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ASSESSMENT OF STRENGTH LIMITING FLAWS IN CERAMIC HEAT EXCHANGER COMPONENTS

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FINAL REPORT

Property Counsel
DOE Field Office, Chicago

For the Period
September, 1984 - June, 1993

WORK PERFORMED UNDER CONTRACT

DE-AC07-84ID12566

and COOPERATIVE AGREEMENT

DE-FC02-89ID12887

BABCOCK & WILCOX COMPANY
Research & Development Division
Lynchburg Research Center
Lynchburg, VA 24506

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For the Period
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By:
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ABSTRACT

The ability to predict energy efficient lifetimes for ceramic heat exchanger components is necessary for their design, manufacture, and sale. The ability to inspect components for critical defects and adherence to specifications is also vital.

This is the final report of a three phase program. In phase 1, various nondestructive evaluation methods were evaluated for use on siliconized silicon carbide heat exchanger tubes. The more promising ones were further developed for use in phase 2. These methods were used to examine samples used in a mechanical testing program, carried out at room temperature and at high temperature, to determine the detectability of defects and the effects of load at high temperature. A model was developed for fast fracture reliability.

During phase 3, the equipment installed during phase 2 was modified for faster, easier ultrasonic scanning and microfocus X-ray computed tomography. Samples were scanned and tested at room temperature and at high temperature, to determine the detectability of defects and the effects of load at high temperature. Models were generated and refined to use the test data to predict lifetimes, both for fast fracture and under high temperature creep.

This report describes the methods used to improve ultrasonic scanning of tubes, to reduce artifacts and enhance defect detection by X-ray computed tomography, to carry out pressure tests and high temperature C-ring tests with acoustic emission monitoring, and to generate a lifetime prediction model. The final model, the verification test results, and a general procedure for establishing specifications and acceptance tests for ceramics are presented.

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FOREWORD

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This report reflects efforts of the following project team members:

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EXECUTIVE SUMMARY

This is the final report for a three phase program. The goal of the program was to be able to ensure an acceptable lifetime for ceramic heat exchanger components. This requires the ability to write component specifications based on a knowledge of the effects of service conditions on the material and the inevitable defects present. It also requires the ability to test the components for compliance to the specifications and for the presence of defects outside the allowable limits. This problem of specifications and testing is common to all advanced materials.

Phase 1 of this program examined the ability of various NonDestructive Evaluation (NDE) methods to locate defects in silicon carbide heat exchanger material. The conclusion was that ultrasonic and X-ray methods are generally useful, although techniques must be adapted for the characteristics of ceramic materials. The Scanning Laser Acoustic Microscope (SLAM) works well for the detection of cracks in the outer surface of tubes, but because of surface roughness and scattering it is not able to pick up inclusions or voids in this particular material.

Ultrasonic C-scan is capable of detecting both inner and outer surface cracks, inclusions larger than normal porosity, regions of high porosity, and variations in wall thickness. Microfocus X-ray is most valuable for detection of high density inclusions and unusual regions of porosity and thickness variation. In all these cases, the defect detection limitations are set by the material characteristics rather than the equipment limitations. The material is nominally 85% silicon carbide, with 15% silicon in the pores. This basic structure results in occasional large pores which are usually but not always filled with silicon. The material may be either extruded or slip cast. Either way results in a somewhat roughened surface. The roughness does not affect the structural integrity but will mask the presence of small cracks. Because of this, the defect threshold size must be set to about 1 millimeter, with anything smaller considered simply part of the normal material condition.

Phase II had three sub-tasks. They were (1) the improvement of NDE capabilities by technology transfer from the national laboratories, (2) the development of a Linear Elastic Fracture Mechanics (LEFM) model for the prediction of tube life, and (3) a material testing program.

NDE capabilities were improved by the addition of Time-of-Flight Acoustic Microscopy

(TOFAM) to the ultrasonic scanning program, and by the addition of Computed Tomography (CT) to the microfocus X-ray system. The TOFAM system was developed at the Idaho National Engineering Laboratory (INEL), Materials Characterization/NDE Section, and the X-ray CT reconstruction programs were supplied by Sandia National Laboratory, Nondestructive Testing Technology Division.

The LEFM model is the basis for establishing acceptance criteria for ceramic heat exchanger tubes. It was created using published crack growth models and material test data.

The testing program used samples taken from silicon carbide heat exchanger tubes. Tests were run both at room temperature and at operating temperature (1230°C) to determine material strength and effect of defects for the LEFM, and also to compare defects found by NDE with the actual strength limiting flaws in the material. This test program was coordinated with other tests on the same material carried out at INEL.

The phase 3 program was a continuation of phase 2. The TOFAM and CT systems were improved and used to select additional samples, an expanded testing program was carried out, and the LEFM model was modified and extended to include statistical properties and creep effects.

At the conclusion of phase 3, five papers based on this work were presented at the 1993 Annual Meeting of the American Ceramics Society. They were part of a special symposium, "Current Capabilities for Non-Destructive Testing and Lifetime Prediction", and will appear in a refereed ACerS book by the same title. The body of those five papers appear in this report, in the sections on time-of-flight acoustic microscopy, X-ray computed tomography, acoustic emission monitoring of testing, application of NDE results to fast fracture modeling, and modeling of high temperature creep effects on lifetime.

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SECTION 1

INTRODUCTION

Advanced ceramic materials are candidates for use in environments that are too hostile for metals. Metals have been developed with amazing properties, but there are limits. For example, the thermodynamic efficiency of a burner or of a heat engine increases with the temperature of the burner air supply or the maximum temperature of the engine. In these applications ceramics can maintain strength to higher temperatures than metals can tolerate. Unfortunately, acceptance of ceramics in these applications has been limited because ceramics are subject to sudden failure from brittle fracture.

This program is motivated by the national importance of the possible applications of ceramics and by the business requirements of producing and selling systems. The conservation of energy through increased thermodynamic efficiency and the conservation of strategic resources used in super alloys are important national priorities. The demonstration of reliable ceramic components is a business necessity, because customers will not buy a product with insufficient or uncertain service life. In order for energy saving or material saving systems to be attractive, the savings must be demonstrably greater than the costs. These include not only initial cost, but also costs of downtime and repair. The challenge is to take the scientifically demonstrated superiorities and deficiencies of ceramic materials through the engineering process to produce commercially successful products.

To produce products with a cost-effective lifetime, it is necessary to specify acceptance criteria for the materials and components. The acceptance criteria must be meaningful for the application, based on material properties and service conditions. It must be possible to verify that the criteria are satisfied by the materials received.

The goal of this program is to produce acceptance criteria and appropriate verification tests for one specific component: silicon carbide heat exchanger tubes. It is hoped that the same general approach will be useful for other materials and applications. The program has three major parts; NonDestructive Examination (NDE), Destructive Testing (DT), and Linear Elastic Fracture Mechanics (LEFM).

Because ceramics are subject to brittle fracture, the assessment of materials for conditions that could lead to failure is vital. This is the task of NDE. This report documents the various NDE techniques used in this program, from the first attempts in phase 1, through the selection and

application of techniques that are useful for phase 2, to improvements to make the testing faster and more flexible in phase 3. Both capabilities and limitations are described.

NDE examination results need to be compared to failure test results, and LEFM requires test data to create and verify the fracture model. This is the task of DT. The testing methods used are described, and results are presented. In addition to determining parameters for the modeling, some testing was carried out in an attempt to better understand the failure mechanisms.

Acceptance criteria must be based on a model that can predict tube lifetime given operating conditions, material characteristics, and possible flaws present. This is the task of LEFM. A model was developed in phase 2, and it was refined and extended in phase 3.

The need for better understanding of ceramic materials is demonstrated by the statistical distribution of fracture toughness test results. The results of a ceramic testing program are often presented as a mean and standard deviation, as is normal practice in other material testing. This form of presentation is useful in many technical applications, because engineering test results usually vary with the normal bell-shaped curve and the standard deviation defines the width of the bell. In the measurement of any property of a physical system, there will be random errors in the measurement and random fluctuations of the property being measured. These measurements are usually close to the average value, with small deviations much more likely than large deviations, so the cumulative results take on the familiar bell curve.

Ceramic test results do not generally produce bell-shaped curves. Fracture toughness testing in this program did not, and, in other published ceramic test reports which present their complete data set, one is most unlikely to find a normal distribution. The usual distribution for ceramic test data is a fairly uniform distribution from a minimum to a maximum, with little evidence of tailing at the edges or peaking in the middle. The distribution looks like a mesa, not a bell. This same kind of result could happen in testing steel, if no controls on heat treating or chemistry were applied. The question is, what are the unknown, uncontrolled parameters in ceramics? The broad, uniform distribution of test results implies a broad, uniform distribution of properties. An understanding of the sources of variation, and especially the causes of unusually low strength in some samples, would be both scientifically interesting and valuable for process control.

These topics are discussed in the following sections. Because of the range and quantity of material presented, each section has been written to be self-contained. It is the intention that

each discussion should be clear to the non-expert while providing sufficient information to allow understanding of motivation and replication of results. We hope that material which is either too obvious or too detailed may be skipped without losing the thread of the argument and still provide an understanding of the general results.

SECTION 2

NONDESTRUCTIVE TESTING

2.1 Preliminary Evaluation of Techniques

In phase 1, four promising technologies were selected for evaluation; ultrasonics, microfocus X-ray, acoustic holography, and Scanning Laser Acoustic Microscopy (SLAM). All of them can be useful, although the techniques require modifications from the mode of employment in metals. It is a general observation that the techniques will probably require further modification and development for each different material or application.

2.1.1 Ultrasonics

Ultrasonic testing (UT) may be used for scanning an area to locate defects or for evaluation of a defect after it has been found. In either case, it is generally carried out with one or two transducers. A single transducer may be used to send pulses and receive the echos (pulse-echo operation), or two transducers may be used, one to send the pulse and the other to receive the echos (pitch-catch operation). The transducers may be focused, or they may be 'plane wave' transducers.

Pulse-echo UT is the most common mode of operation. A short pulse of sound is transmitted, and the distance to a reflector is determined by the round trip travel time. The beam diameter at any distance defines the volume being studied at the corresponding time. The diameter may be calculated from the wavelength, transducer diameter, and focal length, or may be measured experimentally.

In pitch-catch UT, the intersection of the transmit and receive beams defines the volume which is being studied. This kind of UT is generally useful for very short path lengths, in which a pulse-echo transducer would still be ringing from the transmitted pulse when the echo returns. It is also useful for cases in which the direction of the reflected beam is such that the transmitting transducer can not receive the echo. It can be used in cases in which the scattering from closer objects (pores, grain boundaries) obscures returns from deeper defects. It is very useful for detection of delamination in plate materials with the transmitter on one side of the plate and the receiver on the other, because the delamination will cause a loss of signal.

Although tone bursts can be transmitted, in most cases the transducer is excited by a single sharp pulse. The transmitted sound pulse depends upon the resonance frequency and damping of the transducer. Higher damping produces a shorter pulse, which improves separation of multiple returns, but it also decreases sensitivity. Higher frequency gives shorter wavelength, allowing a smaller focal spot size and also allowing better source location, but gives greatly increased scattering from small pores and grain boundaries. This causes noise problems and lack of penetration depth.

Sound may travel in solids as longitudinal waves, shear waves, or surface waves. The modes involved depend on the material characteristics and the angles at which the waves hit surfaces or defects. Mode conversion is possible in any reflection, including scattering from porosity. Waves of different modes travel at different velocities, further complicating test results. Because the critical flaws in ceramics are very small, the tendency is to use the highest frequency possible to maximize the return signal strength.

Silicon carbide samples, both plates and tube sections, were tested. Some samples had cuts or known inclusions. The tube samples were apparently slip cast, and the inner and outer surfaces were quite different. The outer surfaces were very rough on a 10 micron (0.0025") scale, but quite smooth over a larger scale. The inner surfaces were very smooth on a fine scale but had irregular roughness (bumps) over scales of 1 cm (0.5 inch). Some samples were polished and some were tested as received.

Surface wave testing has proven successful on ceramic materials, because the waves restricted to the surface reflect from defects. In the tube samples, the technique proved impractical. The small scale roughness of the outer surface caused too much attenuation for useful scanning, and the large scale roughness of the inner surface made it impossible to maintain the required beam angle. This form of testing was dropped after phase 1.

Ultrasonic measurement of wall thickness worked very well. Thickness is determined by the round trip time for signals bouncing back and forth between the wall surfaces. It can be carried out by directly timing the pulses or by feeding the pulse train into a spectrum analyzer which determines the frequency of the echos. ($\text{Frequency} = 1 / \text{round-trip-time.}$) The sound velocity varies slightly with density, but the variation is too small to matter for this application. Relatively low frequencies can be used to minimize attenuation. The material used in this program has a wall thickness of 6 mm, giving a round trip time of 1 microsecond. A wall thickness resolution of 1% of this value is easily measured with normal laboratory equipment.

Scanning for defects worked best with a 25 MHz transducer with a 50 mm (2 inch) focal length in water. Because of the factor of 8 difference in longitudinal wave velocity between the water and the ceramic, this focal length is about 6 mm (0.25 inch) in ceramic, which is the wall thickness. This frequency gives a wavelength of about 50 microns (0.0012") in silicon carbide. It was selected to optimise defect detection. Many individual pores or grains are detected, so there is no need to go to higher frequency for detection of smaller defects, and attenuation is not too bad, so there is no need to go to lower frequency for better penetration. This frequency is well above the range usually used for metals (1 to 5 MHz) in order to get a short wavelength, scaled for the smaller defects and path lengths in this material. The scanning system was further developed later in the program.

2.1.2 Microfocus X-Ray

X-ray is a well known technique for internal inspection of materials. It is capable of detecting inclusions, porosity, and cracks if they are oriented properly. Two limitations to standard radiography, that appear when the small size of critical flaws in ceramics is considered, may be overcome with microfocus radiography.

In a standard X-ray tube, the radiation source has a diameter of a millimeter or more. This produces an effect called geometric unsharpness. The rays reaching any point on the sample arrive from a cone of angles whose base is the area of the source, and proceed to the film or detector in the same cone. The result is that every point in the sample is projected to a circular area on the detector whose size is given by

$$U_g = f b/a$$

where f is the source spot size, b is the sample-to-detector distance, and a is the source-to-sample distance. Unless the sample is very close to the detector, all edges will be unsharp and small defects will be too blurred for detection.

The other limitation is in the small size of the critical defects. Film must be examined with magnification and real-time monitoring, which uses a phosphor screen and TV camera in place of film, simply doesn't have the resolution. This can be overcome by projection magnification. If we imagine a point-like source of X-rays, all the rays reaching the sample will spread out in a cone with the source at its apex and continue spreading until they reach the detector. Using the same a and b distances as above, this will produce a magnification factor of

$$M = (a + b) / a = 1 + b/a$$

If we compare the magnification factor with the geometric unsharpness, we see that to get a reasonably sharp image of a defect smaller than the source size b/a must be near zero. However, if the source is smaller than the defect magnification will be useful and if the source is much smaller than the defect unlimited magnification is possible. This is the value of microfocus X-ray. It essentially eliminates geometric unsharpness and so allows projection magnification sufficient to make defects easily visible on either film or real-time radiography.

The system used in this program has been used to resolve 3 micron lines and 2 micron spaces, at a projection magnification of 100.

The system was used in scanning plates and tube sections. It showed voids and inclusions quite well, but had two limitations. First, sensitivity to defects was limited by surface roughness. The samples had bumps, pits, and internal pores. Any features smaller than the surface roughness had to be ignored. Second, it was impossible to locate the depth of the flaws. There was no way to tell apart surface pores and internal pores, for instance. These were addressed later in the program by developing a computed tomography system based on the microfocus system.

2.1.3 Acoustic Holography

Acoustic holography is an exact analogy of optical holography. In basic optical holography, an object is illuminated by a laser beam. The reflected laser light is mixed with a reference beam from the same laser and the interference pattern is recorded on film. After development, the film is mounted with the reference beam passing through it. Because the film has a record of the original interference pattern, the light passing through takes on the same pattern. Because the light has the same pattern as the original it propagates the same way, a person looking through the hologram can see the original scene, in three dimensions. White light and reflective holograms are later developments, but the basic principle is the same.

In acoustic holography, a tone burst is transmitted toward the object to be imaged. The return echo is mixed with the same oscillator signal and the resulting interference pattern is recorded over the viewing aperture. The interference pattern is stored in a computer, and corresponds to the developed hologram in the optical case. The mathematics of holography is well understood, so the acoustic hologram is reconstructed mathematically. The distance from the recording plane to the object is known, from the ultrasonic measurements, so it is possible to

reconstruct the plane of the object, forming an image of the object.

This method may be useful in some other ceramic material, but in our silicon carbide samples the scattering from pores and/or grains is so intense that the result was rather like trying to make an optical hologram in a cloud. The scattering randomized the interference pattern and prevented imaging the defects. In retrospect, this is obvious. Focused transducers can locate defects because only a small volume of material is returning sound at any one time. The return from a defect can stand out against the local porosity. In acoustic holography the beam is very broad, rather than focused, and the return from the defect is a small fraction of the returns from the entire volume.

2.1.4 Scanning Laser Acoustic Microscope (SLAM)

The SLAM is a commercial instrument. It is useful for examining a variety of materials, including ceramics, and it has better speed and sensitivity for detecting surface cracks in our material than any other method tried. Images of the area being examined are viewed in real-time on CRT displays.

The system operates by sending a continuous plane wave ultrasonic beam into the bottom of a sample and producing an image of the pattern of ultrasound that emerges from the top. This detects any surface or internal structures that disrupt or modify the sound waves.

The sound waves propagate at an angle so that the emerging waves produce microscopic ripples on the gold surface of a plastic block which serves as a "cover slip". A laser beam scans the cover slip in a TV raster pattern. The ripples produce small variations in the direction of reflection of the beam. The reflected beam passes a knife-edge to a photodetector, whose output is a signal modulated at the same frequency as the modulations of the beam direction. This frequency is the same as the ultrasound except for a small shift caused by the motion of the scanning point on the cover slip. This modulated signal is passed through an amplifier and filter where it is converted to a video amplitude. The CRT sweep is synchronized with the laser sweep and an image of the sound pattern is created.

The angle between the wave direction and the normal to the coverslip is 10° . With the sample parallel to the coverslip, this 10° angle of incidence in water produces a 65° shear wave in silicon carbide. This angled beam is useful for detecting the severest type of defect, a surface connected crack. The sound will not cross the crack, so a large crack produces a distinctive shadow on the SLAM screen.

In some ceramic materials ultrasonic energy is quickly dissipated. This was not a problem in our siliconized silicon carbide samples, but scattering was. The material is 18% silicon, which fills most of the pores in the silicon carbide. At 24 MHz, the lowest frequency our SLAM can use, the wavelength in silicon carbide is 0.325 mm for shear waves and 0.525 mm for longitudinal waves. This is just about the size of the pores, so scattering is very strong. The scattering prevents the detection of defects much below the surface. It also causes problems with the detection of small cracks, because the overlapping scattered beams cause the image to have many small light and dark areas.

A technique was invented to alleviate this problem. As noted above, the relative motion of the laser beam and the sound waves produces a frequency shift between the sound frequency and the light modulation frequency. The electronic filter has a sharp edge at this frequency, with the result that the SLAM is sensitive to sound moving in one direction only. If the ultrasonic transducer is reversed, so that the 10° angle is right-to-left instead of left-to-right, the sound will not be detected. However, the waves that reflect from a crack are reversed and can be detected. A crack now stands out as a bright area in a dark background rather than a dark area in a bright background. This works because most of the scattering which interferes with normal operation is in a generally forward direction. Only a flat vertical surface like a crack will produce a strong reflection in the right direction. Using this technique, it is possible to locate indenter cracks as small as 0.05 mm on a polished surface.

2.1.5 Techniques Developed for Silicon Carbide Tubes

After the initial assessment of potential NDE techniques for silicon carbide tubes, it was clear that additional development was needed for two different applications. The first was for a rapid inspection method and the second was for a detailed inspection compatible with the small size of critical defects and the complexity of the structure on small scales. For rapid scanning, ultrasonic methods were developed, and for detailed analysis a computed tomography system based on microfocus X-ray was developed.

Acoustic holography was not selected because, as described above, it was not a useful technique for this material. SLAM was not pursued because it would have required extensive modification of the instrument to scan long tube sections. The standard instrument is best suited to small flat samples. The work done on tube samples was carried out on cut pieces with semicircular cross sections to allow access to the inner surface.

Additionally, all tube sections were examined by dye penetrant to detect cracks. The dye

penetrant located porosity and linear indications which may have been cracks, but none of them were strength limiting in testing, and none of the linear indications correlated with other defect indications found in other testing. The linear indications were presumably scratches in the green ceramic; there were no actual cracks in any of the material examined.

2.2 Time-of-Flight Acoustic Microscope (TOFAM)

2.2.1 Background

The TOFAM system was developed and built at the Idaho National Engineering Laboratory (INEL)¹. It consists of a control computer and data collection modules in two instrumentation crates. The basic principle is an ultrasonic C-scan of the sample. A C-scan is an image of the object being scanned, showing the areas in which sound is reflected from internal flaws. As the sample is scanned, ultrasonic pulses are sent out and the arrival times of echoes are recorded. The front surface echo is used as the basis for timing echoes from within the sample. Anything arriving between the front and back surface echoes will be color coded in the display to show the depth of the reflector (defect). Other color codes represent the loss of the front surface echo (rough surface or scanning beyond the edge of the sample) or loss of back surface echo (surface roughness, attenuation or deflection of the beam by a misoriented flaw).

The system is highly flexible in setup and operation, which also means its successful use is dependent on the operator's understanding of the computer program, the electronics, and ultrasonic testing. It is a very useful research tool, but not a production line testing instrument. It is the kind of research tool that can be used to design a specialized testing instrument.

The limitations of ultrasonic scanning in siliconized silicon carbide are largely set by the material. Outer surface roughness deflects the beam angles, or prevents penetration at all. Inner surface roughness mimics defects. Porosity produces scattering, and the return signals from pores set the minimum defect signals which can be isolated.

The TOFAM was designed several years ago, using what was then a state-of-the-art computer. Since then, because of the rapid increase in computer speed and memory, and the new languages and development tools in use, the original computer has been replaced with a PC clone, giving much faster processing, more flexible operation, and vastly better data communications.

The scanner for the TOFAM was designed and built by B&W. It has two linear scan axes covering a 15 x 15 cm area and a rotational scan axis. The rotational axis is placed in an

immersion tank, and can accommodate tubes up to 15 cm in diameter and three meters long. The linear scan axes are mounted on a carriage over the tank, and can be manually positioned over any part of the tank.

2.2.2 Modifications to TOFAM

The system has now been modified to scan with multidirectional beams, to transfer scan data to another computer for storage, and to scan more quickly. The multiple beams are controlled by an added multiplexer.

The multiplexer chassis contains eight pulser-receivers. Switches allow selection of any one channel or repetitive cycling through all eight channels. Each transducer operates at a center frequency of 25 MHz, has a diameter of 0.63 cm and a focal length in water of 6.35 cm. The operating frequency of the transducers was chosen to maximize defect sensitivity without significantly increasing effects due to attenuation. The determination of transducer size was, similarly, a compromise between limited available space and insufficiently long near field lengths associated with smaller diameter elements. The transducers are arranged to propagate 60 degree shear waves in the ceramic, all focused at the same spot on the inside surface of the tube. This is accomplished through the use of a specially designed transducer manipulator/holder. This fixture allows each of the eight transducers maximum position adjustability without compromising space requirements. The transducers are positioned every 45 degrees around the central focal spot. Alignment of the transducers is tricky enough that the technique is described in its own section, 2.2.3. Figure 2-1 shows a schematic diagram of the setup. Figure 2-2 shows a transducer holder, and figure 2-3 shows the manipulator/fixture with the 8 transducers mounted.

This fixture is not ideal, because the beam angle calculations presented below had not been carried out. It turns out that this design will not allow all 8 beams to be focused at the same internal point because of interference of the transducers. However, the design allows individual height adjustment and rotation of the eight transducer posts, with setscrew locking. It also allows rotation in a vertical plane by an adjusting screw, with the center of rotation located on the axis of the individual transducer post.

Figure 2-4 shows two of the scans, with "indications", or defects of unknown type. One of the indications is quite large, being detected by several transducers over a range of about 2 cm. The other shows up as two spots. These were produced by two different transducers from the same defect. There are two spots because the transducers are focused at the same point at midwall,

Figure 2-1: Schematic of TOFAM

Figure 2-2: Transducer holder

Figure 2-3: Transducer Fixture

Figure 2-4: Some TOFAM "Indications"

but the defect is at the inner surface as shown by the color code, which indicates depth.

One feature of the system, which is extremely important for rapid data collection but not too obvious, is the ability to collect data with the rotational axis of the tube sample moving either clockwise or counterclockwise. The images are line scans around the tube, with each line offset by a step along the tube. For reasonable ease in locating tube defects on the tubes, all scans should map onto the tube surface in the same manner. This can be done by shifting the tube in the holders, and moving the holders when necessary, but the re-alignment after each move is time consuming. With all motions reversible, tubes can be scanned in one rotational direction and one stepping direction over half of their length, then reversed end for end in the axial holders, and scanned in the opposite rotational and stepping direction for the other half. This allows the data point structure to be consistent on all sections of a tube. Overall examination time per sample is significantly reduced since readjustment of the tube in the axial holders need only be performed once. Very precise centering, within 1 mm, is required to provide a constant distance separation between the transducers assembly and the tube sample, necessary for accurate data collection. Poor positioning alters the beam propagation directions.

New alignment angles for the eight-head multiplexing transducer assembly were calculated as described in section 2.2.3 and utilized in order to get consistent image signals from near inner surface defects on TOFAM scans. All eight transducer channels "see" the same tube area, allowing for maximum clarity as they are cycled through the multiplexer. Image defect resolution is also strongly influenced by adjustment of the delay and signal threshold settings. The instrument still needs careful setup by an experienced operator for each new application, as any ultrasonic technique does.

Tubes are scanned by the TOFAM computer as follows:

- 1) Mark tube in 10 cm segments with water-proof marker.
- 2) Load tube through the two axial holders leaving scan area free for access by transducer assembly.
- 3) Center tube in front holder by reading "run-out" signal from scope trace, then center in rear holder by same procedure.
- 4) Create numbered file on hard disk for segment of tube.
- 5) Program motor controller for linear displacement and rotational direction for proper data collection.
- 6) Scan each of 9 sections in order, monitor for indications.
- 7) Loosen tube from axial holders and reverse end for end to examine the other 9 sections

of the tube.

- 8) Repeat steps 2 through 6 for the second half of the tube.
- 9) Transfer all 18 files to main lab computer for analysis.

The image of the section remains on the monitor for preliminary evaluation before the operator resets for subsequent data files.

This is obviously not a system optimized for large production runs, but it is significantly better than previous tube scanning systems in detection capabilities.

2.2.3 Multi-transducer Alignment

The use of the eight transducer fixture requires aiming the transducer so that all eight beams are focused at or near the same location. This is not difficult for the beams parallel or perpendicular to the axis of the tube, but the beams at 45° are obliquely incident on a curved surface and it is not immediately obvious what the proper angles are. The solution of the general problem is presented here, because we have not seen it elsewhere.

When sound passes from one material into another it obeys Snell's law, given in equation 3. The beam propagates in water with a velocity of V_w at an angle α , and in ceramic with velocity V_c at angle β .

$$(\sin \alpha) / V_w = (\sin \beta) / V_c \quad (3)$$

These angles are measured from the surface normal, in the plane of refraction. The problem of aiming an ultrasonic beam through a curved surface at an arbitrary angle from the axis requires generalizing the expression of Snell's Law to the geometry of the problem. Figure 2-5 shows the geometry and the result of the following description.

The cross product of two vectors ($\mathbf{s} \times \mathbf{n}$) is a vector with magnitude $|\mathbf{s}| |\mathbf{n}| \sin \delta$, where δ is the angle between them, and the cross product's direction is perpendicular to the plane they form. If $\mathbf{s}$ and $\mathbf{n}$ are unit vectors along the ray path and normal to the surface, the magnitude of their cross product is $\sin \beta$, one of the angles for Snell's law. Taking another cross product with the surface normal unit vector to form $\mathbf{n} \times (\mathbf{s} \times \mathbf{n})$ produces a vector parallel to the surface, in the plane of refraction, and of magnitude $\sin \beta$. Using Snell's law, $\sin \alpha = V_w \sin \beta / V_c$, we form the vector

$$\mathbf{p} = \mathbf{n} \times (\mathbf{s} \times \mathbf{n}) V_w / V_c \quad (4)$$

Snell's Law: $\frac{\sin \alpha}{V_w} = \frac{\sin \beta}{V_c}$

$$|(\bar{s} \times \bar{n})| = \sin \beta$$

$$\bar{p} = \frac{V_w}{V_c} \bar{n} \times (\bar{s} \times \bar{n})$$

$$\bar{r} = \bar{p} + (1 - \bar{p} \cdot \bar{p})^{1/2} \bar{n}$$

Figure 2-5: Generalized Expression of Snell's Law

This vector is the surface-parallel component of the refraction unit vector $\mathbf{r}$, with magnitude $\sin\alpha$. The surface normal component will have magnitude $\cos\alpha$, parallel to $\mathbf{n}$. Using the relation $\sin^2\alpha + \cos^2\alpha = 1$, or $\cos\alpha = (1 - \sin^2\alpha)^{1/2}$, we get

$$\mathbf{r} = \mathbf{p} + (1 - \mathbf{p} \cdot \mathbf{p})^{1/2} \mathbf{n} \quad (5)$$

With this expression of Snell's law, we can solve the beam aiming problem by the following procedure. Figure 2-6 shows the general geometry of the problem. The point where all the rays intersect is the origin. The tube axis is parallel to the X axis. The Z axis is the axis of a cone. All eight beams lie on this cone, whose opening angle is the angle of incidence of the rays in the ceramic.

The first step is to find the points on the surface where the beams hit the water-ceramic interface. The equation for the cone is

$$X^2 + Y^2 = (Z \tan\theta)^2 \quad (1)$$

where θ is the cone angle. The equation for the cylinder of radius R with the origin offset from the center in the Z direction by a distance d is

$$Y^2 + (Z + d)^2 = R^2 \quad (2)$$

In equation (1), express X in terms of Y, i.e. $X=Y$ for beam directions at 45° in the X-Y plane. Solve for Y^2 and substitute into (2), giving a quadratic equation in Z. Solve numerically and substitute back into (1) to get the X and Y coordinates.

Given the coordinates X,Y,Z of the point where the beam intersects the surface, the surface normal unit vector is

$$\mathbf{n} = (0, Y/R, (Z+d)/R)$$

For a cone angle θ from the z axis, with a beam angle ϕ in the x-y plane, the unit vector $\mathbf{s}$ is given by

$$\mathbf{s} = (\cos \phi \sin \theta, \sin \phi \sin \theta, \cos \theta)$$

Cone: $X^2 + Y^2 = (Z \tan\theta)^2$

Cylinder: $Y^2 + (Z + d)^2 = R^2$

Figure 2-6: Angled Beam Geometry

Given the numerical values of n and s , and the wave velocities, substitute into the above equations to find p and then r . Once the unit vector r is determined, the ray can be traced up to the plane of the transducers. This allows calculation of the x, y, z coordinates of the transducer face and its angle.

As an aid to setting the angles, which are very critical for high angle shear waves in silicon carbide, we also calculate the rotation and displacement of the transducer holding fixture that will make each beam normal to the surface. The fixture is first put in position at the correct height over the tube. Then it is displaced to the side the distance that will make one of the transducers in the plane perpendicular to the axis be aimed directly at the axis. In this position, the transducer is adjusted for maximum wall thickness multiple echoes, and then returned to scanning position. Alignment for multiple wall thickness echos is a very sensitive means of ensuring that the beam axis is exactly correct. The same is done for the transducer on the other side. Then the fixture is rotated 90° , so that the transducers aimed along the axis are now perpendicular to it, and the procedure is repeated for them. Finally, the same is done for the other 4 transducers at 45° to the axis. Note that these beams are at 45° in the ceramic, but not in the water. The fixture rotation angles must be calculated from the r unit vector.

2.3 Computed Tomography (CT)

2.3.1 Background

One of the purposes of NDE technique development is to evaluate the effects of various defects. When a defect is detected in any material, by any means, whether ultrasonics, X-rays, or otherwise, one of the most important things to determine is its size and shape. To do this, computed tomography capability was added to an existing microfocus X-ray system.

X-ray Computed Tomography (CT) was first developed for use in medicine. Its high resolution images of internal organs have revolutionized diagnosis. More recently, CT has been developed for various industrial applications. CT gives a high resolution image of a slice through the subject, with high contrast sensitivity. No other NDE technique gives comparable data. In standard X-rays, images from all depths in the sample are superimposed on the film. This leads to difficulty in separating one indication (or defect image) from another, as multiple cracks, inclusions, and voids will be superimposed. Separation of indications can be aided by taking X-rays from different angles and observing the displacement of the indications, and this is the basic principle of CT.

CT images are formed by taking line X-ray images through the slice from many directions and mathematically reconstructing the slice through a process called filtered back-projection. B&W received a set of subroutines for the reconstruction process from the Nondestructive Testing Technology Division of Sandia National Laboratory, Albuquerque, New Mexico. They are known as the RECLBL Library of Donner Algorithms, and are written in Fortran.

The reconstructions are performed on the laboratory systems development computer. It is a Hewlett Packard 9000/320, supplemented by an Analogic AP500 array processor and a Datacube Digimax image processor. This equipment is now several years old, so a new system could easily be made both faster and less expensive.

The CT system developed for this project uses an existing radiographic system. The radiographic system has a Feinfocus microfocus X-ray source, which has been used to resolve 3 micrometer lines and 2 micrometer spaces at 100x projection magnification, and a Precise Optics image intensifier used for real time radiography. The radiographic system is used for CT by rotating the object between the source and the image intensifier. A line from the image intensifier image is used as the projection data for a fan beam CT scan, which makes this a "third generation" system. Because the tubes may be very long, they are rotated around a horizontal axis, and the line of data is vertical.

As the tube is rotated in the X-ray beam, the image is monitored by the real time radiography system. The real time image is also fed to the image processing system. The image processor is a VMEbus system controlled by the main computer.

The image processor freezes the image and passes one or more columns of data to the main computer. The main computer then signals the motor controller to rotate the tube to the next angle. Each single column X-ray image collected this way is one of the data sets used by the reconstruction program.

The first step in building the CT system was to get the hardware all working together. This involved setting up software to handle the communications between the various pieces of hardware, etc. After the system worked, the next task was to improve the quality of the images and the speed of operation. Many of the lessons learned during this phase are generally applicable to any industrial CT system.

Reconstruction time was reduced from hours to minutes by putting most of the reconstruction program and data storage in the array processor, thus eliminating, as much as possible,

calculations in the main computer and data transfers between the computer and array processor.

2.3.2 Artifacts and Image Quality

CT images are subject to artifacts, which are image features that differ from the actual cross-section of the object. Artifacts in the reconstructed images were the result of vibration of the sample, inaccuracy in specification of the center of rotation and source-to-object distance, beam hardening, and nonuniformity of the beam intensity over the image.

Vibration artifacts show up as loss of resolution and edge sharpness. They are essentially the same as the blurring in a long exposure photograph when the subject is moving. Vibrations were controlled by stiffening and damping the manipulator with additional bracing.

Errors in the location of the center of rotation and source-to-object distances cause systematic errors in the back projection process, leading to deformation of the shapes of objects. These values were determined easily by experimentation after the reconstruction time was decreased. A tungsten wire was attached to the surface of the tube, and the distance parameters were adjusted to get a circular cross-section of the magnified image.

Beam hardening is the phenomenon of the average X-ray photon energy increasing as the beam passes through material, because the lower energy photons are more strongly absorbed. This produces problems in the preprocessing of the data, before the actual reconstruction.

The input to the reconstruction algorithm from each projection is derived from measurements of X-ray intensities along a line of detectors. The input for each detector should be the integrated attenuation along the line from the source to that detector. If monoenergetic X-rays are used, the X-ray intensity drops exponentially with thickness, and the integrated attenuation is the difference between the logarithm of the intensity without the object and the logarithm of the intensity with the object present. With a broad spectrum of X-rays, photons of each energy are absorbed exponentially, but the low energy photons are absorbed more quickly than the higher energy photons. This results in strong attenuation per distance in thin layers, where attenuation of low energy photons is the predominant loss of intensity, and low attenuation per distance in thick sections, where the low energy photons have been removed and the lower attenuation of the higher energy photons predominates. As a result, if the logarithm of the measured intensity is used as input, the image shows high density for thin sections and low density for thick sections, which is known as beam hardening artifact. Because the edges and corners of an object are generally thin sections for some projection angles, but the center is a

thick section for all angles, this produces high densities at surfaces and low densities inside.

What is needed is a means of correcting the data before the filtered back-projection. The reconstruction algorithm is designed for integrated attenuation as input, which in the case of a single material is equivalent to material thickness. The solution is to generate a graph of X-ray intensity vs. material thickness. Then for any measured intensity the thickness can be read off, or, equivalently, the computer can use the equation of the curve to go from measured intensities to integrated attenuation.

In this system, it is simple to store a frame from the real time images, so a frame containing a step wedge is stored. A step wedge is simply a block of material shaped like a staircase, with different known thicknesses at each step. In this case, the wedge was a stack of bars of varying lengths cut from a tube. This provides reference attenuation, because each step thickness is known and the material is identical. The tubes being scanned present attenuation paths from 1.25 to 4.32 cm, and the step wedge used goes from 1.25 to 4.40 cm in 0.63 cm steps. The measured intensity passing through each step thickness can be used as calibration points on an intensity vs. thickness curve. A curve is fit to the steps, and then the curve is used to adjust the data.

The primary difficulty in this technique is fitting a curve to the steps. Polynomial least square fits are generally inadequate. A low order polynomial will not fit the points very well, leading to significant errors in some of the projections and producing image artifacts. A higher order polynomial can fit all the points exactly, but it may be wildly incorrect between the fitted points. What is required is a model-based curve, which is fit by adjusting parameters.

The model chosen is a double exponential. The intensity decreases exponentially with distance through the material, and the attenuation coefficient also decreases exponentially with distance. This model is not derived from theoretical analysis of the spectrum and cross-sections involved, but from a qualitative analysis of the process. Beam hardening occurs very rapidly at first, and more slowly as the beam gets harder, which is the behavior of a decaying exponential. The model contains sufficient adjustable parameters to give a very good fit to the measured points, using an equation which is constrained to have the right general shape between the points. This is an advantage over a polynomial, whose shape between the points is unconstrained by any information about the physical process being modeled.

This model is used to go from measured intensity I to material thickness T . The equation for the model is $I = (A + I_0 e^{-BF})$, with $F = (1 - e^{-CT})$. I_0 , A , B , and C are the 4 adjustable

parameters. The function F represents the integrated attenuation, which increases rapidly for thin sections and more slowly as the material gets thicker, and the e^{-BF} gives the decrease in intensity caused by the attenuation. I_0 is an overall scaling factor, and A is the offset inherent in working with a digitized video signal. The equation is programmed in a computer, with the measured actual thicknesses and intensities from the step wedge. The operator enters guesses for the adjustable parameters and the computer reports how bad the fit is (error at each point and RMS error). After a number of iterations, it is possible to get a curve which is very close to all the measured points. The values of the adjustable parameters may have little relationship to the values expected from the physical interpretation, but the important fact is the fit to the measured points by a curve constrained to be qualitatively similar to the correct curve. As with any such fit, the curve is only useful within the range of the points to which it was fit. Extrapolations will probably be incorrect. This requires that the range of thicknesses in the step wedge is greater than the range of thicknesses in the sample being scanned.

Another method of making this calculation, which does not require a wedge, can be used if the material dimensions and density are known. Using a part of known uniform density, record an X-ray image, measure the intensity at various points, and calculate the thickness of the projections. This is conceptually similar to a calibration from a wedge, but it avoids the problems of making and mounting the wedge. If many measurements are made, with small differences in thickness, the values can be fit by a stepwise linear function rather than a single curve.

The resolution of the system is primarily limited by the resolution of the real-time X-ray video display, because the pixels of this display are the individual "detectors" used as fan-beam data. The CT images are reconstructed in a 512x512 array, using one point from each line of the real-time X-ray image as data, making each pixel of the image the same size as one pixel of the data set. The entire object to be scanned must be in the image for each angle, to avoid partial scan artifacts, and there are about 480 lines in an image. As a practical matter, most features must occupy several pixels to be distinguishable. This means the resolution is about one percent of the image size in a 512 x 512 pixel image.

Figure 2-7 shows a microfocus X-ray CT scan of the cross-section of a silicon carbide tube containing several cracks. In this and all other tube CT images in this section, the tube is 75 mm (3 inch) outside diameter, with 6 to 7 mm (.025 inch) wall thickness. The slice thickness is 0.2 mm.

The narrowest cracks in figure 2-7 have been measured (by micrography) to have a width of 20

Figure 2-7: CT Image of a Cracked Tube

micrometers. The CT image is generated by computer, so the image is made of an array of squares called pixels. Each pixel has a single gray-scale value representing the shade of the image at its location. In this case the spacing of the pixels corresponds to 200 micrometers on the tube. The 20 micrometer crack is much less than one 200 micrometer pixel wide. However, in the image, the cracks appear to have a width of two pixels (as determined from the matrix of numbers which give the pixel values). This shows that two pixels is the size resolution of the image, which corresponds to a size of 400 micrometers on the tube. This illustrates the difference between resolution and detection by CT. Adjacent cracks would have to be 0.6 mm apart to be resolved, but much smaller defects may be detected. The narrow cracks are only 5% of their image area, but they provide 5% contrast over that area.

Cracks two pixels wide are easily seen because of their linear structure, but defects which are small in all dimensions can be easily missed if they are near a surface where the image intensity is changing rapidly. These are much more readily detected by a data subtraction process. In a material as uniform as a tube, two slices near each other should be nearly identical. If data are collected from two slices, and one data set is subtracted from the other, the only thing left will be the differences, such as defects.

Figure 2-8 shows such an image from a tube section that contained four notches, two inside and two outside, two longitudinal and two axial. Only two of the notches were in each image, so the subtraction produced two positive (white) and two negative (black). In the original image, the notches appear in a uniform gray background, because with the tube subtracted, the defects appear to float in space. The black background has been added to show where the tube was. The other features on the inner surface are produced by roughness, which varies over the 1 mm distance separating the slices. This type of image is very useful for the detection of discontinuities and, because density measurements are not the goal, there is the added advantage of no requirement for beam hardening correction.

Figure 2-8 is also a good demonstration of partial scan artifacts. The black notches have white areas beside them, and the white ones have black areas, because of partial scan, i.e., the reconstruction is lacking data from some angles. In some cases the partial scan artifact will result from the object rotating out of the detector area during part of the scan. In this case, they result from the notches being undetectable during part of the scan, when they are near the edge of the real-time image of the tube and the material is too thick to see them.

The partial scan artifact arises from the nature of the filtered back-projection process. To illustrate, consider an object whose image should be a single white point in a uniform gray

Figure 2-8: CT Image of EDM Notches

background. Imagine also that the object is fixed in space and the source and detectors rotate around it, as in medical CT scanners. The data sets will all have uniform values except for the one point in each that is the image of the object. That image point will be where a line from the source through the object meets the detector array.

To carry out the back-projection, the computer constructs an array of pixels corresponding to physical locations in the object space. The computer uses each data set and the known locations of the source and detectors for that set to draw lines in the image from each point in the data set back to the source (hence the name back-projection). The lines are drawn with the same value as the corresponding point in the data set. In this example, each set will produce one white line and all the rest are uniform gray lines. Where all the white lines meet in the image, there is the image of the object.

Filtering is used to eliminate the major problem with this image: the white lines are all drawn all the way across the image area. The point where they all overlap is the maximum value, but there is still a lot of extra white in the image. The images shown in this report are generated using data taken from about 400 different angles. For our imaginary one point object, this means that at the object point there are 400 lines crossing, and any circle drawn around the object point will intersect lines at 800 points. Any circle of circumference 800 pixels or less will have 1 or more lines per pixel. This is a diameter of over 250 pixels, or half the size of the image. Because of this, the lines in the unfiltered image don't show up as lines, but rather as a fog around the object point. The density of the fog drops off as the inverse of the distance, and doesn't resolve into lines. This results from the data sets which are all gray with one white point, so the filter changes the data sets to surround the white point with black, which shades off to gray with the inverse of the distance from the white point. The black around each white line balances the white from other projection angles, and the result is a clean image of the white point in a gray background. In the case of more complex objects, the process works exactly the same. The filter function for any given point has a positive weight at that point and a negative weight of decreasing magnitude for points further away. This weighting function, or filter, is convolved with the data sets before the back projection and results in an accurate image if the data are measured uniformly from all angles.

The partial scan artifact arises if an image feature is missing from some of the data sets. It may be missing because it was out of the ray paths for part of the rotation, or because it was masked by a high density inclusion, or because the material was too thick for penetration at some orientation. In any case, a white feature will have dark areas in the directions of missing data and light areas perpendicular to that direction because of the effect of the filter function on the

data which is present. And correspondingly, a black feature will have white areas in the direction of missing data.

If the goal is to get a true measure of the density of the material, these artifacts are a serious problem. However, if the goal is to locate small defects, the artifacts act like arrows pointing to the defect and make location of the defects easier.

Besides resolution and contrast, detectability is also limited by noise in the image. One source of noise is photon statistics. The arrival of photons at the detector is random. Over any equal period of time, the number detected will vary with a standard deviation equal to the square root of the average number. As the number increases, the percentage variation decreases. This noise is reduced by averaging a number of image frames, which increases the total number of photons used.

Another major source of noise is produced by the data sampling process. Figures 2-9 A through D show the same image, created by varying numbers of projections. The larger the number of projections, the smoother the image. These images confirm the rule that the number of projections should be similar to the number of rays in each projection.²

Figure 2-10 is an enlarged image of the largest pore we have found inside the tube wall. It was found by accident, because with the real time x-ray, it is impossible to tell whether a pore is at the inside surface, the outer surface, or between as this one is. On this particular scan, we were looking for an acoustic emission source. Unfortunately, this pore was on the wrong side of the tube to be the source.

Figure 2-11 shows an additional artifact suppression technique. The artifact suppressed is the burn-out of the tube edge in the real-time image: that is, because of scattering effects in the scintillator and image intensifier system, the apparent tube edge is displaced from its actual position. The result is that the image shows the tube density going to zero over a thin outer layer, and any defects in the surface appear to be outside the tube. The artifact suppression technique is to pre-process the data, extending the highest density data beyond the edge of the tube. This has the effect of making the tube appear thicker, with surface defects appearing inside. Reconstruction is carried out without beam hardening correction, which also smooths the inner wall transition and emphasizes all defects. The end result is much like the data subtraction method, without the need for a reference scan and without loss of features common to both scans.

A. 55 Projections

B. 110 Projections

C. 220 Projections

D. 440 Projections

Figure 2-9: CT Images with Varying Numbers of Projections

Figure 2-10: Large Pore located by CT

Figure 2-11: Artifact Suppression by Edge Extension

A final consideration in the reconstruction process is the dynamic range of the detectors. All the images shown here were made with 8 bit data. This is responsible for the partial scan artifact that is visible in the images as well as some of the speckle-like noise.

At the angles where the defects are seen through the thinnest material the X-ray intensity is high and the 8 bit numbers are near 256. This allows detection of changes in intensity of near 0.4% where the material cross section is thin. At the angles where the defects are seen through the thickest layers of material, the X-ray intensity is low, the measured intensity is near zero, and resolution is poor. For example, if the intensity measured as an 8 bit number is 10, the resolution is 10% of the thick material. The defects are simply not detectable and the partial scan artifact results. The measured attenuation will make sudden jumps of 10%, rather than change smoothly with thickness, and the resulting discontinuities prevent the filtering from working as intended, leading to excessive apparent noise. If the same data were taken with 14 bit resolution, the same measured point would be 640 out of a maximum range of 16384 instead of 10 of 256 and the image quality limit would be imposed by photon noise rather than data quantization.

SECTION 3

CERAMIC TESTING PROGRAM

This section describes the mechanical testing program and the NDE monitoring of the tests. The mechanical testing was performed on tube sections and C-rings. The primary purpose of the testing was to gather information on the fracture toughness and the distribution of small flaws in the material. The small flaws are strength limiting in this material; they are small in the sense that they cannot be detected by NDE techniques because their size is in the range of normal porosity and surface roughness. They must be handled probabilistically, as described in the section on modeling.

The secondary purpose of the mechanical testing program was to attempt to gather additional information on the nature of the small flaws and the changes introduced by prolonged exposure at high temperatures and loads. This effort was added because of the great scientific interest and potential for process control if these small defects could be located and characterized. The tests were designed to be relatively inexpensive additions to the on-going mechanical testing program. Unfortunately, there were no significant results from these efforts. The attempted experiments are described, with problems encountered, as a guide for possible future more sophisticated attempts.

Tube sections were first scanned with TOFAM and CT to locate unusual areas for pressure testing. During pressure tests of tube sections, AE was used to locate defect areas. Those were then rescanned with TOFAM and CT. The pre-test tube inspection NDE tasks are described in section 2. There are three additional NDE measurements used in the testing program and described here; Acoustic Emission (AE) monitoring, ultrasonic velocity, and elastic modulus.

3.1 Acoustic Emission Monitoring

3.1.1 Background

Acoustic Emission monitoring is useful to detect the sounds produced by crack growth during proof testing or operation of components. The use of multiple sensors allows the location of the source of sound by differences in the time of arrival of the sound at each sensor.

We describe in detail the methods used to monitor acoustic emission during pressure testing of

siliconized silicon carbide tube segments at room temperature, and the monitoring of C-rings of the same material at temperatures up to 1350°C.

Testing results, limitations of the techniques, and suggestions for improvement are presented.

Acoustic Emission monitoring (AE) is a method of monitoring a component to detect crack growth or other damage. The technique is most often applied during a proof test, but it may also be used for monitoring components in use.

Sensors are attached to the test sample to detect the pulse of sound waves generated by the sudden release of energy when a grain or grain boundary fractures. In steel components, the method works very well because a small defect must typically grow fairly large before failure. In monolithic ceramics the problems are greater, because the critical flaw size is smaller and there is less crack growth before failure.

If there are several sensors located where they detect sound from the same event, the source location can be found by triangulation based on the relative arrival times of the sound at the sensors. Use of source location depends on accurate high resolution timing of the pulse arrival, knowledge of the sound propagation velocity, and accurate placement of the sensors. There are various possible means of calculation, depending on the geometry of the part and the locations of the sensors. The failure of a sensor to detect a particular event, and the presence of noise which is confused with events, are the primary limitations on this kind of source location. The standard sound source used in AE calibration is the breaking of a 0.5 mm pencil lead; the pencil lead is broken by pressing it against the sample at several locations to check location accuracy.

The sensors used in this work are piezoelectric, although accelerometers or capacitive sensors may also be used. Sensors are available in a variety of sizes and frequency response ranges. They typically operate in the ultrasonic range, with peak sensitivity at frequencies of 100 kHz to 1 MHz.

If sound pulses cannot be detected at great distances, which happens with materials having high scattering or attenuation of sound, sector location may be used. Sector location simply assumes the source is near whichever sensor detected the pulse first.

3.1.2 Room Temperature Pressure Tests

Tube segments of siliconized silicon carbide were pressure tested to determine the load at which

crack growth begins. The motive was to try to detect crack growth before breaking, in order to study the kinds of flaws present in the material.

Sensors are cemented to a test sample. During a pressure test, sound emitted by the cracking of single grains of material is detected and the difference in time of arrival at the sensors allows calculation of the sound source location. A commercial AE system is used, the Dunegan 8000.

The AE sensors used are sensitive only below 1 MHz. The wavelength of sound in silicon carbide at these frequencies is longer than the cross-sectional dimensions of the test specimens, so it is not clear what modes or propagational velocities are involved. Experiments were carried out to determine the best source location algorithm.

Pencil lead breaks on a tube sample were recorded both with the Dunegan system and with a transient recorder using the same sensors. As expected, the sound arrives in several pulses, which overlap at short propagation distances. The earliest signal travels near the longitudinal wave velocity, which is 11,800 m/sec in this material. The largest amplitude signal travels at 7000 m/sec, which is near the shear wave velocity of silicon carbide. Signals are detected by the AE system when they exceed a threshold value. For sources near a sensor, this may be the fastest wave. For sources at great distances, the largest pulse will be detected. At intermediate distances, the interference of the various pulses makes the timing more unpredictable. Furthermore, the velocities along the length of the tube and around the circumference are not exactly the same, whether for the modes excited or from the difference in inside and outside diameters is not clear.

This makes source location difficult. The AE testing system uses internal proprietary source location algorithms. Because of the difficulty in specifying velocities they could not be used. Instead, the raw information on arrival times was used for our own location algorithms, in conjunction with calibration mapping.

Calibration mapping is a technique often used for such situations. A source location algorithm is selected that gives nearly reasonable values and artificial pulses (pencil lead breaks) are generated over the surface to create a map. The map shows the correspondence of calculated positions with known actual positions of the lead breaks. When real AE events are detected during testing, the calculated position is referred to the calibration map to determine the actual location.

One difficulty in source location algorithms is that the algebra for locating the sources gets

complicated. If more than three sensors are used, a timing error at one sensor can result in errors that vary depending on which way the equations were solved. This can be used to verify that the set of times measured are consistent, but then there is the question of what to do to select the correct values and ignore the bad value. The more sensors used, the worse the problem gets.

A technique that works well with multiple sensors, and improves as more sensors are added, is to use a neural network to do the mapping. The sensor timings and known locations are supplied to the network as the training set, and the network is then used to locate the sources during testing. This takes care of the major problems of source location algorithm development and mapping, and as more sensors are added the effects of errors in any one measurement are reduced. For cylindrical geometries, it works well to have three outputs from the network: the axial position and the sine and cosine of the angular position.

The actual testing was carried out using a fixture capable of pressurizing a 20 cm long tube segment. The tubes were silicon carbide, 90 mm outside diameter and 6 mm wall thickness. The pressure seals were O-rings in steel caps, sealing against aluminum rings attached to the tube segment with epoxy glue. Pressure was supplied by hydraulic fluid, pressurized by a mechanical testing machine that gave computer control over the load and rate of loading. Pressures were increased in steps of about 1 MPa, at about 10 seconds per step, and held constant to detect any AE events. All events from the silicon carbide came within a few seconds of the pressure increases, although there were also events during the holding periods. It was later determined that the events during the holding periods were located either in the pressure seal region or the epoxy seal. The central part of the sample, which had only the silicon carbide material, did not produce events during hold periods.

The sensors were attached to the tube by epoxy glue. They were mounted near the ends of the tube, near the aluminum rings, to avoid damage if the tube suddenly burst. Several of the tubes did burst. The pieces were contained by a 2 cm thick aluminum shell which completely enclosed the ceramic. The shell also provided structural support for assembling the tube and seals before testing. There was a small hole at the bottom of the shell which allowed the sensor cables to be connected to the test instrument, and also allowed oil to drain out after a section of tube burst.

3.1.3 Testing C-rings at High Temperature

Besides the tube section pressure tests, AE has been used on high temperature C-ring tests. The

technique described here has been used up to 1350°C.

The C-ring is monitored by two AE transducers for source location. With only two transducers the source location algorithm is especially simple: The difference in pulse arrival times multiplied by the velocity is the difference in source-to-sensor distances. The only complication is to arrange the system so that the point of equal distance from both sensors, where the sound arrives at both sensors simultaneously, is not in the area of interest. This avoids problems with electronic noise sources, which are detected simultaneously by all sensors.

The transducers are located outside the furnace, on waveguides about 1 meter long. The waveguides are alumina rods with two small boreholes parallel to the axis. A platinum wire runs down one borehole, around the C-ring, and back to the other end of the waveguide, where the ends of the wire are fastened together and spring loaded to keep the rod in contact with the C-ring. Platinum or Pt-Ir thermocouple wire is readily available, and is the only wire we have found that keeps some strength and resists oxidation at these temperatures. Ceramic fiber insulation and fine alumina spaghetti tube are used to prevent the platinum from touching the silicon carbide. If contact is made at high temperature the platinum will form a eutectic and melt. A small piece of glass (from a microscope slide) is placed between the rod and the C-ring, to provide a liquid couplant at high temperature. During assembly, these pieces are held together by adhesives (double sided tape and superglue) which burn off before testing temperatures are reached.

Where the alumina waveguides emerge from the furnace, they are wrapped with ceramic fiber for insulation and to keep the waveguide from rubbing on the bricks, which would cause background noise.

The furnace is electrically heated, and its power supply is a source of noise. The high current, low voltage supply apparently provides pulses of current, or switches the incoming line current quickly. In any case, it radiates noise that can be picked up by the AE system. It was necessary to use an isolation transformer for the AE system and to eliminate all ground loops associated with the sensors and preamps.

During long periods at constant load at high temperature, some AE events are detected. This doesn't happen at room temperature. It may be that this is related to creep, which is known to occur at high temperatures and loads. For long life in service, creep must be avoided.

3.2 Additional Nondestructive Testing

Two other tests were carried out in an attempt to gain more understanding of the effects of prolonged high temperature and load. They were not successful, but the experiments are described for possible future guidance.

3.2.1 Elastic Modulus

During the high temperature testing, the elastic modulus was periodically measured. This is done by measuring the stress-strain curve, using the same testing machine. During long holding times at high temperature, the load was periodically increased. Instead of simply going from one load to a higher load, the testing machine control program was modified to go back to a small load and then up to the new load. During this unload-reload cycle the stress-strain curve was recorded.

It is expected that a C-ring which has suffered creep damage will have a different elastic modulus than one that has not. The stress-strain curves show a variation from one cycle to the next, but nothing systematic. In some cases the modulus increases with time and in other cases it decreases, or varies erratically from one measurement to the next. This does not seem reasonable for the effects of creep in a C-ring. We assume the results are in error, and examined the measurement process more carefully.

The measurements taken to produce each stress-strain curve are stored in a computer file and may be analyzed at any later time. When they are plotted, the points near the high load end of the stress-strain curve form a straight line. The data are not noisy, and can be fit with a least squares tangent modulus line to give high precision. The problem is not in random errors in the data, so the errors must be systematic, and involve the testing setup.

The testing machine is designed for loads 500 to 1000 times greater than those used in this testing, so it is not likely that the strain in the machine itself is significant. However, the curves are measured several hours apart, in a system in which the C-ring and the parts in direct contact with it are at 1350°C (2462°F). It has been observed that thermal expansion in this system continues for at least two days after the operating temperature is reached in the furnace.

The strain measurement is the displacement of a ram, pushing against 1.5 m (5 feet) of steel and ceramic components linked together by both threaded and flat contact interfaces. The total displacement in measuring a stress-strain curve is only about 400 micrometers (0.010"). The

most likely source of systematic error is that the changes in modulus are occurring elsewhere in the mechanical train, possibly in a threaded joint where the continuing thermal expansion produces changes in the load distribution over the threads.

3.2.2 Ultrasonic Velocity

Ultrasonic measurements have recently been shown to be sensitive to the amount of creep in similar material³, so these measurements were also carried out on the samples before and after testing. As in the modulus testing, any effects are easily obscured by measurement errors. In the original work, measurements were taken at many points on uniformly strained material and averaged. In this case this was impossible, because the creep strain distribution on a C-ring is not uniform. The changes in velocity to be expected are on the order of 0.1%, and the variations measured from point to point vary this much. The variations are partly in timing, because 0.1 per cent of the round trip time is only 2 nsec, and partly in the inhomogeneity of the material. The exact velocity measured along any path depends on the volume fractions of silicon, silicon carbide, and porosity along that path.

3.4 Mechanical Testing

The mechanical testing in this program was planned to yield information on the critical stress intensity factor of the material, the effects of various defects, and the effects of temperature and humidity. There are several types of tests, involving pressure tests of tube sections and fracturing of C-rings.

The tubes are 7.6 cm (3 inch) outer diameter, with 0.6 cm (0.25 inch) wall thickness, made of Norton CS101K, which is made of 85% SiC. The 15% pore volume is (mostly) filled with silicon.

The tube section pressure tests were all carried out at room temperature, with AE monitoring. These tests involved several tube sections from each of ten tubes. The sections are 20 cm (8 inches) long. Each end is covered by an aluminum ring 2.5 cm (1 inch) wide epoxied in place for a pressure seal, leaving an exposed test area 15 cm (6 inches) long. The tube sections were internally pressurized with hydraulic fluid (oil). The goal of these tests was to locate active AE sites without fracturing the tube, so that later NDE and C-ring tests could be performed.

The C-rings were cut 1.27 cm (0.50 inch) wide. Rings were tested at room temperature and at 1100°C, 1230°C, and 1350°C. All tests were monitored by AE. The high temperature tests

were either at ambient humidity or room temperature saturated humidity, with air passed through a bubbler. Some of the rings had 0.25 mm (0.010 inch) deep notches on the tensile surface. Others had no notches, to allow testing of natural defects. The notch depth was chosen based on Phase 2 test results, so the rings would almost always break on the notch. Only large natural defects would cause the rings to break elsewhere.

Two of these rings broke away from the notch. One had a fracture origin in a cluster of large grains, and the other had an apparent contamination by silicon nitride and alumina. The size of both these defects was in the range 0.4 to 0.5 mm.

SECTION 4

COMPONENT LIFETIME PREDICTION

4.1 Introduction

Linear Elastic Fracture Mechanics (LEFM) models were presented in phase 2 to predict fast fracture and subcritical crack growth based on stress fields applied to naturally occurring flaws in structural ceramic materials. Comparison of these models with experimental results on C-ring and tube burst tests suggested that deterministic methods alone will not provide an adequate basis for fast fracture or slow crack growth life predictions.

The phase 3 program addressed these needs by incorporating the fast fracture models into a weakest link theory reliability framework. This was accomplished by using the NASA Lewis Research Center's CARES⁴ computer for fast fracture reliability prediction and writing an interface code (ABACARES⁵) to couple CARES with the finite element code ABAQUS⁶.

In phase 2, based on literature findings, we also found that creep damage mechanisms are expected to be life limiting for many structural ceramics in high temperature ($> 1000^{\circ}\text{C}$) service. As a result, in phase 3 we coupled continuum damage mechanics models with ABAQUS stress analysis using a creep subroutine and CARES fast fracture reliability technology to produce a life prediction tool for structural ceramic component design at high temperature. Again, the ABACARES technology developed under this contract played the major role of interfacing ABAQUS and CARES.

In addition, a method was developed to assess the fast fracture reliability of ceramic components considering both the inherent flaws that characterize the material and the occasional more severe flaws that arise from manufacturing or processing anomalies and are detectable using NDE methods developed on this contract. This method demonstrates the application of NDE results to the acceptance criteria for ceramic components.

4.2 Fracture Mechanics

Linear Elastic Fracture Mechanics (LEFM) was used in phase 2 to generate models for predicting fast fracture threshold and slow crack growth tube life. C-rings were tested in

diametral compression to failure. From these data, fracture toughness (K_{Ic}) was calculated based on standard methods reported in the literature. Results from room temperature tests show the anticipated statistical scatter in strength data, and agree quite well with other samples tested in phase 2. Values scatter from 1.9 to 3.1 MPa-m^{1/2} (2.1 to 3.4 ksi-in^{1/2}).

In phase 3, fast fracture reliability models were assembled to incorporate the probabilistic nature of the material failure at low temperature. An ABAQUS to CARES interface program was developed in order to predict fast fracture reliability of ceramic materials. This combination of programs was used to predict the failure in the tube pressure tests, and is sufficiently versatile to predict the reliability of more complex monolithic ceramic structures in the future. The CARES (Ceramic Analysis and Reliability Evaluation of Structures) program was developed by NASA to predict fast fracture failure probability of ceramic structures. It uses the results of finite element analysis (NASTRAN) coupled with two-parameter Weibull statistics. An ABAQUS to CARES interface (ABACARES) was developed on this DOE project to allow fast fracture failure probabilities to be calculated based on ABAQUS finite element results.

The ABACARES interface was developed in FORTRAN on a Solbourne 5/603 UNIX workstation using SunUNIX version 4.1A and ABAQUS version 4.9. This development was based on the following work plan:

1. Port CARES to the Solbourne (UNIX).
2. Execute the Example problems for program verification.
3. Identify finite element input required by CARES.
4. Identify available ABAQUS output required by CARES.
5. Write interface program.
6. Execute the Example problems using ABAQUS/ABACARES/CARES, for verification.
7. Write an ABACARES users manual.

During this development, we kept a continual dialogue with NASA, in order to produce an efficient package which conforms with the current and future releases of CARES. We were informed of NASA's plans for a major modification of the CARES program which corresponded with our timing. Based on this input, we created the ABACARES interface to conform with the new version (CARES3).

The CARES3 program was successfully ported and tested on the Solbourne computer. Test verification runs compared well with the results from the CARES user manual.

CARES requires material identification, stress, volume or area, and temperature data from a finite element analysis. ABAQUS provides stress and temperature data directly. Material identification data can be obtained by grouping the ABAQUS output of similar materials with element sets. Element volumes/areas were not readily available from ABAQUS.

A major effort of the ABACARES interface program was to provide element volumes for the CARES program. Three separate techniques were pursued in order to obtain elemental volumes (and areas). They are highlighted below:

1. Strain Energy & Strain Energy Density Method

Initially considered to be the course of action, this technique does not work. Accurate volumes are only provided for elements of regular size or of constant strain energy density.

This method assumes that element volumes may be calculated using the strain energy and strain energy density as follows:

$$V = E / S$$

where:

V	Element volume
E	Total strain energy for the element
S	Strain energy density for the element

Strain energy and strain energy density are available directly from ABAQUS; however, they are not calculated for the same volume of material. Strain energy density is an integration point variable, while strain energy is a whole element variable. ABAQUS calculates strain energy for the whole element by summing the product of the strain energy and volume for each integration point of the element:

$$E = \sum (V_i * S_i)$$

where:

E	Total strain energy for the element
V _i	Integration point volume
S _i	Integration point strain energy density

This equation reduces to the form $V = E / S$ for only two special cases. First, that each of the integration point volumes are equal, or second that each of the integration point strain energy densities are equal. Thus, the ABAQUS output variables for strain energy and strain energy density may only be used for volume calculations under very specific circumstances, and would impose severe restrictions on FEA modeling. Because of these restrictions, an alternate method of obtaining element volumes was sought.

2. Direct Calculations (Based on NASA routines)

A set of routines were available from NASA to calculate elemental and integration point volumes based on nodal connectivity and coordinates. These routines are based on Jacobian Determinant methods, and are part of the NASTRAN to CARES interface which is provided in CARES.

3. Direct ABAQUS Output (ABAQUS Program Modification)

Ideally, volume data would be directly obtainable from ABAQUS. A letter was sent to Ian Stephenson of Hibbitt, Karlsson, & Sorensen, Inc. (HKS) requesting that volume data for subelements (integration points) be made available as output. This request was received favorably, and they proceeded with this development for ABAQUS version 5.2. However, their timetable did not agree with our schedule.

Of these three techniques, the ability to obtain subelement volumes directly from ABAQUS is most desirable for the ABACARES interface project. First of all, the strain energy density method does not work. Secondly, reduced computation time and an increased number of supported elements, over those supplied by direct calculation methods would result. Finally, the support of volume calculations will be placed upon HKS, not the ABACARES interface.

Because volume data was not available from ABAQUS in a timely fashion, we directly calculated volumes based on the NASA routines. However, it is strongly suggested that the ABACARES program be modified to incorporate the element volume output which is now available in ABAQUS version 5.2. This program revision would provide increased element support. Changes will be minimal, as ABAQUS was developed with this feature in mind.

4.3 Life Prediction Model

4.3.1 Background

At low temperature, fast fracture reliability is a structural integrity issue, but at higher temperature, time dependent deformation and damage of structural ceramics may become the dominant concern. Wiederhorn⁷ has shown that at high temperature, two phase structural ceramics exhibit a time dependent "creep" damage behavior that eventually leads to structural failure. It is the high temperature time dependent behavior that is the subject of this Section.

The approach to developing the life prediction model combines finite element analysis (FEA) stress predictions considering creep behavior, with continuum damage mechanics, and Weibull reliability statistics. The FEA program ABAQUS⁶ is used to predict the time dependent stress states throughout the component considering the redistribution of stress resulting from creep behavior. A continuity parameter is then calculated at each time step following continuum damage mechanics methods⁸. Finally, Weibull statistics are applied to an "effective" stress that is based on the continuity of the material as damage progresses. These statistics are used to predict the reliability at each time step, through the use of the NASA-Lewis computer program CARES, interfaced to ABAQUS with ABACARES.

The continuum damage modeling is basically the same as reported by NASA Lewis⁸ which is a one dimensional analysis. In task 3, this work was extended to two-dimensional cases so it can be applied to tubular components that are commonly required in the power generation and process industries. This extension to two-dimensions was accomplished by representing the two-dimensional state of stress in the tubular component by the Tresca stress⁹.

A methodology for high temperature life prediction based on creep rupture will be described, and an application will be presented. As with most developing materials in structural ceramics, the experimental database is limited. There has been some work in characterizing creep and creep rupture for two phase structural ceramics⁷, and other work in the open literature presents Weibull parameters at ambient temperature¹⁰. However, the method presented here requires further application in order to assess the appropriateness of the approach and the validity of the continuum damage and statistical failure assumptions.

4.3.2 Analytical Approach

The analytical approach is summarized in Figure 4-1. As shown, a creep analysis is performed using the finite element code ABAQUS. The steady state thermo-mechanical load is applied and the analysis is performed for a prescribed number of time steps. The stress is redistributed based on the resulting creep strain at the end of each step in the creep analysis. A state variable (continuity) is then defined to quantify the damage state⁸ of the ceramic material throughout the component volume. This continuity parameter is calculated through the application of a power law that identifies the rate of damage. The power law is extended to two dimensional problems by using the stress state from the creep analysis, and an effective stress is then calculated based on the continuity of the material as the damage progresses. Finally, a Weibull analysis is performed on the effective stress using the CARES⁴ code to predict the fast fracture reliability of each element in the model at the end of each time step. This Weibull procedure assumes that the developing crack growth or flaw enlargement does not significantly change the Weibull parameters. For this analysis, CARES is interfaced with ABAQUS through the use of a modified version of ABACARES⁵ which computes the effective stress and accounts for time dependent reliability⁹.

The heat exchanger tubes being evaluated in this DOE project were made of a slip cast siliconized silicon carbide material. This is a two phase ceramic similar to the Coors RBSC 210 evaluated in the NIST studies⁷, so the data from that report will be used in an analysis demonstration.

Creep Analysis

Industrial ceramic components in service at high temperature can experience creep⁷. The stress field in these components may re-distribute as a result of creep deformations, and this complicates the life prediction calculations. To accommodate this behavior, the life prediction methodology begins with a creep analysis which uses the creep deformations to redistribute the stress in the component before the damage calculations are made for the current time step. Explicit integration methods are used in ABAQUS to determine the stress redistribution over the current time step in the creep analysis. Due to the relatively small inelastic strains involved, this technique was found to be quite stable and convergent.

Figure 4-1: Analytic Approach to Creep Model

To represent the creep behavior, a power law is used in an ABAQUS user subroutine to express steady-state creep strain rate as a function of stress, i.e.,

$$\dot{\epsilon} = c_0 \sigma^m \quad (4.1)$$

Wiederhorn's test results⁷ demonstrate that the creep behavior (strain rate vs. stress) is piecewise continuous as shown in Figure 4-2. From the figure, it can be seen that the creep constants c_0 and m for the siliconized silicon carbide material can have two different values in compression and another value in tension. These variations in creep behavior are handled in the ABAQUS user subroutine by identifying the stress regime in each element and selecting the appropriate values from an input table of creep constants. To determine whether the two-dimensional state of stress is in tension or compression, the normal principle stresses are broken down into two sets - the "hydrostatic" (or dilatoric) component¹¹ and the deviatoric. The sign of the hydrostatic stress then determines whether the element is in tension or compression.

The material constants c_0 and m are determined as described above, the thermo-mechanical loads are applied and creep behavior is calculated by ABAQUS for a specified number of time steps. ABAQUS redistributes the stress at the end of each time step based on the calculated creep strain. The results of the creep analysis are then temperature, volume, creep strain and the redistributed stress for each element at each time step.

Damage Calculation

It is assumed that the evolution of the micro defects represents an irreversible thermodynamic process⁸. On the continuum level this requires the introduction of an internal state variable that serves as a measure of accumulated damage. In a one dimensional (uniaxial) case, let A_0 represent the cross sectional area in an undamaged state. Then denote A as the current cross sectional area in a damaged state where material defects exist in the cross section. A state variable, continuity, can then be defined to quantify the damage state as follows:

$$\psi = A/A_0 \quad (4.2)$$

SILICONIZED SILICON CARBIDE CREEP AT 1300C

Figure 4-2: Strain Rate vs. Stress (From Reference 7)

In our two dimensional case, we consider A_0 to represent the elemental area carrying the load in the undamaged state, and A the elemental area carrying the load in the damaged state. With the definition in Equation 4.2, the continuity is 1 in the undamaged state, and 0 at failure.

For this time dependent analysis, the rate of change of continuity must be specified. The following power law for one dimensional problems was chosen⁵:

$$\dot{\psi} = -B(\sigma/\psi)^n \quad (4.3)$$

The power law is extended to two dimensional problems by representing the stress in Equation (4.3) as the Tresca stress from the creep analysis. The incremental reduction in continuity as time progresses from t_{i-1} to t_i for one time step, is determined by integrating Equation (4.3):

$$\int_{\psi_{i-1}}^{\psi_i} \psi^n d\psi = -B \sigma_i^n \int_{t_{i-1}}^{t_i} dt \quad (4.4)$$

resulting in the following expression for continuity after time step i :

$$\psi_i = [\psi_{i-1}^{n+1} - B\sigma_i^n(n+1)(t_i - t_{i-1})]^{1/(n+1)} \quad (4.5)$$

From Equation (4.5), the continuity can now be calculated for each element for each time step, assuming element stresses to be constant over the time step.

Fast Fracture Reliability

Monolithic ceramic materials exhibit an inherent scatter in strength that can be suitably characterized by the weakest link theory using Weibull statistics⁸. Using Weibull methods, the reliability of a component is⁸:

$$R_i = \exp \left\{ -V_i \left[\frac{\sigma_{i,eff}}{\beta} \right]^\alpha \right\} \quad (4.6)$$

Where $\sigma_{i,eff} = \sigma_i / \psi_i$,

σ_i is the stress for element i,
 ψ_i is the continuity of element i,
 $\sigma_{i,eff}$ is the effective stress, increased by the damage,
 V_i is the volume of element i,
 α is the Weibull modulus, and
 β is the Weibull scale parameter.

Finally, using Equation (4.6), the fast fracture reliability of a component can be calculated for each element at each time step as the product of all the element reliabilities. A modified Weibull based treatment of the failure probability for these cases with non-monolithic increasing stress has been accounted for based on Reference 12.

4.3.3 Demonstration of the Methodology

There is limited data available on creep behavior of two phase ceramic materials. Some of the most appropriate information available is the creep and creep rupture data from tensile coupon specimens developed by NIST for the Coors RBSC 210 material⁷. This data will be used to characterize the creep and damage behavior of the material, and the Weibull parameters will be estimated from the literature¹⁰. This database is limited, and the database of component applications where creep has been observed or measured is even more limited. Due to this lack of component structural performance data, a preliminary demonstration of the methodology will be presented using a simple C-ring test performed at high temperature during phase 2.

Fracture toughness tests were performed on C-rings, but an assessment of the time dependent behavior of the material was beyond the scope of the project. As a result, the elaborate test set up and measurements required to accurately record creep behavior were not performed. However, for our demonstration case, we will make predictions of the C-ring behavior measured in phase 2 testing using the creep/continuum damage methodology described above, and demonstrate that the predictions are consistent with the observed C-ring Behavior. This is not an ideal test case since the measurements in the C-ring tests were not refined enough to observe time dependent behavior, but it will serve to demonstrate the method and give confidence in the predictions. The criteria used for comparison with test data was the computed reliability during

the loading history. Rapid reductions in computed reliability would be used to predict failure conditions.

C-ring Test Conditions

The C-ring was a 3" (76.2 mm) OD, 2.5" (63.5 mm) ID ring that was 0.5" (12.7 mm) wide with a .010" (0.254 mm) deep flaw machined on the O.D. as shown in Figure 4-3. It was tested at a uniform temperature of 1350°C. The C-ring was heated to temperature gradually so the thermal stresses could be neglected in the analysis and only the steady mechanical diametral load would need to be considered. The load was applied in increments according to the following schedule (Table 4.1).

TABLE 4.1 -- Load Schedule

Load	Time at load (hrs)
46 lb (205 N)	18
51 lb (227 N)	6
56 lb (249 N)	17
61 lb (271 N)	5
66 lb (294 N)	14
70 lb (311 N)	3.5
77 lb (343 N)	2
84 lb (374 N)	1 (slow steps until break at 84 lb)

Material Creep Properties

The creep behavior of the material is represented by the data in Figure 4-2. As shown in the figure, the behavior is different in tension than in compression. This behavior was modeled as a piecewise continuous function in ABAQUS's user Creep subroutine. To curve fit the tension data for the Coors RBSC 210 material we assume a power law for the creep behavior as follows:

Tested C-Ring with .010" (0.254 mm) Seeded Flaw

Figure 4-3: C-Ring Configuration

$$\dot{\epsilon} = c_o \sigma^m \quad (4.1)$$

Now, we can determine m as the slope of the plot of $\ln \dot{\epsilon}$ vs. $\ln \sigma$ for RBSC 210 in Figure 4-2. This gives a value of:

$$m = 7.43 \quad (\text{dimensionless})$$

From equation 4.1 and the curve in Figure 4-2, we can determine the corresponding value of c_o to be:

$$c_o = 9.8E-21 \quad \left[\frac{1}{hr \cdot MPa^m} \right]$$

A similar procedure was then used to fit the two sections of the compressive data.

Material Damage Properties

The material damage parameters were defined by the power law in Equation 4.3 that relates the rate of change of continuity to the effective stress. The constants can be evaluated by integrating Equation 4.3 and recognizing that there is complete continuity at the beginning of the analysis before any damage has taken place.

The constants B and n in Equation 4.3 are determined as follows. Integrate Equation (4.3) from 0 to t and observe that at $t = 0$, $\psi = 1$.

$$\int_1^{\psi} \psi^n d\psi = -B \sigma_i^n \int_0^t dt \quad (4.7)$$

We obtain the following expression for continuity:

$$\psi = [1 - B \sigma_i^n (n+1) t]^{1/(n+1)} \quad (4.8)$$

Now, find the time to failure by setting $\psi = 0$ in Equation (4.8).

$$t_f = [B\sigma_i^n (n+1)]^{-1} \quad (4.9)$$

Taking the natural log of Equation (4.9), we can obtain the values of B and n for Coors RBSC 210 from Figure 4-4, which is a plot of $\ln(\text{applied stress})$ vs. $\ln(\text{failure time})$ ⁷.

$$\ln \sigma_i = -1/n \left\{ \ln t_f - \ln[B(n+1)] \right\} \quad (4.10)$$

The slope of the line in Figure 4-4 is $-1/n$. This results in the following value of n:

$$n = 10.08 \text{ (dimensionless)}$$

Now, from Equation (4.9) and the curve in Figure 4-4:

$$B = 1.54E-24 \left[\frac{1}{\text{hr} \cdot \text{MPa}^n} \right]$$

Weibull Parameters

Statistical fracture properties of two grades of siliconized silicon carbide material were determined¹⁰ at ambient temperature at EG&G Idaho as part of a program of evaluating structural ceramics with tubular geometries for heat exchanger applications. These results will be used to estimate the Weibull parameters for this calculation. The two materials are Norton's NC-430, and Coors SC-AXI. For our analysis we will assume our material (Coors RBSC 210) is closer to the Coors SC-AXI and use the appropriate Weibull parameters α and β (Equation 4.6). Reference 10 reports ambient temperature C-ring test data from which siliconized SiC Weibull parameters can be estimated. The general expression for reliability of a component having a varying stress field like a C-ring is as follows.

$$R = \exp \left\{ - \int_V (\sigma/\beta)^\alpha dV \right\} \quad (4.11)$$

Figure 4-4: Applied Stress vs. Failure Time (From Reference 7)

Using the Reference 10 data and Equation 4.11, the following Weibull parameters were determined:

$$\alpha = 15.25 \text{ (dimensionless)}$$
$$\beta = 236.8 \text{ (MPa} \cdot \text{(mm)}^{3/\alpha} \text{)}$$

During the course of making these calculations, we learned that Jadaan¹³ has published high-temperature Weibull parameters for Coors RBSC 210. The use of these material properties provides better results as shown in Reference 9.

Summary of Material Properties

Table 4.2 is a summary of the material properties used in the C-ring test analysis.

TABLE 4.2 -- Material Properties

PROPERTY		UNITS
Elastic	$E = 255E+03$	MPa
	$\mu = .3$	dimensionless
Creep	$m = 27.67 \quad \sigma < -240\text{MPa}$ $C_o = 5.29E-71$	$m = \text{dimensionless}$
	$m = 3.74 \quad -240\text{MPa} \leq \sigma \leq 0\text{MPa}$ $C_o = 4.28E-14$	$C_o = \frac{1}{\text{hr MPa}^m}$
	$m = 7.43 \quad \sigma > 0\text{MPa}$ $C_o = 9.80E-21$	
Continuity	$n = 10.08$	dimensionless
	$B = 1.54E-24$	$\frac{1}{\text{hr MPa}^n}$
Weibull	$\alpha = 15.25$	dimensionless
	$\beta = 236.8$	$\text{MPa (mm)}^{3/\alpha}$

4.3.4 Analysis Results

The finite element model of the C-ring with the .010" (0.254 mm) flaw is shown in Figure 4-5. Taking advantage of symmetry, one quarter of the C-ring was modeled and the load was applied as shown. The figure shows an isometric view of the model and a close-up of the area around the flaw. An analysis was performed using the material properties and load schedule defined above. Three analyses were run to characterize the failure behavior of the C-ring test.

Figure 4-5: Finite Element Model of C-Ring with 0.010" Flaw (Quarter Symmetry)

Fast Fracture Reliability Without Creep

Fast fracture reliability was assessed with ABAQUS and CARES without considering creep or continuum damage. Reliability was plotted vs. applied load in Figure 4-6 for the C-ring with the .010" (0.254 mm) flaw compared with the same C-ring with no flaw. The test C-ring failed at an 84 lb (374 N) load after following the load schedule described above. The results in Figure 4-6 show that the fast fracture reliability is zero for 84 lb (374 N) load, so a failure is expected when the 84 lb (374 N) load is applied. Two assumptions should be noted with regards to Figure 4-6. The Weibull parameters are based on ambient temperature data for a similar SiC material while the actual C-ring test was performed at a temperature of 1300°C. In addition, this fast fracture analysis prediction does not account for any stress redistribution which would be expected to increase the fast fracture reliability in the direction of the un-flawed C-ring analysis results. The actual C-ring loading was applied in steps over more than 65 hours, thus permitting stress redistributions not accounted for in this analysis.

Continuum Damage Reliability With Creep at Constant Load

This analysis examines the behavior when both creep and continuum damage are considered in the reliability calculation. Figure 4-7 is a plot of reliability vs. time for four different loads considering both stress redistribution and damage accumulation. The four loads that were chosen to cause failure in a reasonable time period were 55 lb (245 N), 60 lb (267 N), 65 lb (289 N), and 70 lb (311 N). As shown in the figure, an analysis with a 60 lb (267 N) load predicted an initial reliability of 0.93. This value slowly drops to 0.91 after 90 hours before quickly dropping to zero. Each of these curves display this trend of an initial reliability which drops sharply at failure. Additionally, Figure 4-7 shows that the time to failure is highly dependent upon the load (stress state). This observation suggests that stress redistribution, due to creep effects, are very important for accurate predictions of component life.

Reliability Prediction With the Actual Load History

For the third analysis the experimental loading conditions were applied and creep and continuum damage effects were included. This represents the complete analysis sequence being proposed in this paper. The results are shown in Figure 4-8 as a plot of reliability vs. time. A plot of fast fracture reliability with creep effects, but no continuum damage is shown for comparison, and the load vs. time sequence is also shown on the plot for reference. This figure presents

C-Ring Fast Fracture Reliability

Figure 4-6: Fast Fracture Reliability vs. Load for a C-Ring

C-Ring Continuum Damage Reliability

Figure 4-7: Continuum Damage Reliability (with Creep) vs. Time at Various Loads

C-Ring Reliability

Figure 4-8: C-Ring Analysis vs Actual Load Schedule

several interesting results.

First, it shows that the continuum damage reliability drops from 0.92 after 63.5 hours, to zero after 66 hours, when the C-ring failed. The corresponding drop in fast fracture reliability only reaches a low of 0.76. Next, in both the continuum damage and fast fracture cases, creep effects, which redistribute stresses, are evident in the reliability predictions. This effect is best illustrated when the C-ring was loaded to 70 lb (311 N) during the time from 60 hours to 63.5 hours. The continuum damage model predicts a reliability of 0.92, while fast fracture reliability is 0.98. However, if creep was not considered then the fast fracture reliability for a 70 lb (311 N) load would be 0.5 (see Figure 4-6).

4.3.5 Conclusions

A methodology is presented that predicts the reliability of monolithic structural ceramics that are subject to time dependent (creep) damage. The results of the demonstration analysis with a C-ring cut from a heat exchanger tube show that the predicted behavior is somewhat physically appealing. Accurate predictions were not anticipated in this case since a good material property database was not available for the specific material at the temperature tested, however, the results were sufficiently close to encourage the continued assessment of the method. Additional appropriate test cases need to be developed, and appropriate material properties need to be thoroughly determined to provide an adequate evaluation of the creep/continuum damage approach presented.

With refinements to better address the effects of bi-axial stress fields, and actual material properties, this methodology appears to be quite general and readily applicable to many structural ceramic components and materials. An inherent assumption which may require clarification is the notion that an effective stress can be used with undamaged material Weibull parameters to predict long term fracture reliability. The evolution and coalescence of voids and/or flaw growth in preferential directions may alter the Weibull parameters since the nature of the underlying flaw population as well as applied stress influence these parameters.

4.4 Sensitivity Study of Life Prediction Model

4.4.1 Introduction

The life prediction methodology incorporates continuum damage mechanics within the framework of a weakest link theory (Duffy⁸). For this sensitivity study, the model is further enhanced by incorporating time dependent Weibull statistics (Stanley¹²), in order to account for stress redistribution due to creep.

The life prediction model combines finite element analysis (FEA) stress predictions, considering creep behavior, with continuum damage mechanics, and time dependent Weibull reliability statistics. The FEA program ABAQUS⁶ is used to predict the time dependent stress states throughout the component. Component reliability is then calculated with the computer program LIFE. This software calculates a continuity parameter at each time step, following continuum damage mechanics methods, and utilizes CARES⁴ technology, coupled with time dependent Weibull statistics, to predict component reliability. Thus, it combines the functionality of both ABACARES⁵ and CARES, with extensions for time dependent reliability calculations.

Since creep and creep damage are not easy to measure, data bases are not readily available to assess the validity of continuum damage approaches to predict the time dependent behavior. Until such data is developed, it is prudent to study the behavior of continuum damage models and see how they compare with physical reasoning.

In the following sections, the methodology for high temperature life prediction will be described. This life prediction model, will be used to compare reliability predictions for fast fracture with the reliability associated with continuum damage. The effect of the material parameters, and load history are considered. Additionally, three representations of the multiaxial state of stress in the continuum damage model are studied. Finally, an understanding of the material behavior was obtained⁷, and a comparison of results is made with C-ring test behavior.

4.4.2 Analytical Approach

The analytical approach is the same as presented in Section 4.3 above. Some of that presentation will be repeated here to assemble the creep and damage tools in the framework needed for the sensitivity study. The approach is summarized in Figure 4-1. As shown, a creep

analysis is performed using the finite element code ABAQUS. The steady state thermo-mechanical load is applied and the analysis is performed for a prescribed time. The stress is redistributed based on the resulting creep strain at the end of each time step in the creep analysis. A state variable (continuity) is then defined to quantify the damage state of the ceramic material throughout the component volume. This continuity parameter is calculated through the application of a power law that identifies the rate of damage. Finally, a time dependent Weibull analysis is performed to predict the reliability of each element in the model at the end of each time step. The resulting component reliability is then calculated as the product of the element reliabilities. This Weibull procedure assumes that the developing crack growth or flaw enlargement does not significantly change the Weibull parameters.

Creep Analysis

Industrial ceramic components in service at high temperature can experience creep. The stress field in these components may re-distribute as a result of creep deformations, and this complicates the life prediction calculations. To accommodate this behavior, the life prediction methodology begins with a creep analysis which uses the creep deformations to redistribute the stress in the component before the damage calculations are made for the current time step. Explicit integration methods are used in ABAQUS to determine the stress redistribution over the current time step in the creep analysis. Due to the relatively small inelastic strains involved, this technique was found to be quite stable and convergent.

To represent the creep behavior, a power law is used in an ABAQUS user subroutine to express steady-state creep strain rate as a function of stress, i.e.,

$$\dot{\epsilon} = c_0 \sigma^m \quad (4.1)$$

The test results in Reference 7 demonstrate that the creep behavior (strain rate vs. stress) is piecewise continuous as shown in Figure 4-2. From the figure, it can be seen that the creep constants c_0 and m for the siliconized silicon carbide material can have two different values in compression and another value in tension. These variations in creep behavior are handled in the ABAQUS user subroutine by identifying the stress regime in each element and selecting the appropriate values from an input table of creep constants. To determine whether the state of

stress is in tension or compression, the normal principle stresses are broken down into two sets - the "hydrostatic" (or dilatoric) component¹¹ and the deviatoric. The sign of the hydrostatic stress then determines whether the element is in tension or compression.

The material constants c_0 and m are determined as described above, the thermo-mechanical loads are applied and creep behavior is calculated by ABAQUS for a specified number of time steps. ABAQUS redistributes the stress at the end of each time step based on the calculated creep strain. The results of the creep analysis are then temperature, volume, creep strain and the redistributed stress for each element at each time step.

Damage Calculation

It is assumed that the evolution of the micro defects represents an irreversible thermodynamic process⁸. On the continuum level this requires the introduction of an internal state variable that serves as a measure of accumulated damage. Let A_0 represent the cross sectional area in an undamaged state. Then denote A as the current cross sectional area in a damaged state where material defects exist in the cross section. A state variable, continuity, can then be defined to quantify the damage state as follows:

$$\psi = A/A_0 \quad (4.2)$$

With the definition in Equation 4.2, the continuity is 1 in the undamaged state, and 0 at failure.

For this time dependent analysis, the rate of change of continuity must be specified. Based on stress rupture data (see Figure 4-4), the following power law for uniaxial states of stress was chosen⁸:

$$\dot{\psi} = -B(\sigma/\psi)^n \quad (4.3)$$

In order for the continuum damage model to be useful as a design tool, it has to be generalized from the uniaxial model, to a multiaxial model. This generalization was accomplished by partitioning the three dimensional stress space into specific regions where a given stress state either causes damage to evolve or does not. Three methods of partitioning the stress space were

examined. These methods employed the von Mises, the Maximum Normal, and the Maximum Shear failure theories.

Because ceramics tend to be much stronger in compression than tension, only tensile stresses were considered to contribute to damage, and subsequent component reliability. Thus for compressive stresses, the representative stress in Equation (4.3) was set to zero. This consideration complicates the calculation of the representative stress in each method. The following technique was used to determine the representative stress for the three cases considered:

<u>Case</u>	<u>Failure Theory Basis</u>	<u>Representative Stress</u>
1	von Mises	$\begin{array}{l l} \sigma = \sigma_{\text{Mises}} & \sigma_{\text{hydro}} > 0 \\ \sigma = 0 & \sigma_{\text{hydro}} \leq 0 \end{array}$
2	Maximum Normal Stress	$\begin{array}{l l} \sigma = P_1 & P_1 > 0 \\ \sigma = 0 & P_1 \leq 0 \end{array}$
3	Max Shear Stress	$\begin{array}{l l} \sigma = P_1 - P_3 & P_1 > P_3 \\ \sigma = 0 & P_1 \leq P_3 \end{array}$

Where:

- σ is the representative stress
- σ_{Mises} is the von Mises stress
- σ_{hydro} is the hydrostatic stress
- P_1 is the maximum principle stress
- P_3 is the minimum principle stress

Once the stress is known, the incremental reduction in continuity as time progresses from t_{i-1} to t_i for one time step, is determined by integrating Equation (4.3):

$$\int_{\psi_{i-1}}^{\psi_i} \psi^n d\psi = -B \sigma^n \int_{t_{i-1}}^{t_i} dt \quad (4.4)$$

resulting in the following expression for continuity after time step i:

$$\psi_i = [\psi_{i-1}^{n+1} - B\sigma^n(n+1)(t_i - t_{i-1})]^{1/(n+1)} \quad (4.5)$$

From Equation (4.5), the continuity can now be calculated for each element and for each time step. This equation assumes that element stresses are constant over the time step, which is not the case when creep is considered. As a first approximation, the stress used in Equation (4.5) is the average stress for the current time step.

Continuum Damage Reliability

Monolithic ceramic materials exhibit an inherent scatter in strength that can be suitably characterized by the weakest link theory using Weibull statistics. Using PIA methods, the reliability of element i (R_i) a component, with consideration to continuum damage⁸, is:

$$R_i = \exp \left\{ -V_i \left[\left(\frac{P_{1,i}}{\psi_i \beta} \right)^\alpha + \left(\frac{P_{2,i}}{\psi_i \beta} \right)^\alpha + \left(\frac{P_{3,i}}{\psi_i \beta} \right)^\alpha \right] \right\} \quad (4.12)$$

Where: $P_{x,i}$ are the principle (tensile) stresses for element i ,
 ψ_i is the continuity of element i ,
 V_i is the volume of element i ,
 α is the Weibull modulus, and
 β is the Weibull scale parameter.

For time dependent Weibull analysis this expression must also consider the load history of the component¹². The element reliability at time t is calculated to be the minimum element reliability for all time steps up to t .

Finally, the component reliability at time t is calculated as the product of the element reliabilities at time t .

$$R_c(t) = \prod R_i(t) \quad (4.13)$$

4.4.3 Model Behavior and Implications

The life prediction model will be used to compare reliability predictions for fast fracture with the reliability associated with continuum damage. The effect of the material parameters, load history, etc., are considered. Two basic geometries were studied. The first geometry is that of a unit volume component under uniaxial loading. This simple geometry was used to examine the effect of varying the continuity parameters on life predictions. The second geometry is that of the C-Ring test specimen with a full width flaw, or notch (see Figure 4-3).

Material Properties

The components being evaluated in this project were made of a slip cast siliconized silicon carbide material. This is a two phase ceramic similar to Coors RBSC 210. Table 4.3 lists the material properties used in this demonstration analysis. Young's modulus was measured in phase 2, while the creep response of Coors RBSC 210 was measured by NIST⁷. High temperature Weibull material properties for Coors RBSC 210 were provided by Jadaan¹³. Finally, the continuity data was determined from the NIST measurements, see Figure 4-4.

Table 4.3 Material Properties at 1300°C

PROPERTY		UNITS
Elastic	$E = 255E+03$	MPa
	$\mu = .3$	dimensionless
Creep	$m = 27.67 \quad \sigma < -240\text{MPa}$ $C_o = 5.29E-71$	$m = \text{dimensionless}$
	$m = 3.74 \quad -240\text{MPa} \leq \sigma \leq 0\text{MPa}$ $C_o = 4.28E-14$	$C_o = \frac{1}{\text{hr MPa}^m}$
	$m = 7.43 \quad \sigma > 0\text{MPa}$ $C_o = 9.80E-21$	
Continuity	$n = 10.08$	dimensionless
	$B = 1.54E-24$	$\frac{1}{\text{hr MPa}^n}$
Weibull Parameters	$\alpha = 5.50$	dimensionless
	$\beta = 721.0$	$\text{MPa (mm)}^{3/\alpha}$

Uniaxial Loading

Consider a unit volume tensile specimen subjected to a constant uniaxial stress. Under these ideal conditions the effect of the continuity parameters may be studied directly. Using the mathematical software program MathCad¹⁴, the parameters n and B were varied over a range of stresses in order to study their influence on time to failure, continuity, and component reliability. An understanding of the behavior of the continuum damage model under uniaxial

loading will serve as a foundation as this model is extended into multiaxial stress states.

For the case of a uniaxial stress under a constant load, equation 4.5 may be simplified to determine the time to failure. By noting that continuity is initially one, and is zero at failure, the time to failure may be determined as follows:

$$t_f = \frac{1}{B\sigma^n(n+1)} \quad (4.14)$$

Figures 4-9 and 4-10 plot the time to failure versus stress for various values of n and B . In Figure 4-9 the parameter B is held constant and n is varied from 8 to 12. Similarly, Figure 4-10 holds the value of n constant, and varies B from 5×10^{-25} to 5×10^{-23} . It is evident from Figure 4-9 that the parameter n has a dramatic influence on the time to failure versus stress curve. The parameter B , however, has less effect on the curve.

Another effect which may be observed in Figures 4-9 and 4-10 is the dependency on time to failure versus stress. This effect is also evident in Figure 4-11 which plots continuity (ψ), as a function of time for various levels of stress. This figure shows that the continuity progresses from the undamaged state ($\psi=1$) to the failed state ($\psi=0$) very rapidly. Figure 4-11 also shows that the time to failure is highly dependent upon the stress.

Figure 4-12 is a plot of component reliability versus time for various levels of stress. This plot shows that the reliability is very near one, until failure. By comparing this figure with Figure 4-11 (continuity vs. time), it is evident that the continuity parameter is dominating the reliability equation. The rapid loss in reliability (failure) coincides with the loss in continuity. As seen in Figure 4-12, the time dependent reliability is also highly dependent upon stress. This dependency is due to the effect of the continuity parameter.

Multiaxial Loading

The C-Ring test specimen depicted in Figure 4-3 was used as an example of multiaxial loading. Tested under this DOE flaw assessment program, the sample was subjected to a non-uniform loading history, experienced creep deformation, and contained a full width notch. Additionally, creep rate, creep rupture, and Weibull parameter data was available for this material at the tested

Failure Time vs Stress

$B=1.54E-24$

Uniaxial Loading

Figure 4-9: Failure Time vs. Stress, Various n

Failure Time vs Stress

$n=10.08$

Uniaxial Loading

Figure 4-10: Failure Time vs. Stress, Various B

Continuity vs Time

Uniaxial Loading

Figure 4-11: Continuity vs. Time at Various Stress Levels

Reliability vs Time

Uniaxial Loading

Figure 4-12: Component Reliability vs. Time at Various Stress Levels

temperature of 1300°C. This combination of complex geometry and loading, coupled with a reasonable amount of test data, makes this test specimen a good candidate for model benchmarking.

The finite element program ABAQUS was used to model the C-Ring. Geometric and loading symmetries allowed one quarter of the C-Ring to be modeled with 629 C3D6 and C3D8 elements. Figure 4-13 shows the mesh used for this analysis.

The load history of the specimen is presented in Table 4.4. Except for the last step, the load was applied rapidly at the beginning of each step. During the last step, the load was ramped from 77lb to 84lb over a 1 hour period, when the C-Ring breaks at the 84lb load. For modeling purposes, the 84lb load was then held for an additional 20 hours.

Table 4.4 C-Ring Load History

Step	Load	Time at load (hrs)	Load Type
1	46 lb (205 N)	18	Step
2	51 lb (227 N)	6	Step
3	56 lb (249 N)	17	Step
4	61 lb (271 N)	5	Step
5	66 lb (294 N)	14	Step
6	70 lb (311 N)	3.5	Step
7	77 lb (343 N)	2	Step
8	84 lb (374 N)	1	Ramp (breaks at 84lb)
9	84 lb (374 N)	20	Hold

Figure 4-13: C-Ring Finite Element Model (Symetric Elements not Shown)

Once the stress state versus time was determined for the C-Ring, the reliability versus time was calculated using CARES technology interfacing with ABAQUS with ABACARES technology developed on this contract. First, a fast fracture reliability was calculated (no continuum damage). Then, the continuum damage reliability was calculated based on three separate representations of the multiaxial stress state. Finally, the reliability predictions for these different techniques are compared.

Figure 4-14 is a plot of reliability versus time for the C-Ring. This plot illustrates five separate curves. First, the load history of the C-Ring is represented by the Load curve. This curve shows how the load was applied throughout the test. The arrow pointing to the "Failure" point highlights the point on the Load curve where the actual test specimen broke. A second curve shows the fast fracture reliability versus time. This curve represents the component reliability without consideration for continuum damage. At 80 hours the fast fracture reliability is calculated to be 0.977.

The last three curves in Figure 4-14 plot the continuum damage reliability versus time for the three different representations of multiaxial stress in the continuity equation. The first of these curves is the von Mises curve, and is based on the von Mises stress. This curve is nearly identical to the fast fracture reliability curve. At 80 hours the reliability is calculated to be 0.976, just below the fast fracture value. However, if the analysis were continued beyond 90 hours the von Mises curve would eventually predict failure, whereas the fast fracture curve would show little reduction in reliability. The next curve of interest is the Max Principle curve. This curve is based on the maximum principle stress. In this method the multiaxial stresses are highest. These higher stresses result in premature prediction in failure after 10 hours. In the last curve, Max Shear, the multiaxial stress is lower than the maximum principle stress, yet somewhat higher than the von Mises stress. The failure time predicted for this curve agrees very well with the experimental results.

4.4.4 Conclusions and Recommendations

The use of a continuum damage model for life prediction of structural ceramics seems promising. This methodology appears to be quite general, and readily applicable to many structural ceramic components and materials. A maximum shear stress based theory was used to extend the continuum damage model from uniaxial to multiaxial stress states. The resulting predicted time to failure for the C-Ring demonstration analysis agrees well with the experimental

Continuum Damage Reliability

for Various Failure Theories
at Actual Load Schedule

Figure 4-14: Reliability vs. Time for Various Theories at Actual Load Schedule

results. However, this is only one case, additional test cases need to be developed in order to validate this model.

Material properties for high temperature analysis are often scarce. This method uses material properties derived from common test procedures. New test procedures are not required to obtain the material property data needed for this life prediction methodology.

The study of various representations of the multiaxial stress state was limited. Only one loading condition was examined. Additional cases should be investigated. Furthermore, only the tensile portion of the multiaxial stress states were investigated. As ceramic materials exhibit different behaviors in tension versus compression, other methods of representing the multiaxial stress state should be investigated.

The effect of cyclic loading on life prediction has not been addressed. This type of loading is often present, and should be considered as part of a complete design methodology.

An inherent assumption which may require clarification is the notion that an effective stress can be used with undamaged Weibull parameters to predict long term reliability. The evolution and coalescence of voids and/or flaw growth in preferential directions may alter the Weibull parameters, since the nature of the underlying flaw population, as well as the applied stress, influence these parameters.

SECTION 5

APPLICATION OF NDE RESULTS TO THE ACCEPTANCE CRITERIA FOR CERAMIC COMPONENTS

5.1 Introduction

The use of nondestructive examination methods to characterize flaws in monolithic ceramic materials is an important aspect of the evolving methodologies for the design of structural ceramic components. The influence of the inherent material flaw populations on the reliability of structural components can be characterized with strength based models such as the principle of independent action (PIA) model, or fracture mechanics based models such as the Batdorf reliability model. However, if manufacturing or assembly procedures or processing anomalies occasionally introduce flaws that are more severe than the inherent population in the representative material, then additional measures must be taken to verify acceptability or justify rejecting the component.

A traditional approach to coping with these severe, non representative flaws is proof testing¹⁵ to weed out the weakest components through failure during testing, or to demonstrate increased allowable design strength. As NDE methods advance and become capable of detecting the flaws that are more severe than those inherent in the characteristic material, it is natural to look for other analytical methods to predict their influence on the reliability of the component.

This Section presents such an analytical approach by combining fast fracture reliability predictions of the inherent material flaws using Weibull statistics⁴ with fracture mechanics analysis of the more severe flaws detected with NDE methods. There is a statistical scatter in material toughness (Figure 5-1) and an uncertainty associated with the NDE measurement of the flaw size and location. The fracture toughness and flaw size will therefore both be treated as stochastic parameters in the linear elastic fracture mechanics analysis to produce a reliability prediction associated with the flaw. The resulting method is applicable to the fast fracture reliability prediction of monolithic ceramic components considering both the inherent flaw population and the more severe flaws that are detectable with NDE investigations.

Figure 5-1: Fracture Toughness vs. Failure Load for a Silicon Carbide

Other important design capabilities that may need to be added to the reported methodology depend on the component operating temperature and environment. Such capabilities include high temperature time dependent reliability^{16,17}, slow crack growth, creep assisted crack growth, fatigue¹⁸ and chemical degradation.

At the present time, the limits of flaw size that are detectable with state-of-the-art NDE methods¹⁹ are being improved. Great strides have been made and some work still needs to be done to economically characterize the small flaws necessary to effectively utilize methodologies that account for their individual influence. In the following presentation, it is assumed that any flaws larger than the inherent population in the material are detectable with NDE methods, and an analytical method will be described and demonstrated that combines fast fracture reliability of individual flaws with reliability methods for the normal material.

5.2 Analytical Procedure

The analytical approach to predicting the reliability associated with the inherent flaws in the material is based on methods that calculate the fast fracture reliability or failure probability of macroscopically isotropic ceramic components. It combines probabilistic fast fracture theory from the NASA Lewis CARES⁴ code with Stress analysis results from the ABAQUS⁶ finite element program. The reliability associated with an individual flaw in the component is calculated by Monte Carlo methods with the Lotus add-in program @RISK²⁰. The input to @RISK is the fracture toughness of the material and the flaw size. The fracture toughness is a stochastic parameter determined from test data. The flaw size is also assumed to be stochastic to account for the uncertainty in the NDE characterization of the flaw. Since both input variables are stochastic, the resulting critical stress prediction is stochastic. The reliability of the component can then be calculated as the probability that the critical stress calculated by @RISK is greater than the applied stress.

The reliability is calculated throughout the component for all elements in the finite element model¹⁶. Then at those locations with NDE detected individual flaws, the fast fracture reliability associated with each flaw is calculated with @RISK. The component reliability is calculated as the product of all element reliabilities¹⁶, so the component reliability associated with the individual flaws can be accounted for by multiplying the total component reliability from the inherent flaws by the product of the reliability of the individual flaws.

Inherent Flaws - Strength Based Model

Monolithic ceramic materials exhibit an inherent scatter in strength that can be suitably characterized by the weakest link theory with Weibull statistics⁸. Using Weibull methods, the reliability of an element (R_i) in the finite element model of the component is¹:

$$R_i = \exp \left\{ -V_i \left[\frac{\sigma_i}{\beta} \right]^\alpha \right\} \quad (5.1)$$

Where: σ_i is the stress for element i ,
 V_i is the volume of element i ,
 α, β are the Weibull modulus, and scale parameter.

Using Equation (5.1), the fast fracture reliability of a component (R) can be calculated as the product of all the element reliabilities.

$$R = \prod R_i \quad (5.2)$$

Individual Flaws - Fracture Mechanics Based Model

Given the fracture toughness and flaw size, the critical stress for failure, σ_c , is calculated:

$$\sigma_c = f(a) K_{IC} / \sqrt{\pi a} \quad (5.3)$$

Where $f(a)$ is dictated by flaw type and loading. To be able to simply handle both subsurface and surface flaws, two specific types of flaw models are suggested, and others may be appropriate depending on the specific situation, and the sophistication that is justified in the design analysis.

- The subsurface flaw is assumed to be circular with radius a . The flaw is assumed to be under an applied uniaxial stress which is uniform; stress gradient and multiaxial stress effects were neglected. Neglecting the stress gradient is a reasonable assumption given the small flaw sizes under consideration, and is also conservative if the uniform stress is made equal to the maximum

stress in the stress gradient. It is also conservatively assumed that the flaws were oriented perpendicular to the applied stress, with no mixed mode loading.

- The surface flaw is assumed to be semicircular with radius a . The flaw is also assumed to be under an applied uniaxial stress which is uniform; stress gradient and multiaxial stress effects were neglected. It is also conservatively assumed that the flaws were oriented perpendicular to the applied stress, with no mixed mode loading.

Since the fracture toughness (K_{IC}) data for many two phase structural ceramics exhibits a good deal of scatter, K_{IC} can be represented stochastically. The flaw size (a) is also assumed to be stochastic to account for the uncertainty in the NDE characterization of the flaw. The flaw orientation is also a stochastic parameter, but it is conservatively assumed that the flaws are oriented perpendicular to the applied stress, with no mixed mode loading. The structural reliability associated with an individual flaw in the component is calculated with the Lotus add-in program @RISK²⁰ using Equation 5.3 and stochastic descriptions of fracture toughness and flaw size. The probability distributions for K_{IC} and a are determined for each situation, depending on the material behavior for K_{IC} and the characteristics of the specific NDE method used defining the flaw. The @RISK output gives the component reliability associated with the single flaw. The form of the presentation is the cumulative distribution function for σ_c , and the reliability is calculated as the probability that the critical stress (σ_c) is greater than the applied stress (σ):

$$r = P(\sigma_c > \sigma) \quad (5.4)$$

If there are multiple flaws in the component, the combined reliability resulting from all flaws can be calculated as:

$$r = \prod r_j \quad (5.5)$$

Where r_j are the reliabilities associated with each flaw.

As the NDE flaw characterization is advanced to include flaw orientations, the individual flaw reliability can be further assessed by treating the orientation as a third stochastic variable. This

treatment would involve the use of mixed-mode fracture criteria such as described by Shetty²² and based on the work of Batdorf²³. The Batdorf models and Shetty criteria have been incorporated into the CARES reliability program and have been shown to provide good comparisons⁴ with experimental data on circular disks containing inherent flaw populations.

Component Reliability

The component fast fracture reliability (R_c) due to the inherent flaws in the material is calculated as the product of all element reliabilities¹⁶. The component reliability associated with the individual flaws is then accounted for by multiplying the component fast fracture reliability from the inherent flaws by the product of the reliabilities of the individual flaws.

$$R_c = (\prod R_i) \times (\prod r_j) \quad (5.6)$$

Where R_i are the reliabilities of the elements due to inherent flaw populations and r_j are the fast fracture reliabilities of the individual detected flaws.

5.3 Demonstration of the Methodology

The methodology will be demonstrated by comparing with fast fracture reliability predictions of the C-ring analyzed in Reference 16. In that analysis, the fast fracture reliability was predicted vs applied load for a C-ring with a machined 10 mil deep notch (Figure 4-3) compared to a C-ring with no notch. Those results are presented in Figure 5-2.

For this demonstration, the fast fracture reliability of the C-ring associated with the machined notch will be calculated with linear elastic fracture mechanics (LEFM) using Equation 5.3 in the @RISK program with appropriate probability distributions for the fracture toughness and flaw size. That resulting reliability will be multiplied by the reliability shown in Figure 5-2 for the specimen with no notch:

$$R_c = (\prod R_i) \times r$$

Where $\prod R_i$ is the reliability of the C-ring without the notch and r is the reliability of the C-ring resulting from the LEFM model in @RISK.

Fast Fracture Reliability

C-Ring using PIA Strength Based Model

Figure 5-2: Fast Fracture Reliability of C-Rings

The Stochastic data that characterizes the fracture toughness of the material was determined from test data on 26 C-ring test samples. A plot of fracture toughness vs failure load in the C-ring tests is shown in Figure 5-1. This figure illustrates the statistical scatter in fracture toughness. A goodness of fit check showed that the data fits a lognormal distribution, so the lognormal distribution was used for the fracture toughness in @RISK. The probability density function for a lognormal distribution is:

$$f(K_{IC}) = \frac{1}{K_{IC}\sqrt{2\pi\sigma^2}} \exp \left\{ -\frac{(\ln(K_{IC}) - \mu)^2}{2\sigma^2} \right\} \quad (5.7)$$

Where K_{IC} = fracture toughness
 μ = mean value of K_{IC}
 σ = standard deviation

The probability that the fracture toughness is greater than the mean is found by integrating equation 5.7:

$$P(K_{IC} > \mu) = \int_0^{K_{IC}} f(K_{IC}) d(K_{IC}) \quad (5.8)$$

It was assumed that the flaw size stochastic definition will follow an exponential distribution as suggested by Becher and Pedersen²¹. Using the exponential distribution, the probability that the NDE determined flaw size is greater than the mean value is given by:

$$P(a > a_0) = 1 - e^{-\frac{a}{a_0}} \quad (5.8)$$

The analysis of the C-ring was performed in Reference 16, assuming there were no large NDE detectable flaws. The reliability for the single flaw (.010 in deep machined notch) was determined with @RISK. The Material properties used in the calculations are shown in Table 5.1.

Table 5.1 Material Properties

PROPERTY		UNITS
Elastic	$E = 36.98E+06$ <hr/> $\mu = .3$	psi <hr/> dimensionless
Flaw Size	Exponential distribution Mean = .010	in
Fracture Toughness	Lognormal Fit Mean(ln K_{IC}) = 7.9066 Std Dvn = .1615	ln(psi $\sqrt{\text{in}}$)

5.4 Analysis Results

The results of the analyses are shown in Figure 5-3. This is a plot of reliability vs load for the C-ring with the .010 in notch. The solid line represents these fracture mechanics based analysis results with the inherent flaws treated as though there were no notch, and the notch treated as a separate flaw statistically characterized with the @RISK program.

For comparison, the dashed line represents the results of the strength based analysis from Reference 16, where the notch was modeled in the finite element analysis and the material was assumed to have only the inherent flaws that are characterized with Weibull statistics.

The Lognormal fit to the fracture toughness data shows that there is significant scatter in the data as shown in Figure 5-1 and Table 5.1. The statistical result has been characterized with @RISK.

Comparison of Proposed Model

with Strength Based Model

Figure 5-3: Reliability vs. Load for Notched C-Rings

5.5 Conclusions

A methodology is presented that combines component fast fracture reliability resulting from individual NDE detected flaws using a fracture mechanics based approach with the reliability resulting from the inherent material flaws using a strength based model. The results applied to a machined notch to simulate a flaw show good agreement with Weibull methods⁸.

This method can be used to establish fast fracture acceptance criteria for components with NDE detected flaws that are more severe than the inherent material flaw population.

SECTION 6

CONCLUSIONS AND RECOMMENDATIONS FOR DESIGN AND TEST SPECIFICATIONS

The development of acceptance specifications and acceptance tests for ceramic components is a multi-step process. It is necessary that the part survive the expected operating conditions (including expected transients and unusual conditions). There will inevitably be variations of material properties on a microscopic scale, and many of these variations are "flaws" in the sense that they act as sites of crack initiation. Ceramic materials contain microcracks, grain clusters, porosity, and inclusions. These are probably inevitable, and must be tolerated. The means for dealing with these is statistical analysis.

Some large defects can be detected by NDE techniques. In general, the definition of "large" defects is determined by the condition of the base material. Defect detection is often limited by the need to ignore the general porosity and inhomogeneity of the material, not by the sensitivity limits of the inspection method. The material quality provides a noise background in which small defects are not detected.

The modeling for a component involves material properties and their evolution over the course of the expected lifetime. These can be measured experimentally. Modeling also includes stress distributions from thermal and mechanical loadings. For silicon carbide heat exchangers, fracture toughness is the limiting property in most cases. The part must be able to withstand the effects of defects which are too small to detect by nondestructive testing.

Mechanical loads will determine minimum dimensions for adequate strength, and thermal shock effects may set maximum dimensions. Other dimensions will be set by the requirements of the application and other components. The model must be able to handle these variations, and determine the maximum flaw size allowed for the expected fracture toughness. If this flaw size is smaller than can be detected, it can't be used as a product specification. The alternatives will be to specify a lower fracture toughness or to specify a more homogeneous material that allows detection of the critical flaws.

Inspection Techniques

Several Flaw detection techniques have been evaluated and developed for use in ceramic

materials in the course of this program. Their use as acceptance criteria for production parts will depend on the value of the parts and the consequences of failure.

- Dye penetrant testing is a useful means of detecting surface cracks in many cases. In the samples used in this program, the dye detected porosity at the surface and crack-like features that were apparently artifacts of an extrusion process. None of these were critical flaws, but it seems likely that cracks would have been detected had they been present.
- Acoustic emission monitoring during stress testing should be very useful, if stresses can be generated that are like those encountered in operation.
- X-ray inspection and computed tomography are likely to be too expensive for most uses, but are valuable during the development process. Microfocus sources are needed, with high magnification, because of the small critical flaw sizes in ceramics.
- Ultrasonic methods can almost always be adapted for use in ceramics. Higher frequencies give better resolution but also more scattering and attenuation. Lower frequencies give penetration at the expense of resolution. Beam angles and fixturing must be adapted for the geometry involved and the defects to be detected, but with modern electronics and robotics this can probably be developed for most parts.
- The Scanning Laser Acoustic Microscope is excellent for rapid inspection of parts with suitable geometry if the ceramic doesn't have too much attenuation. This means either relatively small, flat parts or special fixturing to handle more complex geometry. For many potential applications this is probably the best currently available ultrasonic inspection, in terms of speed and thoroughness, but it is necessary to evaluate its use carefully. Its capabilities are a subset of the ultrasonic technique.

The ultrasonic, acoustic emission, X-ray and computed tomography techniques are described extensively in this report. Specific accomplishments are:

Ultrasonic scanning with multiple beams for improved defect detection,

Reduction in data collection time by a factor of 15,

Upgrade of data collection computer system,

Solution of the problem of calculating beam angles in complex geometries,

Method of precisely setting critical beam angles,

Increased speed of X-ray computed tomography reconstructions by a factor of 10,

Developed model for beam hardening correction,

Developed methods to reduce artifacts and increase defect detectability,

Modeling

A methodology is presented that combines component fast fracture reliability resulting from individual NDE detected flaws using a fracture mechanics based approach with the reliability resulting from the inherent material flaws using a strength based model. The results applied to a machined notch to simulate a flaw show good agreement with Weibull methods⁸.

This method can be used to establish fast fracture acceptance criteria for components with NDE detected flaws that are more severe than the inherent material flaw population. This method is generally applicable if time dependent processes such as creep are not present.

In addition, a methodology is presented that predicts the reliability of monolithic structural ceramics that are subject to time dependent (creep) damage. This use of a continuum damage model for life prediction of structural ceramics seems promising. The methodology appears to be quite general, and readily applicable to many structural ceramic components and materials. A maximum shear stress based theory was used to extend the continuum damage model from uniaxial to multiaxial stress states. The resulting predicted time to failure for a C-Ring demonstration analysis agrees well with the experimental results, and the predicted behavior is physically appealing. However, this is only one case. Additional test cases need to be developed in order to validate the model. Accurate predictions were not anticipated in this case since a good material property database on creep was not available for the specific material at the temperature tested.

Material properties for high temperature analysis are often scarce. This method uses material properties derived from common test procedures. New test procedures are not required to obtain the material property data needed for this life prediction methodology.

The study of various representations of the multiaxial stress state was limited. Only one loading condition was examined. Additional cases should be investigated. Furthermore, only the tensile portion of the multiaxial stress states were investigated. As ceramic material exhibit different behaviors in tension versus compression, other methods of representing the multiaxial stress state should be investigated.

The effect of cyclic loading on life prediction has not been addressed. This type of loading is often present, and should be considered as part of a complete design methodology.

An inherent assumption which may require clarification is the notion that an effective stress can be used with undamaged Weibull parameters to predict long term reliability. The evolution and coalescence of voids and/or flaw growth in preferential directions may alter the Weibull parameters, since the nature of the underlying flaw population, as well as the applied stress, influence these parameters.

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