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Assessments of Mechanical and Life Limiting Properties of Two Candidate Silicon Nitrides for Stirling Convertor Heater Head Applications

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January 2006

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Abstract

NASA Glenn Research Center is developing advanced technology for Stirling convertors with a target of significantly improving the specific power and efficiency of the convertor and overall generator for Mars rovers and deep space missions. One specific approach to the target has been recognized as the use of appropriate high-temperature materials. As a series of ceramic material approaches in Advanced Stirling Convertor Development Program in fiscal year 2005, two commercial, structural silicon nitrides AS800 and SN282 were selected and their mechanical and life limiting properties were characterized at 1050 °C in air. AS800 exhibited both strength and Weibull modulus greater than SN282. A life limiting phenomenon was apparent in AS800 with a low slow crack growth parameter $n = 15$; whereas, a much increased resistance to slow crack growth was found in SN282 with $n>100$. Difference in elastic modulus and thermal conductivity was negligible up to 1200 °C between the two silicon nitrides. The same was true for the coefficient of thermal expansion up to 1400 °C.

Introduction

NASA Glenn Research Center is developing advanced technology for Stirling convertors with a goal of substantially improving the specific power and efficiency of the convertor and overall generator. These advances could provide significant performance and mass benefits for Mars rovers and deep space missions and could be allow the use of Stirling radioisotrope power systems for radioisotrope electric propulsion and Venus missions (ref. 1). Performance and mass improvement targets have been established and tasks are underway to fulfill these targets. One of approaches to the targets is to use higher-temperature materials such as superalloys, refractory metal alloys, and ceramic materials (ref. 2). Structural analysis of Stirling Convertor heater heads using superalloys has been made focusing on durability, reliability, and performance (refs. 3 and 4). Of those candidate materials, ceramics offer a number of significant advantages over metal counterparts including lower density, higher-temperature strength, more creep and life-limiting resistances, a wide range of thermal conductivities, and more resistance to corrosive environments. Some previous work for Stirling convertors has also shown that ceramic materials exhibited a better resistance to helium permeability compared to nickel-based superalloy IN718 (ref. 1). A schematic showing a Stirling convertor is presented in figure 1 (ref. 5). The heater head is a component considered to be used with potential structural ceramics.

In the fiscal year of 2005, a new work started to evaluate mechanical properties of two commercial candidate silicon nitrides AS800 and SN282 in Advanced Stirling Technology Program. The major effort was to generate important design data including basic mechanical properties, strength, life prediction parameters, and some other thermal properties such as thermal conductivity and coefficient of thermal expansion. This paper reports the recent results on these mechanical characterizations of the two

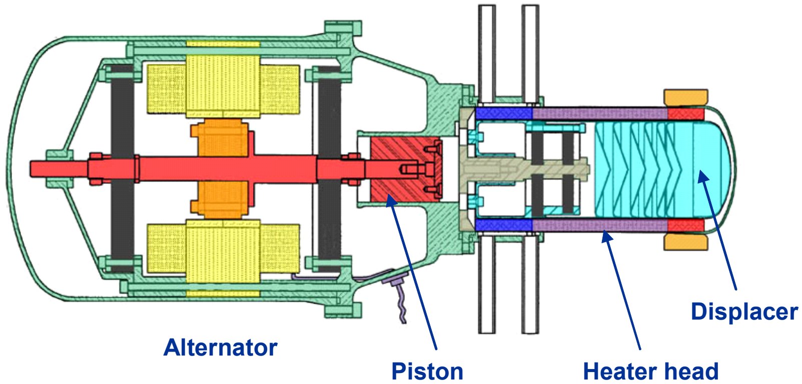


Figure 1.—Schematic of Stirling convertor showing a heater head (ref. 5).

candidate silicon nitrides at 1050 °C that was one of target temperatures set for Advanced Stirling convertors. The candidate advanced structural ceramics have been used in various propulsion programs at NASA Glenn for gas turbine engine applications at higher temperatures 1200 to 1371 °C (refs. 6 to 10).

Experimental Procedures

Materials

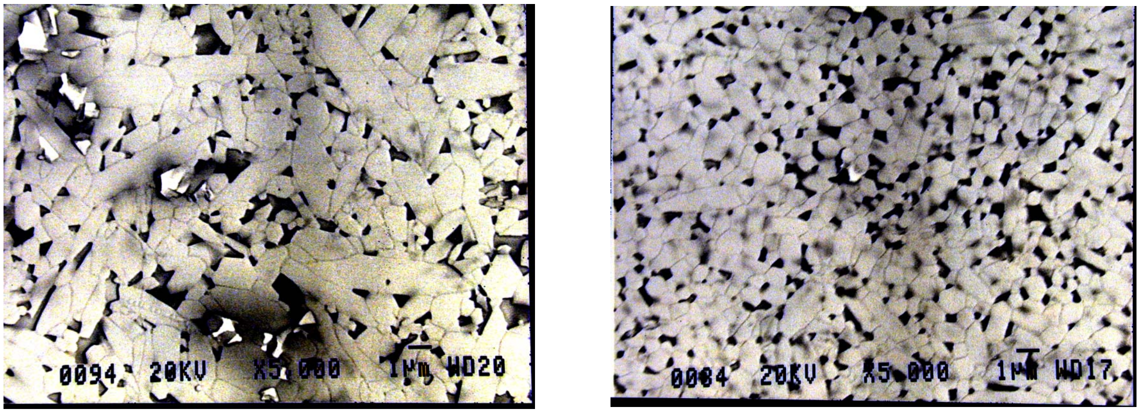
Materials used in this work were commercially available silicon nitrides AS800 (Honeywell, Torrance, California, '99 vintage, gel-cast) and SN282 (Kyocera, Vancouver, Washington, '00 vintage). These two silicon nitrides, both gas-pressure sintered, are currently considered as strong candidate materials for gas-turbine applications in view of their substantially improved elevated-temperature properties (refs. 11 to 13). Both materials are toughened silicon nitrides with microstructures tailored into elongated grain structures. The degree of elongation and the size of grains were greater in AS800 than SN282, as shown in figure 2. The billets for each material were machined into flexure test specimens measuring 3.0 by 4.0 by 45.0 mm, respectively, in thickness, width, and length. The final finishing was completed in a longitudinal direction of test specimens with a no. 500 diamond grinding wheel under the specified conditions in accordance with ASTM standard C1211 (ref. 14). Prior to testing, all AS800 test specimens were annealed at 1200 °C in air for 2 hr to eliminate or minimize damage and/or residual stresses presumably associated with machining. All SN282 test specimens were annealed by the manufacturer prior to testing with proprietary annealing condition.

Basic Mechanical and Physical Properties

Basic ambient-temperature mechanical and physical properties including uniaxial and biaxial flexure strength, elastic modulus, Poisson's ratio, density, Vickers hardness, fracture toughness, and R-curve have been determined from other programs. These basic properties and test methods related will be briefly described in the Results and Discussion section.

Flexure Strength Testing

Strength testing for both AS800 and SN282 flexure test specimens was performed at 1050 °C in air using a SiC four-point flexure fixture with 20-mm inner and 40-mm outer spans in accordance with ASTM C 1211 (ref. 14). An electromechanical test frame (Model 8562, Instron, Canton, Massachusetts) was used in load control with a load rate of 60 N/s which corresponds to a stress rate of 50 MPa/s. Each test specimen was held for about 20 min at 1050 °C for thermal equilibration before testing. A total of 20 test specimens were tested for each material. A limited fractographic analysis was performed after strength testing to determine failure origin and flaw configuration, etc.



(a) AS800 (b) SN282

Figure 2.—Microstructures of AS800 and SN282 silicon nitrides used in this work.

Dynamic Fatigue (Life Prediction; Life Assessment) Testing

Slow crack growth (SCG) behavior of brittle materials can be determined using dynamic fatigue (or called constant stress-rate) testing under a given environment/temperature condition. This test method, as specified in ASTM C 1368 (ref. 15) or C 1465 (ref. 16), determines flexure strengths with several different applied test rates. From the determined strength data as a function of applied test rate, slow crack growth or life prediction parameters of a material can be determined using an appropriate relation. Dynamic fatigue testing was performed in flexure at 1050 °C in air with both AS800 and SN282 silicon nitrides flexure test specimens. For AS800, three different test rates of 0.05, 0.005, and 0.0005 MPa/s were employed in load control of the electromechanical test frame (Model 8562, Instron, Canton, Massachusetts). A total of 20 test specimens were used at a test rate of 0.05 MPa/s while 10 test specimens were used at each of 0.005 and 0.0005 MPa/s. For SN282, two test rates of 0.05 and 0.0005 MPa/s were used with 15 and 10 specimens at respective test rates. To save test time particularly at the lowest test rate of 0.0005 MPa/s in which typical time to failure was around 200 to 300 hr, a preload technique to accelerate testing was applied (refs. 14, 15, and 17 to 19). Preloads ranging from 50 to 80 percent of the failure stress of the material determined at 0.0005 MPa/s were employed. Note that the flexure strength data obtained at 60 N/s (=50 MPa/s) from flexure strength testing were used as one set of the dynamic fatigue data for each material. The test specimen’s configuration and test fixture were the same as those used in flexure strength testing. Also, the thermal equilibration condition at dynamic fatigue testing was the same as that applied in flexure strength testing. Limited optical microscopy was conducted for fractographic analysis.

Elastic Modulus Measurements

Elastic modulus of both AS800 and SN282 silicon nitrides were determined as a function of temperature ranging from 25 to 1200 °C by the impulse excitation of vibration method, ASTM C 1259 (ref. 20), using a flexure specimen configuration with nominal dimensions of 3 by 4 by 45 mm. Output information such as resonance frequency was continuously recorded as a function of temperature via data acquisition incorporated with an automatic excitation system (Model MK5, GrindoSonic, J.W. Lemmens, Belgium). The scatter in elastic modulus of these silicon nitrides was observed negligibly small, typically with a coefficient of variation less than 2 percent (refs. 7 and 8), so that only one specimen was used for each material.

Thermal Properties: Thermal Conductivity and Coefficient of Thermal Expansion

Thermal conductivity of both AS800 and SN282 silicon nitrides was determined at temperatures ranging from 300 to 1200 °C using a diffusion-cooled 3.5 kW CO_2 laser high-heat flux rig (Type DC 035, Rofin, Hamburg, Germany). A disk specimen configuration was utilized, measuring 45 mm in diameter and 2 mm in thickness. The specimen surface heating was provided by the laser beam and the back-side air cooling was used to maintain desired specimen temperatures. A uniform laser power distribution was achieved over 24 mm diameter aperture region of the specimen by using an integrating ZnSe lens combined with specimen rotation. The test rig systems and general procedures for the thermal conductivity measurements have been described elsewhere (ref. 21). Thermal conductivity was determined from the pass-through heat flux and the measured temperature gradient through the specimen thickness under the steady-state laser heating conditions using a one-dimensional heat transfer model (ref. 21).

Thermal expansion experiments to determine the coefficient of thermal expansion (CTE) of two silicon nitrides were conducted using a dilatometer system (UNITHERM, ANTER Corp., Pittsburgh, Pennsylvania) in air at temperatures ranging from 25 to 1400 °C. The nominal dimensions of flexure test specimen were 3 by 4 by 25 mm (length). Longitudinal displacement of the specimen was continuously recorded during the heating/cooling cycle through a data acquisition system combined with an LVDT and temperature measuring devices (ref. 22).

Results and Discussion

Basic Mechanical and Physical Properties

Basic ambient-temperature mechanical and physical properties of both AS800 and SN282 silicon nitrides such as elastic modulus, Poisson's ratio, density, Vickers hardness, flexure (uniaxial and biaxial) strength, fracture toughness, and R-curve have been determined previously and are summarized in table 1 together with test methods used. Note that the R-curve for both silicon nitrides was estimated by the indentation technique (ref. 26) and can be expressed (ref. 27)

$$K_R = k[a]^q \tag{1}$$

where K_R is fracture resistance (MPa $m^{1/2}$), a is crack size (μm), and k and q are R-curve parameters. The values of k and q are listed in table 1.

TABLE 1.—BASIC MECHANICAL AND PHYSICAL PROPERTIES OF AS800 AND SN282 SILICON NITRIDES AT AMBIENT TEMPERATURE (REFS. 7 AND 9)

Material	Elastic modulus[1] E (GPa)	Poisson's ratio[1] ν	Density[2] ρ (g/cm^3)	Hardness[3] H (GPa)	Flexure Strength[4]				Fracture toughness[5] K_{Ic} (MPa√m)	R-curve Parameters[6]	
					Type of loading	Mean strength (MPa)	Weibull modulus	Characteristic strength (MPa)		k	q
AS800 Si_3N_4	309	0.27	3.27	13.6(1.4)	Uniaxial	775(45)	21	795	8.1(0.3)	3.02	0.13
					Biaxial	678(45)	18	698			
SN282 Si_3N_4	304	0.28	3.32	15.3 (0.2)	Uniaxial	595(64)	11	623	5.5(0.2)	2.56	0.10
					Biaxial	426(60)	8	451			

Notes:

1. By the impulse excitation technique, ASTM C 1259 (ref. 20)
2. By mass/volume method
3. By Vickers microhardness indentation, ASTM C 1327 (ref. 23)
4. Uniaxial testing: by four-point flexure with 20/40 mm spans with 3 by 4 by 45 mm bars, ASTM C 1161 (ref. 24) (Number of test specimens used: 20 for each material). Biaxial testing: by ring-on-ring configuration with 20/40 mm rings with 2-mm-thick and 45-mm-diameter disks (Number of test specimens used: 10 for AS800 and 21 for SN282).
5. By single-edge-precracked-beam (SEPB) method, ASTM C 1421 (ref. 25).
6. See the text for descriptions.

The numbers in the parentheses indicate ±1.0 standard deviations.

Flexure Strength

The two-parameter Weibull plots of flexure strength of both AS800 and SN282 silicon nitrides determined at 1050 °C in air are shown in figure 3, where lnln[1/(1-*F*)] was plotted as a function of lnσ_f with *F* and σ_f being failure probability and flexure strength, respectively. Weibull modulus *m* and characteristic strength σ_θ were *m* = 25 and σ_θ = 611 MPa for AS800. For SN282, *m* = 14 and σ_θ = 515 MPa. The mean strength was 598±28 MPa for AS800 and 496±43 MPa for SN282. The Weibull modulus for AS800 and SN282 (*m* = 25 and 14, respectively) at 1050 °C compares well with the values determined at ambient temperature (*m* = 18 to 21 and 8 to 11, respectively, see table 1) and at 1316 °C (*m* = 24 and 12, respectively) for AS800 and SN282 (ref. 28), all with the similar number (= 20) of test specimens used at each test temperature. Failure origins of both silicon nitrides, in many cases, were associated with surface-related defects such as machining flaws, pores, and elongated grains. Typical examples of fracture surfaces are shown in figure 4.

Figure 5 shows a summary of mean flexure strength as function of test temperature, combined with the previously determined strength data at different temperatures (refs. 6, 7, and 28). A general trend was observed for SN282 such that strength decreased monotonically with increasing temperature, as observed in many advanced ceramics. By contrast, AS800 exhibited a strength drop at 1050 °C, deviating from the trend in strength with regard to temperature. The reason is not clear yet; however, it is speculated that intermediate-temperature oxidation might have caused such a strength drop for AS800. It has been reported that intermediate-temperature oxidation around 700 to 1100 °C was responsible for both strength degradation and enhanced slow crack growth for some silicon nitride ceramics (ref. 29).

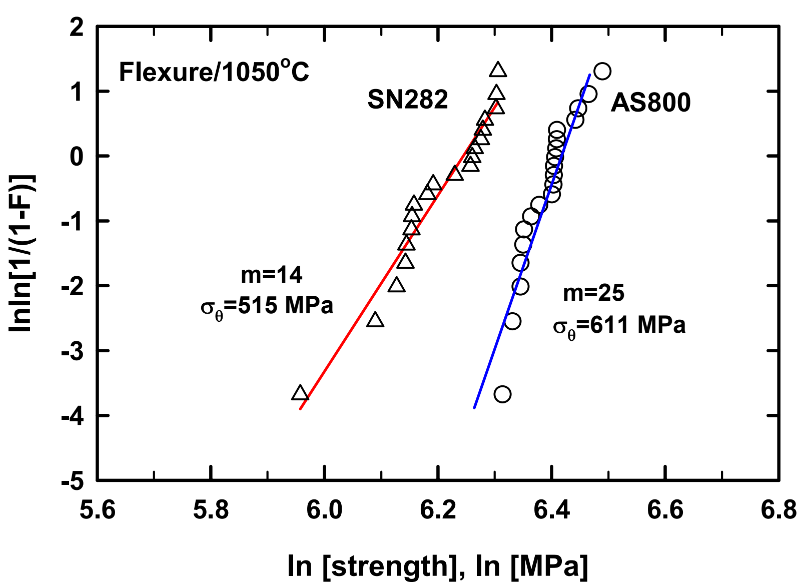
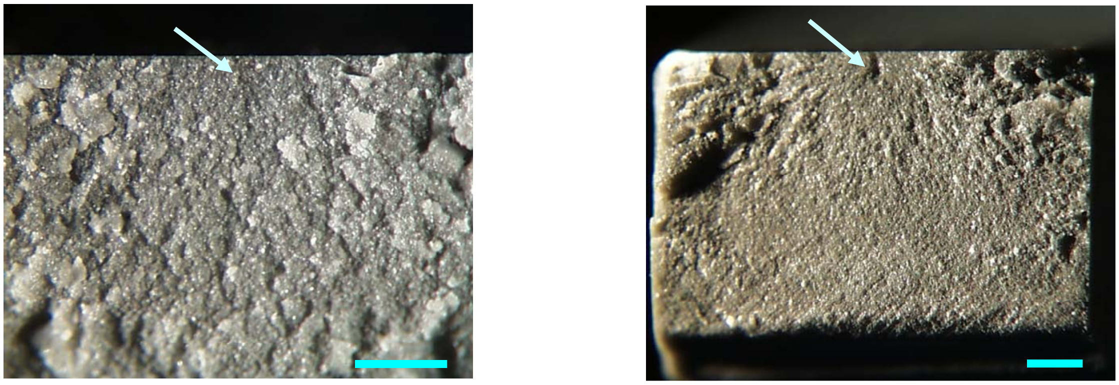


Figure 3.—Weibull distributions of flexure strength determined at 1050 °C in air for AS800 and SN282 silicon nitrides. *m* = Weibull modulus, σ_θ = characteristic strength and *F* = failure probability.



(a) AS800 (b) SN282

Figure 4.—Typical fracture surfaces of specimens tested at 50 MPa/s in flexure at 1050 °C in air: (a) AS800 and (b) SN282 silicon nitrides. Volume flaws located to the top surfaces were responsible for failure, with flaw origins indicated with arrows. Bars in 500 μm.

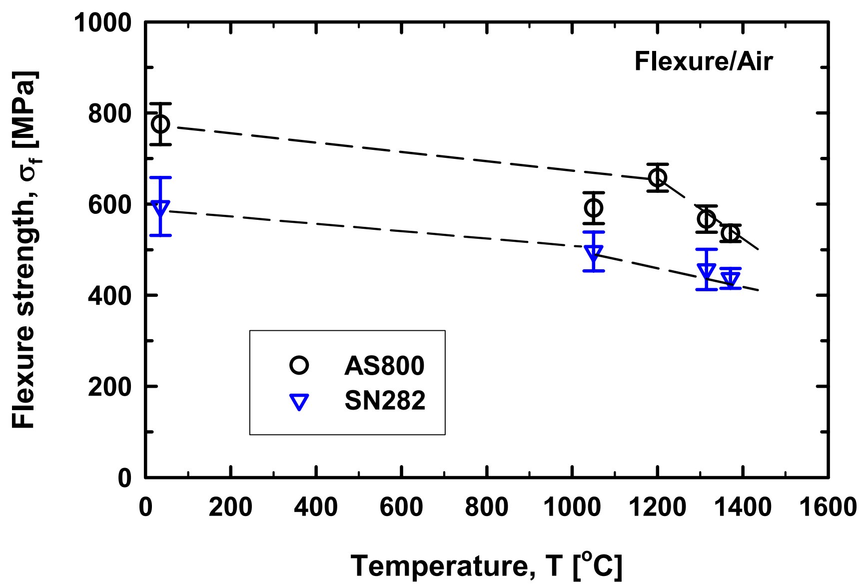
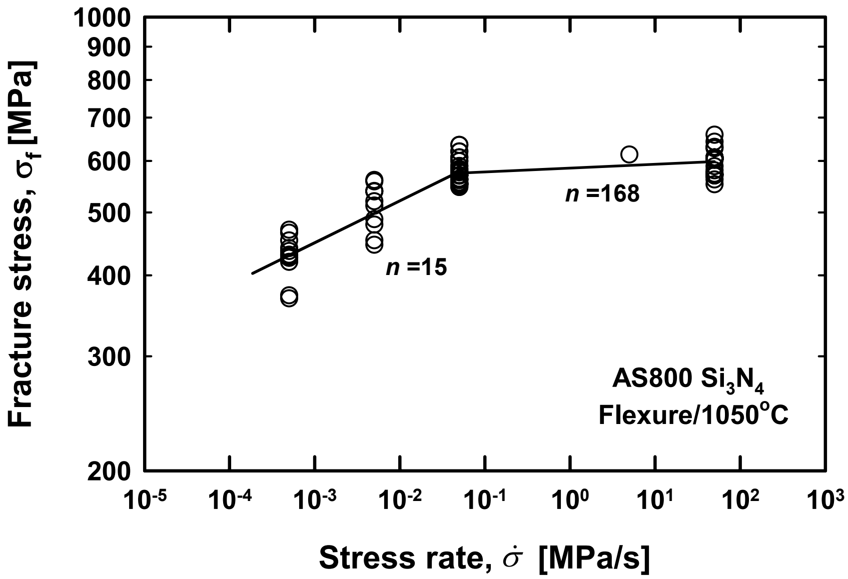


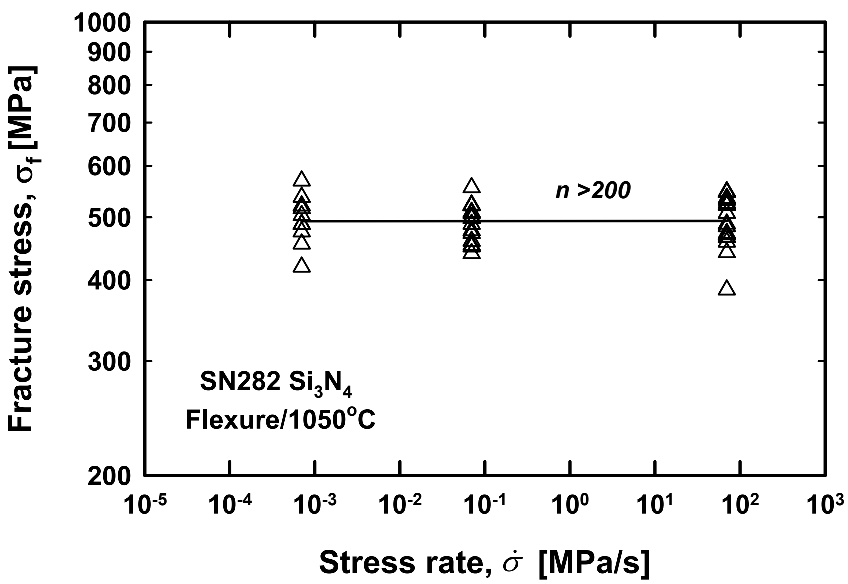
Figure 5.—Summary of flexure strength as a function of temperature for AS800 and SN282 silicon nitrides tested in air. Data, except for 1050 °C, quoted from previous work (refs. 6, 7, and 28).

Dynamic Fatigue

Result of dynamic fatigue testing for AS800and SN282 silicon nitrides at 1050 °C in air are shown in figure 6, where flexure strength was plotted as a function of applied stress rate. For AS800, between applied stresses of 50 and 0.05 MPa/s, there was little strength degradation. However, at applied stress rates ≤0.05 MPa/s, strength degradation took place with further decreasing applied stress rate, showing a susceptibility to slow crack growth. By contrast, no appreciable strength degradation occurred for SN282, indicative of significant resistance to slow crack growth.



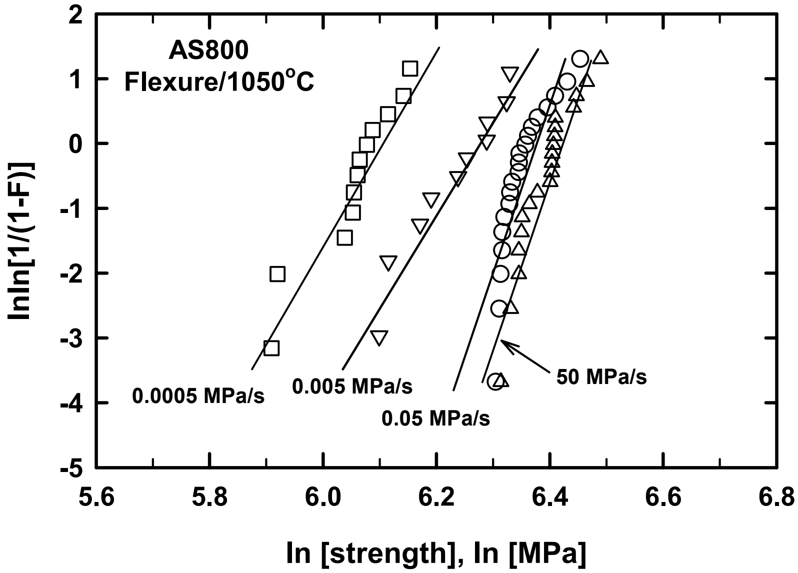
(a) AS800 silicon nitride



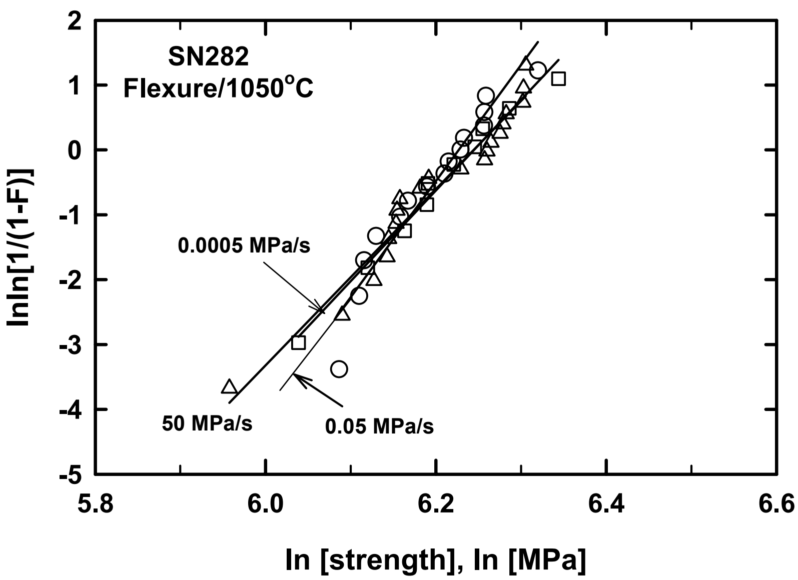
(b) SN282 silicon nitride

Figure 6.—Summary of flexure strength as a function of applied stress rate, determined for AS800 and SN282 silicon nitrides in dynamic fatigue testing in flexure at 1050 °C in air. Slow crack growth parameter n is shown for each material.

Weibull distributions of strength determined at different stress rates are shown in figure 7. Weibull modulus ranged from m = 14 to 26 for AS800 with a somewhat decrease at lower applied stress rates $\dot{\sigma} \leq 0.005$ MPa/s. Weibull modulus for SN282 ranged from m = 14 to 18, almost independent of test rate. Figure 8 shows typical fracture surfaces of AS800 specimens tested at 0.05 and 0.0005 MPa/s. At 0.05 MPa/s, a region of fracture mirror including fracture origin was well defined; whereas, at 0.0005 MPa/s, the fracture mirror region became significant, indicating the occurrence of extensive slow crack growth. No noticeable evidence of slow crack growth was observed from fracture surfaces of SN282 specimens tested at the lowest test rate of 0.0005 MPa/s. If a ceramic flexure specimen is susceptible to

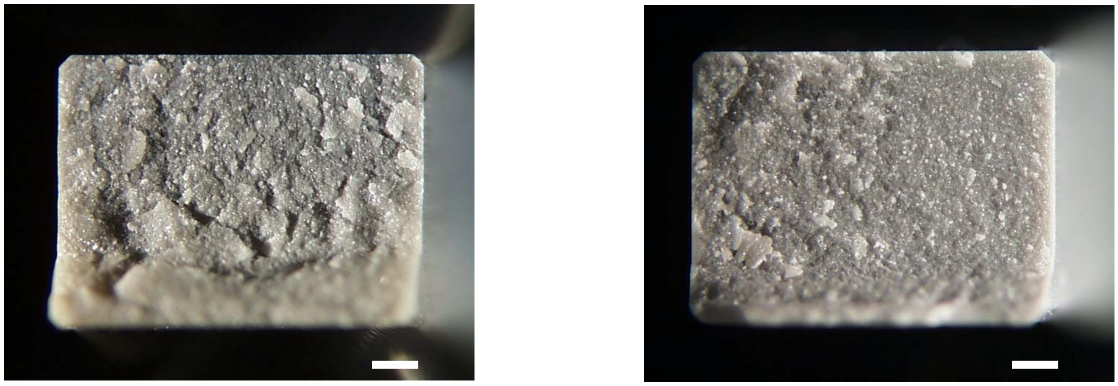


(a) AS800 silicon nitride



(b) SN282 silicon nitride

Figure 7.—Weibull distributions of flexure strength determined for AS800 and SN282 silicon nitrides in dynamic fatigue testing at 1050 °C in air.



(a) Tested at 0.05 MPa/s (b) Tested at 0.0005 MPa/s

Figure 8.—Typical examples of fracture surfaces of specimens tested at 0.005 (a) and 0.0005 MPa/s (b) in dynamic fatigue testing for AS800 silicon nitride at 1050 °C in air. Bars in 500 μm.

creep at elevated temperatures, creep deformation is typically observable at low stress rates ≤0.05 MPa/s during testing (from load-time or displacement-time curve) or from the curvature of specimens tested. However, no noticeable creep deformation was observed for both AS800and SN282 during testing or from specimens tested, indicating that creep was not an issue for the material at this temperature. In fact, creep of many advanced structural silicon nitrides has been known to be negligible below 1200 °C. Hence, slow crack growth can be a major consideration factor for AS800 than creep as far as component reliability/life is concerned.

The basic formulation of slow crack growth (SCG) for advanced monolithic and composite (reinforced with particulates, platelets or whiskers) ceramics at elevated temperatures follows an empirical power-law form

$$v = A[K_I / K_{Ic}]^n \tag{2}$$

where v, K_I, and K_{Ic} are crack velocity, mode I stress intensity factor, and mode I fracture toughness, respectively. A and n are material/environment dependent SCG parameters. In case of dynamic fatigue loading, a constant stress rate ($\dot{\sigma}$) is applied to a test specimen until the test specimen fails. The corresponding fracture strength (σ_f) can be derived from equation (2) as a function of applied stress rate ($\dot{\sigma}$) with some mathematical manipulations to give (ref. 15 and 16)

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D \tag{3}$$

where D is another SCG parameter associated with A, n, and K_{Ic}, inert strength, and crack geometry factor. The SCG parameters n and D can be determined from the slope and intercept by a linear regression analysis when log (*fracture strength*) is plotted as a function of log (*applied stress rate*). Equation (3) is the basis commonly used in dynamic fatigue testing, which has been adopted to determine SCG or life prediction parameters of advanced ceramics in ASTM test standards at both ambient and elevated temperatures (refs. 15 and 16).

The results shown in figure 6 were plotted according to equation (3) with units of MPa for σ_f and MPa/s for $\dot{\sigma}$. For AS800, SCG parameters were found to be n = 15 and D = 699 between $\dot{\sigma}$ = 0.05 and 0.0005 MPa/s, whereas n = 168 and D = 585 between $\dot{\sigma}$ = 50 and 0.05 MPa/s. Particularly, an enhanced susceptibility to SCG with a low SCG parameter n = 15 was noted at the region of lower applied stress rates, which would affect significantly a long term life/reliability of components made out of the material.

The inferior resistance to SCG at 1050 °C may be attributed to intermediate-temperature oxidation, as also observed from the strength test results (see fig. 5) and from other silicon nitride ceramics (ref. 29). More detailed microstructural analysis using SEM, TEM or any appropriate means is required to reveal the exact cause of enhanced SCG behavior of AS800 at 1050 °C. For SN282, SCG parameters were n = 16750 and D = 493. It is generally categorized that significant SCG occurred for n<30, intermediate SCG for 30<n<70, and insignificant SCG for n>70. Hence, virtually no slow crack growth occurred for SN282.

A simplified life prediction diagram, stress rupture, can give a better interpretation of life prediction of brittle materials, which was constructed in figure 9 using the same geometrical and dimensional configurations of test specimens that were employed in dynamic fatigue testing. The prediction was made based on SCG and strength parameters estimated for AS800 and SN282 as a function of applied stress (σ) using a relation e.g., (ref. 30)

$$t_f = f(m, \sigma_i, n, D, F, \sigma) \tag{4}$$

where t_f is life, F is the failure probability, and σ_i is the inert strength. Note that a new SCG parameter of n = 150, instead of n = 16750, was used for SN282 for the sake of a more realistic, conservative estimation of life. The prediction made in figure 9 is valid when the same failure mechanism is operative, irrespective of loading condition, either dynamic fatigue or constant stress (stress rupture). For simplicity, a failure probability of approximately F = 50 percent was used for both silicon nitrides. Of course, different levels of failure probability can be also incorporated. As can be seen from the figure, two different regions of life exist for AS800 because of a transition in the dynamic fatigue behavior shown in figure 6. Therefore, a significant drop in life for AS800 is expected to occur at lower applied stresses. This result prompts an initiation of stress rupture testing to verify the life prediction made based on this dynamic fatigue data. By contrast, as expected, SN282 exhibited a significant resistance to SCG, a very desirable feature of structural components. A detailed life prediction and reliability of ceramic Stirling convertor heater head components can be made using finite element modeling and an appropriate reliability tool such as the *CARES/Life* integrated computer code (ref. 31). Furthermore, accurate evaluation of applied mechanical/thermal stresses acting on the component in service must be exercised in order to better predict the resulting component reliability/life.

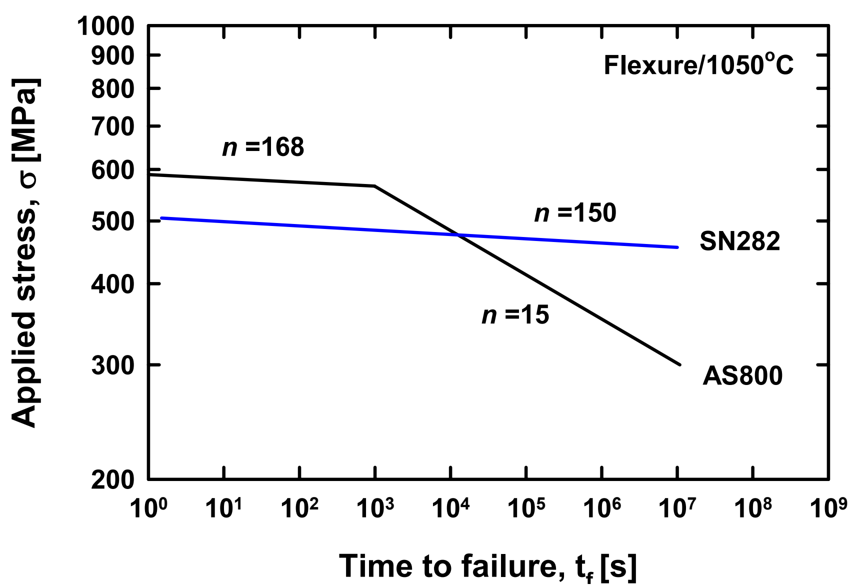


Figure 9.—Simplified life prediction diagram, converted from the dynamic fatigue data for AS800 and SN282 silicon nitrides at 1050 °C in air.

As mentioned in the Experimental Procedures section, preloads were applied to save test time at the lowest test rate of 0.0005 MPa/s in which most (≈ 80 percent) test times in dynamic fatigue testing are consumed. An analytical solution of fracture stress as a function of preload has been made and verified for many brittle materials at ambient or elevated temperatures (refs. 17 to 19). The resulting solution of fracture stress is expressed as

$$\sigma^* = \left(1 + \alpha_p^{n+1}\right)^{\frac{1}{n+1}} \tag{5}$$

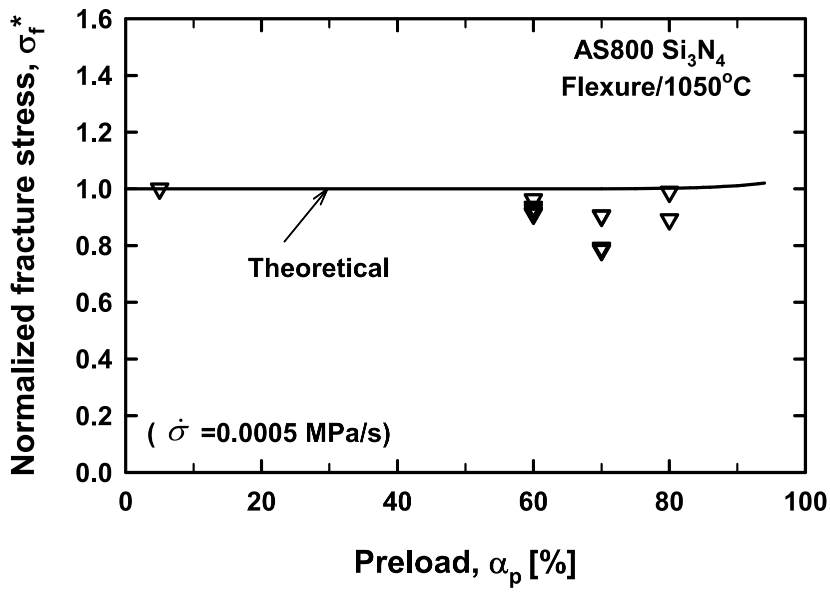
where σ^* is the normalized fracture stress, in which the fracture stress with preload is normalized with respect to the fracture stress without preload, α_p is preloading factor ($0 \le \alpha_p \le 1$), in which the applied preload stress is normalized with respect to the fracture stress without preload. Results of fracture stress as a function of preload, applied at 0.0005 MPa/s for AS800 and SN282, are depicted in figure 10. Also included is the theoretical prediction based on equation (5) together with values of n = 15 and 150, respectively for AS800 and SN282. There seems to exist a discrepancy or a good agreement between the data and the theory for AS800 and SN282. This is due to the fact that reliable strength data without preload were not obtained for both materials since only one test specimen was tested for each material without preload. Of course, more test specimens without preload are required. Nonetheless, the strength data with preload are believed reliable and accurate and can be regarded as representing values at 0.0005 MPa/s, based on our long experience on this technique for various brittle materials. The important thing for this technique is that 50 to 80 percent saving of test time was achieved without changing inherent strength values. Note again that at the lowest stress rate the normal test time for one specimen without preload was approximately 200 to 300 hr (10 days).

Elastic Modulus

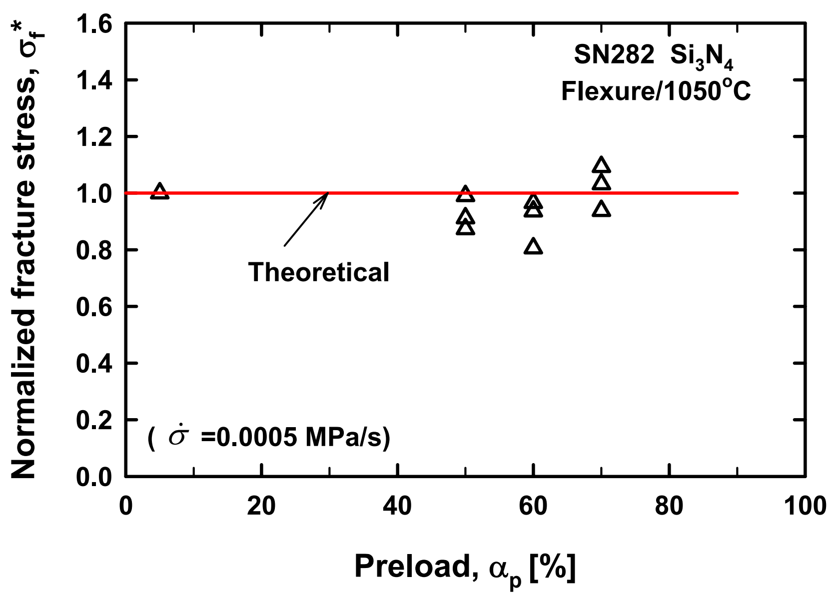
Results of elastic modulus as a function of temperature for both AS800 and SN282 silicon nitrides are shown in figure 11. Elastic modulus of both materials decreased monotonically and linearly up to 1200 °C. The values of elastic modulus at ambient temperature, 310 and 304 GPa for AS800 and SN282, respectively, are in an excellent agreement with those (309 and 304 GPa, respectively; see table 1) determined independently by using a different excitation/acoustic system (Type 2669, Brüel & Kjær, Denmark). The data in figure 11 can be formulated via a regression analysis as

$$\begin{aligned} E &= -0.013\,T + 310 \quad \text{for AS800} \\ E &= -0.012\,T + 304 \quad \text{for SN282} \end{aligned} \tag{6}$$

where E is elastic modulus in GPa and T is temperature in degree Celsius (°C). The correlation coefficient of regression was r^2_{coef}>0.996 for both curves. Elastic modulus degradation from 25 to 1200 °C was about 5 percent for both AS800 and SN200. This negligible degradation in elastic modulus with regard to temperature is typical of many silicon nitrides (refs. 32 and 33). As a consequence, both shear modulus and Poisson's ratio at ambient temperature are expected to remain almost unchanged up to 1200 °C as well. Also note that no appreciable difference in elastic modulus was found between the two materials in the temperature range studied.

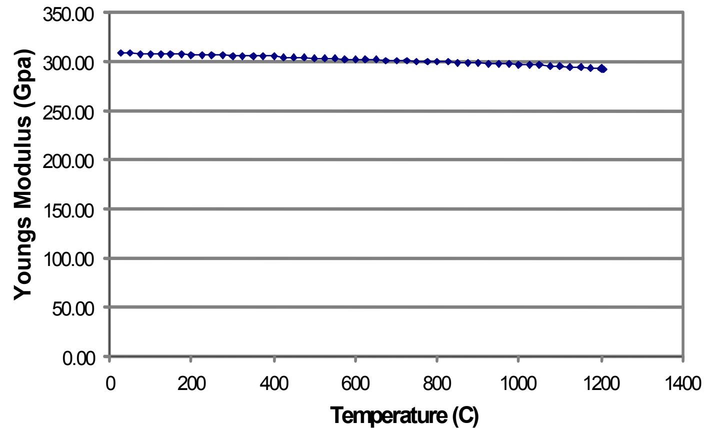


(a) AS800 silicon nitride

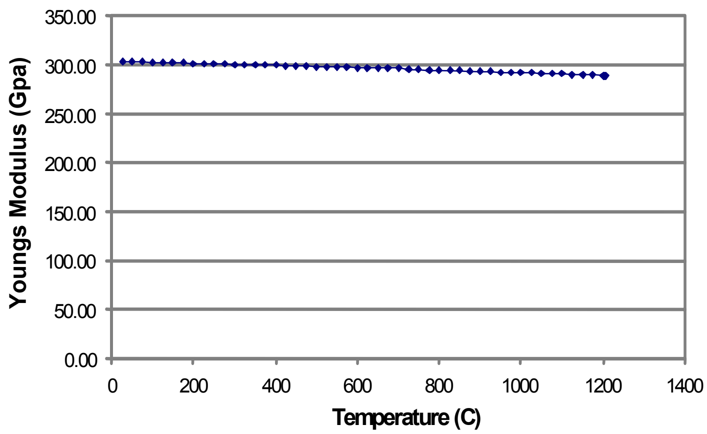


(b) SN282 silicon nitride

Figure 10.—Normalized fracture stress as a function of preload for AS800 and SN282 silicon nitrides in dynamic fatigue testing at 1050 °C in air at the lowest stress rate of 0.0005 MPa/s. The theoretical prediction based on equation (5) is also presented.



(a) AS800



(b) SN282

Figure 11.—Elastic modulus as a function of temperature determined by the impulse excitation of vibration method (ref. 20): (a) AS800 and (b) SN282 silicon nitrides.

Thermal Conductivity and Coefficient of Thermal Expansion

Results of thermal conductivity measurements for both AS800 and SN282 silicon nitrides are summarized in figure 12, where thermal conductivity was plotted against temperature up to 1200 °C. Due to some limitations at lower temperatures inherent in the laser test methodology, it is suggested to utilize the data above 600 °C as valid ones. No difference in thermal conductivity between the two silicon nitrides was observed.

Coefficient of thermal expansion (CTE) as a function of temperature is summarized for both silicon nitrides in figure 13. Thermal expansion data for each material are also included. The value of CTE for both materials increased from 2.5 to 4.6×10^{-6} mm/mm (by 80 percent) when temperature increased from 200 to 1200 °C. Like elastic modulus and thermal conductivity, there was little difference in CTE for a given temperature between two materials, both lying in CTE = 2.5 to 4.6×10^{-6} mm/mm within the temperature range investigated.

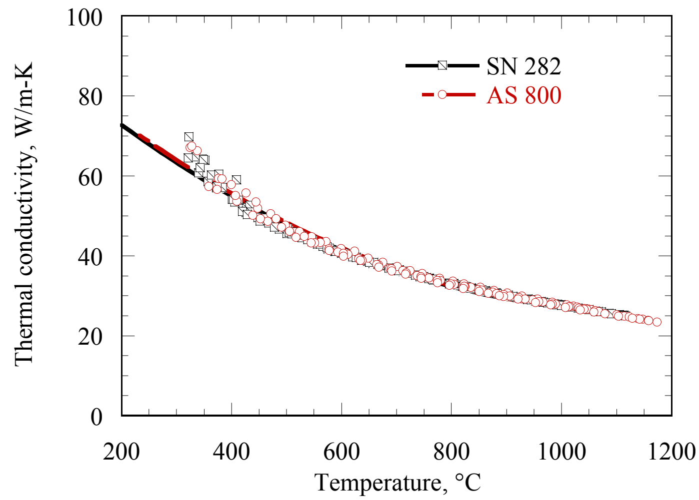


Figure 12.—Thermal conductivity as a function of temperature determined by a laser high heat-flux method for AS800 and SN282 silicon nitrides.

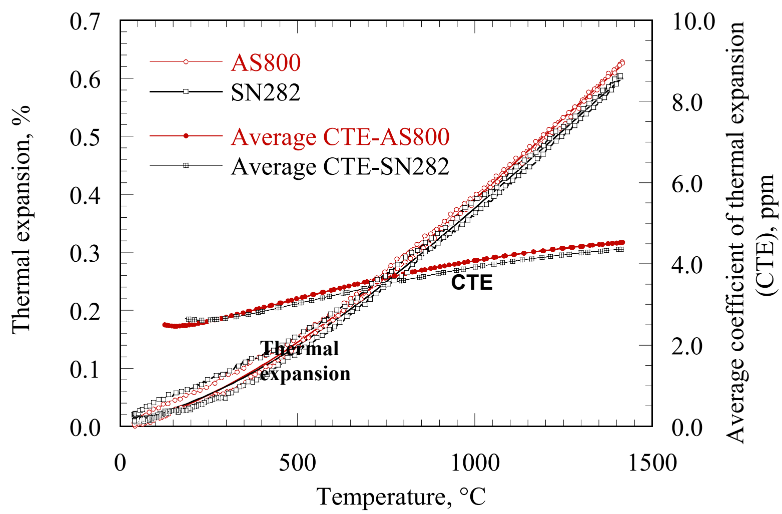


Figure 13.—Coefficient of thermal expansion (CTE) as a function of temperature determined by a dilatometer for AS800 and SN282 silicon nitrides. Thermal expansion data are also included.

Implications

AS800 silicon nitride exhibited better mechanical properties in fracture toughness and strength at ambient temperature than SN282 counterpart. For example, fracture toughness and flexure strength of AS800 are greater by 50 percent and 30 to 60 percent, respectively, as compared with those of SN282. The same is true for Weibull modulus and R-curve. Furthermore, fast-fracture strength (with no presence of slow crack growth) and Weibull modulus at elevated temperatures are greater for AS800 than for SN282. In terms of ambient-temperature mechanical properties and elevated-temperature fast-fracture strength, a preferable choice as a candidate structural material for a Stirling convertor heater head would be AS800 if one does not consider a life limiting phenomenon of the material at elevated temperatures. As seen from the results of this study (see figs. 6 and 9), however, the life limiting phenomenon was apparent

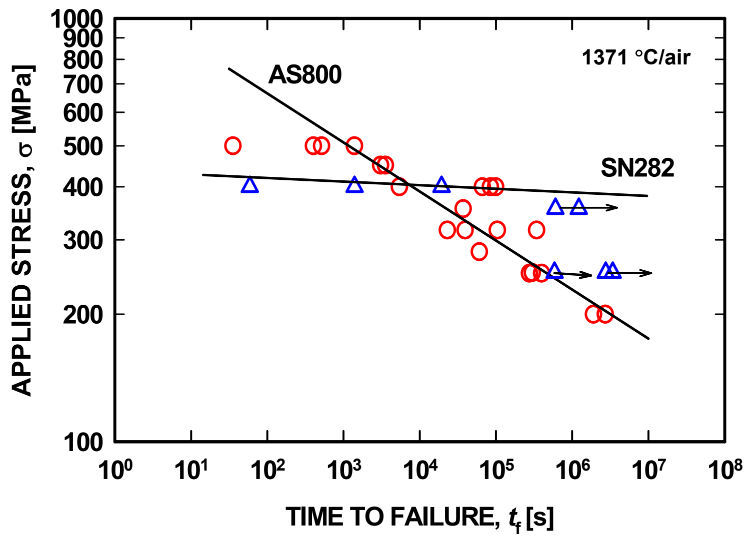


Figure 14.—Stress rupture results of AS800 and SN282 silicon nitrides tested at 1371 °C in air in flexure (ref. 28).

for AS800 even at an intermediate temperature of 1050 °C in air. At temperatures ≥1316 °C in air, resistance to creep, slow crack growth, and environmental degradation (oxidation) has been observed all greater for SN282 than for AS800. One example of stress rupture of AS800 and SN282 previously determined at 1371 °C in air is depicted in figure 14, which shows a significant susceptibility to slow crack growth (or short long-term lives) for SN282, as compared with SN282 counterpart (ref. 28). More work is required in order to characterize life limiting properties of both AS800 and SN282 in different loading (stress rupture, flexure, tension, etc) and environmental (air, helium, vacuum, etc.) conditions. In addition, efforts to perform structural reliability/life prediction analysis together with non-destructive evaluation (NDE) are also required.

Future Work

Future work regarding materials' characterization and basic component design may include following items:

- Fractogractic analysis
- Life assessment: stress rupture and creep for AS800 and SN282
- Environmental effects (air, helium, vacuum) on life/durability
- Non-destructive evaluation (NDE) of materials/components
- Reliability/life prediction of prospective structural ceramic components using an appropriate integrated design code (*CARES/life*)

Summary

Strength and life assessment testing for two commercial silicon nitrides AS800 and SN282 were conducted at 1050 °C in air. Flexure strength and Weibull modulus of AS800 were greater than those of SN282. Susceptibility to slow crack growth that controls life of structural components was apparent for AS800 with a relatively low life prediction parameter $n = 15$, while SN282 exhibited a much increased resistance to life limiting phenomenon. No appreciable difference in elastic modulus or thermal

conductivity was observed between the two silicon nitrides up to 1200 °C. The same was true for coefficient of thermal expansion up to 1400 °C. Although AS800 exhibited greater fast-fracture strength, higher fracture toughness, rising R-curve, and better Weibull modulus, it did not provide a guaranteed resistance to slow crack growth at 1050 °C. More testing and analysis are required to better understand materials' life limiting behavior in different environments and thus to generate a reliable design database for Stirling convertor heater head applications.

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13. ABSTRACT ***(Maximum 200 words)***

NASA Glenn Research Center is developing advanced technology for Stirling convertors with a target of significantly improving the specific power and efficiency of the convertor and overall generator for Mars rovers and deep space missions. One specific approach to the target has been recognized as the use of appropriate high-temperature materials. As a series of ceramic material approaches in Advanced Stirling Convertor Development Program in fiscal year 2005, two commercial, structural silicon nitrides AS800 (Honeywell, Torrence, California) and SN282 (Kyocera, Vancouver, Washington) were selected and their mechanical and life limiting properties were characterized at 1050 °C in air. AS800 exhibited both strength and Weibull modulus greater than SN282. A life limiting phenomenon was apparent in AS800 with a low slow crack growth parameter $n = 15$; whereas, a much increased resistance to slow crack growth was found in SN282 with $n>100$. Difference in elastic modulus and thermal conductivity was negligible up to 1200 °C between the two silicon nitrides. The same was true for the coefficient of thermal expansion up to 1400 °C.

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