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MARTIN MARIETTA

SOLID STATE SINTERING OF SILICON NITRIDE ARL-CR-114

FINAL REPORT

John Mangels
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CERAMIC TECHNOLOGY PROJECT

MANAGED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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SOLID STATE SINTERING OF SILICON NITRIDE
ARL-CR-114

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FINAL REPORT

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"SOLID STATE PROCESSING OF SILICON NITRIDE IN THE SYSTEM $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3\text{-SiO}_2\text{-Mo}_2\text{C}$ "^a

John Mangels and Biljana Mikićelj

ABSTRACT

This report describes the development of Si_3N_4 material compositions in the $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3\text{-SiO}_2\text{-Mo}_2\text{C}$ system with good high temperature stress rupture properties which could be used in engine components. Two distinct processing routes were examined in the course of the program: SSN and SRBSN. SRBSN was chosen for material property optimization. After characterization of two optimized compositions in the above system, demonstration engine components (exhaust valve blanks) were manufactured using the established processing procedures. Dimensional tolerance capabilities of the process were established and material properties of the components were shown to be comparable to those established during material development.

INTRODUCTION

Silicon nitride materials have been under continuous development since the late 1960s, primarily for use in high temperature, structural applications such as heat engines. Many of these compositions are now in commercial production for low temperature, wear applications such as cutting tools and bearings. Automotive components, such as turbocharger rotors, are also in production using silicon nitride. However, the extensive use of silicon nitride components for heat engine applications such as gas turbines has not materialized due to high cost and relatively poor strength and time dependent properties above 1200°C. This limitation of silicon nitride materials arises primarily from the selection of densification aids. These materials, primarily oxides, react with the surface impurities on the silicon nitride to form low melting glasses located at the grain boundaries and unstable second phases which can oxidize at relatively low temperatures.

Gazza¹ determined that silicon nitride compositions which were formulated to lie on the $\text{Si}_3\text{N}_4\text{-Y}_2\text{Si}_2\text{O}_7$ join, Figure 1, exhibited excellent high temperature stress rupture behavior at temperatures exceeding 1200°C. His compositions employed the use of Si_3N_4 plus Y_2O_3 and SiO_2 as the primary densification aids. A minor addition of Mo_2C was also identified as being beneficial to densification.

This program for "Solid State Processing of Silicon Nitride" is based on the work of

^aResearch sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the Ceramic Technology Project of the Propulsion System Materials Program, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

Gazza. The overall goals of this development program were to: (1) develop and scale up a process to fabricate a material having the final composition of silicon nitride plus yttrium silicate ($\text{Y}_2\text{Si}_2\text{O}_7$) with the specific composition of 85.8 mol % Si_3N_4 - 4.73 mol % Y_2O_3 - 9.47 mol % SiO_2 (Gazza's optimum composition), (2) develop a material property data base for the developed process, and (3) demonstrate the production capability of the process by producing an engine component shape.

The program was divided into two technical tasks. Task 1 involved the basic process development and material characterization while Task 2 was devoted to the component demonstration.

The details of each of the tasks are described in the following sections.

Figure 1 Phase diagram of the Si_3N_4 - Y_2O_3 - SiO_2 system. The shaded area highlights the compositions studied by Gazza¹.

TASK 1 - PROCESS DEVELOPMENT AND CHARACTERIZATION

1.0 INTRODUCTION

The objective of Task I of this program was to study the sintering behavior, characterize mechanical properties of a Si_3N_4 material with the general composition of 85.8 mol % Si_3N_4 - 4.73 mol % Y_2O_3 - 9.47 mol % SiO_2 and develop an optimal manufacturing process using this material. In addition to the general composition above, additions of up to 1 mol % Mo_2C were an option. This composition was chosen based on good high temperature stress rupture properties exhibited by materials of this composition¹. In addition, good intermediate and high temperature oxidation resistance was measured previously on a material of this composition¹. Since the material was targeted for heat engine applications, the goals for material key properties are presented in Table 1.

Two approaches to obtaining the required material composition were studied: (1) Sintered Silicon Nitride (SSN) and (2) Sintered Reaction Bonded Silicon Nitride (SRBSN).

Table 1 Proposed goals for the Si_3N_4 material development

Sintered Density	> 98.5 % TD
Phase Composition	$\beta\text{-Si}_3\text{N}_4$ + Yttrium Silicate
Microstructure	Acicular Si_3N_4 grains 1-2 μm in diameter
Fast Fracture 4-pt Flexural Strength	
RT	> 700 MPa
1300°C	> 550 MPa
Stress Rupture Life	
900-1300°C; 300 MPa	> 250 h
900-1300°C; 400 MPa	> 250 h
900-1300°C; 500 MPa	> 100 h
Oxidation Behavior	
900°C	$< 3 \times 10^{-13} \text{ kg}^2\text{m}^{-4}\text{s}^{-1}$
1200°C	$< 1 \times 10^{-12} \text{ kg}^2\text{m}^{-4}\text{s}^{-1}$
1250°C	$< 3 \times 10^{-12} \text{ kg}^2\text{m}^{-4}\text{s}^{-1}$

2.0 EXPERIMENTAL PROCEDURES

2.1 Processing Routes

Table 2 presents basic processing steps used to produce Si_3N_4 of required composition by two alternate processing routes (SSN and SRBSN) used during the program.

Table 2 Processing steps for SSN and SRBSN manufacturing routes

Sintered Silicon Nitride (SSN)	Sintered Reaction Bonded Silicon Nitride (SRBSN)
Raw Powders: Si_3N_4 Y_2O_3 SiO_2 Mo_2C (option) Binders	Raw Powders: Si Y_2O_3 SiO_2 Mo_2C (option) Al_2O_3 (option) Binders
Powder Preparation: Attritor Milling Spray Drying	Powder Preparation: Dry and wet ball milling Spray Drying
Isostatic Pressing	Isostatic Pressing
Binder Removal	Binder Removal
Argon Sintering/Green Machining	Argon Sintering/Green Machining
Gas Pressure Sintering (Nitrogen)	Nitriding
	Gas Pressure Sintering (Nitrogen)

2.2 Raw Material Characterization

Raw Si_3N_4 , Si, and sintering aid powders were characterized by measuring the following:

Particle Size Distribution by dispersing powders in DI water and using Centrifugal Particle Size Analyzer (Horiba CAPA 300),

Surface Area by one-point BET N_2 adsorption method (Beta Scientific Automatic Surface Area Analyzer, Model 4200),

Impurity Content in Si_3N_4 and Si was determined by spectrographic methods (metal impurities). Nitrogen and oxygen contents of the Si_3N_4 powder (Ube SN-10) were determined using the inert gas fusion method (LECO TC-436). Oxygen content was used to correct for the required SiO_2 additions.

2.3 Powder Dispersion and Spray-Drying Characterization

During powder preparation different dispersing techniques were explored. Aqueous slurries of Si_3N_4 and Si with additives were characterized by measuring and adjusting their viscosities, specific gravities, and pH values. A mixture of PVA and PEG was used as a binder for all powders.

An optimal spray drying process was obtained by varying the spray-dryer's inlet temperature, atomizer, and pump speeds.

2.4 Green Body Forming - Isopressing

Autoclave (IP-12-36-30) isopress capable of 30 ksi (20.7 MPa) pressure was used for green body forming. For initial sintering studies rods (4 in. long and .75 in. diameter) were wet-bag isopressed to the appropriate pressure. Rods were sliced into four or more parts that were used in individual experiments. Pressed density of the rods was measured by the hot-wax method.

After sintering cycles were established, billets (4x4x0.5 in.) were dry-pressed followed by isopressing. Alternately, whole isopressed rods were processed without slicing. Billets or rods were used for MOR bar fabrication.

2.5 Binder Burnout Characterization

Binder burnout heating schedule (in air) was developed previously by varying temperatures and times at temperature for the binder system used. For the present system tests were made to assure complete burn out and that the powders (Si , Si_3N_4 , Mo_2C) do not oxidize. To ensure this, some powder lots were analyzed for oxygen content after the binder burnout operation.

2.6 Argon Sintering

This optional operation was included to add green strength to the pressed parts and subsequently allow green machining. It was characterized to control possible shrinkage during the operation. Argon sintered densities were measured using ASTM C373-72 specification (with de-ionized water).

2.7 Nitriding

This operation was used for SRBSN parts (samples) which were pressed from Si powder (with additives). The nitriding cycle was previously developed². Three nitriding furnaces with Mo and/or W heating elements and heat shields were used, and a microprocessor-controlled time-temperature, N₂ pressure demand cycle was employed.

The completion of the nitriding reaction (conversion of Si into Si₃N₄) was determined in three ways:

a. Samples were weighed before and after the nitriding and the reaction completeness expressed as Nitrided Weight Gain (NWG):

$$NWG = \frac{W_{nit}}{W_{init}} \quad (1)$$

The measured NWG was compared to the theoretical NWG for the composition used. For the nitriding to be considered complete, the measured NWG had to be at least 98% of the theoretical value.

b. X-ray diffraction patterns of sample cross-sections were taken to ensure that no Si was present in the sample cores. XRD patterns were also used to measure α - to β -Si₃N₄ ratios, second phase formation, and the condition of Mo₂C after nitriding (in samples containing Mo₂C).

c. Microwave heating was used to ensure that no unreacted Si was present in the sample.

2.8 Gas-Pressure Sintering

All samples and parts were sintered in graphite resistance furnaces with a N₂ gas over-pressure. A two step pressure-temperature cycle was employed to ensure complete densification of the compositions. To abbreviate the sintering run cycle description, the following notation will be employed:

$$T(t1/t2)$$

where T is the final sintering temperature, t1 is the time (in hours) at T and low N₂ pressure, and t2 to the time at T and high N₂ pressure. A range of heating rates, final temperatures, and cooling rates was explored in order to obtain optimal results.

2.9 Post-Sintering Heat Treatment

Selected post-sintering heat treatments were employed to maximize second-phase crystallization and to ensure that the crystallized second-phase is yttrium silicate.

2.10 Sintered Material Property Characterization

2.10.1 Density

Sintered density of parts was measured using the Archimedes principle in DI water.

2.10.2 Phase Composition

XRD patterns of samples sintered under different conditions were taken after grinding 20 mils from the surfaces of pellets or after larger parts were cut into sections suitable for use in the apparatus (Norelco Diffractometer 155-001-02). Grain boundary crystalline phases were identified and the relative degree of crystallinity was determined for the samples. Whenever possible, the reaction products of Mo_2C (forming molybdenum silicides) was identified in the samples as well.

2.10.3 Microstructure, Hardness, Toughness

Samples from different runs were metallographically polished and their optical microstructure was routinely evaluated. Using ASTM B276-79 specification, the size, distribution, and concentration of porosity, metallic, and ceramic second phases in the samples were evaluated. Selected samples were examined in the polished state using an SEM (ISI WB-6) equipped with EDS, Robinson back-scatter detector, and light-element detection capabilities (ultrathin window detector).

Vickers hardness of samples was measured on polished samples using a 5 kg load (HV5). The toughness parameter was estimated from the crack lengths emanating from Vickers indentations³.

2.10.4 Fast Fracture Strength

Room temperature fast fracture strengths were periodically performed after certain milestones were reached in the program. Mil STD 1942B bars were used^b in all cases and a 4-point loading fixture was employed (40/20 mm outer/inner spans, 0.05 mm/min cross head

^b Machined by BOMAS MACHINE SPECIALTIES, INC.

speed). Final high temperature fast fracture strengths were performed on the same size bars and fixture types.

2.10.5 Stress Rupture Life

Stress rupture life was measured on bars of the same size as fast fracture using a fixture with the same span. A low load was placed on the bar at room temperature for alignment. Furnace was heated rapidly to the desired temperature, and after sample equilibration time, the desired load was applied to the sample. A number of stepped stress-rupture tests were performed on bars starting from lower temperatures (1000°C). This was done to test the material's intermediate oxidation and slow-crack-growth resistance. Results are reported as the duration of the test before it's suspension, or the time at which the sample fractured during the test. Permanent strain (ϵ) was measured on some bars after the tests were suspended using the following equation:

$$\epsilon = \frac{4t\gamma}{L^2}$$

where t is the bar thickness, γ arc height, and L the inner span of the fixture.

2.10.6 Fractography

Room temperature fast fracture surfaces were examined using an optical microscope to identify the type (if the flaws are large enough) and location of fracture origin (pore, inclusion, chamfer, surface, sample interior). Some bars were observed under SEM to locate and identify fracture origins.

Failed stress rupture specimens were examined with an optical microscope to determine if failures were due to slow crack growth or fast fracture caused by machining flaws or impurities.

2.10.7 Oxidation Resistance

Selected compositions were subjected to prolonged oxidation in air at temperatures ranging from 900 to 1300°C. MOR bars were heated for time intervals in air after which their mass was measured on an analytical balance. Oxidation kinetics at several temperatures were examined and oxidation constants and activation energies were calculated.

3.0 RESULTS AND DISCUSSION

3.1 Sintered Silicon Nitride (SSN)

3.1.1 Raw Powder Characterization

High quality, commercially available $\text{Si}_3\text{N}_4^{\text{a}}$ powder was chosen for use in the program. Commercially available, high-purity $\text{Y}_2\text{O}_3^{\text{d}}$ powder and fumed SiO_2^{e} were used as standard additives. A commercial source of 1-5 μm $\text{Mo}_2\text{C}^{\text{f}}$ powder was found and this powder was used throughout the program.

Characterization results for the powders are presented in Table 3.

Table 3 Powder characteristics

Powder	Si_3N_4	Y_2O_3	SiO_2	Mo_2C
Purity (%)	98.5	99.9	99.8	99.5
N (%)	>38	n/a	n/a	n/a
O (%)	1.29	n/a	n/a	0.5
Cl (ppm)	100	n.d.	n/a	n/a
Fe (ppm)	<10	<4	n.a	100
Ca (ppm)	30	<1	n/a	n.d.
Al (ppm)	23	n.d.	n/a	n.d.
$\alpha\text{-Si}_3\text{N}_4$ content (%)	95	n/a	n/a	n/a
Surface Area (m^2/g)	11.5	10.6	197	5
Average Particle Size (μm)	1.2	1.9	<0.1	3.2

^a Ube Si_3N_4

^d HCST

^e Cabot Corporation

^f HCST

3.1.2 Compositions

Table 4 presents the four basic compositions employed using the SSN processing route.

Table 4 SSN compositions used in the program

Composition #/ Composition (wt %)	1*	2*	3	4
Si ₃ N ₄	90.21	88.7	88.03	86.6
Y ₂ O ₃	7.81	7.78	7.81	7.78
SiO ₂	1.98	1.99	4.16	4.14
Mo ₂ C	0.0	1.48	0.0	1.48

* In these compositions SiO₂ addition was reduced to account for oxygen content present in the Si₃N₄ powder to give the required composition.

3.1.3 Powder Preparation and Pressing

Due to high surface areas and agglomeration of the powders (especially SiO₂ and Si₃N₄), process development for obtaining uniformly dispersed powder mixes consisted of three iterations (Table 5). Dispersion using solely attritor milling initially resulted in 50-100 μ m porous areas in the sintered samples (Iteration 1). Mixing was improved by ultrasonically dispersing SiO₂ powders before their addition to the Si₃N₄ (Iteration 2). Subsequently, to reduce Si₃N₄ agglomerate size, the powder slurry was circulated through an ultrasonic cavity placed in series with the re-circulating attritor mill (Iteration 3).

Table 5 Powder dispersion development

Iteration	Dispersion Technique
1	Attritor
2	Ultrasonic dispersion of SiO ₂ + Attritor
3	Ultrasonic dispersion of the slurry in line with attritor

The ultrasonic powder dispersion technique (iteration 3) resulted in the following characteristics of the slurries and spray dried powders made from them (Table 6).

Table 6 Slurry and spray-dried powder characterization

Composition	1	2	3	4
Slurry				
Solids Content (Vol %)	45	55	42	50
pH	8.5	8.5	8.5	8.5
Viscosity (cps)	120	150	180	300
Powder				
Bulk Density (g/cm ³)	0.61	0.67	0.58	0.63
Tap Density (g/cm ³)	0.74	0.75	0.7	0.72
Pressed Density @ 15000 psi (g/cm ³)	1.64	1.66	1.62	1.65
Compaction Ratio	2.69	2.48	2.79	2.61

Based on Tables 4 and 6, it is evident that the increase in SiO₂ content increases the slurry viscosities, and decreases the pressed densities of the powder. The presence of Mo₂C also effects slurry and spray-dried powder properties.

Figure 2 shows the uniform distribution of additives in an as-pressed compact of composition 4 powder (iteration 3).

Figure 2 Distribution of additives in a green composition 4 compact (SEM back-scatter image showing atomic number contrast).

3.1.4 Binder Burnout

A previously developed binder burnout schedule, at a temperature of 490°C, that removed 99.9% of the binder from parts up to 3 in. diameter was used. It was shown that at temperatures 20-50°C below the optimum temperature larger parts still contained small amounts of binder. Oxygen contents of pure Si_3N_4 powder before and after the binder burnout operation were compared in Table 7, showing that no measurable oxidation occurred during this operation.

Table 7 Binder burnout effect on oxygen content of Si_3N_4 powder

Sample treatment	O (%)
as received	1.29
after binder burnout	1.28

Pure Mo_2C after a burnout run showed evidence of slight oxidation; however, the XRD patterns confirmed that the oxidation occurred only on particle surfaces.

3.1.5 Argon Sintering

The optional argon sintering operation was performed at a temperature of 1066°C (two hours temperature hold). Measurements indicated that a linear shrinkage of less than 0.2% occurred during this operation. The operation did increase the green-part strength considerably, which was its main purpose.

3.1.6 Gas Pressure Sintering

Initially, a sintering cycle was developed to achieve theoretical densities of compacts. Small compacts of up to 0.5 in. high (sliced from isopressed rods) and 0.75 in. diameter were used in these experiments. After a sintering temperature/time range was developed, larger rod sections and billets were sintered to further optimize the sintering cycle. Sintering behavior was found to be dependent on sample size.

3.1.6.1 Sintering of Samples With up to 0.5 in. Cross Sections

A designed experiment using five variables at two levels was conducted with compositions 1 and 2. The experiment required four sintering runs to complete. The variables were as follows:

1. Maximum temperature (1850, 1900, and 1950°C),
2. Time at Temp. and low pressure (3 and 5 h),
3. Packing Powder Composition (0 and 2% SiO₂ addition),
4. Amount of Packing powder (buried, covered parts),
5. Mo₂C content (0 - composition 1, and 1 mol % - composition 2),
6. Argon Sintering (yes or no)

Variable 4 consisted of either completely burying parts in packed powder (buried) or just covering the part with a layer of powder (covered).

Densities obtained from the experiment are presented in Tables 8 and 9 for non argon-sintered samples (results for argon-sintered samples were identical). These results show that the time at the sintering temperature and low pressure affect the sintered densities of both compositions at 1900 and 1950°C. This suggests that thermal equilibrium was not yet reached in the samples after 3 h at temperature.

The time-at-temperature effect is non-existent when packing powder with 2% SiO₂ addition was used for both compositions. This packing powder promoted densification of both compositions, even when shorter sintering times (or a lower sintering temperature of 1850°C) were used.

In these experiments, densities of above 99.5% were obtained at both sintering temperatures, for both compositions, as long as the samples were equilibrated at the sintering temperatures.

Table 8 Densities of composition 1 (no Mo₂C)

Composition 1 Densities (g/cm ³) $d_h = 3.276 \text{ g/cm}^3$			Sintering				
			1850°C	1900°C		1950°C	
			5 h	3 h	5 h	3 h	5 h
Packing Powder Composition and amount	0% SiO ₂	covered	3.05	2.920	3.278	3.06	3.259
		buried	2.95	2.760	3.266	2.86	3.256
	2% SiO ₂	covered	3.25	3.279	3.271	3.27	3.265
		buried	3.272	3.264	3.262	3.27	3.265

Table 9 Densities of composition 2 (1 mol % Mo₂C)

Composition 2 Densities (g/cm ³) d _m = 3.298 g/cm ³			Sintering Temperature				
			1850°C	1900°C		1950°C	
			5 h	3 h	5 h	3 h	5 h
Packing Powder Composition and amount	0% SiO ₂	covered	3.14	3.04	3.272	3.03	3.273
		buried	3.03	2.90	3.308	3.12	3.200
	2% SiO ₂	covered	3.31	3.29	3.275	3.30	3.300
		buried	3.31	3.30	3.290	3.303	3.283

Densities of compositions 1 and 2 sintered between 1850 and 1950°C do not indicate that Mo₂C acts as a sintering aid.

3.1.6.2 Sintering of Samples With Cross Sections Over 0.5 in.

Based on the results obtained on sintering small samples (Tables 8 and 9), larger rod sections (1 in. long, 1 in. diameter) and billet sections (0.75-1 in. thick) made from compositions 1 and 2 were sintered at 1850°C and 1900°C. Severe bloating was encountered in composition 2 (1 mol % Mo₂C) parts with large cross sections that were sintered using 2% SiO₂ packing powder. Composition 1 parts, and all parts placed in 0% packing powder did not exhibit bloating.

Examination of the interior of the bloated parts showed high levels of unreacted Mo₂C compared to the dense surfaces in which only molybdenum silicides were present. This indicated that SiO₂ from the packing powder preferentially promoted densification of the surfaces of the parts at relatively low temperatures, while the interior of the samples containing Mo₂C were still reacting and generating gases (N₂ and SiO, and CO most likely) which caused the bloating.

To eliminate the cause of bloating in Mo₂C containing compositions and to obtain yttrium silicate as a second phase more consistently in the sintered parts, compositions 3 and 4 were made with approximately 2% higher SiO₂ contents than compositions 1 and 2, respectively (Table 6).

Sintering of large rods made from compositions 3 and 4 at 1800, 1850, and 1900°C in both packing powders did not show any signs of bloating under any of the conditions. Densities

above 99.5% of theoretical were reached for all compositions. For compositions 3 and 4, packing powder with SiO₂ additions no longer acted as a sintering aid.

Based on the sintering, phase composition, and microstructural results, 4x4x0.75 in. billets for room temperature strength evaluation were made from compositions 3 and 4 and were sintered at 1850°C (5/1 schedule) with 0% SiO₂ packing powder in four sintering runs. Some of the billets after sintering had radial cracks which were probably due to a too rapid heating rate. Average billet densities were 3.258 g/cm³ for composition 3 and 3.278 g/cm³ for composition 4 (99.5% of theoretical density in both cases). MIL STD 1942B MOR bars were machined from these billets.

3.1.6.3 Sintering Weight Losses

Weight losses of composition 1 and 2 small pellets ranged between 1 and 5%. Mo₂C containing compositions typically had 0.5-1.0% higher weight losses, a result of the Mo₂C → Mo₃Si₂ reaction which is accompanied by additional weight loss. Weight losses increased at higher sintering temperatures. Packing powder with SiO₂ addition reduced the amount of weight loss by up to 1.0%. Sintering weight losses decreased with the increase in the size of the part.

3.1.6.4 Phase Composition and Microstructure

Phase composition of sintered samples, based on results from all runs, is presented on Table 10. Based on these results, compositions 3 and 4 more consistently contained Y₂Si₂O₇ in the grain boundaries, which was the objective of the program.

Table 10 Phase composition after sintering (compositions 1-4, refer to Table 4)

Composition	Major Phase	1st Minor Phase (in order of occurrence frequency)	2nd Minor Phase
1	β -Si ₃ N ₄	H, Si ₃ Y ₂ O ₃ N ₄ , Y ₂ Si ₂ O ₇	none
2	β -Si ₃ N ₄	H, Y ₂ SiO ₅ , Y ₂ Si ₂ O ₇	MoSi ₂ or Mo ₃ Si ₅
3	β -Si ₃ N ₄	Y ₂ Si ₂ O ₇ , Y ₂ SiO ₅	none
4	β -Si ₃ N ₄	Y ₂ Si ₂ O ₇ , Y ₂ SiO ₅ , H	MoSi ₂ or Mo ₃ Si ₅

Typical optical microstructure of a sintered composition 3 sample (Figure 3) shows a low level of porosity in the 10-25 μm range. In addition, composition 3 (and all compositions with no Mo₂C additions) showed a characteristic macrostructure (coring) with bands of different gray shadings in the part cross sections. Figure 3 shows differences in the metallic phase levels (Si

inclusions) between the core (gray), central band (beige) and the surface layer of the part (gray).

Figure 3 Composition 3 optical microstructure: a. sample core (gray); b. central band (beige); c. surface layer (gray). Mag. 170x.

Composition 4 microstructure (Figure 4) shows an elevated level of B porosity in the sample (BO6-BO8) and a higher concentration of the metallic phase (due to the presence of MoSi_2 and Mo_3Si_5).

Sintered billets of both compositions showed a slightly elevated amount of porosity in the billet interior compared to a less porous 0.125 in. surface layer.

In some samples, isolated porosity larger than $25\text{ }\mu\text{m}$ was found, as well as larger $50\text{-}100\text{ }\mu\text{m}$ metallic inclusions. Metallic inclusions were later identified as Si metal contamination from the spray-dryer. This source of contamination could not be entirely removed even after complete cleaning of the spray-dryer.

Figure 4 Composition 4 optical microstructure. Magnification 170x.

3.1.6.5 Hardness and Toughness

Hardness of compositions 1-4 was in the 1390-1430 kg/mm² range. No sintering temperature dependence was observed. The toughness of the two materials was in the 5.5 to 6.6 MPa·m^{1/2} range.

3.1.6.6 Room Temperature Strength of Compositions 3 and 4

Characteristic and mean strengths, standard deviation, and Weibull moduli of composition 3 and 4 MOR bars are shown in Table 11.

Table 11 Room temperature strengths of composition 3 and 4 materials (see Table 4 for composition)

Composition	Characteristic Strength (MPa)	Mean Strength (MPa)	Standard Deviation (MPa)	Weibull Modulus
3 (15 bars)	468	420	138	3.2
4 (15 bars)	437	408	72	6.3

Examination of the fracture origins for both compositions showed that large pores (30-150 μm) caused about 80% of the fractures, and that the remaining 20% were due to partially metallic Si inclusions.

Strengths reported in Table 11 were not high enough for any high temperature evaluation to be conducted. After reviewing all of the characterization results available it was concluded that although the porosity problem in these samples could probably be overcome by sintering modifications, Si inclusions would always be a potential problem. Based on this, an alternate approach to obtaining the proposed material(s) was attempted. This approach was to obtain the compositions using the Sintered Reaction Bonded Si_3N_4 methodology.

3.2 Sintered Reaction Bonded Silicon Nitride

The basic SRBSN processing steps were presented in Table 1.

3.2.1 Raw Powder Characterization

Commercially available metallurgical grade Si metal powder (99.5% purity) was used for the program. Average particle size of the powder was 3.4 μm with a 3.5 m^2/g surface area. Same grades of powders described in § 3.1.1 were used as sintering aids (Y_2O_3 , SiO_2 , and Mo_2C). High-purity Al_2O_3 powder with 9.3 m^2/g surface area and 1.2 μm average particle size was used in some compositions.

3.2.2 Compositions

Tables 12 and 13 present the compositions studied using the SRBSN route. Compositions are given based on the final Si_3N_4 composition after nitriding.

Table 12 Investigated MTL program SRBSN compositions

COMPOSITION (weight %)	Basic Program Compositions		
	5*	6*	7
Si_3N_4	88.0	86.6	87.1
Y_2O_3	7.8	7.8	7.8
Al_2O_3	0.0	0.0	0.0
SiO_2	4.2	4.1	3.5
Mo_2C	0.0	1.48	1.48
Theoretical Density (g/cm^3)	3.266	3.298	3.290

* Note that the composition 5 (SRBSN) is equivalent to composition 3 (SSN) and 6 (SRBSN) to 4(SSN).

Table 13 Alternative SRBSN compositions which included minor Al_2O_3 additions

COMPOSITION (weight %)	Alternative Compositions						
	First Iteration				Second Iteration		
	8	9	10	11	12	13	14
Si_3N_4	88.0	90.8	85.0	89.8	86.6	87.1	88.6
Y_2O_3	7.8	4.0	7.8	4.0	7.8	7.8	7.8
Al_2O_3	0.0	1.0	1.0	0.0	1.0	0.5	1.0
SiO_2	4.0	4.0	6.0	6.0	4.0	4.0	2.0
Mo_2C	0.2	0.2	0.2	0.2	0.6	0.6	0.6
Theoretical Density (g/cm^3)	3.278	3.238	3.280	3.228	3.291	3.287	3.295

The basic program compositions in Table 12 satisfy the program requirements, and are identical to the compositions used in the SSN route. The alternative compositions were introduced in an attempt to improve the microstructure of the material, and are deviations from the program compositions due to Al_2O_3 additions and the changes in the $\text{Y}_2\text{O}_3/\text{SiO}_2$ ratios. Al_2O_3 was added in an attempt to reduce the volatility of SiO_2 during sintering and improve the microstructure (Al_2O_3 additions were kept at a minimum to restrict the amount of glassy phase formed at the grain boundaries). Mo_2C content was reduced to 0.2% and 0.6%.

3.2.3 Powder Preparation and Pressing

Powders were mixed using a three step process: V-blending and dry ball milling, followed by wet ball milling (binders were added during this step). Using this mixing technique, slurry solid contents of 62% could be obtained with viscosities of 60-80 cps. A free flowing powder was obtained by spray drying. Pressed densities of the powders at 15 ksi were 1.51 and 1.53 g/cm^3 for compositions 6 and 7, respectively. Other compositions had similar pressed densities.

No problems were encountered during the mixing, and the additives were shown to be uniformly dispersed with the Si powder.

3.2.4 Binder Burnout

The same binder burnout schedule used for SSN was used for SRBSN parts. No oxidation of the Si powder was observed during this operation. Complete binder removal from the parts was accomplished.

3.2.5 Argon Sintering

This optional step was performed at 1066°C and it resulted in 0.2-0.4% linear shrinkage. The parts had sufficient strength after the operation to be green-machined. This operation was shown not to affect the sinterability of the parts, therefore it was omitted in the material sintering studies.

3.2.6 Nitriding

Nitriding weight gains for all compositions ranged from 98-99% of the theoretical values. Table 14 illustrates typical weight gains measured on compositions 5-11. Measured weight gains on Table 14 are given for different nitriding runs and different powder batches of the same composition. Similar values were obtained for other compositions (12-14). X-ray diffraction confirmed that no detectable amount of free silicon metal was present in any of the compositions. Nitrided parts were mostly α -Si₃N₄ and β -Si₃N₄ (with 80% α / 20% β weight ratio of about) with minor amounts of H phase. Parts with Mo₂C also had detectable amounts of Mo₂C.

Table 14 Measured nitriding weight gains for different compositions

Nitriding Weight Gain (NWG)	Basic Compositions (see Table 12)			Alternate Compositions			
	5	6	7	8	9	10	11
Nitriding 1	1.521	1.50	1.505	1.521	1.536	1.490	1.522
Nitriding 2	1.520	1.510	1.510	1.520	1.544	1.492	1.534
Nitriding 3	1.513	1.510					
Theoretical Weight Gain	1.542	1.529	1.535	1.542	1.568	1.514	1.559
% of Theoretical	98.44	98.5	98.2	98.61	98.21	98.48	98.01

3.2.7 Gas Pressure Sintering and Room Temperature Strength

SRBSN sintering cycle development was performed using cylindrical rods 0.75 in. diameter and 1-2 in. long. Initially, 5/1 (5 h at low pressure, 1 h at high pressure) sintering cycles developed for the SSN were tried for compositions 5 and 6 (see Table 12). Sintering temperatures from 1850-1950°C were used. However, inconsistent run-to-run densities were obtained. In all cases samples had reached the closed porosity phase before the final pressure application and, therefore, it was necessary to increase the sintering time at high pressure to consistently obtain theoretical densities in compositions 6 and 7. Composition of the packing powder did not effect the densities of the parts (see § 3.1.6.1).

Sintering between 1900 and 1950°C using a 5/2 or 5/3 pressure cycle was found to give consistently high densities (99.5% of theoretical) for compositions 5, 6, 7, and 10 (Figure 5).

It is not clear why composition 8 consistently sintered to lower densities than compositions 5 and 6 although they differed only in the Mo_2C additive amount. Compositions 9 and 11 probably required higher temperatures for densification. No efforts were made to design schedules which would densify compositions 8, 9, and 11 since that was beyond the scope of the program.

Figure 5 Relative densities obtained after sintering SRBSN compositions using three sintering cycles.

Compositions 12-14 sintered to theoretical densities using 1900°C 5/2 or 5/3 cycle.

Preliminary room temperature strength evaluation of compositions 5, 6, and 10, using the 1910°C, 5/3 schedule, was performed (rods were used for machining MOR bars). Room temperature strengths measured on these compositions (sintered using 1910°C, 5/3 cycle) are presented in Table 15. Compared to SSN strengths obtained earlier (Table 11), compositions 6 and 10 showed a significant strength and Weibull modulus improvement over compositions 3 and 4. Composition 5 (no Mo₂C additions) still had a low strength although the Weibull modulus was improved. Porosity was found to cause the majority of composition 5 failures. Examination of fracture origins of composition 6 and 10 fractured bars indicated that porosity and exaggerated grain growth were the major causes of failures (Figure 6).

Table 15 Preliminary RT strengths of SRBSN compositions 5, 6, and 10 after 1910°C, 5/3 sintering (15 bars per composition).

Composition #	5	6	10
Average Strength (MPa)	435	518	532
Characteristic Strength (MPa)	459	534	588
Weibull Modulus	9.1	18	3.8

Based on Table 15 strength results, further strength improvements were attempted using compositions 6, 10, and 14 (the decision was based on microstructure of the compositions and the comparatively low strength values of composition 5). Strength improvements were attempted by: (1) reducing the size of the pores, and (2) minimizing the exaggerated grain growth. This was achieved by further modifications to the 1900°C sintering schedule. Modifications included addition of an intermediate temperature hold during heating (gas-evolving reactions were completed before pore closure), and a reduction of time at sintering temperature by 2 h (3/2 schedule instead of 5/2). Dense billets (3.5x3.5x0.75 in.) from all three compositions were obtained using this cycle, and MOR bars obtained from them exhibited improved strengths (Table 16). Porosity (20-40 μm) was found to be strength limiting for the majority of the bars.

The final modification of the sintering cycle was made by increasing the sintering temperature to 1925°C for compositions 6 and 7. RT strengths from the optimized sintering cycle (Table 17) show a significant improvement in the Weibull modulus of both compositions over the results in Table 16. This cycle was used for the production of MOR bars for high temperature strength and stress rupture testing.

Figure 6 Large grain (150 μm long and 10 μm wide) as a fracture origin in composition 6 (1 mol%Mo₂C, bar fractured at 550 MPa). SEM backscattered image.

Table 16 RT strengths after a modified sintering cycle (hold on heating, 1900°C, 3/2), compositions 6, 10, and 14.

Composition #	6	10	14
Average Strength (MPa)	599	594	617
Characteristic Strength (MPa)	634	626	636
Weibull Modulus	5.3	14	14
# of bars	14	14	16

From the same billets used for machining regular MOR bars, 10 bars were machined with one of the tensile faces left in the as-sintered condition. Table 17 contains the strengths of the as-sintered material surface for comparative purposes. The as-sintered surface strength is 80% of the machined material strength for composition 6. This is a higher number than usually encountered in gas-pressure sintered or glass-encapsulation dipped Si₃N₄ (55-72%) materials⁴.

Table 17 RT strengths of compositions 6 and 7 after the optimized sintering cycle (hold on heating, 1925°C, 3/2).

Composition #	6	6	7
Tensile surface condition	machined	As-sint	machined
Average Strength (MPa)	602	483	595
Characteristic Strength B(63.2) (MPa)	615	494	600
B(1) Strength	467	378	534
Weibull Modulus	17	17	39
# of bars	14	10	14

3.2.8 Sintering Weight losses

Sintering weight losses of compositions 5-7 were typically in the 3-5% range. Generally, the weight losses increased with the increase in the sintering temperature, the time at temperature, and the sample surface area to mass ratio.

Sintering weight loss studies on compositions 8-11 under identical conditions showed that additions of SiO₂ increase sintering weight losses, whereas additions of Y₂O₃ and Al₂O₃ decrease them. It is assumed that additions of Y₂O₃ and Al₂O₃ reduce volatilization of SiO by reacting with the SiO₂ glass (reducing the equilibrium partial pressure of SiO).

3.2.9 Phase Composition and Microstructure

Sintered samples of all compositions had β -Si₃N₄ as the major phase. The second crystalline phases for most compositions were identified as H-phase, Y_{4.67}(SiO₄)₃O (which is the oxidized form of H-phase), and Y₂Si₂O₇. Temperature holds in the cooling portion of the sintering cycle increased the relative amount of the second phase. The optimized sintering cycle for compositions 6, 7, 10, and 13 that included a crystallization step, consistently yielded Y_{4.67}(SiO₄)₃O as the second crystalline phase. Post-sintering heat treatments at 1400°C further increased the crystallization of the grain boundary phase, and in most cases only Y_{4.67}(SiO₄)₃O was detected as the crystallized phase. Using SEM/EDS, Mo was found to be in a silicide form (MoSi₂ or Mo₅Si₃) in the sintered samples.

Optical microstructures of polished samples used for machining MOR bars at different stages of the program are presented on Figures 7 through 9. Figure 7 shows microstructures of compositions 5, 6, and 10 in samples fired using the 1910°C, 5/3 sintering cycle and had bend-bar strengths 535, 518, and 532 respectively (Table 15). Microstructures represent the interiors of the samples. Composition 6 (Figure 7b) showed a relatively high concentration of fine porosity compared to compositions 5 and 10. Composition 10, in addition to porosity, showed evidence of second phase segregation in some areas during firing (gray phase in Figure 7c). This is due to the high additive content of this composition (Table 13).

Figure 7 Optical microstructure of: a) comp. 5, b) comp. 6, c) comp. 10 sintered using the initial sintering cycle (strengths on Table 15). Magnification 170 X.

Figure 8 shows an improved microstructure of compositions 6 and 10 and 14 after a modified sintering cycle (1900°C, 3/2). Compositions 10 and 14 had less porosity in the microstructure compared to composition 6. This is attributed to the presence of 1% Al_2O_3 in compositions 10 and 14. Composition 10 (Figure 8b) again shows the presence of segregated second phase, which is not present in compositions 6 and 14 (due to differences in SiO_2 additions). Improvement in the microstructure lead to higher strengths of the materials (Table 16). Clustered porosity of composition 6 (Figure 8a) was the cause of a low Weibull modulus (5.3).

The final microstructure of composition 6 obtained after the optimized sintering cycle is shown on Figure 9. The average strength of this material was 602 MPa with a Weibull modulus of 71.

Figure 8 Optical microstructures of compositions a) 6, b) 10 and c) 14 after the revised sintering cycle. Magnification 170X.

Figure 9 Microstructure of composition 6 after the optimized sintering cycle. Magnification 170 X.

SEM microstructures of composition 6 and 7 samples sintered using the optimized sintering cycle are shown on Figure 10. Both compositions have acicular Si_3N_4 grains with aspect ratios of 5-20, however the composition 6, overall, has much finer grains (1-2 μm in diameter, Fig. 10a) compared to composition 7 (1-5 μm in diameter, Fig. 10b).

a)

b)

Figure 10 Composition a) 6 and b) 7 SEM microstructures (back-scattered images). Mag. 500 X

3.2.10 Hardness and Toughness

Hardness and toughness values for different SRBSN compositions are shown in Figures 11 and 12, respectively. Hardness of the compositions was in the 1400-1550 kg/mm² range, and it was generally higher for compositions with lower additive amounts (composition 9) and lower for compositions with high additive contents (composition 10).

Figure 11 Hardness of different SRBSN compositions used in the program.

Toughness values of all compositions ranged from 5 to 8 MPa/m (Figure 12). Although the variations from composition to composition are not large, overall, it appears that composition 6 had the highest average toughness values.

Figure 12 Fracture toughness of SRBSN compositions.

3.2.11 Oxidation Resistance

Oxidation weight gains were measured on as machined composition 6 MOR bars, at 1000, 1200, and 1300°C for up to 300 h. Figure 13 shows that the oxidation kinetics follow the parabolic law, and that the oxidation is probably limited by the oxygen diffusion through the surface oxide layer. Data indicate no intermediate temperature oxidation problems for this material. Similar data were obtained on compositions 7 and 10.

From Figure 13, kinetic reaction constants were calculated for the three temperatures ($1.7 \cdot 10^{-11} \text{ kg}^2/\text{m}^4\text{s}$ at 1300°C, $3.8 \cdot 10^{-12}$ at 1200°C, and $1.7 \cdot 10^{-12}$ at 1000°C). These values agree well with the weight gains observed after stress rupture testing and Gazza data¹. The activation energy for the oxidation was calculated to be 120 kJ/mol (Figure 14).

Figure 13 Parabolic oxidation weight gain kinetics fit for composition 6 at 1000, 1200, and 1300°C.

X-ray diffraction of the oxidized surfaces indicated that the major oxidation product was $Y_2Si_2O_7$ (card 22-1103) with strong preferential orientation along the (130) and (220) crystallographic planes. This is due to the formation of elongated $Y_2Si_2O_7$ grains on the part surface (Figure 15).

Figure 14 Temperature dependence of composition 6 oxidation constants for determination of the activation energy.

Figure 15 Oxidized surface of the MoR bar after 250 hrs at 1300°C. Magnification 170 X.

3.2.12 Heat Treatment Effects on Room Temperature Strength

In order to ensure complete crystallization of the grain-boundary phases in compositions 6 and 7, MOR bars produced from the optimal sintering cycles were heat treated in a N_2 atmosphere at 1400°C temperature. Measured room temperature strengths on the heat treated bars (Table 18) were significantly higher (12-22%) than the strength before the heat treatment for both compositions (Table 17). The strength improvement is attributed to the healing of machining induced damage and other flaws near the surface of the material, and/or second-phase crystallization during the heat treatment.

Table 18 Room temperature strength of heat treated Si_3N_4

Composition #	6	7
Average Strength (MPa)	672	739
Characteristic Strength (MPa)	717	758
Weibull Modulus	6.8	14.2
# of bars	20	10

3.2.13 Fast Fracture Strength Temperature Dependence (Compositions 6 and 7)

Elevated temperature strengths were measured on heat-treated MOR bars made from material sintered under optimal sintering schedule. Composition 6 and 7 fast fracture strength temperature dependence is presented in Figure 16. At 1300°C strength for composition 6 was 480 MPa, whereas composition 7 had a lower strength of 370 MPa. (It should be noted that strengths of composition 6 were based on 10 bars at each temperature, and composition 7 strengths only on 5 bars per temperature.)

Figure 16 Temperature dependence of composition 6 and 7 strengths.

Most of the bar failures were porosity or metallic impurity induced.

3.2.14 High Temperature Stress Rupture

Stress rupture properties of compositions 6 and 7 and to a limited extent of compositions 10 and 14, were evaluated in the 1000 to 1300°C temperature range under stress levels of up to 345 MPa.

Initially, stress rupture of post-heat-treated composition 10 and 14 bars were evaluated at 1300°C and loads of 200 MPa (due to their relatively high RT strengths and Weibull moduli - Table 16). Three bars of each of the compositions failed within the first hour of loading. Failure mode was determined to be slow crack growth due to grain boundary oxidation (both compositions contain Al_2O_3 additions). Characterization of compositions 10 and 14 was terminated due to these results.

Composition 6 stress rupture results are presented in Table 19. Most of the results were generated on post-heat-treated MOR bars based on one early failure of as-machined bar at 1300°C (300 MPa) after 28 h (Table 19).

Table 19 Stress rupture properties of composition 6

POST HEAT TREATMENT	TEMPERATURE (°C)	STRESS (MPa)	LIFE UNDER STRESS (h)	STRAIN (%)	WEIGHT GAIN AFTER TEST (%)
Yes	1000 1250	200 200	70 254+	0.37	0.08
	1000 1000 1100	275 310 310	250 236 52+	<0.05	0.01
	1000 1100 1250	310 310 310	140 164 30+ heating el. fail.	0.05	0.03
	1200 1250 1250	275 310 350	150 150+ failed on loading		
	1000	275	130+		
	1200	275	6 min, chamfer mach. flaw		
	1200	310	6 min, surface mach. flaw		
	1250	300	3 min, chamfer mach. flaw		
	1250	300	250+		
	1250	300	250+		
	1250	300	246+	N/A	.07
	1300	200	260+	.87	.084
No	1000 1250	300 300	94 241+	.34	.101
	1250	200	250+	.53	.076
	1300	300	28 failed	N/A	N/A

Most of the tests reported on Table 19 were interrupted if no failure was observed after approximately 250 h at the final temperature. Based on stepped stress rupture tests starting from 1000°C, composition 6 material does not have any intermediate temperature oxidation problems. The material survived stresses up to 310 MPa at 1250°C for over 250 h. Composition 6 had good stress rupture properties at 1300°C up to 200 MPa stress levels. Higher stresses at 1300°C were not attempted due to a permanent strain of 0.87% measured under 200 MPa stress.

Based on the measured permanent strain of the bars, the strain rate of composition 6 at 1250°C and 300 MPa was $0.6-0.9 \times 10^{-9} \text{ s}^{-1}$, comparable to SN251 and GN-10 silicon nitrides³.

All early failures reported in Table 19 were found to be caused by chamfer or surface scratches or heat-treatment-induced impurities (Al and Fe), and are therefore not considered in the general evaluation of the material.

Limited stress rupture test results of the composition 7 material are presented in Table 20 (this composition has a 0.5% lower addition of SiO₂ than composition 6 and was produced in an attempt to improve the creep resistance of the material).

Table 20 Composition 7 stress rupture results

POST HEAT TREATMENT	TEMPERATURE (°C)	STRESS (MPa)	LIFE (h)	STRAIN (%)
yes	1250	350	114+	.5
	1300	300	193+	
	1300	300	1.3, impurity induced	
no	1250	350	failed on loading	
	1300	300	193+	1.0

Limited test results on composition 7 material suggest that the material can sustain stresses up to 300 MPa at temperatures up to 1300°C. At temperatures above 1250°C, the material is limited by a lower fast fracture strength compared to composition 6 (see Figure 16).

TASK I - SUMMARY AND PROPERTY DATA BASE

A process for gas-pressure sintering of composition 6 and 7 reaction bonded Si_3N_4 materials has been developed. Both compositions contain Y_2O_3 , SiO_2 , and Mo_2C sintering additives and differ from each other only in the level of SiO_2 .

The process was shown to be capable of nitriding and sintering rods up to 7 in. length and 1 in. diameter, and billets 4x4x1 in. in size.

Both compositions have been characterized and were found to have comparable room temperature strength (no post-sintering treatment), hardness, toughness, oxidation resistance, and phase composition. Post-sintering heat treatment, which crystallizes the intergranular phases, increased room temperature strengths of composition 6 to 672 MPa and composition 7 to 739 MPa. Microstructure of both materials consisted of acicular $\beta\text{-Si}_3\text{N}_4$ grains; however, composition 7 had coarser grains than composition 6.

High temperature fast fracture strengths of both compositions were good; however, strengths of composition 6 were consistently higher than of 7. In stress rupture, both materials were found to be capable of sustaining stresses of 300 MPa at 1250°C and 250 MPa at 1300°C for over 250 h. Composition 7 appeared to have better stress rupture properties than 6, but more data should be generated to confirm this in the light of its lower high temperature strength. High temperature properties of both materials could be further improved by increasing the purity of the starting powder, and by further fine tuning the microstructural development in the materials.

Material properties of composition 6 and 7 materials are presented in Table 21.

Table 21 Properties of developed composition 6 and 7 Si_3N_4 materials

Property	Composition 6		Composition 7	
Composition				
Si_3N_4 (wt %)	86.6		86.1	
Y_2O_3 (wt %)	7.8		7.8	
SiO_2 (wt %)	4.1		3.5	
Mo_2C (wt %)	1.48		1.48	
Sintered Density (% TD)	>99.5		>99.5	
Phase Composition	$\beta\text{-Si}_3\text{N}_4$ + Yttrium Silicate		$\beta\text{-Si}_3\text{N}_4$ + Yttrium Silicate	
Microstructure	Acicular $\beta\text{-Si}_3\text{N}_4$ grains 1-5 μm in diameter		Acicular $\beta\text{-Si}_3\text{N}_4$ grains 1-2 μm in diameter	
Fast Fracture Flexural Strength (MPa)	Average Strength	Weibull Modulus	Average Strength	Weibull Modulus
RT (as machined bars)	603	17	599	39
RT (heat treated)	672	7	739	14
1000°C	583	-	550	-
1200°C	522	-	490	-
1300°C	480	-	372	-
1350°C	362	-		
Stress Rupture Life (h)				
900-1250°C, 300 MPa	>250		>250	
1300°C, 200 MPa	>250		>250	
900-1300°C, 300 MPa			>200	
Oxidation Rate ($\text{kg}^2/\text{m}^4 \text{ s}$)				
1000°C	$1.7 \cdot 10^{-12}$			
1200°C	$3.8 \cdot 10^{-12}$			
1300°C	$1.7 \cdot 10^{-11}$			
Hardness (kg/mm^2)	1450-1520		1450-1500	
Fracture Toughness ($\text{MPa} \cdot \text{m}^{1/2}$)	6.3-7.5		6.6	

TASK II - DEMONSTRATION COMPONENT PRODUCTION

1.0 Task II Objectives and Component Selection

The objectives of Task II were to demonstrate that the material and process developed in Task I can be used to produce a component to near-net-shape with properties comparable to those demonstrated in Task I.

An exhaust Valve Blank (Ceradyne p/n 78916, Figure 17) was chosen to be the demonstration component. The reliability requirements for this component were: 600 MPa average strength and a minimum B(1) of 450 MPa, Weibull modulus of 17 and operating temperature of 800°C in a reducing, carbon-rich environment. Based on properties demonstrated in Task I, composition 6 satisfied the material requirements (Table 17).

Additional advantages of the chosen demonstration component were that it could be produced using essentially the same processing steps as in Task I, and that the basic material property verification could be easily accomplished by machining MOR bars from the valve stems.

1.1 Fabrication

Valves were processed using the same processing steps described in the previous sections. They were isopressed to near-net-shape to a density of 1.610 - 1.620 g/cm³ (pressed valve blank drawing is in Figure 17). After binder burnout and Ar-sintering, valves were green machined to final shape. Parts were nitrided with average weight gain of 1.508 (composition 6) and 1.510 (composition 7), which is in the range experienced during processing studies.

With the above pressed densities, the linear shrinkage after sintering of the parts was predicted to be approximately 13%.

Figure 17 Drawing of the as-pressed valve blank.

1.2 Sintering

During the sintering of the valves, in addition to material densification, a major requirement was to maintain the straightness of the valve stem, keep the stem perpendicular to the valve face, and avoid any distortions on the valve head. Due to the high additive contents of compositions 6 and 7 and a high length-to-diameter ratio of the parts, this could not be attained without the use of sintering fixtures.

Sintering fixture development involved two iterations.

After adjusting the sintering run parameters, valves of both compositions were sintered. Composition 6 valves were sintered in the production furnace and composition 7 valves in the R&D furnace. Both materials densified, and the valve stem straightness and perpendicularity to the head face could be maintained. A group of sintered composition 7 valves is illustrated in Figure 18.

Figure 18 Sintered exhaust valve blanks, composition 7.

1.3 Sintered Valve Evaluation

Densified valves of both compositions were inspected dimensionally and dye checked for possible cracks.

To illustrate dimensional control achieved, Table 22 presents the dimensions measured on individual parts. It should be noted that parts listed in Table 18 have been sintered in different runs. Based on Table 22 results, dimensional control of as-sintered valves can be kept to about 1.0-1.5% for each of the dimensions. The stem perpendicularity of the parts was better than $\pm 1^\circ$. The major contribution to the dimensional variation was found to be the variability in the sintering weight losses between parts (typically 3-5%).

In addition to dimensional inspection, parts were dye checked in the as-sintered condition for cracks and none were found. Small amounts of dye were absorbed by the surface porosity on the parts, which is typical for all gas pressure sintered parts. Two or three parts from each of the compositions experienced a severe porous reaction layer in the top portion of the stem area. This was attributed to packing powder falling into the fixture during loading.

Table 22 Sintered valve blank dimensions.

Dimension	Perpendicularity (° ')		Length (in.)		Head Diameter (in.)		Stem Diameter (in.)	
Composition	6	7	6	7	6	7	6	7
1	90°10'	90°05'	5.379	5.292	1.398	1.363	.385	.385
2	90°46'	89°50'	5.421	5.319	1.403	1.365	.390	.387
3	90°49'	90°14'	5.388	5.287	1.396	1.365	.387	.384
4	90°14'	90°10'	5.388	5.286	1.397	1.365	.385	.384
5	89°36'	90°20'	5.406	5.307	1.401	1.371	.387	.386
6	89°50'	89°49'	5.383	5.299	1.397	1.363	.386	.386
7	89°56'	89°57'	5.383	5.310	1.395	1.364	.387	.385
8	89°19'	90°03'	5.361		1.394		.386	
9	90°40'		5.388		1.400		.386	
10	89°48'							
Nominal Dimension	90°	90°	5.320±0.030	5.320±0.030	1.379±0.007	1.379±0.007	0.376-0.005+0.01	0.376-0.005+0.010
Average	90°08'	90°03'	5.388	5.300	1.397	1.365	0.3866	0.3853
Standard Deviation	0°32'	0°11'	0.016	0.013	0.003	0.003	0.0015	0.0011

1.4 Material Property Confirmation

1.4.1 Strength at Room Temperature

Room temperature strength of 10 as-machined bars from composition 6 valve stems was evaluated (Table 23). Five valves (2 bars per valve), with each of the valves sintered in a different sintering run under identical conditions, were used. The strengths in Table 22 are comparable to the optimized as-machined bar strength of composition 6 material (see Table 17).

Table 23 Comparison of valve stock material (composition 6) properties to the same material properties evaluated in TASK I

Property (Composition 6)	TASK II	TASK I
Average Strength (Mpa)	654	602
Hardness, HV5 (kg/mm ²)	1480-1550	1520
Toughness (MPa•m ^{1/2})	6.2-7.0	6.5
Phase Composition	$\beta\text{-Si}_3\text{N}_4 + \text{Y}_{4.67}(\text{SiO}_4)_3\text{O}$	$\beta\text{-Si}_3\text{N}_4 + \text{Y}_{4.67}(\text{SiO}_4)_3\text{O}$
Microstructure	Acicular $\beta\text{-Si}_3\text{N}_4$, 1-5 μm in diameter	Acicular $\beta\text{-Si}_3\text{N}_4$, 1-5 μm in diameter

1.4.2 Phase Composition, Microstructure, Hardness and Toughness

X-ray diffraction on valve sections confirmed that the second phase in both valve materials was crystallized and it was identified as $\text{Y}_{4.67}(\text{SiO}_4)_3\text{O}$ in all cases (Table 23).

Optical microstructure was found to be comparable to the optimized composition 6 and 7 materials (see Figure 9). Hardness and toughness of both valve materials were also in ranges previously measured (see Table 23, Figures 11 and 12).

TASK II SUMMARY

It has been demonstrated that both compositions 6 and 7, developed under Task I, can be used to manufacture near-net-shape exhaust valve blanks using gas-pressure sintering of reaction bonded silicon nitride material. Fourteen valve blanks of composition 6 and nine of composition 7 have been delivered to ARL-Watertown as demonstration parts.

Major material characteristics (strength, phase composition, microstructure, hardness, toughness) of the manufactured valves were comparable or slightly better than measured for these materials in Task I.

CONCLUSIONS

The major goals of this development program have been accomplished.

A process was developed whereby a material with the composition of 85.8 mol % Si_3N_4 - 4.73 mol % Y_2O_3 - 9.47 mol % SiO_2 could be processed with properties identical to those that Gazza presented in his original paper. This process was based on the sintered reaction bonded silicon nitride process and involved the following process steps: spray drying, isostatic pressing, binder removal, argon sintering, green machining, nitriding, and gas pressure sintering. This process was capable of producing rods 1 inch in diameter and 7 inches long or billets 3.5x3.5x1.0 inches in size.

A data base of the properties of the material was generated. The microstructural properties, grain size, phase composition, etc., achieved the program goals. The strength and high temperature properties fell short of the objectives because of porosity 10-30 μm in size. These results indicate that more development work is required before these compositions are capable of operating in a heat engine application.

An engine component, an engine exhaust valve blank, was fabricated using the process developed in this program. This demonstration showed that: (1) the properties obtained in the characterization program could be reproduced in a production component, and (2) the dimensions of the valve blank were within $\pm 1.5\%$ of the predicted dimensions. The high and somewhat uncontrolled weight loss of the system during sintering precluded the attainment of higher dimensional control.

The limiting feature of this system is the high degree of reactivity between SiO_2 , Mo_2C , and Si_3N_4 during sintering which generates gaseous species. This gas generation, which occurs prior to and during final densification, contributes to the strength limiting microporosity remaining in the samples. This is also the principal cause of the relatively high dimensional variation of the demonstration component.

The high temperature properties were limited by the relatively low room temperature strength; however, the results were still superior on an absolute basis to many systems containing Y_2O_3 and Al_2O_3 as densification aids. Improvements could be expected if the microstructure control and the overall purity system could be increased.

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