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Evaluation of Silicon Nitride for Brayton Turbine Wheel Application

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Summary

Silicon nitride (Si_3N_4) is being evaluated as a risk-reduction alternative for a Jupiter Icy Moons Orbiter Brayton turbine wheel in the event that the Prometheus program design requirements exceed the creep strength of the baseline metallic superalloys. Five Si_3N_4 ceramics, each processed by a different method, were screened based on the Weibull distribution of bend strength at 1700 °F (927 °C). Three of the Si_3N_4 ceramics, Honeywell AS800, Kyocera SN282, and Saint-Gobain NT154, had bend strengths in excess of 87 ksi (600 MPa) at 1700 °F (927 °C). These were chosen for further assessment and consideration for future subcomponent and component fabrication and testing.

Introduction

Recently, a coupled fluid-structural analysis of the Brayton rotating unit (BRU) turbine wheel concluded that because of the low probability of failure predicted for a silicon nitride (Si_3N_4) wheel at the highest fluid-induced stress location, a properly designed Si_3N_4 rotor could be successful (ref. 1). Thus, Si_3N_4 was chosen as a back-up material for the turbine wheel of the Jupiter Icy Moons Orbiter (JIMO) Brayton cycle nuclear engine. In this application, the component will operate at a maximum temperature of 1700 °F (927 °C) and a maximum stress of 40 ksi (276 MPa), in He-Xe (trace O_2) for >100 000 hr. The temperature and stress conditions are easily achieved with this material, but there are no reports in the literature about the long-term behavior, especially creep (slow crack growth), under these conditions. Reference 1 also emphasizes that a ceramic rotor cannot be directly substituted for a metal part mostly because of ceramic manufacturing limitations, particularly blade thickness.

With manufacturing limitations in mind, the evaluation of five state-of-the-art Si_3N_4 ceramics has begun. Each material has been processed differently by its manufacturer, and thus each represents a different way the turbine wheel can be made. The materials are briefly described in table I. To narrow the choice of materials for long-term testing, the strength of each material was determined by a four-point bend test at 1700 °F (927 °C). There is no existing bend strength data in the literature for any of these materials at 1700 °F (927 °C). A statistical population of each material was tested so that a Weibull modulus could be reliably calculated. The Weibull distribution function, $F = 1 - \exp[-(\sigma_f/\sigma_0)^m]$, where F is the cumulative probability of fracture, σ_0 is the characteristic strength, m is the Weibull modulus, and σ_f is the fracture strength, is used to describe a population density distribution. A larger m indicates a lower scatter or dispersion in the Weibull distribution and a greater material reliability. Thus, desirable materials would have both high strength and high Weibull modulus.

The purpose of this work is twofold. The first is to screen a larger number of candidate materials using a simple, inexpensive test and to choose the best material(s) for the more expensive, long-term sustained tensile-load testing. The second is to determine the baseline properties of the materials at 1700 °F (927 °C).

TABLE I.—CANDIDATE SILICON NITRIDE MATERIALS

Vendor	Material	Designation	Process
Honeywell Ceramic Components	Si_3N_4	AS800	Gas-pressure sintered (GPS)
Ceradyne, Inc.	Si_3N_4	147–31E	Sintered, reaction bonded (SRBSN)
Boride Products	SiAlON	TK4	Sintered
Kyocera Industrial Ceramics	Si_3N_4	SN282	GPS
Saint Gobain Ceramics & Plastics, Inc.	Si_3N_4	NT154	Hot isostatically pressed (HIPed)

Procedures

All high-temperature flexural strength testing adhered to both the ASTM C 1211 and MIL–STD–1492(MR) protocols (ref. 2). Thirty specimens of each of the materials listed in table I were longitudinally ground with a 400-grit diamond wheel to 3 mm high by 4 mm wide by 45 mm long. All long edges had a 0.12-mm bevel. The specimens were heat treated in air at 2100 °F (1150 °C) to relieve residual stress and heal machining damage. The densities of each specimen were determined by dividing the measured weight by the calculated volume. Four-point bend strength was determined at 1700 °F (927 °C) at a crosshead rate of 0.5 mm/min. The inner and outer spans of the flexure fixture were 20 and 40 mm, respectively.

Selected failed specimens of each material were mounted with the two tensile surfaces glued together, and fractography was performed in a scanning electron microscope (SEM). Representative virgin specimens of each material were vacuum-mounted in epoxy; some were polished only and some were polished followed by plasma etching to evaluate their respective microstructure. Both the polished and the polished and etched specimens were examined by both SEM and a field emission scanning electron microscope (FESEM). Selected specimens were examined by energy dispersive analytical x-ray (EDAX) while in the FESEM to discern localized variation in chemical composition.

Results and Discussion

Weibull parameters were determined by the linear regression method from $\ln\ln(1/(1-F)) = m\ln\sigma_f - m\ln\sigma_0$ as shown in figure 1, where the Weibull modulus is obtained directly from the slope and the characteristic strength is calculated from the intercept.

Based upon this initial analysis, the Kyocera SN282 (Kyocera Industrial Ceramics Corp., Vancouver, WA), Honeywell AS800 (Honeywell Ceramic Components, Torrance, CA), and Saint-Gobain NT154 (Saint-Gobain Ceramics & Plastics, Inc., Northborough, MA) had significantly greater average strengths and higher Weibull moduli than the materials from Boride Products (Boride Products, Traverse City, MI) and Ceradyne (Ceradyne, Inc., Costa Mesa, CA). Clearly, the Boride Products material had an average strength below the required 276 MPa. The Weibull modulus of the Ceradyne material (15.89) was a little more than half that of the AlliedSignal material (28.12) and less than half that of the current ductile metal competitors (~40).

Honeywell AS800

AS800 is an in-situ-reinforced silicon nitride produced by gas-pressure liquid-phase sintering with La_2O_3, Y_2O_3, and SrO additives followed by heat treatment (ref. 3). The measured average density of this material was 3.63 g/cm^3 (standard deviation = 0.004). As shown in figure 2(a), the material had a relatively uniform distribution of fine (<5 μm) porosity throughout. At higher magnification, figure 2(b), the typical acicular, interlocking grain structure of an in-situ-reinforced Si_3N_4 is apparent. This structure is due to the α- to β-Si_3N_4 dissolution-reprecipitation sintering mechanism and results from subsequent anisotropic growth of the resultant hexagonal β-Si_3N_4 grains to maximize the low-energy (100) prismatic planes (ref. 3). Lin et al. have described the grain structure to consist of ~80 percent equiaxed, ~0.5-μm

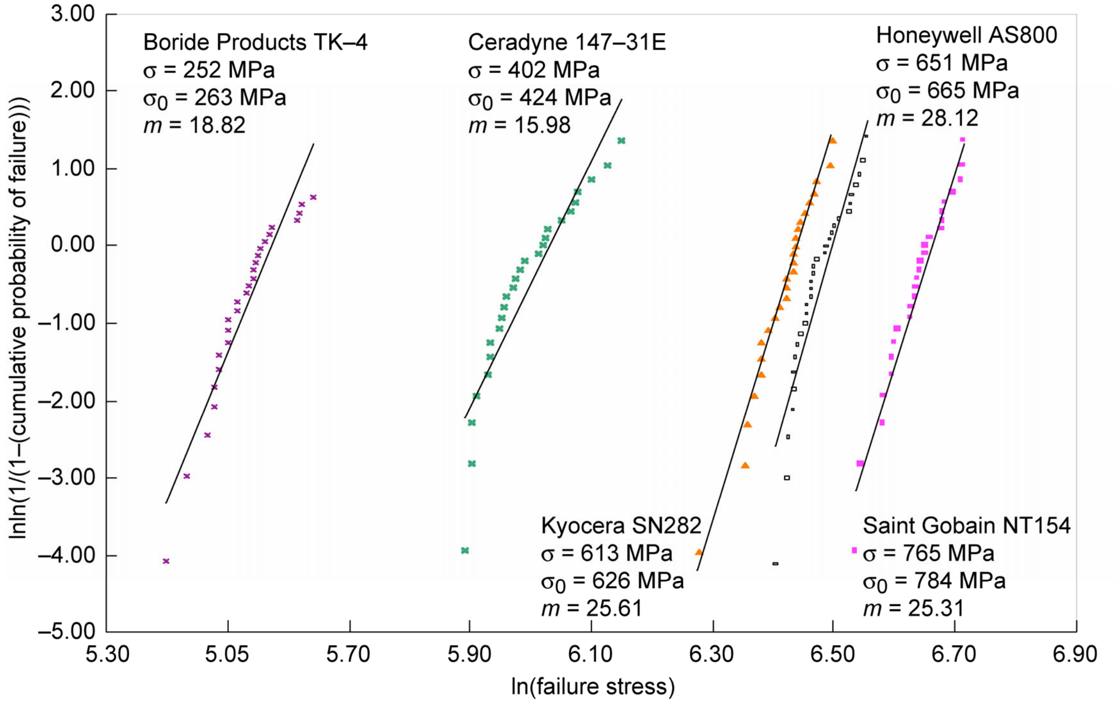

Figure 1.—Weibull plot of five candidate silicon nitride materials after flexure testing in air at 1700 °F (927 °C).

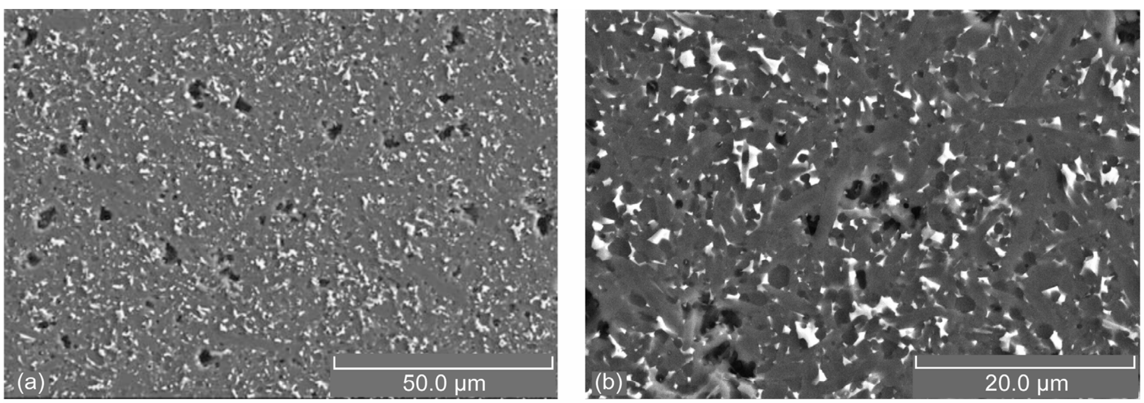

Figure 2.—Field emission scanning electron micrograph of Honeywell AS800 silicon nitride, typical plasma-etched and polished section. (a) Lower magnification showing uniform distribution of fine porosity. (b) Higher magnification showing acicular, interlocking grain structure.

grains, and ~20 percent acicular, 1.5- to 2-μm grains with aspect ratios from 5 to 12 (ref. 4). One benefit of this microstructure compared to fine-grained Si_3N_4 is relatively high fracture toughness, ~8 MPa·m$^{1/2}$ (ref. 5). The EDAX examination of the surface reveals a glassy grain boundary phase containing La, Y, Si, O, and N (fig. 3). Others have identified two crystalline phases, $La_5Si_3O_{12}N$ and either $Y_5Si_3O_{12}N$ (ref. 3) or $Y_{10}Si_7O_{23}N$ (ref. 4), within the grain boundary. This material typically failed from either a surface pore or an exaggerated grain at the surface. A typical fracture origin, a ~100- by 11-μm Si_3N_4 grain, is shown in figure 4(a) and (b). Room-temperature flexural strengths have been reported to be ~800 MPa with a Weibull modulus of 21 (refs. 5 and 6). Reference 6 indicates that the flexure strength at 1700 °F (927 °C) obtained in this study is in agreement with the extrapolated values obtained for AS800 made in 1995. Stepped stress rupture results at 982 °C (essentially no strength degradation at stresses up to 450 MPa for up to 1000 hr) in that same reference provided encouraging justification for continued creep characterization of this material for this application.

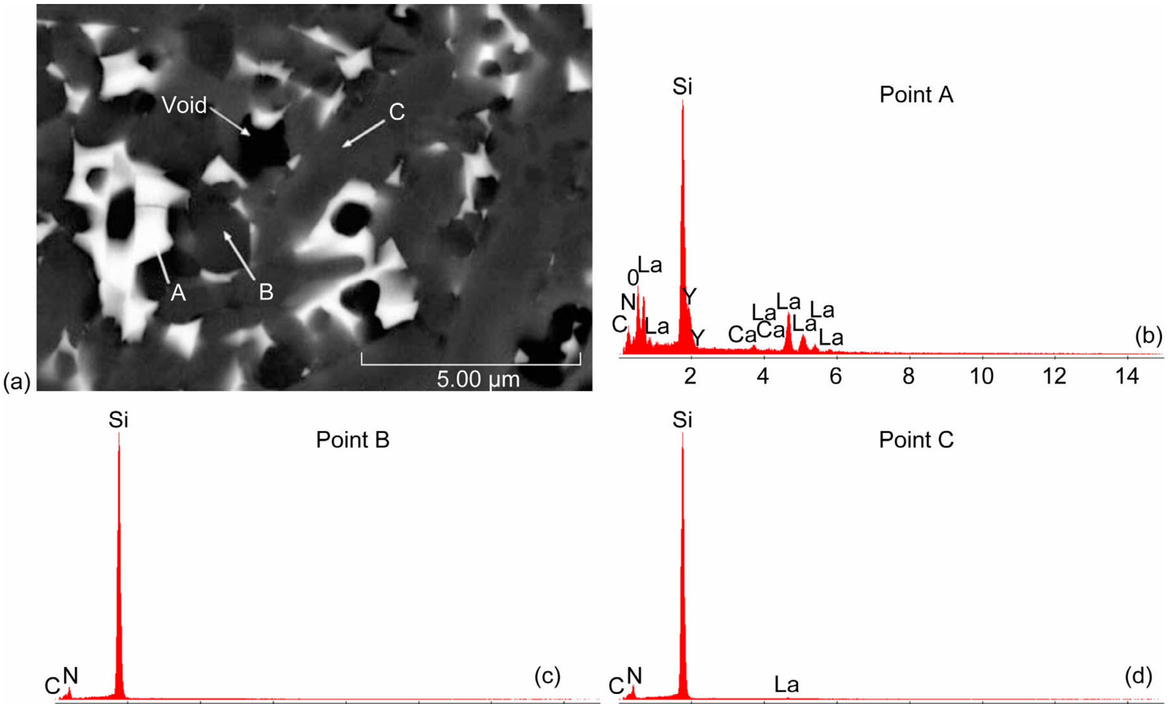

Figure 3.—Microstructural analysis of Honeywell AS800 silicon nitride. (a) Field emission scanning electron micrograph of typical plasma-etched and polished section. (b) Energy dispersive analytical x-ray (EDAX) spectrum of point A. (c) EDAX spectrum of point B. (d) EDAX spectrum of point C.

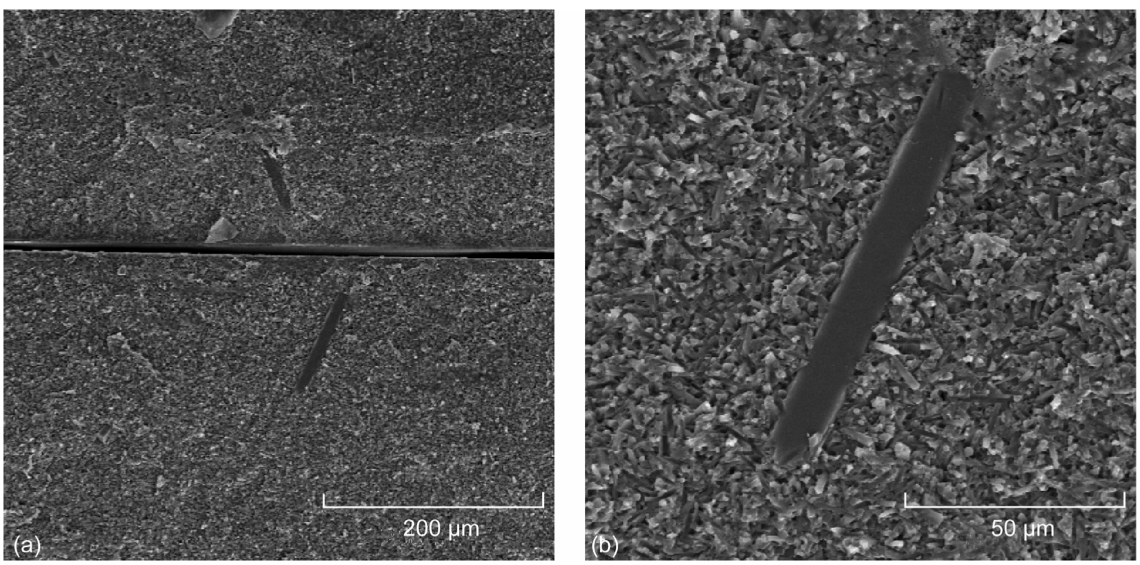

Figure 4.—Scanning electron micrograph of Honeywell AS800 silicon nitride, typical fracture origin. (a) Lower magnification of two halves of failed specimen. (b) Higher magnification of large grain that initiated fracture.

Kyocera SN282

SN282 is another in-situ-reinforced silicon nitride. It is produced by gas-pressure liquid-phase sintering with Lu_2O_3 additive followed by heat treatment (ref. 7). The average density of the material in this study was 3.38 g/cm^3 (standard deviation = 0.005). As shown in figure 5, the material had a relatively uniform distribution of fine (<5 µm) porosity throughout, similar to AS800. Also apparent in figure 5

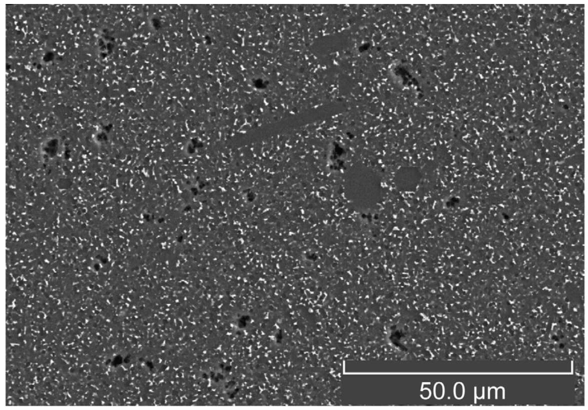

Figure 5.—Field emission scanning electron micrograph of Kyocera SN282 silicon nitride, typical plasma-etched and polished section, showing small, dispersed pores and network of acicular, intertwined grains.

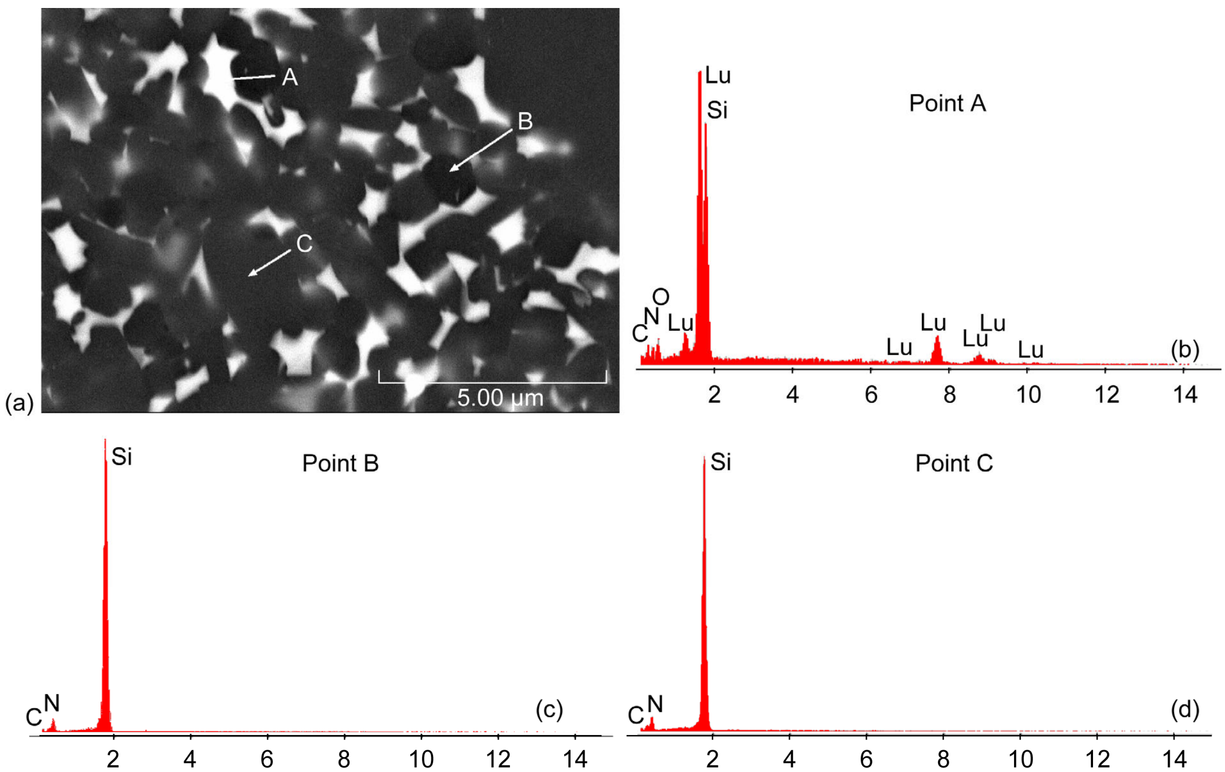

Figure 6.—Microstructural analysis of Kyocera SN282 silicon nitride. (a) Field emission scanning electron micrograph of typical plasma-etched and polished section. (b) Energy dispersive analytical x-ray (EDAX) spectrum of point A. (c) EDAX spectrum of point B. (d) EDAX spectrum of point C.

is the typical acicular, interlocking grain structure of this in-situ-reinforced Si_3N_4. Compared to AS800, SN282 has significantly fewer and larger acicular grains, resulting in a much finer overall microstructure and finer distribution of intergranular glass phase. The EDAX examination of the surface reveals a glassy grain boundary phase containing Lu, Si, O, and N (fig. 6). This is consistent with two crystalline phases, $Lu_2Si_2O_7$ and Lu_2SiO_5 (ref. 7), within the grain boundary. This material typically failed from either a surface pore or an exaggerated grain (or both) at the surface. One such failure origin, both a grain and a pore, is shown in figure 7. Room temperature flexural strengths have been reported to be 595 MPa with a

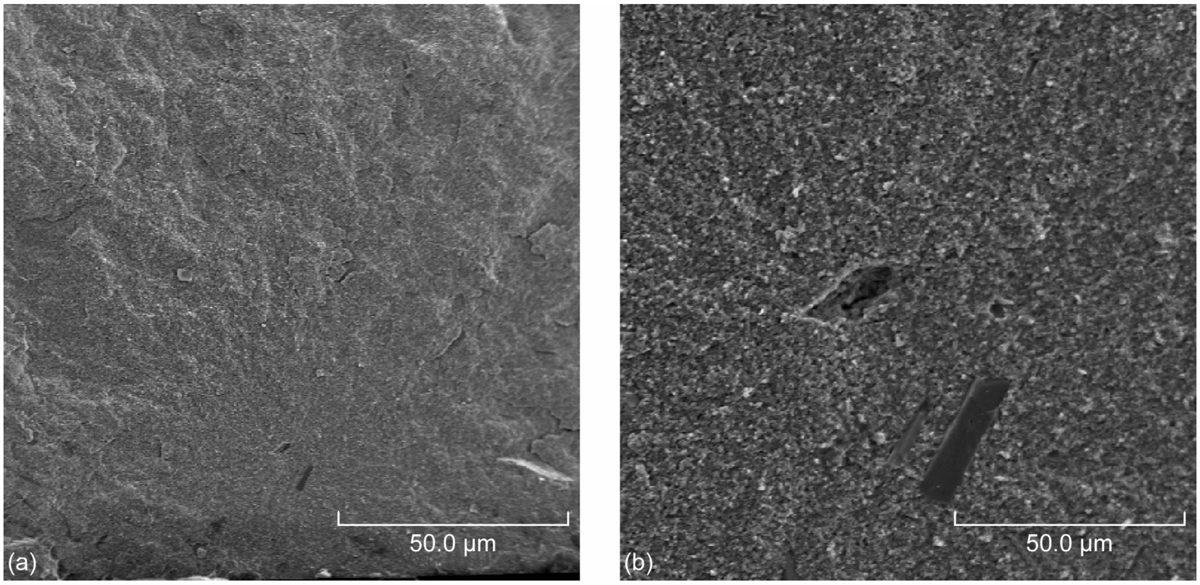

Figure 7.—Field emission scanning electron micrograph of Kyocera NT154 silicon nitride, typical fracture origin of pore and exaggerated grain. (a) Lower magnification. (b) Higher magnification.

Weibull modulus of 11 (ref. 5). Although this is not the highest strength material in the present study, SN282 remains interesting because of its reported improved creep and oxidation resistance at higher temperatures (refs. 8 and 9). It is not known whether these benefits will also be evident at 927 °C. Significantly, the strength does not decrease at 927 °C, which indicates a more refractory grain boundary phase in this material relative to the other materials in this study.

Saint Gobain NT154

NT154 is produced by hot isostatic pressing (HIP) of a glass-encapsulated powder followed by heat treatment. In this case the sintering additive is Y_2O_3, and the resulting grain boundary phase is $Y_2Si_2O_7$ (refs. 10 and 11). The average density of the material in this study was 3.22 g/cm^3 (standard deviation = 0.005). As evident in figure 8, this material departs from the conventionally sintered materials, AS800 and SN282, with a finer microstructure devoid of highly elongated grains. Figure 9 also clearly shows small and dispersed pores, pore clusters, and extremely fine equiaxed grains that fill the space between relatively small acicular grains. There also appears to be substantially less glassy phase, as would be expected from a HIPed material. In contrast to the sintered materials, this microstructure contributes to significantly lower fracture toughness, 5.5 to 6.0 MPa·m$^{1/2}$ (2005, Saint-Gobain Ceramics & Plastics, Inc., Northborough, MA, product literature). Failure in this material can be attributed to pore clusters (fig. 10(a)) and surface machining defects (fig. 10(b)). Room temperature flexural strength is reported to be 900 to 1000 MPa with a Weibull modulus of 10 to 21 (refs. 11 and Saint-Gobain product literature). At 982 °C, the strength drops to 740 MPa with a Weibull modulus of 13 (Saint-Gobain product literature). This reported strength is in agreement with the strength measured in this study, but the modulus determined in this study is significantly higher. The manufacturer also reports a tensile creep rate at 1260 °C of 1.9×10^{-8} s^{-1}, which is very encouraging for our lower temperature application.

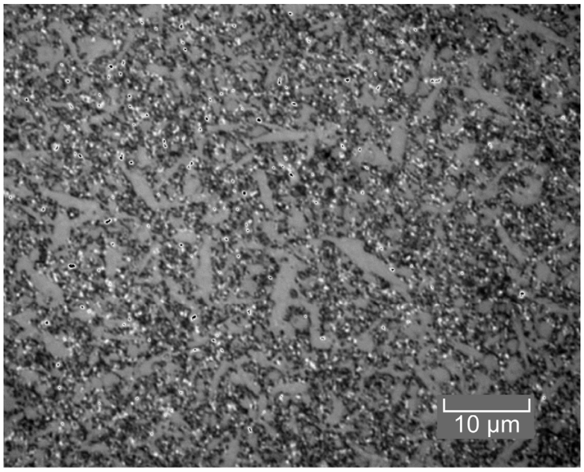

Figure 8.—Optical micrograph of Saint-Gobain NT154 silicon nitride, typical plasma-etched and polished section, showing very small grains and larger, acicular grains.

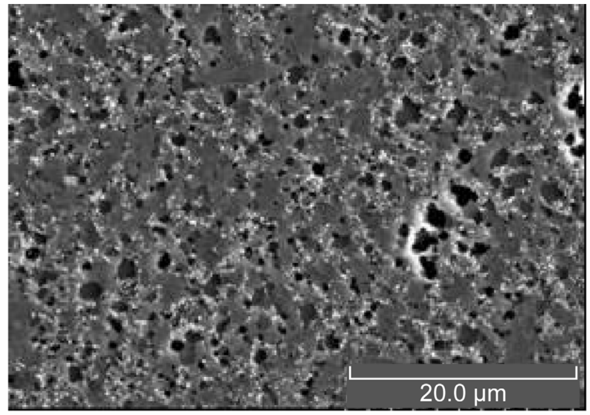

Figure 9.—Field emission scanning electron micrograph of Saint-Gobain NT154 silicon nitride, typical plasma-etched and polished section, showing fine, dispersed porosity and one larger pore cluster.

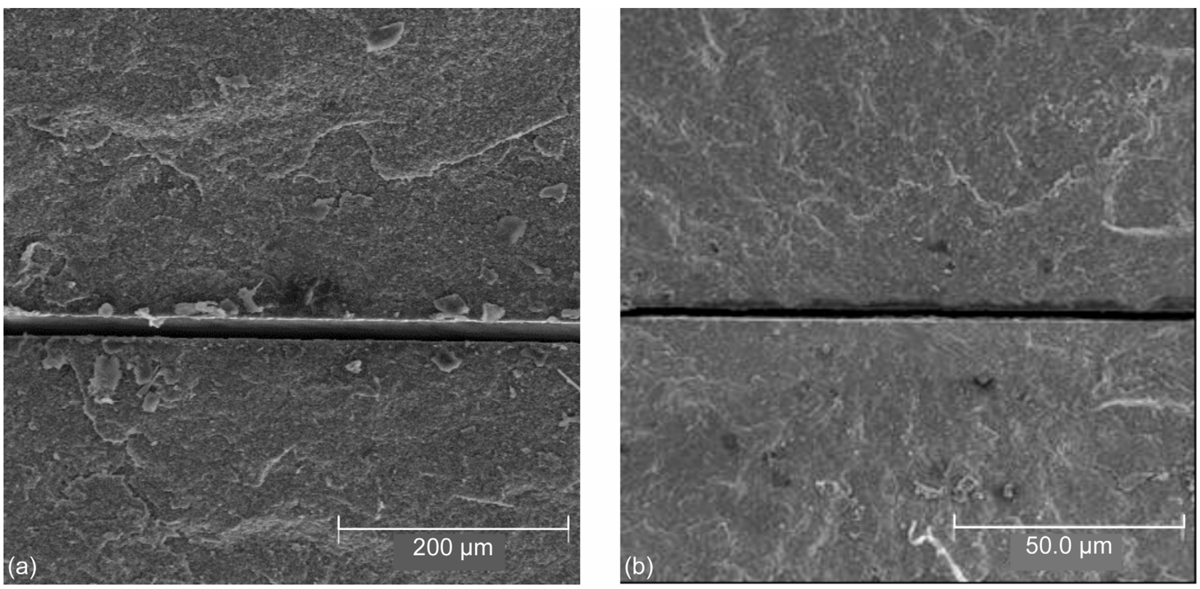

Figure 10.—Scanning electron micrograph of Saint-Gobain NT154 silicon nitride, typical fracture origins. (a) Fracture due to large subsurface pore cluster. (b) Fracture due to machining damage.

Ceradyne 147–31E

The Ceradyne material is made by a sinter reaction bonding (SRBSN) process. Silicon powder is mixed with sintering aids, Y_2O_3 and Al_2O_3 in this case, and formed into a shape. The green preform is then nitrided in a nitrogen atmosphere at temperatures up to 1400 °C. This fabrication process is the easiest and most cost-effective means for making a complex net-shape Si_3N_4 part. The average density of the material in this study was 3.19 g/cm^3 (standard deviation = 0.045). Like the other liquid sintered materials, AS800 and SN282, 147–31E exhibits a relatively uniform distribution of fine (<5 µm) pores and an acicular grain structure (fig. 11). This material has significantly more pores and large acicular grains. The glass phase is less well dispersed, with large pockets of glass dispersed throughout the material (fig. 12). Even though this material has a microstructure similar to AS800 and SN282, the

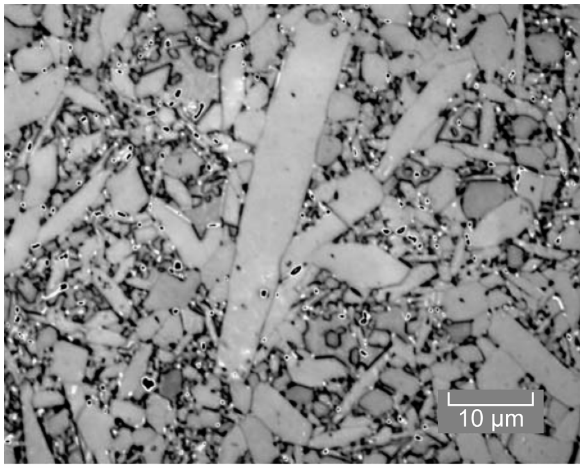

Figure 11.—Optical micrograph of Ceradyne 147–31E silicon nitride, typical plasma-etched and polished section, showing range of grain sizes and shapes.

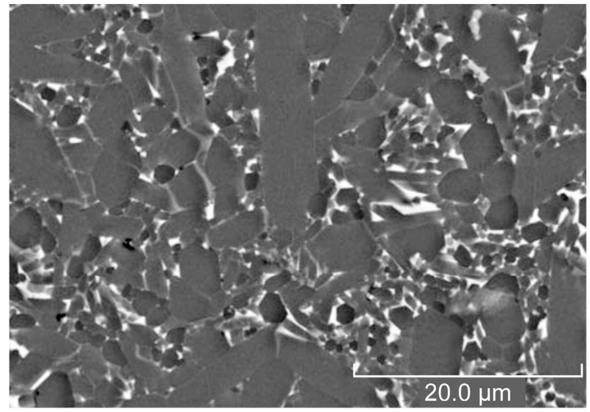

Figure 12.— Field emission scanning electron micrograph of Ceradyne 147–31E silicon nitride, typical plasma-etched and polished section, showing distribution of glassy phase (white).

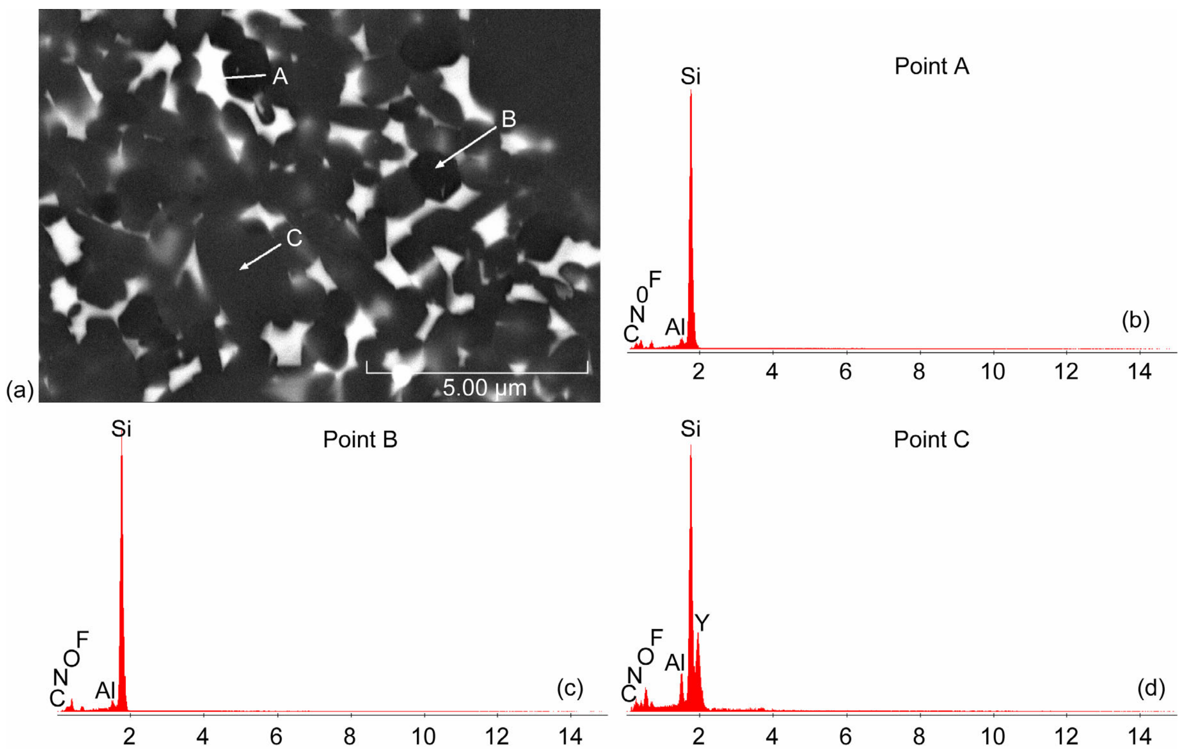

Figure 13.—Microstructural analysis of Ceradyne 147–31E silicon nitride. (a) Field emission scanning electron micrograph of typical plasma-etched and polished section. (b) Energy dispersive analytical x-ray (EDAX) spectrum of point A. (c) EDAX spectrum of point B. (d) EDAX spectrum of point C.

manufacturer reports a fracture toughness of 6.0 MPa·m$^{1/2}$ (2003, Ceradyne, Inc., Costa Mesa, CA, product literature), which is similar to NT154. The EDAX examination of the surface reveals a glassy grain boundary phase containing Y, Al, Si, O, and N (fig. 13). Failure in this material occurred at large subsurface exaggerated acicular grains and large pore clusters (fig. 14). The manufacturer reports a room temperature flexural strength of 700 MPa with a Weibull modulus of 10 to 15 (Ceradyne product literature). A very similar Ceradyne material 147–31N has a reported flexural strength of 618 MPa with a Weibull modulus of 17.2 at 700 °C (ref. 12). This is much better than the values obtained at 927 °C in this study. This may indicate that the glass phase has softened significantly, even at 927 °C.

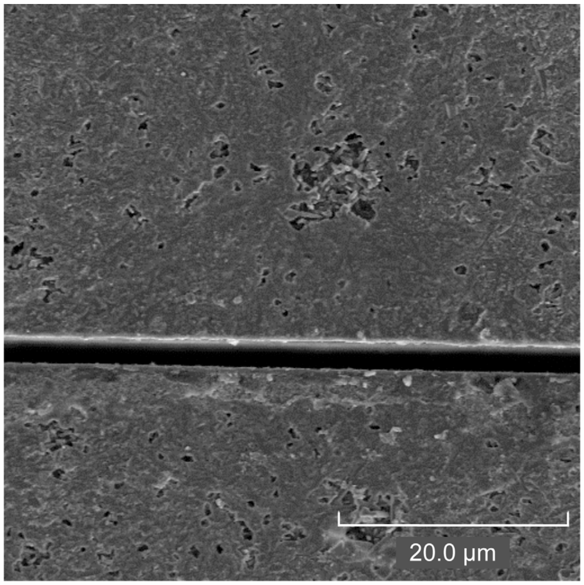

Figure 14.—Scanning electron micrograph of Ceradyne 147–31E silicon nitride, showing large pore cluster as fracture origin.

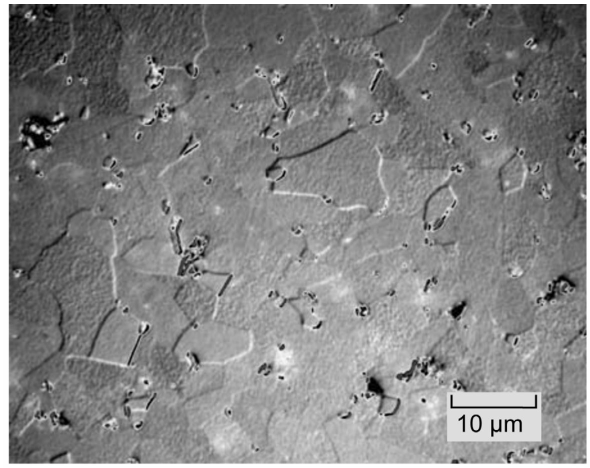

Figure 15.—Optical micrograph of Boride Products TK4 silicon nitride, typical plasma-etched and polished section, showing large, grain-like patches that result from etching process.

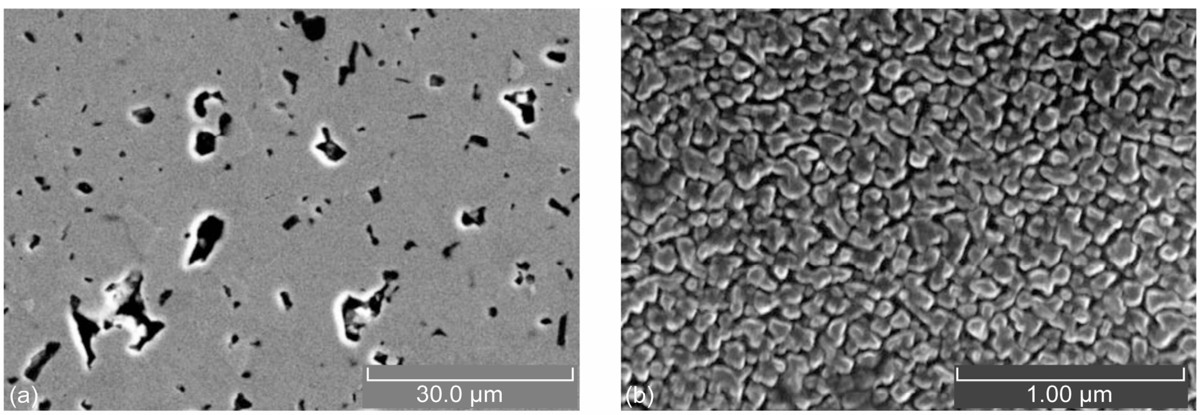

Figure 16.—Field emission scanning electron micrograph of Boride Products TK4 silicon nitride, typical plasma-etched and polished section. (a) Lower magnification showing large (>5 μm) pores. (b) Higher magnification showing very fine grain size (<100 nm).

Boride Products TK4

TK4 is the only SiAlON in this study. This is a sintered material that is a solid solution of Si, Al, O, and N. It consists of 40 percent α-SiAlON and 60 percent β-SiAlON (ref. 13). It is significantly less expensive to process than the other materials in this study. The average density of the material in this study was 3.47 g/cm^3 (standard deviation = 0.062). This material exhibited the largest variation in density of the materials studied. As shown in figure 15, TK4 is characterized by a significant amount of larger (<10 μm) porosity. Evident in the optical micrograph are grain-like patches of material that etch differently. At higher magnification, figure 16(a), both the porosity and patchiness are evident. Within the patchy areas, at very high magnification, is a very uniform, <100 nm, equiaxed fine structure (fig. 16(b)). The typical fracture origin shown in figure 17 is a large area of diffuse porosity that intersects with the tensile surface. Other fracture origins were large, elongated pores that intersected the tensile surface.

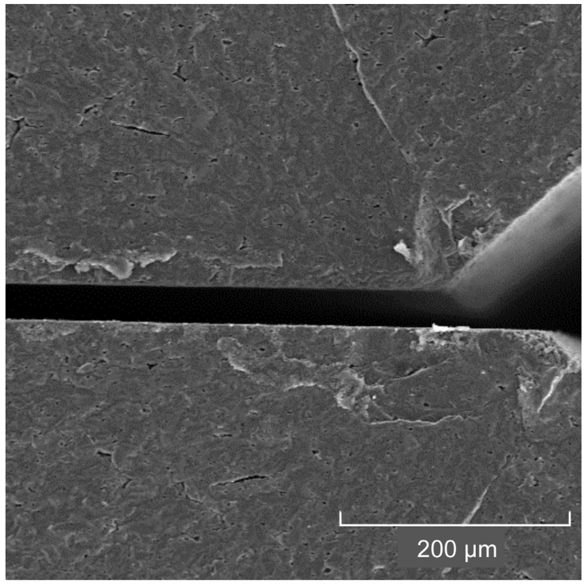

Figure 17.—Scanning electron micrograph of Boride Products TK4 silicon nitride, showing typical fracture origin of large pore intersecting tensile surface. Note large elongated pore away from origin.

These fracture origins are typically much larger (up to 50 μm) than the bulk of the porosity found throughout the material. This excessive porosity is responsible for the significantly lower strength of this material relative to the other materials. This is the only material with a strength less than that required for the application. It is possible that this batch of material had processing problems, since others have measured a room temperature flexural strength of 653 MPa with a Weibull modulus of 12.7 and a 700 °C strength of 691 MPa with a Weibull modulus of 23.9 (ref. 12).

Conclusion

Based upon the results shown in figure 1, the materials from Kyocera, Honeywell, and Saint-Gobain were chosen for further study. The Boride Products material had an unacceptable strength for this application, and the Ceradyne material had the lowest Weibull modulus.

It is recognized that a more thorough screening test is warranted for these materials. Clearly, calculating probability of failure under conditions closer to those to be encountered—276 MPa and 927 °C in He—would provide a more accurate determination of material suitability. Similarly, slow crack growth (SCG) measurements under the same conditions would provide better information about the assumed predominant failure mechanism. However, calculating probability of failure requires a representative component model, and SCG measurement in He is an expensive test that was not undertaken for that reason. Thus, the bend strength and Weibull modulus were chosen as the initial screen, assuming that materials that excel in those attributes will also perform better in probability of failure calculation and SCG measurement.

In the coming months, tensile creep-rupture testing of the Kyocera, Honeywell and Saint-Gobain materials will begin in both air and He-Xe for periods up to 100 000 hr. This will establish which material(s) to consider for future subcomponent and component fabrication and testing.

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13. SUPPLEMENTARY NOTES

14. ABSTRACT
Silicon nitride (Si3N4) is being evaluated as a risk-reduction alternative for a Jupiter Icy Moons Orbiter Brayton turbine wheel in the event that the Prometheus program design requirements exceed the creep strength of the baseline metallic superalloys. Five Si3N4 ceramics, each processed by a different method, were screened based on the Weibull distribution of bend strength at 1700 °F (927 °C). Three of the Si3N4 ceramics, Honeywell AS800, Kyocera SN282, and Saint-Gobain NT154, had bend strengths in excess of 87 ksi (600 MPa) at 1700 °F (927 °C). These were chosen for further assessment and consideration for future subcomponent and component fabrication and testing.

15. SUBJECT TERMS
Silicon nitrides; Weibull density functions; Turbine wheels; Brayton cycle; Sialon; Ceramics

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