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SINTERING OF Si3N4 UNDER NITROGEN PRESSURE. (U)

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DAAG46-79-C-0101

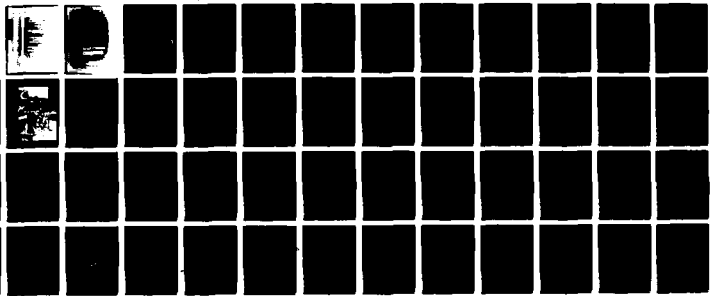
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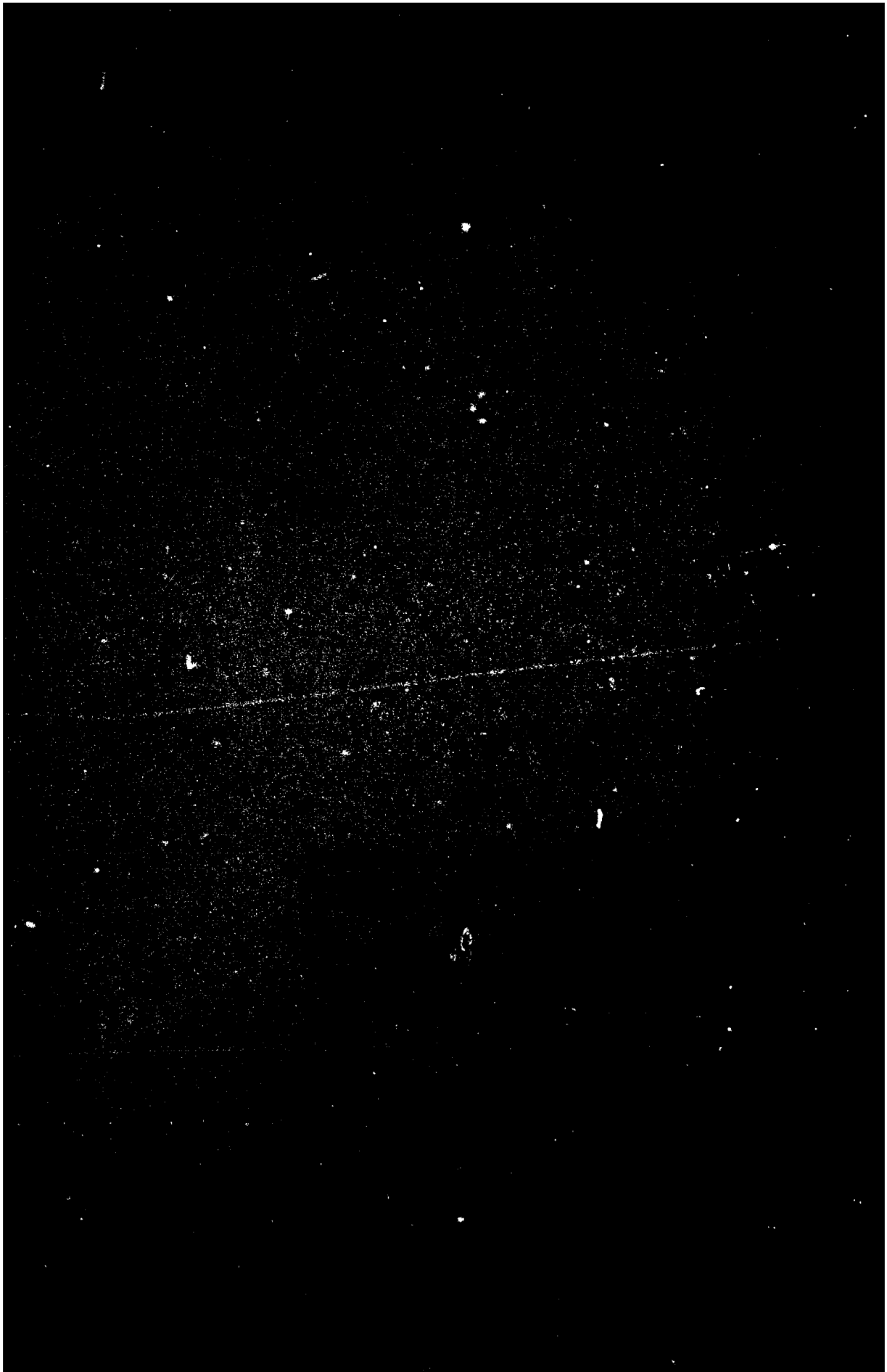
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well as the  $Al_2O_3$  in the mixtures to improve the oxidation resistance of the samples still further, but it was found that it was more difficult to obtain high densities for these materials than it was for  $Si_3N_4 \cdot 15Y_2O_3$  materials. For that reason, preliminary measurements were made on samples containing 15%  $Y_2O_3$ . Strength measurements on as-pressed bars which were cold isostatic pressed and sintered showed that a  $Si_3N_4 \cdot 15Y_2O_3 \cdot 1Al_2O_3$  material with a modulus of rupture as high as 514 MPa (75 ksi) could be prepared.

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FOREWORD

This work has been sponsored by the Army Materials and Mechanics Research Center under AMMRC/DOE Interagency Agreement EC-76-A-1017-002 as part of DOE, Division of Transportation Energy Conservation, Highway Vehicle Systems Heat Engine Program. Mr. George E. Gazza was the Program Monitor.

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Sintering of  $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3\text{-Al}_2\text{O}_3$  Materials  
Under Nitrogen Pressure

SUMMARY

A previous study on contract DAAG46-78-C-0017 showed that a sintered  $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3$  material similar in oxidation resistance to hot-pressed  $\text{Si}_3\text{N}_4$  could be prepared by minimizing the  $\text{Al}_2\text{O}_3$  content in the sample while maintaining high densities. This was achieved by using a high surface area SN402  $\text{Si}_3\text{N}_4$  powder and a high nitrogen overpressure during high temperature ( $1800^\circ\text{C}$ ) sintering. In this study attempts were made to reduce the amount of  $\text{Y}_2\text{O}_3$  as well as the  $\text{Al}_2\text{O}_3$  in the mixtures to improve the oxidation resistance of the samples still further, but it was found that it was more difficult to obtain high densities for these materials than it was for  $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3$  materials. For that reason, preliminary measurements were made on samples containing 15%  $\text{Y}_2\text{O}_3$ . Strength measurements on as-pressed bars which were cold isostatic pressed and sintered showed that a  $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3\cdot 1\text{Al}_2\text{O}_3$  material with a modulus of rupture as high as 514 MPa (75 ksi) could be prepared.

## INTRODUCTION

Silicon nitride is presently being evaluated for use in the Stirling, gas turbine and diesel engines. Of the various forms of silicon nitride, hot pressed silicon nitride has the properties which most nearly meet the requirements for many of these engine applications. The main deterrent to its use is excessive cost. There, therefore, is a great deal of interest in developing a method which is less expensive for producing a high strength silicon nitride material. One of these methods which can produce material with densities which approach those of hot-pressed  $\text{Si}_3\text{N}_4$  materials involves pressureless sintering of cold pressed silicon nitride powders containing sintering aids. In most cases, in these sintering studies, the density which can be achieved is limited by the temperature at which dissociation of the silicon nitride takes place. By heating the samples under high  $\text{N}_2$  pressures, higher sintering temperatures can be used without excessive decomposition of the  $\text{Si}_3\text{N}_4$ .

In a previous study (Refs. 1 and 2),  $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$  materials were selected for study because of the excellent high temperature properties they had when they were hot pressed. That study showed that some  $\text{Al}_2\text{O}_3$  addition was necessary to produce high density material by pressureless sintering, but the oxidation resistant properties of these materials decreased with increasing amounts of  $\text{Al}_2\text{O}_3$  additions. The amount of  $\text{Al}_2\text{O}_3$ , required to obtain high density material, could be reduced by using a GTE SN402 powder instead of an AME powder and by the use of high nitrogen pressure sintering.

In this program, attempts were made to further investigate this system to optimize the properties and obtain reproducible results. Monitoring of the shrinkage factors during sintering were also made to obtain information which could be used in the design of complex molds for cold pressing.

## BACKGROUND

In early studies on pressureless sintering, Terwilliger and Lange heated pellets of AME  $\text{Si}_3\text{N}_4 \cdot 5$  w/o MgO at  $1570^\circ\text{C}$  and obtained 85-90% of theoretical density (Ref. 3). In later studies, higher densities were achieved by Rowcliffe and Jorgensen using GTE and KBI-AME  $\text{Si}_3\text{N}_4$  powders,  $\text{Y}_2\text{O}_3$  additives and higher temperatures (Ref. 4). Ada, Kaneno and Yamamoto (Ref. 5), Buljan and Kleiner (Ref. 6), and Masaki and Kamigaito (Ref. 7) also conducted similar sintering studies, but with a variety of additives and they also produced high density material. Mitomo and co-workers (Refs. 8,9), Priest and Gazza (Ref. 10) and Greskovich, Prochazka, and Rosolowski (Ref. 11) showed that there was a definite advantage in carrying out the sintering under high  $\text{N}_2$  pressures. Priest and Gazza not only obtained very high densities but also showed that there was very little weight loss in the  $\text{Si}_3\text{N}_4$  pellets when high  $\text{N}_2$  pressures were used even at temperatures as high as  $1900^\circ\text{C}$ .

At the same time that these high  $\text{N}_2$  pressure studies were being conducted, a preliminary sintering investigation was also being carried out at UTRC with high  $\text{N}_2$  pressures using GTE and AME  $\text{Si}_3\text{N}_4$  powders and MgO,  $\text{CeO}_2$  and  $\text{Y}_2\text{O}_3$  additives. The objective was to determine which  $\text{Si}_3\text{N}_4$  powders and which additives yielded the highest density products and to ascertain if there was a real advantage in employing  $\text{N}_2$  overpressure during the sintering process.

Some of the materials studies were AME  $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ , AME  $\text{Si}_3\text{N}_4 \cdot 5\text{MgO}$ , AME  $\text{Si}_3\text{N}_4 \cdot 20\text{CeO}_2$ , GTE  $\text{Si}_3\text{N}_4 \cdot 5\text{CeO}_2$ , GTE  $\text{Si}_3\text{N}_4 \cdot 20\text{CeO}_2$ , and GTE  $\text{Si}_3\text{N}_4 \cdot 5\text{MgO}$ . In all of these studies, for the same material, the samples run under high  $\text{N}_2$  pressure (higher sintering temperature) had the highest densities. The highest density was obtained for materials which contained 15%  $\text{Y}_2\text{O}_3$ . Excellent ambient and high temperature properties had been obtained at UTRC for hot pressed samples (Ref. 12) so that the  $\text{Si}_3\text{N}_4$ - $\text{Y}_2\text{O}_3$  system was selected for more detailed pressureless sintering studies.

In a previous program (Ref. 1) a study of pressureless sintering of  $\text{Si}_3\text{N}_4$  powders with various additives was conducted. Of the various additives studied,  $\text{CeO}_2$ , MgO, etc.,  $\text{Y}_2\text{O}_3$  was selected as the one which gave the best preliminary results. Of particular interest were the results using high  $\text{N}_2$  pressures over the specimens to prevent weight loss and thus obtain high densities. On the basis of these results, more detailed high  $\text{N}_2$  pressure studies were conducted using the  $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$  system.

In those studies, it was shown that some other additive, in this case  $\text{Al}_2\text{O}_3$ , was necessary to promote sintering of  $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$  materials unless hot pressing techniques were employed. For the AME  $\text{Si}_3\text{N}_4$  powder used, about 8%  $\text{Al}_2\text{O}_3$  additions resulted in nearly theoretical density samples for  $1730^\circ\text{C}$ , 1 atm  $\text{N}_2$  firings. When 20 atm of  $\text{N}_2$  was used over the samples ( $1800^\circ\text{C}$  firing temperatures) these amounts of  $\text{Al}_2\text{O}_3$  additions could be reduced to 4 to 5%. For GTE SN402 powder studies, the corresponding amounts of  $\text{Al}_2\text{O}_3$  required were 2 to 3% for  $1730^\circ\text{C}$  firings and less than 1% for  $1800^\circ\text{C}$ , 20 atm firings. It was expected that these percentages would vary for different batches of  $\text{Si}_3\text{N}_4$  powders, but they did show a trend.

The oxidation resistance of these  $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3$  materials correlated well with the amount of  $\text{Al}_2\text{O}_3$  in the samples. Pressureless sintered SN402  $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3\cdot 0.6\text{Al}_2\text{O}_3$  showed less weight gain in air than hot pressed  $\text{Si}_3\text{N}_4\cdot \text{MgO}$  over an 80 hr period at  $1350^\circ\text{C}$ . In addition, it was shown that the oxidation resistance improved with increasing density. As was expected, the MOR of the samples also increased with increasing density. Thus, that study showed that it is desirable to use the least amount of  $\text{Al}_2\text{O}_3$  which will produce high density pellets, and high  $\text{N}_2$  pressure sintering is a way of obtaining the high densities with smaller amounts of  $\text{Al}_2\text{O}_3$  additions.

Other items studied in that program involved methods of adding the  $\text{Al}_2\text{O}_3$  and the fabrication of airfoils by cold pressing of the powders and sintering. It was shown that some of the most uniform samples were prepared when the  $\text{Al}_2\text{O}_3$  was added from the balls in the ball milling operation. While this technique appeared to produce a homogeneous distribution of  $\text{Al}_2\text{O}_3$  in the powder mixture, it had the drawback of being dependent on the number of balls used and other variables. Preliminary studies were also conducted on sintering airfoil shapes from the powders studied. The most difficult part of that work was the preparation of dies and developing techniques to form and recover complex shapes. A number of airfoil shapes were prepared from  $\text{Si}_3\text{N}_4\text{-}15\text{Y}_2\text{O}_3$  material by pressureless sintering.

## EXPERIMENTAL PROCEDURES

The powders used in this study were a  $\text{Si}_3\text{N}_4$  powder (high purity, -325 mesh)\* and a SN402  $\text{Si}_3\text{N}_4$  powder\*\*. The yttria was formed by bringing Molycorp CP grade yttrium oxalate up to  $1000^\circ\text{C}$  in air and down over a 24 hr period. The alumina used was Fisher CP grade material. Most powder mixtures were formulated by adding  $\text{Y}_2\text{O}_3$  to each of the  $\text{Si}_3\text{N}_4$  powders, adding methanol as the fluid media and ball milling the powders. The powders were dried and then used in these investigations. The alumina was added to base mixtures by manual mixing of the powders in a mortar and pestle.

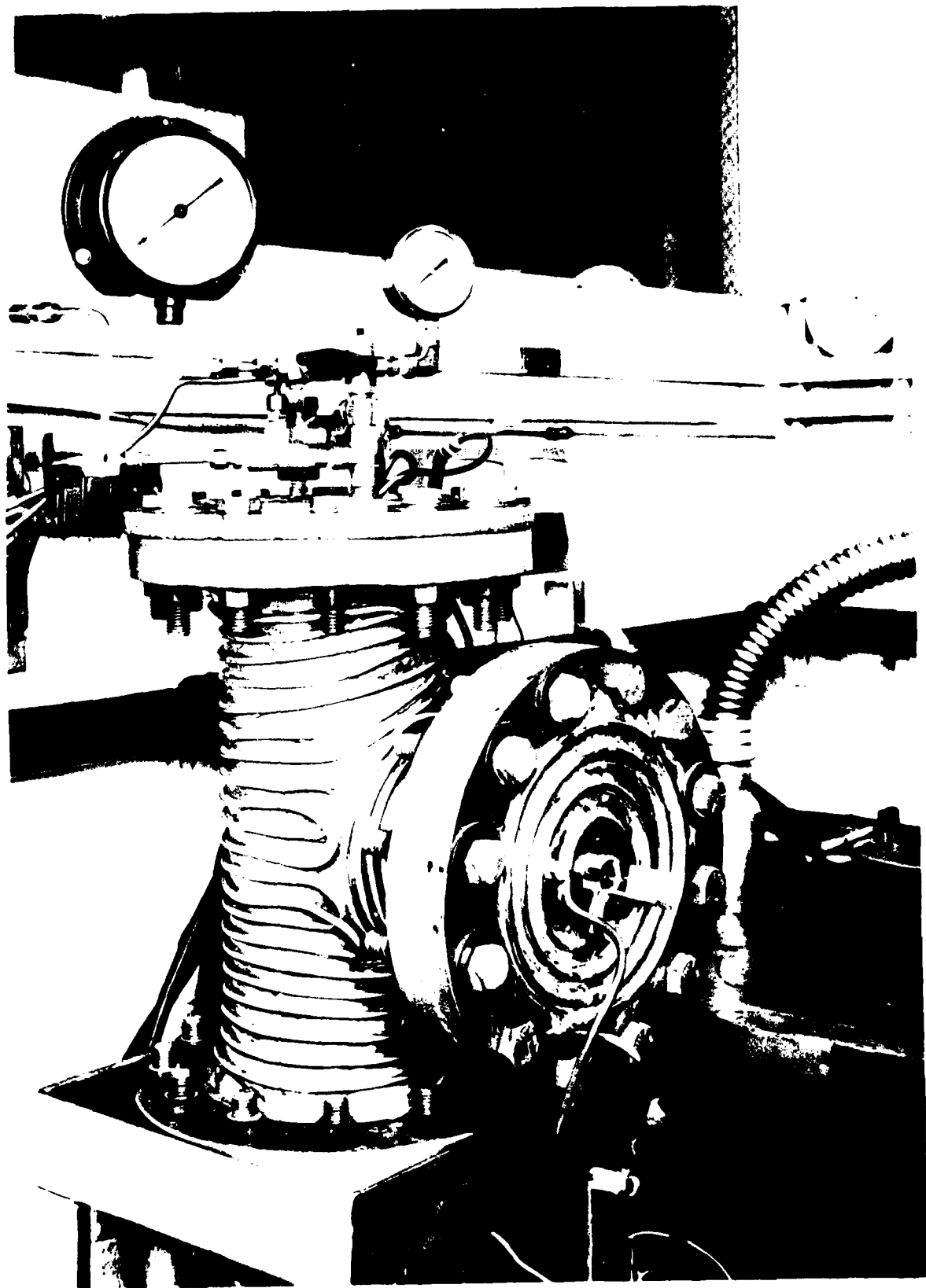
The powders were pressed into pellets or rectangular bars and fired for various times at  $1730$  or  $1800^\circ\text{C}$  in BN crucibles with BN covers. The  $1730^\circ\text{C}$  runs were conducted in a graphite resistance furnace under  $\text{N}_2$  at 1 atm pressure. The temperature of the sample was obtained from an optical pyrometer reading and a plot relating the pyrometer readings to temperature obtained from thermocouple measurements. The high  $\text{N}_2$  pressure,  $1800^\circ\text{C}$ , runs were conducted in a carbon resistance furnace where the temperature was obtained from the power setting and a plot relating the power setting to the melting point of several materials. The high nitrogen pressure sintering apparatus was described in detail in Ref. 1 and is shown in Fig. 1. This unit could be operated at temperatures of  $1800^\circ\text{C}$ , with internal pressures as high as 20 atm. Specimens 5.0 cm in dia by 7.6 cm high could be accommodated within the graphite heating element.

The typical sintering procedure for the high pressure apparatus involved first placing the specimen within the heating chamber and securing the high pressure flanges. A vacuum pump was then used to evacuate the system so that leak-tight conditions could be assured. Nitrogen gas (Matheson Ultra High purity) was then introduced to a pressure of 10 atm and held at this level until the integrity of all connections for water cooling, gas, and power was established. The power was then applied to bring the heating chamber up to the desired level for that particular sintering experiment. A cold nitrogen starting pressure of 10 atm yielded a system pressure of 20 atm at a chamber temperature of  $1800^\circ\text{C}$ . At the end of the sintering interval, the temperature was slowly lowered with the system under pressure. At room temperature, the high pressure gas was vented and the specimen was removed. The densities reported herein were obtained by weighing the samples and using the volume calculated from measured dimensions.

\*Kawecki Berylko Industries of Boyertown, PA

\*\*GTE Sylvania, Precision Materials Group of Towanda, PA

HIGH N<sub>2</sub> PRESSURE FURNACE



## EXPERIMENTAL RESULTS

### Compositional Variations in $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3\text{-Al}_2\text{O}_3$ System

#### Sintering under 1.0 Atmospheres $\text{N}_2$

In a previous program (Ref. 1) it had been shown that  $\text{Si}_3\text{N}_4$  powder when mixed with 15%  $\text{Y}_2\text{O}_3$  and aluminum oxide and pressed and sintered produced high density material. The most oxidation resistant materials were those in which the aluminum oxide content was kept to a minimum. The lowest  $\text{Al}_2\text{O}_3$  content material which still had a high density was obtained using a high surface area SN402  $\text{Si}_3\text{N}_4$  powder.

In this program, it was decided to examine materials prepared using the SN402 powder with decreasing  $\text{Y}_2\text{O}_3$  additions to determine if reducing the  $\text{Y}_2\text{O}_3$  would have the same effect of improving the oxidation resistance of the materials as reducing the  $\text{Al}_2\text{O}_3$  additions did. Base mixtures of 90  $\text{Si}_3\text{N}_4\text{-10Y}_2\text{O}_3$ , 92  $\text{Si}_3\text{N}_4\text{-8Y}_2\text{O}_3$  and 94  $\text{Si}_3\text{N}_4\text{-6Y}_2\text{O}_3$  were prepared. Each of these mixtures were ball milled with aluminum oxide balls for 48 hrs. The desired amount of aluminum oxide was added to these mixtures in varying amounts (1 to 10%) and cold pressed pellets were prepared for sintering. The added aluminum oxide in all cases was mixed with the base powder using a mortar and pestle.

The first three sets of experiments with these base mixtures were all carried out at sintering conditions of  $1730^\circ\text{C}$  for 1 hr under flowing nitrogen. In Table 1 are listed the experimental data for the  $\text{Si}_3\text{N}_4\text{-6Y}_2\text{O}_3$  base materials. The percent shrinkage in height and diameter of each sample in this series was determined. The shrinkage percentage was obtained by taking the difference between the pre- and post-sintered dimension and dividing this difference by the initial or cold pressed pellet dimensions. This information for the  $\text{Si}_3\text{N}_4\text{-6Y}_2\text{O}_3$  base materials is also listed in Table 1. These same experimental data for the  $\text{Si}_3\text{N}_4\text{-8Y}_2\text{O}_3$  base materials are listed in Table 2 and the data for the  $\text{Si}_3\text{N}_4\text{-10Y}_2\text{O}_3$  base materials are presented in Table 3.

In examining the data for the first materials (Table 1) it can be seen that the densities of these pellets did not exceed  $2.70 \text{ g/cm}^3$  and fairly low densities were achieved until approximately 7%  $\text{Al}_2\text{O}_3$  was added. For the  $\text{Si}_3\text{N}_4\text{-8Y}_2\text{O}_3$  base materials (Table 2) reasonable densities ( $>2.90 \text{ g/cm}^3$ ) were reached when 4 to 8%  $\text{Al}_2\text{O}_3$  had been added. It was found that when more than this amount of  $\text{Al}_2\text{O}_3$  was added to these materials the final densities started to decrease. A similar examination of the tabulated data in Table 3 for the  $\text{Si}_3\text{N}_4\text{-10Y}_2\text{O}_3$  base series of materials indicates that densities of over  $3.00 \text{ g/cm}^3$  can be attained with as little as 4%  $\text{Al}_2\text{O}_3$  additions.

Table 1

Sintering Response of  $\text{Si}_3\text{N}_4\text{-6Y}_2\text{O}_3$  Materials

Run Number	% $\text{Al}_2\text{O}_3$ Addition	Initial			Final			Shrinkage	
		Height cm	Dia. cm	Density $\text{g/cm}^3$	Height cm	Dia. cm	Density $\text{g/cm}^3$	Height %	Diameter %
LPS 259	1	.302	.792	1.008	.252	.664	1.327	16.56	16.16
LPS 259	2	.346	.792	.992	.276	.634	1.534	20.23	19.95
LPS 259	3	.398	.792	1.004	.330	.650	1.506	17.09	17.93
LPS 260	4	.316	.792	.963	.245	.624	1.695	22.49	21.21
LPS 260	5	.355	.792	.982	.258	.573	2.119	27.42	27.65
LPS 260	6	.414	.792	.97	.296	.562	2.272	28.50	29.04
LPS 261	7	.270	.792	1.048	.182	.534	2.709	32.59	32.58
LPS 261	8	.331	.792	1.053	.214	.538	2.645	35.35	32.07
LPS 261	9	.354	.792	1.036	.240	.544	2.739	32.20	31.31
LPS 261	10	.392	.792	1.045	.273	.556	2.663	30.36	29.80

Table 2

Sintering Response of  $\text{Si}_3\text{N}_4\text{-8Y}_2\text{O}_3$  Materials  
(Sintered at  $1730^\circ\text{C}$  for 1 hr)

Run Number	% $\text{Al}_2\text{O}_3$ Addition	Initial			Final			Shrinkage	
		Height cm	Dia. cm	Density $\text{g/cm}^3$	Height cm	Dia. cm	Density $\text{g/cm}^3$	Height %	Diameter %
LPS 244	0	.306	.792	1.050	-	-	-	-	-
LPS 234	1	.293	.792	1.066	.223	.625	1.600	23.89	21.09
LPS 242	2	.279	.792	1.136	.196	.563	2.336	29.75	28.91
LPS 243	3	.297	.792	1.094	.206	.566	2.169	30.64	28.54
LPS 245	3	.271	.792	1.156	.213	.544	2.317	21.40	31.31
LPS 241	4	.285	.792	1.133	.185	.530	2.952	35.09	33.08
LPS 246	4.5	.295	.792	1.092	.190	.520	3.023	35.59	34.34
LPS 236	5	.307	.792	1.015	.204	.523	2.921	33.55	33.96
LPS 235	6	.303	.792	1.072	.198	.519	2.896	34.65	34.47
LPS 237	7	.300	.792	1.022	.186	.532	2.902	38.00	32.83
LPS 238	8	.259	.792	1.220	.172	.538	2.847	33.59	32.07
LPS 239	9	.276	.792	1.132	.184	.548	2.746	33.33	30.68
LPS 240	10	.260	.792	1.209	.187	.560	2.579	28.08	29.29

Table 3

Sintering Response of  $\text{Si}_3\text{N}_4$ - $10\text{Y}_2\text{O}_3$  Materials  
(Sintered at  $1730^\circ\text{C}$  for 1 hr)

Run Number	% $\text{Al}_2\text{O}_3$ Addition	Initial			Final			Shrinkage	
		Height cm	Dia. cm	Density g/cm <sup>3</sup>	Height cm	Dia. cm	Density g/cm <sup>3</sup>	Height %	Diameter %
LPS 253	1	.176	.792	1.061	.134	.605	1.785	23.86	23.61
LPS 249	2	.192	.792	1.043	.126	.522	2.744	34.38	34.09
LPS 254	3	.298	.792	1.017	.208	.558	2.365	30.20	29.55
LPS 248	4	.289	.792	1.037	.186	.516	3.116	35.64	34.85
LPS 255	5	.288	.792	1.020	.196	.539	2.804	31.94	31.94
LPS 250	6	.176	.792	1.073	.115	.521	3.032	34.66	34.22
LPS 256	7	.288	.792	1.059	.195	.541	2.869	32.29	31.69
LPS 251	8	.200	.792	1.003	.128	.531	2.858	36.00	32.95
	8	.290	.792	1.447	.197	.536	2.934	32.07	32.32
	8	.323	.792	1.063	.221	.540	2.911	31.59	31.82
	8	.402	.792	1.019	.267	.538	2.934	33.58	32.09
LPS 252	10	.200	.792	1.041	.136	.556	2.597	32.00	29.80

The densities from these tables are shown as functions of aluminum oxide additions in Fig. 2. For ease of comparison a smoothed curve for each of the three series of materials was drawn. The density as a function of aluminum oxide for  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$  materials obtained in the previous program are also shown in Fig. 2. This figure shows that more  $\text{Al}_2\text{O}_3$  is required to produce high density specimens when the amount of  $\text{Y}_2\text{O}_3$  in the mixture is decreased. For the three series of materials studied in this program at the high concentrations (10%) of  $\text{Al}_2\text{O}_3$  additions the final densities began to decrease.

The overall shrinkage percentages for the  $\text{Si}_3\text{N}_4\text{-6Y}_2\text{O}_3$  series of materials (Table 1) are less than those for the other two series. When the final pellet density approaches  $3.0 \text{ g/cm}^3$  these linear shrinkage factors reach 35% (see LPS 246, Table 2 and LPS 248, Table 3).

#### Sintering Under 20 Atmospheres $\text{N}_2$

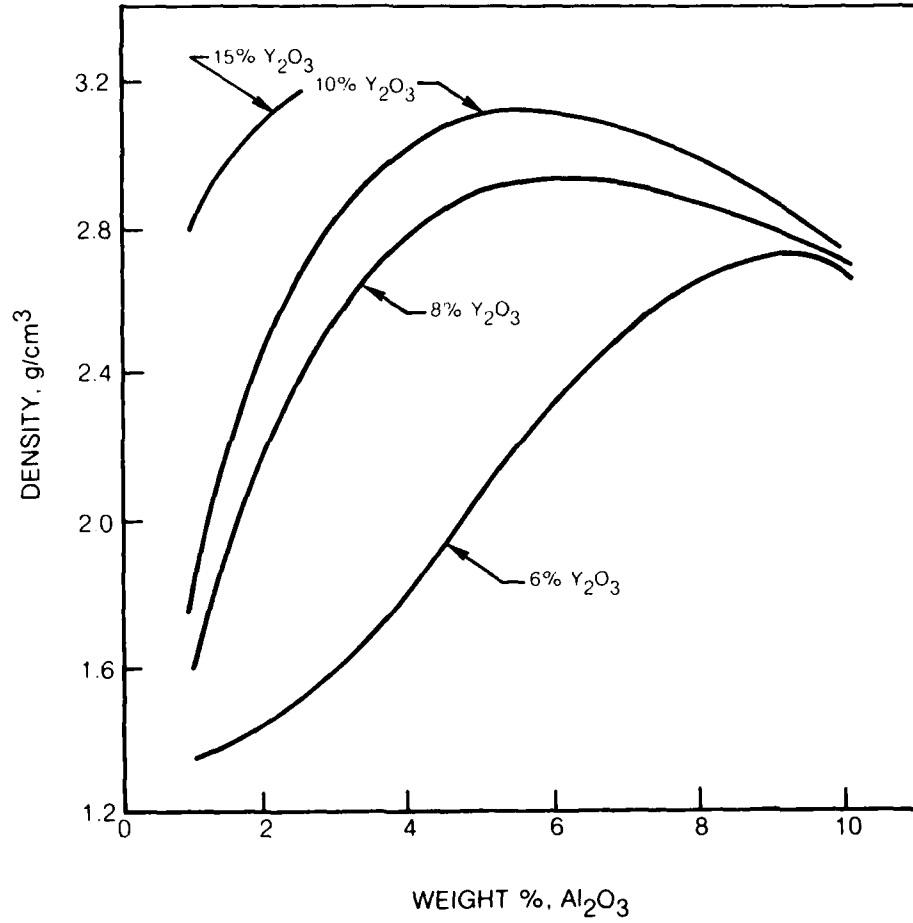
Using these same mixtures, more cold pressed pellets with specific  $\text{Al}_2\text{O}_3$  additions were prepared for higher temperature sintering studies. These series of samples were sintered at  $1800^\circ\text{C}$  for 1 hr under 20 atms of nitrogen. The experimental data obtained from these sintering studies are listed in Table 4. The densities as a function of aluminum oxide addition for these materials as well as for a  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$  material taken from the previous study are shown in Fig. 3. The accompanying shrinkage percentages are listed in Table 4.

In examining the final density data listed in Table 4, it is interesting that no densities in this series fell below  $2.65 \text{ g/cm}^3$ . When comparing these results with those tabulated in Tables 1, 2 and 3, it can be seen that the  $1800^\circ\text{C}$  sintering conditions in most cases yielded samples with higher final densities than those of corresponding compositions sintered at  $1730^\circ\text{C}$ .

The curves of density as a function of aluminum oxides additions shown in Fig. 3 indicate that although the density increases with increasing aluminum oxide addition is fairly linear, there is a crossover between the  $\text{Si}_3\text{N}_4\text{-6Y}_2\text{O}_3$  and  $\text{Si}_3\text{N}_4\text{-8Y}_2\text{O}_3$  base materials at low  $\text{Al}_2\text{O}_3$  additions. The decrease in density for samples with high aluminum oxide additions which was observed with the  $1730^\circ\text{C}$  sintering condition only occurred during the  $1800^\circ\text{C}$  sintering with the  $\text{Si}_3\text{N}_4\text{-10Y}_2\text{O}_3$  base materials. Again it is shown that with lower  $\text{Y}_2\text{O}_3$  content, more  $\text{Al}_2\text{O}_3$  is required to achieve the higher density levels with these mixtures. The shrinkage percentages listed in Table 4 for height and diameters of the pellets were again approximately 35% each for the higher density samples.

DENSITY OF A FUNCTION OF  $\text{Al}_2\text{O}_3$  ADDITIONS FOR VARIOUS  $\text{Si}_3\text{N}_4 - \text{Y}_2\text{O}_3$  MATERIALS

(1730°C SINTERING TEMP., 1 HOUR)



DENSITY AS A FUNCTION OF  $\text{Al}_2\text{O}_3$  ADDITIONS FOR VARIOUS  $\text{Si}_3\text{N}_4 - \text{Y}_2\text{O}_3$  MATERIALS

1800°C SINTERING TEMPERATURE  
 1 HOUR  
 20 ATMOSPHERES  $\text{N}_2$

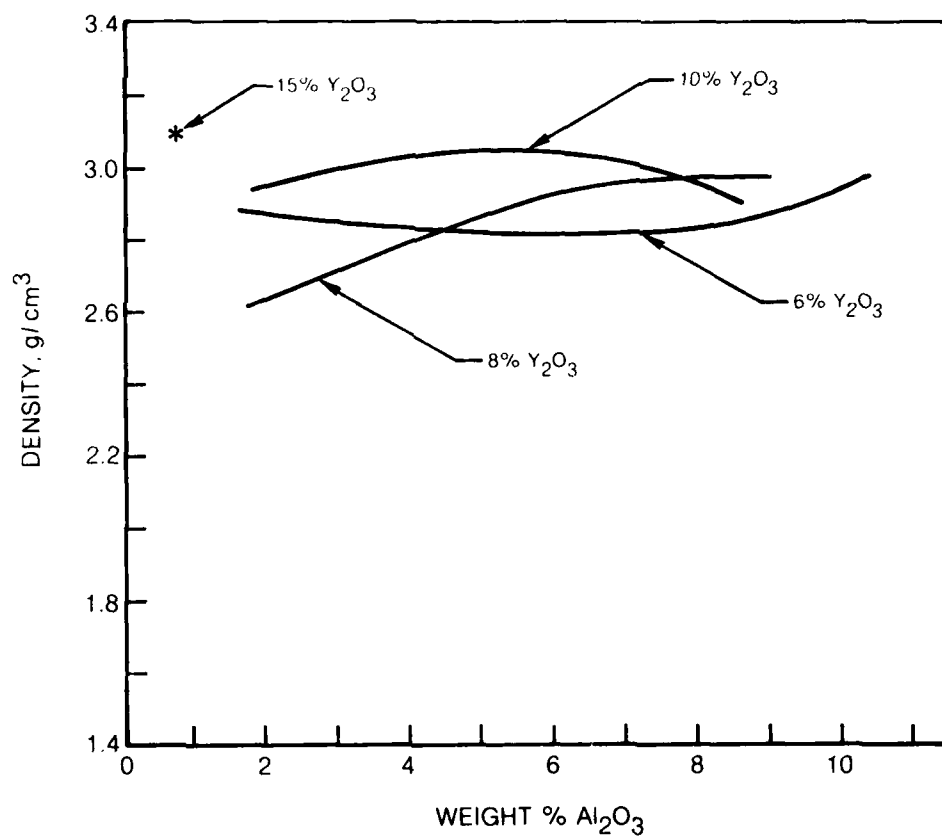


Table 4

Sintering Response of  $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$  Materials  
(Sintered at 1800°C for 1 hr  
20 Atmospheres Nitrogen)

Number	Composition $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$	% $\text{Al}_2\text{O}_3$ Addition	Initial			Final			Shrinkage	
			Height cm	Dia. cm	Density g/cm <sup>3</sup>	Height cm	Dia. cm	Density g/cm <sup>3</sup>	Height %	Dia. %
18A	94:6	2	.279	.792	1.095	.171	.525	2.884	38.71	33.71
15A	94:6	6	.280	.792	1.081	.177	.525	2.849	36.79	33.71
15B	94:6	8	.335	.792	1.080	.222	.532	2.826	33.73	32.83
15C	94:6	10	.400	.792	1.105	.262	.530	2.962	34.50	33.08
18B	92:8	2	.350	.792	1.040	.212	.525	2.653	39.43	33.71
16A	92:8	4	.300	.792	1.008	.185	.530	2.785	38.33	33.08
16B	92:8	6	.340	.792	1.056	.222	.532	2.929	34.71	32.83
16C	92:8	8	.423	.792	1.053	.282	.530	2.970	33.33	33.08
18C	90:10	2	.425	.792	1.048	.266	.520	2.933	37.41	34.34
17A	90:10	4	.270	.792	1.114	.173	.517	3.045	35.93	34.72
17B	90:10	6	.335	.792	1.087	.215	.520	3.046	35.82	34.34
17C	90:10	8	.405	.792	1.097	.273	.530	2.910	32.59	33.08

### Sintering of $\text{Si}_3\text{N}_4$ - $15\text{Y}_2\text{O}_3$ Materials

These preliminary studies indicated that lowering the  $\text{Y}_2\text{O}_3$  content of  $\text{Si}_3\text{N}_4$ - $\text{Y}_2\text{O}_3$  materials made them more difficult to sinter to high densities. The high densities for the lower  $\text{Y}_2\text{O}_3$  content materials could be achieved by increasing the  $\text{Al}_2\text{O}_3$  additions or possibly by modifying the processing conditions. Since it was of more interest at this point in this program to obtain some properties of these materials, samples were prepared in the SN402  $\text{Si}_3\text{N}_4$ - $15\text{Y}_2\text{O}_3$  system where sintering to high densities was easier to achieve. The base mixture ( $\text{Si}_3\text{N}_4$ - $15\text{Y}_2\text{O}_3$ ) was therefore prepared similar to the previously described preparation procedure. The aluminum oxide at levels of 1, 2 and 3% was added and manually mixed with a mortar and pestle prior to cold compaction. For this base mixture series rectangular bars were also sintered to produce samples for mechanical testing.

The sintering for these three aluminum oxide addition series was carried out at  $1730^\circ\text{C}$  for 1 hr under one atmosphere of flowing nitrogen. The experimental data for the  $\text{Si}_3\text{N}_4$ - $15\text{Y}_2\text{O}_3$ - $1\text{Al}_2\text{O}_3$  materials are listed in Table 5. The first two specimens (-A, -B) of each run were rectangular bars that were to be used for mechanical property testing. The remaining specimens (-C, -D, etc.) were of a pellet shape. Where final density information is lacking (such as specimen LPS 332A), in most cases this indicates that the rectangular bar was found to be cracked upon removal from the sintering furnace. In Table 6 are listed the experimental data for the  $\text{Si}_3\text{N}_4$ - $15\text{Y}_2\text{O}_3$ - $2\text{Al}_2\text{O}_3$  materials. Again the first two specimens of each run were rectangular bars and the remaining specimens were of a pellet geometry. Similar experimental data are provided for the  $\text{Si}_3\text{N}_4$ - $15\text{Y}_2\text{O}_3$ - $3\text{Al}_2\text{O}_3$  materials in Table 7. In this table, for experiments LPS 308 through LPS 314, the initial dimensions of the cold press rectangular bars were not recorded at the time of preparation.

In most cases the rectangular bars which were made for physical property measurements were not dimensionally square enough to obtain reliable density data. The density data therefore used for comparisons was always taken from measurements of the pellet specimens. In Table 8 are listed the average densities of pellets from each experimental run and also the average final pellet densities for samples containing the same percentage of aluminum oxide addition to the base mixtures. The density for the material containing 1%  $\text{Al}_2\text{O}_3$  is higher than would be expected from the previous study. The density of the 3%  $\text{Al}_2\text{O}_3$  addition series of materials ( $3.104 \text{ g/cm}^3$ ) is what would be expected from previous studies. The shrinkage factors fall in the same range as those obtained with the previous 6, 8 and 10%  $\text{Y}_2\text{O}_3$  containing mixtures and again the 35% shrinkage takes place when the higher densities are achieved.

Table 5

Sintering Response of  $\text{Si}_3\text{N}_4$ -15 $\text{Y}_2\text{O}_3$ -1Al $_2\text{O}_3$  Materials  
(Sintered at 1730°C for 1 hr)

Specimen Number	Initial				Final				Shrinkage			
	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Length %	Width %	Height %	Dia. %
LPS 331A	.243	3.166	.498	1.025	.156	2.1200	.356	2.864	33.04	28.51	35.80	
331B	.196	3.166	.498	1.118	.126	2.1250	.322	2.873	32.88	35.34	35.71	
331C	.294	(.792)		1.027	.162	(.517)		3.183			44.90	34.72
331D	.275	(.792)		1.036	.183	(.486)		2.899			33.45	38.64
331E	.244	(.792)		1.035	.176	(.492)		2.936			27.87	37.88
LPS 332A	.237	3.166	.498	1.067	.178	-	.347	-		30.32	24.89	
332B	.228	3.166	.498	.987	.147	-	.351	-		29.52	35.53	
332C	.308	(.792)		1.020	.190	(.511)		3.284			38.31	35.48
332D	.298	(.792)		1.008	.204	(.518)		2.840			31.54	34.60
332E	.296	(.792)		1.001	.202	(.510)		2.900			31.76	35.60

Table 6

Sintering Response of  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3\text{-2Al}_2\text{O}_3$  Materials  
(Sintered at 1730°C for 1 hr)

Specimen Number	Initial				Final				Shrinkage			
	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Length %	Width %	Height %	Diameter %
LPS 322A	.239	3.166	.498	.924	.146	2.034	.338	2.869	35.75	32.13	38.91	
322B	.344	3.166	.498	-	.338	-	-	-	-	-	-	37.63
322C	.294	(.792)		1.0	.180	(.494)		3.052			38.77	34.34
322D	.291	(.792)		1.01	.198	(.520)		3.205			31.96	
LPS 323A	.223	3.166	.498	.970	.136	2.056	.338	2.701	35.06	32.12	39.01	
323B	.214	3.166	.498	.889	.131	1.997	.342	2.670	36.92	31.33	38.79	32.83
323C	.292	(.792)		1.048	.207	(.532)		3.009			29.11	36.49
323D	.282	(.792)		1.037	.184	(.503)		2.721			34.75	
LPS 324A	.237	3.166	.498	.937	.159	1.820	.352	2.585	42.51	29.32	32.91	
324B	.185	3.166	.498	1.018	-	-	-	-	-	-	-	35.10
324C	.310	(.792)		1.010	.205	(.514)		2.982			33.87	34.97
324D	.296	(.792)		1.006	.190	(.515)		3.158			34.46	37.63
324E	.284	(.792)		1.015	.194	(.494)		2.929			31.69	
LPS 325A	.226	3.166	.498	.987	.141	2.072	.352	3.200	34.55	29.32	37.61	
325B	.200	3.166	.498	.946	.127	2.058	.348	3.042	35.00	30.12	36.50	34.85
325C	.305	(.792)		1.018	.190	(.516)		2.939			37.70	36.11
325D	.287	(.792)		1.018	.188	(.506)					34.49	36.62
325E	.286	(.792)		.995	.196	(.502)					31.47	

Table 6 (Cont'd)

Specimen Number	Initial				Final				Shrinkage			
	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Length %	Width %	Height %	Diameter %
LPS 330A	.196	3.166	.498	1.140	.139	2.043	.356	2.479	35.47	28.51	29.08	-
330B	.199	3.166	.498	.943	-	-	-	-	-	-	-	37.33
330C	.300	(.792)		1.034	.188	(.523)		3.166	-	-	-	33.96
330D	.286	(.792)		1.019	.184	(.498)		3.025	-	-	-	35.66
330E	.282	(.792)		.998	.176	(.484)		2.793	-	-	-	37.59
LPS 333A	.231	3.166	.498	1.093	.166	2.093	.354	2.843	33.96	28.92	28.14	-
333B	.244	3.166	.498	.899	-	-	-	-	-	-	-	34.44
333C	.302	(.792)		1.028	.198	(.512)		3.238	-	-	-	35.35
333D	.300	(.792)		1.006	.194	(.508)		3.128	-	-	-	35.86
333E	.292	(.792)		1.002	.182	(.494)		2.723	-	-	-	37.63

Table 7

Sintering Response of  $\text{Si}_3\text{N}_4$ -15Y<sub>2</sub>O<sub>3</sub>-3Al<sub>2</sub>O<sub>3</sub> Materials  
(Sintered at 1730°C for 1 hr)

Specimen Number	Initial			Final			Shrinkage					
	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Length %	Width %	Height %	Diameter %
LPS 308					.294	2.044	.322	3.075				
LPS 309					.227	2.240	.320	2.671				
LPS 310A					.143	2.085	.349	2.946				
310B					.146	2.085	.324	3.274				
LPS 311A					.118	2.114	.340	3.120				
311B					.119	1.994	.334	2.773				
LPS 312A					.128	2.054	.341	3.134				
312B					.131	2.054	.337	3.036				
LPS 313A					.107	2.020	.328	2.789				
313B					.108	2.034	.328	2.799				
LPS 314A	.214	3.166	.498		.132	2.079	.334	3.292	34.33	32.93	38.32	
314B	.206	3.166	.498		.125	2.018	.330	3.133	36.26	33.73	39.32	
LPS 315A	.216	3.166	.498	1.018	.118	1.966	.320	3.116	37.90	35.74	45.37	
315B	.183	3.166	.498	1.021	.110	2.006	.323	2.972	36.64	35.14	39.89	
315C	.285	(.792)		1.056	.174	(.488)		3.111			38.94	38.38
315D	.268	(.792)		1.066	.163	(.488)		3.119			39.18	38.38

Table 7 (Cont'd)

Specimen Number	Initial				Final				Shrinkage						
	Length		Width		Length		Width		Length		Width		Diameter		
	Height cm	(Dia) cm	Height cm	(Dia) cm	Density g/cm <sup>3</sup>	Height cm	(Dia) cm	Density g/cm <sup>3</sup>	Height cm	(Dia) cm	Density g/cm <sup>3</sup>	Length %	Width %	Height %	Diameter %
LPS 316A	.200	3.166	.498	1.095	1.055	.119	2.112	3.030	.338	2.099	3.108	33.70	32.13	35.50	34.09
316B	.200	3.166	.498	.946	1.178	.132	2.076	2.870	.340	2.014	2.368	36.39	31.73	30.00	
316C	.282	(.792)		1.088	1.068	.179	(.504)	3.351		(.522)	3.326			32.62	34.09
316D	.264	(.792)		1.082	1.053	.182	(.484)	3.297		(.498)	3.235			34.47	37.12
LPS 317A	.212	3.166	.498	1.055	1.055	.119	2.112	3.030	.348	2.112	3.030	33.29	30.12	43.87	
317B	.168	3.166	.498	1.178	1.178	.132	2.076	2.870	.336	2.076	2.870	34.43	32.53	21.43	
317C	.286	(.792)		1.068	1.068	.179	(.504)	3.351		(.504)	3.351			37.41	36.36
317D	.278	(.792)		1.053	1.053	.182	(.484)	3.297		(.484)	3.297			34.53	38.89
LPS 318A	.200	3.166	.498	1.083	1.083	.132	2.090	2.739	.348	2.090	2.739	34.00	30.12	34.00	
318B	.197	3.166	.498	.998	.998	.163	2.086	2.779	.347	2.086	2.779	17.26	30.33	17.26	
318C	.282	(.792)		1.080	1.080	.190	(.514)	3.044		(.514)	3.044			32.62	35.10
318D	.263	(.792)		1.082	1.082	.174	(.494)	3.000		(.494)	3.000			33.84	37.63
LPS 321A	.269	(.792)		1.038	1.038	.172	(.500)	3.109		(.500)	3.109			36.06	36.87
321B	.270	(.792)		1.044	1.044	.170	(.500)	3.218		(.500)	3.218			37.04	36.87
LPS 328A	.232	3.166	.498	.949	.949	.146	2.104	2.735	.344	2.104	2.735	33.54	30.92	37.07	
328B	.188	3.166	.498	1.138	1.138	.140	2.102	2.580	.353	2.102	2.580	33.61	29.12	25.53	
328C	.306	(.792)		1.008	1.008	.210	(.510)	2.914		(.510)	2.914			31.37	35.60
328D	.278	(.792)		.988	.988	.192	(.522)	2.927		(.522)	2.927			30.94	34.09
328E	.279	(.792)		.982	.982	.185	(.522)	3.025		(.522)	3.025			33.69	34.09
LPS 329A	.173	3.166	.498	.891	.891	.107	2.078	2.472	.359	2.078	2.472	34.37	27.91	38.15	
329B	.160	3.166	.498	.983	.983	.103	2.050	2.704	.346	2.050	2.704	35.25	30.52	35.63	
329C	.288	(.792)		1.018	1.018	.192	(.525)	3.104		(.525)	3.104			33.33	33.71
329D	.369	(.792)		1.033	1.033	.239	(.499)	2.907		(.499)	2.907			35.23	36.99

Table 8

Final Pellet Densities for SN402  $\text{Si}_3\text{N}_4$ -15 $\text{Y}_2\text{O}_3$  with Various  $\text{Al}_2\text{O}_3$   
Additions Sintered at 1730°C for 1 hr

<u>Specimen Run Number</u>	<u>Average Density g/cm<sup>3</sup></u>	<u>% Al<sub>2</sub>O<sub>3</sub> Addition</u>
LPS 331	3.006	1
LPS 332	3.008	1
Group Avg.	3.007	
LPS 322	3.128	2
LPS 323	2.865	2
LPS 324	3.023	2
LPS 325	3.060	2
LPS 326	3.071	2
LPS 327	3.026	2
LPS 330	2.994	2
LPS 333	3.030	2
Group Avg.	3.025	
LPS 315	3.152	3
LPS 317	3.324	3
LPS 318	3.022	3
LPS 321	3.165	3
LPS 328	2.955	3
LPS 329	3.005	3
Group Avg.	3.104	

## Measurement of Properties

The initial measurements of modulus rupture were made on four rectangular bars of  $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3\text{-Al}_2\text{O}_3$  materials. The specimens were cold pressed from one direction in preparation and were slightly distorted after heating. The specimens were not machined, but measured in the as-sintered condition. The results of three point modulus of rupture tests are listed in Table 9. The values of 280 to 370 MPa (40 to 54 ksi) for these samples gave encouragement that good values could be obtained for better prepared samples and samples prepared in the high  $\text{N}_2$  pressure sintering furnace. The results of tests on samples cold compacted under isostatic pressure before sintering are described later.

Rather than wait for samples produced using improved techniques, one of the above described rectangular bars was creep tested at  $1300^\circ\text{C}$  in argon. A sample containing 3%  $\text{Al}_2\text{O}_3$  was chosen because it had the highest density, but as can be seen from data in Table 10 for this creep test and shown in Fig. 4, the creep rate of the sample was high,  $\sim 1 \times 10^{-2}$  in/in hr, compared with hot pressed  $\text{Si}_3\text{N}_4$  (NC 132) which has a creep rate of  $3.2 \times 10^{-4}$  in/in hr at  $1350^\circ\text{C}$  under a similar load of 10 ksi (70 MPa). This would be expected because of the high percentage of  $\text{Al}_2\text{O}_3$  in the sample.

## Oxidation Studies

The oxidation behavior of selected  $\text{Si}_3\text{N}_4$  sintered materials made with the various amounts of  $\text{Y}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$  were examined by measuring the weight gain as a function of time during exposure in air at  $1300^\circ\text{C}$  for 100 hrs. It had been shown in a previous program (Ref. 1) that GTE SN402  $\text{Si}_3\text{N}_4$  bodies prepared from a mixture of  $\text{Si}_3\text{N}_4\text{-}15\text{Y}_2\text{O}_3\text{-}.5\text{Al}_2\text{O}_3$  which had been sintered at  $1800^\circ\text{C}$  under 20 atmospheres of  $\text{N}_2$  was more oxidation resistant than hot-pressed NC-132. The determination of the effect of reducing the  $\text{Y}_2\text{O}_3$  in  $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3\text{-Al}_2\text{O}_3$  materials was of interest in this program.

In Table 11 is listed the final weight gain in  $\text{mg/cm}^2$  after 100 hrs at  $1300^\circ\text{C}$  in air for those specimens studied. The components, the density, and sintering method are also listed in this table. The first four specimens were sintered at  $1800^\circ\text{C}$  for 1 hr under 20 atmospheres of  $\text{N}_2$ . In sample HPS 9B the amount of  $\text{Y}_2\text{O}_3$  was decreased (to 10%) but the  $\text{Al}_2\text{O}_3$  addition had to be increased (to 6%) to obtain the same density that was previously achieved with levels of 1%  $\text{Al}_2\text{O}_3$  additions to the  $\text{Si}_3\text{N}_4\text{-}15\text{Y}_2\text{O}_3$  mixtures. This increase in  $\text{Al}_2\text{O}_3$  content appears to have a negative effect on the oxidation resistance of this material compared to those prepared with the more  $\text{Y}_2\text{O}_3$  but less  $\text{Al}_2\text{O}_3$  (specimens HPS 12, 14A, and 29).

This same effect is apparent for the material prepared at  $1730^\circ\text{C}$ . For  $\text{Si}_3\text{N}_4\text{-}10\text{Y}_2\text{O}_3\text{-}6\text{Al}_2\text{O}_3$  (LPS 250) and  $\text{Si}_3\text{N}_4\text{-}10\text{Y}_2\text{O}_3\text{-}4\text{Al}_2\text{O}_3$  (LPS 248) when compared with LPS 323 ( $\text{Si}_3\text{N}_4\text{-}15\text{Y}_2\text{O}_3\text{-}2\text{Al}_2\text{O}_3$ ) exhibit this behavior. Comparing LPS 250 (6%  $\text{Al}_2\text{O}_3$ ) with LPS 248 (4%  $\text{Al}_2\text{O}_3$ ) also indicates that for materials containing the same amount of  $\text{Y}_2\text{O}_3$  a decrease in aluminum oxide content increased the oxidation resistance.

Table 9

Three Point Modulus of Rupture Test Results for  
 $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$  Materials Sintered at  $1730^\circ\text{C}$  for 1 hr  
(unmachined samples)

<u>Specimen Number</u>	<u>Density</u> g/cm <sup>3</sup>	<u>% Al<sub>2</sub>O<sub>3</sub></u>	<u>Modulus of Rupture</u>	
			<u>ksi</u>	<u>MPa</u>
LPS 311A	3.120	3	49.8	344
LPS 312A	3.134	3	53.7	370
LPS 316A	3.108	3	40.6	280
LPS 322D	3.205	2	53.7	370

**STRAIN vs TIME FOR  $\text{Si}_3\text{N}_4 - 15 \text{Y}_2\text{O}_3 - 3\text{Al}_2\text{O}_3$**

CREEP TESTED AT 1300°C IN ARGON  
STRESS LEVEL 10,000 psi

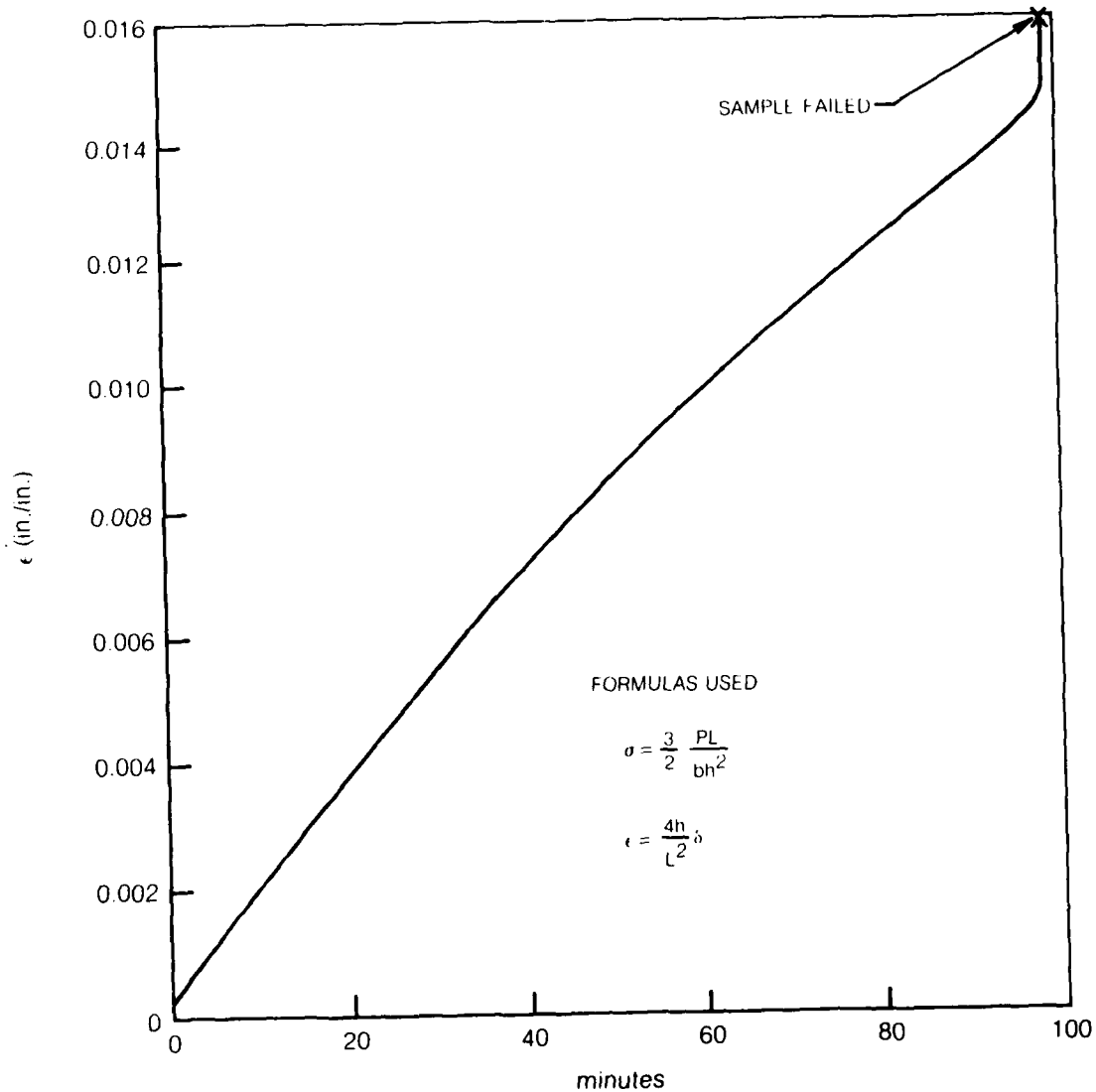


Table 10

Creep in Bending at 1300°C in Argon  
Si<sub>3</sub>N<sub>4</sub>-15Y<sub>2</sub>O<sub>3</sub>-3Al<sub>2</sub>O<sub>3</sub> Materials

Time Min	10,000 psi	
	$\delta_{in}$	$\epsilon_{in/in}$
0	.00049	.00028
5		
10	.00340	.00190
15		
20	.00640	.00370
25		
30	.00970	.00560
35		
40	.01250	.00720
45		
50	.01500	.00860
55		
60	.01725	.00989
65		
70	.01940	.01110

Table 11

Oxidation Weight Gains After 100 hrs  
Exposure in Air at 1300°C

Specimen Number	$\text{Si}_3\text{N}_4:\text{Y}_2\text{O}_3$	$\text{Al}_2\text{O}_3$ Additions	Temp. of Sintering °C	Atmos of $\text{N}_2$	Density $\text{g/cm}^3$	Wt Gain $\text{mg/cm}^2$
HPS 9B	90:10	6	1800	20	3.120	2.60
12	85:15	1	1800	20	3.119	0.32
14A	85:15	.5	1800	20	3.029	0.81
29	85:15	.5	1800	20	2.100	0.64
LPS 250	90:10	6	1730	1	3.033	4.80
248	90:10	4	1730	1	3.116	2.70
LPS 235	92:8	6	1730	1	2.938	0.57
246	92:8	4.5	1730	1	3.023	0.64
LPS 308	85:15	3	1730	1	3.075	3.87
315	85:15	3	1730	1	3.326	4.24
LPS 323	85:15	2	1730	1	2.721	0.80
324	85:15	2	1730	1	3.158	0.14
330C	85:15	2	1730	1	3.166	1.07
LPS 331C	85:15	1	1730	1	3.183	0.74
Norton HP	-	-	-	-	-	0.47
NC-132	-	-	-	-	-	0.53

Examining LPS 308 and LPS 315 ( $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3\text{-3Al}_2\text{O}_3$ ) with the group LPS 323, 324, 330C ( $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3\text{-2Al}_2\text{O}_3$ ) and LPS 331C ( $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3\text{-1Al}_2\text{O}_3$ ) indicates again that within one series of materials where the  $\text{Y}_2\text{O}_3$  content is kept the same, the less aluminum oxide added the better the oxidation resistance of the material. The oxidation resistances of LPS 323, LPS 324 and LPS 331C compare very well with the oxidation resistances of two specimens of hot-pressed NC-132 also shown in this table. The oxidation resistances of the last mentioned specimens also compare favorably with those observed for  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3\text{-.5Al}_2\text{O}_3$  (HPS 14A, 29) which were (listed at the beginning of this table) sintered at  $1800^\circ\text{C}$  under 20 atmospheres of nitrogen.

The behavior of LPS 235 ( $\text{Si}_3\text{N}_4\text{-8Y}_2\text{O}_3\text{-6Al}_2\text{O}_3$ ) and specimen LPS 246 ( $\text{Si}_3\text{N}_4\text{-8Y}_2\text{O}_3\text{-4.5Al}_2\text{O}_3$ ) also show superior oxidation resistances although the amount of  $\text{Y}_2\text{O}_3$  has been decreased (below 15%) and the amount of  $\text{Al}_2\text{O}_3$  increased. This indicates that there are compositions in the  $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3\text{-Al}_2\text{O}_3$  system other than  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3\text{-.5Al}_2\text{O}_3$  which also could have good oxidation resistances. The ease of sintering of the  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$  materials would have to be weighed against the potentially improved oxidation resistance of lower  $\text{Y}_2\text{O}_3$  content materials.

#### Heat Treatment Studies

A heat treatment was employed in an attempt to improve the oxidation resistance and mechanical strength of specimens that had been previously sintered at  $1730^\circ\text{C}$  for 1 hr under flowing nitrogen. Gazza and co-workers have shown that heat treatments improved the properties of hot-pressed  $\text{Si}_3\text{N}_4$  (Ref. 13). The continuous heat treatment chosen consisted of a 20 hr soak at  $1150^\circ\text{C}$  under argon, followed by an 18 hr soak at  $1250^\circ\text{C}$  under a flowing atmosphere of 96%  $\text{N}_2\text{-4\% H}_2$  and then a 36 hr exposure at  $1375^\circ\text{C}$  in this same flowing atmosphere.

The composition chosen for this heat treatment was  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3\text{-3Al}_2\text{O}_3$  because it was easy to sinter to high densities. Seven specimens were treated at one time. The weight and linear measurements of these seven specimens before and after heat treatment are given in Table 12. All seven lost some weight during this heat treatment. Four of them increased in height and width and three increased in diameter. The loss in weight combined with the larger calculated volume resulted in all seven specimens decreasing in density after the heat treatment.

Two of these specimens LPS 316C and LPS 317A were exposed to air at  $1300^\circ\text{C}$ . The results of these tests are listed in Table 13. The effect of density on oxidation resistance can be seen by comparing the weight gain of LPS 317C (density of  $2.607\text{ g/cm}^3$ ) of  $2.06\text{ mg/cm}^2$  with that of LPS 316A (density  $2.941\text{ g/cm}^3$ ) which recorded a weight gain of only  $0.50\text{ mg/cm}^2$  after the 100 hr exposure at  $1300^\circ\text{C}$ .

Table 12

Effect of Heat Treatment on Weight and Dimensions  
of  $\text{Si}_3\text{N}_4$ -15Y<sub>2</sub>O<sub>3</sub>-3Al<sub>2</sub>O<sub>3</sub> Materials

(1150°C Argon 20 hr

1250°C N<sub>2</sub>/4% H<sub>2</sub> 18 hr

1375°C N<sub>2</sub>/4% H<sub>2</sub> 36 hr)

Specimen Number	Before				After					
	Weight gm	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>	Weight gm	Height cm	Length (Dia) cm	Width cm	Density g/cm <sup>3</sup>
LPS 313A	.1977	.107	2.020	.328	2.789	.1938	.129	2.048	.348	2.108
LPS 315B	.1926	.110	2.006	.323	2.972	.1876	.099	2.042	.336	2.761
LPS 315D	.0951	.163	(.488)		3.119	.9011	.161	(.50)		2.880
LPS 316C	.1353	.190	(.522)		3.326	.1321	.199	(.530)		2.941
LPS 317A	.2663	.119	2.112	.348	3.030	.2588	.129	2.132	.361	2.607
LPS 318A	.2630	.132	2.090	.348	2.739	.2598	.146	2.090	.360	2.365
LPS 318C	.1200	.190	(.514)		3.044	.1196	.194	(.526)		2.836

Table 13

Oxidation Weight Gain After 100 hrs Exposure in Air at 1300°C for  
Heat Treated Si<sub>3</sub>N<sub>4</sub>-15Y<sub>2</sub>O<sub>3</sub>-3Al<sub>2</sub>O<sub>3</sub> Materials

Specimen Number	Si <sub>3</sub> N <sub>4</sub> :Y <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub> Addition	Sintering		Atmos N <sub>2</sub>	Density Before Heat Treatment g/cm <sup>3</sup>	Density After Heat Treatment g/cm <sup>3</sup>	Wt Gain mg/cm <sup>2</sup>
			Temp °C					
LPS 316C	85:15	3	1730		1	3.326	2.941	.50
LPS 317C	85:15	3	1730		1	3.030	2.607	2.06
LPS 315 (as sintered)	85:15	3	1730		1	-	3.326	4.24

This same specimen (LPS 316C) can be compared with LPS 315 which gained about 4.0 mg/cm<sup>2</sup>. Both samples have the same composition and were prepared in the same way except that LPS 316C was heat treated as described above. Even though the density of LPS 316C decreased some during the heat treatment it was not as great as for other samples and the result was that the weight gain after 100 hrs exposure to air at 1300°C was only .50 mg/cm<sup>2</sup>. This indicates that the heat treatment can be beneficial if the density of the sample is not reduced to too low a value. This might be accomplished better by using a less severe heat treatment process.

The rectangular bars which had been heat treated (LPS 315B and LPS 318A) were tested for modulus of rupture and are listed in Table 14. Note that the densities of these samples had been decreased significantly by the heat treatment. As would be expected, the strength of materials also decreased. For that reason, the samples were not creep tested.

#### Isostatic Cold Compaction Studies

Near the end of this program an isostatic cold press capable of 210 MPa (30 ksi) became available. Rectangular bar specimens of Si<sub>3</sub>N<sub>4</sub>-15Y<sub>2</sub>O<sub>3</sub> materials with 1, 2 and 3% Al<sub>2</sub>O<sub>3</sub> additions were prepared. These were cold pressed in the normal manner. The cold pressed bars were then placed into individual latex bags which were evacuated and tied off. A group of these samples were then isostatically pressed to 185 MPa (27 ksi) at one time. After removal from the press these samples were sintered in the normal procedure at 1730°C for 1 hr under flowing nitrogen.

The results from the modulus of rupture testing of these bars which were not machined are listed in Table 15. The value of 514 MPa (74.5 ksi) obtained from specimen LPS 364A indicates that this method of preparing samples could lead to the formation of high strength Si<sub>3</sub>N<sub>4</sub> materials especially if the specimens are prepared under N<sub>2</sub> pressure at 1800°C.

#### Sintering of Blade Shapes

To be able to form complex shapes of high density, oxidation resistant silicon nitride bodies without the tedious and expensive machining procedures required with hot-pressed material was one of the goals of this program. Pressureless sintering of the proper chosen composition could produce a usable product directly from the sintering operation if the proper mold was available and shrinkage factors were known.

Table 14

Three Point Modulus of Rupture Test Results for  
 Heat Treated  $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3 - 3\text{Al}_2\text{O}_3$   
 (unmachined samples)

<u>Specimen Number</u>	<u>Density</u>	<u>Density</u>	<u>Modulus of Rupture</u>	
	<u>g/cm<sup>3</sup></u> Before Heat Treatment	<u>g/cm<sup>3</sup></u> After Heat Treatment	<u>ksi</u>	<u>MPa</u>
LPS 315B	2.972	2.761	20.4	141
LPS 318A	2.739	2.365	30.9	213

Table 15

Three Point Modulus of Rupture Test Results for  
Cold Isostatic Pressed and Sintered  
 $\text{Si}_3\text{N}_4$ -15Y<sub>2</sub>O<sub>3</sub> Materials  
(unmachined samples)

<u>Specimen Number</u>	<u>Density</u> g/cm <sup>3</sup>	<u>% Al<sub>2</sub>O<sub>3</sub></u>	<u>Modulus of Rupture</u>	
			<u>ksi</u>	<u>MPa</u>
LPS 364A	3.241	1	74.5	514
LPS 364B	3.283	2	53.0	366
LPS 365B	3.088	2	59.8	413
LPS 364C	3.141	3	62.7	432
LPS 365C	2.995	3	38.1	263

To investigate this process a steel and die plunger assembly was fabricated to cold press silicon nitride powder mixtures into an airfoil shape. The airfoil shape was arrived at by hand machining a long steel plunger to the desired contour. This piece was then used as the tool for an EDM machining process and passed through a steel cube. After machining the steel plunger was cut in two and became the top and bottom plunger for the die cavity. For compacting and cold pressing powders, the 0.0012 cm (0.003 in.) clearance from the EDM machining operation between the cavity and plunger assembly was not a problem.

These cold pressed shapes were then sintered at 1730°C in a graphite tube furnace for 1 hr under an atmosphere of flowing nitrogen. During each blade sintering, a small pellet of the same powder composition was also sintered and its density used as an indication of the final airfoil density.

The silicon nitride powder mixture chosen for this study was made up of 80% AME  $\text{Si}_3\text{N}_4$ , 15%  $\text{Y}_2\text{O}_3$ , and 5%  $\text{Al}_2\text{O}_3$ . Fifty gram lots of mixture were milled for 48 hrs with aluminum oxide mixing balls. This mixture was chosen for the initial experiments because it sintered well, but it did not produce samples with the best strength and oxidation resistance. In Table 16 are listed the experiments made with this mixture and the airfoil shaped die. The initial height of the cold pressed blade and final height after sintering are listed along with similar data for the accompanying pellet. The blade shapes decreased in height an average of 26.7% whereas the pellets showed a decrease in height after sintering of 23.8%. The average diameter shrinkage of the pellets was also 23.8%. These shrinkage factors (23%) are considerably less than those obtained with the GTE SN402 powder with comparable  $\text{Y}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$  content that were listed in Table 7. In those cases recorded shrinkage percentages were in the 35% range for these dimensions. A photograph of typical blades produced is shown in Fig. 5.

Thin slabs were machined from these airfoil shapes and measurements of the modulus of rupture on these pieces yielded values between 345 MPa to 365 MPa (50 ksi to 53 ksi). This is what would be expected for the composition used.

Attempts to sinter a hollow airfoil with this blade shaped die were not successful. The positioning of a dummy core during the cold pressing of the powder could not be controlled. Hollow cold compacts with nonuniform wall thickness cracked upon being exposed to sintering conditions at 1730°C.

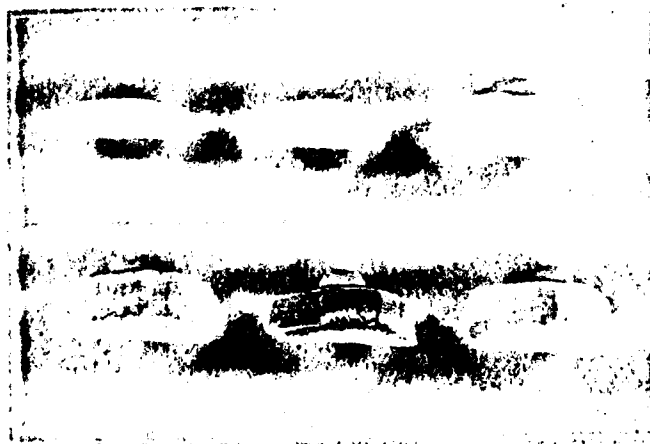
To investigate the general problems associated with sintering hollow shapes, a simple right circular three piece cylindrical steel die was fabricated. This die yielded a cold pressed powder ring with an OD of 2.22 cm (0.875 in.), ID of 1.587 cm (0.625 in.) and heights up to 1.70 cm (0.670 in.).

Table 16

## Sintering Response of Blade Shapes

Specimen Number	Blade		Pellet				Pellet		
	Before Height cm	After Height cm	Before Dia cm	Before Height cm	After Dia cm	After Height cm	Density g/cm <sup>3</sup>	Height %	Diameter %
-15	1.693	1.258	.792	.218	.612	.164	2.937	24.77	22.73
-6	2.010	1.515	.792	.212	.615	.168	2.938	20.75	22.35
-19	2.110	1.524	.792	.217	.602	.168	3.160	22.58	23.99
-20	1.619	1.168	.792	.212	.606	.160	3.243	24.53	23.48
-21	1.774	1.258	.792	.217	.594	.163	3.157	24.88	25.00
-22	.107 .099 .100	.075 .073 .077							
-23	.104 .102	.079 .074							
-24	1.547	1.110	.792	.220	.597	.164	3.204	25.45	24.62
-25	1.346	.992	.792	.204	.602	.156	3.236	23.53	23.99

**BLADE SHAPES MADE BY PRESSURELESS  
SINTERING OF  $\text{Si}_3\text{N}_4 - 15\text{Y}_2\text{O}_3$**



Since a new large container was to be used to contain these rings during sintering there was some concern about temperature uniformity in the larger volume container. To investigate this potential problem, four pellets were prepared from the silicon nitride powder mixture previously described and placed at various locations within this container. For identification after sintering each pellet was cold pressed to a different initial height. In Table 17 are listed the dimensions of these pellets before and after sintering along with their final densities. Shrinkage percentages for the height and diameter are also provided in this table. Both final density and shrinkage percentages for these four pellets were fairly uniform. This would indicate that the new larger container would pose no problem from uneven thermal distribution.

The cold pressed ring compacts produced from this three piece steel die could easily be removed and appeared to be free from cracks prior to sintering. In Table 18 are listed the initial and final dimensions of the rings made with these dies along with the comparable shrinkage percentages for these specimens. No contraction cracks were observed although shrinkage percentages of up to 28% were taking place in thin wall cylinders. For example, in specimen 47, initial wall thickness was 0.3065 cm (0.121 in.) with a final wall thickness of 0.255 cm (0.100 in.) after sintering. With the exception of specimens 44, 50 and 51, the remaining seven experimental runs yielded fairly uniform densities.

Table 17

Verification of Uniform Thermal Distribution  
Within Large Container

<u>Pellet</u>	<u>Before Sintering</u>			<u>After Sintering</u>			<u>Shrinkage</u>	
	<u>Dia.</u> cm	<u>Height</u> cm	<u>Density</u> g/cm <sup>3</sup>	<u>Dia.</u> cm	<u>Height</u> cm	<u>Density</u> g/cm <sup>3</sup>	<u>Height</u> %	<u>Dia.</u> %
A	1.348	.473	1.319	.993	.359	3.182	24.10	26.34
B	1.348	.527	1.317	.991	.410	3.123	22.20	26.48
C	1.348	.581	1.292	.980	.437	3.219	24.78	27.30
D	1.348	.609	1.332	.992	.475	3.130	22.0	26.41

Table 18

## Sintering Response of Ring Shapes

Specimen Number	Before Sintering		After Sintering		Final Density g/cm <sup>3</sup>	Height %	Outer Diameter %	Inner Diameter %
	OD cm	ID cm	OD cm	ID cm				
-36	2.220	1.587	1.607	1.156	2.969	25.49	27.61	27.16
-38	2.220	1.587	1.602	1.135	3.061	26.44	27.84	28.48
-39	2.220	1.587	1.628	1.164	2.978	-	26.67	26.65
-44	2.220	1.587	1.671	1.180	2.601	21.69	24.73	25.65
-46	2.220	1.587	1.700	1.209	3.070	22.36	23.42	23.82
-47	2.220	1.587	1.723	1.214	2.927	21.28	22.39	23.50
-48	2.220	1.587	1.716	1.214	2.786	21.29	22.70	23.50
-49	2.220	1.587	1.718	1.212	2.810	15.50	22.61	23.63
-50	2.220	1.587	1.787	1.260	2.429	11.87	19.50	20.60
-51	2.220	1.587	1.806	1.280	2.392	-	18.65	19.34

## CONCLUSIONS

1. Studies on the  $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3\text{-Al}_2\text{O}_3$  system have shown that reducing the  $\text{Y}_2\text{O}_3$  content of these materials from the  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$  composition made the samples more difficult to sinter. Some improvement in sinterability could be obtained by increasing the  $\text{Al}_2\text{O}_3$  content.
2. Oxidation studies on  $\text{Si}_3\text{N}_4\text{-6Y}_2\text{O}_3$ ,  $\text{Si}_3\text{N}_4\text{-8Y}_2\text{O}_3$ ,  $\text{Si}_3\text{N}_4\text{-10Y}_2\text{O}_3$  and  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$  materials confirmed previous conclusions that the oxidation resistance of these materials improves with increasing density and decreasing amounts of  $\text{Al}_2\text{O}_3$  in the samples. Specimens with oxidation resistance comparable to hot pressed material can be prepared. Since these samples did not have theoretical densities, there appears to be room for further improvements in oxidation resistance.
3. The use of a  $\text{H}_2/\text{N}_2$  high temperature treatment appears to improve the oxidation resistance of these materials if their densities do not decrease to very low values during the heating process. Lowering the density results in a decrease in the strength of the samples. A less severe heat treatment may result in a high strength oxidation resistant material.
4. The density of the samples prepared by pressureless sintering are still below theoretical density. Better processing conditions are needed to optimize the strength of these materials. The use of temperatures  $>1200^\circ\text{C}$  and high  $\text{N}_2$  overpressures could help to achieve these higher strengths.
5. Strengths of 514 MPa (75 ksi) have been obtained for a  $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3\text{-1Al}_2\text{O}_3$  material by using isostatic compaction before sintering. This technique should be used in combination with high  $\text{N}_2$  overpressure sintering so that lower  $\text{Al}_2\text{O}_3$  content materials can be prepared and higher densities can be achieved. This should result in materials with higher strengths as well as better oxidation resistances.

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F. S. Galasso and R. D. Veltri  
Materials Sciences Group  
United Technologies Research Center  
East Hartford, CT 06108  
Technical Report AMMRC-TR 81-28, June 1981  
pp.-illus.-tables, Contract DAAG46-79-C-0101  
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Final Report, September 1979-September 1980

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Key Words

Ceramic materials  
Silicon nitrides  
Sintering  
oxidation  
Yttrium silicon nitrides  
Powders (particles)

A previous study on contract DAAG46-78-C-0017 showed that a sintered  $Si_3N_4 \cdot 15Y_2O_3$  material similar in oxidation resistance to hot-pressed  $Si_3N_4$  could be prepared by minimizing the  $Al_2O_3$  content in the sample while maintaining high densities. This was achieved by using a high surface area SN402  $Si_3N_4$  powder and a high nitrogen overpressure during high temperature (1800°C) sintering. In this study attempts were made to reduce the amount of  $Y_2O_3$  as well as the  $Al_2O_3$  in the mixtures to improve the oxidation resistance of the samples still further, but it was found that it was more difficult to obtain high densities for these materials than it was for  $Si_3N_4 \cdot 15Y_2O_3$  materials. For that reason, preliminary measurements were made on samples containing 15%  $Y_2O_3$ . These measurements showed that by using cold isostatic pressing after cold compaction and before sintering, a  $Si_3N_4 \cdot 15Y_2O_3 \cdot 1Al_2O_3$  material could be prepared with a strength as high as 514 MPa (75 ksi) for unmachined samples.

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