the same temperature, however, a dark ring structure with a lighter center region was observed at the average lowest stressing rate of 0.089 MPa/sec. This structure in PY-6 material has been attributed by Wereszczak et. al.⁵ to stress assisted corrosion, which developed into cracks causing premature failure of the material (Figure 19). At 1400°C, however, surface pitting and oxidized regions were observed which substantially decreased the strength of the material (Figure 20). Visual analysis of the MOR bars did not indicate any permanent deformation in the specimens at any condition. Further examination by an optical comparator revealed specimens tested at 1400°C and a 0.035 MPa/min stressing rate exhibited only minor curvature, indicating the specimen crept during testing. The creep resulted in an irreversible plastic deformation of the material which shifted the neutral axis of the MOR bar. Furthermore, some discoloration was observed on the fracture surface of several specimens tested at these conditions (Figure 21).

3.2.1.2 Flexural Dynamic Fatigue Testing at ORNL. Dynamic fatigue testing was conducted at ORNL, under a user agreement with HTML, in four-point flexure using 3 x 4 x 50 mm longitudinally machined MOR specimens. The specimens were tested with an outer span of 40 mm and an inner span of 20 mm using alpha-silicon carbide fixtures at 1000°C, 1200°C, and 1400°C in ambient air (25°C, 35-50% relative humidity) and 99.999% purity argon (Ar), respectively. The environmental flexure testing was conducted within an alumina retort (diameter = 100 mm), which was enveloped by an alpha-silicon carbide load train. The retort was surrounded by a resistively heated calmsell furnace and the entire unit was vertically mounted on a universal testing machine. The purity of the argon was independently verified with an oxygen analyzer which continually measured the oxygen content of the inert gas entering the retort. A slight positive pressure of approximately 2 kPa was maintained while the gas flow rate was adjusted to 0.5 ml/sec. The chamber was purged and back-filled with argon a minimum of three times prior to each test. Data acquisition was computer automated and included the tracking of load, load ram displacement, oxygen content, and test time. Further details of this work are described in Reference 5.

At ORNL, 51 MOR specimens were loaded to failure using stressing rates of 0.001, 0.5, and 37 MPa/sec at the previously mentioned temperatures and environments. Table 6 summarizes the results of testing and failure analysis. Test data on individual specimens and compiled in Appendix IV. Figure 22 shows both the environment and temperature affected the strength of the material as function of stressing rate. Power law regression curve fits are shown on the graph for the three temperatures and the two environments. Specimens tested in argon consistently exhibited higher strengths at the two lower stressing rates when compared to those specimens tested in air at the same temperatures. The line for the 1000°C specimens tested in argon is flat, indicating a very high slow crack exponent N and no susceptibility to SCG. The higher average fast fracture strength at 37 MPa/sec of 775 MPa at 1000°C for the specimens tested in argon compared to 603 MPa measured in air, a drop of 22% in strength, is evidence that the air environment causes weakening in PY-6 silicon nitride even at temperatures as low as 1000°C. This weakening is more pronounced in air at 1400°C, where the fast fracture strength dropped about 26% in air relative to argon at 37 MPa/sec.

Figure 17. MOR bar tested at a rate of 0.212 MPa/sec (0.00508 mm/min) at 1000°C shows softening and bubbling of the material.

Figure 18. MOR specimen tested at 1200°C at a average stress rate of 1.55 MPa/sec (cross-head speed 0.0508 mm/sec). Specimen failed from an internal inclusion.

Figure 19. Stress assisted oxidation of the MOR at 1200°C and 0.089 MPa/sec in air. Note the dark gray/black ring around a center light region. Surface pitting of the tensile surface is also visible.

TE94-2400

Figure 20. Optical micrographs of specimens tested at 1400°C at an average stressing rate of 0.035 MPa/sec. Surface pitting of the tensile surface is also visible.

Table 6. Average dynamic fatigue behavior of MOR specimens characterized at ORNL

Temperature—°C	Stress rate—MPa/sec	Environment	No. of specimens	Average strength*		Standard deviation		Failure time—sec	Fractography observations
				ksi	MPa	ksi	MPa		
1000	37	Air	3	87.45	603	2.21	15	16	Fast fracture from corner or edge machining flaw or internal defect
	0.5	Air	3	74.21	512	3.84	26	1023	
	0.001**	Air	2	77.45	534	0.44	3	534,000	
1200	37	Air	3	82.62	570	10.11	70	.15	Fast fracture from corner or edge machining flaw or internal defect. Fracture from a corner or edge damage zone
	0.5	Air	3	59.90	413	5.99	41	826	
	0.001**	Air	3	53.08	366	2.17	15	366,000	
1400	37	Air	3	67.34	464	6.35	44	13	Fast fracture from corner or edge machining flaw or internal defect. Fracture from a corner or edge damage zone
	0.5**	Air	3	37.37	258	5.22	36	515	
	0.001**	Air	3	28.43	196	1.89	14	196,000	
1000	37	Argon	2	112.40	775	4.06	28	21	Fast fracture from corner or edge machining flaw or internal defect
	0.5	Argon	2	102.83	709	3.92	27	1418	
	0.001	Argon	3	96.66	710	2.54	18	710,333	
1200	37	Argon	3	84.46	582	4.90	34	16	Fast fracture from corner or edge machining flaw or internal defect
	0.5	Argon	3	73.05	504	10.71	74	1007	
	0.001**	Argon	3	95.04	655	5.20	36	655,333	
1400	37	Argon	3	90.94	627	8.17	56	17	Fast fracture from corner or edge machining flaw or internal defect. Pitting observed on the corners of two specimens
	0.5**	Argon	3	78.37	540	4.80	33	1081	
	0.001**	Argon	3	53.90	372	1.07	7	371,667	

* As-measured strength of the specimens

** Permanent deformation observed. No correction has been made for curvature in the measured strength of the specimen.

TE94-2401

Figure 21. MOR specimen tested at 1400°C; 0.035 MPa/sec showing a large prominent gray ring around the light center region.

Figure 22. Dynamic fatigue behavior of PY-6 MOR bars tested in air and argon at ORNL.

The slower stressing rates (0.5 and 0.001 MPa/sec) resulted in further weakening of the material, except in the case of specimens tested at 1000°C in argon. The MOR specimens crept at the two lower stressing rates at 1400°C both in argon and air, but the strength in air was only half that in argon at the same conditions. It is noteworthy that average MOR strengths were similar for the tests conducted at 1200°C in air and at 1400°C in argon at 0.001 MPa/sec. This suggests the effects of creep damage alone at 1400°C in argon are as severe as the combined effects of both oxidation and creep damage at 1200°C in air.

The results of the failure analysis of the specimens are summarized in Table 6. In general, the specimens tested at 37 MPa/sec failed from a machining induced defect at the corner or edge of the tensile surface at all temperatures. Fracture surfaces of the specimens were evaluated for three ambient air conditions, namely 1200°C, 0.001 MPa/sec; 1400°C, 0.001 MPa/sec; and 0.5 MPa/sec showed damage zones. At 1400°C, the presence of cracks on the tensile surface can be seen as a stress assisted oxidation damage zone (Figure 23). A visual change in the microstructure and color was also revealed by polishing the cross section of specimens (Figure 23[b]). It is believed stress corrosion degradation of the material occurs in the presence of oxygen; the intergranular regions of the grain boundary provide a diffusion path for the oxygen while metal cation impurities move to the specimen surface from the bulk interior. The observed damage zone had a different appearance than the SCG zones identified in sintered silicon nitride.^{6,7}

To further characterize the discolored region, X-ray diffraction (XRD) analysis of the tensile surface of the specimens tested at 1400°C in air and argon was conducted after removing a 75 micron surface layer. The XRD results showed the amorphous yttrium-silicate had been converted to beta-Y₂Si₂O₇ and alpha-Y₂Si₂O₇ for the specimens tested in air and argon, respectively. Pure alpha-Y₂Si₂O₇ is stable in the range of 25 to 1225°C, while pure beta-Y₂Si₂O₇ is stable between 1225 and 1445°C. If the transformation from beta to alpha was to occur around 1225°C, then one would only expect the alpha phase to be present; however, Falk and Dünlop⁸ have reported the presence of beta phase on the surface and alpha in the interior of the specimen in their studies with pressureless sintered silicon nitride at 1400°C in air. They reported that the beta phase was stabilized at the surface on the silicon nitride specimens by the extra impurity cations diffused from the bulk to the surface while the alpha phase was present in the bulk interior. In the present study the beta phase was identified in the discolored region and may be indicative of cation impurities having diffused to the surface and stabilized the high temperature phase. No discolored ring nor beta-phase were observed in the specimens characterized in the argon environment at 1400°C.

A comparison of the flexural dynamic fatigue studies, conducted both at Allison and ORNL, from 1000°C to 1400°C revealed similar SCG behavior of the PY-6 injection molded and HIPed silicon nitride. The material degraded both with temperature and stressing rate. The lowest stressing rate at ORNL was more than one order of magnitude slower than that used at Allison (0.001 MPa/sec at ORNL versus 0.035 MPa/sec at Allison), which additionally revealed significant creep enhanced damage and stress assisted oxidative corrosion and degradation of the material. Specimens tested at Allison did not demonstrate any evidence of creep at lower temperatures and stressing rates, except for the influence of stress assisted oxidative pitting. Some curvature of MOR bar was measured at 1400°C and 0.035 MPa/sec.

At the lower stressing rate, the time-to-failure of ORNL specimens was 35 times greater than the time-to-failure for specimens evaluated at Allison. This is believed to have resulted in larger visible damage zones not only at 1400°C but also at lower temperatures. Even though the starting composition of the PY-6 material tested at both the laboratories was identical, the differences in the grain boundary composition of the HIPed material tested at Allison and ORNL could be different. Specimens might have been HIPed in separate batches, and the stacking sequence even in the same HIP furnace run may have caused variations in the grain boundary devitrification and crystallization due to temperature gradient or other unknown factors. Overall, it is believed the study at the two laboratories was successful and complimentary and added

Figure 23. Fractography of a specimen tested at 1400°C and 0.001 MPa/sec in air (a) fracture surface showing the damage shown, (b) polished cross-section with ring around the perimeter, and (c) side view of the specimen depicting parallel Mode I cracks on the tensile surface of the MOR bar.

significantly to a better understanding of the flexural dynamic fatigue behavior of PY-6 material. The testing at both the laboratories improved the robustness of the data base which is extremely important when deployment of new brittle silicon nitride materials is being envisioned in future heat engine products.

3.2.2 Tensile Dynamic Fatigue Evaluation

Tensile dynamic fatigue studies were conducted using both the button-head and dogbone specimens to assess if the two specimen geometries gave comparable SCG results. Fabrication of the button-head specimens is substantially more expensive than the dogbone specimens, thus, data base generation is expensive. Ceramic rotors for heat engines are generally 7.5 cm to 15.0 cm (3 in. to 6 in.) in diameter and only small specimens, such as dogbone size, can be dissected from the hub of the rotor to measure the properties of the material and compare them with the material properties used in the design of the component. Only MOR bars are currently machined and tested to qualify the component process and material quality. This is suitable only for fast fracture qualification of the material. For time dependent qualification of the material, tensile testing of the material must be conducted. Dogbone tensile specimens are relatively cheaper to machine and are potentially suitable for component qualification. The button-head specimens were fabricated from injection molded and HIPed rods, whereas the dogbone specimens were machined from similarly processed rectangular billets. The machining of the button-head specimens was done in-house by GTE, whereas the dogbone specimens were machined by Bomas Specialty Machining, Boston, Massachusetts. All the specimens were examined by the various NDE techniques prior to mechanical testing.

3.2.2.1 Tensile Dynamic Fatigue of Button-Head Specimens at SoRI. SoRI tested 55 button-head specimens from 1000 to 1400°C at loading rates ranging from 0.115 MPa/sec to 69 MPa/sec. The testing was conducted in the gas-bearing facility described in Section 2. The stress-strain behavior of each specimen was recorded. Stress-strain plots of all the specimens tested at 6.9 MPa/sec and 0.115 MPa/sec from 1000 to 1400°C are shown in Figure 24. The ultimate strength, strain-to-failure, modulus, and failure time for each specimen was calculated, and Table 7 summarizes the results. Details of the individual specimen results are shown in Table 8 and Appendix V. The average specimen strength at 1000°C dropped from 395.4 MPa at 69 MPa/sec to 290.3 MPa/sec at 0.115 MPa/sec, a drop of almost 27% in strength. At 1100 and 1200°C, the average strength decreased 11% and 7%, respectively, at 0.115 MPa/sec compared to 69 MPa/sec. The average strength, however, also dropped 27% at 1300°C at 0.115 MPa/sec compared to 69 MPa/sec. Similarly, at 1400°C the strength dropped 44% at the lowest stressing rate relative to 69 MPa/sec test data. Based on the strength results, it is believed that the material fracture mode may be changing between 1200 and 1300°C, in particular at 0.115 MPa/sec. The material exhibited linear elastic stress-strain behavior between 1000 to 1200°C except at a 0.115 MPa/sec stressing rate at 1200°C. At 0.115 MPa/sec, slight deviation from linearity was beginning to be observed at 1200°C, which was further amplified at 1300°C indicating intergranular grain boundary material softening causing decrease in the modulus of the material. In fact, at 1400°C the material exhibited nonlinear behavior at both the 6.9 MPa/sec and 0.115 MPa/sec, again reflecting the viscous flow and grain boundary sliding at the grain boundaries. This was also observed even at 69 MPa/sec (Figure 12). The average strain-to-failure ranged from 0.0009 to 0.0014 at all temperatures and stressing rates except at 1400°C and 0.115 MPa/sec where the average strain-to-failure increased to 0.0018 in. with a corresponding decrease of about 50% in the modulus of the material, which is probably due to both SCG and creep damage accumulation of the material and has been found to occur concurrently in silicon nitride materials.

TE94-2404-5
Part 1 of 5

Figure 24. Stress-strain behavior of button-head specimens tested at (a) 6.9 MPa/sec and (b) 0.115 MPa/sec (page 1 of 5).

Figure 24. Stress-strain behavior of button-head specimens tested at (a) 6.9 MPa/sec and (b) 0.115 MPa/sec (page 2 of 5).

Figure 24. Stress-strain behavior of button-head specimens tested at (a) 6.9 MPa/sec and (b) 0.115 MPa/sec (page 3 of 5).

Figure 24. Stress-strain behavior of button-head specimens tested at (a) 6.9 MPa/sec and (b) 0.115 MPa/sec (page 4 of 5).

Figure 24. Stress-strain behavior of button-head specimens tested at (a) 6.9 MPa/sec and (b) 0.115 MPa/sec (page 5 of 5).

Table 7. Average dynamic fatigue behavior of button-head tensile specimens

Temperature—°C	Stress rate—MPa/sec	No. of specimens	Average strength		Modulus—GPa	Strain to failure—in./in.	Failure time—sec
			ksi	MPa			
1000	69	5	57.35	395.4	295.8	0.0014	6
1000	6.9	2	39.57	272.8	332.6	0.0009	40
1000	0.115	2	42.11	290.3	287.9	0.0011	2524
1100	69	5	45.87	316.3			5
1100	6.9	2	47.72	329.0	275.5	0.0014	48
1100	0.115	3	41.34	285.1	274.4	0.0011	2479
1200	69	5	45.23	311.8			5
1200	6.9	2	37.60	259.3	258.8	0.0010	38
1200	0.115	5	42.24	291.2	291.5	0.0010	2532
1300	69	5	55.07	379.7			6
1300	6.9	2	46.53	320.8	327.4	0.0011	46
1300	0.115	3	40.16	276.9	267.9	0.0014	2408
1400	69	5	43.74	301.6			4
1400	6.9	2	35.50	244.8	300.1	0.0011	35
1400	0.115	4	24.41	168.3	157.5	0.0018	1464

Failure analysis of all the specimens was conducted using optical micrography to ascertain the failure location and fracture controlling defect. A limited number of specimens were examined by SEM. At the 6.9 MPa/sec stressing rate, the fracture surfaces from 1000 to 1200°C appeared similar to those resulting from monotonic tensile testing at 69 MPa/sec. At 1300°C, however, internal pitting and weakening of the material was observed (Figure 25), and the fracture surfaces appear to be smoother than the torturous surface observed at 69 MPa/sec indicating change in the failure mechanism. No classic SCG features were visible. At 1400°C both optical and SEM micrography reveal a damage zone similar to 1300°C specimens (Figure 26). At the lowest stressing rate (0.115 MPa/sec) failure surfaces both at 1000 and 1100°C appear identical to those of the two faster rates (Figure 27). At 1200°C, the presence of SCG from a pre-existing defect is indicated with the inception of grain boundary softening at 0.115 MPa/sec (Figure 28), which is evident from the stress-strain data shown in Figure 24 (b). At 1300°C and 0.115 MPa/sec, the failure occurs from a combination of SCG and grain boundary softening (Figures 29 and 30) which decreases the material modulus. At 1400°C and 0.115 MPa/sec, both SCG and interstitial multi-grain junction creep cavities (Figure 31) contribute to the degradation of the strength and modulus of the material.

Table 8. Dynamic fatigue behavior of button-head tensile specimens.

Specimen number	Temperature— °C	Stress rate— MPa/sec	<u>Strength</u>		Modulus— GPa	Strain- to-failure— in./in.	Failure time— sec	Failure origin	Failure location from center of gage— mm	
			ksi	MPa					+	-
493B	1000	69	45.15	311.27	329.4	0.0010	5	II		29.21
118B	1000	69	65.55	451.97	293.1	0.0016	7	SF	7.62	-
121B	1000	69	65.55	451.96	288.8	0.0016	7	SF		27.18
52A	1000	69	43.49	299.86	300.4	0.0010	4	SI	9.40	
05A	1000	69	67.00	461.94	267.5	0.0019	7	SF		26.67
51A	1000	6.9	41.69	287.45	333.9	0.0009	42	SF		15.24
54A	1000	6.9	37.44	258.15	331.3	0.0008	37	SF	2.54	
60A	1000	0.115	46.01	317.24	283.7	0.0012	2759	SF		28.45
568B	1000	0.115	38.20	263.39	292.2	0.0009	2290	SF		3.81
655B	1100	69	45.16	311.38	301.1	0.0010	5	SI		13.21
53A	1100	69	45.38	312.90	248.4	0.0013	5	SF	31.24	
482B	1100	69	46.25	318.89	304.0	0.0010	5	SF		17.78
653B	1100	69	43.35	298.90	259.0	0.0012	4	IP		25.40
454B	1100	69	49.22	339.37			5	IP		
67A	1100	6.9	40.72	280.76	-	-	41	Failed at two locations within gage		
569A	1100	6.9	54.71	377.23	275.5	0.0014	55	II	3.05	
547A	1100	0.115	37.24	256.77	212.0	0.0012	2233	See note 1		
549B	1100	0.115	44.15	304.41	313.3	0.0009	2647	See note 1	27.94	
556A	1100	0.115	42.64	294.00	297.9	0.0010	2557	II		12.70
484B	1200	69	45.65	314.76	306.1	0.0010	5	SF	21.59	
520B	1200	69	44.94	309.86	292.9	0.0011	4	SF		25.91
520A	1200	69	45.35	312.69	300.0	0.0011	5	SF		24.13
490A	1200	69	44.85	309.24	298.8	0.0010	4	SF		23.62
518B	1200	69	45.34	312.62	295.7	0.0011	5	Failed at two locations within gage		
558A	1200	6.9	38.69	266.77	265.2	0.0010	39	See note 1	21.59	
558B	1200	6.9	36.50	251.67	252.3	0.0010	36	See note 1		17.02
554B	1200	0.115	34.33	236.71	264.4	0.0009	2058	See note 1		12.70
546B	1200	0.115	46.36	319.65	304.3	0.0012	2780	II		19.05
564B	1200	0.115	59.13	407.70	306.0	0.0012	3545	Failed at two locations within gage		
521A	1200	0.115	33.20	228.91	253.3	0.0006	1991	IF		
534A	1200	0.115	38.17	263.18	329.4	0.0008	2289	SF		25.40

Table 8 (cont).

Specimen number	Temperature— °C	Stress rate— MPa/sec	Strength		Modulus— GPa	Strain- to-failure— in./in.	Failure time— sec	Failure origin	Failure location from center of gage— mm	
			ksi	MPa					+	-
192A	1300	69	55.62	383.50	299.7	0.0013	6	II	13.97	
184B	1300	69	55.12	380.05	305.7	0.0013	6	SF		27.94
332A	1300	69	53.83	371.16	306.2	0.0012	5	II	5.33	
54B	1300	69	58.10	400.60	280.6	0.0014	6	Failed at two locations within gage		
110A	1300	69	52.69	363.30	304.3	0.0012	5	II		
553A	1300	6.9	49.01	337.92	325.6	0.0011	49	II		13.21
549A	1300	6.9	44.04	303.66	329.2	0.0009	44	SF		29.46
565A	1300	0.115	48.80	336.48	258.0	0.0016	2926	SF		3.05
574A	1300	0.115	34.84	240.22	241.3	0.0012	2089	See note 1		16.51
575A	1300	0.115	36.84	254.01	304.4	0.0012	2209	II	17.78	
662A	1400	69	43.83	302.21	321.0	0.0010	4	II	5.08	
661A	1400	69	43.96	303.10	316.0	0.0010	4	II	12.19	
664A	1400	69	43.70	301.31	319.2	0.0010	4	II	10.92	
329B	1400	69	42.97	296.28	319.0	0.0010	4	SI		
334A	1400	69	44.23	304.97	276.6	0.0012	4	II		20.83
545A	1400	6.9	38.96	268.63	309.5	0.0012	39	SF	1.27	
548A	1400	6.9	32.04	220.92	290.6	0.0010	32	See note 1		2.54
526A	1400	0.115	22.09	152.31	183.4	0.0015	1324	See note 1	2.54	
571A	1400	0.115	22.62	155.96	131.8	0.0017	1356	SF	1.27	
564A	1400	0.115	26.08	179.82	157.3	0.0021	1564	SF	5.08	
81C	1400	0.115	26.86	185.20	-	-	1610	SF	8.89	

+: Towards top of the loading rod

SF - Surface machining flaw

-: Towards bottom of the loading rod

SI - Surface inclusion

IP - Internal pore

II - Internal inclusion

Note 1: Specimen packing opened up in transit from Southern Research Institute to Allison. This caused mix up in specimens and difficulty in matching broken ends of specimens to identify failure origin. In some cases it was possible to identify flaw location.

(a)

(b)

TE94-2405

Figure 25. Fractography of specimens tested at 1300°C and 6.9 MPa/sec (a) failed at 303.7 MPa from surface flaw and (b) failed at 337.9 MPa from internal inclusion.

TE94-2406

Figure 26. Fractography of specimen tested at 1400°C and 6.9 MPa/sec with strength of 268.8 MPa.

TE94-2407

Figure 27. Fractography of specimen tested at 1100°C and 0.115 MPa/sec. The specimen failed from an internal inclusion.

TE94-2408

Figure 28. Fractography of a specimen tested at 1200°C and 0.115 MPa and failed from an internal flaw at 228.9 MPa. The defect is very porous.

TE94-2409

Figure 29. Fractography of a specimen characterized at 1300°C and 0.115 MPa/sec. The strength was 336.5 MPa. Failure occurred via SCG and creep damage.

TE94-2410

Figure 30. Fractography of a specimen tested at 1300°C and 0.115 MPa/sec and failed from an internal inclusion at 254.01 MPa.

Figure 31. Fractography of a specimen tested at 1400°C and 0.115 MPa/sec and failed from a surface flaw at 156 MPa.

TE94-2411

Based on the results it is evident that classic SCG features are not evident in a majority of the specimens. The degradation due to dynamic fatigue testing conditions is caused by primary SCG between 1000 and 1200°C. The stress-strain behavior in this temperature regime is linear at all stressing rates, except at 1200°C and 0.115 MPa/sec when the stress-strain curve becomes slightly non-linear indicating limited softening of the glassy grain boundary phase. At 1300 and 1400°C, both SCG and creep damage accumulation further degrades the strength and modulus of the material.

3.2.2.2 Tensile Dynamic Fatigue of Dogbone Specimens at NIST. The dogbone specimens were characterized at the National Institute of Standards and Technology (NIST) from room temperature to 1400°C at varying loading rates from 0.062 MPa/sec to 16 MPa/sec. The testing was conducted using an Instron testing facility. Rectangular billets of 6 cm x 5 cm x 1.5 cm nominal size of PY-6 silicon nitride were injection molded and HIPed. The 50 mm width of the billet was selected to match with the 50 mm length of the specimen. This minimized the machining of the specimens. Microradiography inspection of the billets at GTE indicated the presence of voids and surface cracks in some billets. Ten billets with no visible voids or cracks were selected and shipped to NIST for the fabrication of the dogbone specimens. The average density of the billets was measured to be 3.2548 gm/cc. Dogbone specimens of similar length as that of the billets were machined at Bomas Specialty Machining (Figure 32). The cross section of the billet was reduced to that of the final tensile specimen with a shaped grinding wheel. A thin wafering blade then cut specimens of the desired thickness from the block. After the tensile specimens were sliced from the block, holes were machined into the specimens; accurate machining of the holes along the center line of the specimen was essential for good alignment. The percent of bending was held between 1 to 3% depending on the applied stresses used. At lower applied stresses the bending was higher and visa-versa.

All the specimens were inspected by FPI, microradiography, and acoustic microscopy for surface and volume flaws. Figure 33 shows typical photographs of two faces of the ten machined specimens after FPI from a billet. A large amount of penetrant was retained by the specimens indicating the presence of minute surface machining damage. The average density of the dogbone specimens was measured to be 3.2626 gm/cc. The specimens were further examined by microradiography using optimized MOR parameters which did not reveal any large defects. Acoustic microscopy of the two faces of the specimens was conducted using a 50 MHz F#0.8 surface wave transducer with a water path of 5.08 mm. The acoustic images (Figure 34 [a] and [b]), indicated minor variation in the surface finish of the two faces of the ten specimens. The side walls of the specimens were also investigated by acoustic microscopy for the presence of any surface defects (Figure 35). Enlargement of the acoustic images of the specimens revealed the presence of a surface defect in the side wall of one specimen (Figure 36) about 85 microns in size. The majority of the specimens did not reveal any large discrete flaws.

Table 9 summarizes the results of dynamic fatigue testing at NIST. Figure 37 shows the data graphically and indicates the strength does not decrease significantly as a function of stressing rate at 1200°C. This implies that the material is not very susceptible to SCG at this temperature. At 1300 and 1400°C, the material strength degrades similarly as a function of loading rates. Overall, a large drop in strength is observed from 1000 to 1400°C. The decrease in strength is a consequence of subcritical crack growth and of creep damage accumulation, which reduces the strength of silicon nitride at a lower stressing rate. At temperatures of about 1200°C, transgranular fracture dominates, whereas above 1260°C, the fracture becomes primarily intergranular. Above 1260°C, both SCG and cavitation contribute to the degradation of the material.

TE94-2412

Figure 32. Schematic of dogbone tensile specimen tested at NIST.

TE94-2413

Figure 33. Optical bottom photograph of specimens after FPI inspection.

Figure 34. Ultrasonic C-scan surface wave images of the back surface of dogbone specimens.

Figure 35. Surface wave acoustic microscopy of the two side walls of the gage area of dogbone specimens machined from billet 65R.

Figure 36. Acoustic microscopy characterization of the gage area of a dogbone specimen using surface wave.

Table 9. Dynamic fatigue behavior of dogbone tensile specimens characterized at NIST

Specimen number	Tempera- ture—°C	Stress rate— MPa/sec	<u>Tensile strength</u>		Failure time—sec
			ksi	MPa	
37R7	RT	16	61.60	424.7	27
47R4	RT	16	61.78	426.0	27
65R10	RT	16	69.30	477.8	30
47R7	1000	16	56.79	391.6	24
36R3	1000	16	56.43	389.1	24
65R7	1150	0.062	62.57	431.4	6958
46R9	1200	16	55.66	383.8	24
61R4	1200	0.062	55.94	385.7	6221
46R1	1200	0.062	50.82	350.4	5652
46R2	1300	16	43.73	301.5	19
35R8	1300	16	52.60	362.7	23
61R7	1300	16	49.60	342.0	21
47R9	1300	0.062	33.23	229.1	3695
40R8	1300	0.062	33.37	230.1	3711
35R2	1350	0.062	25.09	173.0	11
40R3	1350	0.062	25.96	179.0	11
39R5	1400	16	38.43	265.00	17
40R10	1400	16	34.04	234.70	15
39R1	1400	0.062	18.93	130.50	2105
61R10	1400	0.062	19.71	135.90	2192

Figure 37. Dynamic fatigue behavior of PY-6 Si_3N_4 dogbone specimens.

The comparison of the dynamic fatigue results obtained on the button-head and the dogbone specimens are depicted in Figure 38. The power law curve fit line are almost parallel for both sets of data at all temperatures, clearly indicating excellent correlation and fracture from identical failure mechanisms. At 1200°C the curve fit line is nearly flat, indicating a very large SCG exponent (N) and showing the material is resistant to SCG at this temperature. Both at 1300 and 1400°C, the curve fit lines for both types of specimens are again parallel providing further proof that the dogbone specimen is suitable for assessing the average performance of the material in dynamic fatigue testing. This is very encouraging since dogbone specimens can be dissected from ceramic rotors and time dependent behavior and the quality of the component material can be assessed.

3.3 UNIAXIAL CREEP AND STRESS RUPTURE

Reliable usage of structural silicon nitride components in vehicular and stationary power generating heat engines require a long-term data base at elevated temperatures between 1150 to 1400°C to ensure structural integrity of the material in field applications. Silicon nitride materials have been known to fail from SCG and creep rupture time-temperature dependent failure modes. It has been reported by many workers that both SCG and creep may be occurring concurrently in the current silicon nitride materials and identification of exact temperatures to isolate the regime of the individual failure mechanism may not be obvious. During this program, a systematic effort was made to assess the tensile stress and creep rupture behavior of injection molded and HIPed PY-6 silicon nitride material using both the button-head and dogbone tensile specimens. Testing of the two specimen geometries was conducted to determine the suitability of the dogbone specimen to generate reliable creep data for component design and process qualification. Dogbone specimen fabrication is relatively cheap and is readily adaptable to use for process qualification.

Figure 38. Comparison of dynamic fatigue behavior of button-head and dogbone specimens of PY-6 Si₃N₄ material.

3.3.1 Creep Rupture Testing Using Button-Head Specimens

In Table 10, 95 tensile button-head specimens were characterized from 1149 to 1400°C at SoRI to assess the creep and stress rupture behavior of the material. The average measured density of the specimens was 3.2596 gm/cc using the Archimedes method. The average ultrasonic longitudinal velocity was measured to be 1.0655 cm/μsec (0.4195 in/μsec) using the time of flight measurement method. No correction was made for acoustic diffraction. The dimensions and concentricity of each specimen were determined prior to testing as described in Section 3.1.2. In general, the total run out of the specimen/pullrod assembly was maintained to around 75 μm to 100 μm. The specimens were nondestructively inspected using FPI, microradiography, and acoustic microscopy. The specimen and grip alignment procedure was described in Section 3.1.2.

Three deadweight loading creep testing systems were assembled and equipped with the high temperature heating, gripping, and clip-on strain measurement method described in Section 3.1.2. Strain was collected on both sides of the gage in each specimen, averaged, and digitally collected and stored on an IBM-PC based data acquisition system. As a back up, the strain was also recorded on an x-y recorder. Figure 39 shows the high temperature creep testing set up and loading system. The precision of the strain measurement system was experimentally determined to be ± 50-75 μin/in. The temperature gradient, measured by reading the temperature differences between the top, middle, and bottom of the gage section, was on the order of 28°C. To achieve this gradient, the top and the bottom loading rods were heated to 800 - 875°C. At the set point, the controller maintained temperature to within 5°C.

Typical creep strain versus time curve are given in Figures 40 and 41. The creep rate was calculated as a linear least square fit to the minimum slope of the curve. Table 10 summarizes the creep test data and failure analysis results of all the specimens. The selection of the applied

Figure 39. Creep rupture test set up at SORL.

Figure 40. Typical creep strain versus time curves at 1200, 1260, and 1300°C.

Figure 41. Typical creep strain versus time curves at 1370 and 1400°C.

Table 10. Creep rupture characterization of button-head specimens at SoRI

Specimen number	Temperature— °C	Applied stress— MPa	Creep rate— 1/sec	Total strain— ($\mu\epsilon$)	Time-to-failure— hr	Failure origin	Failure mechanism	Location from center of gage—mm		Remarks
								+	-	
550B	1149	229.92	7.74 E-10	1020	3.02	SF	Stressed oxidation		19.20	
572A	1149	239.79	5.52 E-09	1140	41.9	IF	Stressed oxidation		21.23	
547B	1149	248.08	--	--	0.003	SF	Failed while loading			
546A	1149	249.13	--	--	0.003	SF	Failed while loading	10.31		
341A	1200	124.78	9.37 E-11	530	>240	No failure				
635B	1200	149.75	9.95 E-10	2710	>233	No failure				
517B	1200	159.84	3.61 E-10	1730	>360	No failure				
525A	1200	174.33	7.97 E-09	890	18.96	SF	SCG	29.03		
636A	1200	174.69	4.02 E-10	1240	>389	No failure	Grain boundary cavities			
454B	1200	177.12	--	--	0.008	SF	Failed while loading	13.23		
452A	1200	177.74	--	--	0.733	SF			17.58	Note 11
629A	1200	180.75	--	--	>291	No failure				Note 1
514A	1200	184.99	3.40 E-07		0.009	SF at 45° loading axis		11.46		
448B	1200	190.03	2.68 E-07	--	0.159	SI		17.30		
460A	1200	199.25	7.63 E-09	1700	2.06	SF	SCG			
552A	1200	201.59	--	--	0.003	SF	Failed while loading	35.43		
565B	1200	215.11	8.55 E-11	1110	>612	No failure				Note 26
454A	1200	220.75	--	--	0.0087	SI	Failed while loading		2.46	
463B	1200	225.46	2.27 E-08	1600	0.55					
453A	1200	250.49	4.92 E-08	1700	0.48	SF	SCG, rough surface at origin		12.50	
90A	1200	289.59	6.10 E-10	1570	>266	No failure				Note 2
533B	1260	125.05	3.29 E-10	--	>721	No failure				Note 14
533A	1260	141.07	--	--	4.42	SF	Rough/glassy surface at origin	27.69		Note 1
471A	1260	141.22	3.36 E-08	910	>27.37	No failure				Note 1
488B	1260	149.94	2.21 E-08	490	0.82	SF	Rough/glassy surface at origin		4.52	
479A	1260	155.06	1.43 E-10	1650	>703	No failure				
458A	1260	160.81	5.87 E-07	1250	0.122	SF	Rough/glassy surface at origin	9.78		

Table 10 (cont)

Specimen number	Temperature— °C	Applied stress— MPa	Creep rate— 1/sec	Total strain— ($\mu\epsilon$)	Time-to-failure— hr	Failure origin	Failure mechanism	Location from center of gage—mm		Remarks
								+	-	
466B	1260	165.93	5.01 E-09	1300	5.12	SF		31.65		
515A	1260	175.20	4.54 E-10	3380	>1154	No failure	Two grain interfacial cavitation			Note 13
463A	1260	175.26	6.52 E-10	2010	364		GB interstitial cavitation/interface			Note 9
532A	1260	176.21	4.58 E-09	820	2.73	SF	Rough surface at origin		12.52	
486A	1260	177.71	71.6 E-09	1060	26.24	IF	SCG			
528A	1260	180.04	9.15 E-08	850	0.475	SF		2.16		
456A	1260	185.19	3.64 E-07	990	0.205	IF			0.46	
567B	1260	200.52	2.05 E-10	1220	306	No failure				
553B	1260	226.19	2.95 E-07	950	0.135	SF			17.25	
559B	1260	252.07	--	--	0.003	SF	Failed while loading		15.24	
468B	1300	140.04	--	--	2.03	SF			31.55	
531B	1300	140.49	6.68 E-10	3890	306.1	SF		0.64		
626B	1300	149.97	9.51 E-10	3200	>473	No failure	Grain boundary cavitation			
634B	1300	150.74	1.21 E-09	2750	>624	No failure				
453B	1300	152.27	--	--	0.003	SF		28.45		Note 19
487A	1300	155.22	5.4 E-08	860	0.55	SF				
488A	1300	155.89	--	--	0.003	SF		29.18		Note 19
524B	1300	159.70	4.22 E-08	1670	1.87	SF			14.25	Note 20
519A	1300	160.53	9.21 E-08	750	0.182	II		3.18		
467A	1300	164.70	--	--	109.41	SF		38.43		Note 1
523A	1300	164.80	1.88 E-08	1410	4.99	SF	Two interfacial cavitation	10.80		Note 20
626A	1300	173.15	--	--	0.003	SF at angle	Broke while loading		25.88	Note 16
491B	1300	174.88	--	740	0.003	SF		30.18		Note 11
474A	1300	175.06	3.91 E-08	610	0.535	SF			22.38	
188B	1300	175.27	1.63 E-09	5490	772.0	IF	Interfacial cavitation			
457B	1300	176.23	9.73 E-08	1130	0.364	SF			28.78	
514B	1300	178.08	8.07 E-08	1230	0.826	SF		3.71		

Table 10 (cont)

Specimen number	Temperature— °C	Applied stress— MPa	Creep rate— 1/sec	Total strain— ($\mu\epsilon$)	Time-to-failure— hr	Failure origin	Failure mechanism	Location from center of gage—mm		Remarks
								+	-	
341B	1300	180.17	6.04 E-08	1710	3.3		GB interstitial cavitation			
343B	1300	184.60	2.40 E-07	1930	0.96					
77A	1300	199.54	1.54 E-07	1250	0.69	SF		1.98		
334B	1300	249.31	4.73 E-07	1670	0.24	SF			0.37	
201A	1300	275.22	1.38 E-05	1100	0.013		Viscous flow and GB interstitial cavitation			
493A	1338	159.88	2.08 E-07	1960	1.44	II			17.02	
464B	1370	65.55	--	--	>617.2	II	Specimen broke in several pieces on disassembly			Note 12
522B	1370	70.47	8.41 E-10	1470	>287	No failure	Cavitation			Note 18
84B	1370	73.91	6.02 E-09	7010	287	SF	Intertecial Cavitation			
80A	1370	74.67	1.19 E-09	--	>387	No failure	Specimen broke in several pieces on disassembly			
457A	1370	78.78	--	--	0.003	SF	Broke while loading	2.67		Note 15
57A	1370	79.51	2.51 E-09	7200	>587.5	No failure				Note 21
476A	1370	85.19	1.58 E-09	2920	>413	No failure	Specimen broke in several pieces on disassembly			Note 22
55A	1370	86.46	--	1470	0.006		Broke while loading		22.94	Note 5
90A	1370	86.95	5.99 E-09	00	>101	No failure	Failed			Note 3
602B	1370	90.33	4.59 E-07	3400	7.87	SF				
521B	1370	90.69	--	--	>105.63	SF	Stress-oxidation		0.61	Note 1, Note 8
535B	1370	95.29	4.44 E-09	1340	>73.94	No failure				Note 1
522A	1370	95.37	9.02 E-10	480	>146	No failure				Note 10
490B	1370	99.75	1.69 E-07	1300	0.61	SF		9.88		
524A	1370	105.37	1.95 E-07	3770	8.13		Viscous flow and intestacial Cavitation			
563A	1370	109.83	8.35 E-10	8270	>997.6	No failure				

Table 10 (cont)

Specimen number	Temperature— °C	Applied stress— MPa	Creep rate— 1/sec	Total strain— ($\mu\epsilon$)	Time-to-failure— hr	Failure origin	Failure mechanism	Location from center of gage—mm		Remarks
								+	-	
573B	1370	120.43	6.79 E-11	1600	>1865	No failure	Specimen broke in several pieces on disassembly			
561B	1370	125.32	7.36 E-08	1050	2.18	SF		7.62		
576A	1370	131.20	1.84 E-09	--	230	SF			2.54	Note 24
192B	1400	75.09	1.97 E-09	10390	844		Two grain interfacial Cavitation			
486B	1400	79.80	--	--	>266	No failure				Note 1
497A	1400	79.85	4.36 E-09	4360	>217.5	No failure				
59A	1400	84.66	2.24 E-07	3990	1.8	SF	Rough origin	2.51		
523B	1400	85.66	4.07 E-08	1160	4.5					
612B	1400	90.23	1.10 E-08	5920	65.74	IF	Stress-oxidation	6.68		
611A	1400	95.47	5.01 E-08	2970	10.12	SF	Rough origin	6.78		
195B	1400	99.98	3.58 E-08	3100	12.97		Two grain interfacial Cavitation			8
530B	1400	110.33	3.93 E-08	1310	2.89					
552B	1400	114.9	3.10 E-08	--	172.3	SF	Two grain interfacial Cavitation		4.83	
171A	1400	125.35	3.51 E-07	2960	1.96		Viscous flow, cavitation			
548B	1400	130.13	4.96 E-08	660	4.18	SF	Stress-oxidation	11.43		
60A	1400	150.66	2.10 E-06	1380	0.025					
340A	1400	174.37	1.33 E-05	1310	0.015	SF	Rough origin			
621A	1427	70.49	--	--	>14.74	No failure	Temperature overshoot			Note 17
525B	1500	90.33	2.4 E-07	2350	0.84	SF	Stress-oxidation	7.98		Temp- erature overshoot
+: -: SF: SI: IF:	Towards top of loading rod Towards bottom of loading rod Surface flaw caused by machining damage Surface inclusions Internal flaw					II: SCG:	Internal inclusion: Slow crack growth			

Table 10 (cont)

Notes:

1. Clip-on flags slipped after x hours.
2. First test run. After 266 hours, raised temperature to 1370°C and lowered stress to 86.95 MPa.
3. Heater element connection arcing after 101 hours, test stopped.
4. Heater failure after 387 hours; amplifier problem.
5. One of the first tests run. Time scale too large for accurate creep strain data reduction. Estimate time to failure at 22 seconds.
6. Failed while loading.
7. Failed before last weight put on, 35 pounds applied. Estimate time to failure at 30 seconds.
8. Heater element connection arcing caused specimen failure at 105 hours.
9. Connection arcing after 146 hours. Connection replaced and test restarted.
10. Heater element connection arcing after 146 hours, test stopped.
11. Test too short to obtain accurate creep rate.
12. Failed before last weight put on, 15.5 pounds applied. Estimated time to failure at 11 seconds.
13. Utility company had to turn water off at 170 hours. Test stopped and restarted.
14. Utility company had to turn water off at 95 hours. Clip-on flags slipped on restart. Test continued for stress-rupture data.
15. Utility company had to turn water off at 19 hours. Clip-on flags slipped on restart. Test continued for stress-rupture data.
16. Failed before last weight put on, 34.5 pounds applied. Estimate time to failure at 10 seconds.
17. Temperature overshoot to 1427°C. Test planned for 1370°C. Test stopped.
18. Williamson optical went out after 287 hours causing temperature overshoot and premature failure.
19. Failed while loading.
20. Possible bad alignment caused quick time to failure. Frame recalibrated.
21. Test stopped after 587.5 hours due to heater failure.
22. Test stopped after 413 hours due to heater failure.
23. Failed while loading.
24. Flags slipped but specimen ran for 88 hours. Restarted and specimen failed after 230 hours.
25. Failed while loading.
26. Test stopped after 612 hours due to heater failure.

stress levels were picked based on the fast fracture data and the need to generate a large range of time-to-failure for modeling; however, this was found to be slightly difficult to follow primarily because of the low Weibull modulus of the material. Specimens expected to fail in long periods of time actually failed in short time and visa-versa at all temperatures. NDE performed prior to testing did not reveal any large defect to cause premature failure. It is, therefore, believed that great care must be taken to utilize such test data for design of ceramic components. The distribution of failure time must be included in the probability of failure or survival calculations of the components.

The primary creep region in the specimens ranged from very short time to 500 hours or more at the various temperatures (Figures 40 and 41) similar to the measurement made by several other workers on various silicon nitrides. The material exhibited both primary and steady-state secondary creep regions similar to other silicon nitride materials, including NT154. The scatter and variation in the primary creep region of specimens at a given temperature and applied stress is significantly influenced by the variations in the grain boundary phase which pronounces effects the creep behavior of the material causing variability in the kinetics of the grain boundary devitrification and recrystallization which ultimately controls the creep nature of the material.

The minimum creep data as a function of applied stress are depicted in Figure 42 with rather interesting results. The Norton power law ($\dot{\epsilon} = C \cdot \sigma^n$) regression for the data at 1300 to 1400°C revealed parallelism in the curve fit lines, and is believed to be a consequence of similar failure mechanisms operating in the material. The data at 1370°C are mostly scattered around the 1400°C data and the curve fit line. From these results it is clear that identical failure mechanisms control rupture of the material both at 1370 and 1400°C. The data at 1200 and 1260°C have much larger scatter but do show comparative slopes of the curve fit lines. In fact, about 50% of the data at 1260°C is clustered with the 1200°C and the other half with 1300°C data, revealing both SCG and creep rupture may be occurring at 1260°C depending on the test conditions. Consequently, it appears 1260°C is the key transition temperature where the failure mode probably switches from an SCG mechanism to creep rupture. These findings are consistent with the results of other researchers in similar type of silicon nitrides.⁹ The data can also be represented by the Monkman-Grant plot equation ($t_f = A \dot{\epsilon}^{-m}$), which depicts the test results as pooled into two temperature ranges, namely 1149 to 1260°C and 1300 to 1400°C (Figure 43). The stratification of the data of the two groups shows consolidated creep formulations could be developed by multiplying the Monkman-Grant equation with the Arrhenius factor ($\exp(-Q/RT)$). The stress dependence of the time-to-failure is very intriguing (Figure 44), where the data at 1300°C falls in with the 1149 to 1260°C data. At 1300°C, short time failures at high stresses may be caused by SCG and beginning of primary creep, whereas at low stresses and long failure times, the primary failure is most probably from creep rupture of the material. By combining the Monkman-Grant and Norton equation with the Arrhenius factor, life time of components at a given applied stress and temperature can be calculated. This methodology is further discussed and developed in Section 5.

Seven specimens that did not fail during creep rupture testing between 1200 and 1427°C were loaded to failure at room temperature to measure their retained tensile strength. Table 11 summarizes the results and Figure 45 shows the linear stress-strain behavior of the specimens. In general, the room temperature retained strength of the specimens was higher than the material strength at the respective temperatures. The modulus and the strain-to-failure were equivalent to the room temperature values. Other failed specimens tested at these conditions revealed SCG and/or presence of cavitation between 1260 and 1400°C. These data show that when components are used in steady-state conditions in automotive or industrial engines that are periodically shut off, the room temperature fast fracture strength may improve.

Figure 42. Minimum creep rate versus applied stress as a function of temperature.

Figure 43. Monkman-Grant behavior showing time to failure as a function of minimum creep rate (some tests were stopped before failure was reached; however, those with $t_f > 200$ hours were used in the regressions and are included on the plot).

Figure 44. Stress rupture behavior of PY-6 silicon nitride.

Table 11. Room temperature residual strength of uniaxial creep specimens that did not fail during testing.

Specimen ID	Strength —ksi	Strength —MPa	Modulus —msi	Modulus —GPa	Strain to failure	Remarks
635B	69.67	480	50.27	347	0.0014	>233 hr at 149.75 MPa, 1200°C
629A	65.86	454	49.48	341	0.0014	>291 hr at 180.75 MPa, 1200°C
471A	64.46	444	49.04	338	0.0013	>27 hr at 141.22 MPa, 1260°C
634B	44.94	310	49.53	342	0.0009	>624 hr at 150.74 MPa, 1300°C
486B	34.07	235	52.74	364	0.0007	>266 hr at 79.80 MPa, 1400°C
497A	55.27	381	46.28	319	0.0012	>218 hr at 79.85 MPa, 1370°C
535B	64.55	445	49.93	344	0.0013	>74 hr at 95.29 MPa, 1370°C
621A	59.01	407	48.62	335	0.0012	>15 hr at 70.49 MPa, 1427°C

Figure 45. Residual room temperature strength of unfailed button-head tensile specimen during creep rupture testing.

Failure analysis of all the specimens was conducted to ascertain the fracture initiating site using optical micrography. Selected specimens were examined by SEM fractography and TEM to determine the failure mechanism and microstructural composition. Optical micrography of specimens tested at 1149°C revealed fast fracture features (Figure 46). When the specimens failed at short times at high stresses between 1200 and 1400°C, the failure was predominantly controlled by both surface and internal flaws associated with an oxidized SCG zone (Figure 47). Specimens tested at 1200°C at both low and high stresses, and failed in a short time, revealed a differing texture (smooth and torturous) of the fracture surface, possibly indicating change in the failure mechanism such as transgranular to intergranular (Figure 48). At 1260°C, SEM fractography detected the presence of two grain interfacial cavitation in a specimen tested at 175 MPa for 364 hours and examined perpendicular to the loading axis (Figure 49). Similarly, Figure 50 shows a large population of distributed interfacial cavities after 1154 hours at 1260°C. The beginnings of interstitial cavities and viscous flow were observed at 1300°C after 0.013 hours at an applied stress of 275 MPa. A large density of interfacial cavities of approximately 40 nm to 160 nm and a variety of shapes (round, ellipsoidal, irregular shape) were revealed at 164.8 MPa after 4.99 hours, as well as after 770 hours at 175.3 MPa (Figure 51). Similarly, at 1370°C the material exhibited a stressed oxidation damage area along with two grain junction interfacial cavitation and softening of the bulk material (Figure 52). Fractography disclosed a similar failure mechanism at 1400°C both at various stresses and life times (Figure 53).

(a)

(b)

TE94-2426

Figure 46. Optical micrographs of specimens tested at 1149°C (a) 249.1 MPa, $t_f = 0.003$ hr, (b) 239.8 MPa, $t_f = 41.9$ hr.

1200° C

$\sigma_f = 190 \text{ MPa}$, $t_f = 0.16 \text{ Hrs.}$

1300° C

$\sigma_f = 178 \text{ MPa}$, $t_f = 0.826 \text{ Hrs.}$

1260° C

$\sigma_f = 185 \text{ MPa}$, $t_f = 0.205 \text{ Hrs.}$

1400° C

$\sigma_f = 174 \text{ MPa}$, $t_f = 0.015 \text{ Hrs.}$

Figure 47. Optical micrographs of button-head creep rupture specimens which failed in less than 1 hr.

VS93-3303
TE94-2427

Figure 48. Typical lower power SEM photographs of specimens tested at 1200°C.

Figure 49. Creep rupture of button-head tensile specimens tested at 1260°C ($\sigma_a = 175$ MPa). Failure time = 364 hr. Initiation of interfacial two-grain junction cavities.

TE94-2430

Figure 50. SEM micrograph of interfacial cavities in specimen 515A tested at 1260°C $\sigma_a = 175.2$ MPa, $t_f > 1154$ hr.

TE94-2431

Figure 51. SEM micrograph of interfacial cavities in specimens tested at 1300°C.

TE94-2432

Figure 52 Typical SEM photos of a specimen tested at 1370°C, specimen No. 84B, $\sigma_a = 73.9$ MPa, $t_f = 287$ hr.

2.89 HRS
110 MPa

45 HRS
86 MPa

844 HRS
75 MPa

VS92-3657
TE94-2433

Figure 53. Excessive creep cavitation in uniaxial tensile specimens tested at 1400°C was detected by SEM. Specimens were prepared perpendicular to the loading axis.

Transmission electron microscopy (TEM) and EDS analysis of several creep tested specimens was conducted at ORNL under an user agreement. Table 12 summarizes the results of the specimens investigated and gives the primary microstructural observations. Fracture surfaces of all specimens were also characterized in the SEM.

Because the originally fractured creep specimens tended to be oxidized, obscuring details of the failure mechanisms, fresh surfaces were prepared for scanning electron microscopy by fracturing the tensile bar to expose a perpendicular surface parallel to the tensile axis and near the original plane of fracture. Specimens were coated with a thin layer of carbon to minimize charging effects, and observed in a Hitachi S-800 field emission gun SEM. This method allowed lenticular cavities that tend to form in two-grain junctions aligned normal to the tensile axis to be observed more easily.

Creep specimens after failure were analyzed by TEM characterization. The as-received sample microstructure was represented by a TEM specimen cut from the butt-end of a specimen that did not undergo high temperature testing. Thin slices were cut from the creep button-head specimens so the subsequent thin foil TEM specimens represented a plane parallel to and containing the tensile axis, and a location within 1 mm of the fracture surface. The thin slices were core-drilled to 3 mm diameter, ground and polished using standard techniques (a graded series of diamond polishes from 30 mm to 1 mm), dimpled using a Gatan dimpler, and ion-milled to perforation using a Gatan Duo Ion Mill with argon ions at 6 kV and 12 deg incidence.

The electron transparent specimens were observed using either a JEOL 2000 FX analytical electron microscope or a Hitachi HF-2000 field emission gun (FEG) analytical electron microscope. Both microscopes were operated at 200 kV, and EDS were recorded using a Kevex thin window detector on the 2000 FX or a Noran thin window detector on the HF-2000. EDS data were analyzed using NIST desk-top spectrum analysis software run on a Macintosh computer. A probe size of 20 nm was obtained on the 2000 FX, while the FEG of the HF-2000 permitted a

Table 12. Summary of TEM and EDS results of PY-6 creep specimens

Sample No.	Temperature—°C	Treatment time—hr	MPa	Observations
351-A	1200	As-received, butt end	175	Glassy GB phase
636-A		>389		Minor interstitial voids, Polyx-tal GB
463-A		364		Beginnings of two-grain cavitation (heater failed at 146 h), GB single x-tal, individual pockets are different single x-tals
626-B	1300	>473	175	Number two-grain cavitation, but much triple point voids. X-tallized GB in glass matrix in pockets (pockets not fully x-tallized)
192-B	1400	844	75	Two x-talline GB phases, one is $Y_2Si_2O_7$, other is $Y_4Si_3O_{12}$

probe size of 2 nm to be obtained. For quantitative analysis, the Cliff-Lorimer ratio method for thin foil quantitation was used. In this method, the ratio of compositions of any two elements in a thin specimen is related to the intensity of characteristic X-ray peaks from the two elements by a "k-factor" so $CA/CB = k_{AB}(IA/IB)$. k-factors were obtained using a standard of $Y_2Si_2O_7$ previously characterized by X-ray diffraction methods. Quantitative results were obtained from the as-received specimen and from a specimen tested at high temperature and low stress. Spectra from all other specimens were qualitatively compared to these results. In general, spectra were acquired from grain boundary pockets near a hole edge, so the specimen area would be thin enough to minimize any chance of sample self-absorption effects to occur. However, since the SiK α peak at 1.74 keV and the YLa peak at 1.92 keV are almost linearly self-absorbed, this was found not to be a problem with these analyses.

General microstructural and EDS observations are described below:

As-Received Specimen, 351-A: This specimen showed a simple microstructure of β - Si_3N_4 grains in a glassy matrix or grain boundary phase (GB). Because the electron beam in point mode tended to damage the GB glass, EDS spectra were obtained from GB pockets containing considerable glass so the beam could be spread to 5 nm or so. This optimized the potential to obtain spectra representative of the existing material. Figure 54 shows a typical spectrum from the $Y_2Si_2O_7$, and the spectrum obtained from the glassy GB phase in sample 351-A. The correspondence of spectra suggests that the GB composition is, though still a glass, consistent with the $Y_2Si_2O_7$ composition. Full quantitation of the glassy GB phase in 25 separate pockets gave a composition (normalized to yttrium) of Y: 1.00 ± 0.0 , Si: 1.099 ± 0.025 and O: 3.69 ± 0.05 . This is an excellent match to the composition $Y_2Si_2O_7$ (the oxygen results are less reliable because oxygen X-rays are more easily absorbed by the heavier elements).

Specimen 636-A (1200°C, 175 MPa): This specimen ran 389 hours without creep failure. Some interstitial voids were noted in the grain boundary pockets in the SEM, Figure 55 (a). The GB phase showed significant crystallization, with a polycrystalline structure developing. Some residual glass was found in the GB pockets. Figure 55 (b) shows a typical pocket bounded by β - Si_3N_4 grains. The large upper grain is oriented to a $\langle 001 \rangle$ zone axis, so the boundary with the GB pocket is viewed edge-on. EDS spectra were acquired from the labeled spots. They showed a variation in composition from grain to grain, but in general two compositions were seen. Figure 56 shows the spectrum from area A, which is consistent with compositions of areas B, E, and F. Figure 57 shows the spectrum of area C which was the same as area D. For completeness, Figure 58 (a) shows the spectrum of the β - Si_3N_4 grain and Figure 58 (b) shows the hole count, or spectrum acquired with the beam placed in the hole in the specimen. This latter spectrum shows there are essentially no significant contributions to the sample spectra from spurious or systems peaks generated in some microscopes from improperly collimated radiation originating in the upper column of the instrument. Note the carbon peak, which varies from point to point, likely originates from the unavoidable buildup of contamination when the probe is focused to a fine point for acquisition of spectra with high spatial resolution.

Specimen 463-A (1260°C, 175 MPa): This specimen ran for 364 hours to failure at a temperature slightly elevated over that of sample 636-A. Interestingly, the major GB pockets in this specimen were essentially totally recrystallized but showed large single crystals instead of a fine-grained polycrystalline microstructure as seen in 636-A. Figure 59 shows an extensive region of GB pocket material surrounded by β - Si_3N_4 grains. Figure 60 (a) and (b) show the diffraction patterns obtained from areas A and B. These patterns are identical, showing that the pocket material is a large single crystal extending entirely around a small beta grain within the pocket. Figure 61 (a) shows the EDS spectrum from area A can be compared to the spectrum from a standard $Y_2Si_2O_7$ spectrum of Figure 61 (b). The significantly lower Si composition is consistent with a Y_2SiO_5 composition for this crystalline phase.

Figure 54. (a) $\text{Y}_2\text{Si}_2\text{O}_7$ standard spectra, 10 spectra collected; (b) as received PY-6 Specimen 3561A spectra, 25 spectra collected.

Figure 55. (a) SEM fractography and (b) TEM thin-foil photographs of fracture surface Specimen 636A tested at 1200°C, 175 MPa, > 389 hr.

Figure 56. EDS spectra of Specimen 636A in four spots showing identical composition.

Figure 57. EDS spectra of Specimen 636A showing similar composition in two areas (c) and (d).

Figure 58. (a) Spectrum of β -Si₃N₄ and (b) hole count in spectrum.

TE94-2439

Figure 59. TEM photograph of a region with a β -Si₃N₄ grain surrounded by grain boundary phase in location A, B, and C. Specimen 463A tested at 1260°C under 175 MPa and failed after 364 hr.

Figure 60. Diffraction patterns of areas A and B indicating presence of identical material in Specimen 463A.

Figure 61. Comparison of (a) EDS spectra of area A and (b) EDS spectra of Y₂Si₂O₇ standard.

Figure 62 (a) shows a two-grain junction with an apparent crack, and at higher magnification (Figure 62 [b]), it is apparent that some lenticular voids are beginning to form. The EDS spectrum from area A in this figure is shown in Figure 62 (c). A small peak of Y is seen along with the expected Si and N peaks from the β -Si₃N₄ material. A trace of O is also evident, indicating the two-grain junction originally had some of the Y-Si-O phase, probably amorphous, present. Only a few of these areas were seen in this specimen; they suggest just the onset of cavitation in this sample.

Specimen 192-B (1400°C, 75 MPa): This specimen ran 844 hours to creep failure, under test conditions of high temperature but low stress level. Fractographic analysis of this specimen indicated extensive creep damage and cavitation and viscous flow of the GB material (Figure 63). The nominal cavity size ranged from 40 nm to 300 nm with varying aspect ratios. EDS analyses of this specimen showed an interesting bi-modal distribution of composition (Figure 64). Sampled were 40 GB pockets, and when the data were properly grouped the results showed one

Figure 62. TEM micrograph of (a) two-grain junction with a crack, (b) magnification of crack region showing beginning of lenticular cavities, and (c) EDS spectra of region (a) showing Si_3N_4 with traces of yttrium and oxygen.

75 MPa, 844 HRS

VS92-3656
TE94-2443

Figure 63. Fractography of specimen 192B showing interfacial cavitation and grain pull-out.
Test temperature: 1400°C, $\tau_a = 75$ MPa, $t_f = 844$ hr.

Figure 64. EDS spectra of grain boundary pocket in specimen 192B tested at 1400°C.

phase (26 analysis points) to be consistent with $\text{Y}_2\text{Si}_2\text{O}_7$ (Y: 1.00 ± 0.0 ; Si: 1.085 ± 0.7 ; and O: 3.67 ± 0.142). The second phase (14 analysis points) gave a composition of Y: 1.0 ± 0.0 , Si: 0.75 ± 0.024 ; and O: 2.995 ± 0.048 . This composition is consistent with $\text{Y}_4\text{Si}_3\text{O}_{12}$.

3.3.2 Creep Rupture Tensile Testing of Dogbone Specimens

The creep rupture characterization of injection molded and HIPed PY-6 silicon nitride material was performed at the NIST from 1200 to 1400°C using dogbone flat tensile specimens. In creep, 38 specimens were tested, 21 of these were tested to failure yielding data for creep rupture analysis. Test time ranged from 5 minutes to 2500 hours.

Creep rupture testing was conducted using hot grips systems developed and assembled at NIST. Creep deformation was measured continuously and recorded on a IBM-PC computer using a laser extensometer that followed the relative displacement of two flags attached to the gage section of each specimen. The laser extensometer consisted of a detector and HeNe laser (<1 mW). The accuracy of the laser extensometry for measurements at room temperature was better than + 2 micron. Both the detector and the laser were protected from the heat of the furnace by water-cooled plates attached to the front of each unit. A schematic representation of the apparatus is shown in Figure 65. For ease of loading, a clamshell furnace was used in the creep apparatus. The tensile specimen was attached to the two silicon carbide loading rods in a pin and clevis arrangement using sintered alpha-silicon carbide pins. Pin loading reduced flexural strains during testing to less than 2% of the total creep strain. The silicon carbide loading rods extended out of the furnace and were connected via simple universal joints to an air piston that applied the load to the specimen (upper rod) and to a load cell that monitored the applied load on the specimen (lower rod). Using an optical pyrometer, no temperature difference was detected within the gauge section of the tensile specimen. More details of the test methodology are described in Reference 10.

The test conditions and creep data are summarized in Table 13. The creep curves for the specimens are compiled in Appendix VI. The material exhibited both primary and steady-state creep. The nature and extent of the two creep regimes depend on the applied stress and temperature. For example, Figure 66 (a) and (b) depicts specimens tested at 1400°C that failed after 2511 hours at 72 MPa and 23.5 hours at 86 MPa, respectively, with long primary creep regions. In fact, Figure 66 (b) only shows primary transient creep. These data are representative of much of the

Figure 65. Schematic of creep testing set-up at NIST.

Table 13. Creep rupture characterization of dogbone tensile specimens at NIST

Specimen	Temperature— °C	Applied stress—MPa	Time-to failure—hr	Creep rate— x10E (-9)/sec	Percent total strain (ϵ_f)
37R9	1200	250	>2.6	--	>0.04
39R7	1200	275	6.0	34	—
47R8	1200	275	7.8	55	--
47R5	1250	180	4.87	3.82	--
61R3	1250	190	664	1.54	--
39R4	1300	104	>888	0.22	>0.2
40R4	1300	125	96.5	6.24	0.16
46R8	1300	125	>885	2.05	>0.6
37R8	1300	150	63.4	3.21	0.15
63R5	1300	150	>987	0.26	>0.4
39R3	1300	180	1.6	53	<0.01
35R6	1300	180	8.0	43	0.18
36R8	1300	215	0.25	--	--
46R3	1300	215	1	--	--
63R9	1300	215	12.5	44	0.3
39R10	1350	60	1.0	--	--
35R10	1350	72	>1035	1.82	>1.0
37R3	1350	72	>1203	0.07	0.4
35R9	1350	86	527	1.62	0.8
46R7	1350	86	>1034	0.78	>0.76
47R10	1350	104	32.9	13.9	0.2
46R6	1350	104	56.9	22	0.32
36R7	1350	104	68.8	12.7	0.3
65R3	1350	104	>526	0.68	>.5
61R9	1350	104	>887	0.64	>0.58
36R2	1350	125	15.4	104	0.5
37R6	1350	125	24.4	29.2	0.18
47R1	1350	150	1.4	477	0.26
47R2	1350	200	0.08	3050	0.06
63R3	1350	200	0.34	650	0.05
65R4	1400	72	>2511	1.75	>2.2
40R9	1400	86	4.0	--	<0.01
35R1	1400	86	23.5	54	0.7
61R2	1400	86	278.5	9	1.25
39R2	1400	104	3.0	.00	0.35
35R4	1400	104	3.3	296	0.4
63R8	1400	125	1.28	464	0.2
63R7	1400	125	2.25	300	0.25

Figure 66. Typical creep data obtained on NIST specimens. Very long primary transient creep and the absence of tertiary creep before failure is characteristic of silicon nitride.

data presented in the literature. Tensile creep tests on PY-6 silicon nitride suggest this material exhibits a long primary creep stage, which can take up more than half the total creep life. In some grades of silicon nitride, the entire creep life may consist of primary creep¹⁰⁻¹³; in others, steady-state creep may be apparent after one-half the specimen lifetime.^{9, 10, 14} This type of variability was observed in PY-6 material for specimens lasting for 500 to 1000 hours. For such specimens a distinct break was observed at approximately 200 to 300 hours (Figure 67). This change in slope is probably a consequence of devitrification¹⁰ at grain boundaries during annealing. Devitrification modifies the composition of the glass in the grain boundaries and increases its viscosity, thus increasing resistance to creep. Such devitrification is observed in all grades of silicon nitride, unless annealing is used as a part of the manufacturing cycle. The fact a distinct break in the creep curves is observed for some specimens and not for others, suggests the glass composition and structure of the PY-6 was variable from specimen to specimen. This may also be partially responsible for the large variation in time-to-failure with only a small change in applied stress between 1200 and 1400°C in PY-6 silicon nitride material.

The minimum creep rate versus applied stress data is graphically plotted in Figure 68. The curve fit lines from 1300 to 1400°C are nominally parallel to each other, indicating the failure mechanisms are identical in this temperature regime. The data for 1200°C fall far apart from 1300 to 1400°C and are consistent with the findings on button-head specimens observed during this program and the results of other workers.⁹ This effect is attributed to the change in failure mechanisms from creep cavitation to crack growth. In fact, the test results on button-head specimens showed the transition occurs between 1200 and 1300°C. Figure 69 shows stratification in the stress rupture behavior of the material between 1300 and 1400°C, indicating identical failure mechanisms. Monkman-Grant plots show the excellent correlation between the button-head and dogbone specimens with a slope varying between 1 and 1.3 (Figure 70). This is additional proof that the data generated by the two specimen designs is identical within the large scatter, typical characteristic of ceramic materials. Improved quality control and processing may tighten the scatter band and further improve the reliability of silicon nitride materials.

Figure 67. Long-term creep curves showing a sharp break in the slope of the curves. This behavior was observed in about half of the specimens tested and may be due to devitrification at grain boundaries during testing. Test conditions: 86 MPa, 1350°C.

Figure 68. Minimum creep rate versus applied stress.

Figure 69. Applied stress versus time-to-failure.

TE94-2449

$$t_f = A \cdot \dot{\epsilon}^m$$

Figure 70. Creep rupture data follow Monkman-Grant model both for button-head and dogbone specimens.

Microstructural and fractographic analysis were conducted using both SEM and TEM methods. Fractography revealed failure predominantly occurred from surface defects. Microstructural and TEM were conducted to ascertain the nature and composition of the grain boundary along with the fracture controlling mechanism across the test temperature range. Analytical TEM was used to investigate the effect of microstructure on the tensile creep of GTE PY-6 silicon nitride. The results were obtained from selected tensile test samples crept at 1200, 1250, 1350, and 1400°C. At 1400°C, creep lifetimes of examined samples ranged from 3.5 to over 2500 hr and strains ranged from a few tenths of a percent to over 2%. In all cases, TEM samples were made from sections cut parallel to the stress axis; standard ceramographic techniques, including ion milling, were used to obtain thin foils. Primary emphasis was placed on characterizing the cavity damage that develops during tensile creep.

Examination of PY-6 revealed a microstructure consistent with liquid phase sintering, as the silicon nitride grains are surrounded by an intergranular second phase. The silicon nitride grains range in size from a few hundred nanometers to over three microns, tending to be somewhat equiaxed in shape. Compared to other, similar grades of silicon nitride, PY-6 appeared to contain fewer elongated, whisker-shaped grains. As with other grades, the smaller grains tend to be clustered in regions interstitial to the larger grains. These interstitial regions, as well as isolated triple or multi-grain junctions, also contain most of the volume of the intergranular, second phase. Although originally a glass, rapid devitrification at the test temperatures (except 1200°C.) occurs and results in the formation of crystalline nitrogen apatite (H-phase) and delta phase, yttrium disilicate within these interstitial regions. Both direct observation and EDS analysis indicate the presence of narrow (approximately 1 nm) glassy interphases separating most silicon nitride grains. More generally, however, the interface structure in PY-6 was variable, ranging from narrow and glassy (1 nm) to wide and largely crystalline (>10 nm) (due to devitrification). As illustrated in Figure 71, such variations in interface structure occur even along the same interface. Although the composition of the residual glass could not be quantified, EDS results suggested a composition close to yttrium disilicate $Y_2Si_2O_7$.

TE94-2451

Figure 71. Interface separating silicon nitride grains in PY-6. Within central region (G) interphase is glassy and typically about 1 nm thick; within outer regions (X) interphase is thicker (>10 nm) and contains crystalline second phase.

Tensile creep of PY-6 results in various forms of cavitation depending on temperature and applied stress. At 1400°C and stresses above 100 MPa, creep rupture occurs at relatively low strains, apparently due to interfacial cavitation. Two forms of cavities are observed at interfaces aligned normal to the stress axis. At narrow, glassy interfaces cavitation results in high densities of discrete, ellipsoidal cavities of 100 to 300 nm dimensions (Figure 72). Similar interfacial cavities form in NT154, and their nucleation and growth appeared to be identical for both materials. In addition, crack-like cavities of grain-size lengths also form, apparently by rapid diffusive growth, within the largely crystalline second phase originally present within wider interfaces (>10 nm) (Figure 73). In contrast, samples tested at lower stresses at 1400°C, or at comparable stresses at 1350 and 1250°C, exhibit only discrete cavities at narrow, glassy interfaces; crack-like cavity growth was not evident. In addition to interfacial cavitation, large scale (>300 nm) cavities at multigrain junctions were also found in PY-6 after creep at temperatures above 1250°C (Figure 74). As in NT154, the growth of these large cavities results in the removal of the second phase (which is largely crystalline) from multigrain junctions and the density of these cavities increases with strain. Examination of one sample that failed after 8 hours at 1200°C revealed little, if any, cavitation, suggesting failure at this temperature occurred by SCG from pre-existing flaws.

In summary, creep of PY-6 at temperatures of 1250 to 1400°C results in discrete interfacial cavities at narrow (1 nm or less) two grain boundaries and interstitial cavities at multigrain junctions. The nucleation and growth of interfacial cavities is identical to that in NT154. Apparently, limited transport through the nearly "pure" yttrium silicate glass phase unique to these two grades restricts cavity growth into and along the silicon nitride grains and results in the formation of discrete cavities of nearly uniform size. While the average size of these interfacial cavities increases with increasing temperature, the density of cavities both within individual interfaces and overall decreases with increasing temperature. In contrast, the relative extent (as evidenced by cavity size and density) of interstitial cavitation appears to increase with increasing temperature. Most notably, there is a significant increase in the population of large cavity sizes (0.5 to 1.0 microns) with increasing temperature, regardless of strain. As such,

TE94-2452

Figure 72. Crack-like interfacial cavity produced in PY-6 during creep at 1400°C. With reference to Figure 71, rapid cavity growth within wider portions of the interface results in crack-like morphology.

TE94-2453

Figure 73. Pair of interfaces containing discrete, ellipsoidal shaped interfacial cavities typically produced at narrow glassy interfaces (1 nm) in PY-6 during tensile creep. Similar phenomenon is observed in NT154. Interfacial cavitation of this type was found in all samples tested at 1250 to 1400°C. Test conditions for this specimen were 1350°C, 86 MPa, test stopped after 1034 hr. Tensile stress axis is vertical to the figure, i.e., specimen was cut parallel to the tensile axis.

TE94-2454

Figure 74. Large-scale multigrain junction cavity in PY-6. Diffusive transport of second phase from triple and multigrain junctions during creep results in cavities of varied size and shape.

this increase in average cavity size reflects the strong temperature dependence of cavity growth rate within the second phase at multigrain junctions. At 1400°C and sufficiently high applied stresses, rapid cavity growth within the second phase also occurs within relatively wide (10 nm or more) two grain boundaries in PY-6 to form crack-like cavities. While these cavities do not appear to grow with increased strain, their presence should contribute to the creep rupture process.

Button-head and dogbone specimens depicted similar creep behavior of the PY-6 material between 1250 and 1400°C. The data generated by SoRI and NIST, using two vastly differing experimental set-ups, provided similar results within scatter. This indicates that dogbone specimens, when dissected from components, can be utilized to qualify material and processes.

3.4 OXIDATION

The effects of oxidation on the mechanical behavior of structural silicon nitride materials must be fully understood prior to the introduction of these materials in automotive and industrial engine applications. Many researchers have studied the oxidation behavior of silicon nitride materials fabricated by variety of processing methods such as pressureless sintered¹⁵, hot pressed^{16, 17}, and reaction bonding.¹⁸ In addition, many workers also studied the effects of oxidation on the retained MOR strength of these materials.¹⁹⁻²¹ In general, a protective SiO₂ or silicate layer is formed due to "passive" oxidation on the surface of the specimens which retards the rate of oxidation of the material. The formation of the oxide layer on nonoxide ceramics follows linear, parabolic, logarithmic, power law, or mixed growth laws.²² The applicability of each law depends on the composition and recrystallization behavior of the surface layer. A homogeneous layer with constant properties and composition over time will follow the parabolic law, while a constant change in the properties of the layer with time will result in logarithmic, power law, or a mixture of both. In real life applications, no one law is followed exactly. Severe active oxidation of silicon nitride normally occurs at low oxygen partial pressure, resulting in weight loss due to the formation of gaseous silicon monoxide (SiO). At low temperatures, oxidation has been reported to improve the strength of silicon nitride material by healing the surface or blunting surface flaws, whereas at elevated temperatures strength decreases due to the formation of new flaws during thermal exposure.

During this program, oxidation studies were conducted in a static air environment from 1000 to 1400°C. A total of 130 Type-B specimens were exposed at various temperatures from 1 to 500 hours in a programmable Lindberg furnace. Dimensions and density of all the specimens were measured. The weight before and after heat treatment was measured with an accuracy of ± 1 mg. The specimens were examined by microfocus X-ray before and after heat treatment. The specimens were placed in the furnace on alpha silicon carbide bars to facilitate air flow around each specimen. Retained strength of the specimens was measured in 4-point bending at room temperature with an outer span of 40 mm and inner span of 20 mm at a cross-head speed of 0.508 mm/min (0.020 in./min). The thickness of the oxide layer was measured from SEM micrographs of the fractured specimens. The weight change per unit surface area, oxide layer thickness, and room temperature retained strength of all the specimens were measured as a function of time and temperature.

NDE of the specimens was conducted by film microradiography after oxidation but before mechanical testing. The results indicated that surface forming small pits within the oxide layer were very difficult to discern from the background fussy X-ray image of the oxidized specimen. The tensile surface of five selected specimens was examined by acoustic microscopy at 50 MHz, detecting many singular or clustered surface anomalies (Figure 75). These anomalies were developed at the surface due to the oxidation of the material. The acoustic image of one MOR bar surface had many more clusters than the other four, indicating the microstructural chemistry variations in the specimens effect the oxidation kinetics. Based on these results, it was concluded

Figure 75. Surface wave acoustic imaging of oxidized MOR bar specimens.

that both microfocus X-ray and acoustic microscopy were unsuitable techniques for detecting and discerning discrete fracture initiating defects in oxidized PY-6 silicon nitride MOR specimens.

The test conditions and average measured results of the oxidation study are summarized in Table 14. Substantial scatter was observed in the weight change, both as a function of temperature and time with no apparent trend. This may be due to variations in the material microstructure, subtle variability in the grain boundary chemistry, or bulk impurities such as iron (Fe) significantly altering the diffusion kinetics of species²³ and, consequently, the oxidation behavior of the material. The thickness of the oxide layer increased linearly with increasing temperature for specimens exposed for 100 hours (Figure 76). Limited data after 500 hours exposure indicated some acceleration in the linear growth rate of the oxide layer thickness at 1300 and 1400°C (Figure 76). The presence of distributed iron impurity in this material is postulated to decrease the dissociation or oxidation temperature of silicon nitride and increase the transport rate of oxygen through the material, causing a rapid rate of oxidation. At 1200 and 1400°C, the oxide layer thickness can be represented both by a logarithmic and power function of time equations, respectively (Figure 77). In general, both functional forms provide similar results within scatter and experimental error. The data at various temperatures can be combined with the Arrhenius factor ($\exp(-Q/RT)$) and an empirical relationship between the oxide layer thickness, exposure time, and temperature can be established. Similarly, the room temperature retained flexural strength of the material decreases as a function of temperature after 100 hours of exposure (Figure 78). After 500 hours, however, the strength either did not change or increased up to 1200°C due to surface oxide layer buildup which sealed the surface. Above 1200°C, the retained strength decreased after 500 hours, indicating formation of pits or fracture of the surface oxide layer due to thermal expansion mismatch between the substrate and the surface layer. Section 5 of this report describes the theoretical basis and assumptions behind a proposed oxidation model.

Table 14. Average oxidation behavior of PY-6 silicon nitride in air

Temperature—°C	Exposure time—hr	Number of specimens	Weight change per unit area —mg/cm ²	Oxide layer thickness —μM	Flexural strength		Summary of failure analysis
					ksi	MPa	
1000	100	5	0.0000	0.6	123.09	848.72	SFC(2), SI(1), SF(1), UI(1)
	500	5	0.0000	0.0	164.03	1131.00	SFC(1), SF(4)
1100	100	5	0.0163	1.0	104.26	718.89	SFC(2), SF(3)
1150	100	5	0.2692	1.6	96.00	661.93	SFC(3), SF(1), II(1)
1200	1	5	0.0163	0.5	106.85	736.73	SFC(2), SF(3)
	3	5	0.0217	1.3	91.06	627.86	SFC(1), SF(4)
	6	5	0.0514	0.7	69.64	480.20	SF(5)
	10	5	0.0108	0.8	78.39	540.52	SF(5)
	50	5	0.0081	1.4	95.26	656.79	SFC(2), SF(3)
	100	5	0.4025	1.4	107.28	739.67	SFC(2), SF(3)
	200	5	0.0298	1.8	88.68	611.45	SFC(2), SF(3)
	500	5	0.0299	1.8	102.30	705.35	SFC(1), SF(4)
1260	100	8	-0.2620	2.0	94.86	654.03	SFC(3)
1300	100	5	0.3383	2.6	103.10	710.61	SFC(1), SF(4)
	500	5	0.0259	3.1	74.90	516.41	SF(4), SI(1)
1371	100	8	0.0694	2.3	98.08	676.30	SFC(3), SF(5)
1400	1	5	-0.5297	3.4	108.31	746.77	SFC(4), SF(1)
	3	5	-0.0510	2.2	113.26	780.96	SFC(5)
	6	5	0.1027	2.8	109.56	755.45	SFC(3), SF(1), II(1)
	10	5	0.0636	1.4	104.92	723.41	SFC(2), SF(3)
	50	5	0.8552	2.4	101.47	699.64	SF(4), UI(1)
	100	5	0.2548	3.2	99.14	683.55	SFC(1), SF(4)
	500	5	-0.2374	4.8	92.36	636.80	SFC(1), SF(4)

SFC - Surface machining induced flaw at the corner

SF - Surface machining flaw

SP - Surface pore

SI - Surface inclusion

II - Internal inclusion

IP - Internal pore

UI - Unidentified or lost origin

Figure 76. Oxidation behavior of PY-6 Si_3N_4 as a function of temperature after 100 and 500 hours exposure in air.

TE94-2457

Figure 77. Effect of time and temperature on oxide layer thickness in MOR bars. Data fitted to (a) logarithmic law and (b) power law.

Figure 78. Room temperature MOR retained strength decreases after 100 hours oxidation in air as a function of temperature.

The failure of the specimens predominantly occurred from the inside corner of the MOR bar within the inner span. SEM fractography of specimens exposed at 1000°C for 100 hours and 500 hours showed no surface pitting or debonding of the surface oxide layer with the silicon nitride substrate (Figure 79). Specimens exposed at 1200°C for 100 hours showed near-surface degradation of the material as well as disbonding of the surface layer from the silicon nitride substrate, which decreased the strength of the material (Figure 80). The pitting in the oxide layers, randomly observed at 1400°C, further decreased the strength (Figure 81).

X-ray diffraction studies revealed the presence of cristobalite, β - Si_3N_4 , and β -yttrium disilicate in the oxide layer of specimens. The as-received material primarily had only β - Si_3N_4 phase (Figure 82 [a]). After 100 hours exposure at 1000°C, β - $\text{Y}_2\text{Si}_2\text{O}_7$ and β - Si_3N_4 phases were detected (Figure 82 [b]). The α - $\text{Y}_2\text{Si}_2\text{O}_7$ phase is stable between 25 and 1225°C, whereas β - $\text{Y}_2\text{Si}_2\text{O}_7$ phase is stable in the 1225 to 1445°C temperature range. Both at 1300 and 1400°C, several peaks of cristobalite and β - $\text{Y}_2\text{Si}_2\text{O}_7$ along with β - Si_3N_4 were observed (Figure 83). It was also discovered that the amount of cristobalite increased with a corresponding decrease in the silicon nitride phase, indicating the dissociation of silicon nitride in the presence of oxygen at ambient pressure after 100 hours as a function of temperature (Figure 84). Cristobalite is known to undergo a reversible alpha-to-beta polymorphic transformation with about 5.4% volume change resulting in surface residual stresses, which may cause cracking of the surface oxide layer and disbonding from the silicon nitride substrate.

3.5 MULTIAXIAL TESTING

Gas turbine components are normally subjected to multiaxial stresses during operation, requiring understanding of the material behavior under complex stress states. During this program,

100 HOURS, $\sigma_f = 976.3$ MPa

500 HOURS, $\sigma_f = 1202.4$ MPa

VS94-0928
TE94-2459

Figure 79. Fractography of 1000°C oxidized MOR specimens tested at room temperature.

$\sigma_f = 740.04 \text{ MPa}$

$\sigma_f = 693.7 \text{ MPa}$

VS94-0927
TE94-2460

Figure 80. Oxidation of PY-6 Si₃N₄ at 1250°C after 100 hours in air.

100 HOURS, $\sigma_f = 541.3$ MPa

500 HOURS, $\sigma_f = 608.5$ MPa

VS94-0926
TE94-2461

Figure 81. Oxidation of PY-6 at 1400°C after 100 hours and 500 hours in air (note surface pitting).

Figure 82. X-ray diffraction of MOR specimens (a) as received and (b) after 100 hours exposure at 1000°C.

Figure 83. X-ray diffraction of MOR surface after 100 hours at (a) 1300°C and (b) 1400°C. Both cristobalite and B-Y₂Si₂O₇ were detected.

Figure 84. X-ray diffraction shows increase in the amount of cristobalite after 100 hours of oxidation in air as a function of temperature (note decrease in β -Si₃N₄ content).

efforts were concentrated toward measuring the biaxial fast fracture strength, mixed-mode fracture toughness, and biaxial creep rupture behavior of the PY-6 silicon nitride material at room and elevated temperatures. The data base was utilized to develop ceramic life prediction methods to predict performance of the various type of verification test articles and assess their correlation with the experimental results. All the material characterization for this task was conducted at Battelle Research Laboratories, Columbus, Ohio.

3.5.1 Biaxial Fast Fracture Strength

Biaxial fast fracture strength of 18 circular disks at room temperature was measured at Battelle Laboratories, using a ball-on-ring arrangement at a loading rate of 0.25 mm/min (0.01 in/min) (Figure 85). The surfaces were first rough machined until the thickness was within 0.125 mm (0.005 in.) of the final thickness. The last 125 micron (0.005 in.) was removed using a 400 grit wheel at a 1.25 mm (0.050 in.) cross feed and 7.5 micron (0.0003 in.) feed per pass. Specimens were flooded with coolant during machining. Disks were rotated after each pass to provide a uniformly machined surface. This provided specimens with a 400 grit surface finish. The machined surface was referred to as the tensile surface for testing. The opposite surface was just rough ground to maintain flatness and thickness within dimensional requirements. The nominal diameter of the machined disks was 3.182 cm (1.25 in.) and 0.253 cm (0.100 in.) thickness. In these experiments the support radius was 25.4 mm and the loading ball radius was 6.35 mm. Figure 85 shows the experimental set-up and the related equations to calculate the strength of the disk. Test results are summarized in Table 15. The average measured strength was 727 MPa (105.4 ksi) with a standard deviation of 44.9 MPa. The Weibull modulus (m) was calculated to be 17.2 with a characteristic strength of 776 MPa using MLE method with suspended item analysis. In general, the disk specimens typically failed in five or six segments (Figure 86). Examination of the samples by low-power microscopy did not show any significant differences in the fracture

$$\sigma_{max} = \frac{3P(1+\nu)}{4\pi t^2} \left[1 + 2 \ln \frac{a}{b} + \frac{(1-\nu)}{(1+\nu)} \left\{ 1 - \frac{b^2}{2a^2} \right\} \frac{a^2}{R^2} \right]$$

TE94-2465

Figure 85. Biaxial strength measurement of a circular disk specimen using ball-on-ring test.

Table 15. Fast fracture biaxial strength of PY-6 silicon nitride circular disks at 25°C.

Specimen number	Diameter— cm	Thickness— cm	Strength— MPa	Failure origin
3-1	3.185	0.251	759.4	SF
3-2	3.183	0.251	715.6	SF
3-9	3.188	0.257	765.7	SF
3-23	3.180	0.251	705.5	SF
3-34	3.183	0.249	714.5	SF
3-37	3.183	0.251	671.8	SF
3-38	3.178	0.251	775.6	SF
3-77	3.185	0.251	755.0	SF
4-81	3.185	0.254	813.4	SF
4-82	3.178	0.254	768.0	SF
4-83	3.183	0.254	685.1	SF
4-84	3.185	0.254	782.0	SF
4-86	3.185	0.255	846.5	SF
4-88	3.178	0.253	825.9	SF
4-90	3.178	0.253	779.7	SF
4-92	3.185	0.253	750.6	SF
4-93	3.180	0.253	736.3	SF
4-98	3.185	0.254	720.7	SF
Average	3.182	0.253	754.0	
Standard deviation	0.003	0.002	45.86

(a)

(b)

TE94-2466

Figure 86. Fast fracture biaxial testing of disks at room temperature (a) failed in six pieces and (b) failed in five pieces.

morphology between the specimens. All the specimens failed from surface flaws. Fractographic analysis revealed the failure originated from machining damage and propagated parallel to the grinding marks (Figures 87 and 88). It is evident that biaxial strength measurement is sensitive to the surface condition of the specimen.

Flexural specimens were dissected from biaxial disks to compare the Weibull properties of the two types of specimens. The surface of the biaxial disks was first prepared to simulate a standard biaxial test specimen as described above. Subsequently, two specimens, nominally $31.75 \times 5.08 \times 2.54$ mm in size, were prepared from each disk. A total of 20 specimens were prepared and tested at room temperature in four-point bending using an outer span of 27.94 mm and inner span of 16.76 mm at a loading rate of 0.25 mm/min (0.01 in./min). The results of the testing are summarized in Table 16. The average transverse strength of the bars was measured to be 638.4 MPa (92.6 ksi) with a standard deviation of 35.2 MPa. The failure of the specimens initiated from surface machining defects or damage, as observed for the biaxial disks. The Weibull modulus (m) of 20.2 was calculated by the MLE method with a corresponding characteristic strength of 655 MPa. The Weibull modulus of 17.2 for the disk and 20.2 for the bars indicates reasonable agreement within scatter between the two types of specimens with similar surface finishes. The failure in all the specimens initiated from a surface machining flaw and propagated parallel to the grinding marks similar to the disks.

TE94-2467

Figure 87 . Low magnification scanning electron micrographs of silicon nitride biaxial tested specimens.

TE94-2468

Figure 88 . High magnification scanning electron micrographs of silicon nitride biaxially tested specimens.

Table 16. Fast fracture strength of MOR specimens machined from circular disks.

Specimen number	Depth— cm	Width— cm	Strength— MPa	Failure origin
4-61A*	0.2535	0.5095	614.26	SF
4-61B*	0.2525	0.5098	602.86	SF
4-63A	0.2522	0.5098	604.07	SF
4-63B	0.2522	0.5095	627.35	SF
4-65A	0.2525	0.5093	651.64	SF
4-65B	0.2525	0.5093	665.41	SF
4-71A	0.2515	0.5090	613.28	SF
4-71B	0.2565	0.5093	611.16	SF
4-72A	0.2532	0.5090	586.44	SF
4-72B	0.2522	0.5090	604.97	SF
4-74A	0.2515	0.5090	620.22	SF
4-74B	0.2515	0.5090	597.08	SF
4-76A	0.2517	0.5090	669.78	SF
4-76B	0.2515	0.5090	608.65	SF
4-77A	0.2517	0.5093	692.53	SF
4-77B	0.2515	0.5093	682.36	SF
4-78A	0.2517	0.5090	681.33	SF
4-78B	0.2515	0.5090	671.13	SF
4-80A	0.2507	0.5077	676.91	SF
4-80B	0.2507	0.5093	686.52	SF
Average	0.2521	0.5092	638.4	
Standard deviation	0.0012	0.0004	35.2	

* - Two specimens "A" and "B" were machined from each disk.

SF - Surface machining defect

Type-B MOR bars were also transversely machined and characterized in four-point bending to assess their strength and compare their Weibull behavior with the two Allison prepared test specimens mentioned above. First, the bars were rough machined using a 180 grit wheel until the specimens dimensions were within 50 to 75 micron of the desired dimensions. Subsequently, a 320 grit wheel was used to machine the remaining material at a cross feed rate of 250 $\mu\text{m}/\text{sec}$ to remove 2.5 μm (0.0001 in.) material per pass. The edges of the bars were then beveled. The four-point flexural strength was measured with an outer span of 40 mm and inner span of 20 mm at a cross-head speed of 0.508 mm/min. The results of the testing are summarized in Table 17. The average strength was measured to be 652.2 MPa (94.59 ksi) with a standard deviation of 101.0 MPa. The Weibull modulus (m) was calculated by the MLE method to be 7.96 with a characteristic strength of 702 MPa. All the specimens fractured from machining induced surface defects.

Table 17. Fast fracture MOR strength of transverse ground Type B specimens at room temperature.

Specimen number	Width— mm	Thickness— mm	Strength— ksi	Strength— MPa	Failure origin
34.2171	4.0157	3.0378	116.47	803.03	SF
34.2172	4.0183	3.0353	100.90	695.72	SF
34.2173	4.0157	3.0328	84.37	581.71	SF
34.2174	4.0183	3.0404	91.19	628.73	SF
34.2175	4.0157	3.0328	89.08	614.23	SF
34.2176	4.0183	3.0328	89.08	614.23	SF
34.2177	4.0132	3.0302	86.14	593.92	SF
34.2178	4.0157	3.0429	70.79	488.11	SF
34.2179	4.0107	3.0277	119.50	823.97	SF
34.2180	4.0132	3.0302	104.00	717.05	SF
34.2181	4.0157	3.0277	88.86	612.67	SF
34.2182	4.0183	3.0277	94.05	648.51	SF
34.2183	4.0157	3.0277	88.86	612.67	SF
34.2184	4.0183	3.0378	105.95	730.55	SF
34.2185	4.0157	3.0429	92.13	635.26	SF
34.2186	4.0208	3.0378	87.11	600.61	SF
34.2188	4.0157	3.0353	100.97	696.16	SF
34.2189	4.0183	3.0353	90.45	623.63	SF
34.2190	4.0107	3.0302	105.64	728.37	SF
34.2211	4.0183	3.0328	87.46	603.01	SF
34.2212	4.0183	3.0328	79.08	545.23	SF
34.2213	4.0157	3.0302	110.75	763.64	SF
34.2214	4.0157	3.0302	119.68	825.17	SF
34.2215	4.0157	3.0302	79.26	546.49	SF
Average			94.59	652.22	
Standard deviation			14.65	100.99	

SF - Surface machining defect

Figure 89 shows the two-parameter Weibull distribution and curve fit line for four test specimens including the fast fracture longitudinally ground specimens. The data indicate the biaxial disks and MOR specimens dissected from the similarly machined disks provide almost identical Weibull modulus. The sensitivity of the Weibull modulus to the surface finish indicates the material data for component design must be obtained from identically machined surface finished specimens. Changes in the surface finish can cause large variations in Weibull properties and thus significantly effect component failure and survival probability predictions.

VS93-3934
 TE94-2469

Figure 89. Comparison of fast fracture strength of biaxial disks and transverse ground bars.

3.5.2 Mixed Mode Fracture of Through-Cracks (Chevron Notch)

Mixed-mode fracture toughness of silicon nitride was measured at room temperature, 1200, and 1300°C with chevron notch (CN) disk specimens (Figure 90). The testing involved compressing the disk specimen diametrically, and noting the maximum load at unstable fracture in the case of fast fracture experiments. The room temperature testing and analysis followed the procedure described by Shetty, et. al and others.²⁴⁻²⁶ In this test, the specimen is placed with the notch at an acute angle to the loading axis and loaded along a diameter in compression, with the proportions of tensile and shear stress on the crack being controlled by choice of the angle. By rotating the plane of the notch at different angles with respect to the loading axis, the mode mixity (quantified through the ratio K_{II}/K_I) can be varied over a wide range; here K_{II} and K_I are the Mode II and Mode I stress intensity factors, respectively). The CN produces stable crack growth during loading, so there is a sharp crack in the specimen at the point of unstable fracture. This is unlike the case of single-edge notch beam geometries, where the sharpness of the machined notch can influence the measured fracture toughness of the material.

The CN disks were loaded to failure at a cross-head speed of 4.2 $\mu\text{m}/\text{sec}$ at four angles from 0 to 23 deg with the loading axis at room temperature. At times, the crack became unstable prior to complete fracture of the chevron region for specimens that underwent oblique loading resulting in crack deflection. In such cases, the crack length at fast fracture was determined from the examination of the fracture surface. The specimen dimensions and data are reported in Table 18.

Figure 91 shows the fit of the data to the expression describing typical mixed-mode behavior:²⁷

$$K_{Iq}/K_{Ic} + (K_{IIq}/CK_{Ic})^2 = 1$$

Figure 90. The chevron notched, diametrically compressed crack disk specimen.

Table 18. Summary of diametral compression experiments at 25°C

Specimen	Angle —deg	Diameter —in.	Thickness —in.	Crack length— in. (a)	Load —lb	K_{Iq} — MPa√m	K_{IIq} — MPa√m
3-27	0	1.253	0.1000	0.625(b)	800	6.18	0
3-48	0	1.253	0.0999	0.625(b)	855	6.61	0
3-26	0	1.251	0.0999	0.625(b)	815	6.32	0
3-72	9	1.253	0.1003	0.45	1150	5.63	4.44
3-47	9	1.251	0.1002	0.46	1110	5.55	4.40
3-25	9	1.251	0.1008	0.36	1200	4.98	3.75
3-51	14	1.252	0.1004	0.42	1150	4.28	6.22
3-61	14	1.253	0.0998	0.42	1180	4.42	6.41
3-45	23	1.254	0.1005	0.36	1180	1.54	8.26
3-52	23	1.253	0.0991	0.39	1120	1.47	8.50
3-53	23	1.251	0.0998	0.36	1220	1.60	8.63

(a) Crack length at onset of unstable growth.

(b) Crack length between ends of chevron; other specimens failed before chevron had completely cracked.

Figure 91. Mixed-mode testing of chevron notch disk specimens in diametral compression at room temperature with through-cracks.

where K_{Iq} and K_{IIq} are the opening and shear fracture toughness components in mixed-mode loading, while C is the ratio of stress intensity at failure in pure shear (K_{IIc}) to pure-opening-loading toughness (K_{Ic}). For the silicon nitride tested here, K_{Ic} is $6.4 \text{ MPa m}^{1/2}$ and $C = 1.57$. The K_{Ic} value obtained for this silicon nitride is comparable to NT154 and other silicon nitrides of the same class. The value of C is in the mid range of brittle materials.²⁸

High temperature fracture toughness was measured at 1200 and 1300°C using the CN disk specimen shown in Figure 90. The specimen was heated in a compact setup utilizing induction heating (Figure 92). In this setup, two SiC platens were used to compress the disk specimen; compression achieved on a tensile creep frame using special fixtures. The specimen was induction heated utilizing a tubular SiC susceptor. The tubular susceptor had a slit of approximately 0.25 inch width along its entire length, the slit allowing for visual observation of the crack in the specimen. An insulating felt was wrapped around the susceptor to reduce heat loss from the susceptor and to prevent heating of the induction coils, which were at close proximity of the susceptor. A telescoping arrangement was used to monitor crack growth, although the glow at very high temperature made crack length observation difficult; sometimes the sample was cooled before viewing the crack. A thermocouple was used to monitor and control temperature. The photograph in Figure 93 shows the experimental arrangement.

In fast fracture testing, a sharp crack is automatically generated at the point of unstable fracture. Thus, in Mode I, the crack length at the point of instability is $2a_f$ (Figure 92) and this value is used along with the maximum load to determine the fracture toughness (K_{Ic}) of the material. Under mixed mode loading, the crack may become unstable before reaching $2a_f$, and in that case the crack length at fast fracture is determined from examination of the fracture surface; the length is larger than $2a_1$ but less than $2a_f$.

Figure 92. Experimental setup to conduct high temperature mixed-mode fracture toughness testing.

TE94-2473

Figure 93. Photograph of experimental system to measure elevated temperature fracture toughness using chevron notch disk specimens.

The notched specimens were placed in the above fixture with notch at either 0 or 23 deg angle to obtain pure bending or pure shear loading. A slight load was placed on the specimen to maintain the angle during heating and temperature stabilization. In some cases the specimen was held at load for a period, unloaded prior to fracture, and examined microscopically for any crack growth. No stable crack growth was detected in any of these cases. Disk specimens with CN were tested at 1200 and 1300°C at the two angles. The results are shown in Table 19. At 1200°C, the average K_{IC} was found to be $4.5 \text{ MPa} \cdot \text{m}^{1/2}$ and at 1300°C was calculated to be $5.4 \text{ MPa} \cdot \text{m}^{1/2}$.

Table 19. Summary of diametral compression fast fracture testing at elevated temperatures.

Specimen number	Temperature—°C	Angle—deg	Diameter—in.	Thickness—in.	Crack length—in.	Load—lb	K _{Iq} —MPa·m ^{1/2}	K _{IIq} —MPa·m ^{1/2}
3-30	1200	0	1.25	0.1053	0.625	673	4.96	0
3-40	1200	0	1.25	0.0808	0.540	520	4.30	0
34.2273	1200	0	1.25	0.0800	0.625	585	5.67	0
3-29	1200	23	1.25	0.0800	0.625	371	0.29	5.59
3-81	1200	23	1.25	0.0805	0.630	441	0.31	6.74
3-84	1200	23	1.25	0.0800	0.625	431	0.32	6.56
34.2271	1300	0	1.25	0.8000	0.620	562	5.40	0
34.2272	1300	0	1.25	0.8000	0.630	552	5.40	0
34.2274	1300	0	1.25	0.8000	0.630	564	5.52	0
34.2251	1300	23	1.25	0.0803	0.625	500	0.37	7.58
34.2252	1300	23	1.25	0.0800	0.630	517	0.37	7.95
34.2253	1300	23	1.25	0.0798	0.630	501	7.72	7.72

3.5.3 Mixed Mode Fracture of Surface Cracks (Vickers Indents)

Circular disk specimens were surface cracked using a Vickers indenter and a load, $P = 40$ kg, in order to measure the fracture toughness associated with controlled surface flaws. A hardness impression and the associated cracks are shown in Figure 94. The dimensions in this micrograph provide a toughness estimate; using hardness of 14.5 GPa (obtained from the indent size of Figure 94), the fracture toughness estimate is calculated from the formula:

$$K_{Ic} = 0.16 (P/a^{3/2})(E/H)^{1/2}$$

where $2a$ is the indentation crack length. The K_{Ic} estimate is $5.1 \text{ MPa} \cdot \text{m}^{1/2}$ or about 80% of the large crack value measured using the diametral-compression specimens. This result is consistent with the general indentation toughness experience, according to which data are generally 80 to 100% of the through-crack value. More accurate values are obtained by fracturing the indented specimen.

When the indented disks were loaded in diametral compression, it was found that failure occurred at the bearing surfaces because of the very high toughness of the silicon nitride. Therefore, the disks were cut into MOR bars with the indents at their centers in order to obtain data. The bars were loaded to failure at a rate of $8.5 \times 10^{-4} \text{ mm/sec}$ in a specially-designed four-point-bend fixture which minimizes experimental errors. The results are summarized in Table 20. Failure originated at the indents on all specimens. Most cracks ran either along the indent diagonal or very close to the diagonal. The one specimen, where the crack ran along one edge of the indent, exhibited somewhat higher strength than the others.

Combining the average fracture strength for the zero degree cracks with the indent load and applying Chantikul, et al.'s equation for strength of an indented specimen²⁹

$$K_{Ic} = 0.59 (E/H)^{1/8} (\sigma P^{1/3})^{3/4}$$

Figure 94. Vickers indentation and associated cracks on the surface of MOR specimens dissected from circular disks with a surface finish similar to disks tested for fast fracture biaxial strength. The MOR specimens were tested with varying angles of the cracks with respect to the loading axis.

Table 20. Strength of Vicker's indented surface cracked silicon nitride MOR bars at 25°C.

Specimen number	Angle—deg (a)	Width—in.	Thickness—in.	Failure load—lb	Strength—MPa
3-15(b)	0	0.2001	0.0944	80.5	217
3-55(b)	0	0.2001	0.0943	80	200
3-70	0	0.1998	0.0947	75	217
3-71(b)	15	0.1999	0.0950	69	230
3-22(c)	15	0.2000	0.0944	75.5	231
3-11	30	0.2001	0.0948	92	264
3-43(b)	30	0.2000	0.0945	81.5	235
3-60(b)	45	0.2002	0.0940	95	277
3-59	45	0.2000	0.0940	88	257
3-56(d)	45	0.2004	0.0945	110	317

- (a) Smaller angle between the line perpendicular to specimen axis and one diagonal of the hardness impression.
- (b) Crack ran close to the diagonal of the indent; in all other specimens the crack ran along the diagonal.
- (c) Crack ran along one edge of the indent.
- (d) Indent was about 0.050 in. off-center in the width direction.

K_{IC} was found to be $6.7 \text{ MPa} \cdot \text{m}^{1/2}$, which is about 5% higher than the through-crack value obtained using the disk specimen. Figure 95 shows variation in strength with the orientation of indenter cracks relative to the tensile axis. The line used to fit the data is based on the following equation expressed in strength terms:

$$\sigma_f \cos^2 \Theta / \sigma_u + (\sigma_f \sin \Theta \cos \Theta / C \sigma_u)^2 = 1$$

where Θ is the shallowest crack angle between the maximum tensile stress direction and one indenter diagonal, σ_f is the corresponding tensile fracture strength, and σ_u is fracture strength for $\Theta = 0$. The advantage of the above is that the crack length at fracture (which likely is larger than the indented crack length because of the contribution of residual stresses) does not enter into the equation. As shown in Figure 95, the value of C for surface cracks was between 1.25 to 1.5 (depending on the data reduction scheme used) compared to 1.57 for the through-cracks discussed earlier. Physically, the lower value of C indicates a greater sensitivity of small flaws to shear stresses than large cracks. However, it is important to note the failure load of an inclined crack is not very sensitive to differences in C ; for example, the 20% difference in C between surface (assuming $C = 1.25$) and through-cracks translates into a 10% difference in failure loads for $\Theta = 45$ deg.

Figure 96 shows the effect of temperature on the ratio $C = K_{IIc}/K_{IC}$. Obviously, the value of C at 1200°C is lower than at room temperature or at 1300°C . The temperature dependence of K_{IIc} is similar to K_{IC} since Figure 96 suggests the ratio is fairly insensitive to temperature. This result implies shear loadings have the same qualitative effect on toughness as tensile loadings, which is not unexpected for an elastic material, since loadings induce an asymmetric tensile stress field at the crack tip, whose magnitude is comparable to the opening-mode stress field. These shear-induced tensile stresses also are the cause of the apparent decrease in opening-mode fracture toughness with an increasing shear component of loading.

Figure 95. Mixed mode testing of MOR bars with surface cracks (Vickers indents) at room temperature.

Figure 96. Shear-mode/opening-mode fracture toughness ratio.

Two other mechanisms affecting toughness should be mentioned: rubbing and bridging. Both effects decrease the crack-tip stress intensity. Rubbing occurs when the crack-opening displacement is smaller than the asperities on the opposing crack faces.³⁰ This effect comes into play at low Mode I/Mode III ratios and causes K_{IIc} values associated with low K_{Ic} levels to be higher than they would be in the absence of rubbing. If rubbing were absent, the K_{IIc}/K_{Ic} ratio would be closer to unity than the values reported here. The occurrence of bridging at 1000°C and above has been suggested by Wereszczak, et. al.⁵ to account for rising R curves. This mechanism occurs when the crack bypasses some grains, leaving behind an island of unbroken material that restrains crack opening and further crack advance. Like rubbing, this mechanism reduces the stress intensity and increases the apparent toughness.

3.5.4 Biaxial Creep

Biaxial creep and stress rupture behavior of circular disks of 31.75 mm (1.25 in.) diameter and 2 mm (0.080 in.) thickness was studied at Battelle Laboratories using a ball-on-ring arrangement. The schematic of the test facility is shown in Figure 97. The specimen was supported by silicon carbide tubes, plate, and ring of 15.625 mm (0.625 in.) diameter at the bottom. The specimen was loaded with a 4.826 mm (0.190 in.) diameter silicon nitride ball at the top and pushed by a silicon carbide rod. The testing was conducted in a resistance furnace with molybdenum di-silicide elements and capable of going up to 1700°C. Displacement was measured by a spring-loaded LVDT probe with an alumina arm at the bottom center of the disk; however, contact with the specimen was with a silicon nitride point attached to the alumina arm in order to reduce unwanted reactions with the specimen. A photograph of the experimental biaxial creep measurement system is shown in Figure 98. Experiments were conducted at 1200, 1300, and 1400°C. The loading fixture, with the specimen in place, was held at temperature for approximately 1/2 hour in order to equilibrate the system. Dead weight loading was increased at a loading rate of 2 lb/sec until either the specimen broke or the selected creep load was reached. The experiments were performed in air for up to 200 hours, after which they were considered runout.

TE94-2477

Figure 97. Test setup for biaxial creep experiments using ball-on-ring arrangement; heated in furnace with MoSi_2 elements.

TE94-2478

Figure 98. Setup for the biaxial creep experiments.

Tables 21 and 22 show the test data at 1200 and 1300°C for three types of tests. One group of specimens failed at a very short time at load or while loading. In the second group, one specimen at each temperature was step loaded to ascertain the stress envelope for testing. The third group of specimens consisted of constant load creep tests. Figure 99 is a typical load-displacement curve for specimens which failed in a short time at 1200 and 1300°C. The load/time curve is linear since the tests were conducted in load control mode. Figure 100 shows a typical load/displacement record for a typical creep test, with a linear regression fit line through the data. The flat creep rate line shows the material is creep resistant at these temperatures and test setup. Figure 101 shows the stress rupture plot at 1200 and 1300°C. The first step loading results at both temperatures exceeded the lowest short time to failure or fast fracture stress. Incremental loading to beyond the highest fast fracture stress produced no failure at 1200°C, even though the total time under stress was over 200 hours. At 1300°C, the step-loaded specimen broke after 74 hours at stress above strengths of 7 of the 8 fast fracture (short time failure) specimens, having previously survived 72 hours at lower stresses. The average stress of the specimens which broke after short time test at 1200°C was about 549 MPa and at 1300°C was 500 MPa. Comparison of these results with the room temperature measured strength of 727 MPa clearly shows the material was degraded at elevated temperatures. Figure 102 shows cracks (bright white regions, see arrows) at the center of the tensile surface of two specimens tested at 1300°C. Dark field microscopy provided better definition of microcracks than under the bright-field

Table 21. Biaxial creep behavior of PY-6 silicon nitride at 1200°C.

Specimen number	Thickness— in.	Applied load—lb	Applied stress— MPa	Displacement rate—in./sec	Failure time—hr
34.2266	0.071	191	515	7.9E-5	0.025
34.2267	0.070	234	639	8.0E-5	0.033
34.2269	0.070	228.5	624(a)	7.0E-5	0.032
34.2560	0.084	250	436(a)	(b)	-
34.2610	0.070	195	533	(b)	0.025
34.2562	0.084	200(d)	349(d)	(b)	>200
34.2577	0.100(e)	252(d)	350(d)	(b)	>200
34.2270	0.070	180(d)	492(d)	7.2E-9	>168
34.2240	0.070	180(d)	492(d)	6.8E-9	>246
34.2250	0.070	Step (c,d)	Load (c,d)	-	(c)

(a) Broke on loading.

(b) Lvdrt malfunction.

(c) Stress (MPa)/time (hrs) history was: 491/24; 518/24; 573/24; 600/72; 627/8; 655/19; 682/9; 709/19.

(d) Specimen unloaded without fracture.

(e) Support ring diameter was 0.920 in., in all other experiments support ring diameter was 0.625 in.

Table 22. Biaxial creep rupture behavior of PY-6 silicon nitride at 1300°C.

Specimen number	Thickness— in.	Applied load—lb	Applied stress— MPa	Displacement rate—in./sec	Failure time—hr
34.2242	0.0908	297	429	(a)	0.042
34.2244	0.0800	200	391	(a)	0.11
34.2268	0.0703	278(b)	>759	8.9E-5	(b)
34.2600	0.0704	218	595	(a)	(c)
34.2601	0.0704	205	560	(a)	4.2E-3
34.2609	0.0700	201	458	(a)	(c)
34.2611	0.0700	209	570	8.8E-5	0.025
34.2241	0.0915	299(b)	327	(a)	>200
34.2243	0.0908	220(b)	318	(a)	>200
34.2247	0.0800	182(b)	355	3.2E-8(e)	>360
34.2563	0.0800	204(b)	380	2.6E-9	>304
34.2564	0.0800	222(b)	430	8.3E-10	>456
34.2567	0.0800	235(b)	460	(a)	>231
34.2583	0.0704	200(b)	539	(a)	>220(f)
34.2619	0.0790	188(b)	387	8.3E-10(e)	>75
34.2260	0.0706	Step 230	Load 614	(a)	74(d)

(a) Gage malfunction.

(b) Unloaded before fracture.

(c) Broke during loading.

(d) Prior to final load specimen was held successively at 480, 530, and 555 MPa for 24 hr each.

(e) Erratic displacement record: tabulated rate is final displacement divided by test time.

(f) Furnace problems during test.

Figure 99. Representative load/displacement/time record for fast fracture tests at 1200 and 1300°C.

Figure 100. Representative load/time/displacement/time record for creep tests at 1200 and 1300°C.

TE94-2481

Figure 101. Biaxial stress rupture (a) 1200°C and (b) 1300°C.

(a)

(b)

(c)

TE94-2482

Figure 102. Dark field optical photograph at the center of the tensile surface (a) Specimen 34.2563, (b) Specimen 34.2564, and (c) is the bright field photograph of Specimen 34.2564 showing grinding marks and creep cracks.

conditions. In Figure 102 (a), the finer lines in the background that are oriented in the 7 o'clock - 1 o'clock direction are grinding marks. Although some of the cracks are along those grinding marks, there are crack branches which do not follow grinding marks, thus, unlike room temperature fracture tests, where the crack initiation appeared to be along the grinding direction, crack initiation in creep appeared not to be influenced as much by the grinding direction. Figure 102 (c) shows the bright field of counterpart of Figure 102 (b), and shows clearly that the grinding direction had no influence on the growth morphology of creep cracks.

The data at 1400°C are assembled in Table 23. Typical load/displacement curves for short time-to-failure or fast fracture and for longer failure times are shown in Figure 103. The total displacement is much higher than the corresponding testing at 1200 and 1300°C, clearly indicating increased creep deformation at this temperature. Figure 104 shows the stress-rupture time curve fit line showing the material life increased with decreasing applied stress, which is typical of ceramic materials.

IV. NONDESTRUCTIVE EVALUATION DEVELOPMENT

Structural silicon nitride test specimens and components usually fail from both surface and volume defects. The flaws are introduced during the various stages of the fabrication process such as molding, drying, debinder removal (if any), sintering, and machining. The most common flaws present in today's high strength silicon nitride materials are iron (Fe) based inclusions, naturally occurring small voids, cracks, and machining induced surface damage. The first two types of flaws are found both on the surface and within the volume of the material, whereas the machining induced defects (e.g., cracks and grinding damage) occur only on the surface. The inclusions result from Fe impurities in the starting powder or from abrasion of the molding machinery. Nondestructive detection of these defects can screen out defective parts and substantially reduce variability, offering the potential for increased operating stresses. Additionally, the a priori knowledge of the defect size, distribution, and characteristics could potentially provide important information about the flaw population that affects the probability of detection (POD), intrinsic material strength, slow crack growth, and large scatter in time-to-failure during stress rupture testing and component evaluation.

Table 23. Biaxial creep rupture behavior of PY-6 silicon nitride at 1400°C.

Specimen number	Thickness— in.	Fracture load—lb	Fracture stress— MPa	Displacement rate—in./sec	Failure time— hr
34.2248	0.070	272	742	1.1E-4	0.042
34.2249	0.070	274	747	1.2E-4	0.053
34.2258	0.069	218	612	1.0E-6	8.3
34.2259	0.070	210	573	4.2E-6	1
34.2261	0.069	190	533	1.7E-6(a)	4.7
34.2262	0.070	175	477	(b)	100

(a) Erratic displacement recorded; tabulated rate is final displacement divided by test time.

(b) Gage malfunction.

Figure 103. Representative load/displacement curves for (a) short time to failure and (b) long time to failure at 1400°C.

TE94-2483

Figure 104. Biaxial stress rupture behavior at 1400°C.

During this program, nondestructive evaluation (NDE) techniques including visual, fluorescent penetrant inspection (FPI), microfocus X-ray radiography, and acoustic microscopy were used to examine MOR bars, button-head and dogbone tensile specimens, and biaxial disks prior to testing. The NDE development efforts concentrated primarily to (i) fabricate reference specimens, (ii) optimize microradiography inspection parameters, (iii) develop a 100 MHz acoustic microscopy (AM) data acquisition and imaging system, (iv) optimize the AM parameters for surface and volume inspection of specimens, and (v) determine the defect detection capability of both the monochromatic and polychromatic X-ray computed tomography (CT) techniques.

4.1 FABRICATION OF REFERENCE SPECIMENS

Three type of reference specimens, namely MOR bars, button-head tensile specimens, and biaxial disks, were designed and fabricated to optimize various NDE technique parameters. Calibration standards of single crystal C-axis sapphire were fabricated for ultrasonic imaging to determine the surface and subsurface resolution of the acoustic microscopy technique.

4.1.1 Reference Standards with Surface Defects

Five MOR bars with 5 micron to 100 micron diameter and varying aspect ratio surface holes were fabricated as shown in Figure 105. Table 24 shows the layout of each bar. The holes were drilled by an Excimer laser at Resonatics, Inc., using programmed x-y location of each hole. The laser parameters were developed for each size and depth of hole to create a quality straight hole. Optical micrography revealed chatter and damage on the surface around the hole. In addition, the bottom of the holes was tapered which decreased their effective depth and volume. Similar holes of 10 to 100 micron diameter and varying depth were induced in the gage area of the uniaxial button-head specimens. One specimen had 50 holes each of 25, 50, and 100 micron diameter and depth, Figure 106. Another specimen, Figure 107, had 19 holes each of 10, 25, and

Figure 105. Layout of excimer laser-drilled holes on the surface of as-machined MOR specimen.

Figure 106. Layout of surface-drilled holes in tensile specimens.

REGION #	DIA. x DEPTH μm	# OF HOLES
1	50 x 50	19
2	25 x 25	19
3	25 x 10	19
4	10 x 20	19
5	10 x 10	19

Figure 107. Layout of surface-drilled holes in tensile specimens.

VS91-1611
TE94-2487

Table 24. Dimension and layout of laser-drilled holes in MOR bars.

Specimen number	Pattern number	Zone number	Hole diameter— μm	Hole depth— μm
76 & 77	1	1	5	5
		2	5	10
		3	5	20
79 & 81	1	1	10	5
		2	10	10
		3	10	20
82 & 83	8	1	25	25
		2	25	50
283 & 284	8	1	50	25
		2	50	50
285 & 287	11	1	100	100

50 micron diameter with varying aspect ratios to ascertain the effect of the depth on flaw detection. Optical micrography, Figure 108, disclosed that the holes were irregular and did not have flat bottoms in the button-head specimens. Biaxial circular disk were machined to the required surface finish for multi-axial testing by Battelle. Subsequently, laser-drilled surface holes of 25 to 100 micron diameter and depth (aspect ratio = 1) were induced to simulate a standard for disk inspection prior to testing, Figure 109.

4.1.2 Reference Standards with Volume or Internal Defects

Fabrication of reference standards with isolated internal or volume defects has been a major historic problem in structural ceramic materials. During this program, internal defects in Type-B MOR bars, button-head tensile specimens, and rectangular 15 mm thick billets were simulated with laser-drilled and pilot holes. The pilot holes were induced by Bullen Ultrasonics, Eaton, Ohio, and laser-drilled holes by an Excimer laser at Resonatics, Inc, respectively. The MOR specimens with laser-drilled holes of 10 to 100 micron diameters and 250 micron to 2 mm depth were fabricated with an aspect ratio of ranging from 1 to 290 to assess the subsurface detection capability. Reference specimens with pilot holes and laser-drilled holes in flat rectangular billets were fabricated. Four zones with seven pilot holes each were fabricated at various depths ranging from 0.5 mm to 3 mm. Excimer laser-drilled holes, each with a 1 mm depth and either a 50, 100, or 250 μm diameter, were drilled into the pilot holes. Button-head tensile specimens with pilot/laser-drilled holes were also similarly fabricated.

100 X 100 μm

50 X 50 μm

VS91-1605
TE94-2488

LASER DRILLED HOLES: NOT FLAT BOTTOM VARIABLE DIAMETER/DEPTH

Figure 108. Photograph of laser-drilled surface holes showing damage around the holes.

TE94-2489

Figure 109. Biaxial disk with simulated surface holes. Fifty (50) holes of each size were produced.

4.1.3 Fabrication of Ultrasonic Imaging Resolution Standards

Lithographic methods have been used for creating surface and subsurface void arrays in many high temperature ceramic materials such as glasses, structural ceramics, and ceramic-metal interfaces. The combination of photolithographic methods with ion beam etching and hot pressing provides the methodology to fabricate reference standards with internal voids. These methods summarized here are described in greater detail elsewhere.^{31, 32} Single crystal C-axis (perpendicular to flat faces) sapphire was used to make the resolution standards since its ultrasonic velocity is similar to that of silicon nitride. Single crystal wafers were acquired from Meller Optics. The specimens were prepared at the University of California, Berkeley.

Two 2 in. x 2 in. square single crystal C-axis sapphire wafers (2 mm and 4 mm thick) were sectioned to produce 25.4 mm by 25.4 mm (± 1.5 mm) pieces for lithographic processing, ion beam etching, and bonding. Three reference specimens with subsurface voids at 2 mm, 4 mm, and 8 mm below the entry surface were produced. One 4 mm thick piece was used to produce a surface standard.

Using a mask containing a 1951 Air Force Resolution Pattern, the desired defect pattern for NDE studies was transferred to four 25.4 mm by 25.4 mm wafers of the 2 mm and 4 mm thickness for the standards. Changes in the photoresist composition and the method of exposure required modification of the previously used conditions for exposing and developing the photoresist. After developing the photoresist, ion beam milling was used to prepare a patterned sapphire substrate. Optical microscopy was used to verify the pattern quality. A profilometer was used to determine the etched feature depth, to assure a pattern depth within the 0.2 to 0.9 μm range as planned. Samples were then cleaned to remove organics and residual trace metals using standard microfabrication cleaning procedures.

Etched and unetched substrates were configured to produce a 25.4 mm by 25.4 mm bicrystal. Three assemblies were prepared to produce a 2 mm/2 mm (2/2), a 2 mm/4 mm (2/4), and a 4 mm/4 mm/2 mm (4/4/2) bonded sample. Bonding conditions involved applied stresses of the order of 10-20 MPa for periods of 1 to 4 hr at temperatures between 1150-1300°C. After cooling, optical microscopy was used to determine the quality of the bond. Relatively more difficulty was encountered in producing well bonded interfaces than in previous studies of this type. As a result, for two of the three bonds it was necessary to repeat the hot pressing of the samples to improve the quality of the interface. This second pressing die improved the area fraction bonded, but for the 2/2 specimen induced the development of a twin (Figure 110).

Three bonded and one surface sapphire standards for acoustic microscopy, containing the desired interfacial defect structure, were produced. Bonded standards (with buried test patterns) were produced by the sequential application of photolithography, ion beam etching, and hot pressing.

4.2 MICROFOCUS RADIOGRAPHY

A Feinfocus microfocus 160 kV and 2 mA system, Figures 111 and 112, was used to optimize the flaw detection sensitivity. This system has 6 deg of freedom, which includes linear translation in x,y,z directions and two angular motions (0 and $\emptyset$) around the Z-axis. In addition, the linear translation of the imaging plane lends itself to provide the 6th deg of freedom. The system has a beryllium window. Kodak-M type film was used for recording the images.

A parametric study was conducted to optimize the microfocus X-ray image contrast to detect small flaws. Two PY-6 silicon nitride 3 mm thick bars were taped together in a step manner and radiographed at various accelerating voltages (kV) at 10x. The film density in the region of one bar was kept constant to around 2.7 by changing the current and film exposure. The film density of the two bar region was measured as a function of kV. The difference in the film density at two successive kVs was determined. Figure 113 shows that 70 kV was the optimum voltage at which there was maximum density difference, i.e., maximum contrast, which is required for detecting

USAF TARGET 1951

NUMBER OF LINES PER MILLIMETER IN USAF
RESOLVING POWER TEST TARGET 1951

GROUP NUMBER							
ELMT NO.	0	1	2	3	4	5	6
1	1.00	2.00	4.00	8.0	32.0	32.0	64.0
2	1.12	2.24	4.49	8.98	17.95	36.0	71.8
3	1.26	2.52	5.04	10.1	20.16	40.3	80.6
4	1.41	2.83	5.66	11.3	22.62	45.3	90.5
5	1.59	3.17	6.35	12.7	25.39	50.8	102.
6	1.78	3.56	7.13	14.3	28.51	57.0	114.

VS93-3952 TE94-468

Figure 110. Sapphire reference standard for ultrasonic image calibration.

Figure 111. Real-time microfocus X-ray system.

Figure 112. Schematic of ceramic component inspection technique by projection microradiography using Feinfocus X-ray system.

Figure 113. Optimization of X-ray image contrast for the inspection of MOR bars and biaxial disks.

small fracture controlling flaws in structural ceramic materials. The density difference between 70 kV and 77 kV accelerating voltage was around 1.6%. This voltage range provided the maximum contrast.

Further defect detection and contrast optimization studies were performed on MOR bars with laser-drilled surface holes by varying the X-ray tube voltage, current, exposure time, and magnification of the image. It was experimentally established that 78 kV and 0.2 mA resulted in optimum detection of surface holes, which is consistent with the contrast studies described earlier. Table 25 summarizes the results obtained on various type of specimens. X-ray detected 50 micron diameter and 50 micron deep holes with a thickness sensitivity of 1.69% and equivalent penetrometer sensitivity (EPS) of 1.19% in MOR bars. Microscopy revealed that the subsurface quality of the laser-drilled holes was very irregular; they were shallower in depth than planned, were drilled at an angle, and not flat at the bottom at all. This resulted in detection of 43 holes out of 50. The 25 micron diameter holes were not detected because of their shallow depth and lower than anticipated effective volume. Khandelwal³³ has demonstrated detection of 25 micron surface holes and internally seeded voids using the same experimental system and Kodak-M film. The radiographic detection of the holes in button-head specimens depends on their radial orientation and location with respect to the X-ray beam because of their irregular shape, size, and cumulative geometrical effects. Only 100 micron nominal diameter and depth holes were detected in the gage region with a thickness sensitivity of 2.13% and EPS of 1.5%. Holes in the biaxial disk were detected with a thickness sensitivity of 1.42% and EPS of 1%. However, the 32 out of 50 holes of 50 micron diameter were only detected compared to 43 in MOR bars. This may be due to the increased thickness of the biaxial disks and/or the variable quality of the holes.

4.3 ACOUSTIC MICROSCOPY

Failure of high strength ceramic material normally is initiated from naturally occurring and machining induced surface defects. One of the most sensitive NDE techniques to characterize such surfaces is acoustic microscopy. The fracture controlling flaw size in today's silicon nitride is very small (1 to 50 μm). A 100 MHz computer controlled acoustic microscopy (AM) system was

Table 25. Detection of surface holes by microfocus X-radiography.

Specimen type	<u>Nominal flaw*</u>		<u>Number of flaws</u>		<u>Percent — thickness</u>	
	Diameter— μm	Depth— μm	Present	Detected	Sensitivity	EPS
MOR bar (15x) T = 3 mm	100	100	100	100	3.38	2.39
	50	50	50	43	1.69	1.19
Tensile (2x) T = 4.76 mm	100	100	50	36	2.13	1.50
Biaxial disk (8x) T = 3.5 mm	100	100	50	50	2.85	2.01
	50	50	50	32	1.42	1.0

*Actual holes: Not flat bottom, shallow depth, angular, irregular

developed and implemented at Allison to characterize PY-6 ceramic test specimens prior to testing (Figures 114 and 115). This system utilizes Panametrics 100 MHz pulser/receiver, single shot 100 MHz peak detector with Zero-Span auxiliary equipment which increases the dynamic range of the peak detector output, 50 MHz transducers, and a 486/33 MHz Dell computer control data acquisition and imaging software. The system utilizes a PixelWorks imaging board (1280 x 768), Panametrics Multiscan version 2.1 software for data acquisition, image processing and display on Mitsubishi 15 in. Diamond monitor Model HL6605 (1280 x 1024). The system is equipped with a 232 megabyte hard drive, a 44 megabyte Bernoulli removal cartridge drive system, and an Fujitsu 128 megabyte magneto-optical disk drive with removal disks to store large amounts of C-scan imaging data. The image storage capability of the system enables precision scanning of specimens to detect minute defects, as necessary. The immersion tank, 16.5 x 15.5 x 11.5 in., was fabricated from anodized aluminum. The scanner has x, y, z, and θ axis of scanning. All the axes are programmable and computer controlled. The scanning speed of the system can be varied up to 6 in./sec with a resolution of at least 5 μm in x, y, and z directions, respectively. Specimens can be inspected at a rotory speed of 360 deg/sec with 0.005 deg resolution. C-scan setup and image data can be acquired and stored on the disk in real time. It can be presented on the screen or hard copy generated using a Seiko Model 5504 color printer. A 50 MHz 0.2 in. (5.08 mm) focal length surface wave transducer ($F\# = 0.8$) was predominantly used for the inspection of specimen surfaces.

Acoustic microscopy (AM) examination of the MOR bar, button-head tensile specimen, and biaxial disk reference standards with surface holes was conducted. MOR specimens with surface holes were examined using a 50 MHz transducer with a focal length of 5.08 mm and crystal diameter of 6.35 mm ($F\#0.8$). The specimens were scanned parallel to their length and machining direction. The transducer was incremented in the width direction. Acoustic microscopy detected 25 x 25 μm size holes with clarity (Figure 116). Smaller 10 microns holes could not be discerned

Figure 114. Schematic of 100 MHz acoustic microscopy system.

Figure 115. A 100 MHz acoustic imaging system developed at Allison for the inspection of ceramic specimens.

from the background microstructure signal of the longitudinally machined surface. The button-head tensile specimens with 10 to 100 micron surface holes were also evaluated by acoustic microscopy using the F#0.8 transducer. The specimens were radially scanned by rotating the specimen and keeping the transducer stationary. Subsequently, the transducer was incremented to a new axial location and the specimen was rotated and inspected. Figure 117 shows a C-scan image of a tensile reference standard, shown in Figure 106, which illustrates that all the 50 holes each 25, 50, and 100 μm sizes were readily detected by AM. Another tensile specimen standard, Figure 107, with 10 to 50 micron size holes specimens was also characterized. All the 10 micron diameter x 20 micron deep holes were detected (Figure 118). Acoustic microscopy detected 25 μm diameter holes in the biaxial disk specimen. The results for all three types of specimens are summarized in Table 26. Microfocus X-ray detected 50-100 micron surface flaws whereas acoustic microscopy detected 10 micron surface voids. Acoustic microscopy detected 25 x 25 micron surface holes in the biaxial circular disk (Figure 119) using the setup for MOR bars. Comparative analysis of the detectability of surface holes in three different surface finish specimens by acoustic microscopy was significantly affected by the surface finish (Table 26) as expected. The surface finish of the tensile and biaxial specimens was better than MOR bar specimens. Ten micron diameter holes were detected in tensile specimens compared with 25 micron holes in MOR bars.

Acoustic microscopy detected 25 micron diameter and deep subsurface holes readily in MOR bars using a 50 MHz transducer with a focal length (F) of 12.7 mm and a diameter (d) of 6.35 mm (F#2.0) at a water path of 12.7 mm (Figure 120). Smaller holes of 10 micron nominal diameter could not be discerned from the background microstructure. Again, the holes were imperfectly drilled as described previously. The rectangular billet with deeper subsurface holes was examined using a 25 MHz nominal frequency 3 in. in focal length transducer with a water path of about 2.75 mm. The 50 μm diameter holes were readily detected at a subsurface depth of 14 mm (0.55 in.) (Figure 121).

Table 26. Detaching of surface holes by acoustic microscopy.*

Specimen type	<u>Nominal hole size**</u>		Flaws present	Flaws detected
	Diameter— μm	Depth— μm		
MOR bar	100	100	100	100
	50	50	100	50
	25	25	50	49
Tensile	100	100	50	50
	50	50	50	50
	25	25	50	50
	25	10	19	19
	10	20	19	18
	10	10	19	9
Biaxial disk	25	25	49	49

*Transducer: 50 MHz, F#0.8

**Actual flaw shape/size were uncontrolled

TE94-2494

Figure 116. Detection of 25 μm x 25 μm surface holes in MOR bars.

Figure 117. Detection of 25-100 μm diameter and depth surface holes in the gage area of the but-ton-head tensile specimens.

Figure 118. Detection of 10 μm diameter holes in the gage area of the button-head specimen by surface wave acoustic microscopy (AM).

Figure 119. Acoustic microscopy detected 25 μm surface holes in the biaxial disks.

TE94-2496

HOLE #	HOLE LOCATION FROM TOP SURFACE (d), mm
1	<0.25
2	0.25
3	0.50
4	0.75
5	1.00
6	1.25
7	1.50
8	2.00

NOMINAL HOLE DIAMETER = 25 μ m

Figure 120. Acoustic microscopy detected micron internal hole 2 mm below the surface.

HOLE #	HOLE DEPTH, mm		DISTANCE FROM SURFACE (d)
	COUNTERBORE	LASER	
1	3.00 mm	1.00	11.00 mm
2	2.75 mm	1.00	11.25 mm
3	2.50 mm	1.00	11.50 mm
4	2.00 mm	1.00	12.00 mm
5	1.50 mm	1.00	12.50 mm
6	1.00 mm	1.00	13.00 mm
7	0.50 mm	1.00	13.50 mm
8	0.00 mm	1.00	14.00 mm

NOMINAL COUNTERBORE DIAMETER = .500 mm
 NOMINAL LASER HOLE DIAMETER = 0.050 mm

Figure 121. Ultrasonic detected 50 μ m diameter holes 14 mm below the surface.

Sapphire resolution standards (25.4 mm x 25.4 mm) were examined by acoustic microscopy to determine the spatial resolution capability of separating surface and subsurface defects. Three reference specimens were evaluated: (i) surface etched pattern on a 4 mm wafer; (ii) 2 mm/2 mm specimen with pattern at the interface; and (iii) 4 mm/2 mm specimen with pattern on the 4 mm wafer. Optical micrography measured the depth of the surface etched pattern on the 4 mm wafer to be between 0.2 μm and 0.9 μm . The specimen was evaluated using the 50.0 MHz, F#0.8 transducer at a water path of 5.04 mm with an increment of 12.5 micron (Figure 122). The image data were collected by focusing the beam at the water-sapphire interface and gating the reflected signal. The -6dB focal spot diameter at the interface was calculated to be approximately 30 micron. Magnification of the image on screen revealed that Group 3 Element 6 line pairs 70 μm apart were resolved, which is consistent with the literature.³⁴ The shallow depth of the pattern made it difficult to resolve smaller line-pairs. The second sapphire standard (2 mm/2 mm) with the pattern 2 mm below the surface was evaluated using a 50 MHz transducer with a 12.7 mm focal length and crystal diameter of 6.35 mm (F#2.0). The transducer was focused at the bonded interface of the two wafers where the etched pattern was located. The scanning increment was 12.5 micron at a water path of 1 mm, which was experimentally optimized for best results. The line pair in Group 2 Element 2 with 223 micron separation were resolved (Figure 123). Many other voids were also detected because of somewhat poor bonding of the sapphire wafer in the unetched area. In addition, the twin developed during fabrication also was detected. The third sapphire target (4 mm x 2 mm) was interrogated by entering the acoustic beam from the 4 mm wafer side. Here, a 50 MHz nominally 3.81 cm focal length transducer was focused 4 mm below the surface at the two wafer bonded interface. The scanning increment was 25 micron and the water path was 12.7 mm. The line-pair in Group 1 Element 6 were resolved at distance of 280 microns (Figure 124).

Figure 122. Acoustic C-scan in age of the surface etched sapphire wafer. No image processing was performed. On magnification, Group 3, Element 6 line-pair 70 μm apart was resolved.

Figure 123. Acoustic microscopy detected 223 micron apart line-pair 2 mm below the surface in a sapphire standard.

Figure 124. Acoustic microscopy resolved Group 1, Element 6 line-pair 4 mm below the surface. The line-pair was 280 micron apart.

4.4 COMPUTED TOMOGRAPHY (CT)

The feasibility of using computed tomography (CT) to nondestructively examine ceramics has been tested in a number of studies.³⁵⁻⁴² CT was found to be a very successful technique for measuring the uniformity of ceramic material and long-range density gradients³⁵⁻³⁸, for detecting voids and cavities in ceramic objects^{35, 36}, as an aid in developing finite element modeling⁸, and for evaluating ceramic behavior during processing.^{39, 40, 42} CT has been used for the evaluation of ceramics at various processing stages, and for detecting different types of defects.

Of interest in ceramics are distributed defects and localized defects. Distributed defects can be nonuniformities in material composition and/or material densification that appear as density gradients. Localized defects include 2-D defects, such as cracks, and 3-D (volumetric)

defects, such as voids, cavities, and inclusions. CT is especially valuable for examining distributed defects, being practically the only technique that can measure density gradients with accuracy required in high-quality ceramics, nondestructively, and with information on defect geometry. CT is also a promising method for detecting 2-D and 3-D localized defects (cracks, voids, and inclusions): it can provide information about defect geometry and is potentially much more sensitive than other techniques, such as radiography.

To apply CT as a method of quality control, one has to evaluate its sensitivity quantitatively. The determination of the size of the smallest defects that can be detected and measured in a CT image is particularly important for structural ceramics, and especially when the defect is smaller than the CT spatial resolution. This is because structural ceramics are replacing traditional materials in a variety of critical applications, while critical defect sizes in structural ceramics are small, usually smaller than the CT scanner resolution. Critical defect sizes are in a range of a few dozen μm (hundredths of a mm), while the spatial resolution of typical CT scanners is in the range of 1 mm or 0.1 mm. The question of whether CT sensitivity is sufficient to detect small defects in ceramics, and more generally, the CT defect detectability limits for small localized defects, is therefore of crucial importance.

During this program, the problem was theoretically and experimentally evaluated. A detailed theoretical study was conducted at Chalk River Laboratories under the guidance of Dr. B. D. Sawicka to ascertain detectability limits of voids and iron-based inclusions that are commonly found in today's silicon nitride materials. Experimental studies were also conducted to assess flaw detection sensitivity and resolution capabilities of two CT technologies, namely the Aracor Tomoscope System and Chalk River Nuclear Laboratories Ir-192 isotope based system.

4.5 REFERENCE STANDARD EVALUATION BY ARACOR TOMOSCOPE CT SYSTEM

The Tomoscope Microfocus CT System (TOMO) installed at the Air Force Wright Laboratory, Dayton, has a microfocus X-ray radiation source that provides a 200 kVp (195 nominal) polychromatic X-ray beam at 0.45 mA. The current capabilities for the TOMO include data correction and image enhancement software, up to 2048 by 2048 pixel reconstructed image data sets, slice thickness from 50 microns to 1 mm, and a resolving power of 25 microns. TOMO is a third generation scanner that obtains data by rotation only (Figure 125). The specimen or component to be inspected is mounted on a turntable located between the X-ray fan beam source and the detector array. The data are collected after each incremental rotation and stored digitally on the computer. The data are subsequently processed and reconstructed using sophisticated algorithms to generate a CT image.

A reference specimen with simulated laser-drilled holes of 25 to 500 micron diameter and 3 mm deep were made in a rectangular billet of PY-6 silicon nitride with a maximum diagonal dimension of 88 mm (Figure 126). Five holes of each of the five diameters were fabricated at a constant depth of 3 mm. The large holes, 250 μm and 500 μm , were machined by Bullen Ultrasonics, Eaton, Ohio, while the 25 μm to 100 μm size holes were drilled by Resonatics, Inc., using the Excimer laser. This diagonal dimension is called "chord length." In general, flaw detection and resolution sensitivity decrease with longer chord length. In addition, X-ray beam hardening effects also start to play a role in defect detection sensitivity. The aforementioned specimen was evaluated with X-ray computed tomography (CT) using Tomoscope Microscopic CT System (TOMO). The Tomoscope system was experimentally determined to have insufficient power, 195 kV, to penetrate a test block of 88 mm to detect the holes with a reasonable signal-to-noise ratio (SNR). The specimen was machined to effectively decrease the chord length to 55 mm. The phantom was evaluated using 195 kV tube voltage, 1.0 mm slice thickness, pixel size of 0.038 mm, and 2048 samples. Imagery of the new phantom showed considerably more detail. The 500 micron diameter holes were detected and resolved (Figure 127). The 250 micron and 125 micron diameter holes were detected but not resolved. Smaller 25 micron and 50 micron holes were not detected. The specimen was further machined (Figure 128) to decrease the diagonal dimension of

TE94-2499

Figure 125. Schematic of a third generation CT system at ARACOR.

TE94-2500-5

Figure 126. Layout of reference standard with varying size and spaced holes.

Figure 127. Computer tomography Aracor Tomoscope. Detected and resolved 500 μ m diameter holes.

TE94-2502

Figure 128. Layout and size of reference specimen after machining.

the specimen to determine the detection limit and resolution capability of the Tomoscope. Billets with 25, 50, and 100 micron diameter holes were evaluated. The slice thickness of 1.0 mm with a pixel size of 40 micron was used. Figure 129 shows the optical and CT image of the 100 micron diameter holes. It is evident that 100 micron holes were detected. The outer hole appears as a moon shape in the CT image. The blurring between the second and third holes appears in the CT image. Optical examination of the specimen revealed that a shallow hole between the second and third hole was present. The shallow hole produced a partial volume effect in the CT image. This resulted in a slight reduction of the CT value in the region that appears as a slight blurring of the third hole toward the second. The diameter of the hole on the surface was measured to be 274 micron. Examination of the bottom of the hole revealed a tapered hole with decreasing diameter at the bottom. The optical and CT image of the billet with 50 μm diameter holes is shown in Figure 130. Optical measurements yielded 150 μm surface diameter of the hole. The outer two holes were detected and resolved by CT. Measurement of the holes from CT image was nearly 120 μm . The optical micrograph shows that three holes are clustered together. The optical and CT image of the 25 micron hole billet is shown in Figure 131. Again, three holes are clustered together and seen as a single elongated hole. The single outermost hole was detected that establishes the flaw detection capability of the system.

Optical Image

CT Image

TE94-2503

Figure 129. Detection of 100 μm diameter holes by Tomoscope at Aracor.

TE94-2504

Figure 130. Detection of 50 µm diameter holes by Tomoscope at Aracor.

TE94-2505

Figure 131. Detection of 25 μm diameter holes by Tomoscope at Aracor.

4.6 THEORETICAL AND EXPERIMENTAL CT STUDIES AT CHALK RIVER LABORATORIES, CANADA

During this program, the technical effort at Chalk River Laboratories, Canada, concentrated on theoretically assessing the effect of pixel size, image noise, signal-to-noise ratio, and the type of defect (air bubbles, iron, iron oxide, iron silicide) on defect detection in silicon nitride material. In addition, experimental studies were conducted using Ir-192 isotope to assess the defect detection capability of the system and to compare the experimental results with theoretical predictions.

A set of five ceramic rectangular specimens with laser-drilled holes were evaluated for experimental tests on defect detectability limits. These specimens contained cylindrical holes, 3 mm long and from 25 μm to 500 μm in diameter (d), that could simulate cracks. Each specimen contained holes of one size, separated by 2d to 10d diameter distances. Although the hole separation for small defect diameters was too small to resolve them, at least some of the defects were sufficiently separated to provide a useful detectability test. CT measurements were performed using two different resolutions and image noise.

CT examinations were performed using AECL's first-generation CT scanner (Figure 132).⁴³ The scanner employs an Ir-192 source, a CsF detector, a set of tungsten collimators that form the photon radiation beam, a rotate-translate table that provides mechanical movement of the object for scanning, and a computer-controlled table movement and data acquisition system. The scanner assures ideal beam conditions by collimating the beam from both the detector and source sides to form a pencil beam. This results in no crosstalk and very low scattered-radiation background. The rotate-translate table provides high mechanical accuracy and ideal data collection geometry. These data collection conditions result in images free from artifacts that are typical for high-generation scanners. The use of an isotopic source, rather than an X-ray generator, is

TE94-2506

Figure 132. Photograph of a Chalk River Laboratory CT scanner.

advantageous when high sensitivity in the detection of density variation is required. An isotopic source provides a photon beam of well-defined energy spectrum and stable intensity, so that beam hardening (BH) due to a multi-energy spectrum can be ideally corrected. This scanner provides virtually artifact-free CT images. CT data measured using an isotopic source can provide a measurement of absolute density of the examined object.

Two basic CT scan parameters are spatial resolution and image noise. Spatial resolution is defined by the size of the scanning beam, which should be correlated with sampling distance. The image can be reconstructed using various sizes of the reconstruction matrix and correspondingly various pixel sizes. In this work, pixel size in the reconstructed image was always such as to be consistent with the image's spatial resolution. This means that images were reconstructed using a pixel size Dt equal to half the beam size and equal to the sampling distance, and that the scans were performed with a sufficient number of projections.

Density resolution (i.e., image contrast) is defined by the image noise. Defined on a pixel-to-pixel basis, image noise can be measured as a standard deviation in the density value from pixel to pixel in a uniform object. Image noise is caused primarily by counting statistics. That portion due to counting statistics can be calculated from the following formula:

$$s \sim \{\mu Dt (I m t)^{0.5}\}^{-1} \quad (4)$$

where μ is the linear absorption coefficient, I is the intensity of the beam transmitted through the investigated object, Dt is the sampling distance (pixel size), m is the number of projections, and t is the data acquisition time per ray. Image noise can be measured as fluctuations in the density value from pixel to pixel, in a CT image of an ideally uniform object. Image noise is increased due to inaccuracies of the scanner and image reconstruction procedure (inaccuracy in detector readings, cross talk between detectors, mechanical inaccuracy in scanner operation, and errors in reconstruction procedure, such as uncorrected BH effects). All these effects were negligible in the present work. Our first-generation scanner, providing ideal beam conditions and mechani-

cal accuracy, produced CT images that were artifact free and featured image noise due to statistical fluctuations only. BH corrections were made by software. By changing the data acquisition time or the number of projections, images with different image noise were measured. Image noise varied from 0.5 to 16% in different images.

In this work, CT data were collected using 180 to 720 projections, and either 128 or 256 rays in each projection. Sampling was performed twice per beam width (this means that the distance between consecutive rays in one projection was twice as small as the beam width). CT images were reconstructed in grids of square pixels, using either 128×128 or 256×256 pixels, the numbers being consistent with the number of rays in the scan. In this way, the pixel size in the image was consistent with the scan resolution. The spatial resolution of the image, equal to the beam width, was equal to two pixels.

A pixel in a CT image is, in reality, a voxel (volume element), which has the "height" in a direction perpendicular to the scan plane. The voxel "height" is defined by the beam size in this direction. This in-slice thickness was either 1 mm or 3 mm in various scans. Since we are referring to 2-D images only, in the following, the terms pixel and voxel are used synonymously.

CT images were calculated from the measured projection data using a filtered back-projection method and Ram-Lak filter. BH due to the multi-energy spectrum of an Ir-192 source was corrected by software, assuming that the specimens were composed of pure silicon nitride (Si_3N_4).

A model was developed to assess CT detectability limits and perform calculations. An individual defect can be treated as isolated if it is separated from another defect by a distance of at least two pixels (spatial resolution). In reality, due to partial volume effects, a minimal distance of two to four pixels is required. If this distance is smaller, defects are not individually resolved, and defect clusters will not appear to be different from individual defects.

An individual defect is detected in a CT image if its signal is larger than the image noise. Then the following formula applies, which can be derived from geometrical considerations:

$$(1 - (\mu_{\text{def}}/\mu_{\text{m}})) \cdot (V_{\text{def}}/V_{\text{voxel}}) \geq h \cdot s \quad (5)$$

where μ_{def} and μ_{m} are linear absorption coefficients of defect and matrix materials, V_{def} is the defect volume within one voxel, V_{voxel} is the voxel volume, and h is the signal-to-noise ratio. As shown in the above formula, defect detectability depends on a relative difference between the absorption coefficients of the defect and object, the defect size relative to the voxel size, and the image noise. Using this formula, one can calculate the smallest defect size V_{def} that can be detected in a CT image, depending on all involved parameters. A defect does not have to be larger than the pixel size to be detected and even sized. Two basic CT parameters are explicit in this formula: the spatial resolution (defined by the voxel size V_{voxel}), and the image noise, s , relative to the absorption coefficient.

In this work, numerical calculations were performed for several typical voxel sizes and a range of image noise values, and for defects of two geometries in a silicon nitride matrix. Calculations were performed for $h = 2, 3 \dots 10$. Defects having size smaller than the pixel size were of interest. Two defect geometries were: (1) long cylinders and (2) spheres. Cylinders were assumed to be oriented perpendicularly to the scanning plane, and to be longer than the in-slice thickness (1 mm). These cylindrical defects can simulate long cracks: although a cylindrical shape imposes a circular cross section, the same result is obtained for any cross section shape, assuming the same cross-sectional area. Calculations for cylindrical voids were verified experimentally. Spheres simulated 3-D defects of various shapes: the results are applicable to any defect shape, assuming the same defect volume. To evaluate the dependence of the defect detectability limits on a defect chemical composition, calculations for 3-D defects were performed for different defect materials: iron, several iron oxides, and several typical iron-oxygen-silicon compounds (iron silicide and iron silicates).

Experimental verification of the performed calculations was obtained by CT measurements performed on five ceramic billets with laser-drilled cylindrical holes. The measured images were free from artifacts, which are usually present in images measured by higher generation scanners. Comparison of the experimental results with the calculated data confirmed the accuracy and applicability of the proposed theoretical approach.

Calculations for defect detectability limits were conducted assuming the defect was to be a cylinder with a diameter d , and the length equal to or larger than the in-slice thickness; the cylinders are perpendicular to the scanning plane. Calculations of defect detectability limits were performed for rectangular pixels with a pixel size of $0.125 \times 0.125 \times 1 \text{ mm}^3$ and $0.25 \times 0.25 \times 1 \text{ mm}^3$. The results for selected values of image noise " σ " and the signal-to-noise ratio " η " are shown in Tables 27 and 28, and in graphs in Figures 133 and 134 (in the figures curves represent calculated data, and dots represent experimental data.)

Table 27. The calculated minimal defect size for cylindrical voids in a silicon nitride matrix, detectable in a CT scan with rectangular voxels ($0.250 \times 0.250 \times 1 \text{ mm}^3$) and image noise σ . The cylinder length is more than 1 mm. Scan resolution is consistent with the pixel size; η defines the signal-to-noise ratio.

σ —%	Defect diameter— μm		
	$\eta = 2$	$\eta = 3$	$\eta = 4$
10	110	140	160
5	80	100	110
3	60	75	90
2	50	60	70
1	35	45	50
0.5	25	30	35
0.2	16	20	22
0.1	11	14	16

Table 28. The calculated minimal defect size for cylindrical voids in a silicon nitride matrix, detectable in a CT scan ($0.125 \times 0.125 \times 1 \text{ mm}^3$) and image noise σ . The cylinder length is more than 1 mm. Scan resolution is consistent with the pixel size; η defines the signal-to-noise ratio.

σ —%	Defect diameter— μm		
	$\eta = 2$	$\eta = 3$	$\eta = 4$
10	60	70	80
5	40	50	55
3	30	40	40
2	25	30	35
1	20	22	25
0.5	13	15	35
0.2	8	10	22
0.1	6	7	16

Figure 133. Minimal defect size calculated for a cylindrical defect detectable in a CT scan with rectangular voxels ($0.25 \times 0.25 \times 1 \text{ mm}^3$) as a function of the image noise. Defects are air cylinders ($>1 \text{ mm}$ long) in a silicon nitride matrix. Scan resolution is consistent with the pixel size. η defines the signal-to-noise ratio. As explained in the text (Section 5.2), black dots mark defects clearly observed in CT scans, crossed-detected with some uncertainty, and open dots - not detected.

Figure 134. Same as Figure 133, but for a voxel of $0.125 \times 0.125 \times 1 \text{ mm}^3$.

To verify the accuracy of the calculations and the proposed approach, measurements were performed on a set of five ceramic specimens that had laser-drilled cylindrical holes. CT measurements were performed using two different CT resolutions and various image noises. The pixel size was $250 \times 250 \mu\text{m}^2$ and $125 \times 125 \mu\text{m}^2$, with an in-slice thickness of 1 mm in both cases (i.e., rectangular voxels). CT scans were performed sometimes on individual specimens, but mostly for a set of two specimens placed together on the scanning table. Image noise varied from 0.6 to 10% between scans.

CT detected and resolved all large holes ($500 \mu\text{m}$ to $100 \mu\text{m}$). The pattern of the hole spacing is shown in Figure 135. Larger holes are separated in the image by, correspondingly, 20, 10, 6, and 4 pixels (1 pixel = $125 \mu\text{m}$), which corresponds to their spacing equal to, respectively, 10 defect diameters, 5, 3 and 2. For a specimen having $100 \mu\text{m}$ holes, this sequence corresponds to the spacing of 1 mm, 0.5 mm, $300 \mu\text{m}$ and $200 \mu\text{m}$, which makes the last two holes unresolved, because they are separated by fewer than two pixels; this agrees with the CT result in Figure 135.

Small holes ($25 \mu\text{m}$ to $100 \mu\text{m}$) were mostly not resolved in any scan, because they were separated from each other by a distance too small relative to the pixel size; (holes to be resolved, i.e., detected as individual defects in a CT image, must be separated by more than two pixels). For a $250 \mu\text{m}$ pixel scan, the largest distance between $25 \mu\text{m}$ holes was one pixel, between $50 \mu\text{m}$ holes was two pixels, and between $100 \mu\text{m}$ holes was four pixels; only the latter could be resolved. For a $125 \mu\text{m}$ scan, the largest distances were two pixels, four pixels, and eight pixels, correspondingly. In those cases there was a chance to detect individually a $100 \mu\text{m}$ hole and one $50 \mu\text{m}$ hole, but $25 \mu\text{m}$ holes could not be resolved (Figure 136). Holes unresolved visually in CT images might be detected as a cluster ($25 \mu\text{m}$ in a $125 \mu\text{m}$ pixel image and $25 \mu\text{m}$ and $50 \mu\text{m}$ in a $250 \mu\text{m}$ pixel image). In such a case, one can get an estimate of an individual defect detectability from density profiles across a cluster region. A density profile crossing a cluster of holes would show jumps corresponding to individual holes in a cluster; if the amplitude of this jump was above the image noise, the defect would be assumed to be individually detectable (although not resolved). To better present defect detection for unresolved defects, some density profiles in two perpendicular directions are shown next to the CT images.

Tables 29 and 30 summarize experimental results on defect detectability. Defects are either clearly detected, detected with some uncertainty ("detected"), or undetected. The experimental results are marked, respectively, as black points, crossed points, and open points in Figures 133 and 134. Only some of the results are shown; not included are data for $250 \mu\text{m}$ and $500 \mu\text{m}$ holes (detected in all the scans, independent of the image noise), and a $100 \mu\text{m}$ hole in a CT scan with very large noise (noise 16%, hole not detected).

As shown in Figures 133 and 134, all points for detected defects lay above, and for undetected defects below, the theoretically calculated curves for the signal-to-noise ratio between 2 and 4. Thus, the h value equals between 2 and 3, which agrees with the expectations based on the image noise defined by statistics only.

By comparing experimental data in Tables 29 and 30 with calculated data in Tables 27 and 28, and from similar comparison in Figures 133 and 134, it is apparent that the calculations provided accurate estimates of the smallest detectable cylindrical voids in a silicon nitride matrix.

Theoretical calculations for spherical voids and iron based inclusions were performed to assess the detectability of such commonly occurring defects in silicon nitride materials. The dependence of the defect detectability limits on chemical composition was studied using iron and several iron-oxygen-silicon compounds. According to the phase diagram, various iron oxides, silicides, and silicates could be formed. Calculations were performed for CT images with rectangular voxels, and with an in-slice thickness of 1 mm. The defect was assumed to be a spherical inclusion of diameter d , containing a chemically uniform material. Calculated maximum pixel size for defect detection is summarized in Figure 137 for voids and inclusions of various chemical compositions and diameter up to $200 \mu\text{m}$, and for a CT scan using rectangular voxels and image noise from 0.25 to 3%.

(a)

(b)

(c)

TE94-2509

Figure 135. Depicted are: (a) CT scan of two ceramic blocks, with series of 250 μm and 100 μm holes. (pixel size 125 μm , image noise 5%. Density profile along horizontal line crossing); (b) 250 μm holes; and (c) 100 μm holes. Horizontal scale is in pixels. All holes are clearly distinguished.

(a)

50 μm

(b)

(c)

TE94-2510

Figure 136. Depicted are: (a) CT scan of two ceramic blocks with penes of 50 μm and 100 μm holes. (pixel size 125 μm , image noise 1.5%); (b) detectability of 50 μm holes (other holes appeared clustered); and (c) density profile along horizontal line crossing 50 μm holes Pixel 8 representing one hole. Pixel 12-16 show clustered holes.

Table 29. Summary of the experimental data for CT scans performed on ceramic specimens with laser-drilled holes (cylindrical voids). The voxel size was $0.125 \times 0.125 \times 1 \text{ mm}^3$, image noise σ as specified. Three columns list defects clearly detected (1), detected with some uncertainty (2), and not detected (3) in a CT image. Scan 1275 was measured using a thinner slice (about 0.5 mm), and this measurement is not included in a graph verifying calculated defect detectability.

CT scan No.	σ —%	Defects clearly detected	Defects detected	Defects not detected
1246	5	100, 250 μm		
1247	3.5	100, 250 μm		
1248	5.5		50 μm	25 μm
1249	10			50, 25 μm
1277	1.5	100, 50 μm		
1280	2.5	50 μm	25 μm	
1281	4	50 μm		25 μm

Table 30. Summary of the experimental data for CT scans performed on ceramic specimens with laser-drilled holes (cylindrical voids). The voxel size was $0.25 \times 0.25 \times 1 \text{ mm}^3$, image noise σ as specified. Three columns list defects clearly detected (1), detected with some uncertainty (2), and not detected (3) in a CT image.

CT scan No.	σ —%	Defects clearly detected	Defects detected	Defects not detected
1237	0.8	500, 250 μm		
1238	1.3	500, 250 μm		
1240	1.4	250, 100 μm		
1241	0.7	250, 100 μm		
1243	2	250 μm		50 μm
1245/08	1.4	250 μm	50 μm	
1245/18	1	250, 50 μm		
1245/89	0.8	250, 50 μm		
1250	0.9	100, 50 μm		
1251	3.1	100 μm		50 μm
1252	4.5	100 μm		50 μm
1252	6.2		100 μm	50 μm
1275	2	100, 50 μm		
1276/180	1	100, 50 μm		
1276/360	0.6	100, 50 μm

TE94-2511

Figure 137. Calculated maximal pixel size for defect detection of a 3-D defect in a CT image with rectangular voxels ($\Delta t \times \Delta t \times 1$ mm). Defects are spherical inclusions in a silicon nitride matrix. Defect material as specified (iron, air, iron oxides). σ is the image noise. The signal-to-noise ratio η between 2 and 4 was assumed to provide the useful range in a range of CT scanners.

Calculations were also performed for two cubic voxels: $0.25 \times 0.25 \times 0.25 \text{ mm}^3$ and $0.125 \times 0.125 \times 0.125 \text{ mm}^3$. The results are summarized in Tables 31 and 32. Representative data for $h = 3$ are given, and for defects with various chemical compositions, as before (air, iron, iron oxides, iron silicate, and silicides). Calculated maximum pixel sizes for defect detection are summarized in Figure 138 for voids and inclusions of various chemical compositions, diameter up to $200 \mu\text{m}$, and image noise from 0.25 to 3%.

Table 31. The calculated minimal defect size for a 3-D defect detectable in a CT scan with cubic voxels ($0.25 \times 0.25 \times 0.25 \text{ mm}^3$) and image noise σ . Scan resolution is consistent with the pixel size. The 3-D defect is a void or inclusion in a silicon nitride matrix. Defect material as specified. Data are for the signal-to-noise ratio η equal to 3.

σ —%	Defect diameter—mm					
	Air	Iron	Iron oxides	FeSi	FeSiO ₃	Fe ₂ SiO ₄
10	0.21	0.19	0.24	0.22	0.53	0.30
5	0.17	0.15	0.19	0.17	0.41	0.24
3	0.14	0.13	0.16	0.15	0.35	0.20
2	0.12	0.11	0.14	0.13	0.31	0.18
1	0.10	0.09	0.11	0.10	0.25	0.14
0.5	0.08	0.07	0.09	0.08	0.19	0.11
0.2	0.06	0.05	0.07	0.06	0.14	0.08
0.1	0.05	0.04	0.05	0.05	0.11	0.07

Table 32. The calculated minimal defect size for a 3-D defect detectable in a CT scan with cubic voxels ($0.125 \times 0.125 \times 0.125 \text{ mm}^3$) and image noise σ . Scan resolution is consistent with the pixel size. The 3-D defect is a void or inclusion in a silicon nitride matrix. Defect material as specified. Data are for the signal-to-noise ratio η equal to 3.

σ —%	Defect diameter—mm					
	Air	Iron	Iron oxides	FeSi	FeSiO ₃	Fe ₂ SiO ₄
10	0.10	0.09	0.12	0.12	0.26	0.15
5	0.08	0.08	0.10	0.09	0.20	0.12
3	0.07	0.06	0.08	0.08	0.17	0.10
2	0.06	0.05	0.07	0.07	0.15	0.09
1	0.05	0.04	0.06	0.05	0.12	0.07
0.5	0.04	0.04	0.05	0.04	0.10	0.06
0.2	0.03	0.03	0.03	0.03	0.07	0.04
0.1	0.02	0.02	0.03	0.03	0.05	0.03

Figure 138. Calculated maximal pixel size for defect detection of a 3-D defect in a CT image with cubic voxels ($\Delta t \times \Delta t \times 1$ mm). Defects are spherical inclusions in a silicon nitride matrix. Defect material as specified (iron, air, iron oxides). σ is the image noise. The signal-to-noise ratio η between 2 and 4 was assumed to provide the useful range in a range of CT scanners.

In conclusion, comparison of the experimental data with calculated CT defect detectability limits provided a test of the accuracy of the performed calculations and theoretical approach. The experimental results were obtained for ceramic specimens with defects of controlled sizes and simple geometry. Cylindrical holes of 50 μm diameter were detected and resolved. There was good agreement between experimental results and calculated data. This indicated that calculations provided accurate estimates of the defect detectability limits.

The present results raise the question of whether typical CT scanners are suitable to examine ceramics. Typical CT scanners feature a spatial resolution of 0.1 to 1 mm and image noise of 1 to 3%. As our calculations show, the smallest voids detectable in a silicon nitride matrix at such parameters are 400 to 600 μm at 1 mm (cubic) pixel size, and 50 to 65 μm at 0.1 mm (cubic) pixel size. Inclusions might be more difficult to detect than voids; depending on chemical composition, the smallest detectable inclusions might be up to 2.5 times larger. The presence of scanner artifacts makes these values still worse. Critical defects in ceramics are small, of the order of 25 μm to 50 μm . To detect a 25 μm void, one needs a pixel size smaller than 45 μm to 80 μm at an image noise in the range from 3 to 0.5%.

In summary, NDE techniques were developed to show the capability of detecting 10 to 50 micron size flaws. Acoustic microscopy has been demonstrated to detect 10 μm surface holes and 50 μm diameter subsurface holes at a depth of 14 mm. Microradiography detected 50 μm surface holes. Computed tomography detected 25 micron discrete voids at ARACOR, whereas the detection at Chalk River was limited to 50 mm with clarity. The 25 μm void was detected with limited detectability. The optimized acoustic microscopy and microradiography techniques were used to inspect the specimens.

V. ANALYTICAL LIFE PREDICTION MODELS

An important task in the development of the life prediction models has been the enhancement of time independent fast fracture, as well as the capability to analyze the time dependent failure mechanisms including SCG, creep, and oxidation.

In fast fracture, the corner (edge) failure has been recognized as separate from both surface and volume failures. The Allison version of CARES utilizes this information in the probabilistic analysis of the component.

For materials not shear sensitive, a theoretical argument indicates that the Weibull modulus is independent of temperature, and the use of a consolidated Weibull modulus reduces the amount of material testing required. The consolidated modulus capability has been added to the CARES fast fracture methodology.

In SCG, CARES now determines required material properties from either static or dynamic fatigue test specimens. This information, along with finite element analysis, allows CARES to compute the time dependent failure probability of a component. The SCG theory also incorporates an activation energy to reduce the size of the test matrix.

For both creep and oxidation, the data reduction package determines the material parameters from specimen testing. The life models determine the time dependent failure probability of a component. Specifically, the creep model has the capability of determining not only probabilistic parameters but also the material constants which can be applied to find the time dependent deformation of a component.

The following sections give a detailed development of the methodology.

5.1 FAST FRACTURE MODEL

5.1.1 Corner Failure Modes

The PY-6 injection molded and HIPed Type-B MOR bars were characterized from room temperature to 1400°C. A total of 320 specimens were evaluated at a constant cross-head speed of 0.5

mm/min in a servo-hydraulic test system. The data regression for surface, volume, and corner failures was accomplished using the CARES program and the maximum likelihood estimator (MLE) methodology with suspended items.

At room temperature, specimen failures are particularly influenced by surface or corner features. While the remaining specimens failed from surface flaws, 80% of the specimens failed from corner flaws (Table 33). The higher values of corner flaw stress intensity factors, as compared to surface or volume stress intensity factors, result in lower strength for the same size and shape of flaw and are partially responsible for the observed results. The failures are also influenced by the process-induced naturally occurring defects including machining marks on the surface, as well as residual stresses. None of the specimens at room temperature failed from volume flaws because of the smaller stress intensity factor and the small volume under high stress. Even at elevated temperatures, only a few volume failures were observed. Above 1000°C, about 13% of the specimens failed from volume flaws, 62% failed on the surface, and corner failures were found in 25% of the specimens.

Table 33. PY-6 Type-B MOR bar fast fracture data summary

Temperature— °C	Flaw type	Number of specimens	Weibull exponent	Characteristic strength— ksi/MPa
25	Surface	16	8.65	159/1096
	Corner	64	5.91	133.6/921
	Volume	0	----	----
1000	Surface	7	14.5	124.3/857
	Corner	9	8.76	124.5/858
	Volume	4	20.9	126.7/874
1100	Surface	12	14.66	92.68/639
	Corner	6	16.44	96.66/666.5
	Volume	1	-----	----
1200	Surface	52	11.28	99.32/685
	Corner	21	6.64	115.4/796
	Volume	15	11.21	111/765
1300	Surface	20	17.85	88.81/612
	Corner	3	11.21	105.5/727
	Volume	6	29.91	92.86/640
1400	Surface	54	12.95	72.66/501
	Corner	21	11.6	78.76/543
	Volume	4	11.98	90.24/622

Based on CARES data reduction using MLE with suspended items.

The CARES program was modified to account for the corner flaw population. In this process MOR failures are identified as either surface, volume, or corner failures. The MLE method is used with two other failure modes treated as suspended items for the third. Unlike surface or volume failures, the corner failure is associated with length. Once the characteristic strength has been found, the unit characteristic strength of the corner may be found from the following equations:

$$S_o = S_{th} * l_{eff}^{1/m} \quad (6)$$

$$l_{eff} = 2*(L_2*m + L_1)/(m+1) \quad (7)$$

Where:

S_o = unit characteristic strength

S_{th} = characteristic strength

L_2, L_1 = inner and outer spacing between 4 point loads

l_{eff} = effective length for the MOR bar

m = Weibull modulus

The Weibull probability of survival resulting from component corner features may be found by:

$$POS_i = \text{Exp}(-l_i * (S_i/S_o)^m) \quad (8)$$

$$POS = POS_1 * POS_2 * \dots * POS_n \quad (9)$$

If S_i is the stress on a small elemental length (l_i) then the component reliability resulting from all corner features is the product of individual probabilities of survival obtained from Equation 8. The NASTRAN finite element code output provides both element length and stress, which can be used as input to the Allison version of CARES.

5.1.2 Consolidation of the Weibull Exponent

One of the problems incurred in the development of a ceramic design system is the large amount of testing necessary to obtain the fast fracture Weibull properties at operational temperatures. A possible approach in reducing the data requirements involves consolidating the elevated temperature data using a single Weibull modulus. The observed PY-6 surface Weibull exponent is 8.6 at room temperature. At elevated temperatures, the mean Weibull exponent remains in a tight range of 11 to 14 from 1000°C to 1400°C, except for the value of 18 at 1300°C. This suggests that it may be reasonable to consolidate the elevated temperature data using a single value of (m) (Figure 139).

For a shear insensitive material such as PY-6, a theoretical argument may be made that the Weibull exponent is independent of temperature. For simplicity, consider that two specimens with differing flaw sizes normal to the applied stress fail in fast fracture at the same temperature. The strengths are given by the equations:

$$S_1 = K_{ic} / (Y * c_1^{0.5}) \quad (10)$$

$$S_2 = K_{ic} / (Y * c_2^{0.5}) \quad (11)$$

Where c_1 and c_2 are the critical flaw sizes at failure.

Figure 139. PY-6 silicon nitride MIL-B MOR bar fast fracture Weibull modulus based on surface failures.

The ratio of the strengths depends only upon the flaw size ratio.

$$S_1/S_2 = (c_2/c_1)^{0.5} \quad (12)$$

Using the Weibull two parameter equation along with a median ranks equation allows the solution of the Weibull modulus m for the test temperature. This solution is given in Equation (15).

$$1 - P_{fi} = \exp(-(S_i/Sch)^m) \quad (13)$$

$$P_{fi} = (i - 0.3)/(N + 0.4) \quad (14)$$

where:

P_{fi} = failure probability of i th specimen

i = specimen rank

N = number of specimens tested

S_i = strength of i th specimen

Sch = specimen characteristic strength at test temperature

$$m = \ln(\ln(1/(1 - P_{f1}))/\ln(1/(1 - P_{f2}))) / \ln(S_1/S_2) \quad (15)$$

The Weibull modulus is dependent only on the individual specimen failure probabilities and the ratio of the square root of the flaw sizes. Since these variables are independent of the test temperature, the same two specimens tested at a different temperature would demonstrate an identical Weibull modulus.

Since the PY-6 mixed mode testing has shown the material to be relatively insensitive to shear stresses, the Weibull exponent is expected to be temperature independent. Individual specimen strengths may be normalized to a single base temperature by multiplying by the ratio of K_{ic} at the base temperature to K_{ic} at the test temperature. Alternatively, if a number of specimens have been tested at each temperature, the average strengths are proportional to K_{ic} and the ratio of mean stresses may be used to normalize all data to the base temperature. This was done for the elevated temperature PY-6 data. Once fast fracture strengths are normalized to a single temperature, the CARES program using the MLE methodology may be used to find the consolidated Weibull exponent, m_c . If all failures result from a single population, the Weibull characteristic strength is determined at each temperature by using the equation:

$$S_{ch} = S_m / \Gamma(1 + 1/m_c) \quad (16)$$

Where:

S_m = mean strength at each temperature
 Γ = Gamma function

If suspended items exist, it is first necessary to find the mean strength after accounting for the suspended items. This is done by using the MLE method and finding the characteristic strength, S , and the unconsolidated, m , and then the mean strength is given by:

$$S_m = S \Gamma(1 + 1/m) \quad (17)$$

and the characteristic strength corresponding to m_c is again obtained from Equation 16.

Finally, at each temperature a Weibull plot is constructed to verify the consolidated m results in a reasonable fit of the original data. This process is illustrated in Figure 140. In this case, surface failures (no censored data) were used to determine a PY-6 Weibull consolidated exponent of 11.97. The consolidated exponent was used with the characteristic strength at each temperature from 1000°C to 1400°C to develop Figure 140. The figure illustrates that the fit using the consolidated m_c value works well even for the bulk of the 1300°C fast fracture data, where a large discrepancy might have been expected, and at other temperatures, the fit is even better. Figure 139 also shows that the Weibull moduli computed at the individual temperatures of 1200°C and 1400°C agree well and are quite close to the overall value of the Weibull modulus. At each of these temperatures, over 50 specimen tests were available, resulting in a low scatter of the estimated moduli values.

The room temperature surface Weibull exponent ($m = 8.6$) is smaller than that observed at elevated temperatures. It is believed that at higher temperatures the process and machining induced surface defects are modified and probably blunted, which decreases the percentage of corner failures and reduces the scatter in the surface strength as evidenced by an increased Weibull modulus at elevated temperatures.

While a theoretical argument suggests that m is independent of temperature, such an argument assumes the critical flaw is unaffected by the environment. This may not always be true. Thus, it is essential that a final Weibull plot be used to verify the fit after the consolidated Weibull constants are obtained. Table 34 summarizes the Weibull constants for both consolidated and unconsolidated Weibull exponents. Note the characteristic strength is relatively unaffected by the use of the consolidated modulus; however, for PY-6 the elevated temperature consolidated Weibull exponent would result in a rather poor fit of the room temperature data.

Figure 140. Comparison of predicted FF data fit versus experimental results for PY-6 Type-B MOR specimens with surface failures using the consolidated Weibull modulus of 11.97.

Table 34. For PY-6 Type-B MOR bar, a comparison of the consolidated and individual temperature surface Weibull fast fracture material constants.

Temperature —°C	Flaw type/ number of specimens	Baseline		Consolidated m	
		Weibull exponent	Character. strength— ksi/MPa	Weibull exponent	Character. strength— ksi/MPa
1000	Surface/7	13.54	117.2/808.1	11.97	117.7/811.5
1100	Surface/12	13.91	89.67/618.3	11.97	90.17/621.7
1200	Surface/52	10.46	95.94/661.5	11.97	95.45/658.1
1300	Surface/20	18.80	86.77/598.3	11.97	88.04/607.0
1400	Surface/54	12.02	70.93/489.1	11.97	70.96/489.3

Based on CARES data reduction using MLE without suspended items.

5.1.3 Room Temperature Fracture Toughness and Shear Sensitivity

5.1.3.1 Brazilian Cracked Disk Testing. The Mode I fracture toughness and the relative shear sensitivity of PY-6 material at room temperature was determined as part of the investigation of a fast fracture methodology. Eleven ceramic disks were precracked with chevron through notches and tested with compressive loads applied at various angles. The Brazilian cracked disk test results are used to compute the values of KI and KII for the material. Depending on the values of KI and KII, the relative influence due to crack opening and shear on fracture toughness can be determined. Table 35 summarizes the test information and the computed values of KI and KII. Values of KI and KII were computed using Equations 19 through 23 and the methodology developed in References 26 and 44. Figure 141 shows the fit of the data to Equation 18, from Reference 45 which describes typical mixed mode behavior.

$$KI/K_{Ic} + (KII/c*K_{Ic})^2 = 1 \quad (18)$$

Where:

K_{Ic} = plain strain fracture toughness

K_{IIc} = pure shear fracture toughness

c = K_{IIc}/K_{Ic}

The room temperature value of K_{Ic} was found to be 6.44 MPa-m^{0.5}, while c was determined to be 1.52 for the through-crack in PY-6 material. The c value of 1.52 illustrates that PY-6 is relatively insensitive to shear stresses acting on a flaw. The c value for the Garrett NT154 material is about 2.0, also indicating a low shear sensitivity. Equations 24 through 29 define the regression methodology used to find K_{Ic} and c using Equation 18.

5.1.3.2 Equations and Theory—Brazilian Cracked Disk.

- Computation of KI and KII^{26, 44}

$$KI = (P/R/B)*(a/Pi)^{0.5}*NI(a/R, Ang) \quad (19)$$

Table 35. Summary of PY-6 room temperature Brazilian cracked compressive disk test results.

Specimen	Angle— deg	Diameter— in./mm	Thick— in./mm	Crack length— in./mm	Load— lb/Newt.	KI— MPa-m ^{0.5}	KII— MPa-m ^{0.5}
3-27	0	1.253	0.100	0.625	800	6.13	0
		31.83	2.54	15.88	3559		
3-48	0	1.253	0.0999	0.625	855	6.56	0
		31.83	2.54	15.88	3803		
3-26	0	1.251	0.0999	0.625	815	6.27	0
		31.78	2.54	15.88	3625		
3-72	9	1.253	0.1003	0.45	1150	5.53	4.50
		31.83	2.548	11.43	5115		
3-47	9	1.251	0.1002	0.46	1110	5.44	4.46
		31.78	2.545	11.68	4938		
3-25	9	1.251	0.1008	0.36	1200	4.93	3.76
		31.78	2.560	9.144	5338		
3-51	14	1.252	0.1004	0.42	1150	4.17	6.24
		31.80	2.550	10.67	5115		
3-61	14	1.253	0.0998	0.42	1180	4.30	6.43
		31.83	2.535	10.67	5249		
3-45	23	1.254	0.1005	0.36	1180	1.47	8.20
		31.85	2.553	9.144	5249		
3-52	23	1.253	0.0991	0.39	1120	1.3	8.43
		31.83	2.517	9.91	4982		
3-53	23	1.251	0.0998	0.36	1220	1.53	8.57
		31.78	2.535	9.14	5427

Figure 141. Results of mixed mode room temperature fast fracture testing of PY-6 compressively loaded diametrical through-cracked disks used to determine both the Mode I and shear fracture toughness.

$$K_{II} = (P/R/B) \cdot (a/\pi)^{0.5} \cdot N_{II}(a/R, \text{Ang}) \quad (20)$$

$$N_I = \sum A_i(\text{Ang}) \cdot T_i(a/R) \cdot (a/R)^{2i-2} \quad (21)$$

$$N_{II} = 2 \cdot \sin(2\text{Ang}) \sum B_i(\text{Ang}) \cdot S_i(a/R) \cdot (a/R)^{2i-2} \quad (22)$$

Where:

P = applied load producing fast fracture failure

$\pi = 3.14159$

R = disk radius

a = crack half length

B = disk thickness

Ang = angle between crack plane and load vector (see Table 37)

N_I, N_{II} = intermediate variables

i = index of terms in summation, i = 1, 5

Table 36 provides the values of S_i and T_i as a function of a/R , while Table 37 defines the equations necessary to compute A_i and B_i .

- Newton forward interpolation

S_i and T_i are found for nontabulated values of a/R by using the Newton forward interpolation method. The following equations apply if $y = f(x)$ and x_i are monotonically increasing at equal intervals, h:

$$Dy_1 = y_2 - y_1 \quad (23)$$

$$Dy_2 = y_3 - 2y_2 + y_1$$

$$Dy_3 = y_4 - 3y_3 + 3y_2 - y_1$$

$$Dy_4 = y_5 - 4y_4 + 6y_3 - 4y_2 + y_1$$

$$Dy_5 = y_6 - 5y_5 + 10y_4 - 10y_3 + 5y_2 - y_1$$

$$u = (x - x_1)/h$$

$$y = y_1 + u(Dy_1 - 0.5Dy_2 + Dy_3/3 - Dy_4/4 + Dy_5/5) + u^2(0.5Dy_2 - 0.5Dy_3 + 11/24Dy_4 - 5/12Dy_5) + u^3(Dy_3/6 - Dy_4/4 + 7/24Dy_5) + u^4(Dy_4/24 - Dy_5/12) + u^5Dy_5/120$$

Table 36. Tabulation of constants S_i and T_i used in computing fracture toughness of a notched disk

a/R	T1 S1	T2 S2	T3 S3	T4 S4	T5 S5
0.1	1.014998	0.503597	0.376991	0.376991	0.314159
	1.009987	0.502341	0.376363	0.376363	0.314159
0.2	1.060049	0.514907	0.382430	0.383392	0.318086
	1.039864	0.509959	0.379956	0.380584	0.316245
0.3	1.135551	0.533477	0.391640	0.393835	0.325033
	1.089702	0.522272	0.386086	0.387518	0.320834
0.4	1.243134	0.559734	0.404603	0.408597	0.334831
	1.160796	0.539824	0.394822	0.397403	0.327411
0.5	1.387239	0.594892	0.421949	0.428353	0.347941
	1.257488	0.563966	0.406869	0.410966	0.336447
0.6	1.578258	0.642124	0.445387	0.454861	0.365559
	1.390654	0.597985	0.424037	0.430072	0.349219

Table 37. Equations to compute A_i and B_i used to determine the fracture toughness of a through-cracked disk; $S = \sin(\text{Ang})$ $C = \cos(\text{Ang})$

A1	$1 - 4S^2$
A2	$8S^2 (1 - 4C^2)$
A3	$-4S^2 (3 - 36C^2 + 48C^4)$
A4	$-16S^2 (-1 + 24C^2 - 80C^4 + 64C^6)$
A5	$-20S^2 (1 - 40C^2 + 240C^4 - 448C^6 + 256C^8)$
B1	1
B2	$-5 + 8C^2$
B3	$-3 + 8(1 - 2C^2)(2 - 3C^2)$
B4	$3 + 16(1 - 2C^2) - 12(1 - 2C^2)^2 - 32(1 - 2C^2)^3$
B5	$5 - 16(1 - 2C^2) - 60(1 - 2C^2)^2 + 32(1 - 2C^2)^3 + 80(1 - 2C^2)^4$

In using these equations the S_i or T_i are considered as y_i while tabular values of a/R are the x_i . x is the value of a/R used in the interpolation.

- Method of determining K_{Ic} and K_{IIc}

Equation 18, describes the relationship between K_I and K_{II} in mixed mode behavior.⁴⁵ The best fit of all test data pairs K_{IIi} and K_{Ii} thus evaluates K_{Ic} and $K_{IIc} = c \cdot K_{Ic}$. The technique elected was to select a curve fitting the above equation, which would minimize the sum of the squares of the normal distance from the curve to the data points. This method does not bias the value of either K_{Ic} or K_{IIc} .

The equation to be fitted, Equation 18, is written in the form:

$$Y = m \cdot X^2 + b \quad (24)$$

Where:

$$X = K_{II}$$

$$Y = K_I$$

$$m = -K_{Ic}/K_{IIc}^2$$

$$b = K_{Ic}$$

The function to be minimized can be written as:

$$\sum (X - X_i)^2 + (Y - Y_i)^2 = \sum d^2 \quad (25)$$

or
$$\sum (X - X_i)^2 + \sum (m \cdot X^2 + b - Y_i)^2 = \sum d^2$$

If the partial derivative with respect to both m and b is set to zero and the two simultaneous equations are solved, Equations 26 and 27 can be derived:

$$m = (N \cdot \sum X^2 \cdot Y_i - \sum X^2 \sum Y_i) / (N \cdot \sum X^4 - \sum X^2 \cdot \sum X^2) \quad (26)$$

$$b = (\sum X^4 \sum Y_i - \sum X^2 \sum X^2 Y_i) / (N \sum X^4 - \sum X^2 \sum X^2) \quad (27)$$

The values of X , which are abscissas on the curve at a minimum distance from the X_i, Y_i data points, must be found. An equation may be found for each value of X by again taking the partial derivative of the summation and setting to zero. The following set of N cubic equations result.

$$X^3 + X(1 + 2m^*b - 2m^*Y_i)/(2m^2) - X_i/(2m^2) = 0.0 \quad (28)$$

Thus for a system of N data points, a system of $N+2$ equations are available to solve for the N values of X and the optimum fit parameters b and m (the equation just derived can also be obtained from analytical geometry considerations which require the slope of the line through the data point be the negative reciprocal of the curve slope at the intersection point).

An iterative process can be used to efficiently solve the system of $N+2$ nonlinear equations. Initial values of b and m may be determined by taking the X s to be equal to the data points X_i . At this point, the N cubic equations in X may be solved to obtain new values, and m and b are then recalculated. This continues until m and b are virtually unchanged in successive passes, at which time the best fit curve has been found. A test problem with 30 data points was found to converge in about 10 passes.

- Cubic equation solution

Solution techniques to determine the roots of a cubic equation are standardized in mathematical texts. Equation 29 describes the method. The cubic equation to find X may be written in the form:

$$\begin{aligned} X^3 + aX + c &= 0.0 \quad a = (1 - 2m^*Y_i + 2m^*b)/(2m^2) \\ c &= -X_i/(2m^2) \\ T &= c^2/4 + a^3/27 \end{aligned} \quad (29)$$

- Case I – $T > 0$ (1 real root)

$$\begin{aligned} T1 &= -0.5*c + T^{0.5} \\ T2 &= -0.5*c - T^{0.5} \\ X &= T1^{1/3} + T2^{1/3} \end{aligned}$$

- Case II – $T = 0$ (2 real roots)

$$\begin{aligned} T1 &= (-0.5*c)^{1/3} \\ X &= 2*T1 \\ X &= -T1 \end{aligned}$$

- Case III – $T < 0$ (3 real roots)

$$\begin{aligned} \cos(z) &= -0.5*c/(-a^3/27)^{0.5} \\ C &= 2*(-a/3)^{0.5} \\ X &= C*\cos(z/3) \\ X &= C*\cos(z/3 + 120 \text{ deg}) \\ X &= C*\cos(z/3 + 240 \text{ deg}) \end{aligned}$$

If more than a single root is found, Equation 25 is used to determine the value of X resulting in the lowest value of d.

5.1.3.3 Indented Bend Bar Test to Determine Fracture Toughness and Shear Sensitivity. The Mode I fast fracture toughness and the relative shear sensitivity were also determined using a Vickers indentation test on four point bend bars. Orienting the crack formed along the diagonal of the Vickers indentation at an angle to the maximum bending stress results in a mixed mode fast fracture failure.

First Chantikul, et al.'s equation²⁹ for the strength of an indented specimen was used to determine K_{ic} for the part through-crack formed by the Vicker's indentation.

$$K_{ic} = 0.59 (E/H)^{1/8} S^{3/4} P^{1/4} \quad (30)$$

Where:

E = elastic modulus

H = measured hardness

S = average fast fracture strength for bars with cracks normal to the specimen axis

P = indentation load

For PY-6 the following values were obtained, resulting in a computed value of K_{ic} = 6.7 MPa-m^{0.5}.

E = 300 Gpa

H = 14.5 Gpa

S = 211.3 MPa (Table 38)

P = 3.92 E-4 MNewtons

Table 38. Fast fracture bend strength of indented room temperature PY-6 bars

Specimen	Angle—deg	Width— in./mm	Thick— in./mm	Load— lb./Newt	Strength— MPa
3-15	0	0.2001	0.0944	80.5	217
		5.083	2.398	358	
3-55	0	0.2001	0.0943	80	200
		5.083	2.395	356	
3-70	0	0.1998	0.0947	75	217
		5.075	2.405	334	
3-71	15	0.1999	0.0950	69	230
		5.077	2.423	307	
3-22	15	0.2000	0.0944	75.5	231
		5.080	2.398	336	
3-11	30	0.2001	0.0948	92	264
		5.083	2.408	409	
3-43	30	0.2000	0.0945	81.5	235
		5.080	2.400	363	
3-60	45	0.2002	0.0940	95	277
		5.085	2.388	423	
3-59	45	0.2000	0.0940	88	257
		5.080	2.388	391	
3-56	45	0.2004	0.0945	110	317
		5.090	2.400	489	

This value of K_{IC} , 6.7 for the part through-crack, is approximately 4% greater than the value of 6.44 found from the through-crack disk specimen.

The value of c , the ratio of the shear toughness to that of the Mode I fracture toughness, is found directly by selecting the value of c that will allow Equation 31 to best fit the indented bar test results.

$$S_f/S_u \cdot \cos^2(A_c) + (S_f \sin(A_c) \cos(A_c)/c S_u)^2 = 1 \quad (31)$$

A_c = angle between crack normal and specimen axis

S_u = fracture strength at a zero angle

S_f = fracture strength at the crack angle

Table 38 summarizes the test data, while Figure 142 illustrates the fit for values of $c = 1.25$ and 1.5. Testing at 45 deg tends to indicate that the value of 1.25 is reasonable. At other angles a value of c equal to the value ($c = 1.52$) obtained from the Brazilian cracked disk test provides a better fit. The lower value of c would indicate a greater shear sensitivity for smaller flaws, but still confirms that the material is not shear sensitive.

5.2 SLOW CRACK GROWTH METHODOLOGY

5.2.1 Uniaxial Slow Crack Growth Theory

Two experimental methods are commonly used to study the SCG behavior in ceramic materials.

- the constant load test (static fatigue)
- the constant loading rate test (dynamic fatigue)

Figure 142. Results of mixed mode room temperature fast fracture testing of PY-6 Vicker's indented Type-B MOR bars used to determine both the Mode I and the shear fracture toughness for part through-cracks.

In the constant load test, the rupture time of the specimens is determined at various applied stress levels. Dynamic fatigue testing involves the measurement of failure strength at various stressing rates. The same material data are obtained from both types of testing. First, the applicable static fatigue analytic methodology will be developed.

5.2.1.1 Static Fatigue Methodology. The governing static fatigue Equation 32 was obtained from Reference 46. This equation utilizes an activation energy term that is used to consolidate data obtained at various temperature levels into a single relationship. This feature reduces the amount of test data required to a limited number of temperature levels. To fit data at a single temperature, the activation energy term is set equal to zero and the material constants B and N are obtained from a fit of the simplified equation.

Constant load:

$$T_f = B \cdot S_i^{N-2} \cdot S^{-N} \cdot \exp(Q/RT) \quad (32)$$

Where:

T_f = time to failure (constant load test)
 S_i = initial strength (fast fracture)
 S = applied stress
 T = temperature (absolute)
 Q = activation energy (material constant)
 R = universal gas constant 0.0083 K-Joules/(Mole-deg K)
 B, N = material constants

The solution technique used to determine the three material constants requires Equation 32 to first be linearized, which allows the least square minimization technique to be applied. This approach is applied by taking the natural logarithms of both sides of the equation; a rearrangement of the equation then allows a 'best' fit of the data by finding material constants which minimize the scatter in terms of the natural log of applied stress. The linearized equation follows:

$$Y = C_1 + C_2 \cdot X_1 + (1+2C_2) \cdot X_2 + C_3 \cdot X_3 \quad (33)$$

Where:

$Y = \ln(S)$
 $X_1 = \ln(T_f)$
 $X_2 = \ln(S_i)$
 $X_3 = 1./T$
 $C_1 = \ln(B)/N$
 $C_2 = -1./N$
 $C_3 = Q/(R \cdot N)$

The constants C_1 , C_2 , and C_3 are found by first writing an expression for the square of the sum of the deviations. By taking the partial derivative with respect to each of the constants and setting to zero, a set of three simultaneous linear equations are obtained. This set of equations is then solved to find the three material constants.

$$\text{Sum} = \sum (C_1 + C_2 \cdot X_{1i} + (1+2C_2) \cdot X_{2i} + C_3 \cdot X_{3i} - Y_i)^2 \quad (34)$$

i = ith test sample

N_s = number of samples tested

After setting the partial derivatives to zero:

$$C1*Ns + C2* \sum X1i+2*X2i + C3 \sum X3i = \sum Yi - X2i \quad (35)$$

$$C1*\sum X1i+2*X2i+C2* \sum (X1i+2*X2i)^2 + C3 \sum X3i*(X1i+2*X2i) = \sum (Yi-X2i)*(X1i+2*X2i)$$

$$C1*\sum X3i + C2* \sum X3i*(X1i+2*X2i) + C3 \sum X3i^2 = \sum Yi*X3i-X2i*X3i$$

Equation 35 is solved for C1, C2, and C3, then N, Q, and B are determined by rearranging Equation 33 into 36.

$$\begin{aligned} N &= -1/C2 \\ B &= \text{Exp}(-C1/C2) \\ Q &= -R*C3/C2 \end{aligned} \quad (36)$$

5.2.1.2 Dynamic Fatigue Methodology. Dynamic fatigue tests determine the SCG material constants similar to static fatigue, only the testing procedure and the form of the governing equation change. It should be noted that the material constants should be identical, regardless of which test is used. Specimen testing consists of applying stress at a constant loading rate and measuring the fracture strength. The applicable equation, which also uses an activation energy to consolidate temperature data, follows:

$$Sf = (B*(N+1)*Si^{N-2} * \dot{S})^{1/(N+1)} * \text{EXP}(Q/RT*(1+N)) \quad (37)$$

Where:

$\dot{S}$ = applied stress rate
Sf = fracture strength

As with the static fatigue testing, a solution technique to determine the three material constants requires that Equation 37 be linearized prior to the application of least square regression optimization techniques. Again the linearization is accomplished by taking the natural logarithms of both sides of the equation. The linearized equation follows:

$$Y = CA + (1-3*CN)*X1 + CN*X2 + CEX*X3 \quad (38)$$

Where:

$Y = \text{Ln}(Sf)$
 $X1 = \text{Ln}(Si)$
 $X2 = \text{Ln}(\dot{S})$
 $X3 = 1/T$
 $CA = \text{Ln}(B*(N+1))/(1+N)$
 $CN = 1/(1+N)$
 $CEX = Q/(R*(1+N))$

The constants CA, CN, and CEX are found by writing an expression for the square of the sum of the deviations. By taking the partial derivative, with respect to each of the constants and setting to zero, a set of three linear equations results. This set of equations is solved to find the three optimum material constants:

$$\text{Sum} = \sum (CA + (1-3*CN)*X1i + CN*X2i + CEX*X3i - Yi)^2 \quad (39)$$

i = i th test sample

N_s = Number of samples tested

After setting the derivatives to zero:

$$CA \cdot N_s + CN \cdot \sum X_{2i-3} \cdot X_{1i} + CEX \cdot \sum X_{3i} = \sum Y_i - X_{1i} \quad (40)$$

$$CA \cdot \sum X_{2i-3} \cdot X_{1i} + CN \cdot \sum 9 \cdot X_{1i}^2 + X_{2i}^2 - 6 \cdot X_{1i} \cdot X_{2i} + CEX \cdot \sum X_{2i} \cdot X_{3i} - 3 \cdot X_{1i} \cdot X_{3i} = \sum Y_i \cdot X_{2i-3} \cdot X_{1i} - X_{1i} \cdot X_{2i+3} \cdot X_{1i}^2$$

$$CA \cdot \sum X_{3i} + CN \cdot \sum X_{2i} \cdot X_{3i} - 3 \cdot X_{1i} \cdot X_{3i} + CEX \cdot \sum X_{3i}^2 = \sum X_{3i} \cdot Y_i - X_{1i} \cdot X_{3i}$$

Equation 40 is solved for CA , CN , and CEX , then N , Q , and B are determined by rearranging Equation 38 into Equation 41.

$$\begin{aligned} N &= (1/CN) - 1 \\ Q/R &= CEX/CN \\ LN(B) &= CA/CN + LN(CN) \end{aligned} \quad (41)$$

5.2.1.3 Slow Crack Growth Methodology Specimen Correlation. Dynamic fatigue behavior of four groups of PY-6 specimens was measured and analyzed using the dynamic fatigue model (Equation 37). The most comprehensive data set was developed based on 120 Type-B MOR bars which failed on the surface. Testing occurred in the temperature range of 1200 to 1400°C. A second set of 29 Type-B MOR bars were tested from 1000 to 1400°C, under a user agreement at Oak Ridge National Laboratories and directed by Dr. M. K. Ferber. A third set of 28 button-head tensile specimens were tested by Southern Research Institute (SoRI) at various temperatures and loading rates. While a final group of 18 dogbone tensile specimens were tested at NIST under Dr. S. M. Wiederhorn.

In order to avoid the inclusion of creep failures in the SCG model, specimens which contained creep damage were deleted. The maximum temperature is limited to 1200°C except for the NIST data which include 1300°C failures at times less than 3 hours. Metallurgical examination of the 1300°C button-head tensile bars has not identified the presence of creep damage at such short times. Table 39 summarizes the material constants obtained from each data set.

Table 39. A comparison of PY-6 dynamic fatigue SCG material constants obtained using differing specimens and test facilities

Source	Temperature —°C	No. of specimens	Specimen type	Norton exponent— N	Coefficient MPa ² — sec— B ₁	Activation energy KJ/mole— Q
Allison	1200	47	Type-B MOR	27.9	--	--
Oak Ridge	1000-1200	20	Type-B MOR	29.5	1.3E-3	164
SoRI	1000-1200	16	Button-head Tensile	31.1	2.8E-4	207
NIST	1000-1300	10	Dogbone Tensile	29.8	8.98E-2	165

All four data sets produce values of the crack velocity exponent, N , which differ by a maximum of 10%. The activation energy, Q , was also consistent. The Allison MOR data set included only 1200°C information, which does not permit the computation of an activation energy. The value of the coefficient, B , which is quite sensitive to the fast fracture strength estimate, was less consistent.

Figure 143 uses the material constants derived from dynamic test results to predict static test results. In the figure, the dynamic fatigue constants determined from the 2 Type-B MOR and the NIST dogbone specimen tests are used to predict the button-head static fatigue data. The 1200°C button-head initial fast fracture strength of 311 MPa (Table 40) is used in Equation 32, the static fatigue equation. Two of the three predictions are within 20 MPa of the baseline button-head static predictions. Predictions made using the Allison MOR bars were about 45 MPa too high. The fact that N is consistent produced the similar trends in terms of strength versus time.

Figure 143. Prediction of PY-6 button-head tensile specimen SCG static fatigue behavior at 1200°C using dynamic fatigue test material constants.

Table 40. Initial strength of PY-6 specimens tested in dynamic fatigue

Temperature —°C	Allison Type-B — MPa	Oak Ridge Type-B — MPa	NIST dogbone—MPa	SORI Button-head—MPa
1000	--	755.3	390.4	379.4
1100	--	585.7	387.1	313.7
1150	--	--	385.4	--
1200	617.8	617.8	383.8	311.2
1300	578.4	578.4	335.4	383.9

Overall, considering the small size of some of the data sets, the results appear consistent and yield SCG constants which can be used in a probabilistic model. Both the static and dynamic fatigue data regression models have been added to the CARES program.

One important point should be noted. The SCG equations using an activation energy to consolidate temperature data allow limited testing to reasonably define the material properties over a large temperature range. Such an approach compromises fit at any single temperature. In particular, the equations assume that the slope of strength versus temperature, the crack velocity exponent, is temperature independent. Data generally show that this slope increases at higher temperatures, but within reasonable temperature ranges this does not introduce a great deal of error.

If a component is designed to operate at a single temperature, material testing and data regression may be directed to that single temperature. Decisions of this type are engineering decisions, specific to the design situation and vary accordingly. By making available general regressions employing activation energy, single temperature data may be consolidated by fixing the value of activation energy, while limited data within a large temperature range may be consolidated to allow for a component which is expected to experience a large range of operation.

The button-head tensile specimens were tested both in dynamic and static fatigue. An analysis of the static data, using Equation 32 resulted in SCG constants somewhat different from those obtained from the dynamic tests. As can be seen in Table 41, the crack velocity exponent (N) is 50.9 versus 31.1 from dynamic testing.

When static test data were predicted by using dynamic material constants at 1200°C in Figure 144, the dynamic fatigue model predicted higher strengths than were observed. This may indicate a higher level of environmental damage associated with the constant load tests, and therefore for PY-6 the static SCG fatigue data should be used in order to ensure an accurate component life prediction.

5.2.1.4 Probabilistic Slow Crack Growth Model. After SCG data have been analyzed it is possible to approximate the Weibull modulus representing the scatter in strength at a fixed point in time using the following equations:⁴⁹

$$\begin{aligned} \text{Sum} &= \sum ((S_{fi} - S_{fp}) / S_{fp})^2 \\ C_v &= (\text{Sum} / (N_s - 3))^{0.5} \\ m_{SCG} &= 1.2 / C_v \end{aligned} \quad (42)$$

Where:

S_{fi} = observed static strength
 S_{fp} = predicted static strength
 N_s = number of specimens tested
 C_v = static coefficient of variation
 m_{SCG} = Weibull modulus in SCG

Table 41. A comparison of PY-6 1200 C SCG material constants obtained from static and dynamic testing of button-head tensile specimens

Type of test	Number of specimens	Crack velocity exponent (N)	Activation energy — KJ/M (Q)	Coefficient — MPa ² -sec (B1)
Dynamic fatigue	16	31.1	207	2.8E-4
Static fatigue	31	50.9	587	1.88E-25

Figure 144. Comparison of PY-6 button-head tensile specimen SCG static fatigue behavior at 1200°C with predictions made using the static fatigue model along with material constants obtained from both static and dynamic test.

Fundamental to the application of SCG to a complex structure requiring a finite element analysis is the concept that the Weibull modulus applicable to SCG is the same as that obtained from fast fracture testing. This follows from the idea that the fast fracture modulus obtained from fracture testing describes the initial distribution of component flaws and the growth of these pre-existing flaws under load should not appreciably change this distribution.

Usually, the value of the SCG modulus agrees reasonably well with that obtained in fast fracture. Occasionally, however, the SCG Weibull modulus appears much higher than that obtained in fast fracture. If this happens, conservative design practice suggests 'adjusting' the value of N in SCG until the value of m in SCG is similar to the fast fracture value.

5.2.2 Application of Slow Crack Growth Methodology to Component Design

Static fatigue methodology has been incorporated into the CARES software, allowing the prediction of the time dependent failure probability of a component subjected to a constant loading. This situation closely approximates the operation of a gas turbine engine component.

In general, while the loading is constant, stresses vary throughout the component, thus MSC-NASTRAN is used to determine the component stresses as well as elemental areas and volumes.. The following set of equations are then used to develop an equation allowing the time dependent failure survival probability to be determined for a single element.⁴⁷

$$dc/dt = A \cdot K_I^N \quad (43)$$

$$K_I = Y \cdot S_a \cdot c^{0.5}$$

$$K_{Ic} = Y \cdot S \cdot c^{0.5}$$

$$P_o = \text{Exp}(-\text{Area}(S_a/S_o)^m)$$

Where:

dc/dt = crack growth rate

c = crack length

A = crack velocity coefficient

N = crack velocity exponent

K_{Ic} = Mode I critical fracture intensity

K_I = Mode I fracture intensity

S = strength

S_a = applied stress

S_o = unit characteristic strength

Area = elemental surface area

Y = magnification factor

P_o = fast fracture probability of survival (Weibull)

m = fast fracture Weibull modulus

The final equation applicable to an element may be written in the following form:

$$POS = P_o (1 + t \cdot S_o^2 / B1 (-\ln(P_o) / \text{Area})^{2/m} m / (N-2)) \quad (44)$$

Where:

POS = time dependent failure probability

$B1$ = slow crack growth coefficient at element temperature

t = time of applied static load

The value of P_o may be computed by any of several FF methods available in CARES, and then the survival probability of an element after time, may be found. The component survival probability is the product of the elemental survival probabilities. Two SCG material properties are required N , the crack velocity exponent, and $B1$, the coefficient. If an activation energy is not used $B1 = B$. Otherwise, if an activation energy is used $B1$ is obtained from Equation 45 which accounts for the variation with temperature:

$$B1 = B \cdot \text{EXP} (Q/RT) \quad (45)$$

For shear insensitive materials such as PY-6 and NT154, P_o is conservatively obtained using the Weibull PIA model.

5.2.3 Slow Crack Growth Methodology. Application to a General Mission

From Reference 48, the slow crack growth failure probability may be computed by using a transformation of the stress distribution at failure to the equivalent effective stress distribution at time $t = 0$ and then employing fast fracture methodologies.

Equation 46 defines this effective stress in the most general form:

$$Seq, o = \left(\frac{\int_0^{tf} S^N dt}{B1} + S(tf)^{N-2} \right)^{\frac{1}{N-2}} \quad (46)$$

Where:

B1, N = SCG material constants
 tf = failure time
 S(tf) = stress at failure
 S = time dependent stress

For a component subjected to several time periods of constant load, followed by a final load to failure, this relationship can be integrated to the form:

$$\text{Seq, o} = (t_1 S_1^N / B_1 + t_2 S_2^N / B_1 + \dots S(t_f)^{N-2} / (N-2)) \quad (47)$$

Where:

t1, t2.... = time spent at each load condition
 S1, S2.... = stress at each condition

In Equation 47 the time spent changing stress levels is neglected. The stresses must also monotonically increase with time, otherwise beneficial residual stresses formed at the crack tip may retard the crack growth rate.

This equation can be used to predict component failure such as a rotating turbine disk. A typical disk is spun at two or more rotational speeds, but the Allison Cares program is currently limited to the analysis of failure probability for only a single loading condition. Thus, it was necessary to operate CARES at each rotational speed or operating point, find the survival probability after a fixed time, and compute the overall value of B1 applicable to the component and mission. The finite element program also defined the peak local stress used in finding the characteristic strength and the overall value of B1. The equations follow:

$$S_{th} = S_{max} / (\ln(1/POS))^{1/m} \quad (48)$$

Where:

Smax = the maximum component stress
 POS = the fast fracture survival probability
 Sth = characteristic strength

$$B_i = t_i S_{max_i}^2 / (((\ln(1/POS))^{(N-2)/m} / (S_{max} / S_{th})^{N-2}) - 1.0) \quad (49)$$

Where:

POS = CARES probability of survival after time ti at Smaxi

It was necessary to slightly modify Equation 47 to account for differing overall values of B for the differing load conditions:

$$\text{Seq, o} = (t_1 S_1^N / B_1 + t_2 S_2^N / B_2 + \dots S(t_f)^{N-2} / (N-2)) \quad (50)$$

Where:

B1, B2 ... = overall values of B for each loading condition
 S1, S2 ... = maximum stress for each loading condition

Finally a probability of survival could be computed by using the Weibull equation:

$$POS = \text{Exp} (\text{Seq,o} / S_{th})^m \quad (51)$$

In the verification section this methodology is shown to result in failure predictions near those observed.

5.3 CREEP ANALYSIS METHODOLOGY

5.3.1 Creep/Stress Rupture Model

At extremely high temperatures, particularly at low stress levels and high lives, the creep damage mechanism becomes of importance. For the high strength silicon nitride materials, PY-6 and NT154, the onset of creep occurs between 1300 and 1400°C.

While there are a number of equations which adequately describe the creep rupture phenomenon, the 'classical' Norton equation⁴⁶ was chosen to describe the relationship between creep rate and applied stress. The 'classical' Monkman-Grant equation was chosen to relate rupture time to creep rate. By combining these equations a relationship between rupture time and applied load is obtained:

$$Cr = B * S^N * EXP(-Q/RT) \quad \text{Norton Equation} \quad (52)$$

$$Tf = A * Cr^a \quad \text{Monkman-Grant} \quad (53)$$

$$Tf = A * B^a * S^a * N * EXP(-a * Q/R * T) \quad \text{Combined} \quad (54)$$

Where:

- Cr = creep rate 1./hr
- S = static applied stress, strength, MPa
- T = absolute temperature, K
- Tf = rupture time, hr
- N = Norton constant (material constant)
- a = Monkman-Grant material constant (about -1)
- A = material coefficient, hr^{1+a}
- B = material coefficient, MPa^{-N}/hr
- Q = activation energy (material constant), KJ/gm Mole
- R = universal gas constant 0.0083 KJ/(gm Mole-K)

When both creep rate and rupture data are available it is necessary to fit the data in such a way as to obtain an optimum estimate of the five material constants. This is done by first writing Equations 52 and Equation 54 with the applied stress on the left side of the equation. Both equations are linearized by taking the natural logarithms of the equations. Minimizing scatter in terms of the logarithm of the stress determines material constants. This is done by writing expressions for the sum of the deviations for each equation, adding the two sums and forming a set of five linear equations by taking the partial derivatives with respect to each constant and setting equal to zero:

$$Y = C1 + C2 X1 + C3 X2 + 0 X3 \quad \text{(From Equation 52)} \quad (55)$$

$$Y = C4 + 0 X1 + C3 X2 + C5 X3 \quad \text{(From Equation 54)} \quad (56)$$

Where:

- $Y = \ln(S)$
- $X1 = \ln(CR)$
- $X2 = 1/T$
- $X3 = \ln(Tf)$
- $C5 = 1/aN$
- $C1 = -\ln(B)/N$
- $C2 = 1/N$
- $C3 = Q/NR$
- $C4 = C1 - C5 \ln(A)$

$$\text{Sum1} = \sum_{Nb} (C1 + C2 X1i + C3 X2i - Yi)^2 \quad (57)$$

$$\text{Sum2} = \sum_{Na} (C4 + C3 X2i + C5 X3i - Yi)^2$$

$$\text{Sum} = \text{Sum1} + \text{Sum2}$$

Setting the five partial derivatives to zero results in five linear equations, Equations 58 through 62:

$$C1 Nb + C2 \sum_{Nb} X1i + C3 \sum_{Nb} X2i + C4 * 0 + C5 * 0 = \sum_{Nb} Yi \quad (58)$$

$$C1 \sum_{Nb} X1i + C2 \sum_{Nb} X1i^2 + C3 \sum_{Nb} X1i * X2i + C4 * 0 + C5 * 0 = \sum_{Nb} X1i * Yi \quad (59)$$

$$C1 \sum_{Nb} X2i + C2 \sum_{Nb} X1i * X2i + C3 \sum_{Nc} X2i^2 + C4 \sum_{Na} X2i + C5 * \sum_{Na} X2i * X3i = \sum_{Nc} X2i * Yi \quad (60)$$

$$C1 * 0 + C2 * 0 + C3 \sum_{Na} X2i + C4 Na + C5 * \sum_{Na} X3i = \sum_{Na} Yi \quad (61)$$

$$C1 * 0 + C2 * 0 + C3 \sum_{Na} X2i * X3i + C4 \sum_{Na} X3i + C5 * \sum_{Na} X3i^2 = \sum_{Na} Yi * X3i \quad (62)$$

Where:

Na = number of failure times

Nb = number of creep rate data points

Nc = Na + Nb

After solving Equations 58 through 62 for the constants C1...C5, the fundamental material constants can be found:

$$N = 1/C2 \quad (63)$$

$$Q/R = C3/C2$$

$$\ln(B) = -C1/C2$$

$$a = C2/C5$$

$$\ln(A) = (C1 - C4)/C5$$

Not only is this methodology incorporated into the Allison CARES program but additional flexibility has been added, allowing the engineer to separately regress creep rate information and stress rupture data. Furthermore, the software, if requested, will search for outliers and systematically remove them from the data set. The user may fix the values of either the Monkman-Grant exponent, a , the Norton exponent, N , or the activation energy, Q .

Table 42 summarizes creep rate and rupture time information on a number of PY-6 tensile bars tested at Southern Research Institute. A complete regression of both the creep rate information and the rupture time data was made to generate the material constants. The resultant material constants, applicable from 1300°C to 1400°C follow:

$$A = 1.177 \text{ E } -4 \text{ sec}^{a+1}$$

$$B = 4.54 \text{ E } 26 \text{ MPa}^{-N} \text{ -sec}$$

$$N = 14.61$$

$$Q = 2010 \text{ KJ/Mole}$$

$$a = -1.1544$$

Table 42. Creep rupture of PY-6 button-head tensile bars tested by Southern Research Institute

Specimen ID	Temperature— °C	Applied stress— MPa	Creep rate — 1/sec	Rupture time —hours
TNAX341B	1300	180.17	6.04 E-08	3.30
TNAX188B	1300	175.27	1.63 E-09	772
TNAX523A	1300	164.8	1.88 E-09	4.99
TNAX634B	1300	150.74	1.21 E-09	624
TNAX626B	1300	149.97	8.99 E-10	473
TNAX531B	1300	140.49	6.68 E-10	306.1
TNAX467A	1300	164.7		109.41
TNAX524A	1370	105.37	1.95 E-07	8.13
TNAX490B	1370	99.75	1.69 E-07	0.610
TNAX522A	1370	95.37	9.04 E-10	146
TNAX535B	1370	95.29		73.94
TNAX602B	1370	90.33	4.59 E-07	7.87
TNAX90A	1370	86.95	6.03 E-09	101
TNAX476A	1370	85.19	1.58 E-09	413
TNAX57A	1370	79.51	2.51 E-09	587.5
TNAX80A	1370	74.67	5.68 E-09	387
TNAX84B	1370	73.91	6.02 E-09	287
TNAX522B	1370	70.47	8.41 E-10	287
TNAX521B	1370	90.69		105.63
TNAX576A	1370	131.2		230
TNAX563A	1370	109.83	8.35 E-10	997.6
TNAX340A	1400	174.37	1.33 E-05	0.015
TNAX60A	1400	150.66	2.10 E-06	0.025
TNAX548B	1400	130.13	4.96 E-08	1.48
TNAX171A	1400	125.35	3.51 E-07	1.96
TNAX552B	1400	114.9	3.10 E-08	172.3
TNAX530B	1400	110.33	3.93 E-08	2.89
TNAX195B	1400	99.98	3.58 E-08	12.97
TNAX611A	1400	95.47	5.01 E-08	10.12
TNAX612B	1400	90.23	1.10 E-08	65.74
TNAX523B	1400	85.66	4.07 E-08	4.50
TNAX59A	1400	84.66	2.24 E-07	1.80
TNAX497A	1400	79.85	4.36 E-09	217.5
TNAX192B	1400	75.09	2.02 E-09	844.
TNAX486B	1400	79.80		266.

Failure data at 1300°C shorter than 3.3 hours was considered to be slow crack growth failure.

Upon examination several observations may be made. The value of the Monkman-Grant exponent, a , -1.15, is quite close to the value of -1 which is often assumed when data are limited. The value of the activation energy is fairly large, showing the sensitivity of creep and rupture to temperature. The Norton exponent, N , of 14.6, defines a slope somewhat steeper than is generally observed in SCG data.

Figure 145 illustrates the fit of the material model to the stress rupture data at 1300°C, 1370°C, and 1400°C. Visually, the fit is reasonable at all three temperatures. Figure 146 compares the relationship between creep rate and applied stress to that predicted by the material model; again the results look quite good. Figure 147 shows the fit of the Monkman-Grant relationship to failure time versus creep rate information. The Monkman-Grant relationship is temperature independent and the data exhibit a fairly large scatter in creep rates; however, the prediction appears to fit the mean behavior reasonably well. The overall quality of the data fit is particularly encouraging since both creep and rupture time data were incorporated into the relationship.

5.3.2 Statistical Stress Rupture Failure Model

The large amount of scatter observed in the stress rupture strength of the button-head tensile rupture bars (Figure 145) suggests that a prudent component design practice should incorporate a statistical 'weakest link' concept. Such an approach, applicable to stress rupture, was developed and incorporated into the Allison CARES program.

The methodology utilizes 1) the rupture/creep material model and 2) finite elemental stresses, areas, and volumes which are obtained from a NASTRAN analysis of the component. The approach is inherently conservative since the predicted stresses are purely elastic and the stress analysis does not account for the beneficial effect of stress redistribution resulting from creep relaxation.

Figure 145. Predicted versus observed stress rupture failure time for PY-6 button-head tensile bars that failed at elevated temperature.

Figure 146. Predicted versus observed creep rates for PY-6 button-head tensile bars that failed from stress rupture at elevated temperatures.

Figure 147. Comparison of predicted and observed creep results for PY-6 button-head tensile bars based on the application of the Monkman-Grant theory to elevated temperature rupture.

The development of the equations applicable to a statistical creep model follow. First a fundamental part of this methodology is Equation 54. This relationship can be interpreted as relating the average strength in stress rupture to the time spent at a particular temperature level. Upon rearranging Equation 54, Equation 64 may be derived.

$$S_m = (A \cdot B a)^{-1/aN} T_f^{1/aN} \exp(Q/NRT) \quad (64)$$

Where:

S_m = mean strength at time T_f

An assumption is made that at all fixed time "slices" the distribution of strength, but not the mean strength, is constant and described by a Weibull distribution. Thus the Weibull modulus related to creep, m_{cr} , is constant with time. This is similar to the standard practice of characterizing the time dependent SCG strength distribution with the fast fracture Weibull modulus when developing the SCG statistical theory. After a coefficient of variation is determined from the mean creep strength and the data scatter, it is then possible to estimate the value of m_{cr} .

For each tensile test specimen the mean predicted strength is:

$$S_{mi} = (A \cdot B a)^{-1/aN} T_{fi}^{1/aN} \exp(Q/NRT_i) \quad (65)$$

Where:

S_{mi} = predicted mean strength for the i th specimen

T_{fi} = time of load of the i th specimen

T_i = exposure temperature of the i th specimen

A coefficient of variation may be written as:

$$C_v = \left(\sum_{N_{pts}} ((S_{mi} - S_i)/S_{mi})^2 / (N_{pts} - 3) \right)^{0.5} \quad (66)$$

Where:

S_i = observed strength of the i th specimen

N_{pts} = total number of test specimens

From Weibull theory⁴⁹, a Weibull modulus related to the variation of creep strength is approximately:

$$m_{cr} = 1.2/C_v \quad (67)$$

Weibull theory⁴⁹ also relates a unit characteristic strength to specimen geometry, mean strength, and Weibull modulus. Since the mean strength is time dependent and temperature dependent, the unit characteristic strength is also a function of time and temperature. Assuming creep is primarily a volume related parameter (a similar relationship may be used for surface related failures) Equation 68 applies:

$$S_{ocr} = S_m \cdot V_{eff}^{1/m_{cr}} / \Gamma(1 + 1/m_{cr}) \quad (68)$$

Where:

V_{eff} = specimen effective volume

Γ = Gamma function

S_{ocr} = unit characteristic strength in creep

For each of the three principal stresses (Weibull PIA⁵⁰) acting on a finite element of volume V_i , the survival probability may be written as:

$$POS_{ij} = \text{EXP}(-V_i(S_{ij}/S_{ocr})^{mcr}) \quad (69)$$

Where:

S_{ij} = the j th ($j = 1, 3$) principal stress acting on the i th element

POS_{ij} = the probability of survival of the i th element resulting from the j th principal stress

The component survival probability is the product of all the POS_{ij} ; thus at a given time, the survival probability of a component may be computed. Since S_{ocr} also depends upon the local element temperature, this value will vary with location as well as time.

5.4 OXIDATION

As part of this effort, the effect of elevated temperatures and exposure time on the room temperature fast fracture strength of PY-6 was defined both experimentally and analytically. This information was incorporated into a statistical oxidation model which permits the determination of time dependent failure probability as a result of oxidation exposure.

5.4.1 Oxidation Failure Model

The oxidation model is based on four assumptions.

- Oxide thickness increases with time following a log linear relationship.
- Temperature dependence is assumed to be a thermal activation process with an Arrhenius type of temperature dependence.
- The flaw resulting in failure is proportional to the oxide layer thickness.
- Linear elastic fracture mechanics relate fracture strength and flaw size.

The model development and basic ideas follow⁴⁷, with a single major difference, the paper suggested that the accumulation of an oxide coating followed a parabolic diffusion law so the thickness was proportional to the square root of exposure time. Test results showed that for the PY-6 silicon nitride material the rate of oxide accumulation was much less, but was approximately proportional to time raised to a power.

The equations describing the average behavior of oxide thickness growth and the effect of oxidation on room temperature strength can be developed in the following way. First, the empirical equation describing the development of the oxide layer is:

$$Th = C \cdot t^b \cdot \text{EXP}(-Q/R/T) \quad (70)$$

Where

Th = oxide thickness, μm

t = time, hr

T = absolute temperature, K

Q = activation energy (material constant), KJ/Gm Mole

b = diffusion exponent (material constant)

R = universal gas constant, 0.0083 KJ/Gm Mole - K

C = thickness coefficient (material constant), $\mu\text{m}/\text{hr}^b$

From fracture mechanics, the room temperature strength of a specimen with a critical flaw of size, c , may be written as:

$$S_{rt} = K_{Ic} / Y_o * c^{-0.5} \quad (71)$$

Where:

K_{Ic} = room temperature fracture toughness

Y_o = magnification factor

Assuming the critical flaw size in oxidation is proportional to the oxide thickness leads to Equation 72, which relates the measured room temperature strength to the exposure time and temperature.

$$S_{rt} = E * t^{-0.5b} * \text{EXP}(0.5 * Q / R / T) \quad (72)$$

Where:

E = the strength coefficient, a material constant, $\text{MPa-hr}^{0.5b}$

Equation 72 may be used with experimental specimen data to determine the three material constants. This is accomplished by first linearizing Equation 72 by taking the natural logarithm of the equation.

$$Y = C_o + C_1 * X_1 + C_2 * X_2 \quad (73)$$

Where:

$$Y = \ln(S_{rt})$$

$$C_o = \ln(E)$$

$$X_1 = \ln(t)$$

$$C_1 = -0.5b$$

$$X_2 = 1/T$$

$$C_2 = Q/2R$$

Equation 73 is then used in writing an equation for the sum of the variations from the mean equation. By taking a partial derivative of this expression for each material constant and setting to zero, three linear simultaneous equations result. The solution of this equation set determines the optimum values of C_o , C_1 , and C_2 . Finally the basic material constants may be obtained by relating b , Q , and E to these values.

$$\text{Sum} = \sum (C_o + C_1 * X_{1i} + C_2 * X_{2i} - Y_i)^2 \quad (74)$$

Where:

Sum = the sum of the variances from the mean

i = i th specimen

N_s = number of specimens

Setting the partial derivatives of 74) to zero results in Equations 75

$$C_o N_s + C_1 * \sum X_{1i} + C_2 * \sum X_{2i} = \sum Y_i \quad (75)$$

$$C_o \sum X_{1i} + C_1 * \sum X_{1i}^2 + C_2 * \sum X_{1i} * X_{2i} = \sum X_{1i} * Y_i$$

$$C_o \sum X_{2i} + C_1 * \sum X_{1i} * X_{2i} + C_2 * \sum X_{2i}^2 = \sum X_{2i} * Y_i$$

Equations 76, obtained from Equations 73 are used to find Q/R , E , and b .

$$\begin{aligned} Q/R &= 2 \cdot C_2 \\ E &= \text{Exp}(C_0) \\ b &= -2 \cdot C_1 \end{aligned} \quad (76)$$

In developing the material model, 65 Type-B specimens were exposed to temperatures of 1260, 1300, 1371, or 1400°C for periods of time varying from 1 to 500 hours. The thicknesses of the oxide layers were measured and the room temperature flexural strengths of the specimens were also measured.

Since both the oxide thicknesses and strengths are measured, a comparison of predicted activation energies, Q , and the exponent, b , are obtained independently from the two tests. In applying the data to a statistical model the values obtained from MOR testing are fundamental. The values obtained from oxide thickness measurements are provided for comparison and as a partial substantiation of the material model.

Material constants from
thickness measurement

$$\begin{aligned} C &= 167 \mu\text{m}/\text{hr}^b \\ b &= 0.11 \\ Q &= 63 \text{ KJ/Mole} \end{aligned}$$

Material constants from
MOR testing

$$\begin{aligned} E &= 1050 \text{ MPa-hr}^{0.5b} \\ b &= 0.067 \\ Q &= -8 \text{ KJ/Mole} \end{aligned}$$

The low value of activation energy obtained from the MOR tests reflects the relative temperature insensitivity of the strength within this temperature range.

Activation energy obtained from thickness measurements shows the rate of oxide thickness does increase with temperature, as would be expected. The slight increase in 1400°C strength, which is responsible for the negative activation energy obtained from MOR testing, probably results from scatter within the small data sets.

Values of b obtained from both types of testing, while not exactly identical, agreed reasonably well and differed greatly from a value of 0.5, which would have been correct had the parabolic diffusion law been applicable. As illustrated in Table 43 and Figure 148, the use of the regression constants obtained from bend testing allow a very reasonable prediction of average strength behavior (each average is based on from 4 to 10 test specimens). A standard deviation on strength is calculated to be 11.7% corresponding to a Weibull modulus of about 10.3. The fact that this exceeds the room temperature fast fracture surface Weibull modulus of 8.65 indicates the oxidation process results in a greater consistency of critical surface flaw sizes.

While the room temperature fast fracture strength is predicted by the model, the oxide thickness does not correlate as well (see Table 44 and Figure 149). Based upon measured thickness data, a standard deviation is 26.4% compared to a value of only 11.7% for the strength data. In Table 45 the strengths of the individual specimen tests were compared to the ± 1 standard deviation values. As expected, about 70% of the data lay within this scatter band. Examining the predictions made at each of the four temperatures shows the percentage of specimen strengths in the predicted scatter band varied from 60 to 80%, indicating the data fit was not strongly biased at a particular temperature. This is consistent with the small activation energy, which implies little variation of strength within the test temperatures.

5.4.2 Probabilistic Oxidation Model

Once the basic oxidation strength model has been developed, a probabilistic oxidation model may be generated. This model is applicable to components which are exposed to a constant temperature environment.

Table 43. A comparison of PY-6 Type-B MOR bar room temperature fast fracture strength after temperature exposure with the predicted strength

Temperature —°C	Time— hr	Average strength—MPa	Predicted strength—MPa	Ratio actual/predicted
1260	100.00	659.68	653.05	1.01
1300	100.00	710.61	658.42	1.08
1300	500.00	564.56	624.09	0.90
1371	100.00	669.60	667.40	1.00
1400	1.00	746.77	781.97	0.95
1400	3.00	780.96	753.90	1.04
1400	6.00	755.45	736.72	1.03
1400	10.00	723.41	724.30	1.00
1400	50.00	699.64	686.54	1.02
1400	100.00	683.55	670.89	1.02
1400	500.00	636.80	635.91	1.00

Figure 148. Predicted versus observed room temperature fast fracture strength of PY-6 Type-B MOR bars after exposure to oxygen at 1400°C.

Table 44. A comparison of PY-6 Type-B MOR bar predicted oxide thickness with the measured value

Temperature —°C	Time— hr	Average thickness— μm	Predicted thickness— μm	Ratio actual/predicted
1260	100.00	1.91	1.95	0.98
1300	100.00	2.56	2.22	1.16
1300	500.00	3.06	2.63	1.16
1371	100.00	2.30	2.73	0.84
1400	1.00	1.67	1.80	0.92
1400	3.00	2.70	2.03	1.33
1400	6.00	2.80	2.18	1.28
1400	10.00	1.70	2.31	0.74
1400	50.00	2.40	2.74	0.87
1400	100.00	3.20	2.96	1.08
1400	500.00	4.25	3.51	1.21

Figure 149. Predicted versus measured PY-6 Type-B MOR bar oxide thickness after exposure to air at elevated temperatures.

Table 45. A comparison of PY-6 Type-B MOR bar room temperature fast fracture strength with the predicted strength after exposure to oxygen at elevated temperatures

Temperature —°C	Time— hr	Specimen strength— MPa	Predicted + 1 standard deviation	Predicted - 1 standard deviation	Is data in scatter band?
1260	100	636.89	729.20	576.91	Y
1260	100	545.20	729.20	576.91	N
1260	100	577.14	729.20	576.91	Y
1260	100	634.61	729.20	576.91	Y
1260	100	576.17	729.20	576.91	N
1260	100	701.69	729.20	576.91	Y
1260	100	700.37	729.20	576.91	Y
1260	100	809.92	729.20	576.91	N
1260	100	662.81	729.20	576.91	Y
1260	100	751.96	729.20	576.91	N
1300	100	762.35	735.19	581.65	N
1300	100	710.49	735.19	581.65	Y
1300	100	642.21	735.19	581.65	Y
1300	100	764.51	735.19	581.65	N
1300	100	673.49	735.19	581.65	Y
1300	500	520.55	696.86	551.33	N
1300	500	586.31	696.86	551.33	Y
1300	500	554.03	696.86	551.33	Y
1300	500	597.35	696.86	551.33	Y
1371	100	657.91	745.22	589.59	Y
1371	100	548.61	745.22	589.59	N
1371	100	702.61	745.22	589.59	Y
1371	100	677.76	745.22	589.59	Y
1371	100	611.89	745.22	589.59	Y
1371	100	598.42	745.22	589.59	Y
1371	100	720.77	745.22	589.59	Y
1371	100	674.42	745.22	589.59	Y
1371	100	687.51	745.22	589.59	Y
1371	100	816.11	745.22	589.59	N
1400	1	784.94	873.14	690.80	Y
1400	1	822.87	873.14	690.80	Y
1400	1	731.89	873.14	690.80	Y
1400	1	834.24	873.14	690.80	Y
1400	1	559.93	873.14	690.80	N
1400	3	891.19	841.80	666.00	N
1400	3	841.32	841.80	666.00	Y
1400	3	585.74	841.80	666.00	N
1400	3	709.24	841.80	666.00	Y
1400	3	877.29	841.80	666.00	N
1400	6	692.55	822.61	650.82	Y
1400	6	765.61	822.61	650.82	Y
1400	6	890.49	822.61	650.82	N
1400	6	754.04	822.61	650.82	Y

Table 45 (cont).

Temperature —°C	Time— hr	Specimen strength— MPa	Predicted + 1 standard deviation	Predicted - 1 standard deviation	Is data in scatter band?
1400	6	674.55	822.61	650.82	Y
1400	10	758.05	808.75	639.85	Y
1400	10	706.13	808.75	639.85	Y
1400	10	694.90	808.75	639.85	Y
1400	10	875.19	808.75	639.85	N
1400	10	582.80	808.75	639.85	N
1400	50	688.65	766.58	606.49	Y
1400	50	751.41	766.58	606.49	Y
1400	50	723.70	766.58	606.49	Y
1400	50	643.56	766.58	606.49	Y
1400	50	690.90	766.58	606.49	Y
1400	100	753.89	749.11	592.66	N
1400	100	650.56	749.11	592.66	Y
1400	100	754.06	749.11	592.66	N
1400	100	717.98	749.11	592.66	Y
1400	100	541.26	749.11	592.66	N
1400	500	622.03	710.05	561.76	Y
1400	500	693.88	710.05	561.76	Y
1400	500	608.47	710.05	561.76	Y
1400	500	593.40	710.05	561.76	Y
1400	500	666.21	710.05	561.76	Y

The development of the equations applicable to a statistical model follow. A fundamental part of this methodology is Equation 72 which relates the average room temperature strength to the elevated temperature exposure time and the temperature. For a shear insensitive material, the strength at temperature will be proportional to the ratio of the Mode I critical fracture intensities; thus the elevated temperature mean strength may be written as:

$$St = Kic(T)/Kic(RT) * E * t^{-0.5b} * EXP(0.5 * Q/R/T) \quad (77)$$

Where:

St = elevated temperature strength

Kic(T) = Mode I fracture intensity at temperature

Kic(RT) = Mode I fracture intensity at room temperature

If a coefficient of variation is first determined, it is then possible to derive an estimation of the average Weibull modulus applicable to the strength variation at a fixed exposure time.

For each tensile test specimen, the mean room temperature strength is predicted by Equation 72 to be:

$$Srti = E * t_i^{-0.5b} * EXP(0.5 * Q/R/T_i) \quad (78)$$

Where:

t_i = time of exposure of the i th specimen
 T_i = exposure temperature of the i th specimen

A coefficient of variation may be found from the following equation:

$$C_v = (\sum ((S_{rti} - S_i)/S_{rti})^2 / (N_s - 3))^{0.5} \quad (79)$$

Where:

S_i = observed strength of the i th specimen at room temperature

From Weibull theory⁴⁹, a Weibull modulus related to the variation of strength and assumed to be independent of temperature is approximately:

$$m_o = 1.2/C_v \quad (80)$$

Weibull theory⁴⁹ also relates a unit characteristic strength to specimen geometry, mean strength, and Weibull modulus. Since the mean strength is dependent on exposure time and temperature, the unit characteristic strength is also a function of time and temperature.

$$S_{oo} = S_t * A_{spec}^{1/m_o} / \Gamma(1 + 1/m_o) \quad (81)$$

Where:

A_{spec} = specimen effective surface area
 Γ = Gamma function

For each of the principal stresses (Weibull PIA) acting on a finite element of area A_i , the survival probability may be written as:

$$POS_{ij} = \exp(-A_i * (S_{ij}/S_{oo})^{m_o}) \quad (82)$$

Where:

S_{ij} = the j th ($j = 1, 3$) principal stress acting on the i th element

The component survival probability is the product of all the POS_{ij} , and after a given time at temperature, the survival probability of a component may be computed. At the start of operation this survival probability is computed to be one, as would be expected.

It should be noted that this methodology does not apply in situations where the presence of stress accelerates the rate of oxidation of a flaw. Such situations can be considered using a sub-critical crack growth methodology in which the specimen test data include the effects of oxidation.

As a general ceramic design methodology, statistical approaches encompass the proposed component lifing methodology. Survival probabilities associated with fast fracture define the time independent failure situation. The time dependent failure probability is determined by the survival probabilities associated with SCG, creep rupture, and oxidation. Depending upon the temperatures and materials, one or all three of these failure mechanisms may affect ceramic component reliability.

VI. CONFIRMATORY TEST CORRELATION

An important element of the monolithic ceramic lifing program was the verification of the life prediction models. These confirmatory specimens were designed to produce both fast fracture and time dependent failures. The life prediction models were used to predict the life of each test. The predictions were compared to the observed results in order to determine the degree of correlation.

During the course of this program, the Allison ceramic supplier GTE discontinued all efforts to develop and supply ceramic materials. As a result, while flat circular plate specimens were available for biaxial load tests, PY-6 spin disk specimens of acceptable quality for test were not delivered. Through the program monitor, and with the aid and cooperation of AlliedSignal Aerospace Company, Norton NT154 spin test data and a uniaxial material data base were transferred to Allison to allow for verification of the methodology.

Table 46 summarizes the various multiaxial verification tests involved in the program. The tests include both fast fracture and SCG. The 1371°C spin disk was expected to fail from creep during testing but analysis indicated that, although creep was observed in uniaxial specimens, failure was predicted much more closely by the SCG model.

Table 46. A summary of the multiaxial slow crack growth and fast fracture verification tests for PY-6 and NT154 materials

Component	Type of loading	Material	Temperature— °C	Predicted failure mode
Flat circular plate	Center ball	PY-6	Room temperature	Multiaxial fast fracture
Flat circular plate	Center ball	PY-6	1200	Multiaxial slow crack growth
Flat circular plate	Center ball	PY-6	1300	Multiaxial slow crack growth
Flat circular plate	Uniform pressure	NT154	Room temperature	Multiaxial fast fracture
Spin disk	Rotation	NT154	Room temperature	Multiaxial fast fracture
Spin disk	Rotation	NT154	1204	Multiaxial slow crack growth
Spin disk	Rotation	NT154	1371	Multiaxial slow crack growth

6.1 FAST FRACTURE BIAXIAL CONFIRMATORY TESTS

6.1.1 Ball-on-Ring Confirmatory Testing

Battelle Research Laboratories of Columbus, Ohio, conducted the biaxial strength measurements at room temperature using flat circular PY-6 disk specimens of 31.82 mm nominal diameter and 2.5 mm thickness. The disks surfaces were machined using a protocol developed at Battelle. These specimens were tested with a ball-on-ring arrangement, loaded at the center by a 6.35 mm diameter ball, and supported at a 25.4 mm diameter. MOR specimens, used to predict the biaxial strength and verify the fast fracture models, were cut from machined disks to ensure both types of specimens had identical surface characteristics.

Maximum disk stress occurs on the tensile surface at the center of the disk where both radial and tangential stresses are equal and given⁵¹ as:

$$S_{max} = 3P(1+\mu)/4\pi t^2 (1 + 2 \ln a/b + (1-\mu)/(1+\mu) * (1-b^2/2a^2)a^2/R^2) \quad (83)$$

Where:

P = applied load

t = disk thickness

a = support radius

b = radius of center load (b=t/3)

R = disk radius

μ = Poisson's ratio (0.28 at room temperature)

π = 3.14159

Table 47 summarizes the disk test data along with the computed maximum stress.

Table 47. A summary of the room temperature PY-6 circular flat plate ball-on-ring fast fracture surface test results

Specimen	Load— lb/Newton	Diameter— in./mm	Thickness— in./mm	Strength— MPa
4-81	570/2535	1.254/31.85	0.1000/2.54	813.44
4-82	538/2393	1.251/31.78	0.1000/2.54	767.97
4-83	480/2135	1.253/31.83	0.1000/2.54	685.06
4-84	548/2438	1.254/31.85	0.1000/2.54	782.04
4-86	600/2669	1.254/31.85	0.1005/2.55	846.48
4-88	572/2544	1.251/31.78	0.0995/2.53	825.90
4-90	540/2402	1.251/31.78	0.0995/2.53	779.70
4-92	520/2313	1.254/31.85	0.0995/2.53	750.63
4-93	510/2269	1.252/31.80	0.0995/2.53	736.32
4-98	505/2246	1.254/31.85	0.1000/2.54	720.68
3-1	520/2313	1.254/31.85	0.099/2.51	759.38
3-2	490/2180	1.253/31.83	0.099/2.51	715.63
3-9	549/2442	1.255/31.88	0.101/2.57	765.67
3-23	483/2148	1.252/31.80	0.099/2.51	705.47
3-34	478/2126	1.253/31.83	0.098/2.49	714.53
3-37	460/2046	1.253/31.83	0.099/2.51	671.82
3-38	531/2362	1.251/31.78	0.099/2.51	775.64
3-77	517/2300	1.254/31.85	0.099/2.51	755.00

At a radius, r , exceeding b , the radial and tangential stresses are obtained using the following three equations:⁵¹

$$C = 3P(1+\mu)/4\pi t^2 \quad (84)$$

$$S_r = C(2 \ln a/r + 0.5(1-\mu)/(1+\mu)(a^2-r^2)b^2/r^2R^2) \quad (85)$$

$$S_t = C(2 \ln a/r + 5(1-\mu)/(1+\mu)(4-b^2/r^2)a^2/R^2) \quad (86)$$

Where:

S_t = tangential stress

S_r = radial stress

At a radius less than b , stresses attenuate less rapidly than given by Equations 85 and 86.⁵¹ A reasonable, but slightly high, estimation of stresses in this local region is that both radial and tangential stresses remain constant and equal to S_{max} at the center.

After several iterations, a promising approach was found allowing the prediction of the biaxial results from uniaxial MOR testing. Initially, Weibull shape and scale parameters obtained from room temperature testing of longitudinally ground Type-B MOR bars were used to predict the biaxial strengths, but results were not encouraging. In particular, the room temperature Weibull modulus of 8.6 differed greatly from the value of 17 found for the disk specimens. Fractographic examination of the ball-on-ring specimens showed failure initiated on the tensile surface along machining marks. To better approximate the observed biaxial results, a set of Type-B MOR bars were manufactured with 320 grit transverse machining and evaluated at room temperature. When these data were analyzed the Weibull modulus was still found to be approximately 8, again resulting in poor correlation. The predicted characteristic strength using the Weibull parameters of the transversely ground Type-B bars was 979 MPa, 26% high (Table 48).

Table 48. Comparison of the room temperature PY-6 ball-on-ring predicted surface characteristic strength with the measured value

Baseline MOR specimen	Applied theory	Weibull exponent —m	Unit characteristic strength—MPa-m ² /m	Effect area—mm ²	Predicted characteristic strength—MPa	Measured characteristic strength—MPa
Battelle* MOR	Weibull PIA	20.19	412.6	4.58	759	776
Battelle* MOR	Stress nor. Mach Marks	20.19	412.6	2.29	785	776
Allison** MOR	Weibull PIA	7.96	218.0	6.32	979	776

* Specimens cut from flat plates

** Standard Type-B MOR specimens

Twenty MOR specimens were machined from ball-on-ring disk specimens. The specimen surface was first ground and polished identical to the biaxial disk specimens. MOR bars were then dissected from the disks so machining marks were transverse to the specimen length. These MOR bars varied minimally in material and surface finish from the disk specimens, and truly represented the surface condition of the biaxial disk specimen. The variables which tend to obscure the verification of a multiaxial predictive methodology were minimized. The Weibull modulus of 20 observed on these MOR specimens, tested in a four-point bending mode, agreed well with that of the disks.

Material properties from the MOR specimens cut from the disks were used to compute the disk characteristic strength using effective areas from two differing multiaxial models. In the first model, only the stress normal to machining marks was assumed to influence the fast fracture failure probability. In the second model, the effective area calculation was based upon the Weibull principal of independent action (PIA). Characteristic strength of the ball-on-ring specimens was predicted using the aforementioned two model approaches and Weibull properties of the transversely ground MOR specimens cut from the flat disks. Results were quite encouraging. The first model predicted a characteristic strength of 785 MPa versus an experimental value of 776 MPa, a difference of only 1%. The PIA value of 759 was within 2% of the measured value. This clearly indicates the degree of difficulty in predicting surface component strengths from the original uniaxial MOR specimen test data. It was necessary to machine MOR specimens from the actual component and test in a manner consistent with the component in order to achieve correlation. It must be recognized that high strength ceramic surface properties are very sensitive to surface forming and machining differences.

The effective area of the Battelle MOR bars was 87.84 mm^2 based upon failure of the ground top surface only (Equation 89). The characteristic strength is related to unit characteristic strength by Equation 87, which was applied to the data in Table 48:

$$S_{th} = S_o / (A_{eff})^{1/ms} \quad (87)$$

Where:

S_{th} = characteristic strength
 S_o = unit characteristic strength
 ms = surface Weibull modulus

In Figure 150, a model based on stresses normal to the machining marks is used along with MOR specimen properties obtained from bars machined from the disks to predict a 90% scatter band for the ball-on-ring disks. This was done by first using Equation 88 to convert the MOR measured strengths to that of the ball-on-ring specimens, which have a smaller effective area. Because of the machining marks on the top surface of the MOR bar, all failures originate on this surface and the effective area is based only on this surface. Equation 89 defines the method of computing this effective area.

$$S_b = S_s (A_s / A_b)^{1/ms} \quad (88)$$

Where:

S_s = measured specimen strength, MPa
 S_b = ball-on-ring strength, MPa
 ms = specimen Weibull exponent, 20.19
 A_s = specimen effective area based only on top surface, mm^2
 A_b = effective area of ball-on-ring, mm^2

Figure 150. PY-6 biaxial fast fracture room temperature ball-on-ring confirmatory test prediction of surface failure probability versus experimental results.

$$As = w L2 + w (L1-L2)/(1+ms) \quad (89)$$

Where:

L1 = outer load span = 28 mm
 L2 = inner load span = 16.8 mm
 w = width = 5.08 mm

Table 49 summarizes the MOR test data, as well as the transformation of strength to that of the smaller ball-on-ring specimens. The strength transformation is based upon the following values:

$As = 87.84 \text{ mm}^2$
 $Ab = 2.29 \text{ mm}^2$
 $ms = 20.19$

The CARES program was then used to find the predicted scatter band. When the measured disk results are plotted the correlation appears to be reasonable. All of the disk data lie within the 90% confidence band with a Weibull slope that is visually close to that predicted.

6.1.2 Fast Fracture Analysis of NT154 Pressurized Flat Plate

Experience has shown that the prediction of biaxial fast fracture failures associated with the surface tends to be difficult. For PY-6 ball-on-ring circular disks, two items were required to make an accurate prediction from uniaxial data.

- failures occurred parallel to machining marks, allowing a failure model to be specifically developed for this situation
- uniaxial specimens were cut from actual disks and machined so the machining marks were normal to the bend axis.

A detailed discussion of the steps necessary to accurately predict the fast fracture biaxial ball-on-ring room temperature testing of PY-6 circular plates was presented in the previous section.

Table 49. Room temperature surface fast fracture strength of PY-6 MOR specimens cut from ball-on-ring flat circular plates

MOR specimen	Strength of MOR bar — MPa	Predicted strength of ball-on-ring specimen — MPa
1	614.3	735.9
2	602.9	722.2
3	604.1	723.7
4	627.4	751.6
5	651.6	780.7
6	665.4	797.1
7	613.3	734.7
8	611.2	732.2
9	586.4	702.5
10	605.0	724.7
11	620.2	743.0
12	597.1	715.3
13	669.8	802.4
14	608.6	729.1
15	692.5	829.6
16	682.4	817.5
17	681.3	816.2
18	671.1	804.0
19	676.9	810.9
20	686.5	822.4

In predicting the biaxial fast fracture of a different NT154 verification test, specimens representative of the specific surface were not available. In this situation, predictions were made using the most conservative available model along with the 'worst case' material data. After examining the surface failure data available from E-bars, Type-B MOR specimens, and tensile bar specimens, it was found that the tensile specimens resulted in the lowest strength predictions. Also the Weibull PIA model was the most conservative of the available material fast fracture models. Even with this conservativeness, the strength was still overpredicted.

Based upon previous experience, a conservative approach was utilized to predict the biaxial fast fracture strength of the Garrett NT154 pressurized circular plate. After examining the results of the normal stress criteria and a number of other models, it was determined that the Weibull PIA model was the most conservative.

Equations defining the stress distribution of a simply supported circular plate loaded with a uniform pressure were obtained from Reference 52. The radial stress, S_r , and the tangential stress, S_t , are functions of radius only and are given by Equations 90 through 94.

$$S_r = S_b (1 - a(r/r_1)^2) \quad (90)$$

$$S_t = S_b (1 - b(r/r_1)^2) \quad (91)$$

$$S_b = 3pr_1^2/8t^2 (2(1-\mu) + (1+3\mu)(r_2/r_1) - 4(1+\mu)(r_2/r_1)^2 \ln(r_2/r_1)) + p(3+\mu)/4(1-\mu) \quad (92)$$

$$a = 3p(3+\mu)r_1^2/8t^2 S_b \quad (93)$$

$$b = 3p(1+3\mu)r_1^2/8t^2 S_b \quad (94)$$

Where:

S_b = peak stress developed in the plate center

r = radial location

r_1 = support radius

μ = Poisson's ratio

p = applied pressure

r_2 = disk radius

t = disk thickness

a, b = parameters which determine the stress gradients.

The relatively simple form of Equations 90 and 91, when substituted into the integral defining the effective area, allows for closed form solutions with both the axial and radial stress fields. When the PIA model is used the effective area is the sum of these two areas. Equations 95, 96, and 97 give the effective area solutions.

$$A_r = \pi r^2/a(ms+1) \quad (95)$$

$$A_t = \pi r^2/b(ms+1) \quad (96)$$

$$A(\text{PIA}) = A_r + A_t \quad (97)$$

Where:

$\pi = 3.14159$

ms = surface Weibull exponent

$A(\text{PIA})$ = effective area to be used in a PIA model

A Weibull MLE analysis of tensile surface failures resulted in the values shown in Table 50, and the E-bar and Type-B constants are also indicated. Note that the E-bar and Type-B MOR specimens have nearly identical surface Weibull properties. Tables 51, 52, and 53 itemize the surface data used in the prediction, and suspended items were not used. As indicated, the most conservative application of data was to be applied. The use of suspended items resulted consistently in a higher mean strength and was thus disregarded.

Table 50. Room temperature NT154 fast fracture material constants obtained from E-bar, Type-B, and tensile button-head surface failures

Specimen type	Weibull exponent— ms	Weibull unit characteristic strength— MPa-mm ² /ms
Tensile	6.219	2313
E-bar	8.105	1803
Type-B	8.070	1818

Table 51. Room temperature NT154 surface fast fracture strength from MOR testing of E-bars

Specimen ID	Strength— ksi/MPa	Failure location
LPEL-1	100.6/693.6	Surface
LPEL-4	97.3/670.9	Surface
LPEL-5	82.0/565.4	Surface
LPEL-7	103.0/710.2	Surface
LPEL-8	107.3/739.8	Surface
LPEL-12	101.4/699.2	Surface
LPEL-14	91.9/633.6	Surface
LPEL-15	116.4/802.6	Surface
LPEL-16	116.1/800.5	Surface
LPEL-22	87.2/601.2	Surface
LPEL-23	125.4/864.6	Surface
LPEL-36	92.7/639.2	Surface
LPEL-37	107.5/741.2	Surface
LPEL2-2	96.2/663.3	Surface
LPEL2-3	115.5/796.4	Surface
LPEL2-5	109.3/753.6	Surface
LPEL2-9	100.9/695.7	Surface
LPEL2-11	127.9/881.9	Surface
LPEL2-15	131.1/903.9	Surface
LPEL2-19	131.4/906.0	Surface
LPEL2-22	113.0/779.1	Surface
LPEL2-31	105.1/724.7	Surface
LPEL2-37	110.5/761.9	Surface
LPEL2-38	113.9/785.3	Surface
LPEL2-40	122.2/842.6	Surface
LPEL2-43	142.1/979.8	Surface
LPEL2-49	112.5/775.7	Surface
LPEL2-54	133.1/917.7	Surface
LPEL2-59	117.7/811.5	Surface
LPEL2-63	107.2/739.1	Surface
LPEL3-2	58.2/401.3	Surface
LPEL3-4	95.4/657.8	Surface
LPEL3-9	103.3/712.3	Surface
LPEL3-14	106.9/737.1	Surface
LPEL3-18	115.3/795.0	Surface
LPEL3-24	109.6/755.7	Surface
LPEL3-25	117.0/806.7	Surface
LPEL3-38	109.4/754.3	Surface

Table 52. Room temperature NT154 surface fast fracture strength from Type-B MOR testing

Specimen ID	Stress— ksi/MPa
LPBL- 3	71.9/495.8
LPBL-10	143.2/987.4
LPBL-14	89.1/614.3
LPBL-20	159.4/1099.1
LPBL-24	139.4/691.2
LPBL-29	130.7/901.2
LPBL-32	172.4/1188.7
LPBL-33	151.3/1043.2
LPBL-35	167.5/1154.9
LPBL-36	142.6/983.2
LPBL-37	159.4/1099.1
LPBL-39	136.2/939.1
LPBL-41	157.8/1088.0
LPBL-44	144.3/994.9
LPBL-48	157.8/1088.0
LPBL-50	127.0/875.7
LPBL-51	144.3/995.0
LPBL-164	137.3/946.7

Table 53. Room temperature NT154 surface fast fracture strength from button-head tensile specimens

<i>Suspended Items Not Included</i>	
Specimen ID	Stress— ksi/MPa
LPTEN3 62	88.8/612.2
LPTEN3 67	97.2/670.2
LPTEN3 84	133.5/920.5
LPTEN3 98	146.8/1012.2
LPTEN3 99	96.2/663.3
LPTEN3 108	116.5/803.3
LPTEN3 117	101.4/699.2
LPTEN4 3	90.8/626.1
LPTEN4 5	144.8/998.4
LPTEN4 7	127.9/881.9
LPTEN4 8	121.7/839.1
LPTEN4 14	101.6/700.5
LPTEN4 21	128.9/888.8
LPTEN4 43	109.8/757.1
LPTEN1 33	86.1/593.7
LPTEN2 13	108.5/748.1
LPTEN2 25	91.6/631.6
LPTEN2 76	91.7/632.3
LPTEN2 151	102.3/705.4
LPTEN3 101	104.7/721.9
LPTEN3 102	101.9/702.6

Table 54 summarizes the fast fracture failure strength obtained from the circular pressurized plates. Equation 97 was applied in the calculation of the equal biaxial peak center stress using the thickness and failure pressure information from Table 54. In addition to the information in the table, the following information describing the support radius, the disk radius, and Poisson's ratio is necessary.

$$\mu = 0.27$$

$$r_1 = 24.13 \text{ mm}$$

$$r_2 = 25.23 \text{ mm}$$

A Weibull MLE analysis of the plate strength information results in a Weibull modulus of 10.404 and a characteristic strength of 808.6 MPa. All plate failures origins were on the tensile surface.

The values of a and b can be shown to depend only upon μ , r_1 , r_2 , and t . The dependence on t is very weak, and t varies only slightly from specimen to specimen. Since the other variables are constant from specimen to specimen, the values of a and b are nearly constant, and thus the

Table 54. Room temperature NT154 surface fast fracture test results obtained from flat circular plates loaded with an increasing uniform pressure until failure

Specimen ID	Fracture pressure— psi	Calculated thickness*— mm	Fracture strain	Fracture strength— MPa
LPPB 5	1998	3.192	0.002169	958.7
LPPB 6	1514	3.194	0.001641	725.3
LPPB 7	1432	3.193	0.001553	686.4
LPPB 8	1746	3.193	0.001894	837.1
LPPB 12	1662	3.188	0.001809	799.6
LPPB 13	1464	3.197	0.001584	700.1
LPPB 14	1729	3.196	0.001872	827.4
LPPB 15	1775	3.198	0.001920	848.6
LPPB 16	1549	3.199	0.001674	739.9
LPPB 17	1765	3.199	0.001908	843.3
LPPB 18	1882	3.200	0.002033	898.6
LPPB 19	1498	3.200	0.001618	715.1
LPPB 20	1338	3.201	0.001444	638.2
LPPB 21	1788	3.195	0.001937	856.1
LPPB 22	1752	3.199	0.001893	836.7
LPPB 23	1510	3.199	0.001632	721.3
LPPB 24	1578	3.196	0.001709	755.4
LPPB 25	1159	3.191	0.001259	556.5
LPPB 26	1514	3.193	0.001642	725.7
LPPB 27	1402	3.186	0.001528	675.4
LPPB 28	1736	3.188	0.001889	834.9
LPPB 29	1429	3.193	0.001550	685.1
LPPB 30	1423	3.192	0.001545	682.9
LPPB 31	1800	3.191	0.001955	864.1
LPPB 40	1687	3.192	0.001831	809.3
LPPB 41	1426	3.191	0.001549	684.6
LPPB 42	1751	3.190	0.001903	841.1
LPPB 43	1489	3.189	0.001617	714.7
LPPB 44	1774	3.192	0.001926	851.3
LPPB 45	1368	3.193	0.001484	655.9
LPPB 46	1602	3.187	0.001744	770.8
LPPB 47	1655	3.187	0.001802	796.5
LPPB 48	1668	3.188	0.001815	802.2
LPPB 49	1583	3.177	0.001735	766.8
LPPB 50	1668	3.168	0.001838	812.4
LPPB 51	1559	3.168	0.001718	759.3
LPPB 52	1794	3.169	0.001976	873.4
Average =			0.001746	771.6

* The thickness information was not directly available but a correlation in the following form had been provided:

$$e t^2 = 0.1106 E^{-4} p \quad \text{where } e = \text{measured strain, } e t^2 \text{ was in mm}^2, p = \text{psi}$$

Table 54 (cont).

From this a calculated thickness in mm was obtained for each specimen.

$$t = (0.1106 E^{-4} p/e)^{0.5}$$

The elastic modulus was found to be about 322.6 GPa for the circular plate by relating the average center biaxial stress to the average measured strain.

effective disk areas equation 97 vary only with the Weibull exponent, ms. The parameters a and b are computed using the average thickness of 3.191 mm.

$$a = 1.0082$$

$$b = 0.55805$$

Three sets of uniaxial NT154 fast fracture material data were available. The button-head tensile specimen data were the most conservative and were first used in the analysis. As can be seen in Table 55, the predicted characteristic strength of 805.6 MPa was only slightly below the observed disk value of 808.6 MPa. As illustrated in Figure 151, the Weibull slope of 6.2 is less than the value of 10.4 measured for the circular disks. This results in conservative predictions of strength at failure probabilities below 63%.

From our previous experience, even the use of conservative specimen data tended to overestimate the surface fast fracture strength, but this was not the case for the NT154 pressurized circular plate. To complete the analysis, it was decided to make predictions using the two other (less conservative) data sets. These were the E-bar and Type-B MOR specimens.

As can be seen in Table 55, the characteristic strength predicted from E-bar and Type-B specimen data was 2-3% higher than observed in the circular plates. But, as can be seen in Figure 151, the E-bar prediction, with a Weibull modulus of 8.1, came closer to predicting the test results than did the tensile bar data.

At the low failure probabilities where components are designed, even the MOR predictions were conservative, and thus, for the plates, the use of the most conservative data was not required. When initially designing a component, the worst material properties should be used until information is available to indicate the approach is overly conservative.

Table 55. Room temperature NT154 pressurized flat plate surface characteristic strength predicted from uniaxial Weibull test and the PIA methodology

Specimen type	Weibull modulus—ms	Disk effective area—mm ²	Predicted disk characteristic strength	Measured disk characteristic strength—MPa	Percent difference predicted versus measured
Tensile	6.219	705.4	805.6	808.6	-0.4%
E-bar	8.105	559.3	826	808.6	+2.1%
Type-B	8.070	561.4	830	808.6	+2.6%

Figure 151. NT154 biaxial fast fracture room temperature pressurized flat circular plate confirmatory test.

6.1.3 Room Temperature NT154 Spin Test Disks

An important portion of the ceramic life prediction methodology development process was the verification of representative engine components. The fast fracture lifing methodology validation process involved the testing of NT154 disks spun to burst at room temperature. Both volume and surface failures were observed.

While a total of 14 disks were tested, 5 were not considered in the analysis. Of the five disks, four had a different heat treat. The fifth disk failure originated on the shaft at a very low rpm, suggesting an outlier or possibly a different failure mechanism (imbalance, frequency problems, etc). Table 56 summarizes information for the nine disks analyzed. Even though the peak internal stress was almost 15 % higher than that of the surface, six disks failed at the surface, while only three failed internally.

Table 56. Room temperature fast fracture strength of NT154 spin disks

Disk ID	Failure speed—rpm	Location	Peak stress per FEM analysis —MPa	
			Internal	Surface
21	82,800	Surface	302.4	263.9
18	99,100	Surface	433.2	378.1
4	92,800	Surface	379.9	331.6
36	96,600	Surface	411.6	359.3
9	105,000	Surface	486.3	424.5
17	102,600	Surface	464.4	405.3
3	107,100	Internal	506.0	441.6
31	90,200	Internal	358.9	313.2
34	88,500	Internal	345.5	301.6

6.1.3.1 Internal Failure Fast Fracture Analysis of NT154 Disks. Two types of NT154 MOR room temperature bars demonstrated volume failures. These were the E-bars and 4-in. long bars cut from actual disks. Unlike the Type-B bar, the thickness exceeded the width resulting in relatively high effective volumes. Table 57 describes the important features associated with these NT154 MOR test specimens. Tables 58 and 59 summarize the E-bar and 4-in. bar fast fracture test results used to determine the Weibull parameters.

Fifteen E-bars failed internally, while the remainder consisted of surface and corner failures. Using the CARES MLE methodology with suspended items, the Weibull modulus and characteristic strength were determined for the internal failures (Table 60).

Of the 24 4-in. bars, 6 failed internally, while the remainder failed either on the surface or at a corner. The CARES MLE methodology was used to determine the fast fracture material parameters (Table 60).

In addition to characterizing the Weibull parameters of each data set, a consolidated data set was formed (Table 60). This was accomplished by normalizing the strength measured on the smaller 4-in. bars to what would be expected if the material were formed into the larger E-bar. At this point, CARES using the MLE methodology is applied to the combined data set. The following two equations were used to normalize the strength of the 4-in. specimens.

$$V_e = 0.5 \cdot w \cdot h \cdot (L_1 + m_v \cdot L_2) / (m_v + 1)^2 \quad (98)$$

Where:

V_e = Effective volume of 4 point bend MOR specimen
 w = Specimen width
 h = Specimen depth
 L_1 = Outer load span
 L_2 = Inner load span

$$S(\text{E-bar}) = S_4 \cdot (V_4 / V_e)^{1/m_v} \quad (99)$$

Where:

$S(\text{E-bar})$ = normalized strength of 4-in. bar
 S_4 = measured MOR strength of 4-in. bars
 V_4 = effective volume of 4-in. bars
 V_e = effective volume of E-bars

Table 57. Dimensions and load spans of two types of NT154 fast fracture MOR bars

ID	Outer load span		Inner load span		Width		Depth	
	in.	cm	in.	cm	in.	cm	in.	cm
4 in. bars cut from disks	3.5	8.89	1.75	4.445	0.40	1.02	0.525	1.334
E-bars	5.0	12.70	2.5	6.35	0.35	0.889	0.7205	1.830

Table 58. Summary of NT154 room temperature E-bar MOR fast fracture strength

Specimen ID	Strength— ksi/MPa	Failure location
LPEL-1	100.6/693.6	Surface
LPEL-4	97.3/670.9	Surface
LPEL-5	82.0/565.4	Surface
LPEL-7	103.0/710.2	Surface
LPEL-8	107.3/739.8	Surface
LPEL-9	109.3/753.6	Surface
LPEL-11	118.9/819.8	Corner
LPEL-12	101.4/699.2	Surface
LPEL-13	80.1/552.3	Internal
LPEL-14	91.9/633.6	Surface
LPEL-15	116.4/802.6	Surface
LPEL-16	116.1/800.5	Surface
LPEL-17	98.8/681.2	Surface
LPEL-18	105.2/725.4	Corner
LPEL-19	76.9/530.2	Corner
LPEL-20	82.8/570.9	Corner
LPEL-21	117.9/812.9	Corner
LPEL-22	87.2/601.2	Surface
LPEL-23	125.4/864.6	Surface
LPEL-24	99.3/684.7	Internal
LPEL-25	119.5/823.9	Internal
LPEL-26	117.4/809.5	Corner
LPEL-29	77.8/536.4	Corner
LPEL-35	81.0/558.5	Corner
LPEL-36	92.7/639.2	Surface
LPEL-37	107.5/741.2	Surface
LPEL2-2	96.2/663.3	Surface
LPEL2-3	115.5/796.4	Surface
LPEL2-5	109.3/753.6	Surface
LPEL2-6	89.3/615.7	Corner
LPEL2-7	112.0/772.2	Corner
LPEL2-8	91.5/630.9	Internal
LPEL2-9	100.9/695.7	Surface
LPEL2-10	118.0/813.6	Surface
LPEL2-11	127.9/881.9	Surface
LPEL2-12	71.3/491.6	Corner
LPEL2-13	63.4/437.1	Corner
LPEL2-15	131.1/903.9	Surface
LPEL2-16	97.9/675.0	Corner
LPEL2-19	131.4/906.0	Surface
LPEL2-20	109.8/757.1	Surface
LPEL2-21	100.9/695.7	Internal
LPEL2-22	113.0/779.1	Surface
LPEL2-23	122.2/842.6	Surface
LPEL2-24	57.9/399.2	Corner

Table 58 (cont)

Specimen ID	Strength— ksi/MPa	Failure location
LPEL2-25	128.9/888.8	Corner
LPEL2-26	104.7/721.9	Internal
LPEL2-27	102.5/706.7	Corner
LPEL2-28	134.5/927.4	Corner
LPEL2-29	86.6/597.1	Corner
LPEL2-30	50.6/348.9	Corner
LPEL2-31	105.1/724.7	Surface
LPEL2-36	64.2/442.7	Corner
LPEL2-37	110.5/761.9	Surface
LPEL2-38	113.9/785.3	Surface
LPEL2-39	101.2/697.8	Internal
LPEL2-40	122.2/842.6	Surface
LPEL2-41	80.5/555.04	Surface
LPEL2-42	103.3/712.3	Corner
LPEL2-43	142.1/979.8	Surface
LPEL2-46	99.8/688.1	Surface
LPEL2-48	117.7/811.5	Corner
LPEL2-49	112.5/775.7	Surface
LPEL2-50	66.0/455.1	Corner
LPEL2-51	92.9/640.5	Internal
LPEL2-52	115.0/792.9	Internal
LPEL2-54	133.1/917.7	Surface
LPEL2-55	125.6/866.0	Internal
LPEL2-57	75.1/517.8	Corner
LPEL2-58	126.5/872.2	Corner
LPEL2-59	117.7/811.5	Surface
LPEL2-63	107.2/739.1	Surface
LPEL2-64	95.3/657.1	Corner
LPEL2-65	113.4/781.9	Corner
LPEL3-1	115.4/795.7	Internal
LPEL3-2	58.2/401.3	Surface
LPEL3-3	116.7/804.6	Surface
LPEL3-4	95.4/657.8	Surface
LPEL3-5	110.2/759.8	Internal
LPEL3-8	80.4/554.4	Corner
LPEL3-9	103.3/712.3	Surface
LPEL3-10	118.7/818.4	Internal
LPEL3-11	127.4/878.4	Corner
LPEL3-12	132.1/910.8	Corner
LPEL3-14	106.9/737.1	Surface
LPEL3-15	86.2/594.3	Corner
LPEL3-16	98.3/677.8	Corner
LPEL3-17	117.3/808.8	Corner
LPEL3-18	115.3/795.0	Surface
LPEL3-20	128.0/882.6	Internal

Table 58 (cont)

Specimen ID	Strength— ksi/MPa	Failure location
LPEL3-21	70.1/483.3	Corner
LPEL3-23	143.8/991.5	N/A
LPEL3-24	109.6/755.7	Surface
LPEL3-25	117.0/806.7	Surface
LPEL3-31	72.6/500.6	Corner
LPEL3-33	126.1/869.5	Internal
LPEL3-34	85.5/589.5	Side inclusion
LPEL3-36	109.3/753.6	Surface
LPEL3-37	133.0/917.0	Surface
LPEL3-38	109.4/754.3	Surface

Table 59. Summary of NT154 room temperature 4-inch bar MOR fast fracture strength

Specimen ID	Strength (ksi/MPa)	Failure Location
G51-1	116.8/805.3	Surface
G51-2	107.5/741.2	Corner
G51-3	113.8/784.6	Surface
G51-4	83.7/577.1	Internal
G51-5	82.1/566.1	Corner
G51-6	114.8/791.5	Surface
G47-1	106.9/737.1	Internal
G47-2	121.1/835	Corner
G47-3	100.5/693	Internal
G47-4	92.9/640.5	Internal
G47-5	58.3/402	Corner
G47-6	114.5/789.5	Surface
G34-1	124.3/857.0	Surface
G34-2	86.0/593.0	Surface
G34-3	108.3/746.7	Internal
G34-4	114.6/790.2	Internal
G34-5	120.2/828.8	Corner
G34-6	109.8/757.1	Surface
G22-1	124.5/858.4	Surface
G22-2	121.7/839.1	Corner
G22-3	114.5/789.5	Surface
G22-4	111.9/771.6	Surface
G22-5	101.0/696.4	Corner
G22-6	65.5/451.6	Corner

Table 60. NT154 room temperature fast fracture material properties for internal failures of MOR specimens

ID	Weibull modulus— mv	Characteristic strength— MPa	Specific effective volume $\text{m}^3 \times 10^{-7}$	Unit characteristic strength $\text{MPa} \cdot \text{m}^3/\text{mv}$
E-bar	7.977	995.22	6.395	166.5
4 in. bar	7.295	925.38	4.0677	123.14
Combined E-bar and 4 in. bar	7.0649	986.54	7.199	133.24

Since the effective volumes depend upon the value of the consolidated Weibull modulus, mv, an iteration is required in addition to the CARES analysis. An estimation of mv must first be made. Using this value, the 4-inch specimen strength is normalized to that of the E-bar, and a CARES regression results in a new estimation of mv. The normalization is then redone and CARES is reapplied. This process converges quickly to the stable value of mv, which is shown in Table 60.

Once fast fracture material properties are determined, a NASTRAN finite element analysis of the spin disk is completed at a single rotor speed of 50,000 rpm.

Using the FEM output stress and volume information as CARES input, a failure probability based on Weibull PIA was determined. This information, in conjunction with the NASTRAN peak internal stress, allows the computation of the disk effective volume. The effective volume is independent of speed for an isothermal spin disk. Equation 100 provides the relationship used to determine the effective volume, and Table 61 summarizes the results.

$$V_{\text{eff}} = \ln(1/(1-P_f)) / (S_{\text{max}}/S_0)^{mv} \quad (100)$$

Where:

P_f = failure probability (From CARES)

S_{max} = peak volume stress

S_0 = unit characteristic strength

Table 61. NT154 room temperature spin disk internal stresses and computed FF failure probability at 50,000 rpm

Weibull exponent	Unit characteristic strength $\text{MPa} \cdot \text{m}^3/\text{mv}$	Peak stress— MPa	CARES volume failure probability	Spin disk effective volume $\text{m}^3 \times 10^{-4}$
7.295	123.14	110.279	0.58008 E-4	1.2971
7.977	166.5	110.279	0.14063 E-5	0.37588
7.0649	133.238	110.279	0.339727 E-4	1.2925

Once the effective volume is available, the probability of survival at any other disk speed may be found using Equations 101 and 102. Equation 101 determines the peak stress at speeds other than the baseline, since mechanical rotational stresses are proportional to the square of the rpm. Equation 102 is the classical Weibull statistical equation.

$$S(\text{rpm}) = S_{\text{ref}} (\text{rpm}/50,000)^2 \quad (101)$$

$$\text{POS} = \text{Exp}(-V_{\text{eff}} (S(\text{rpm})/S_o)^{mv}) \quad (102)$$

Where:

S_{ref} = peak volume stress at 50,000 rpm

$S(\text{rpm})$ = peak volume stress at any rotor speed

rpm = rotor speed to which stress is scaled

POS = Weibull cumulative survival probability

The survival probabilities for the three disks that experienced volume failures may be obtained from median ranks. In the median rank analysis the six disks that experienced surface failures are treated as suspended items. Table 62 compares the predicted failure probabilities obtained from E-bar, 4-in. bar, and consolidated data with the experimental results.

The results indicate the experimental results are bracketed by predictions based on the E-bar and 4-in. bar data. The predictions made from the 4-in. bars are conservative, while the E-bar predictions are optimistic. For the combined data set, the observed and predicted results agree well. Figure 152 contains a combined Weibull plot constructed with the 90% scatter band. For the combined data set, after the disk failure data are corrected for the effective volume, only disk number 3 lies outside the scatter band. This disk is approximately 10% stronger than predicted.

In summary, the application of a consolidated MOR data set, along with the CARES PIA methodology, predicted the observed NT154 spin disk internal fast fracture failures reasonably well.

6.1.3.2 Fast Fracture Analysis of the NT154 Spin Disks Surface Failures. Prior correlative efforts have shown that the accurate prediction of biaxial fast fracture at the component surface tends to be difficult and should be treated conservatively. Based upon this experience, if a specimen data base specific to the component surface is not available, the most pessimistic data applied in the most pessimistic manner should be used to estimate surface strength, but even this estimate can be unconservative.

Table 62. Comparison of NT154 room temperature spin disk internal fast fracture experimental and predicted failure probabilities

Disk ID	Stress—MPa	Experimental failure probability	Predicted failure probability		
			4-inch bar	E-bar	Combined
3	506.0	0.618	0.98	0.23	0.80
31	358.9	0.204	0.27	0.017	0.13
34	345.5	0.086	0.21	0.013	0.10

Figure 152. NT154 biaxial fast fracture room temperature spin disk internal failure confirmatory test.

For a shear insensitive material, the Weibull PIA methodology is the most pessimistic analytical approach. A CARES analysis of disk failure probability using NT154 Type-B, Mil-E, and tensile bar Weibull data showed the tensile bar results, without suspended items, was most conservative. Table 53 summarizes the tensile bar test data.

The fast fracture button-head tensile bar surface failure material data were used to analyze the NT154 room temperature spin disk verification tests.

The Weibull MLE analysis of the button-head specimens yielded surface constants as follows:

$$m_s = 6.22$$

$$S_o = 250.83 \text{ MPa-m}^2/m_s$$

From finite element analysis and the CARES PIA model, the peak surface strength was found to be 96.254 MPa at 50,000 rpm, and the surface failure probability was found to be 0.56137 E-4 . Using an equation similar to Equation 100 an effective area is 0.02170 m^2 .

A comparison of disk failure probability obtained from median ranks and that computed from the tensile bar specimen data shows the strength prediction is still nonconservative (Table 63). The mean strength has been overestimated approximately 15%.

To summarize, predictions of surface multiaxial strength are often optimistic, even when conservative uniaxial fast fracture information is used. Such was the case with NT154 spin disks where tensile bar strength data still overestimated the disk strength by about 15%.

Table 63. Comparison of NT154 room temperature spin disk surface fast fracture experimental and predicted failure probabilities

Disk ID	Stress—MPa	Experimental failure probability	Predicted failure probability from tensile bar data
9	424.5	0.758	0.444
17	405.3	0.622	0.349
18	378.1	0.485	0.243
36	359.3	0.348	0.184
4	331.6	0.211	0.116
21	263.9	0.074	0.029

6.2 SLOW CRACK GROWTH BIAXIAL VERIFICATION

6.2.1 Ball-on-Ring Testing of PY-6 Material at 1200°C

Biaxial static fatigue tests on flat circular plates using the ball-on-ring arrangement were conducted by Battelle Laboratories at 1200°C. The specimens had a 31.75 mm nominal diameter and a 1.78 mm thickness. Support was at 15.9 mm. Table 64 summarizes the loads, geometries, and peak stress. Equation 83 with a Poisson's ratio of 0.28 was applied to calculate the maximum stress developed at the plate center.

Table 64. PY-6 1200°C SCG static surface strengths of flat circular plates loaded by a ball-on-ring fixture

ID	Thickness— in./mm	Fracture load— lb/Newtons	Fracture stress— MPa	Failure time— hr
34.2266	0.071/1.803	191/850	503	0.025
34.2267	0.070/1.778	234/1041	637	0.033
34.2269	0.070/1.778	228.5/1016	622	0.032
34.2610	0.070/1.778	195/867	533	0.025
34.2562	0.084/2.1336	200/890	356	200*
34.2577	0.100/2.54	252/1121	348	200*
34.2270	0.070/1.778	180/801	490	168*
34.2240	0.070/1.778	180/801	490	246*

* Specimen unloaded without failure.

At elevated temperatures, available fast fracture data tends to indicate a lower sensitivity to the surface preparation; certainly after oxide begins to form, this would be expected to be true. As a result both fast fracture and SCG information obtained from button-head tensile specimens in static loading were used to predict the biaxial results.

The effective area of the ball-on-ring circular disk specimens was obtained by using equations 83 through 86 which defined the radial and tangential stress fields. The tensile specimen surface Weibull modulus of 5.4 was used in the computations along with a failure model, based upon room temperature results, which consider only the stresses acting normal to the machining marks. The fast fracture strength of the circular plates was then calculated by using Equation 103 along with the tensile bar Weibull properties at 1200°C. The circular plate fast fracture strength was predicted to be 981 MPa at 1200°C.

$$S_i = S_o * \Gamma(1 + 1/ms) / A_{eff}^{1/ms} \quad (103)$$

Where:

ms = Tensile bar surface Weibull modulus = 5.412

Γ = Gamma function = 0.922

S_o = tensile bar unit characteristic strength = 94.84 MPa-m²/ms

A_{eff} = effective area of circular plate = 2.077 E-6 m²

SCG under static loading follows Equation 32 when an activation energy is used to consolidate several temperatures.

To obtain SCG material constants, 1200°C, 1260°C, and 1300°C static fatigue data obtained from button-head tensile specimens were regressed. Table 65 summarizes the 1200°C data, while the remainder of the Southern Research test data used in this regression are shown in Table 66. In Figure 153 a regression of these data, which minimize the scatter on strength, results in the following values for the material properties Q/R, B, and N.

$$Q/R = 475,686 \text{ K}$$

$$\ln(B) = -349.39 \text{ MPa}^2\text{-hr}$$

$$N = 68.3$$

The values of the tensile bar initial strengths used in the regression are tabulated below.

<u>Temperature—°C</u>	<u>Strength—MPa</u>
1200	324
1260	362
1300	387

Figure 153 shows the fit of the model to the tensile bar results. The predicted fast fracture strength of the circular plate, 981 MPa, is inserted into Equation 32 along with the values of Q/R, B, and N from the tensile bar data. This yields a prediction of strength versus time for the biaxial circular plate specimens (Figure 154). The model results in a reasonable correlation, with the line going through the mean of the short time data and lying above the long-time non-failure test data. Thus, at 1200°C a prediction of the time dependent strength for a biaxial plate in bending, using SCG and fast fracture information from tensile bar testing produced a reasonable correlation.

Table 65. PY-6 1200°C SCG static fatigue test results of button-head tensile specimens with surface failures

ID	Stress—MPa	Failure time— hr
453A	250.49	0.48
463B	225.46	0.55
460A	199.25	2.05
448B	190.03	0.159
514A	184.99	0.009
636A	174.69	389 *
525A	174.33	18.96
517B	159.84	360 *
635B	149.75	232 *
341A	124.78	240 *
90A	289.59	266 *
454A	220.75	0.008
552A	201.59	0.003
629A	180.75	291 *
452A	177.74	0.733
454B	177.12	0.008

* Test stopped without failure

Figure 153. PY-6 button head tensile specimen slow crack growth model prediction versus stress rupture tensile specimen data from 1200°C to 1300°C.

Table 66. PY-6 1260 and 1300°C SCG static fatigue test results of button-head tensile specimens with surface failures.

ID	Temperature— °C	Stress— MPa	Failure time— hr
553B	1260	2261.9	0.135
567B	1260	200.52	306
456A	1260	185.19	0.205
528A	1260	180.04	0.475
486A	1260	177.71	26.24
532A	1260	176.21	2.73
463A	1260	175.26	364
515A	1260	175.20	1154
466B	1260	165.93	5.12
458A	1260	160.81	0.122
479A	1260	155.06	703
488B	1260	149.94	0.820
471A	1260	141.22	27.37
533B	1260	125.05	721
533A	1260	141.07	4.42
201A	1300	275.22	0.013
334B	1300	249.31	0.240
77A	1300	199.54	0.690
341B	1300	184.60	0.960
514B	1300	178.08	0.826
457B	1300	176.23	0.364
474A	1300	175.06	0.535
519A	1300	160.53	0.182
524B	1300	159.7	1.87
487A	1300	155.22	0.55
491B	1300	174.88	0.003
626A	1300	173.15	0.003
488A	1300	155.89	0.003
453B	1300	152.27	0.003
468B	1300	140.04	2.03

Figure 154. Comparison of predicted and observed slow crack growth behavior for PY-6 ball-on-ring flat circular disks tested at 1200°C and 1300°C.

6.2.2 PY-6 Ball-on-Ring Testing at 1300°C

At 1300°C, Battelle Laboratories also tested the biaxial fatigue of flat circular plates using the identical ball-on-ring test arrangement described at 1200°C. Table 67 describes the circular plate geometry and the peak elastic bending stresses computed using Equation 83 and the Poisson's ratio of 0.28.

Similar to the 1200°C prediction, both fast fracture and SCG information obtained from button-head tensile specimens in static loading were used to predict the biaxial results.

The effective area of the ball-on-ring circular disk specimens was estimated by the same methodology as at 1200°C. Since the geometries were nearly identical, a value of 2.077 E-6 m^2 was again used.

At 1200°C, the surface fast fracture information was well defined with 53 surface failures resulting in a Weibull modulus of 5.412. At 1300°C, the Weibull modulus of 6.44 was defined by the surface failure of only four specimens. Since specimens with similar flaw distributions should have a Weibull modulus independent of temperature, 5.412 was considered to be a better estimator at 1300°C. The characteristic strength was obtained by rationing by the mean strengths, resulting in a value of:

$$S_o = 113.36 \text{ MPa-m}^{2/5.412}$$

The fast fracture strength of the circular plates was then calculated by using Equation 103 along with the tensile bar Weibull properties at 1300°C. The circular plate fast fracture strength was predicted to be 1173 MPa at 1300°C. It may be observed that the plate strength is computed to be higher at 1300°C than at 1200°C. This follows from the tensile bar data which also showed higher strengths at 1300°C.

Table 67. PY-6 1300°C SCG static surface strengths of flat circular plates loaded by a ball-on-ring fixture

ID	Thickness— in./mm	Fracture load— lb/Newtons	Fracture stress— MPa	Failure time— hr
34.2242	0.0908/2.306	297/1321	441	0.042
34.2244	0.0800/2.032	200/890	399	0.11
34.2268	0.0703/1.786	278/1237	749	b
34.2600	0.0704/1.788	218/970	586	a
34.2601	0.0704/1.788	205/912	551	0.0042
34.2609	0.0700/1.778	201/894	547	a
34.2611	0.0700/1.778	209/930	569	0.025
34.2241	0.0915/2.324	229/1019	334	200.0 b
34.2243	0.0908/2.306	220/979	327	200.0 b
34.2247	0.0800/2.032	182/810	363	360.0 b
34.2563	0.0800/2.032	204/907	407	304.0 b
34.2564	0.0800/2.032	222/988	443	456.0 b
34.2567	0.0800/2.032	235/1045	469	231.0 b
34.2583	0.0704/1.788	200/890	537	220.0 b
34.2619	0.0790/2.007	188/836	386	75.0 b

a Broke during loading

b Unloaded before fracture

The predicted initial strength of the circular plate, 1173 MPa, is used in Equation 32 along with the values of material constants previously determined from the tensile bars to allow a prediction of strength versus time for the biaxial circular plate. This is shown graphically in Figure 154 and compared with the circular plate test results. As at 1200°C, the prediction appears reasonable. The line goes through the mean of the short time data and lies above the most of the longer time specimens, which were not tested to failure. The flatness of the line indicates a very large life scatter at the same stress levels.

It should be noted that the use of an elastically calculated stress is inherently conservative at high lives where, at 1300°C, any creep relaxation will reduce the surface bending stresses. This may partially explain the large number of run-outs.

6.2.3 Biaxial SCG Verification—NT154 Spin Disk Testing at 1204°C

A number of the NT154 spin disks were subjected to a 1204°C environment and various rotational speeds, resulting in both fast fracture and time dependent failure modes. The dominant time dependent failure mechanism at this temperature is slow crack growth. Since only one SCG failure was internal, all analysis was based upon the surface. Analysis consisted of four important steps:

- obtain fast fracture material constants
- obtain slow crack growth material constants
- use MSC-NASTRAN to define stress and geometry input to CARES
- use CARES to determine the predicted disk failure probability

Consistent with the room temperature approach, the tensile specimen data were first used to estimate the disk fast fracture strength. This did not work well. Of 20 test specimens, only 2 failed on the surface (Table 68). As a result of the low number of surface failures, the Weibull MLE analysis was limited to two approaches. In the least conservative of these, the 18 volume failures were considered to be suspended items. The most conservative approach assumed surface failures would occur at the same stress as the observed 18 internal failures.

The suspended MLE methodology used with the tensile specimen data resulted in a predicted disk mean fast fracture strength of 442 MPa. The spin disks tested to fast fracture failure were found to have a mean strength of 276 MPa. Thus the least conservative approach overpredicted strength by about 60%.

Based upon room temperature experience, the more conservative approach was expected to result in the better estimate of the disk surface Weibull properties. In this case, using internal failures as surface failures, the predicted disk strength of 325 MPa was still 18% higher than the observed strength.

The primary objective of the 1200°C spin disk testing was the verification of the SCG methodology. Consequently, the best available estimation of disk fast fracture properties was used to avoid introducing other difficulties. This was done by using fast fracture material constants obtained from the disk testing in the SCG analysis. Table 69 summarizes the fast fracture disk specimens used to obtain the material constants.

Table 68. NT154 button-head tensile fast fracture specimen strength at 1204°C

Specimen ID	Fracture strength— ksi/MPa	Failure type
LPTEN139	78.3/539.9	Volume outside gage
LPTEN145	60.3/415.8	Volume
LPTEN229	66.3/457.1	Volume
LPTEN259	61.4/423.4	Volume
LPTEN296	65.3/450.2	Volume
LPTEN2141	71.2/490.9	Volume outside gage
LPTEN2143	79.3/546.8	Volume
LPTEN2158	72.0/496.4	Surface
LPTEN336	52.8/364.1	Volume
LPTEN346	74.3/512.3	Volume
LPTEN349	73.4/506.1	Volume
LPTEN356	80.1/552.3	Volume outside gage
LPTEN372	76.2/525.4	Volume outside gage
LPTEN378	86.9/599.2	Volume
LPTEN381	96.4/664.7	Volume
LPTEN385	71.8/495.1	Surface
LPTEN392	67.5/465.4	Volume
LPTEN3110	85.0/586.1	Volume
LPTEN3131	69.5/479.2	Volume
LPTEN3134	61.8/426.1	Volume

Table 69. NT154 1204°C spin disk fast fracture test results

Disk-ID	Failure speed—rpm	Peak surface stress— ksi/MPa	Failure location
32	76,000	32.94/227.1	Surface
33	76,000	32.94/227.1	Surface
13	83,000	39.033/269.1	Surface
23	83,000	39.033/269.1	Surface
37	74,000	31.298/215.8	Volume
29	80,400	36.707/253.1	Volume
6	93,000	48.667/335.6	Volume

Three disks which failed at the shaft/fillet were not included in this table because of the possibility of unknown bending stresses caused by resonance. One disk which failed at the surface at 60,800 rpm was considered to be an outlier.

Analysis of the disk data using the MLE method with the volume failures taken as suspended items resulted in the following material constants:

$$S_o - 76,590 \text{ psi-in}^2/\text{m} = 155.23 \text{ MPa-m}^2/\text{ms} \\ \text{ms} - 6.0$$

Surface stresses are caused by the superposition of rpm induced stresses and small thermally induced stresses. As a result the peak stress may be computed by an equation of the form:

$$S_p = A \cdot \text{rpm}^2 + B \quad (104)$$

where

A and B are constants specific to the disk with a fixed thermal gradient

These constants are obtained from disk finite element analysis at two different rotational speeds. Table 70 summarizes the stresses used to obtain the values of A and B as well as a back prediction of stresses from the equation. In Table 69, stresses at other speeds were obtained from the Equation 104 using the values of:

$$A = 5.474 \text{ E-9 ksi-minute}^2 = 3.774 \text{ E-8 MPa-minute}^2 \\ B = 1.3223 \text{ ksi} = 9.117 \text{ MPa}$$

While the disks exhibited surface failures, the tensile bars from which SCG constants were to be obtained failed primarily as a result of internal flaws. This was judged to be a relatively minor problem. Once the volume related SCG constants were obtained, the surface constants could be estimated. Since the crack velocity exponent, N, is a basic material property, it is expected to be substantially the same for both surface and volume. Since the coefficient, B, is related to crack amplifying factors, the surface value may be estimated using fracture mechanics in the following manner:

$$B = 2 / ((N-2) \cdot K_{Ic}^{(N-2)} \cdot Y^2 \cdot A) \quad (105)$$

Table 70. Comparison of NT154 1204°C spin disk surface stresses obtained from NASTRAN and back predicted from Equation 99

NASTRAN surface stresses—ksi/MPa	Revolutions per minute	Surface stress values back-predicted—ksi/MPa
31.298/215.8	74,000	31.298/215.8
36.709/253.1	80,400	36.707/253.1
48.667/335.6	93,000	48.667/335.6

A, Kic, and N are material properties that are nearly the same for both surface and volume. Only Y differs.

$$Y_{surf}/Y_{vol} = 0.71 P_i^{0.5}/0.6366 P_i^{0.5} \text{ (fracture mechanics)} \quad (106)$$

$$Y_{surf}/Y_{vol} = 1.1153$$

$$B_{surf} = B_{vol}/1.244 \quad (107)$$

The equation for static fatigue requires the initial (fast fracture) strength of the specimen to be known prior to the regression of static data to determine the constants N, B, and Q/R.

Table 68 summarizes the tensile fast fracture data used to determine the strength as well as the specimen values of m. Using an MLE analysis, with both surface failures as well as out-of-gage failures treated as suspended items, the following material properties were found:

$$S_{th} = 555.74 \text{ MPa}$$

$$mv = 6.957$$

$$S_i = S_{th} \cdot \Gamma(1 + 1/mv) = 520.2 \text{ MPa}$$

Static fatigue data, identified in Table 71, was regressed to obtain the SCG material constants. Since the basic SCG theory assumes the fast fracture Weibull Modulus applies to the static fatigue data, the value of N was chosen so the static fatigue m was identical to the fast fracture value. The value of B at a specific temperature is given by Equation 45. B obtained from volume data is corrected to a surface value by dividing by 1.244. After the regression, the following values were obtained.

$$T = 1477 \text{ K}/(1204^\circ\text{C})$$

$$N = 14.1$$

$$B = 1.729 \text{ ksi}^2\text{-hr.}/82.20 \text{ MPa}^2\text{-hr}$$

$$Q/R = 10430.34 \text{ K}$$

$$B_t = 1.729 \cdot \text{Exp}(10430.34/1477) = 2017 \text{ ksi}^2\text{-hr}/95,890 \text{ MPa}^2\text{-hr}$$

$$B(\text{surface}) = 2017/1.244 = 1621 \text{ ksi}^2\text{-hr}/77,060 \text{ MPa}^2\text{-hr}$$

The final material constants used in the SCG spin-disk analysis follow:

$$S_o = 76.590 \text{ ksi-in.}^2/\text{ms}/155.23 \text{ MPa-m}^2/\text{ms}$$

$$ms = 6.00$$

Table 71. NT154 button-head tensile SCG stress rupture internal failure results at 1149 and 1204°C

Specimen ID	Temperature— °C	Failure stress— MPa	Failure time— hr
2-56	1204	415	1.3
3-73	1204	400	1.1
3-121	1204	385	501*
3-75	1204	375	252
3-48	1204	375	525
3-118	1204	375	120
3-116	1204	375	141
2-149	1204	375	66
3-54	1204	375	43
3-66	1204	375	24
3-34	1204	375	19
2-146	1204	375	12
2-164	1204	375	0.84
2-72	1204	375	0.50
1-38	1204	375	528*
3-83	1204	375	266
3-76	1204	375	253
3-71	1204	375	153
3-93	1204	375	80
2-93	1204	375	40
3-124	1204	375	7
3-95	1204	375	6
1-43	1204	375	0.40
2-28	1204	350	612*
2-71	1204	350	2.3
3-39	1204	345	636
2-30	1204	340	566
3-37	1204	340	177
2-36	1204	337	593*
3-19	1204	335	469
2-74	1204	325	68
2-31	1204	315	594*
1-3	1204	300	517*
3-41	1149	450	7.0
2-47	1149	450	0.08
2-75	1149	445	0.02
2-32	1149	440	376
3-31	1149	435	594*
3-109	1149	425	509*
2-62	1149	425	32

* Test Stopped

$$N = 14.1$$

$$B(\text{surface}) = 1621 \text{ ksi}^2\text{-hr}/77,060 \text{ MPa}^2\text{-hr}$$

The Allison version of the CARES program computes the time dependent probability of failure of a statically loaded component by applying equations of the following form to each finite element and finding the product of the survival probabilities.

$$S_{i, o} = (S^{N-2} + S^N * t/B)^{1/(N-2)} \quad (108a)$$

$$POS_i = \text{Exp}-A_i (S_{i,o}/S_o)^m \quad (108b)$$

Where:

$S_{i, o}$ = effective stress at time = 0

S = element stress

A_i = element area

t = time

POS_i = probability of survival of i th element

Both fast fracture and slow crack growth disk component probabilities of survival obtained from CARES are given in Table 72.

From the information in Table 72, values of characteristic strength, S_{th} , and SCG coefficient, B_c , may be obtained based upon the component peak stress and the probability of survival. This is accomplished by rearranging both the fast fracture and SCG failure probability equation and deriving Equations 110 and 112, which use the known values of S_{max} , t , POS , m , and N to find S_{th} and B_c .

$$POS = \text{Exp}(- (S_{max}/S_{th})^m) \quad (\text{fast fracture}) \quad (109)$$

$$S_{th} = S_{max} / (\text{Ln}(1/POS))^{1/m} \quad (\text{from 109}) \quad (110)$$

Table 72. Predicted surface SCG probability of survival of NT154 1204°C spin disks using CARES PIA methodology

Revolutions per minute	Failure time—hr	Surface POS	Peak stress— ksi/MPa
67,000	0.0	0.958909	25.895/178.5
	150.0	0.724461	
71,000	0.0	0.919244	28.917/199.4
	27.33	0.725877	
	100.00	0.551931	
75,000	0.0	0.849891	31.973/220.5
	10.0	0.646863	
	27.0	0.505957	
	50.0	0.401393	

$$S_{c, o} = (S_{\max}^{N-2} + S_{\max}^N * t/B_c)^{1/(N-2)} \quad (108a)$$

$$POSc = \text{Exp}-(S_{c,o}/S_{th})^m \quad (111)$$

$$B_c = t * S_{\max}^2 / (((\ln(1/POSc))^{(N-2)/m} / (S_{\max}/S_{th})^{(N-2)}) - 1) \quad (112)$$

Equation 112 is obtained from Equations 108a and 111. Table 73 summarizes the computed values of characteristic strength and B_c . It should be noted that S_{th} is slightly dependent on rpm, since the small amount of thermal stress does not scale with rpm. B_c is slightly dependent on time only because the first term in the SCG equation, $S_{\max}^{N-2}$, has not been ignored.

Table 74 defines the rpm levels and the hold time for the four disks that failed on the surface as a result of SCG. Since only disk 22 is held at a constant rpm until final failure, the previous equation for $S_{i, o}$ must be modified for the more complex missions. If the time spent moving from one rpm to another is neglected and if the loads are constantly increasing, an equation of the following more general form may be derived:

$$S_{i, o} = (S_f^{N-2} + S_{\max 1}^N * t_1/B_{c1} + \dots + S_{\max n}^N * t_n/B_{cn})^{1/(N-2)} \quad (113)$$

S_f = final mission stress

$t_1 \dots t_n$ = time intervals

$S_{\max 1} \dots S_{\max n}$ = peak stress at each rpm

$B_{c1} \dots B_{cn}$ = slow crack growth coefficient at each rpm

Table 75 summarizes the stresses, slow crack growth constants, values of $S_{i, o}$, and the computed probability of survival of the four disks. Since a mean disk life corresponds to a probability of survival of 50%, the prediction was conservative on three of four spin disks. Only disk 22 failed sooner than expected. Table 74 compares predicted and observed lives. For disk 22, the mean life was predicted to be 116 hr at 71,000 rpm while the observed life was 27 hr.

Disk 7 was spun for 100 hr at 71,000 rpm, 10 hr at 75,000 rpm, and finally burst at 82,000 rpm. The predicted life of 100 hr at 71,000 rpm followed by 2.7 hr at 75,000 rpm was slightly conservative. It should be observed that disk 7 was spun for 110 hr at speeds greater than or equal to 71,000 rpm, while disk 22 failed in 27 hr at the same 71,000 rpm. This illustrates a life scatter in the NT154 spin disks greater than four.

Table 73. Predicted surface values of NT154 1204°C spin disk fast fracture characteristic strength and SCG coefficient from CARES PIA analysis

Revolutions per minute	Failure time—hr	B_c —ksi ² -hr/MPa ² -hr	S_{th} —ksi(MPa)
67,000	150.0	1675.09/79,635	43.928 (302.9)
71,000	27.33	1655.86/78,721	43.678 (301.2)
	100.00	1656.95/78,773	
75,000	10.0	1624.95/77,252	43.275 (298.4)
	27.0	1626.70/77,335	
	50.0	1627.25/77,361	

Table 74. Applied load spectrum for four NT154 spin disks which failed on the surface in SCG at 1204°C

Disk ID	rpm $\times 10^3$	Load time hours	Predicted mean load time—hr
41	67/75	150/27	150/16
22	71	27.33	116
7	71/75/82	100/10/burst	100/2.7
19	75/84.5	50/burst	23.5

Table 75. Predicted failure probability for four NT154 spin disks which failed on the surface in SCG at 1204°C

Specimen ID	K-rpm	Stress— ksi (MPa)	Bc-ksi ² -hr (MPa ² -hr)	Sth— ksi (MPa)	Si,o— ksi (MPa)	Predicted POS*
41	75	31.973 (220.5)	1626.7 (77,335)	43.275 (298.4)	41.378 (285.3)	0.4657
	67	25.895 (178.5)	1675.1 (79,636)			
22	71	28.917 (199.4)	1655.9 (78,723)	43.678 (301.2)	36.130 (249.1)	0.7259
7	82	38.12 (262.9)	-----	43.275 (298.4)	42.313 (291.7)	0.4174
	75	31.973 (220.5)	1624.9 (77,249)			
	71	28.917 (199.4)	1656.9 (78,771)			
19	84.5	40.408 (278.6)	-----	43.275 (298.4)	44.063 (303.8)	0.328
	75	31.973 (220.5)	1627.3 (77,364)			

* N=14.1, m=6.0

In Figure 155 the CARES predictions are compared to the actual disk fast fracture failures (the small dependence of the effective area on rotational speed was ignored). Using median ranks computations, along with the effective time zero stress, the SCG spin disk failure data fast fracture were plotted. As can be seen, the SCG test results lie within the 90% confidence band and do not differ greatly from the mean of the fast fracture predictions. This tends to show that the proposed SCG methodology gives reasonable results.

6.2.4 NT154 Spin Disk Testing at 1371°C (2500°F)

The high temperature life prediction methodology was applied to five NT154 disks rotated to high speeds at 1371°C (2500°F). The speed was maintained until failure occurred. Table 76 summarizes the test results.

This testing was done by the Garrett Auxillary Power Division under their ceramic life program and furnished to Allison for an independent analysis.

While creep damage mechanisms are important at this temperature, an SCG analysis resulted in predictions close to the observed test results. A creep analysis was not particularly accurate. All disk failures occurred at the surface, thus the analysis was limited to SCG at the surface.

Since fast fracture failure data obtained from identical disks tested at 1204°C showed a surface strength about 25% lower than predicted from tensile specimens; it was decided to use this disk information and scale to the 1371°C temperature. For NT154 which is not shear sensitive, it has been shown that the same population of specimens tested at different temperatures have the following characteristics:

- The Weibull modulus is unchanged.
- The strength (or characteristic strength) is proportional to K_{Ic} .

Figure 155. NT154 biaxial SCG spin disk surface failure confirmatory test at 1204°C.

Table 76. NT154 spin disk 1371°C surface static failure data used to verify SCG methodology

Disk ID	Failure speed— rpm	Failure time— hr	Peak surface stress—MPa*
8	41,800	6.966	79.35
26	50,000	11.08	107.66
24	50,000	0.0166	107.66
14	55,000	0.95	127.40
15	45,000/50,000	10.0/0.40	89.81/107.66

* Obtained from an MSC/NASTRAN finite element analysis.

Stresses consist of a small amount of thermally induced stress as well as much larger rpm induced stresses.

At 1204°C, an MLE Weibull analysis indicated the following surface fast fracture properties:

$$S_o = 155.23 \text{ MPa-m}^{2/m}$$

$$m = 6.0$$

The surface Weibull modulus, 6.0, was retained at 1371°C. The unit characteristic strength was reduced by the ratio of K_{Ic} at 1371°C to that at 1204°C. Table 77 shows the test data used to determine the average K_{Ic} values. The revised value of S_o was estimated to be:

$$S_o \text{ (at 1371°C)} = 155.23 \times 5.789 / 6.139 = 146.38 \text{ MPa-m}^{2/m}$$

Table 77. NT154 K_{Ic} values obtained from notched MOR bar tests at 1204°C and 1371°C

1204°C		1371°C	
Specimen-ID	$K_{Ic} - \text{ksi-in.}^{0.5}$	Specimen ID	$K_{Ic} - \text{ksi-in.}^{0.5}$
LPCHV35	6.323	LPCHV33	5.892
LPCHV37	5.928	LPCHV34	5.928
LPCHV38	6.215	LPCHV59	6.036
LPCHV51	6.215	LPCHV60	6.071
LPCHV52	6.107	LPCHV63	6.395
LPCHV53	5.748	LPCHV64	5.209
LPCHV55	6.251	LPCHV66	4.994
<u>LPCHV56</u>	<u>6.323</u>		
Average	6.139	Average	5.789

While the disks exhibited surface failure, the tensile bars, from which SCG constants were to be obtained, failed primarily as a result of internal flaws. Correction of SCG volume constants to the surface was done in the same manner at 1371°C as was done at 1204°C. N , the crack velocity exponent, was assumed to be the same for both surface and volume. The surface constant B is obtained from the volume B by dividing by 1.244. This approach is based on fracture mechanics considerations.

The governing equation for static fatigue requires that the initial (fast fracture) strength of the specimen to be known prior to the regression of static data which determine the constants N , B , and Q/R .

While the fast fracture strength of the tensile bar was available at 1204°C (2200°F) it was not available at higher temperatures. Since Type-B strength data were available at both 1204°C and 1371°C, the tensile bar strength was estimated at 1371°C by ratioing by the Type-B MOR strengths. A linear interpolation/extrapolation was used to determine tensile bar strengths at 1295°C, 1315°C, and 1400°C. Table 78 summarizes the final estimated strengths of the tensile bars.

Static fatigue data, obtained by holding tensile bars at a constant load until failure, were available at 1295, 1315, 1371, and 1400°C. These data were regressed using the slow crack growth equation (Equation 32).

The activation energy term, $\text{Exp}(Q/RT)$, is used to consolidate the effect of temperature. Implicit in this formulation is the fact that the crack velocity exponent, N , is independent of temperature.

Table 79 summarizes the data used in the regression to determine the material constants. Figure 156 demonstrates that the static fatigue data fit the SCG equation quite well at 1315 and 1371°C. At 1400°C, specimens loaded at less than 120 MPa exhibited a steeper slope, indicating possible long time creep behavior. On the plot, two of the points shown represent 16 points at the same load (a logarithmic life average is used only for plotting purposes, the individual points are used in the data regression). As can be seen, the 1371°C fit goes through both consolidated data points. The following values of SCG constants were found:

$$B_v = 2.0974 \text{ E } -81 \text{ MPa}^2\text{-hr (volume)}$$

$$B_s = 1.686 \text{ E } -81 \text{ MPa}^2\text{-hr (surface)}$$

$$N = 17$$

$$Q/R = 300,506 \text{ K}$$

Table 78. Fast fracture volume strength of NT154 button-head tensile specimens at elevated temperature

Temperature— °C	Button-head tensile bar strength—MPa	Type-B MOR bar strength—MPa
1204	520	670.2
1295	503 (est)	-----
1315	499 (est)	-----
1371	488 (est)	629.5
1400	483 (est)	-----

(est)—Estimated from 1204°C data and MOR results.

Table 79. Stress rupture failure data for NT154 button-head tensile bars used to determine SCG constants at 1371°C

Specimen ID	Test temperature— °C	Applied stress— MPa	Failure time— hr
3-29	1400	150	80
1-21	1400	140	96
1-10	1400	130	192
2-125	1400	130	139
1-4	1400	125	341
2-114	1400	110	411
2-68	1400	100	462
2-123	1400	90	476*
2-35	1400	85	597*
2-55	1371	210	1.4
1-9	1371	190	68
1-6	1371	180	62
3-32	1371	180	64
2-118	1371	180	28
2-140	1371	180	25
2-60	1371	180	19
1-8	1371	180	14
2-91	1371	180	8.6
1-7	1371	180	6.8
2-116	1371	180	6.8
2-34	1371	180	5.6
3-69	1371	180	1.8
1-19	1371	180	1.6
1-16	1371	180	0.84
2-72	1371	180	0.24
2-69	1371	180	0.24
2-109	1371	180	0.14
1-209	1371	160	209
2-126	1371	155	24
2-113	1371	150	208
2-134	1371	150	171
3-33	1371	150	75
3-50	1371	145	443
2-120	1371	145	385
2-7	1371	145	373
2-12	1371	145	368
2-92	1371	145	368
2-63	1371	145	293
1-13	1371	145	266
3-35	1371	145	236
2-57	1371	145	364
2-136	1371	145	122
2-162	1371	145	117
2-101	1371	145	112

Table 79 (cont)

Specimen ID	Test temperature— °C	Applied stress— MPa	Failure time— hr
1-17	1371	145	111
2-50	1371	145	90
2-46	1371	145	73
2-45	1371	145	57
2-124	1371	140	586
2-117	1371	135	587
2-130	1371	135	280
2-119	1371	130	588
2-121	1371	125	728
1-2	1315	250	24
1-24	1315	240	2.9
2-64	1315	235	52
2-111	1315	232.5	297
2-37	1315	230	244
2-112	1315	230	168
2-128	1315	227.5	20
1-15	1315	225	675*
1-11	1295	375	0.35
1-23	1295	300	0.40

* Test stopped.

Figure 156. NT154 button-head tensile specimen slow crack growth model prediction versus measured stress rupture tensile data from 1315 to 1400°C.

In applying the CARES program to the 1371°C disk, the surface value, B_s , at 1371°C is equal to $B_s \cdot \exp(Q/(RT))$, where $T = 1644$ K and the activation energy term Q/R which was used to consolidate the testing at different temperatures is removed. Thus:

$$B_s \text{ (at 1371°C)} = 0.040864 \text{ MPa}^2 \cdot \text{hr}$$

Table 76 summarizes the loadings and failure time of disks spun in a 1371°C environment. These results indicate a large scatter in observed lives even at the same loading. At 50,000 rpm, the stronger disk lasted nearly 700 times as long as the weaker one. These types of results are not unusual for the time dependent behavior of high strength ceramics.

In determining the failure probability of the spin disks at the demonstrated failure time, a NASTRAN finite element analysis is used to define both the stress fields and the areas and volumes associated with each finite element. The CARES program is then used.

The Allison CARES program computes the time dependent probability of failure of a statically loaded component by applying Equations 108a and 108b to each finite element and finding the product of the survival probabilities. Within CARES, since the material is shear insensitive, the PIA approach, which treats each principal stress separately, was used. This approach often tends to yield the most conservative results in a multiaxial loading situation.

The SCG and fast fracture failure probabilities predicted from the material data and the CARES program are shown in Table 80. The prediction is generally optimistic. Table 81 puts these results in perspective. In this table, predicted mean lives as well as predicted lives corresponding to an expected failure rate of 5% are shown. The observed results lie approximately within this band, indicating the predicted lives were optimistic. The observed lives of disks 26 and 14 were quite close to the mean life prediction, while the lives of disks 8 and 24 were near the 5% failure life. Disk 15 life was well within the predicted limits. Figure 157 illustrates the results.

From the fast fracture survival probability in Table 80 and the peak surface stress from Table 79, the disk characteristic strength, S_{th} , may be computed from Equation 110.

An equivalent time zero stress which accounts for the effect of SCG may be found from the following equation:

$$S_e = S_{th} \cdot (\ln(1/POS))^{1/m} \quad (114)$$

Where:

S_e = equivalent time zero stress

POS = probability of survival after static load to observed failure time

Table 80. Predicted survival probability using CARES of 5 NT154 spin disks which failed on the surface at 1371°C from SCG

Disk ID	Failure speed— rpm	Failure time— hr	Survival probability	
			Fast fracture	SCG
8	41,800	6.966	0.999789	0.952289
26	50,000	11.08	0.998212	0.516195
24	50,000	0.0166	0.998212	0.952132
14	55,000	0.95	0.994415	0.406340
15	45,000/50,000	10.0/0.40	0.998212	0.790360

Table 81. NT154 spin disks tested at 1371°C and failed on the surface have lives that are roughly bounded by the mean and 5% failure probability lives predicted by CARES using the SCG methodology

Disk ID	Failure speed—rpm	Predicted 5% failure life —hr	Observed life—hr	Predicted mean life—hr
8	41,800	7.85	6.966	4,300
26	50,000	0.0186	11.08	10.16
24	50,000	0.0186	0.0166	10.16
14	55,000	0.0007	0.95	0.40
15	45,000/50,000	0.22/0.009	10.0/0.40	121/4.9

Figure 157. Comparison of observed NT154 1371°C spin disk lives with both the mean life and the 5% failure life predicted from SCG methodology and the CARES program.

Table 82 summarizes the values of the equivalent time zero stress for the five disks.

Figure 158, which compares the effective time zero stress (corrected for the effective surface areas) to the fast fracture prediction with 90% confidence limits, shows the predicted results lie within the confidence bounds, again verifying a reasonable correlation.

Table 82. Effective time zero stresses, calculated from CARES results for NT154 spin disks at 1371°C, for comparison with fast fracture results

Disk ID	<u>Survival probability</u>		Peak stress— MPa	Characteristic strength— MPa	Effective stress— MPa
	Fast fracture	SCG			
8	0.999789	0.952289	79.35	325.21	196.65
26	0.998212	0.516195	107.66	308.98	288.40
24	0.998212	0.952132	107.66	308.98	186.94
14	0.994415	0.406340	127.40	302.32	297.08
15	0.998212	0.790360	107.66	308.98	242.76

Figure 158. Comparison of NT154 1371°C spin disk SCG time zero effective strength versus predicted fast fracture strength. Test results lie within the predicted 90% confidence band.

VII. CONCLUSIONS

7.1 MULTIAXIAL FAST FRACTURE

7.1.1 High Strength Silicon Nitride is Shear Insensitive

The high strength silicon nitride materials analyzed in this program, GTE PY-6 and Norton NT154, demonstrated a very low shear sensitivity. In particular, for PY-6, the diametral disk compressive test indicated a value of $c = 1.52$ at room temperature. The value of c in excess of 2 was measured for NT154; c is the ratio of pure shear stress intensity at failure to pure-opening-loading toughness.

7.1.2 The Accurate Prediction of Biaxial Surface Fast Fracture Strength is Difficult

Experience has shown the prediction of biaxial fast fracture surface reliability at room temperature tends to be difficult. Component surfaces differed from specimen surfaces in terms of residual stress, microstructure, and machining induced flaws. In particular, the biaxial stress fields result in stresses acting across machine marks. Uniaxial specimens are manufactured by using longitudinal machining processes which minimize the effects of machining marks on strength. Examination of a PY-6 room temperature circular plate indicated the failure occurred across machining marks.

A fast fracture strength analysis was conducted for three multiaxial components, which failed at the surface, constructed from both PY-6 and NT154 materials. The application of conservative uniaxial specimen data and the Weibull PIA methodology produced the best correlation with the observed fast fracture results.

7.2 BIAXIAL SLOW CRACK GROWTH

SCG methodology using uniaxial specimen material constants gave a reasonable prediction of the observed lives for both NT154 and PY-6 components tested in a temperature range of 1200 to 1371°C, even though metallurgical observations indicate the presence of creep damage above 1300°C.

7.3 CREEP DEFORMATION AND FAILURE

Uniaxial specimen testing confirmed that creep deformation can be described reasonably well with a 'classical' Norton stress dependency and an Arrhenius temperature dependency. The Monkman-Grant relationship correctly relates the rupture time to the observed creep rate.

7.4 OXIDATION IN THE ABSENCE OF STRESS

The oxidation model indicates that the room temperature fast fracture strength is linear with elevated temperature exposure time raised to a negative power. An activation energy term can be used to consolidate temperature data, although for PY-6 material the relationship between strength and exposure time is nearly temperature independent from 1260 to 1400°C.

VIII. RECOMMENDATIONS

8.1 MULTIAXIAL FAST FRACTURE

Shear insensitive fast fracture models should be used in the analysis of the high strength silicon nitride materials such as PY-6 and NT154. The Weibull PIA model is the most conservative

of these models and best predicts the biaxial fast fracture strengths of components tested within this program. For these reasons, this model is recommended in the preliminary design process until experience with the actual part indicates this model is too conservative.

The accurate prediction of the biaxial surface strength of a ceramic component is difficult, and in the initial design a conservative approach should be applied. If possible, uniaxial specimens should be obtained from actual components with machine marks transverse to the specimen. If this is impractical, the use of the lowest strength data available is recommended. Button-head tensile bars consistently showed the lowest fast fracture surface Weibull properties of the uniaxial specimens tested in this program.

For some fast fracture data sets, the handling of volume failures as suspended items in the MLE determination of surface Weibull properties increases the calculated value of characteristic strength. While the use of suspended items is fundamentally sound, it has been found within this program that the additional 'safety factor' obtained by not using suspended items has resulted in a better prediction of the measured fast fracture strength of the biaxial verification components.

To summarize, based on verification testing and analysis, it is preferred that initial fast fracture surface analysis of the high strength silicon nitride components be based on:

- the Weibull PIA model
- specimens carefully prepared to duplicate the component surface properties

If these types of specimens are not available, use data from the lowest strength specimens. Suspended items should not be considered if their use increases the computed characteristic strength of the test bars.

8.2 BIAxIAL SLOW CRACK GROWTH

SCG methodology gave a reasonable prediction of the observed lives of both NT154 and PY-6 verification components in the temperature range of 1200 to 1371°C, even though metallurgical observations reveal the presence of creep damage above 1300°C.

Based on these test observations, it is recommended that above 1300°C both probabilistic creep and SCG analysis should be conducted. The more conservative result should be used to estimate the life of the component. It is also recommended that the stress analysis be based on an elastic FEM analysis, without creep redistribution. At this time, the additional conservativeness appears necessary to avoid a design with a high risk of premature failure.

The most difficult portion of the SCG methodology was obtaining a reliable estimate of the component surface fast fracture strength. It is recommended that component fast fracture tests be conducted to verify the reliability of predictions based upon specimen data.

A second difficulty was encountered in determining the NT154 SCG surface failure constants. Available tensile bar specimens failed internally, requiring an approximation of the surface properties. In the initial design of a component it is necessary to estimate the time dependent failure probability resulting from both internal and surface failures. Thus, there is a need for specialized specimen designs to characterize each of these types of failures.

8.3 CREEP DEFORMATION AND FAILURE

Ceramic materials exhibit a great deal of scatter in the time to rupture. As a result, rupture failures have a probability of occurring in a region with stresses less than the component maximum. Because of this, it is recommended that an analysis of creep damage be based on a probabilistic model. Such a model has been developed and incorporated into the CARES program.

8.4 OXIDATION IN THE ABSENCE OF STRESS

A probabilistic oxidation model applicable only to the component surface was incorporated into CARES.

The failure probability resulting from oxidation should not always be applied. For example, specimens tested in air may fail on the surface from SCG. In this situation, the effect of oxidation is already included in the test data as part of the SCG failure mechanism.

It is recommended that the oxidation damage methodology be applied only when the other time dependent failure modes, SCG or creep, do not contain the surface oxidation effects. In this situation, the component survival probability is the product of the survival probability from oxidation combined with the probability of survival from the other time dependent modes.

IX. SUMMARY

This comprehensive ceramic methodology development program has resulted in the completion of several major tasks. These include:

- The generation of an extensive material data base characterizing a high strength advanced silicon nitride material, GTE PY-6, applicable to efficient high temperature ceramic components.
- Implementation into the CARES program durability models describing significant time dependent ceramic failure mechanisms and allowing the extraction of the applicable material constants from basic specimen tests.
- The addition of probabilistic models into the CARES program, to allow for reliable, efficient design of ceramic components, accounting for the damage caused by time dependent failure modes.
- The use of subcomponent test results to verify the design methodology as well as provide information to aid in the 'fine tuning' of the design system approach.

9.1 MATERIAL DATA BASE

GTE PY-6 silicon nitride material was extensively characterized from room temperature to 1400°C. Properties obtained included the Mode I fracture toughness and the Mode II shear toughness in mixed mode failure as well as properties related to the failure modes of fast fracture, subcritical crack growth, creep, and oxidation.

At room temperature, diametrically compressive loaded chevron-notched disks resulted in the measurement of mixed mode fracture toughness. Notched specimen tests measured the pure opening-loading Mode I toughness, K_{Ic} , at elevated temperatures.

Button-head tensile bars and Type-B MOR specimens were tested at room temperature, 1000, 1100, 1200, 1300, and 1400°C to obtain the Weibull two-parameter fast fracture material constants at each temperature.

Tensile specimens tested under constant loading at elevated temperatures resulted in the development of slow crack growth material constants. Creep rate information, as well as rupture time information, is available between 1300 and 1400°C.

At elevated temperature both MOR specimens and tensile bar specimens subjected to a constant rate of loading provided an alternate approach to obtain SCG constants. Results obtained at four test facilities using three types of specimens were quite consistent, however, results did differ for the statically loaded tensile bars.

At temperatures ranging from 1000 to 1400°C, 125 Type-B MOR bars were oxidized for times as long as 500 hours. Measurements of room temperature fast fracture strengths as well as the thickness of oxide layer was recorded. A model was developed that related the observed strength to the temperature and the duration of exposure.

9.2 DETERMINATION OF TIME DEPENDENT MATERIAL PROPERTIES.

As part of this program, computer algorithms were incorporated into CARES. These algorithms allowed the reduction of specimen data to produce material models applicable to SCG, creep, and oxidation.

The SCG theory, added to CARES, is based upon the well accepted relationship that the crack velocity is proportional to the Mode I stress intensity raised to a power, the crack velocity exponent. An activation energy term has been shown to consolidate specimen data over a range of differing temperatures, and this is also incorporated into the basic theory. The analyst is given the choice of either using the activation energy to consolidate data or of reducing data at each temperature. In addition, the activation energy or the crack velocity exponent may be fixed in order to apply 'engineering judgement' to the development of the material models.

Specimen creep rate behavior is assumed to follow the 'classical' Norton law, wherein the creep rate is proportional to the applied stress raised to a power. An activation energy term is applied to consolidate different temperatures. The Monkman-Grant equation relates rupture time to the creep rate raised to a power. In general, a total of five material constants are needed to completely relate the creep rate and rupture time to the applied stress and temperature loading.

While both the rate and failure time information may be consolidated together, the flexibility to fit this information separately has been incorporated into the code. The analyst may also individually fix the Norton exponent, the Monkman-Grant exponent, or the activation energy.

An oxidation model was developed and incorporated into the CARES code. This model assumes a diffusion law applies to the oxidation process. With this model, the logarithm of the oxide layer thickness is proportional to the logarithm of exposure time. Since the critical flaw was assumed proportional to the oxide thickness, an equation resulted relating fast fracture strength to exposure time raised to a power. An activation energy term is also available to consolidate data for multiple temperatures. This technique has been shown to model the degradation of strength with time and temperature for PY-6.

9.3 PROBABLISTIC TIME DEPENDENT MODELS

In a manner similar to the fast fracture failure probability, the CARES program has been coded to compute the time dependent failure probability of a component.

The program uses material model information described previously, and the output of a component finite element stress analysis. This output defines elemental stresses, areas, volumes, and temperatures. At a specified point in time, the survival probability of the component can be determined resulting from the combined SCG, creep, or oxidation damage.

While the overall survival probability of a component is the product of the three different time dependent damage mechanisms, this requires some clarification. Usually, specimens tested in SCG or creep are exposed to an oxidizing environment. Thus, SCG or creep data derived from such specimens already implicitly contain the effects of the environment. The addition of oxidation damage to the creep or SCG failure probabilities in effect 'double count' the oxidation damage and should be avoided. Since SCG involves the time dependent growth of initial flaws, the subcritical crack growth failure probability at time zero is the fast fracture failure probability. As a result, fast fracture is used only to predict the component initial time zero failure probability.

At this time, the stresses in a component are assumed to be constant with time, i.e. creep stress relaxation is not taken into account. The methodology currently used, thus, has a conservative bias in estimating the failure probability.

9.4 SUBCOMPONENT MODEL VERIFICATION

A number of subcomponent test results were analyzed to verify the capabilities of the proposed failure models. These tests are summarized in Table 83, some additional information follows.

In biaxial fast fracture at room temperature three components were examined:

- a flat circular plate of PY-6 loaded by a center ball with an outer simple diameter support, so as to produce peak surface stress at the center
- a flat circular plate of NT154 loaded by uniform pressure with an outer diameter simple support, so as to produce peak stresses on the surface at the center
- an NT154 spin-disk, while the highest stresses are internal, more disks experience a surface failure; both surface and volume failures are analyzed

Table 83. Confirmatory test correlation summary

Component	Material	Temperature —°C	Failure mode	Correlation
Flat disk	PY-6	20	FF/surface	Reevaluation of MOR material data required
Flat disk	PY-6	1200	SGG/surface	Correlation using existing tensile stress rupture specimens
Flat disk	PY-6	1300	SGG/surface	Correlation using existing tensile stress rupture specimens
Flat disk	NT154	20	FF/surface	Correlation using existing tensile and MOR specimens
Spin disk	NT154	20	FF/surface	Prediction using tensile specimen data strength 15% above observed
Spin disk	NT154	20	FF/volume	Correlation using existing MOR specimens
Spin disk	NT154	1204	FF/surface	Predicted strength 26% above observed; 1204°C disk fast fracture strength was used in 1204°C and 1370°C slow crack growth disk correlations
Spin disk	NT154	1204	SGG/surface	Correlation using 1204°C fast fracture disk strength and projected surface slow crack growth values estimated from 1204°C tensile stress rupture volume specimens
Spin disk	NT154	1371	SGG/surface	Correlation using corrected 1204°C fast fracture disk strength and projected surface slow crack growth values estimated from 1371°C tensile stress rupture volume failures

The prediction of the fast fracture strength distribution of several of the biaxial components using the Weibull methodology worked well. These specimens included:

- the flat circular PY-6 plate loaded biaxially, which failed on the surface at room temperature
- the flat NT154 pressurized plate uniformly loaded at room temperature, which failed on the tensile surface
- the NT154 disks, which were spun at room temperature and failed internally

The analysis of the fast fracture strength of NT154 spin disks that failed at room temperature on the surface was optimistic. The strength estimated from button-head tensile specimens was about 15% high. The button-head specimens have the lowest fast fracture strength of available specimen data. An additional safety factor was added by not using volume failures as suspended items in the determination of Weibull material constants. Inclusion of volume failures results in an even higher estimation of the characteristic strength.

If data obtained from specimens cut from the actual component were not available, the button-head specimen data without suspended items were used. For the flat NT154 pressurized plate, the characteristic strength was estimated within about 1/2% with this approach.

The following component failures were reasonably described with the use of multiaxial SCG theory.

- the 1200 and 1300°C PY-6 flat circular plates loaded by a center ball and an outer simple support resulting in a time dependent failure under a static load
- an NT154 spin-disk spun at 1204°C that failed at the surface; rotation speeds are held constant for a period of time and then stepped up to a new constant speed until a time dependent failure occurred
- an NT154 spin-disk spun at 1371°C which failed at the surface; rotation speeds are held constant until a time dependent failure occurred; originally the intention of this test was to produce a creep failure, SCG methodology correlated with the observed results

While the multiaxial SCG theory worked reasonably well, two difficulties were encountered in the analysis of the high temperature NT154 spin disks.

- Even the use of conservative tensile bar fast fracture data tended to overpredict the observed disk strengths.
- While the disks failed on the surface, the tensile bars failed internally.

The application of the SCG methodology requires a knowledge of the Weibull fast fracture material properties. The fast fracture strengths demonstrated by the NT154 spin disks were about 15% lower than those conservatively predicted from uniaxial tensile specimens. The demonstrated disk fast fracture properties were thus incorporated into the time dependent analysis; this approach worked well.

The button-head tensile bars failed internally rather than on the surface. The use of internal SCG material constants to estimate the surface values seemed to work reasonably well, however, had surface specimen failure data been available, the credibility of the analysis would have strengthened.

In the design of new parts, it is necessary to be able to determine the effects of both surface and volume reliability upon the design life requirements. This requires specimen data to determine time dependent material constants of both surface and volume. It may be necessary to design specific specimens which fail in each of these modes.

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NOMENCLATURE

C	Celsius
CARES	ceramic analysis and reliability evaluation of structures
cm	centimeter
F	Fahrenheit
FEM	finite element model
FF	fast fracture
Gamma	gamma function
K	degrees Kelvin
KJ	kilojoules
ksi	kilopounds per square inch
m	meter
MLE	maximum likelihood estimator
mm	millimeter
MOR	modulus of rupture
Mole	molecular weight
MPa	megaPascal
MSC-NASTRAN	NASTRAN code maintained by the MS Corporation
Mum	micrometers
NIST	National Institute of Standards
NASTRAN	general usage FEM code
PIA	principle of independent action
POS	probability of survival
PY-6	GTE silicon nitride material
NT154	Norton silicon nitride material, tested by AlliedSignal Aerospace
SCG	slow crack growth
SoRI	Southern Research Institute

SYMBOLS

A	oxidation strength coefficient, crack velocity coefficient, Monkman-Grant coefficient, rpm stress coefficient
a	support radius of ball-on-ring specimen, crack half length, Monkman-Grant exponent, pressurized plate stress constant
Ab	ball-on-ring effective area
Ac	angle between crack normal and specimen axis
Aeff	effective area
Ai	parameter to find KI, area of ith element
Ang	angle between crack plane and load vector
A(PIA)	effective area for PIA model
Ar	effective area of pressurized plate due to radial stress
As	specimen effective area (based on top surface)
At	effective area of pressurized plate due to tangential stress

b	radius of center loading for ball-on-ring specimen, oxidation growth rate exponent, pressurized plate stress constant
B	slow crack growth coefficient, disk thickness, creep rate coefficient, thermal stress term
Bc	slow crack growth coefficient of component
Bi	parameter to find KII
B1	slow crack growth coefficient corrected for temperature
Bs	slow crack growth coefficient for surface failure
Bt	slow crack growth coefficient corrected for temperature
Bv	slow crack growth coefficient for volume failure
c	crack length, K_{IIC}/K_{IC}
C	oxide thickness coefficient, ball-on-ring stress constant
c1,c2	crack length
Cr	creep rate
Cv	static coefficient of variation
dt	time increment
E	elastic modulus, oxide strength coefficient
Exp(X)	e^x
h	MOR specimen thickness
H	measured hardness
i	specimen rank 1,2,3...N
KI	Mode I fracture intensity
KII	Mode II fracture intensity
K _{IC}	Mode I fracture toughness
K _{IC} (T)	Mode I fracture toughness at temperature
K _{IC} (RT)	Mode I fracture toughness at room temperature
K _{IIC}	pure shear fracture toughness
L1	outer load spacing on a MOR specimen under 4-point load
L2	inner load spacing on a MOR specimen under 4-point load
li	length of ith element
leff	effective length of corner for Type-B MOR specimen
m	Weibull modulus
mc	consolidated Weibull modulus
mcr	Weibull modulus related to creep strength scatter
mo	Weibull modulus in oxide statistical model
ms	surface Weibull modulus, specimen Weibull modulus
mv	volume Weibull modulus
Mu	Poisson's ratio
N	crack velocity exponent, total number of data points, Norton creep exponent
NI, NII	intermediate variables to find KI,KII for compressive disk
Npts	total number of data points
Ns	number of samples
p	pressure
P	load, indentation load
Pf	failure probability
Pfi	failure probability of ith specimen
Pi	3.14159
Po	fast fracture probability of survival
POS	probability of survival
POSi	probability of survival of ith element
POSij	probability of survival of ith element from jth principal stress

Q	activation energy
r	radial location on flat disk
r1	support radius on pressurized circular plate
r2	radius of pressurized circular plate
R	disk radius , universal gas constant
rpm	rotor rotational speed
S	strength, characteristic strength, applied stress
•	
S	applied stress rate
Sa	applied stress
Sb	ball-on-ring strength, peak stress on pressurized plate
Sch	specimen characteristic strength at test temperature
Seq,o	effective time 0 stress (SCG)
Sf	fracture strength, fracture strength at crack angle (indented MOR bar), final mission stress (SCG)
Sfi	observed static strength
Sfp	predicted static strength
Si	stress on ith element, specimen, ff strength, parameter used to find KII
Sij	jth principal acting on ith element
Si,o	effective time 0 stress (SCG) for ith element
Sm	mean strength at a single temperature, at time
Smi	observed mean strength of ith specimen
Smax	maximum stress
So	unit characteristic strength
Socr	unit characteristic strength in creep statistical model
Soo	unit characteristic strength in oxide statistical model
Sr	radial stress
Sref	reference peak stress (at 50,000 rpm for room temperature spin disks)
S(rpm)	stress at any rpm for room temperature spin disks
Srt	strength of oxide specimens at room temperature
Ss	specimen strength
St	elevated temperature strength of oxidized specimens, tangential stress
Sth	characteristic strength
S(tf)	stress at failure in SCG
Su	fracture strength at a zero angle (indented MOR bar)
S1, S2	strength of specimen 1,2
t	thickness of ball-on-ring specimen, time
T	absolute temperature
Th	oxide thickness
Ti	parameter used to find KI
Tf,tf	failure time
Tfi	failure time of ith specimen
Ve	effective volume of 4-point bend MOR specimens
VE	effective volume of E-Bar MOR specimens
Veff	effective volume
V4	effective volume of 4 inch MOR specimens
Vi	volume of ith element
w	MOR specimen width
Y	geometry factor

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