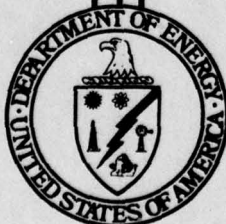


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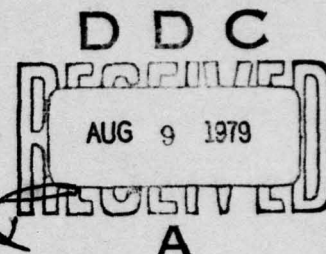


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**SINTERING OF Si_3N_4 UNDER
NITROGEN PRESSURE**

LEVEL *14*

June 1979



**UNITED TECHNOLOGIES RESEARCH CENTER
EAST HARTFORD, CONN. 06108**

Final Report — March 1978 — March 1979

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Watertown, Massachusetts 02172**

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Department of Energy

Division of Transportation Energy Conservation
Heat Engine Highway Vehicle Systems Program

U. S. DEPARTMENT OF ENERGY

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ABSTRACT

✓ way showed less weight gain in air than hot pressed $(\text{Si}_3\text{N}_4)(\text{MgO})$ over an 80 hr period at 1350°C . ↙

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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 Si_3N_4 Under Nitrogen Pressure

SUMMARY

A study of pressureless sintering of Si_3N_4 powders with various additives has been conducted. Of the various additives studied, CeO_2 , MgO , etc., Y_2O_3 was selected as the one which gave the best preliminary results. Of particular interest were the results using high N_2 pressures over the specimens to prevent weight loss and thus obtain high densities. On the basis of these results, more detailed high N_2 pressure studies were conducted using the $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ system.

In these studies, it was shown that some additional additive, in this case Al_2O_3 , was necessary to promote sintering of $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ materials unless hot pressing techniques were employed. For the AME Si_3N_4 powder used, about 8% Al_2O_3 additions resulted in nearly theoretical density samples for 1730°C , 1 atm N_2 firings. When 20 atm of N_2 was used over the samples (1800°C firing temperatures) these amounts of Al_2O_3 additions could be reduced to 4 to 5%. For GTE SN402 powder studies, the corresponding amounts of Al_2O_3 required were 2 to 3% for 1730°C firings and less than 1% for 1800°C , 20 atm firings. It is expected that these percentages may vary for different batches of Si_3N_4 powders, but they do 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 Al_2O_3 in the samples, the pressureless sintered SN402 $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ showing less weight gain in air than hot pressed $\text{Si}_3\text{N}_4 \cdot \text{MgO}$ over an 80 hr period at 1350°C . In addition, it was shown that the oxidation resistance improved with increasing density. As would be expected, the MOR of the samples also increased with increasing density. Thus, this study showed that it is desirable to use the least amount of Al_2O_3 which will produce high density pellets, and high N_2 pressure sintering is a way of obtaining the high densities with smaller amounts of Al_2O_3 additions.

Other things studied in this program involved methods of adding the Al_2O_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 Al_2O_3 was added from the balls in the ball milling operation. While this technique

appeared to produce a homogeneous distribution of Al_2O_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 above. The most difficult part of this work was the preparation of dies and developing techniques to form and recover complex shapes. A number of airfoil shapes have been prepared from $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ material by pressureless sintering.

INTRODUCTION

Silicon nitride is presently being evaluated for use in 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 that the cost of fabricating complex shapes from the hot pressed material is excessive. There is, therefore, 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 Si_3N_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. In this study some of the samples were heated in high N_2 pressures so that higher sintering temperatures could be used without excessive decomposition of the Si_3N_4 .

BACKGROUND

In early studies on pressureless sintering, Terwilliger and Lange heated pellets of AME $\text{Si}_3\text{N}_4 \cdot 5 \text{ w/o MgO}$ at 1570°C and obtained 85-90% of theoretical density (Ref. 1). In later studies, higher densities were achieved by Rowcliffe and Jorgensen using GTE and KBI-AME Si_3N_4 powders, Y_2O_3 additives and higher temperatures (Ref. 2). Ada, Kaneno and Yamamoto (Ref. 3), Buljan and Kleiner (Ref. 4), and Masaki and Kamigaito (Ref. 5) also conducted similar sintering studies, but with a variety of additives and they also produced high density material. Priest and Gazza (Ref. 6) using oxide additives and Greskovich, Prochazka, and Rosolowski (Ref. 7) using nonoxide additives showed that there was a definite advantage in carrying out the sintering under high N_2 pressures. Priest and Gazza not only obtained very high densities but also showed that there was very little weight loss in the Si_3N_4 pellets when high N_2 pressures were used even at temperatures as high as 1900°C .

At the same time that these high N_2 pressure studies were being conducted, a preliminary sintering investigation was also being carried out at UTRC with high N_2 pressures using GTE and AME Si_3N_4 powders and MgO , CeO_2 and Y_2O_3 additives. The objective was to determine which Si_3N_4 powders and which additives yielded the highest density products and to ascertain if there was a real advantage in employing N_2 overpressure during the sintering process.

Some of the materials studies were AME $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$, 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 N_2 pressure (higher sintering temperature) had the highest densities. The highest density was obtained for materials which contained Y_2O_3 and excellent ambient and high temperature properties had been obtained at UTRC for hot pressed samples (Ref. 8) so the $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$ system was selected for more detailed pressureless sintering studies. A list of these runs are given in Appendix A together with sintering data.

EXPERIMENTAL PROCEDURES

The powders used in this study were Si_3N_4 powders bought from AME (high purity, -325 mesh), a SN402 Si_3N_4 powder bought from GTE, and two Si_3N_4 powders supplied by AMMRC. One of the AMMRC powders was obtained by passing AME powder through an air classifier so that only 1.39% of the material had a particle size of greater than 40μ and 50% had a particle size less than 0.7μ . The other Si_3N_4 powder was obtained from Stark by AMMRC. It contained about 96% α phase and consisted of 2 to 3μ size particles.

The yttria was formed by bringing Molycorp CP grade yttrium oxalate up to 1000°C and down over a 24 hr period. The alumina used was Fisher CP grade material.

Some powder mixtures were formulated by adding 15 w/o Y_2O_3 to each of the Si_3N_4 powders, adding methanol as the fluid media and ball milling the powders. The powders were dried and then used in these investigations. Other powder compositions were prepared by mixing the powders in a mortar and pestle.

The powders were pressed into pellets .786 cm in diameter and fired for various times at 1730 or 1800°C in BN crucibles with BN covers. The 1730°C runs were conducted in a graphite resistance furnace under 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 N_2 pressure, 1800°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.

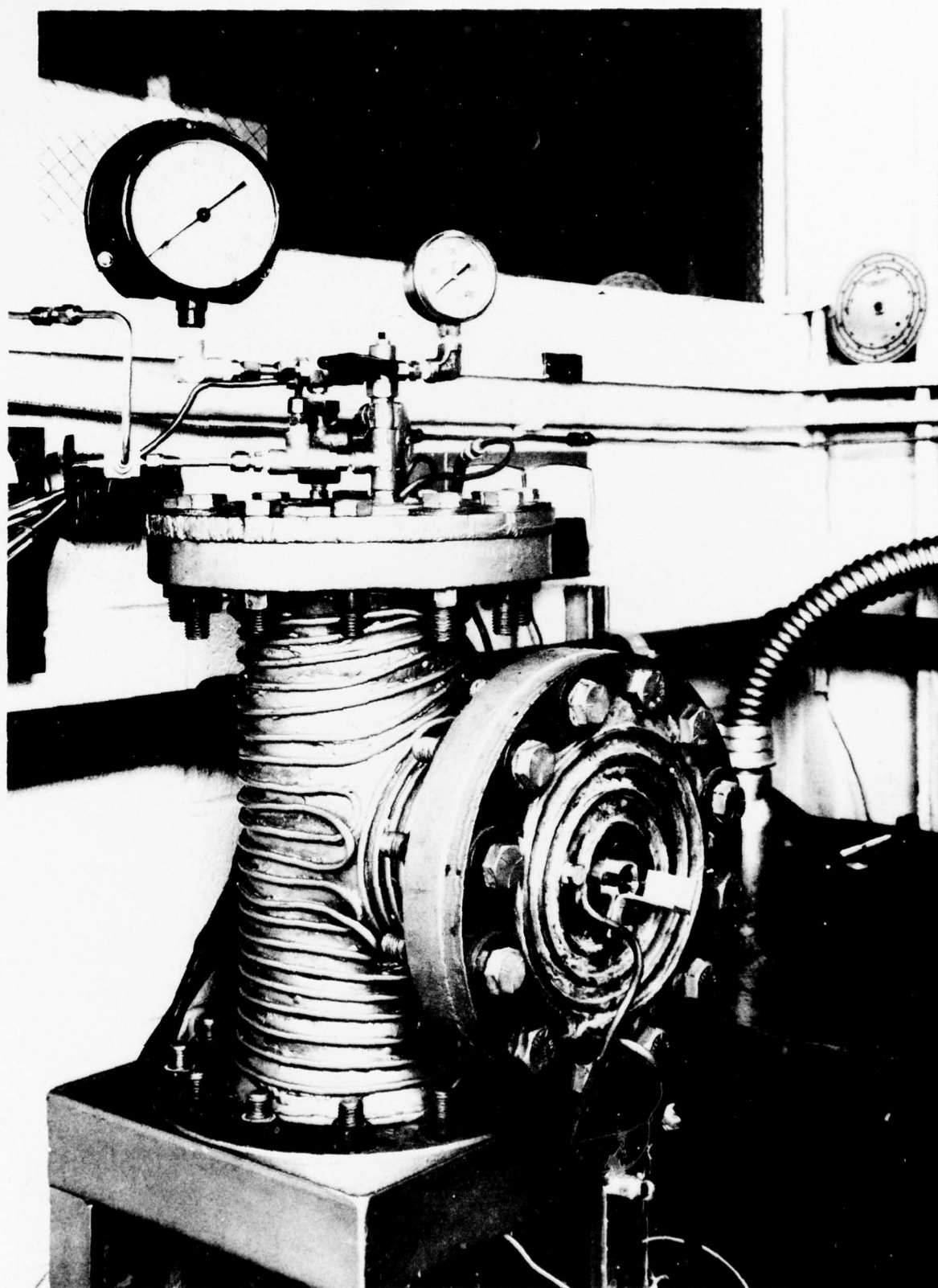
The densities reported herein were obtained by weighing the samples and using the volume calculated from measured dimensions.

The high nitrogen pressure sintering apparatus is shown in Fig. 1. This unit can be operated at temperatures of 1800°C , with internal pressures as high as 20 atm. Specimens of 5.0 cm in dia by 7.6 cm high can be accommodated within the graphite heating element.

The typical sintering procedure 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 was then introduced up to a pressure of 10 atm and held at this level until the integrity of all connections for water cooling, gas, and power

FIG. 1

HIGH N₂ PRESSURE FURNACE



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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 yields a system pressure of 20 atm at a chamber temperature of 1800°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.

EXPERIMENTAL RESULTS

Sintering Studies

The initial studies on this program were directed toward determining which parameters were important in the sintering of the compacted powders of $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ materials. Five, 10 and 15 w/o Y_2O_3 were added to Si_3N_4 . The mixtures were compacted and sintered at 1730°C . The $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ sintered the best and was selected as the material to use in further studies.

Two powder samples of $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ material (A & B) which were prepared from AME Si_3N_4 and Y_2O_3 by ball milling for 25 and 60 hrs respectively were available from previous UTRC hot pressing experimental work. Four new AME $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ samples were prepared. Samples 1 and 2 were ball milled with WC balls in a plastic container for 48 hrs and samples 3 and 4 were ball milled with alumina-silica balls and an Al_2O_3 ($85\text{Al}_2\text{O}_3 \cdot 15\text{SiO}_2$) container. In samples 1 and 3, new K106 AME Si_3N_4 powder was used while an old Si_3N_4 powder was used in samples 2 and 4. The AME $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ (Powder B) powder sintered the best; over 90% of the theoretical density was achieved at temperatures as low as 1730°C , (see Table 1). One of the samples was found to have a density as high as 3.28 g/cm^3 when the rough surface of the sample was sanded smooth. Powder A also sintered, but 1800°C high N_2 pressure sintering was needed to achieve high densities (Table 2). While powder B sinters better than powder A, Fig. 2 shows that the surface of the compact formed from it is much rougher with small voids being visible. The data from the other fired pellets are shown in Table 3. As can be seen, none of those samples sintered very well, but the samples heated at 1800°C under 20 atm of N_2 had the highest densities. X-ray diffractometer tracings showed that some conversion from α to β Si_3N_4 had taken place, but unreacted Y_2O_3 was still present.

Chemical analyses, X-ray diffraction analyses and SEM analyses of the powders A, B, 1, 2, 3 and 4 were conducted to determine what differences existed in these powders which would make them behave so differently in the sintering studies.

SEM photographs of the powders are shown in Figs. 3-8. Sample 4 consisted of large aggregates and sintered poorly, but the particles in samples 1, 2 and 3 are finer and they also sintered poorly. Sample A consists of relatively large particles and sample B contains fine particles; sample B sinters better than sample A. It is obvious from the above data that it is difficult to correlate the particle sizes with powder sinterability in these studies. It appears that there are more important factors which control the sintering behavior of these powders.

Table 1

Data for Sintered (B) $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ Pellets
(8.2 Al_2O_3)

<u>Sample</u>	<u>Firing Temp (°C)</u>	<u>N₂ Pressure</u>	<u>Time (hr)</u>	<u>Density (g/cm³)</u>
LPS-1	1730	1 atm	$\frac{1}{2}$	3.10
LPS-2	1730	1 atm	$\frac{1}{2}$	3.04
LPS-3	1800	20 atm	$\frac{1}{2}$	3.19

Table 2

Data for Sintered (A) $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ Pellets
(3.3 Al_2O_3)

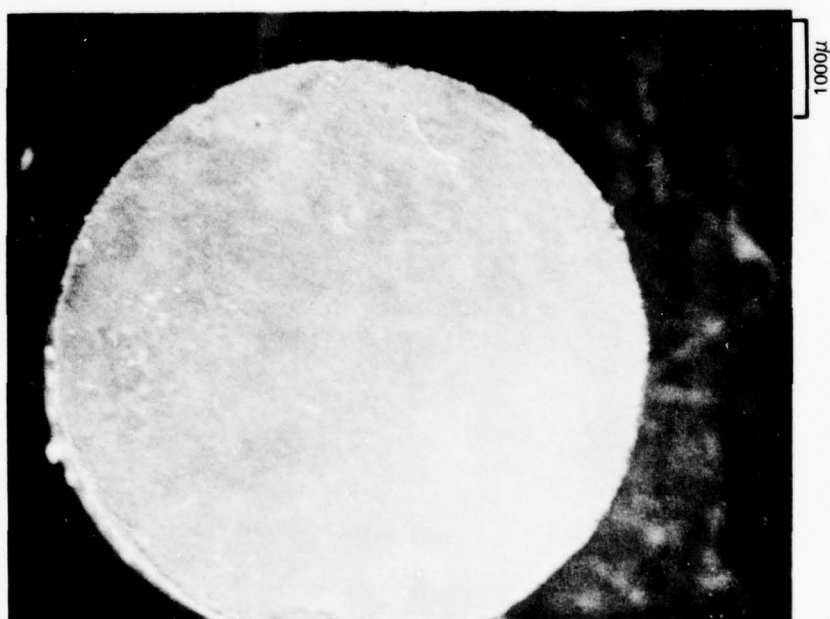
<u>Sample</u>	<u>Firing Temp (°C)</u>	<u>Firing Time (hrs)</u>	<u>Density¹ (g/cm³)</u>	<u>Density² (g/cm³)</u>	<u>Apparent Porosity (%)</u>
LP 7	1730	1	2.5	2.5	15.6
LP 9	1730	2	2.7	2.7	13.3
LP 10	1730	6	2.1	2.3	20.1
LP 11	1800	1	2.9	3.1	1.6

¹Density calculated using measured volume

²Density calculated using liquid displacement

PHOTOMICROGRAPHS OF FIRED $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3$ PELLETS

a) MADE FROM SAMPLE A POWDER



b) MADE FROM SAMPLE B POWDER



FIG. 2

Table 3

Data for Sintered $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ Pellets
Prepared Using Powders 1, 2, 3 and 4

Made from #1 powder (WC balls and K106 AME Si_3N_4 used)

<u>Sample</u>	<u>Firing Temp ($^{\circ}\text{C}$)</u>	<u>Gas Pressure</u>	<u>Time (hr)</u>	<u>Density (g/cm^3)</u>
LPS 19	1730	1 atm	1	1.22
LPS 29	1800	20 atm	1	1.75

Made from #2 powder (WC balls and old Si_3N_4 used)

LPS 20	1730	1 atm	1	1.24
LPS 30	1800	20 atm	1	1.94

Made from #3 powder (Al_2O_3 - SiO_2 balls and K106 Si_3N_4 used)

LPS 22	1730	1 atm	1	1.22
LPS 32	1800	20 atm	1	2.04

Made from #4 powder (Al_2O_3 - SiO_2 balls and old Si_3N_4 used)

LPS 23	1730	1 atm	1	1.27
LPS 33	1800	20 atm	1	2.30

FIG. 3

SEM PHOTOGRAPHS OF SAMPLE A POWDER, $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$



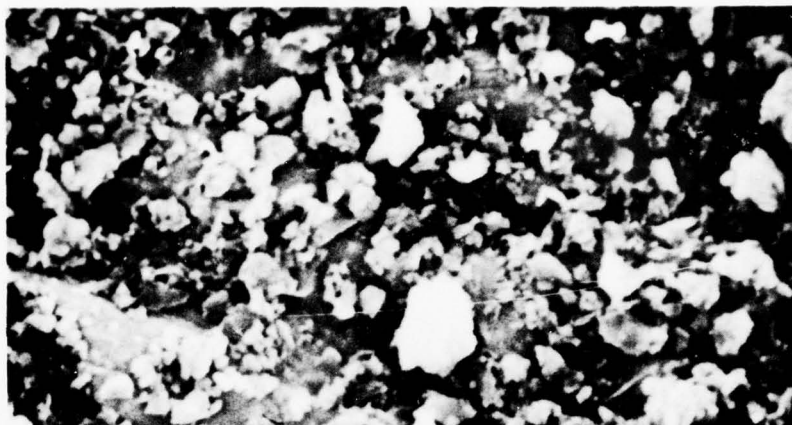
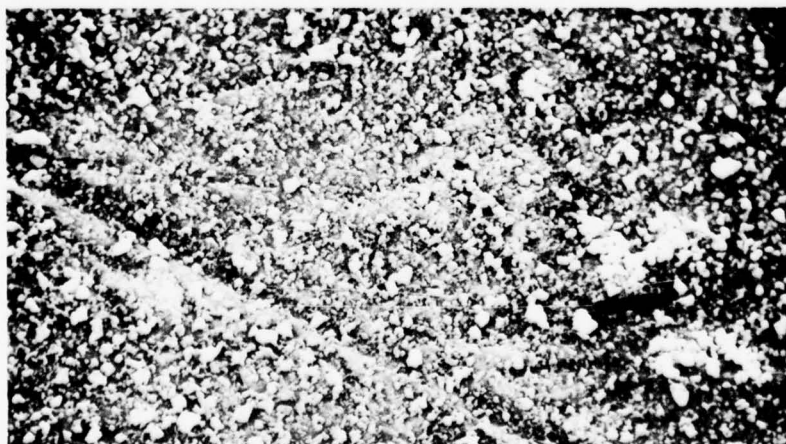
2μ



4μ



10μ

SEM PHOTOGRAPHS OF SAMPLE B POWDER, $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ 2 μ 4 μ 10 μ

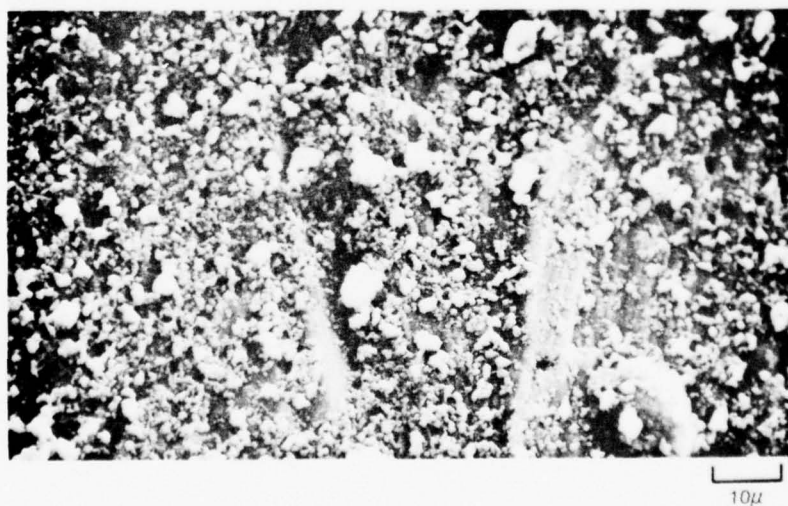
SEM PHOTOGRAPHS OF SAMPLE 1 POWDER, $K_{106}Si_3N_4 \cdot 15Y_2O_3$ 

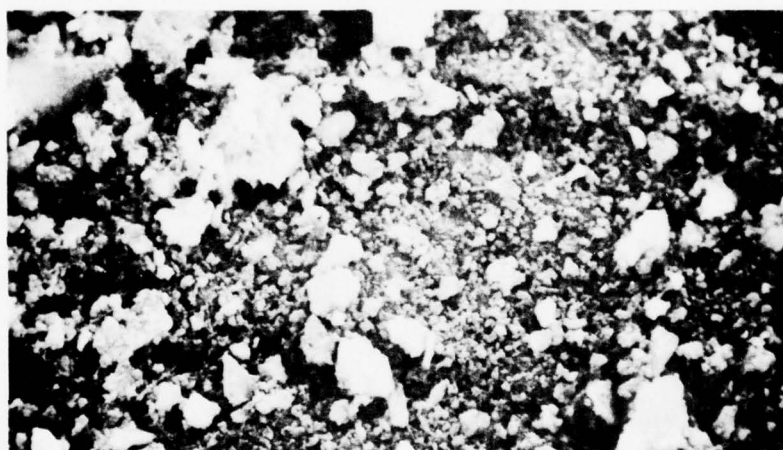
FIG. 6

SEM PHOTOGRAPHS OF SAMPLE 2 POWDER, OLD $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$

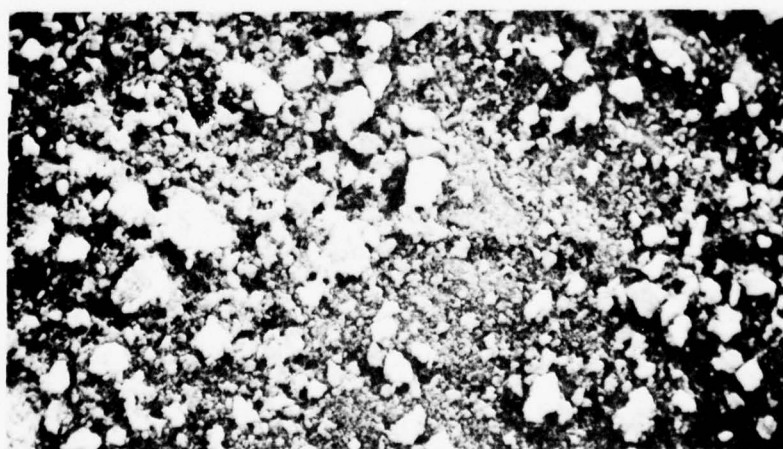
WC BALLS USED



2μ



4μ



10μ

FIG. 7

SEM PHOTOGRAPHS OF SAMPLE 3 POWDER, K106 $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$

Al_2O_3 BALLS USED

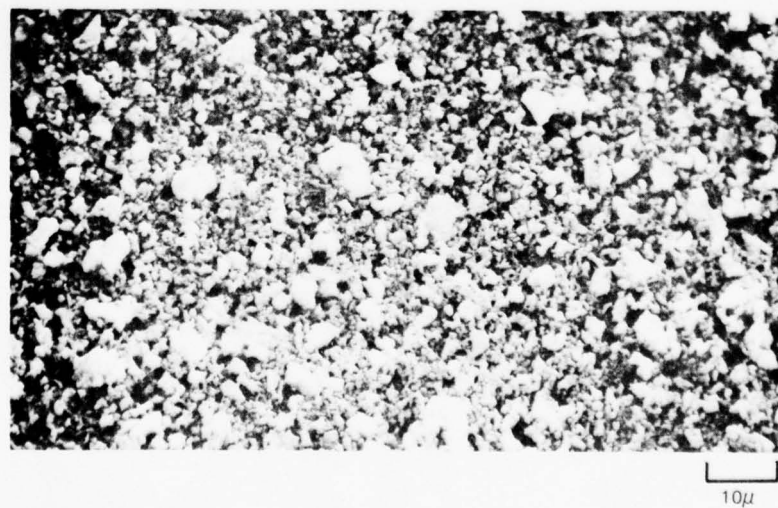
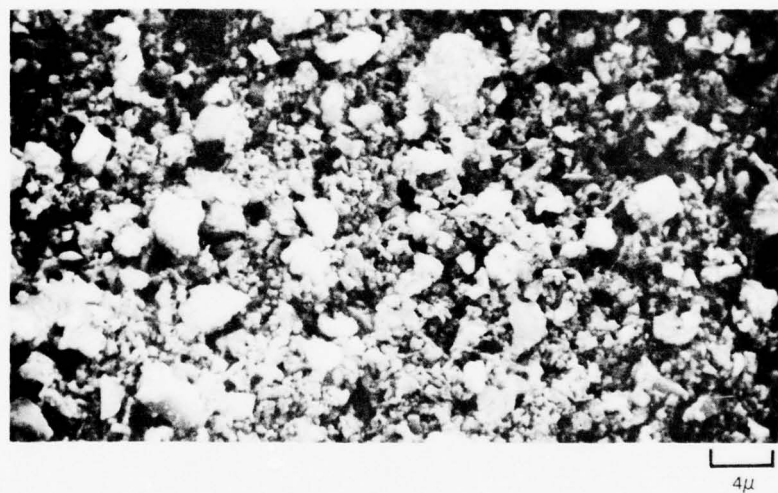
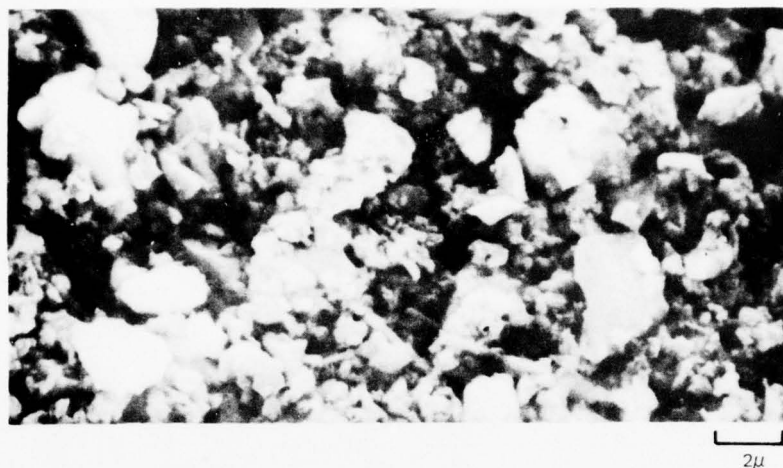
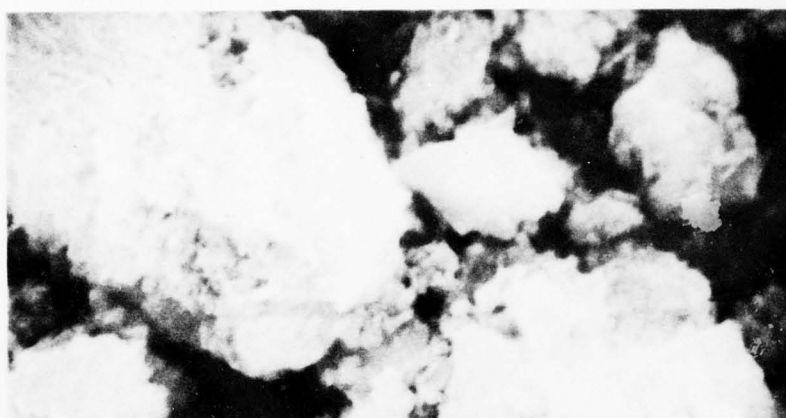


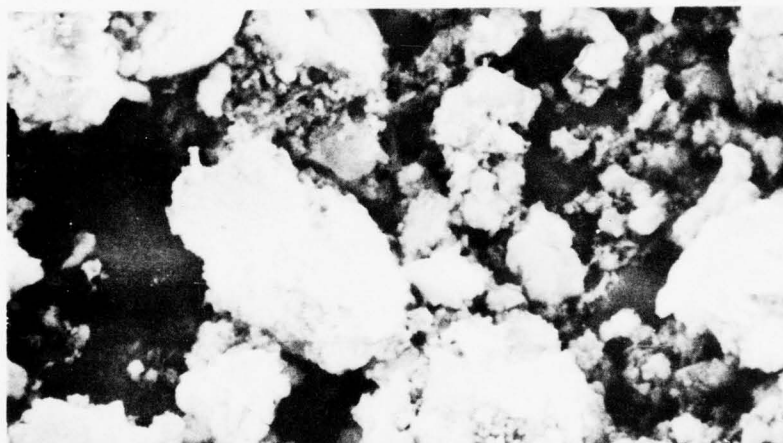
FIG. 8

SEM PHOTOGRAPHS OF SAMPLE 4

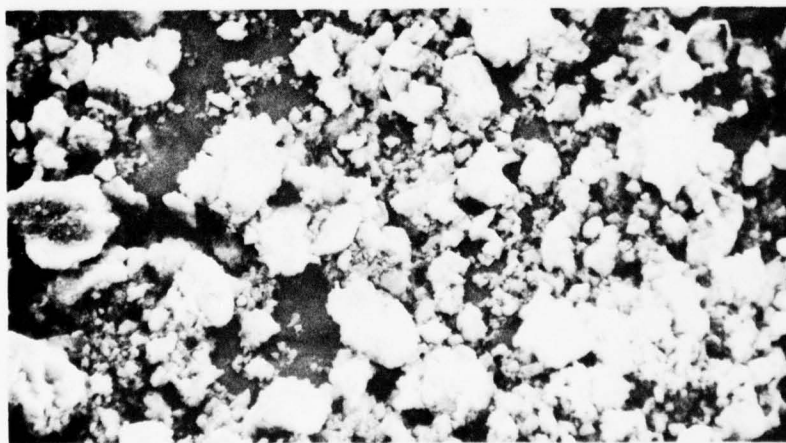
OLL $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ Al_2O_3 BALLS USED



2 μ



4 μ



10 μ

X-ray diffraction analyses of the powders showed no evidence of major differences in the powders. The α Si_3N_4 content of the powders varied but could not be correlated with the sintering behavior of the powders.

Chemical analyses of the starting older and new silicon nitride powders indicated a SiO_2 content of 3.4% for the former and 1.5% for the new K106 powder. (See Table 4) Since powders A, B, 2 and 4 contained old Si_3N_4 powder, it could be assumed that they contained at least 3.4% SiO_2 .

Another difference was found in the Al content of the mixed powders. Note in Table 5 that samples 1, 2, 3, 4, A and B contain increasing amounts of aluminum in that order, and the sinterability of the powders also improves in the same order. Since it can be assumed that the Al is picked up from the alumina-silica balls and ceramic container, it would be expected that the ball milling time is an important consideration. From the chemical analyses of the powders (Table 5), it can be seen that the Al content does not correlate well with the time of milling. However, these powders were prepared differently. As will be seen later, it does correlate when the same conditions are used in the ball milling operation. It does appear that the Al is picked up during milling and the sintering behavior of the powders improve with increasing Al content.

Silica and Alumina Additions

In the sintering studies it was shown that Al_2O_3 from the balls in the ball milling operation and from the starting powders promoted sintering. It was not known whether the Al_2O_3 additions alone would cause sintering or whether the SiO_2 additions also were required. These studies were conducted to answer this question.

Data from these studies are summarized in Table 6. In the first series of preparations, SiO_2 was added to a $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powder designated #1 (containing 1.5% SiO_2 , .2% Al_2O_3) which sintered very poorly at 1730°C . As can be seen from the densities, the addition of SiO_2 alone caused the #1 powder to sinter a little better, but still not very well.

In the next series of preparations, both SiO_2 and Al_2O_3 powders were added to #1 $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powder and to a mixture of K106 Si_3N_4 and Y_2O_3 . As can be seen in Table 6, when about 3-4% Al_2O_3 was present the pellets sintered well ($D = 2.3 \text{ g/cm}^3$) and when 8% Al_2O_3 was added the pellets sintered still better ($D = 2.9 \text{ g/cm}^3$).

Table 4

Major Impurities in Si_3N_4 Powder Lots (wt %)

<u>Si_3N_4 Powder</u>	<u>Ca</u>	<u>Al</u>	<u>Fe</u>	<u>O_2</u>	<u>(SiO_2)</u>
Old AME Si_3N_4	.001	.05	.15	1.8	3.4
K106 AME Si_3N_4	<.05	.10	.20	0.8	1.5

Table 5

Major Impurities in $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ Powders (wt %)

<u>Sample</u>	<u>Ca</u>	<u>Fe</u>	<u>Mg</u>	<u>Cr</u>	<u>Ni</u>	<u>Ti</u>	<u>Cu</u>	<u>Al</u>	<u>Milling Time (hrs)</u>
1	.1	.2	.01	.01	.01	<.01	.02	.09	48
2	.1	.2	.02	.03	.2	.05	.015	.11	48
3	.1	.2	.02	.03	.3	<.01	<.01	0.40	48
4	.1	.2	.05	.02	.03	.02	<.01	0.78	48
A	.4	.2	.2	.06	.05	.05	.02	1.74	25
B	.1	.2	.2	.02	.03	.02	<.01	4.36	60

Table 6

Data Showing the Effect of Additions on the Density
of Sintered $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$

<u>SiO₂ Additions</u>					
<u>Sample</u>	<u>Powder</u>	<u>Sintering Temp (°C)</u>	<u>Density (g/cm³)</u>	<u>% SiO₂</u>	<u>% Al₂O₃</u>
LPS19	#1 $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$	1730	1.22	1.5	.2
LPS37	#1 + SiO_2	1730	1.64	2.5	.2
LPS35	#1 + SiO_2	1730	1.70	6.5	.2
<u>SiO₂ and Al₂O₃ Additions</u>					
LPS40	#1 + SiO_2 + Al_2O_3	1730	2.30	6.5	4.2
LPS41	K106 Si_3N_4 + $15\text{Y}_2\text{O}_3$ + SiO_2 + Al_2O_3	1730	2.30	6.5	4
LPS42	K106 Si_3N_4 + $15\text{Y}_2\text{O}_3$ + SiO_2 + Al_2O_3	1730	2.90	6.5	8
<u>Al₂O₃ Additions</u>					
LPS75	P-3(K106 Si_3N_4 + $15\text{Y}_2\text{O}_3$ mix)	1730	1.74	1.5	2.2
LPS79	P-3 + Al_2O_3	1730	2.17	1.5	3.2
LPS80	P-3 + Al_2O_3	1730	2.66	1.5	4.2
LPS1	B	1730	3.10	-	8.2
LPS70	SN402· $15\text{Y}_2\text{O}_3$	1730	2.70	<3	.6
LPS105	SN402· $15\text{Y}_2\text{O}_3$ + Al_2O_3	1730	3.00	<3	1.6
LPS114	SN402· $15\text{Y}_2\text{O}_3$ + Al_2O_3	1730	3.21	<3	2.6

In the last series, Al_2O_3 powder was added to $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ mixtures without additional SiO_2 being added (see Table 6). These studies showed that the Al_2O_3 additions alone to the powders produced good sintering in powders which did not sinter well without the additions. These results are summarized in Fig. 9. The figure shows clearly that the addition of SiO_2 alone doesn't improve the sintering behavior of $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powder a great deal, the addition of both SiO_2 and Al_2O_3 does improve the sintering behavior, and the addition of Al_2O_3 alone seems to be the most effective. Because of the residual amount of SiO_2 in the starting Si_3N_4 powder, it is difficult to determine if any SiO_2 at all is required since some of the SiO_2 is present in all of these mixtures. Figure 9 also shows that about 3-4% Al_2O_3 is needed to get some sintering and around 8% Al_2O_3 in the powder mixture results in nearly theoretical density pellets being formed from 1730°C firings.

In Fig. 10 the advantage of being able to use a 1800°C firing temperature and high N_2 pressures is shown. Under these conditions, the very high density ($>3 \text{ g/cm}^3$) in the pellets can be achieved with only 3-4% Al_2O_3 additions to AME Si_3N_4 powder with Y_2O_3 .

Comparison of the Sintering Behavior of AME $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ and GTE $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ Powders

Sintering Behavior

When SN402 Si_3N_4 powder was used instead of AME Si_3N_4 powder in the $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ mixtures only 1.6% Al_2O_3 additive was required to get high densities ($\sim 3.2 \text{ g/cm}^3$) at 1730°C , see Fig. 9. As can be seen, this compares with about 8% Al_2O_3 needed for AME Si_3N_4 containing powders. If 1800°C , high N_2 pressure firings were used, the amount of Al_2O_3 required drops to 0.6% for SN402 in comparison to 3-4% Al_2O_3 for AME containing powders (see Fig. 10). The need for less Al_2O_3 for SN402 Si_3N_4 containing materials can probably be attributed to the amorphous nature and/or the fine particle size of the powder. It should be noted that even when SN402 Si_3N_4 was used some Al_2O_3 was needed; a sample containing .01% Al_2O_3 did not densify at 1730°C (LPS-65). The advantage of using less Al_2O_3 in the mixture can be seen in the oxidation resistances of the materials.

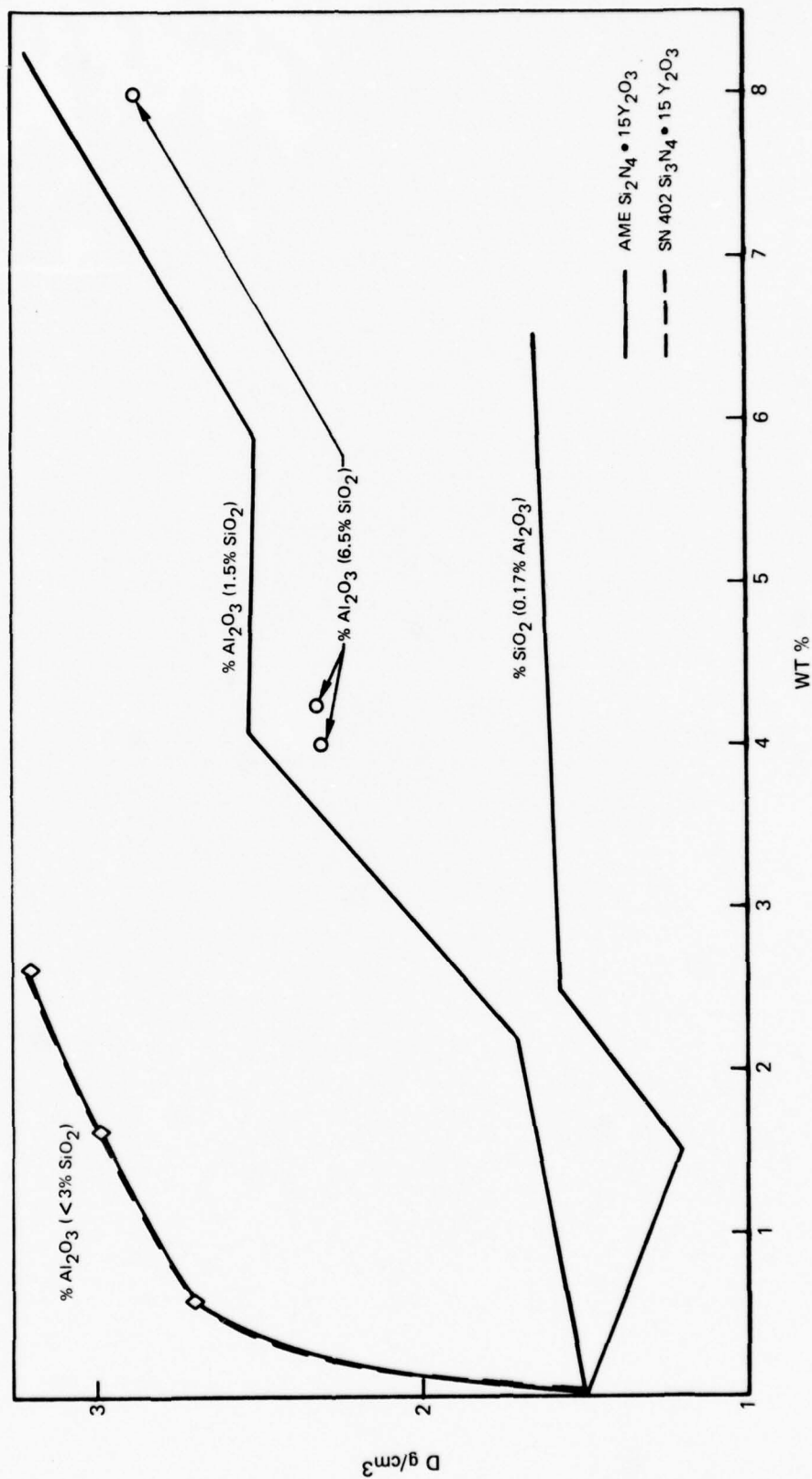
A better comparison of the sintering behavior and handleability of the $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powders containing either SN402 Si_3N_4 or the AME Si_3N_4 powder is illustrated in Fig. 11. As can be seen, the powder containing AME Si_3N_4 cold compacts ($D = 1.5 \text{ g/cm}^3$) better than the powder containing SN402 powder ($D = 1.0 \text{ g/cm}^3$). The SN402 $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ material then sinters better and has a higher density for 1730°C firings ($2.8 \text{ vs } 2.5 \text{ g/cm}^3$). For 1800°C firings the two powders yield about the same density pellets.

FIG. 9

DENSITY OF SINTERED $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ AS A FUNCTION OF WT% Al_2O_3 AND SiO_2 IN THE POWDER

(1730°C SINTERING TEMP)

1 HR



79-02-35-6

DENSITY OF SINTERED $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ AS A FUNCTION OF WT% Al_2O_3 IN THE POWDER

(1800°C SINTERING TEMP, 300 PSI N_2)

1 HR

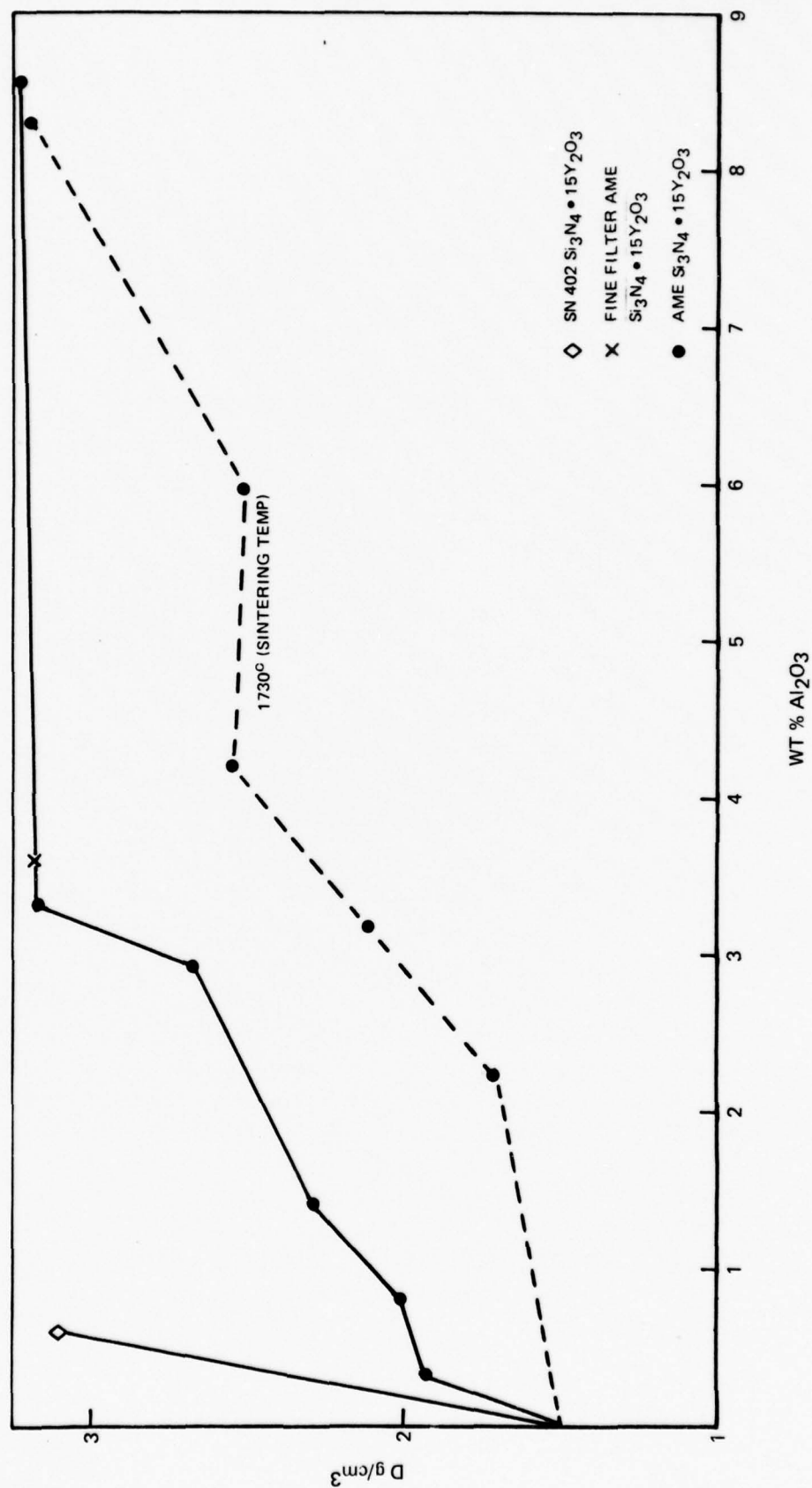
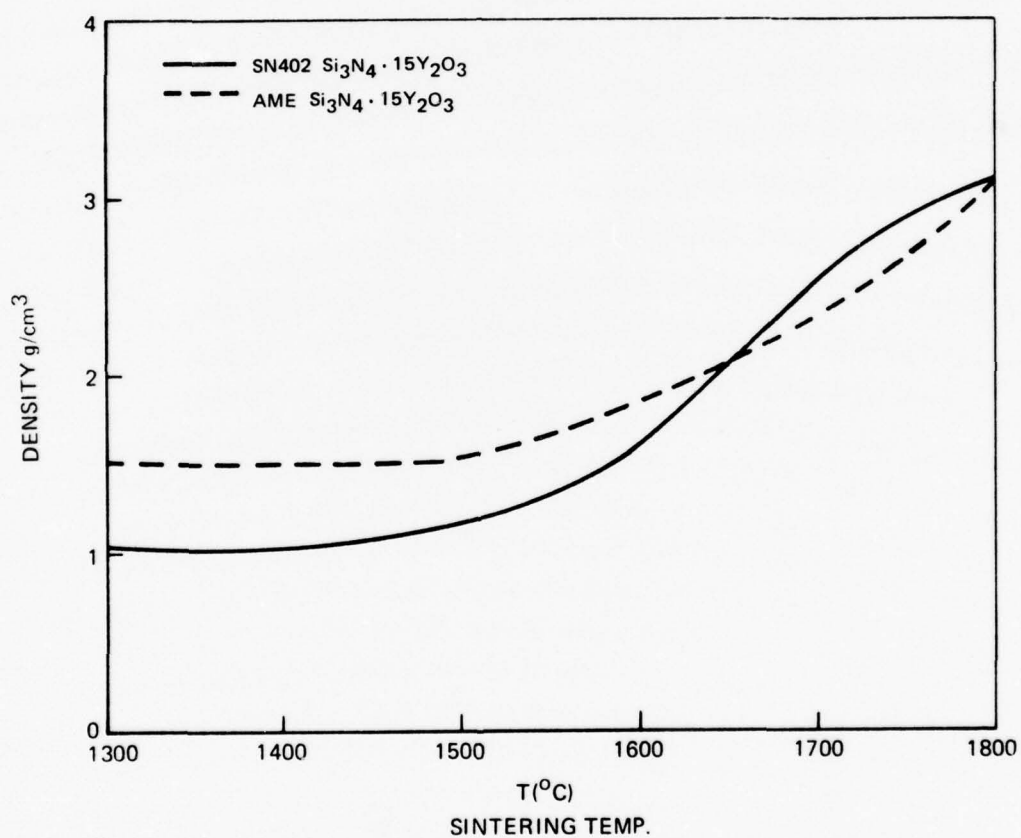


FIG. 10

79-02-35-3

DENSITY OF $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ MATERIAL AS A FUNCTION OF
SINTERING TEMPERATURE

(1HR)



For these two powders studied, the (P-2) $\text{SN402 Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ material contained only 0.6% Al_2O_3 while (A) AME $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ material contained 3-4% Al_2O_3 . From previous data it has been shown that when the SN402 $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powder contains 3% Al_2O_3 it sinters even better than this P-2 powder.

From preliminary data, it appears that fine AME Si_3N_4 and Stark Si_3N_4 powder behave more like the as purchased AME powder than SN402 Si_3N_4 in $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ materials. More detailed studies are needed to determine if there are differences in the sintering behavior of these powders.

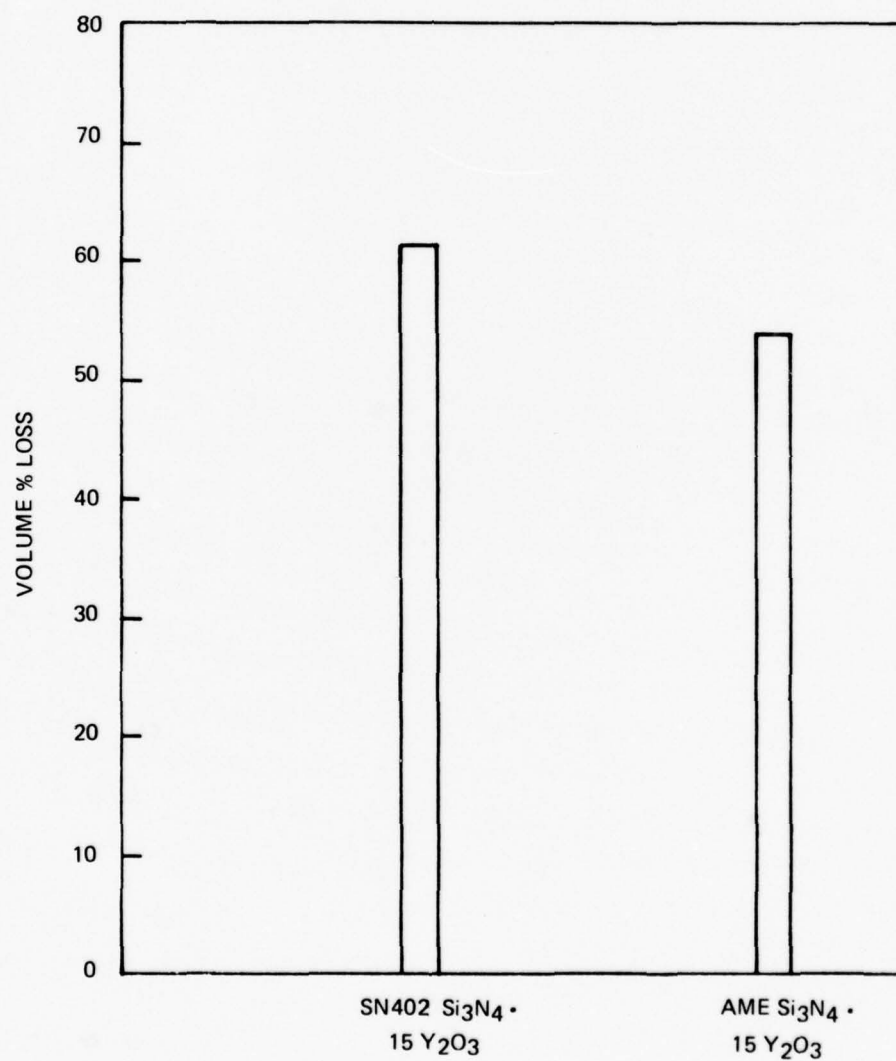
Volume and Weight Loss During Sintering

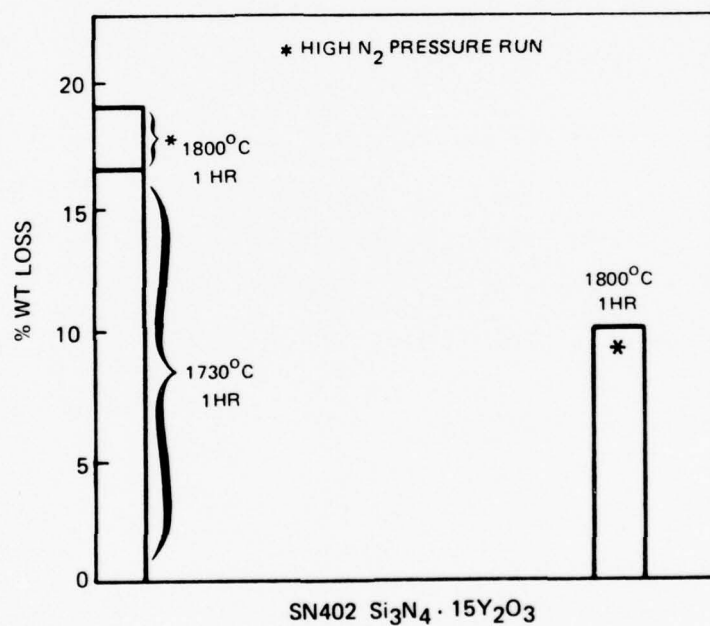
Although SN402 $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powders require less Al_2O_3 to produce high density material, one drawback is that they lose slightly more volume (shrinkage) and more weight during sintering than the AME Si_3N_4 containing powders. (See Fig. 12.) The greater shrinkage can be attributed in part to the difficulty of cold compacting the powder to high green densities. (See Fig. 11) This material also loses more weight (10% vs 2% for AME $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ at 1800°C for 1 hr, for example). Figure 13 of this report shows that 17% of its weight was lost when the material was heated at atmospheric pressure, while only 12% of its weight was lost when it was heated under 20 atm of N_2 pressure. When a sample heated to 1730°C was reheated at 1800°C under high N_2 pressures only about 2% more weight was lost. The density of the pellet heated in two steps to 1800°C was a little lower than that of the pellet heated to 1800°C in a single step. There is still not enough information to draw a conclusion as to whether a high N_2 pressure high temperature treatment of a $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ pellet sintered at a lower temperature will produce very high density material. This could be useful if it were possible; a lower density prefired material could be machined and then refired at a high temperature under high N_2 pressure to form a final desired shape.

Oxidation Studies

A detailed study of the oxidation behavior of samples made from powder A was conducted. X-ray diffraction analysis of the powder mixture before firing indicated that it contained mostly α Si_3N_4 with a small amount of β Si_3N_4 and Y_2O_3 . When the powder was fired in pellet form in N_2 at low temperatures ($1500, 1600^\circ\text{C}$) for 1 hr some conversion from α Si_3N_4 to β was detected in the pellet along with some unreacted Y_2O_3 and $\text{Si}_3\text{Y}_2\text{O}_3\text{N}_4$. When the pellets were fired at 1730 and 1800°C for 1 hr, all of the α Si_3N_4 was converted to β and only a small amount of $\text{Si}_3\text{Y}_2\text{O}_3\text{N}_4$ was found.

VOLUME % LOSS FOR $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ PELLETS DURING 1730°
FIRING FOR 1 HR IN N_2



% WT LOSS DURING SINTERING OF Si_3N_4 MATERIAL

Although some $\text{Si}_3\text{Y}_2\text{O}_3\text{N}_4$ (reported to be poor in oxidation resistance at 1000°C) was present in the high density pellets, oxidation studies on these pellets at 1000°C did not indicate any catastrophic oxidation behavior. (See Fig. 14) At 1350°C in air, the high density samples also showed good oxidation resistance. As can be seen in Fig. 15, the rate of weight gain for the pellets decreases with increasing density of the sample. The oxidation data for hot pressed NC-132 $\text{Si}_3\text{N}_4\cdot\text{MgO}$ are included for comparison purposes.

The significance of being able to reduce the Al_2O_3 content in these powders can be deduced from the oxidation data presented in Figs. 14 and 16. Note that for the pellets produced with $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3$ powders, the oxidation resistance improves with decreasing amounts of Al_2O_3 in the sample. Thus, while the powder B will produce high density pellets at only 1730°C it gains the most weight when heated at 1350°C in air. The other two samples require 1800°C firings to produce high density pellets, but they have good oxidation resistant properties. The GTE $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3$ is particularly impressive, gaining less weight at 1350°C than typical commercial hot pressed $\text{Si}_3\text{N}_4\cdot\text{MgO}$. Hot pressed $\text{Si}_3\text{N}_4\cdot\text{Y}_2\text{O}_3$ exhibits better oxidation resistance than this pressureless sintered sample, but it is still an experimental material.

Addition of Alumina

Since it has been learned in this study that $\text{Si}_3\text{N}_4\cdot 15\text{Y}_2\text{O}_3$ powder does not sinter well without some Al_2O_3 addition, consideration was given to the best method of adding the Al_2O_3 . For the SN402 Si_3N_4 powder only a very small amount of Al_2O_3 was required for sintering and this was easily picked up during ball milling. When AME powder was used, 3 to 4% Al_2O_3 was needed to form high density pellets at 1800°C and higher Al_2O_3 percentages were required if 1730°C firings were to be used.

Sintering studies on this program have shown that the Al_2O_3 additions can be made by mixing the powders in a mortar and pestel. While the high densities were achieved with these powders, regions of low melting areas on the pellets were observed; the best results were obtained when the Al_2O_3 was picked up from the alumina balls in the ball milling operation. (Powder A, for example, yielded pellets with very smooth surfaces.)

In the first attempt to investigate the Al_2O_3 pickup, 20 alumina balls were used in the mixing operation for a 50g load of Si_3N_4 and Y_2O_3 powders. Powder samples were taken at various times and pellets were made and heated at 1730°C . None of these sintered pellets were found to have high densities (see Table 7). From Fig. 17, it can be seen that even after 48 hrs, critical amounts of Al_2O_3 to cause good sintering were still not present.

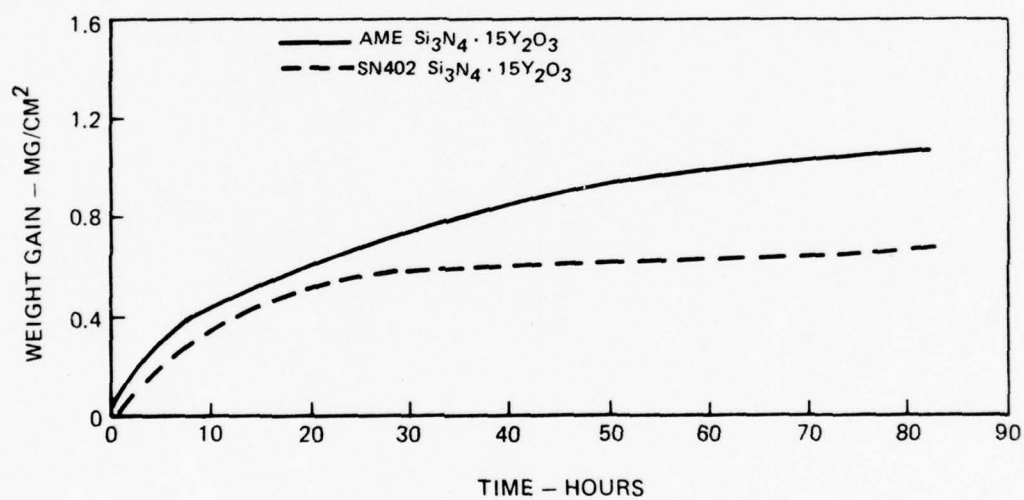
WEIGHT GAIN FOR $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ VS TIME, EXPOSURE AT 1000°C IN AIR

FIG. 15

WEIGHT GAIN FOR $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$ (A) VS. TIME, EXPOSURE AT 1350°C IN AIR

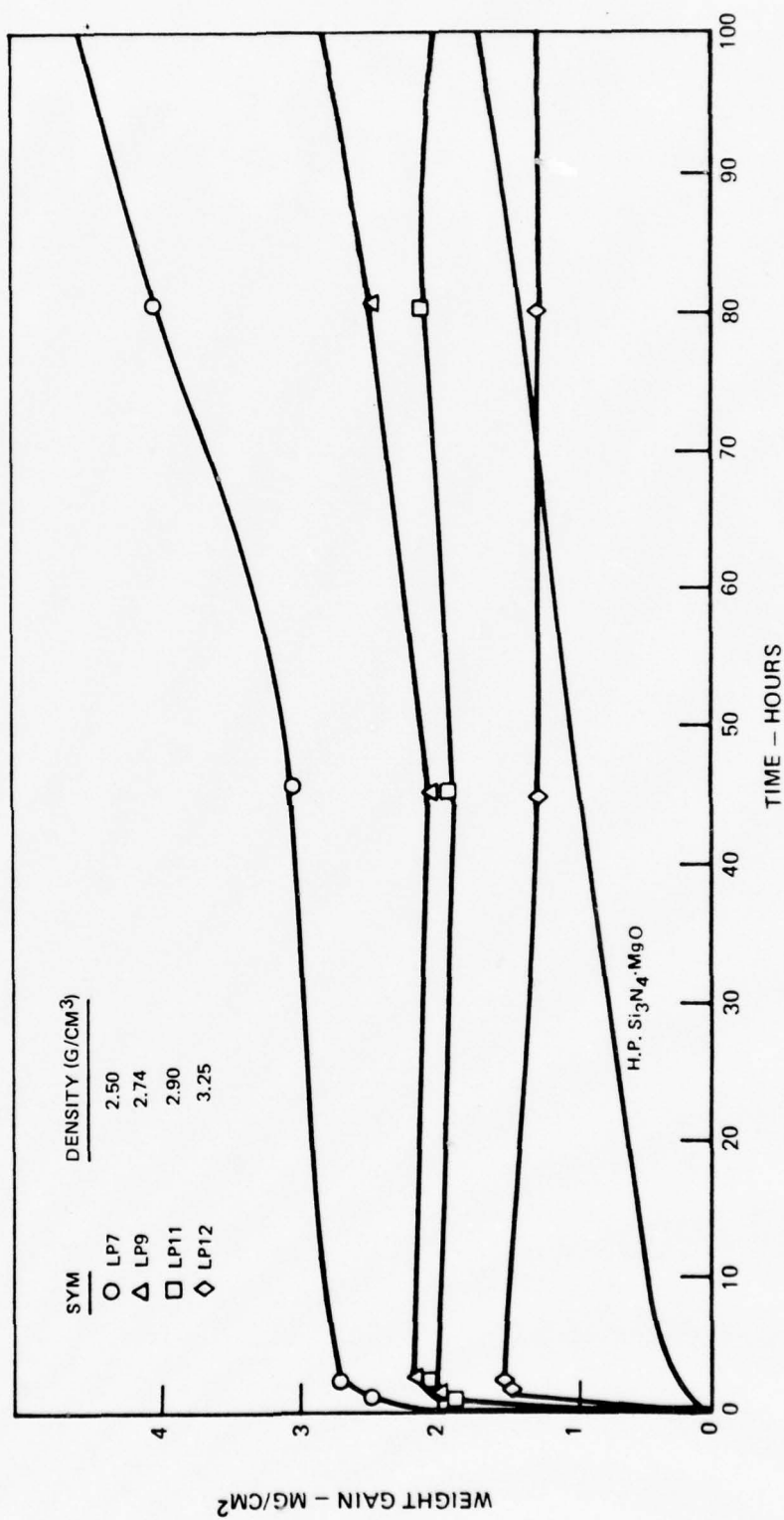
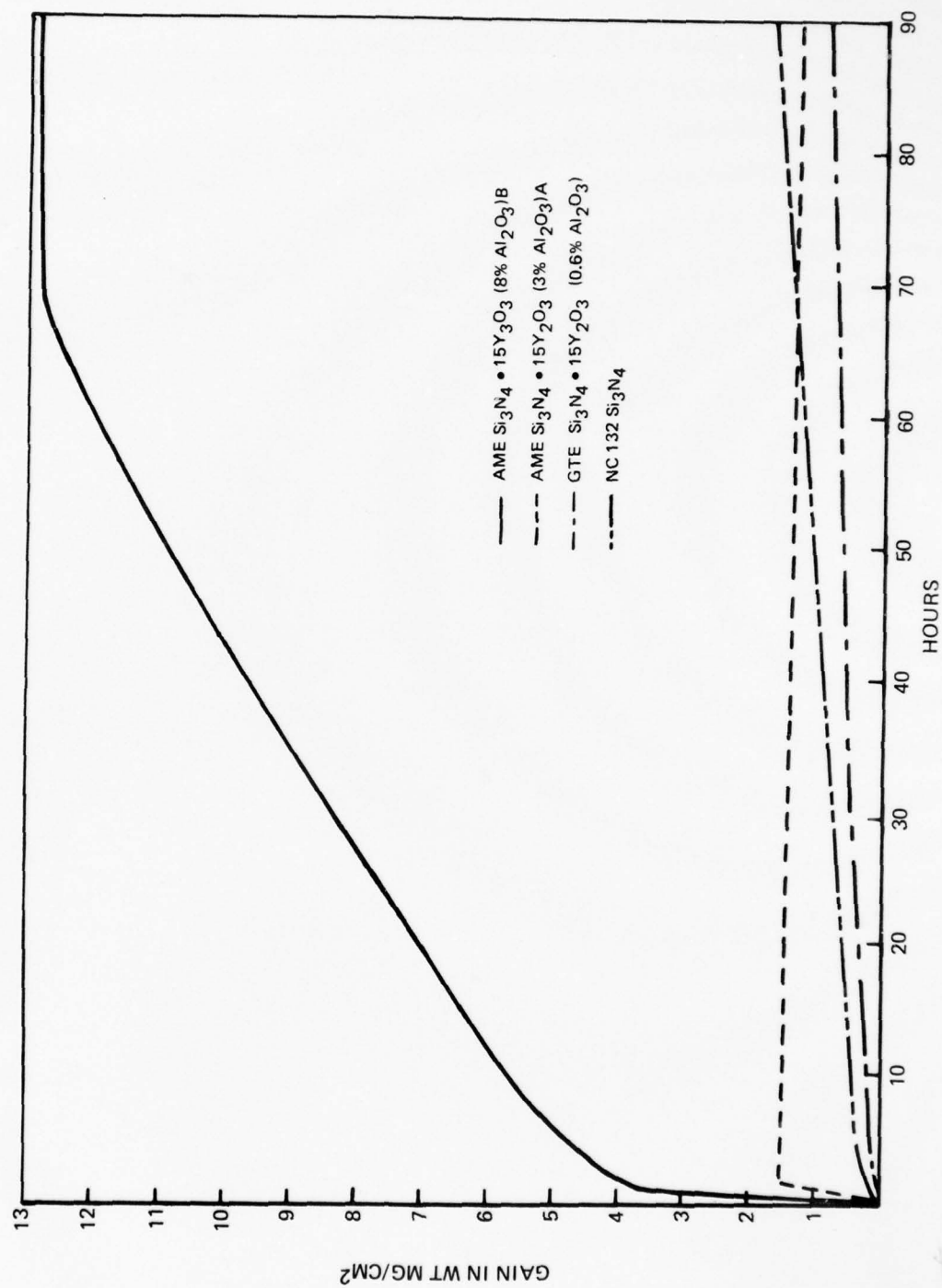


FIG. 16

WT GAIN FOR $\text{Si}_3\text{N}_4 \bullet 15\text{Y}_2\text{O}_3$ VS TIME, EXPOSURE AT 1350°C IN AIR

79-02-35-5

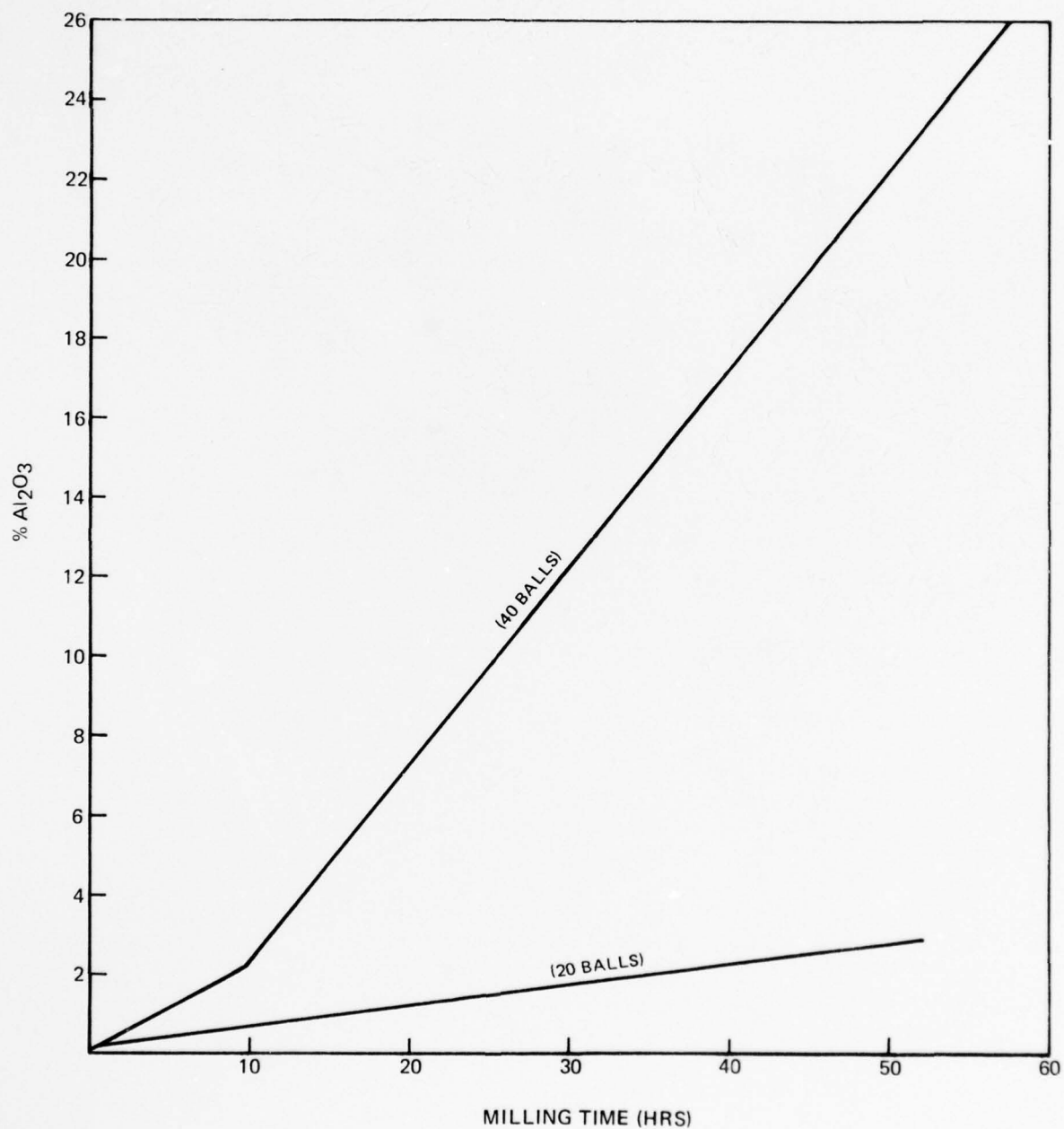
% Al_2O_3 PICK UP AS A FUNCTION OF BALL MILLING TIME

Table 7

Data Showing the Effect of Ball
Milling on the Density of Sintered $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$

(20 balls used)

<u>Sample</u>	<u>Powder</u>	<u>Milling Time (hrs)</u>	<u>Firing Temp (°C)</u>	<u>Density (g/cm³)</u>	<u>% Al₂O₃</u>
LPS 78	K106 $\text{Si}_3\text{N}_4 + 15 \text{Y}_2\text{O}_3$	10	1730	1.46	.82
LPS 77	"	24	1730	1.53	
LPS 76	"	48	1730	1.57	
LPS 75	"	60	1730	1.73	3.0

(40 balls used)

LPS 82	K106 $\text{Si}_3\text{N}_4 + 15 \text{Y}_2\text{O}_3$	10	1730	1.84	2.3
LPS 83	"	24	1730	3.24	
LPS 84	"	48	1730	3.30	20.2

These experiments were repeated with 40 balls and the results now showed that the powders produced high density pellets after they had been mixed for over 10 hrs (Table 7). This agrees with what is shown in Fig. 17. Note that after 10 hrs of mixing the amount of Al_2O_3 added to the powder increases to greater than 3%, and after a few more hours of mixing too much (to produce uniform pellets) Al_2O_3 was added. Thus, this study shows that the Al_2O_3 can be added by the ball milling operation, but there is some question as to how well controlled this technique can be. It may be that additions using Al containing solutions or other methods of producing a uniform mixture may be required to reproducibly control the Al_2O_3 content of the powder.

Strength Measurements

A rectangular bar (LPS-97) as prepared from an AME $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powder containing about 9% Al_2O_3 was found to have a RT (MOR) strength of 324 MPa (48 ksi). A rectangular bar made from powder A (LPS-53) with about 3.3% Al_2O_3 had a strength of 473 MPa (70 ksi). The true value was probably higher since the test specimen broke along a surface crack which was present on the specimen before testing. Approximately 540 MPa (80 ksi) was obtained for samples made from SN402 $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powder containing .6% Al_2O_3 . The pellets made from powder A are much smoother (less surface imperfections) than those produced from powders containing more Al_2O_3 and this was true of pellets made from GTE powder. This would explain the higher strength observed for the lower Al_2O_3 content materials. In addition, it is felt that the ability to lower the Al_2O_3 content of the pellets should also improve the high temperature properties of the materials.

For comparison purposes, specimens were prepared with $\text{Si}_3\text{N}_4 \cdot 5\text{MgO}$ powder. In the initial tests it was found that it was difficult to produce large enough cold pressed specimens which could be fired to produce MOR samples. In order to solve this problem, the powder was warm pressed in carbon dies at 538°F for 15 min. A specimen produced in this manner was heated at 1730°C in 1 atm of N_2 and another at 1800°C in 20 atm of N_2 . The first specimen was found to have a strength of 380 MPa (55 ksi) and the latter 262 MPa (38 ksi). The lower strength of the sample produced at 1800°C can probably be attributed to the warpage caused by the high temperature firing. It appears from these preliminary results that the selection of the $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ system to investigate high strength materials was justified.

Preparation of Airfoil Shapes

The first attempt to produce a blade shape involved slip casting of the powder, but because no pressure was used, porosity was present in the sample.

Next, attempts were made to prepare a mold in which the powder could be cold pressed before firing. An outline of this work on the molds is presented below.

Mold #1

Mold #1 was made from dental plaster using a small ceramic blade to reproduce. A cardboard tube 7.5 cm in diameter was cut into two (2) pieces 7.5 cm high; then the blade was coated with mold release and held in position on a piece of modeling clay on wax paper. A cardboard tube was placed over the blade and the dental plaster was poured around the blade filling the cardboard tube. After the first half was set, a piece of mylar was cut to fit around the blade and the other piece of cardboard tubing was placed over the first piece and dental plaster was poured around the blade filling the cardboard tube. This mold did not work as the plaster chipped too easily and a good blade shape was not achieved.

Mold #2

This mold was made much the same way as mold #1 except epoxy patch was used instead of dental plaster. The cardboard tubing was 3.75 cm by 2.5 cm high. The mold was satisfactory and an airfoil shape was pressed but it could not be released from the mold without breaking it.

Mold #3

Vertical Type - This mold was made by using an existing rectangular shaped mold of metal 5 cm long by 2.5 cm wide by 2.5 cm high (inside dimensions: .5 cm wide by 3.1 cm long). The base section of this mold was removable. A ram or plunger was made from a graphite bar 2.2 cm wide by 3.1 cm thick by 4.7 cm long. This ram was filed and sanded to form an airfoil shape. The ram was fitted to the rectangular mold and coated with mold release; then the open area was filled with epoxy patch. All pieces could be taken apart.

The powder was poured in the top of the mold with the epoxy pieces in place and then compressed with the graphite ram. The bottom of the mold was then removed and the pressed powder pushed through. The epoxy would also push through and because the graphite was not that precise in shape (as a machined piece would be), the powder tended to hang up in the trailing edge.

Mold #4

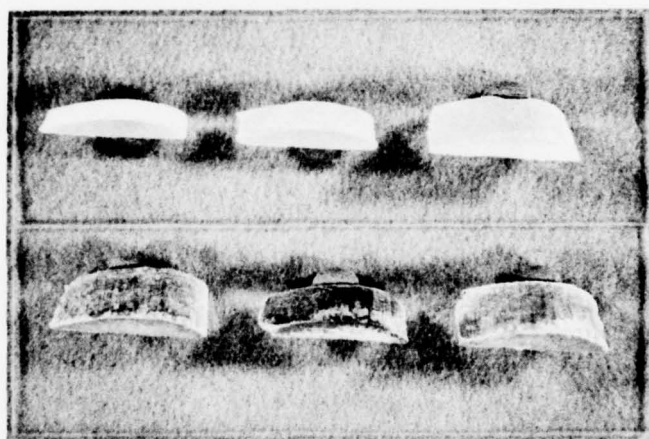
The last mold was made using a .63 cm x 2.5 cm aluminum bar which was cut to make a rectangular mold the same size as mold #3. This mold contained the same bottom piece as was used in mold #3. An airfoil shaped piece was found and reworked to use as a ram or plunger. Because the airfoil was both concave and convex in shape, it was filled with epoxy to make one flat surface.

The airfoil or blade was then coated with mold release and set up in the rectangular mold (flat edge against one flat side of the mold) with the bottom, coated with mold release, in place.

The rest of the mold cavity was then filled with epoxy patch. Making the mold in this way gave a metal finish against the epoxy insuring a smoother surface. The airfoil cavity was filled with powder, then pressed with the airfoil shaped plunger. The bottom was removed and the pressed powder pushed through - the epoxy stays in place.

A series of blade shapes pressed in this mold from $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ powder and fired at 1730°C is shown in Fig. 18. The powders were selected to give high density material during pressureless sintering.

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



CONCLUSIONS AND RECOMMENDATIONS

As can be seen from these studies, high density $\text{Si}_3\text{N}_4 \cdot 15\text{Y}_2\text{O}_3$ pellets can be formed by pressureless sintering if small amounts of Al_2O_3 are added to the powders. Some insight as to the amount of Al_2O_3 required was gained to date. The amount of Al_2O_3 needed was dependent on the source of Si_3N_4 powder used. In future studies, the amount of Al_2O_3 required should be investigated further so that it can be determined for even different Si_3N_4 powder (i.e. different particle size material) from the same source. The work should also be directed toward learning how to decrease the amount of Al_2O_3 additions and still obtain high density material, which this study has shown improves properties.

Methods of adding the Al_2O_3 should be explored. Ball milling with Al_2O_3 balls produced good results, but a more reliable method is needed.

Gas heat treatments also should be explored as a method of improving the properties of already sintered material. These studies should be compared with results obtained by G. Gazza (Ref. 9) of AMMRC on hot-pressed $\text{Si}_3\text{N}_4 \cdot \text{Y}_2\text{O}_3$ materials.

Once good materials are prepared and the amount of additives required are reduced, the creep properties should be investigated and the materials should be used to form complex shapes.

ACKNOWLEDGEMENT

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APPENDIX A

Data for Pressureless Sintering Runs

Run	Powder*	Temp (°C)	Initial Density (g/cm ³)	Final Density (g/cm ³)	Time (hr)
LPS 1	(B) AME Si ₃ N ₄ ·15Y ₂ O ₃ (8.2Al ₂ O ₃)	1730	1.35	3.04	11
2	(B) AME Si ₃ N ₄ ·15Y ₂ O ₃ (8.2Al ₂ O ₃)	1730	1.44	3.10	½
3	(B) AME Si ₃ N ₄ ·15Y ₂ O ₃ (8.2Al ₂ O ₃)	1800	1.43	3.19	½
4	Fine filter AME Si ₃ N ₄ ·15Y ₂ O ₃ (3.6Al ₂ O ₃)	1730	1.56	2.65	½
5	Starck Si ₃ N ₄ ·15Y ₂ O ₃	1730	1.66	2.00	½
6	Fine filter AME Si ₃ N ₄ ·15Y ₂ O ₃ (3.6Al ₂ O ₃) AMMRC	1800	1.55	3.09	½
7	Starck Si ₃ N ₄ ·15Y ₂ O ₃	1800	1.67	1.96	½
9	Starck Si ₃ N ₄ ·15Y ₂ O ₃	1730	1.64	2.18	1
10	Fine filter Si ₃ N ₄ ·15Y ₂ O ₃ (3.6Al ₂ O ₃)	1730	1.48	2.33	1
11	(B) AME Si ₃ N ₄ ·15Y ₂ O ₃ (8.2Al ₂ O ₃) new cruc.	1730	1.58	1.66	½
12	(B) AME Si ₃ N ₄ ·15Y ₂ O ₃ (8.2Al ₂ O ₃) new cruc.	1730	1.58	no data	1
13	(B) AME Si ₃ N ₄ ·15Y ₂ O ₃ (8.2Al ₂ O ₃) new cruc.	1730	1.40	2.38	½
14	Starck Si ₃ N ₄ ·15Y ₂ O ₃	1800	1.66	1.77	
15	Starck Si ₃ N ₄ ·15Y ₂ O ₃	1800	1.61	2.63	½
16	Starck Si ₃ N ₄ ·15Y ₂ O ₃	1800	1.68	2.67	1
18	Starck Si ₃ N ₄ ·15Y ₂ O ₃ + SiO ₂	1730	1.25	2.78	1
19	(1) AME Si ₃ N ₄ ·15Y ₂ O ₃ (.17Al ₂ O ₃)	1730			
20	(2) AME Si ₃ N ₄ ·15Y ₂ O ₃ (.21Al ₂ O ₃)	1730	1.49	1.24	1
22	(3) AME Si ₃ N ₄ ·15Y ₂ O ₃ (.76Al ₂ O ₃)	1730	1.44	1.22	1
23	(4) AME Si ₃ N ₄ ·15Y ₂ O ₃ (1.46Al ₂ O ₃)	1730	1.53	1.27	1
24	(B) AME Si ₃ N ₄ ·15Y ₂ O ₃ (8.2Al ₂ O ₃)	1730	1.46	3.03	1
25	(4) AME Si ₃ N ₄ ·15Y ₂ O ₃ (1.46Al ₂ O ₃)	1730	1.53	1.40	1
27	(4) AME Si ₃ N ₄ ·15Y ₂ O ₃ (1.46Al ₂ O ₃)	1730	1.53	1.31	1
28	(1) AME Si ₃ N ₄ ·15Y ₂ O ₃ (.17Al ₂ O ₃)	1730	1.38	1.27	1
29	(1) AME Si ₃ N ₄ ·15Y ₂ O ₃ (.17Al ₂ O ₃)	1800	1.53	1.75	1
30	(2) AME Si ₃ N ₄ ·15Y ₂ O ₃ (.21Al ₂ O ₃)	1800	1.5	1.94	1
32	(3) AME Si ₃ N ₄ ·15Y ₂ O ₃ (.76Al ₂ O ₃)	1800	1.53	2.04	1
33	(4) AME Si ₃ N ₄ ·15Y ₂ O ₃ (1.46Al ₂ O ₃)	1800	1.49	2.33	1
34	SN402 Si ₃ N ₄ ·10Y ₂ O ₃ packed in powder	1730		2.9	

<u>Run</u>	<u>Powder</u>	<u>Temp</u> <u>(°C)</u>	<u>Initial</u> <u>Density</u> <u>(g/cm³)</u>	<u>Final</u> <u>Density</u> <u>(g/cm³)</u>	<u>Time</u> <u>(hr)</u>
LPS 35	(1) + 5 SiO ₂ (.17Al ₂ O ₃)	1730	1.47	1.75	1
36	(1) + 10 SiO ₂ (.17Al ₂ O ₃)	1730	1.43	1.39	1
37	(1) + 1 SiO ₂ (.17Al ₂ O ₃)	1730	1.49	1.64	1
39	(1) + 4 Al ₂ O ₃ (4.2Al ₂ O ₃)	Incomplete data		1.53	
40	LPS 35 mix + 5 Al ₂ O ₃	Incomplete data		2.3	
41	AME Si ₃ N ₄ + 15Y ₂ O ₃ + 5SiO ₂ + 5Al ₂ O ₃	Incomplete data		2.3	
42	AME Si ₃ N ₄ + 15Y ₂ O ₃ + 5SiO ₂ + 8Al ₂ O ₃	1730	1.5	2.89	1
43	Starck Si ₃ N ₄ + 5SiO ₂ + 5Al ₂ O ₃ + 15Y ₂ O ₃	1730		2.85	1
45	AME Si ₃ N ₄ .5Y ₂ O ₃	1730		1.4	1
46	AME Si ₃ N ₄ .10Y ₂ O ₃	1730		0.9	1
47	AME Si ₃ N ₄ .15Y ₂ O ₃	1730		1.67	1
49	LPS 46 mix + 5 SiO ₂ + 5 Al ₂ O ₃	1730	1.5		1
50	LPS 45 mix + 5 SiO ₂ + 5 Al ₂ O ₃	1730	1.38	1.07	1
51	LPS 47 mix + 5 SiO ₂ + 5 Al ₂ O ₃	1730	1.03	1.6	1
52	(A) AME Si ₃ N ₄ .15Y ₂ O ₃ (3.3Al ₂ O ₃) AMMRC	1800	1.49		1
53	(A) AME Si ₃ N ₄ .15Y ₂ O ₃ (3.3Al ₂ O ₃) Cruc. B	1730	1.43	2.8	1
54	Fine filter AME Si ₃ N ₄ .15Y ₂ O ₃ (3.6Al ₂ O ₃) AMMRC	1800	2.16	3.15	1
55	Fine filter AME Si ₃ N ₄ .15Y ₂ O ₃ (3.6Al ₂ O ₃)	1730	1.9	2.79	1
56	AME Si ₃ N ₄ + 10 Y ₂ O ₃ + 5 SiO ₂ + 4 Al ₂ O ₃	1730	1.45	1.64	1
57	AME Si ₃ N ₄ + 5 Y ₂ O ₃ + 5 SiO ₂ + 4 Al ₂ O ₃	1730	1.39	1.51	1
58	AME Si ₃ N ₄ + 15 Y ₂ O ₃ + 5 SiO ₂ + 4 Al ₂ O ₃	1730	1.44	2.07	1
59	Same as LPS 58	1730	1.02	1.87	1
60	Same as LPS 59	1730	1.04	2.04	1
61	(P1) AME Si ₃ N ₄ + 15 Y ₂ O ₃ (2.8Al ₂ O ₃)	1730	1.55	1.67	1
62	(P1) (2.8Al ₂ O ₃)	1730	1.55	1.65	1
63	Fine filter Si ₃ N ₄ .15Y ₂ O ₃ (3.6Al ₂ O ₃)	1800	1.065	2.85	1
64	(P1) (2.8Al ₂ O ₃)	1800	1.543	2.69	1
65	SN402 Si ₃ N ₄ + 15 Y ₂ O ₃ (<.02Al ₂ O ₃)	1730	1.07	1.06	1
66	SN402 Si ₃ N ₄ + 10 Y ₂ O ₃ (1.67Al ₂ O ₃) (H.P. material)	1730	1.09	2.46	1

Run	Powder	Temp (°C)	Initial Density (g/cm ³)	Final Density (g/cm ³)	Time (hr)
LPS 67	AME Si ₃ N ₄ + 5 MgO	1800			1
68	SN402 Si ₃ N ₄ + 10 Y ₂ O ₃ , 38ksi	1800	1.11	2.84	1
69	AME Si ₃ N ₄ + 5 MgO, 55 ksi	1730			1
70	(P2) SN402 Si ₃ N ₄ + 15 Y ₂ O ₃ (.6Al ₂ O ₃)	1730	1.16	2.70	1
71	(P2)	1800	1.16	3.10	1
72	AME Si ₃ N ₄ •5CeO ₂	1730	Broke		1
73	AME Si ₃ N ₄ •15Y ₂ O ₃ ball, cruc. A milled 48 hrs + 48 hrs, 40 balls	1730	Shattered		1
74	(B) AME Si ₃ N ₄ •15Y ₂ O ₃	1730	1.42	3.05	1
75	P-3 AME Si ₃ N ₄ •15Y ₂ O ₃ ball milled 60 hrs, 20 balls(3Al ₂ O ₃)	1730	1.49	1.73	1
76	P-3 - 48 hr mix	1730	1.40	1.57	1
77	P-3 - 24 hr mix	1730	1.41	1.53	1
78	P-3 - 10 hr mix(.82Al ₂ O ₃)	1730	1.48	1.46	1
79	P-3 - 60 hr mix + 1 Al ₂ O ₃	1730	1.45	2.17	1
80	P-3 - 48 hr mix + 2 Al ₂ O ₃	1730	1.50	2.34	1
81	P-3 - 60 hr mix + 48 hrs	1730	1.58	2.66	1
82	AME Si ₃ N ₄ •15Y ₂ O ₃ , 10 hrs(2Al ₂ O ₃) DP-1 40 balls	1730	1.44	1.84	1
83	DP-1 24 hrs	1730	1.51	3.24	1
84	DP-1 48 hrs(20Al ₂ O ₃)	1730	1.55	3.30	1
85	DP-1 60 hrs	1730	1.55	3.18	1
86	(3) AME Si ₃ N ₄ •15Y ₂ O ₃ (.76Al ₂ O ₃) + 2SiO ₂ + 5 Al ₂ O ₃	1730	1.44	2.23	1
87	(3) + 5 Al ₂ O ₃	1730	1.58	2.53	1
88	(3) + 5 Al ₂ O ₃ flat cruc.	1730	0.791	1.76	1
89	DP-2 AME Si ₃ N ₄ •15Y ₂ O ₃ + 5 Al ₂ O ₃ , 48 hrs, 20 balls	1730	1.48	3.20	1
90	DP-1-24 AME Si ₃ N ₄ •15Y ₂ O ₃ 40 balls rect. sample	1730	1.31	1.93	1
91	DP-1-48 40 balls rect. sample	1730	1.36	2.81	1
92	DP-1-60 40 balls rect. sample	1730	1.28	2.92	1
93	DP-1-24 40 balls rect. sample	1730	1.37	2.91	1
94	DP-2-48 20 balls rect. sample	1730	1.44	2.95	1
95	(3) + 5 Al ₂ O ₃	1730	1.46	2.38	1
96	DP-1-24 40 balls rect. sample	1730	1.08	2.60	1
97	DP-1-48 40 balls rect. sample	1730	1.31	3.05	1
98	DP-1-60 40 balls rect. sample	1730	1.21	2.88	1
99	P-2 rect. sample	1800	0.98	2.95	1

<u>Run</u>	<u>Powder</u>	<u>Temp</u> <u>(°C)</u>	<u>Initial</u> <u>Density</u> <u>(g/cm³)</u>	<u>Final</u> <u>Density</u> <u>(g/cm³)</u>	<u>Time</u> <u>(hr)</u>
LPS 100	P-2 rect. sample	1800	0.997	2.88	1
102	P-2	1730	1.11	2.47	1
103	SN402 Si ₃ N ₄ •10Y ₂ O ₃ 24 hrs	1800	1.03	2.71	1
104	P-2 A-B	1800	1.09	3.18	1
105	P-2 + 1 Al ₂ O ₃	1730	1.14	3.01	1
106	P-2 + 2 Al ₂ O ₃	1730	1.15	3.15	1
109	P-2 Cruc. C	1730	1.12	2.45	1
110	P-2 packed in powder	1730	1.09	2.35	1
113	P-2 rect. sample	1800	0.95	3.10	1
114	P-2 + 2 Al ₂ O ₃	1730	1.13	3.21	1
116	P-2 + 2 Al ₂ O ₃ rect. sample	1730	0.974	2.58	1
117	(B)	1730	1.36	3.2	1
118	blade				
119	P-2 rect. sample	1800	Broke in Half		1
120	P-2 rect. sample	1800	0.994	3.03	1
121	blade				
122	P-2	1730	1.13	2.44	1
123	LPS 122 reheat	1800	2.44	2.91	1
124	blade				
125	SN402 Si ₃ N ₄ •15Y ₂ O ₃ + .1 Al ₂ O ₃	1730	1.11	1.07	1
126	SN402 Si ₃ N ₄ •15Y ₂ O ₃ + .5 Al ₂ O ₃	1730	1.11	1.25	1
127	Same as LPS 125	1730	1.13	1.01	1
128	SN402 Si ₃ N ₄ •15Y ₂ O ₃ + 1 Al ₂ O ₃	1730	1.14	1.31	1
129	SN402 Si ₃ N ₄ •15Y ₂ O ₃ +22 Al ₂ O ₃	1730	1.16	1.62	1
130	P-2 rect. sample, 70 ksi	1800	0.863	3.18	1
131	blade				
132	P-2	1400	1.00	1.69	1
133	P-2	1600	1.09	1.38	1
134	P-2	1500	1.08	1.61	1
135	P-2	1730	1.10	2.26	1
136	P-2	1400	1.08	1.02	1
137	P-2	1500	1.08	1.16	1
138	blade				
139	P-2	1600	1.08	1.65	1
140	P-2	1730	1.08	2.82	1
141	SN402•15Y ₂ O ₃ + .1 Al ₂ O ₃	1730	1.12	1.12	1
142	SN402•15Y ₂ O ₃ + .5 Al ₂ O ₃	1730	1.15	1.26	1
143	SN402•15Y ₂ O ₃ + 2 Al ₂ O ₃	1730	1.15	1.76	1
144	SN402•15Y ₂ O ₃ + 3 Al ₂ O ₃	1730	1.18	2.27	1
145	P-2	1800	1.08	3.11	1

<u>Run</u>	<u>Powder</u>	<u>Temp</u> <u>(°C)</u>	<u>Initial</u> <u>Density</u> <u>(g/cm³)</u>	<u>Final</u> <u>Density</u> <u>(g/cm³)</u>	<u>Time</u> <u>(hr)</u>
LPS 146	SN402 Si ₃ N ₄ •5CeO ₂	1730	1.08	0.95	1
147	SN402 Si ₃ N ₄ •10CeO ₂	1730	1.15	1.35	1
148	SN402 Si ₃ N ₄ •15CeO ₂	1730	1.17	1.89	1
149	New P-2, 24 hrs + 5 Al ₂ O ₃ SN402 Si ₃ N ₄ •15Y ₂ O ₃	1730	1.19	2.88	1
150	New P-2	1730	1.14	1.87	1
151	New P-2	1800	1.16	3.08	1
152	LPS 140 reheated	1800	2.82	3.00	1
153	Fine filter AME Si ₃ N ₄ •15Y ₂ O ₃ (3.6Al ₂ O ₃) + 5 Al ₂ O ₃	1730	1.40	2.14	1
154	Fine filter AME Si ₃ N ₄ •15Y ₂ O ₃ (3.6Al ₂ O ₃) + 5 Al ₂ O ₃	1800	1.50	2.75	1
155	Stark Si ₃ N ₄ •15Y ₂ O ₃ + 5 Al ₂ O ₃	1800	1.97	2.37	1
156	Stark Si ₃ N ₄ •15Y ₂ O ₃ + 5 Al ₂ O ₃	1730	1.91	2.42	1
157	AME Si ₃ N ₄ •15Y ₂ O ₃ + 5 Al ₂ O ₃	1800	1.47	2.77	1
158	AME Si ₃ N ₄ •15Y ₂ O ₃ + 5 Al ₂ O ₃	1730	1.50	2.05	1
159	AME Si ₃ N ₄ •15Y ₂ O ₃ + 6 Al ₂ O ₃	1800	1.50	2.69	1
160	AME Si ₃ N ₄ •15Y ₂ O ₃ + 6 Al ₂ O ₃	1730	1.49	2.30	1

*Al₂O₃ in mixture given in parenthesis when known

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SINTERING OF Si_3N_4 UNDER NITROGEN PRESSURE
F. S. Galasso and R. D. Veltri
United Technologies Research Center
East Hartford, Connecticut 06108
Technical Report AMMRC CTR 79-37, June 1979, 44 pp -
illus.-tables, Contract DAAG46-78-C-0017
Final Report, March 1978 to March 1979

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Ceramic materials
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Sintering
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Yttrium silicon nitride
Milling
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This study showed that some Al_2O_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. The oxidation resistance of these materials improved with decreasing amounts of Al_2O_3 in the samples and with increasing density. The amount of Al_2O_3 required to obtain high densities was less for $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$ pellets heated under high N_2 pressures. SN402 $\text{Si}_3\text{N}_4\text{-15Y}_2\text{O}_3$ pellets heated in this way showed less weight gain in air than hot pressed $\text{Si}_3\text{N}_4\text{-MgO}$ over an 80 hr period at 1350°C.

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