NASA Technical Memorandum 87092

Reliability of Two Sintered Silicon Nitride Materials

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August 1985

NASA

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SUMMARY

Two types of sintered silicon nitride were evaluated in terms of reliability: an experimental, high pressure nitrogen sintered material and a commercial material. The results showed wide variations in strength for both materials. The Weibull moduli were 5.5, 8.9, and 11 for the experimental material at room temperature, 1200, and 1370 °C, respectively. The commercial material showed Weibull moduli of 9.0, 8.6, and 8.9 at these respective temperatures. No correlation between strength and flaw size was noted for the NASA experimental material which was extensively evaluated. Based on the above data, this paper discusses the applicability of the Weibull and Griffith theories to processing defects on the order of 100 μm or less in size.

I. INTRODUCTION

Silicon nitride, like other ceramics targeted for structural applications, is presently an unreliable material. The reasons for this poor reliability, i.e., the large range of strength values, are: (1) the fact that ceramics contain a wide variety of processing flaws; and (2) the fact that ceramics are brittle and consequently are not as "forgiving" of flaws as their ductile metal counterparts. Research in recent years has done much to evaluate the extent of the problem (refs. 1 and 2) with the result that experimentation is now being conducted on processing methodology (refs. 3 to 6). Specifically, this involves the understanding and control of defect formation in ceramic bodies and the manufacture of spherical powder particles of small size and narrow size distribution. Along other lines, efforts have been made to understand the failure process and reliability in terms of fracture mechanics (refs. 1, 7 to 13). The thrust of the latter effort is the assumption that a basic understanding of failure would suggest further means of improving the strength and reliability of ceramics.

Various statistical measures of reliability have been used to describe ceramics, the most popular of which is the two parameter (slope and characteristic strength) Weibull modulus. Basically, this is a measure of the dispersion of the strength distribution and is analogous to the standard deviation of the normal distribution. In this application of the Weibull distribution, a third parameter, the location parameter or expected minimum strength value is assumed equal to zero. Small Weibull moduli, in the range of 5 to 12, are usually obtained for ceramics, indicating poor reliability. In contrast, metals generally have moduli over 30. The Weibull modulus is based on the weakest link concept, meaning that a test specimen will fail immediately

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when the stress intensity at one of the flaws reaches the critical value for failure (ref. 14). The present research has included this modulus as a rough indicator of Si_3N_4 reliability since much of the reliability data from other sources has been based on this number. However, it should be kept in mind that the Weibull theory was developed for specimens tested in tension and without an interacting flaw population and that the development of better statistical measures is needed.

One goal of the present work was to determine baseline mean strength and Weibull modulus for two types of silicon nitride: an experimental, high pressure nitrogen sintered material (produced at Lewis) and a commercial sintered material. The materials selected for this study are of interest because they represent current experimental material and a recent generation of Si_3N_4 considered for structural applications. Further goals were to examine the causes of failure in these materials, and to correlate strength with flaw size. Previous studies on the effects of flaw type and size on strength have used either specimens seeded with defects in the range, 100 to 800 μm (ref. 15), or specimens having machining-induced surface cracks (ref. 16). In this study, the flaws are typical processing defects and range in size from 10 to 100 μm. Three test temperatures were used: room temperature, 1200, and 1370 °C.

II. EXPERIMENTAL PROCEDURE

1. Materials

The experimental, high pressure nitrogen sintered material (designated NASA 6Y) was fabricated according to the following procedure. Silicon nitride, silica, and yttria powders were ground together as 100 g charges in 1 liter RBSN mills[1] filled with HPSN media.[2] The media charge was 800 g of 1 cm by 1 cm HPSN cylinders and the milling fluid was 1/2 liter of 100 percent ethanol. Milling time was 24 hr. After milling, the powder-ethanol slurry was transferred to a Rotovapor[3] for removal of ethanol vapor. This was followed by drying the powder in a vacuum oven at 110 °C. The powders were then sieved through a 100 mesh screen.

Table I gives the chemical analyses of the starting powders and summarizes the charge weight percentages before milling. It also gives the specific surface area, carbon analysis, and trace element analysis after milling. The specific surface areas were determined by the 3-point BET method. Oxidation occurs during milling as evidenced by an increase in the oxygen content. Pickup from wear of the Si_3N_4 milling hardware averaged 3.6 h. Table I

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[1]Ceramic Systems Incorporated, Detroit, MI - Chemical analysis of the mill indicate: 0.005 percent C, 1.04 percent O_2, and expressed in parts per million: 1000 Al, 710 Ca, 410 Cr, 500 Cu, 6000 Fe, 350 Mg, 310 Mn, 70 Ni, 250 Ti and 110 V.

[2]Advanced Ceramic Systems, Ypsilanti, MI - Chemical analysis of the media indicate: 5.25 wt % O_2, 0.46 percent C, 0.8 percent Al, and, expressed in parts per million: 670 Ca, 30 Cu, 2000 Fe, <2 Ti, 370 Cr, 130 Mg, 90 Mn, <10 V, <20 Zr, 2000 Mo, 240 Ni, 130 Zn, <30 Pb, <60 Sn.

[3]Rotavapor, Prieser Scientific, Louisville, Kentucky.

also gives the calculated mole percentages for the Si_3N_4-SiO_2-Y_2O_3 experimental composition (NASA 6Y) in the sintered condition. These calculations are based on initial charge composition, Si_3N_4 enrichment due to pickup from the milling hardware, oxygen and yttrium analyses after sintering and reduction of some SiO_2 by carbon assumed to occur during sintering. The AME-KBI SI_3N_4 powder was chosen for this study because of its equiaxed crystal morphology and better compactibility in comparison to other powders which contain high aspect ratio crystals.

The powder composition was compacted at 21 MPa into 2.6 g test bars 3.81 by 0.79 by 0.45 cm using a two-way action tungsten carbide-lined die. The die-pressed bars were vacuum sealed in thin-wall latex tubing and isostatically cold-pressed at 414 MPa. Green density averaged 1.91 g/cc (60.4 percent of the calculated theoretical density,[4] 3.16 g/cc). Weighed and measured bars were sintered 15 at a time in a tungsten cup with a loose-fitting tungsten lid in a water cooled double-wall furnace. The bars were separated from one another and from contact with the tungsten cup by BN discs. The loaded tungsten cup was placed on a tungsten pedestal with the axis of the pedestal coincident with a 10.2 cm diameter tungsten mesh heater. Surrounding the heater were concentric W and Mo radiation shields. Sintering temperatures were monitored and controlled with W-5Re/W-26Re thermocouples, with temperature for this study held at 2140 °C. Sintering time was 1 hr in a static N_2 pressure of 2.5 MPa. Heating from R.T. to 2140 °C was at an approximately linear rate and accomplished in 45 min. Nitrogen pressure rise was also approximately linear, increasing from 0.7 MPa at the start of heating to 2.5 MPa within 60 min.

The bars were weighed and measured both before and after sintering to determine weight loss and shrinkage. The major faces of the bars were longitudinally ground with a 400 grit diamond wheel while the four long edges were beveled 0.12 mm. Final test bar dimensions were 3.0 by 0.56 by 28 cm. Bar densities, calculated from dimensions and weights, averaged 3.12 g/cc (98.7 percent of the calculated theoretical density).

The Lewis 6Y test bars for this study were made from five of twelve identically processed powder batches. Process reliability for these 12 powder batches was found to be excellent based on a comparison of the following values: Si_3N_4 media plus mill wear; specific surface area; oxygen content; and spectrographic analysis of ground powder. Additional test bar comparisons, based on average values and standard deviations, were made for: green density; sinter weight loss; dimensional shrinkage; and final density.

The commercial sintered Si_3N_4 (GTE AY-6) was obtained in August, 1982, as fifty longitudinally surface ground bars, 5.2 by 0.64 by 0.32 cm in size. The bars had been cut from cold-pressed and sintered 11.4 by 5.8 by 0.95 cm plates. Long edges of these bars were beveled 0.12 mm. At Lewis, these bars were cut in two, resulting in a 100 test bar lot. Bar densities, calculated

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[4]Theoretical density was calculated by the law of mixtures assuming the presence of Y_2O_3 and SiO_2 in addition to Si_3N_4.

from dimensions and weights, averaged 3.20 g/cc (98.2 percent of the reported theoretical density, 3.26 g/cc).

2. Testing and Analysis

Four-point flexural strength tests were conducted at a crosshead speed of 0.51 mm/min with inner and outer spans of 9.53 and 19.05 mm respectively. Room temperature tests were conducted with steel fixtures. Elevated temperature tests were conducted using SiC fixtures in a SiC muffle furnace mounted on a testing machine.[5] All tests were conducted in air.

X-ray diffractometer scans of ground surfaces of test bars showed ß-Si_3N_4 as the major phase for both 6Y and AY-6. A minor amount of Si_2N_2O was detected in the AY-6 composition.

For microstructural characterization, two-stage carbon replicas (made from polished and etched cross sections of test bars) were examined in the transmission electron microscope. Etching involved immersion in fused KOH for about 45 sec. Fracture surfaces were examined in a scanning electron microscope. The magnification was checked with NBS standards to assure the accuracy of flaw size measurements to within +10 percent.

III. RESULTS AND DISCUSSION

1. Statistics of Strength

A summary of strength data is given in table II. This includes statistical data for the two materials at room temperature, 1200, and 1370 °C. Statistical analysis consisted of F-tests (comparisons of variance) and Student's t tests (comparison of means) at the 95 percent significance level. From the t-tests it was determined that: (1) the room temperature strength are equal for AY-6 and 6Y; (2) the 1200 and 1370 °C strengths of 6Y are greater than that of AY-6; and (3) there is no loss in 6Y strength when the temperature is increased from 1200 to 1370 °C. These differences result from differences in the refractory nature of the grain boundary phase (ref. 17) where Y_2O_3-SiO_2 (NASA 6Y) produces a more refractory phase than Y_2O_3-Al_2O_3-SiO_2[6] (GTE AY-6).

The strength relationships may be seen graphically in the Weibull plots in figure 1. These are plots of ln ln [1/(1-P)] versus ln strength in which the slope is the Weibull modulus, m. The equation showing the relationship between the probability of failure, P, and the Weibull modulus is:

$$P = 1 - \exp\left[-kV\left(\sigma_m/\sigma_o\right)^m\right]$$

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[5] Instron Corporation, Canton, Massachusetts.

[6] It is assumed that the commercial material contains SiO_2, but the quantity is not known.

where

P = cumulative probability of failure
k = load factor derived for 3 or 4-point bending
V = volume under tensile stress
σ_m = maximum tensile stress in test bar
σ_o = normalizing stress (indirect measure of mean fracture stress)
m = Weibull modulus (ref. 14)

According to theory, a higher Weibull modulus indicates a more reliable material. However, since there appears to be no statistical test for the rigorous comparison of two Weibull moduli, the use of these numbers to determine which of two or more materials is most reliable is questionable. As mentioned previously, other tests must be developed to fully describe the reliability of brittle materials. Until that point, the evaluation of these materials may be made on the basis of F-tests and minimum strength level (to be specified by design engineers) (ref. 14) in cases where the Weibull modulus is small (ref. 18). For the materials studied, the application of such an analysis to the data in figure 1 and table II indicates that: (1) the standard deviation (strength spread) and the minimum strength decrease with temperature for both materials; (2) the standard deviation is significantly higher for 6Y at room temperature and 1370 °C; and (3) the 6Y minimum strength is below that of AY-6 at room temperature but above it at 1200 and 1370 °C. From this, it appears that 6Y is of technical interest, primarily for its high temperature strength.

Fractography. - Shown in figure 2 are plots of strength versus (flaw radius)$^{1/2}$ for 6Y at room temperature, 1200, and 1370 °C. This includes all observable fracture origins within 100 μm of the specimen surface (24 origins found out of 60 bars tested for room temperature, 27 out of 58 for 1200 °C, and 14 out of 29 for 1370 °C). The region from 0 to 100 μm from the surface is the first of fourteen 100 μm zones which extend from the surface of the specimen to its neutral axis. This was regarded as a region of uniform stress as a simplification. The flaw dimension which is plotted lies perpendicular to the test bar tensile face. No correlation between strength and flaw size was obtained for any of these temperatures. In addition, there was no correlation between defect type (pore,[7] Fe-base inclusion) and strength in the defect range studied. In work on AY-6 material by Pasto et al. (ref. 19), a calculated flaw radius was reported to correlate with room temperature flexural strength. Flaw radii in this study were calculated based on fracture toughness determined by indentation technique. AY-6 was also evaluated in the present study on a limited basis. The data is shown in figure 3. It consists of representative fractures from randomly selected regions of the strength range. The 1200 °C data is limited to four points and 1370 °C data is absent because slow crack growth had masked processing flaws in many instances. For the AY-6 room temperature and 1200 °C results at least, there appears to be some correlation between strength and flaw size. This could be attributed to a more uniform flaw character in this material. The information obtained in the present study contrasts to earlier studies on larger flaws (refs. 1 and 15) in which a correlation was obtained between defect type and strength.

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[7]Pores, agglomerates, and low density regions were grouped together on the basis of expected thermal expansion behavior.

The fact that the NASA 6Y data show a lack of correlation between strength and flaw size or flaw type, may be explained by reference to literature on the applicability of Griffith theory to inhomogeneous brittle materials (refs. 1,7, 8,10,12, and 14). General reasons for the lack of correlation can include differences in: (1) grain size; (2) internal stress, often due to thermal expansion differences between the matrix material and a second phase or impurity; (3) flaw population in the vicinity of the fracture origin; (4) flaw shape effects; and (5) flaw orientation (flaws which are not contained in the plane perpendicular to the maximum tensile stress are not stressed in a typical K_I mode). For 6Y, elements 2 to 5 in this list are thought to apply.

Typical processing flaws are shown in figure 4. Figures 4(a) and (b) are representative of pores and Fe-base inclusions found in 6Y while figure 4(c) is most representative of porosity in AY-6. Fewer inclusions were found in the AY-6 specimens indicating better process control. However, the porosity observed in this material was generally larger than that of 6Y (refer to figs. 2 and 3). Grain size is shown in table III and general microstructure in figure 5 for 6Y and AY-6. From these it can be seen that AY-6 has a slightly coarser microstructure but that the two materials are fairly similar.

The implication of these results is that the iterative processing/testing approach, currently used in DOE-funded contracts (ref. 20), is presently the best way to improve ceramic reliability, especially in materials showing varied flaw character.

IV. CONCLUSIONS

The room temperature strengths of NASA 6Y (Si_3N_4-SiO_2-Y_2O_3) and GTE AY-6 (Si_3N_4-SiO_2-Al_2O_3-Y_2O_3) are equiavlent. At 1200 and 1370 °C, NASA 6Y is significantly stronger than GTE AY-6. In addition NASA 6Y composition does not show a significant drop in strength from 1200 to 1370 °C.

There is no correlation between strength and flaw size or type for the materials and processing defects which were examined. This results from the wide variation in flaw character.

The next step in studying and improving the reliability of ceramics should be the production of material having fewer, smaller (10 μm), and ideally, more uniform defects. Research in this area is currently being conducted at NASA Lewis and in a number of other laboratories.

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TABLE I. - CHARACTERIZATION OF SILICON NITRIDE, OXIDE POWDERS, AND SI_3N_4-SiO_2-Y_2O_3 COMPOSITION AFTER MILLING AND SINTERING

Material or Composition	Mill charge, wt %			As received or milled condition chemical analysis			Specific surface area, m^2/gm	Sintered condition[a] phase content, mol %		
				Oxygen wt %	Carbon wt %	Spectro analysis, ppm				
	Si_3N_4	SiO_2	Y_2O_3					Si_3N_4	SiO_2	Y_2O_3
Si_3N_4[b]	-----	----	----	0.89	0.18	240 Al, 60 Ca, 20 Co, 50 Cr, 1280 Fe, 20 Mg, 30 Mn, 40 Ti, 40 V, 590 Y, 30 Zn, 10 Zr	4.7	-----	----	----
SiO_2[c]	-----	----	----	----	0.16	220 Al, 190 Ca, 30 Cr, 50 Cu, 50 Fe, 130 Mg, 90 Mn, 340 Na, 40 Ti	166	-----	----	----
Y_2O_3[d]	----	----	----	----	0.11	60 Cu, 60 Mg, 40 R.E. oxides	7.5	-----	----	----
COMP 6Y	89.6	4.2	6.2	4.54	0.39	420 Al, 120 Ca, 60 Co, 100 Cr, 1280 Fe, 60 Mg, 70 Mn, 40 Ti, 60 V, 4.3 percent Y, 20 Zn, 80 Zr, 90 Mo, 180 Ni	15.3	85.1	11.1	3.8

[a]Calculated allowing for Si_3N_4 pickup during milling, weight loss during sintering and O, C, and Y analyses after sintering.
[b]AME-KBI, High Purity, 99.5 percent; 83.7 percent α, 15.7 percent β, 0.6 percent Free Si.
[c]Apache Chemicals Inc., Code 6846, 99.99 percent.
[d]Molycorp, 5600, 99.9 percent.

TABLE II. - STRENGTH STATISTICS

Material	MOR test temperature, °C	Number of tests	Average strength, MPa	Standard deviation, MPa	Weibull modulus
NASA 6Y	Room temperature	60	548	117	5.5
	1200	58	393	53.3	8.9
	1370	29	382	39.2	11
GTE AY-6[a]	Room temperature	30	523	71.0	9.0
	1200	29	289	41.1	8.6
	1370	25	193	25.6	8.9

[a]A room temperature flexural strength of 552 MPa and a Weibull modulus of 11 were reported for ten bars (0.127 by 0.259 by 2.5 cm), tested in 4-point flexure with spans of 1.016 and 2.286 cm (19).

TABLE III. - GRAIN SIZE AND MORPHOLOGY OF SINTERED SI_3N_4

Material	Equiax grain size range, μm	Elongated grain width range, μm	Aspect ratio of elongated grains
6Y	0.4 to 4.4	0.4 to 4.0	1:2 to 1:8
AY-6	0.3 to 8.0	0.3 to 4.5	1:3 to 1:8

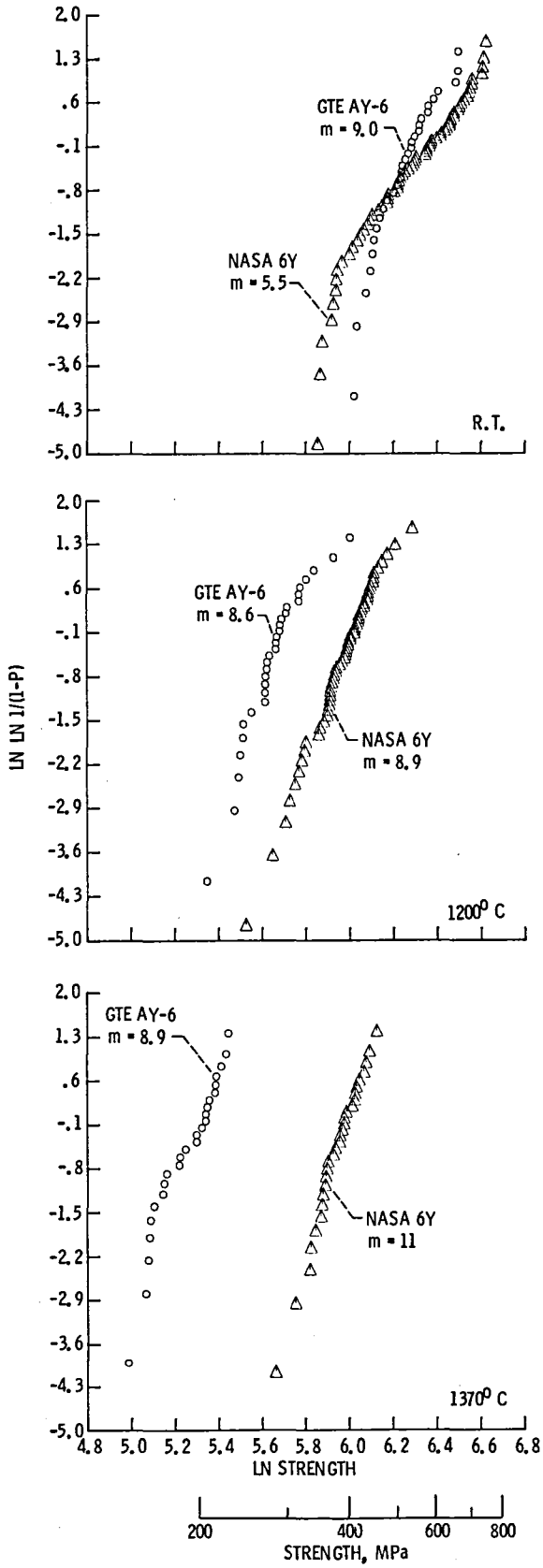

Figure 1. - Weibull plots for NASA 6Y and GTE AY-6 sintered Si_3N_4.

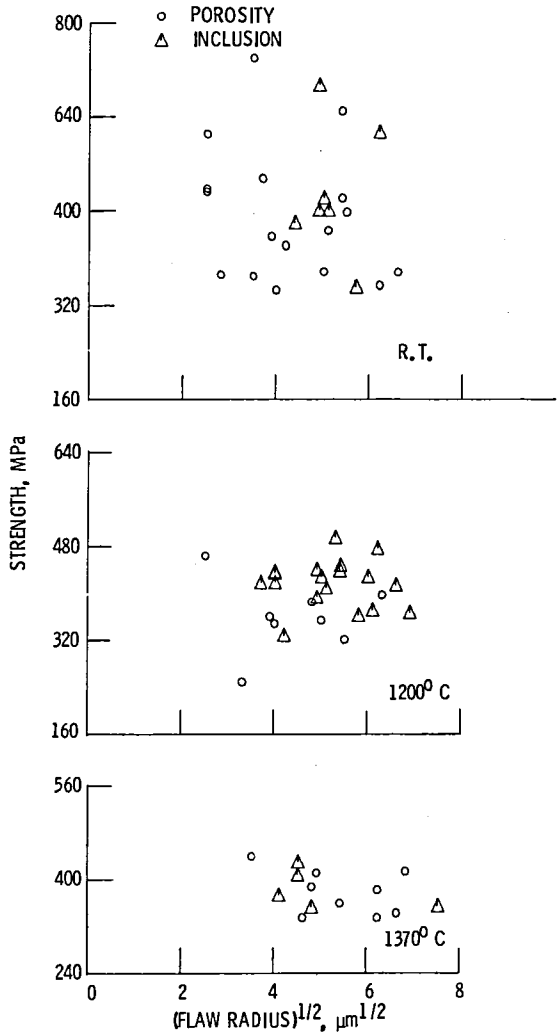

Figure 2. - Strength vs. (flaw radius)$^{1/2}$ for NASA 6Y sintered Si_3N_4.

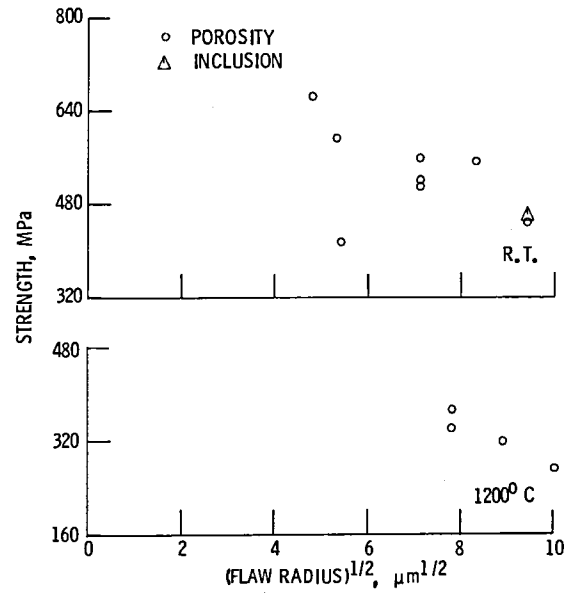

Figure 3. - Strength vs. (flaw radius)$^{1/2}$ for GTE AY-6 sintered Si_3N_4.

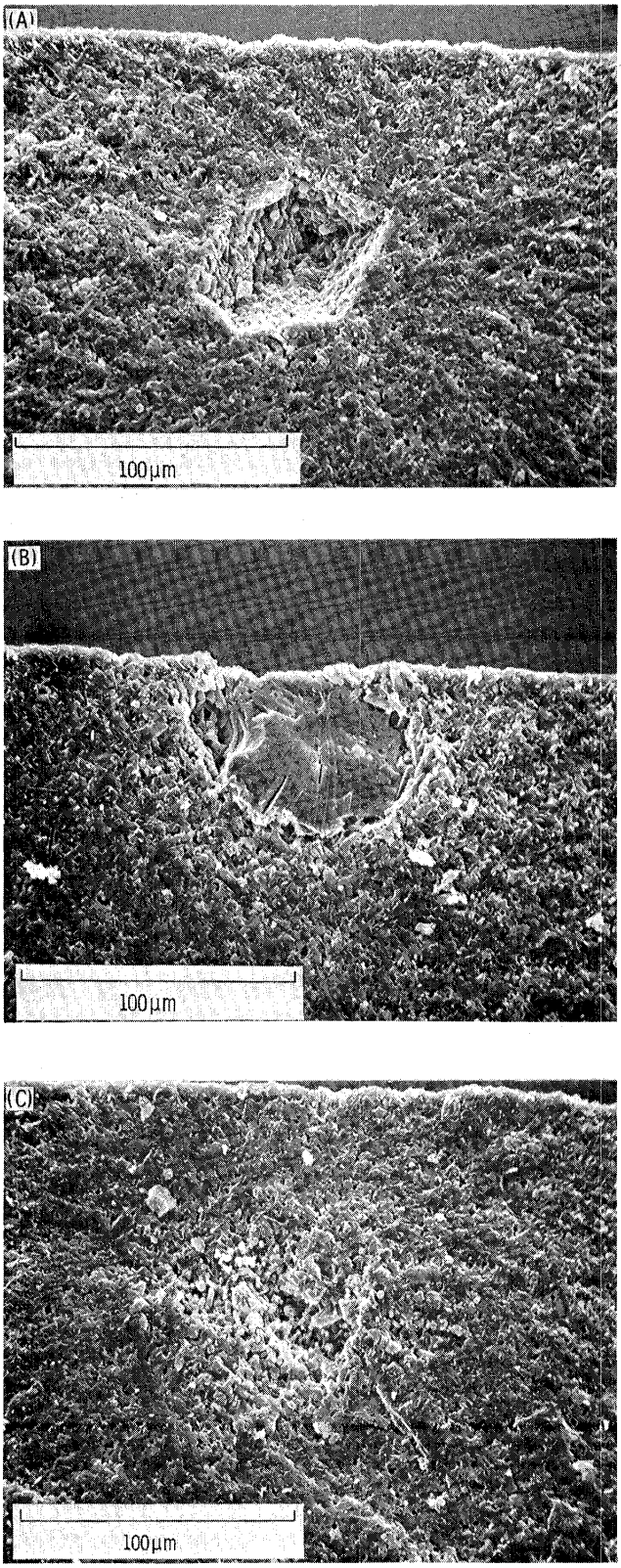

Fig. 4. - Scanning electron micrographs of typical processing flaws in sintered Si_3N_4. (A) pore and (B) Fe-base inclusion in NASA 6Y; (C) pore in GTE AY-6.

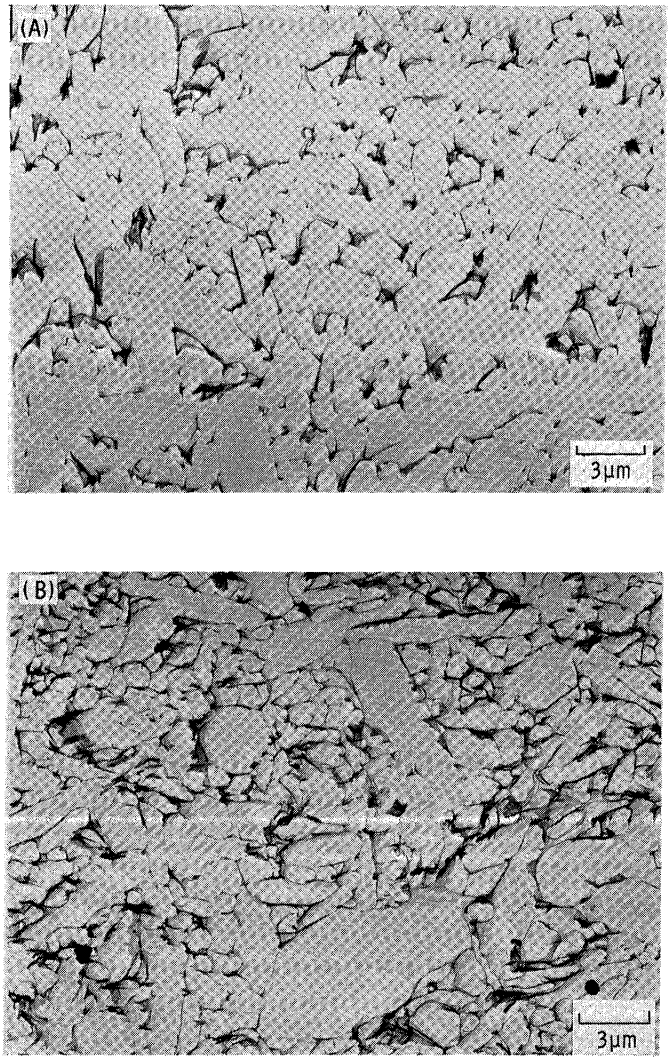

Fig. 5. - Transmission electron micrographs of replicas of polished and etched sintered Si_3N_4. (A) NASA 6Y, (B) GTE AY-6.

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
NASA TM-87092		

4. Title and Subtitle	5. Report Date
Reliability of Two Sintered Silicon Nitride Materials	August 1985
	6. Performing Organization Code: 533-05-12

7. Author(s)	8. Performing Organization Report No.
Diane M. Mieskowski, William A. Sanders, and Laurie A. Pierce	E-2325-1
	10. Work Unit No.

9. Performing Organization Name and Address	11. Contract or Grant No.
National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135	
	13. Type of Report and Period Covered: Technical Memorandum

12. Sponsoring Agency Name and Address	14. Sponsoring Agency Code
National Aeronautics and Space Administration Washington, D.C. 20546	

15. Supplementary Notes

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16. Abstract

Two types of sintered silicon nitride were evaluated in terms of reliability: an experimental, high pressure nitrogen sintered material and a commercial material. The results showed wide variations in strength for both materials. The Weibull moduli were 5.5, 8.9, and 11 for the experimental material at room temperature, 1200, and 1370 °C, respectively. The commercial material showed Weibull moduli of 9.0, 8.6, and 8.9 at these respective temperatures. No correlation between strength and flaw size was noted for the NASA experimental material which was extensively evaluated. Based on the above data, this paper discusses the applicability of the Weibull and Griffith theories to processing defects on the order of 100 μm or less in size.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Reliability; Silicon nitride; Flexural strength; Statistics; Weibull modulus	Unclassified - unlimited STAR Category 27

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of pages	22. Price*
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