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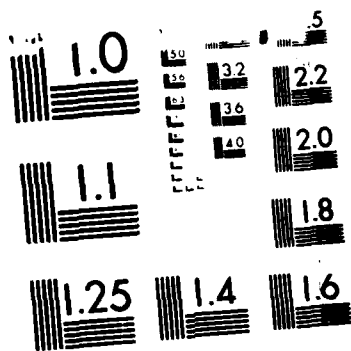
EFFECT OF OXIDATION ON THE DENSIFICATION OF SINTERABLE  
RBSN (REACTION-BONDED SILICON NITRIDE)(U) ARMY  
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ABSTRACT

Predoped RBSN bars containing 6%Y<sub>2</sub>O<sub>3</sub> and 1%Fe<sub>2</sub>O<sub>3</sub> were sintered to high density using a two-step pressure/temperature cycle. Some bars were preoxidized at 1000°C prior to sintering to increase the oxygen content (SiO<sub>2</sub>) of the specimens and to determine its effect on sintering behavior. Modulus of rupture and stress-rupture data were obtained on both as-received and heat-treated specimens

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## INTRODUCTION

Sintering of silicon nitride usually involves mixing silicon nitride powder with an appropriate additive, e.g., MgO or  $Y_2O_3$ , cold pressing the mixture, then sintering to achieve densification. An alternate approach involves the sintering of reaction-bonded silicon nitride (RBSN) where the densification aid has been added to the silicon prior to nitridation or infiltrated into the RBSN subsequent to nitridation. A major advantage cited for the sintering of doped RBSN is the relatively low percent shrinkage (6 to 8%) during densification which produces improved dimensional control. Another difference to be noted between the sintering processes is the compositional and phase development sequence occurring prior to and during sintering. Since quaternary oxynitride compounds are formed during the nitridation step of predoped RBSN, the reaction kinetics will differ for each sintering process. Those kinetics are influenced by the amount and stability of each component or compound formed and the reaction path. Additionally, high nitrogen gas pressures and appropriate cover powders are necessary for stabilizing specimen compositions at high sintering temperatures.

In the present study, reaction-bonded silicon nitride bars that had been fabricated from injection-molded silicon mixed with 6% $Y_2O_3$ , and 1% $Fe_2O_3$  were sintered to high density using  $N_2$  gas pressures up to 8.0 MPa and temperatures between 1875°C and 2000°C. The starting  $SiO_2$  content of some specimens was increased by oxidation prior to sintering. Densified specimens were tested at temperatures between 700°C and 1200°C in stress rupture for up to 1000 hours under stress (300 MPa). Room temperature modulus of rupture (RT MOR) was determined for surviving stress-rupture specimens and on specimens not subjected to high temperature testing. Fracture surfaces were examined by SEM to define fracture origins.

## EXPERIMENTAL PROCEDURE

Injection molded, predoped RBSN bars containing 6% $Y_2O_3$  and 1% $Fe_2O_3$  were supplied by the Garrett Turbine Engine Company, Phoenix, AZ, (identified as code 9) for study. X-ray diffraction (XRD) analysis of as-received bars indicated that they were typically 80% alpha phase. The remainder was essentially beta phase and yttrium-nitrogen apatite ( $Y_5Si_3O_{12}N$ ) with a small amount of free silicon also detected. The densities of the specimens were between 2.4 and 2.5 g/cc. Some of the bars were oxidized at 1000°C, in air, for times ranging from 10 to 120 minutes. The increases in  $SiO_2$  content were estimated from observed weight gains. Both oxidized and unoxidized specimens were imbedded in packing powder in a covered RBSN crucible during sintering. The composition of the packing powder was 64% $Si_3N_4$ -25%BN-6% $Y_2O_3$ -5% $SiO_2$ . It was used to protect the specimens from carbon in the atmosphere and provide a partial pressure of SiO around the specimens to restrict the loss of  $SiO_2$  by reducing reactions cited in the technical literature.<sup>1,2</sup> High  $N_2$  gas pressures were used to suppress the decomposition of  $Si_3N_4$  which could occur at the sintering temperatures utilized, i.e., 1875°C to 2000°C. Two-step sintering procedures were used which involved using a relatively low  $N_2$  gas (Figure 1) pressure (2.0 MPa) during the first sintering step then raising the pressure to 5.5 MPa in the second step. Sintering temperatures ranged from 1875°C to 2000°C. Two temperature levels were generally used for the sintering process where the change in temperature level corresponded with changing the gas pressure level. The first

1. LANGE, F. F. *Silicon Nitride Polyphase Systems: Fabrication, Microstructure, and Properties*. International Metals Reviews, v. 25, no. 1, 1980, p. 1-20.
2. MESSIER, D. R., and DeGUIRE, E. J. *Thermal Decomposition in the System Si-Y-Al-O-N*. J. Amer. Cer. Soc., v. 67, no. 9, 1984, p. 602-605.

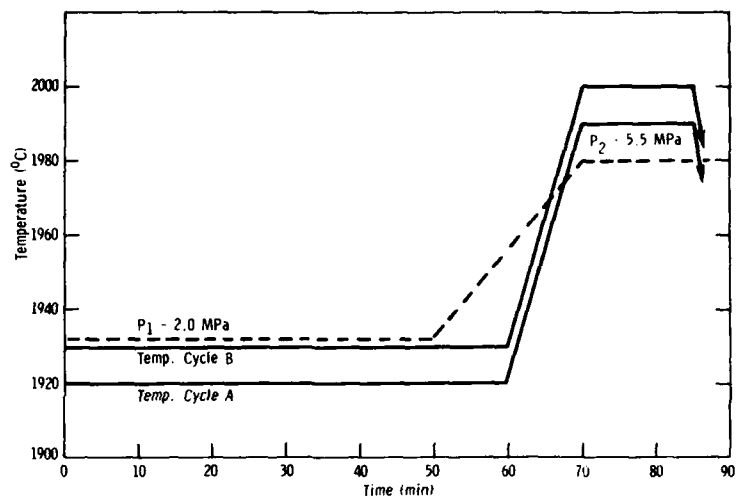


Figure 1. Two-step temperature/pressure cycles used for sintering.

temperature level employed was in the range of 1875°C to 1940°C, while the second temperature step was usually higher, 1970°C to 2000°C. Hold times of 45 to 60 minutes were used for step 1, and 15 to 30 minutes for step 2. For most sintering cycles, a crystallization treatment was performed by holding at a temperature between 1075°C and 1200°C for 60 minutes subsequent to the high temperature sintering steps. Some sintered bars were tested without machining to determine RT MOR. Bars were also machined to dimensions of 2.54 mm thick x 3.81 mm wide x 50 mm long for stress-rupture testing. The bars were placed under a 300 MPa flexure stress during the stepped temperature stress-rupture (STSR) procedure<sup>3</sup> from 800°C to 1200°C at 24-hour increments. After STSR, the bars were held at 1200°C under stress for up to 1000 hours unless failure occurred. Bars surviving 750 to 1000 hours were unloaded, cooled, and removed from the furnace for determination of retained RT MOR. Similar isothermal stress-rupture experiments were also conducted at 1000°C and 700°C to determine whether any intermediate temperature degradation occurred.

#### OXIDATION OF RBSN STARTING SPECIMENS

The use of oxidation reactions to alter the SiO<sub>2</sub> content of Si<sub>3</sub>N<sub>4</sub> compositions has been previously reported by Greskovich<sup>4</sup> and Evans and Moulson.<sup>5</sup> In this study, some of the as-received, reaction-bonded Si<sub>3</sub>N<sub>4</sub>-6%Y<sub>2</sub>O<sub>3</sub>-1%Fe<sub>2</sub>O<sub>3</sub> bars were oxidized in air at 1000°C for various times up to 120 minutes before sintering. The weight changes produced by oxidation were used to estimate the increase in SiO<sub>2</sub> content. Figure 2 shows the percent weight gain for the oxidation time used at 1000°C. Parabolic kinetics are followed with little increase in SiO<sub>2</sub> content produced after approximately 60 minutes. The increase in SiO<sub>2</sub> content after 60 minutes is estimated to be 1.0 to 1.50 percent.

3. QUINN, G. D. *Characterization of Turbine Ceramics After Long-Term Environmental Exposure*. U.S. Army Materials Technology Laboratory, AMMRC TR 80-15, April 1980.
4. GRESKOVICH, C. D., and PALM, J. A. *Development of High Performance Sintered Si<sub>3</sub>N<sub>4</sub>*. General Electric Co., Contract DAAG46-78-C-0058, Final Report, AMMRC TR 80-46, September 1980.
5. EVANS, J. R. G., and MOULSON, A. J. *The Effect of Impurities on the Densification of Reaction-Bonded Silicon Nitride (RBSN)*. *J. of Mat. Sci.*, v. 18, no. 12, 1983, p. 3721-3728.

Both oxidized and as-received RBSN were densified in two-step sintering processes as described previously and illustrated in Figure 3. As shown in the figure, the oxidized bars attained higher sintered densities than those not pretreated. The best results appear to be obtained using 30 to 60 minutes oxidation time. An increase of 3 to 5% in density is noted for oxidized specimens compared with unoxidized. Weight change measurements on the sintered bars revealed that unoxidized bars always gained weight (up to 2%) during sintering while oxidized bars usually lost weight (up to 2.5%) depending on the amount of preoxidation (percent increase in  $\text{SiO}_2$  content). As the specimen becomes silica-rich, lower melting compositions may be formed. Also, preoxidation of the specimens may result in compositions where the partial pressure of  $\text{SiO}$  produced in the specimen is greater than that produced by the cover powder resulting in weight loss during sintering.

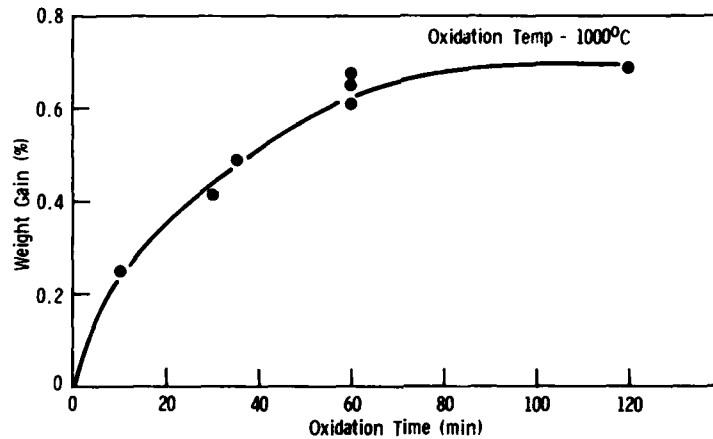


Figure 2. Effect of oxidation on as-received RBSN specimens.

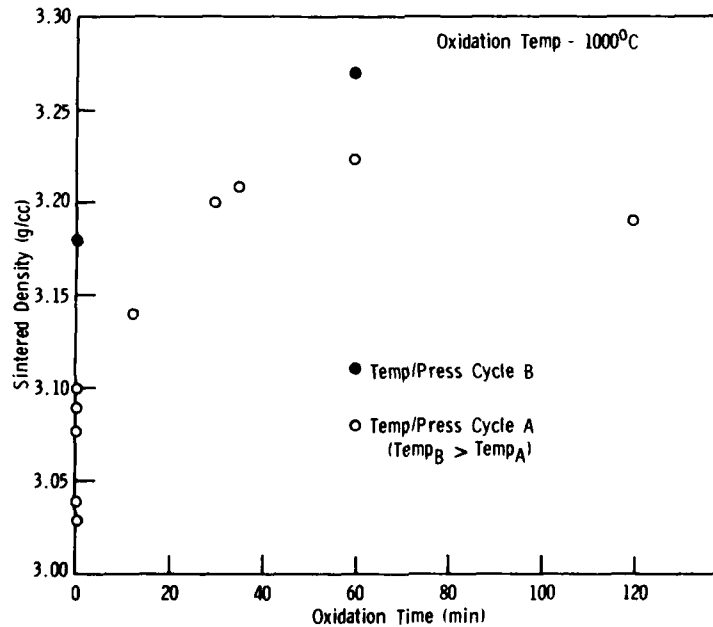


Figure 3. Effect of preoxidation time and sintering cycle on sintered density.

X-ray diffraction results on sintered specimens indicate that grain boundary phases found are similar to those in unsintered specimens that have not been pre-oxidized. The principal boundary phase appears to be  $Y_5(SiO_4)_3N$  which may remain after sintering or convert to  $Y_{4.67}(SiO_4)_3O$ . The preoxidized specimens exhibit a change in the grain boundary phase after sintering from  $Y_5(SiO_4)_3N$  to  $Y_2SiO_5$  and occasionally some  $Y_2Si_2O_7$ . All of the  $\alpha$ - $Si_3N_4$  converted to the  $\beta$  form.

### Mechanical Properties

Some bars were tested in the as-sintered condition to determine RT modulus of rupture. Values ranged from 490 to 670 MPa. Fracture origins were observed to be primarily pores/pore clusters remaining in the specimens after sintering.

Other specimens were machined to dimensions previously cited for stress-rupture testing at temperatures of 700°C, 1000°C, and 1200°C under a 300 MPa stress. The specimens were held under these temperature/stress conditions for 750 to 1000 hours as shown in Table 1. Stepped-temperature stress-rupture (STSR) procedures from 800°C were used in 100°C/24-hour increments prior to isothermal testing at temperatures of 1000°C and 1200°C. Specimens surviving the stress-rupture test for 750 to 1000 hours were unloaded, cooled, and removed from the furnace for measurements of weight change, permanent strain, and retained RT MOR, also shown in Table 1.

Table 1. CREEP, OXIDATION, AND MOR DATA

Stress Rupture (Temp. °C)	Time (hr)	$\epsilon$ (hr <sup>-1</sup> ) (% Perm. Strain)	Oxidation Rate Const. (kg <sup>2</sup> m <sup>-4</sup> s <sup>-1</sup> )	RT MOR After Stress- Rupture Test
700	750	n.m.	n.m.	570
1000	750	n.m.	$2.0 \times 10^{-13}$	390-510
1200	1000	$1.0-2 \times 10^{-6}$ (0.1-0.2%)	$6.5 \times 10^{-13}$	486-670

(STSR from 800°C prior to all tests at 1000°C and 1200°C)

$\sigma = 300$  MPa

Measurements of weight gain on stress-rupture bars indicated that they have good oxidation resistance. Oxidation rate values calculated for specimens surviving at 1000°C and 1200°C were  $2.0 \times 10^{-13}$  and  $6.5 \times 10^{-13}$  kg<sup>2</sup>m<sup>-4</sup>s<sup>-1</sup>, respectively. Essentially no change in weight was noted at 700°C within the limits and accuracy of measurement. Specimens appeared uniform in surface color with a very thin, coherent oxidation layer.

The amount of permanent strain in each stress-rupture bar was determined by measuring bar curvature under a microscope and by a photographic technique.<sup>3</sup> Within the limits of measurement, bars tested at 1200°C for 1000 hours exhibited 0.1 to 0.2% permanent strain. An upper limit strain rate of 1.5 to  $2.0 \times 10^{-6}$ /hr was calculated from these measurements. It is regarded as an upper limit because the measurement includes both the primary and secondary (steady state) creep stages. Permanent strain values were not measurable for specimens tested at 700°C and 1000°C (values less than 0.1%).

Retained RT MOR values are shown in Table 1 for stress-rupture survivors. Values range from 390 to 670 MPa and are primarily strength limited by residual pores as observed at the fracture origins (Figure 4). Occasionally, an agglomerate, as shown in Figure 5 was observed at the origin of failure.

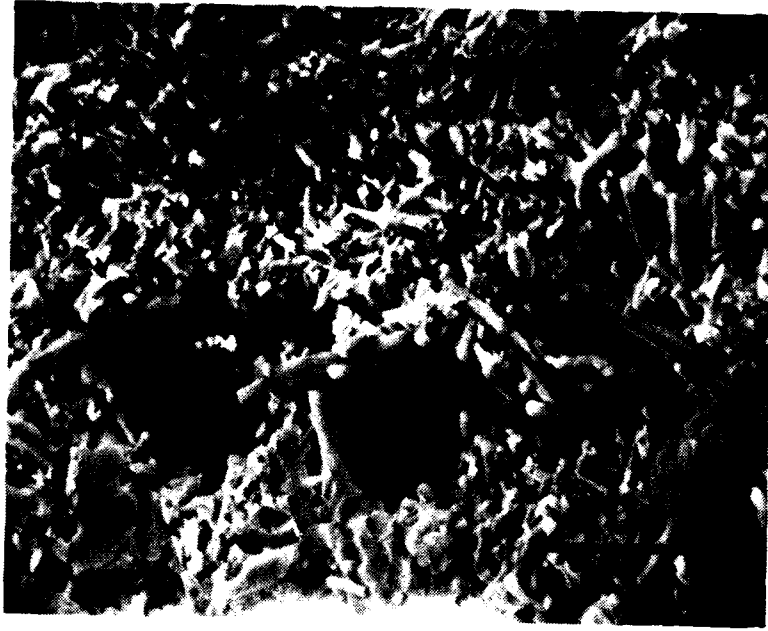


Figure 4. Pore cluster at fracture origin of sintered specimen.

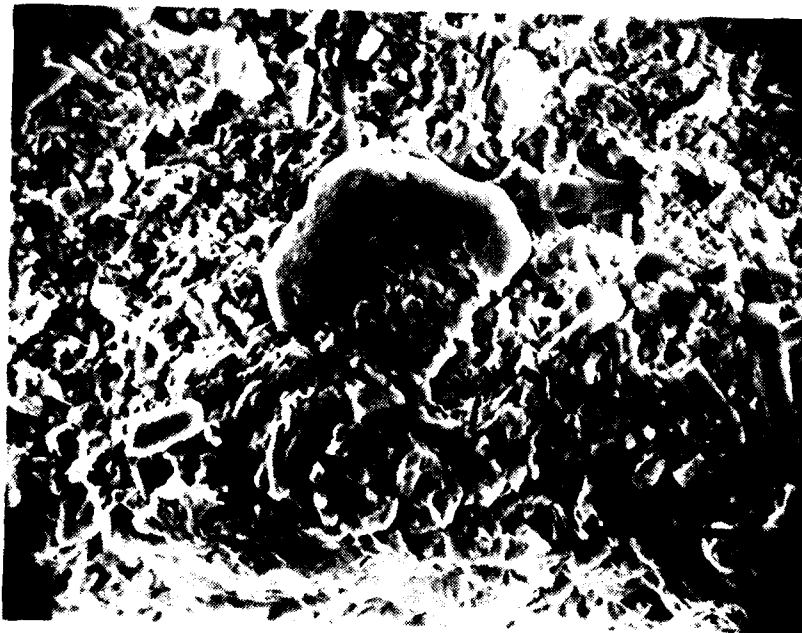


Figure 5. Agglomerate at fracture origin of sintered specimen.

## SUMMARY

Reaction-bonded  $\text{Si}_3\text{N}_4$  bars containing 6% $\text{Y}_2\text{O}_3$  and 1% $\text{Fe}_2\text{O}_3$  were sintered to high density (>97%) using high  $\text{N}_2$  pressures (up to 8.0 MPa) and temperatures of 1875°C to 2000°C. Bars which were oxidized before sintering had higher sintered densities than those which were not preheat treated. The development of more silica-rich compositions resulted in the formation of yttrium-silicate phases which appear to promote densification for the sintering parameters used in this study.

Stress-rupture tests on sintered bars (with and without preoxidation) show no intermediate temperature (700°C to 1000°C) degradation under stress (300 MPa) for times of 750 to 1000 hours. The bars exhibited good oxidation resistance to 1200°C temperatures and low residual permanent strain was noted after 1000 hours at 1200°C. Retained MOR values after stress-rupture exposure at 700°C, 1000°C, and 1200°C were similar to as-sintered material values. Strength-limiting defects were usually residual pores and pore clusters.

## ACKNOWLEDGMENT

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