SILICON NITRIDE-ALUMINUM OXIDE SOLID SOLUTION (SiAION) FORMATION AND DENSIFICATION BY PRESSURE SINTERING

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Stirred-ball-mill-blended $\text{Si}_3\text{N}_4$ and $\text{Al}_2\text{O}_3$ powders were pressure sintered in order to investigate the mechanism of solid-solution formation and densification in the $\text{Si}_3\text{N}_4-\text{Al}_2\text{O}_3$ system. Powder blends with $\text{Si}_3\text{N}_4:\text{Al}_2\text{O}_3$ mole ratios of 4:1, 3:2, and 2:3 were pressure sintered at 27.6 MN/m$^2$ (4000 psi) pressure at temperatures to 1700°C (3090°F). The compaction behavior of the powder blends during pressure sintering was determined by observing the density of the powder compact (ram travel) as a function of temperature and time starting from room temperature. This information, combined with the results of X-ray diffraction and metallographic analyses regarding solutioning and phase transformation phenomena in the $\text{Si}_3\text{N}_4-\text{Al}_2\text{O}_3$ system, was used to describe the densification behavior.
SILICON NITRIDE - ALUMINUM OXIDE SOLID SOLUTION (SiAION)

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SUMMARY

In this experimental study, precompacted mixtures of silicon nitride (Si$_3$N$_4$) and aluminum oxide (Al$_2$O$_3$) powders were pressure sintered under vacuum in order to investigate the mechanism of formation and densification in the Si$_3$N$_4$-Al$_2$O$_3$ system. Intensive mixing and comminution of the fine-particle-size powders was attained by using a stirred-ball mill.

Powder blend compacts of Si$_3$N$_4$ plus Al$_2$O$_3$ in mole ratios of 4:1, 3:2, and 2:3 were vacuum pressure sintered at temperatures from 1100$^\circ$C to 1700$^\circ$C (2010$^\circ$F to 3090$^\circ$F) for as long as 2 hours at a pressure of 27.6 MN/m$^2$ (4000 psi). Pore-free compacts with only a trace of second phase were obtained at temperatures as low as 1500$^\circ$C (2730$^\circ$F) with the 40-mol% Si$_3$N$_4$ - 60-mol% Al$_2$O$_3$ composition. (All Si$_3$N$_4$-Al$_2$O$_3$ compositions are in mole percent and will hereinafter be given in the form 40Si$_3$N$_4$-60Al$_2$O$_3$.)

Ram travel recorded during pressure sintering indicated high densification rates in the temperature range 1400$^\circ$C to 1700$^\circ$C (2550$^\circ$F to 3090$^\circ$F) with a maximum at 1500$^\circ$C (2730$^\circ$F). Densification rates and end-point densities were correlated with the results of X-ray diffraction and metallographic analyses to characterize the solutioning and phase transformations in the Si$_3$N$_4$-Al$_2$O$_3$ system.

INTRODUCTION

Silicon nitride (Si$_3$N$_4$) in both the pressure-sintered (hot pressed) and reaction-sintered forms offers a great potential for use in hot-flow-path components in gas

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turbine engines (refs. 1 to 6). Although the strength properties of Si₃N₄ materials are
good, materials of higher strength are desired. For hot-pressed Si₃N₄ (HPSN), high-
temperature strength begins to be impaired above approximately 1200°C (2190°F) be-
cause of a glassy, silicate, grain boundary phase (ref. 7). This silicate phase is a con-
sequence of an addition of magnesium oxide (MgO) required for obtaining high density.
Reaction-sintered Si₃N₄ (RSSN) does not suffer from high-temperature-strength degra-
dation since it is essentially free of any glassy grain boundary phase. But RSSN, by
the nature of its fabrication process (nitriding of silicon powder compacts), is at pre-
sent limited to about 70 percent of theoretical density and is thus not as strong as HPSN.
However, the strength of RSSN is insensitive to increasing temperature, and indica-
tions are that 75-percent-dense RSSN is actually stronger than HPSN above approxi-
mately 1400°C (2550°F) (ref. 8). It follows that a material should have the high den-
sity and high strength of HPSN with the strength maintenance exhibited by RSSN.

In developing a material for quantity use, such as for the hot-flow-path components
of gas turbine engines, an economical production method is a major consideration.
Parts can be more easily fabricated to shape from RSSN than from HPSN. Slip casting
or injection molding is used for RSSN parts in comparison to the more expensive hot-
pressing procedure, which must be followed by costly diamond grinding of parts. Thus
RSSN, although not as strong as HPSN, is considered an attractive material for certain
turbine components.

With these considerations in mind, it is understandable that a paper by Jack and
Wilson (ref. 9) describing the excellent properties of pressureless-sintered Si₃N₄ base
materials of low porosity (viz 7 percent) evoked considerable interest. Jack and Wilson
describe a Si₃N₄ material of an expanded beta (β) Si₃N₄ crystallographic form, termed
β' (beta prime), which resulted from the solutioning of aluminum oxide (Al₂O₃) into
Si₃N₄ during sintering of Si₃N₄-Al₂O₃ powder mixtures. Such sintered bodies (termed
"SiAlON ceramics" by Jack and Wilson) were found to have a lower coefficient of ther-
mal expansion than HPSN, which is good from thermal shock considerations. And these
sintered bodies did indeed exhibit excellent thermal shock resistance. The sintered
SiAlON bodies were also found by Jack (ref. 10) to be more oxidation resistant than HPSN
in 1300°C (2370°F), 150-hour oxidation tests. He observed that perhaps the greatest
advantage of the SiAlON material over HPSN was its ease of fabrication. And he re-
lated this sinterability to the promotion of diffusion by vacant lattice sites. Very re-
cently, Drew and Lewis (ref. 11) have found metallographic evidence for liquid-phase
sintering of sintered and hot-pressed Si₃N₄-Al₂O₃ powder mixtures.

The interest generated in SiAlON materials by the Jack and Wilson paper (ref. 9)
has resulted in efforts by several research groups to consolidate SiAlON bodies (refs. 12
to 14). Arrol (ref. 12) confirms the superior oxidation resistance of SiAlON over HPSN
that is reported in reference 9. But he indicates that impurities are responsible for the
sintering ease of certain SiAlON's and that higher-density (purer) SiAlON's are not so easily sintered. As reported in reference 9, SiAlON's were found to be more easily hot pressed than HPSN; some SiAlON bodies exhibited superior creep resistance to HPSN at 1225° C (2240° F) and 69-MN/m² (10 000-psi) load. Lange (ref. 13) also found it difficult to obtain high-density SiAlON bodies by sintering but did obtain high-density SiAlON bodies by hot pressing. Contrary to Jack and Wilson (ref. 9), Lange found that his hot-pressed SiAlON of the same nominal composition as that of Jack and Wilson had a greater coefficient of thermal expansion than HPSN. Likewise, Crandall, Hed, and Shipley (ref. 14) found it difficult to sinter SiAlON, achieving at best only 80 percent of theoretical density. However, they did obtain pore-free compacts by hot pressing.

From this brief summary of the results obtained by Arrol (ref. 12), Lange (ref. 13), and Crandall (ref. 14), the high sinterability of SiAlON reported by Jack and Wilson (ref. 9) is seen to be problematical. In references 12 to 14, it was commonly concluded that the type and amount of second phases (some possibly liquid) occurring with the SiAlON (Si₃N₄-Al₂O₃ solid solution) major phase are very important, as are starting material purities and consolidation process conditions.

The study reported herein was conducted to provide some further insight into the mechanism of the formation and densification of SiAlON material. In this study, intensive stirred-ball-mill blending of starting powders assured excellent mixing and contact of Si₃N₄ and Al₂O₃ powders. In order to characterize the kinetics of reaction and densification, powder blends of various Si₃N₄:Al₂O₃ mole ratios were pressure sintered in vacuum at temperatures from 1100° to 1700° C (2010° to 3090° F). Press ram travel due to the application of a constant pressure (a measure of powder compaction) was recorded during heating and at equilibrium temperature. Powder compaction rates and end-point densities were correlated with X-ray diffraction and metallographic analyses to describe SiAlON formation and densification as a function of temperature and Si₃N₄:Al₂O₃ mole ratio.

MATERIALS

The starting materials were Plessey Co., Ltd., α-Si₃N₄ and Cabot Corp. AlON γ-Al₂O₃ powders. The Si₃N₄ powder was reported by the supplier to be 94 percent α-phase plus 6 percent β-phase. It was also determined at the NASA Lewis Research Center to contain 0.92 wt % oxygen and 0.22 wt % carbon by inert-gas fusion and induction heating - chromatographic finishing, respectively. The Si₃N₄ powder contained 50 to 260 ppm calcium, 800 to 1200 ppm aluminum, and 300 to 500 ppm iron as determined by semiquantitative spectrographic analyses also performed at Lewis. The Al₂O₃ powder was reported by the supplier to be essentially 100 percent γ-phase and to
have a metal impurity content of less than 0.1 percent. The morphology and size ranges of the as-received powders were characterized by transmission electron microscopy (TEM). Plessey α-Si₃N₄ consists of fibrous and flake-like crystallites (fig. 1(a)). The flakes range in size from 0.02 to 1.0 micrometer and the fibers from 0.04 to 0.3 micrometer with a wide variation in length. The γ-Al₂O₃ powder particles are round in shape (fig. 1(b)) and are positively charged (according to the supplier). Particle size ranges from 0.005 to 0.09 micrometer and is predominantly in the 0.01- to 0.02-micrometer range.

APPARATUS AND PROCEDURE

Each powder mixture (50-g batch weight) with the desired Si₃N₄:Al₂O₃ mole ratio was blended and ground at a controlled speed in a nickel stirred-ball mill with 3-millimeter-diameter steel grinding balls (ball-to-powder-weight ratio of 44:1) and a liquid solution of 3 percent alcohol in heptane. In order to control the amount of iron pickup to a relatively constant level, 0.4 to 0.8 wt %, the blending time was varied between 1 to 2 hours, depending on the Si₃N₄:Al₂O₃ mole ratio. It had been found in a preliminary experiment that for a given blending time the amount of iron pickup increased with the concentration of Si₃N₄ in the powder mixture. After blending, the powder mixture was dried in air and then in vacuum for at least 24 hours. Based on a 100-mol %-Si₃N₄ blending, oxygen pickup for the Si₃N₄-Al₂O₃ mixtures is estimated to range from about 1.6- to 2.5-wt % oxygen. A transmission electron microscope characterization of the 40Si₃N₄-60Al₂O₃ mixture after stirred-ball-mill blending is shown in figure 2. The very fine Al₂O₃ particles remain unchanged, but the rod-like Si₃N₄ particles have been broken. The large particles are probably agglomerates of Si₃N₄ particles with adhering Al₂O₃ particles.

A schematic of the vacuum induction furnace used for pressure sintering is shown in figure 3. The furnace, which is induction heated with a graphite susceptor, was run under a vacuum of 4 to 0.006 N/m². A port in the water-cooled manifold that is welded to the top plate connects the vacuum system to the furnace. The graphite susceptor, 15.6 centimeters (6 1/8 in.) in diameter and 40.6 centimeters (16 in.) long, and the carbon susceptor extension rest on the water-cooled bottom plate. The susceptor is positioned inside a 25.4-centimeter- (10-in.-) diameter, 81.3-centimeter- (32-in.-) long, fused-quartz tube. A water-cooled induction coil, powered by a 9600-hertz, 50-kilowatt motor-generator, is wrapped around the quartz tube. Annular grooves with silicone rubber gaskets are located in the water-cooled top and bottom plates. These grooves secure the quartz tube and tightly seal the vacuum chamber. The annular space between the susceptor assembly and the quartz tube is filled with carbon black insulation.
A graphite cloth backed by a segmented graphite ring prevents the carbon black insulation from being blown out during evacuation. Four thick tie rods between the top and bottom plates align the furnace and ensure rigid assembly. The graphite die body rests on a carbon block which clears the susceptor by 0.16 centimeter (1/16 in.). The carbon block rests on the water-cooled base. Above the die are two carbon radiation shields.

The rectangular die opening dimensions are 6.35 centimeters by 1.27 centimeters (2.5 in. by 0.5 in.). Approximately 48 grams of powder were packed in the die, which was coated with boron nitride. The dies packed with powder were first cold pressed by using a hydraulic hand press with 13.8 MN/m² (2000-psi) pressure. The die was then loaded into the hot press and 27.6 MN/m² (4000-psi) pressure was applied on the powder compact under vacuum. The 27.6 MN/m² (4000-psi) pressure was held during both the heating and temperature holding periods. Pressure was completely removed at the onset of cooling.

The sample (powder compact) temperature was measured by a thermocouple placed in a hole in the center of the die body, with the thermocouple hot junction 0.5 centimeter (0.20 in.) from the sample. Temperatures were also monitored with an optical pyrometer by sighting the top of the die through a glass prism mounted on the furnace cover. Temperatures could be generally maintained within 10° C (±18° F) of the desired holding temperature by controlling the power supply setting.

One of the main objectives of this study was to examine the compacting behavior of the powder compact during both the heating and temperature holding periods of a pressure sintering run. This was accomplished by monitoring the ram travel shown on a dial gage mounted on the ram with reference to the top of the press. Thus, ram travel, temperature, and time-elapsed data starting from the moment when furnace power was turned on were recorded every 5 minutes during each pressure sintering run.

In order to make meaningful analyses on ram travel - temperature - time data, a fixed heating schedule was followed for all pressure sintering runs in this study. It was accomplished by setting the furnace power input at a certain high rating and then letting the temperature rise naturally. The power input was subsequently reduced to a lower level when the sample temperature (temperature at the center of the die) approached the desired holding temperature. Temperature control by following this procedure was quite reproducible. Figure 4 shows a typical thermal history for a pressure sintering run to 1700° C (3090° F) with a hold at this temperature. For other 1700° C (3090° F) runs in this study, the maximum temperature deviation from this curve was generally ±10° C (±18° F). The same heating schedule was used for all pressure sintering runs.

In order to provide a common base for comparing the compaction behavior among pressure sintering runs, ram travel data from each run were subsequently converted to the transient relative density \( \rho_r(T,t) \) of the sample at the corresponding transient tem-
perature $T$ and elapsed time $t$. This conversion was accomplished by the procedures outlined in the following paragraph.

The transient relative density of a sample of a given composition is defined as

$$\rho_r(T, t) = \frac{\rho(T, t)}{\rho_m} \times 100$$

(1)

where $\rho(T, t)$ is the calculated transient density of the sample based on the ram travel datum at the transient temperature $T$ and the elapsed time $t$ and $\rho_m$ is a measured density. This density $\rho_m$ was obtained from a sample having the same composition as the one under investigation but pressure sintered at the most severe conditions used in this study (2 hr at 1700° C (3090° F) and 27.6 MN/m$^2$ (4000 psi)). These conditions rendered pore-free microstructures for all three compositions chosen for investigation in this study. Measured densities for these pore-free bodies are listed in Table I for the respective compositions. Furthermore, both $\rho(T, t)$ and $\rho_m$ can be expressed in terms of sample weight and sample dimensions:

$$\rho(T, t) = \frac{W}{A_x H_t}$$

(2)

$$\rho_m = \frac{W}{A_x H_f}$$

(3)

where $W$ is the sample weight, $A$ the cross-sectional area of the sample as defined by the die opening, $H_t$ the transient thickness of the sample, and $H_f$ the measured thickness (at room temperature) of the sample which had been pressure sintered under the most severe conditions and was pore free. The transient relative density $\rho_r$ is then, by substitution of equations (2) and (3) into equation (1), expressed as

$$\rho_r(T, t) = \frac{H_f}{H_t}$$

(4)

RESULTS AND DISCUSSION

Most pressure-sintering (hot pressing) studies on SiAlON materials to date have concerned the end-point density of the sample after a 1- to 2-hour heating at tempera-
tures of 1700° C (3090° F) or higher and pressures of 27.6 MN/m² (4000 psi) or higher. The holding temperatures and times used in hot-pressing SiAlON materials have been chosen somewhat arbitrarily based on the experiences gained from hot-pressing studies on Si₃N₄ with MgO as a sintering aid.

In order to provide a better understanding of the compaction behavior of SiAlON materials during pressure sintering, it was decided to investigate the density-temperature-time relation throughout the entire pressure-sintering period at 27.6 MN/m² (4000 psi) to 1700° C (3090° F) and also during a 2-hour hold. The density-time-temperature results were correlated with X-ray diffraction analyses and with metallographic analyses of the pressure-sintered samples by scanning electron, light, and transmission electron microscopy. Composition effects on compaction were included in this study by varying the Si₃N₄:Al₂O₃ mole ratio. Therefore, the compaction behavior of the Si₃N₄ and Al₂O₃ powder mixtures can be better understood on the basis of the observed solutioning, phase transformation, microstructure changes, and other physiochemical phenomena.

Relative Density - Transient Temperature Curves

It has been established that Si₃N₄ powders of commercial purity without additives do not densify at all under conventional pressure sintering (hot pressing) conditions (ref. 15). However, when Al₂O₃ powder is mixed with Si₃N₄ powder, full-density bodies (SiAlON) can be obtained by conventional hot-pressing methods (refs. 12 to 14). It is therefore clear that interaction between Al₂O₃ and Si₃N₄ powder particles in a mixed powder sample plays an important role in enhancing densification. This interaction effect was revealed by comparing the relative density - transient temperature (or time) curves of a series of pressure sintering runs under the same conditions but with increasing Al₂O₃ content in the powder mixtures, ranging from 100-percent Si₃N₄ to 100-percent Al₂O₃ powders.

Figure 5 shows the relative density - transient temperature curves for the heating period of pressure sintering runs of 100-percent Si₃N₄ and 100-percent Al₂O₃ as received from the suppliers. No significant densification was observed during the subsequent 2-hour hold at 1700° C (3090° F) in either run. Theoretical densities of Si₃N₄ and Al₂O₃ were used for \( \rho_m \) to obtain values of \( H_f \). These \( H_f \) values were used to calculate the relative densities \( \rho_F \) (by eq. (4)) that were used in plotting the curves of figure 5.

The 100-percent Si₃N₄ curve is essentially horizontal with a slightly negative slope, confirming the nondensifying nature of the material as pointed out previously. The slightly negative slope of the curve is attributed to the thermal expansion of the hot-
pressing system as temperature increased. Distinct densification stages were observed in the relative density - transient temperature curve of the 100-percent $\text{Al}_2\text{O}_3$ run. The initial stage in $\text{Al}_2\text{O}_3$ densification has been described as resulting from particle sliding, fragmentation, and plastic flow by dislocation motion, with particle sliding being the main contributor to densification (ref. 16). It is not known whether initial densification is enhanced by the transformation of $\gamma$-$\text{Al}_2\text{O}_3$ to $\alpha$-$\text{Al}_2\text{O}_3$, which occurs between 750° C (1390° F) and 1200° C (2190° F) (ref. 17). But Rice (ref. 18) and Gazza, Barfield, and Preas (ref. 19) report no appreciable differences in minimum hot-pressing parameters required for equivalent densification whether a $\gamma$-$\text{Al}_2\text{O}_3$ or an $\alpha$-$\text{Al}_2\text{O}_3$ powder is used as the starting material. A stage of inhibited densification for 100-percent $\text{Al}_2\text{O}_3$ from approximately 1200° to 1350° C (2190° to 2460° F) is indicated by the plateau region on that curve in figure 5. This behavior, also noted for our $\text{Si}_3\text{N}_4$-$\text{Al}_2\text{O}_3$ mixture, is similar to that reported by Rice (ref. 18). He found that inhibited densification coincided with heavy outgassing during an interval of the heating period in vacuum hot pressing. Above 1350° C (2460° F) our $\text{Al}_2\text{O}_3$ resumed densification, reaching a relative density of 88 percent at 1700° C (3090° F). As noted previously, little additional densification occurred during the subsequent 2-hour holding period. Failure to achieve higher relative density is believed to be due to trapped gaseous impurities, as has been discussed by Rice (ref. 18). These impurities, in quantity, are related to the surface area of the ultrafine $\gamma$-$\text{Al}_2\text{O}_3$ powder used. At conditions similar to our pressure sintering conditions, $\text{Al}_2\text{O}_3$ can be consolidated to close to theoretical density (ref. 20).

Three powder mixtures with decreasing $\text{Si}_3\text{N}_4$-$\text{Al}_2\text{O}_3$ mole ratios of 4:1, 3:2, and 2:3 (20-, 40-, and 60-mol % $\text{Al}_2\text{O}_3$) were chosen for study. These three powder mixtures are listed in table I, along with the composition and density values expressed in three different forms. Calculation of the complete solid solution ($\beta'$) densities given in table I was based on the SiAlON formula given by Jack and Wilson (ref. 9) and on lattice parameters determined for our pressure-sintered SiAlON compositions. The solubility of $\text{Al}_2\text{O}_3$ in $\text{Si}_3\text{N}_4$ at 1750° C (3180° F) has been reported to be 67 mol % (ref. 21). Therefore, in order to ensure reasonably complete solutioning, the upper limit of $\text{Al}_2\text{O}_3$ content in the mixtures chosen for study was set at 60 mol %.

Relative density - transient temperature curves for pressure sintering runs for the three mixtures, 100-percent $\text{Si}_3\text{N}_4$, and 100-percent $\text{Al}_2\text{O}_3$ during the heating period (from room temperature to 1700° C (3090° F)) are shown in figure 6. Figure 7 shows the relative density - time curves for the three mixtures in the remaining period (2-hr hold at 1700° C (3090° F)) of their pressure sintering runs. Light microscope examination made of the three pressure-sintered samples held at 1700° C (3090° F) showed a pore-free microstructure for all three. As previously mentioned for the pressure-sintered 100-percent-$\text{Si}_3\text{N}_4$ and 100-percent-$\text{Al}_2\text{O}_3$ runs, measured density $\rho_m$ values
(table I) for these three pressure-sintered samples were used in calculating relative densities, based upon light microscope examination of the samples showing them to be pore free.

It might be expected that the major densification in hot pressing SiAlON-forming powder mixtures would occur during the holding period at the 1700°C (3090°F) and above temperatures which have been used. However, we found that the major amount of densification in all three mixtures (fig. 6) took place during the heating period. More significantly, on all three curves shown in figure 6, 90 percent or more of the total amount of densification (final density minus the initial as-cold-pressed density) was accomplished in the temperature range 1400°C to 1700°C (2550°F to 3090°F), which constitutes the last 30 minutes of the heating period. This 30-minute time span was obtained from figure 4 by noting the time difference between 1400°C and 1700°C (2550°F and 3090°F) on the heating schedule curve. The 2-hour hold at 1700°C (3090°F) following the heating period resulted in a density increase of only 2 percent or less of the final density, from a relative density of 98 to 100 percent, in the two lower-Al₂O₃-content samples and no increase in the third sample. Therefore, it is clear that at temperatures of 1700°C (3090°F) or higher, it is not necessary to employ a hold time for 1 to 2 hours in pressure sintering to produce dense SiAlON bodies, as has been the practice in the field.

A closer examination of the relative density - temperature curves in figure 6 reveals that, as the Al₂O₃ content in the sample increases, the curve more closely resembles the 100-percent-Al₂O₃ curve. On the 80Si₃N₄-20Al₂O₃ curve, the sample essentially does not densify until the temperature reaches 1450°C (2640°F), above which temperature a high rate of densification occurs. On the 60Si₃N₄-40Al₂O₃ curve, an initial rise in relative density occurs, beginning at about 1000°C (1830°F) and ending at about 1250°C (2280°F). This initial density rise is followed by an arrest in the temperature range 1250°C to 1400°C (2280°F to 2550°F) before the fast rise in relative density begins. On the 40Si₃N₄-60Al₂O₃ curve, the initial rise in relative density begins at 1000°C (1830°F) and is followed by an arrest from 1200°C (2190°F) to about 1300°C (2370°F) before the major increase in density begins. As for the 100-percent-Al₂O₃ curve, the initial rise in relative density between about 900°C and 1200°C (1650°F and 2190°F) is followed by an arrest before another rise in density begins at about 1350°C (2460°F).

The pressure-sintered Si₃N₄-Al₂O₃ samples which have been discussed were initially mechanical mixtures of Si₃N₄ particles and Al₂O₃ particles before interaction occurred between them. It is reasonable to assume that in a mechanical mixture of different powders the contribution of each powder constituent to the way the mixture compacts is related to the volume percentage of each particular powder constituent present in the mixture. As stated previously, we observed that as the amount of Al₂O₃ powder
in the Si₃N₄-Al₂O₃ powder mixtures increased, the resultant relative density - transient temperature curves (fig. 6) became more similar in form to the 100-percent-Al₂O₃ curve. Initially, the characteristic features of Al₂O₃ powder compaction were only slightly reflected in the 80-mol %-Si₃N₄ - 20-mol %-Al₂O₃ mixture, which contained 12.3-vol % Al₂O₃ (table I). However, the occurrence of enhanced densification in the 80Si₃N₄-20Al₂O₃ mixture in comparison to 100-percent Al₂O₃ and 100-percent Si₃N₄ is evident beginning at 1400°C (2550°F) (fig. 6). At this temperature there is a fast and sustained rise in the relative density - transient temperature curve for the 80Si₃N₄-20Al₂O₃ material. As the volume percentage of Al₂O₃ increases to 41.8 percent in the 40Si₃N₄-60Al₂O₃ mixture (table I), the Al₂O₃ particles, being much finer both before and after stirred-ball-mill mixing with the coarser Si₃N₄ (fig. 2), are able to surround the larger Si₃N₄ particles. This explains why the characteristic compaction behavior of Al₂O₃ is clearly seen for the 40Si₃N₄-60Al₂O₃ material. This material in the 1300°C to 1700°C (2370°F to 3090°F) temperature range also has a greater relative density than the 100-percent-Al₂O₃ sample - again indicating the enhanced densification. X-ray analysis results (discussed in the next section) shed more light on the type of reactions taking place in each temperature range during pressure sintering.

Densification at Holding Temperatures Below 1700°C

It has been stated in the preceding discussion that it might not be necessary to employ temperatures as high as 1700°C (3090°F) to produce dense SiAlON bodies by pressure sintering and also that a very active densification mechanism begins to operate in the temperature range 1350°C to 1700°C (2460°F to 3090°F). Therefore, in an attempt to provide more information regarding the compaction behavior of Si₃N₄-Al₂O₃ powder mixtures at temperatures below 1700°C (3090°F), a series of pressure sintering experiments with lower holding temperatures were made on samples with 40Si₃N₄-60Al₂O₃ composition. In each such pressure sintering run, the heating schedule shown in figure 4 was followed to the desired holding temperature and then a 2-hour hold at that temperature was made.

The transient relative density of the sample throughout the entire pressure sintering run was calculated for each experiment following the procedures outlined in equations (1) to (4). For each lower-temperature pressure sintering run, the relative density - temperature data in the heating period closely reproduced the 40Si₃N₄-60Al₂O₃ curve in figure 6 up to the desired holding temperature level. During the subsequent 2-hour holding period the sample might or might not increase its relative density depending on the level of the holding temperature. For a particular holding temperature, the relative density of a sample at the end of a 2-hour holding period minus its relative
density at the onset of the 2-hour hold is termed the "net change in relative density" during that 2-hour period. In figure 8, the net change in relative density of a sample is plotted against the corresponding holding temperature. A strong time- and temperature-dependent densification mechanism is clearly operative from approximately 1350° to 1700° C (2460° to 3090° F). Below this temperature range, the sample essentially did not increase its density during a 2-hour hold. Here a less time-dependent mechanism caused the sample to densify to a certain amount depending on temperature alone. From light microscope examination, samples with holding temperatures of 1500° C (2730° F) and higher were essentially pore free. Thus, the "net change in relative density" was slight at holding temperatures above 1550° C (2820° F) because the sample had attained a high relative density during the heating period. And so the possibility of further densification in the holding period was negligible. This is why at 1700° C (3090° F) the net change in relative density was zero. The high mobility in the Si₃N₄-Al₂O₃ system above about 1350° C (2460° F) suggests its utilization for mechanical forming such as forging and extrusion.

X-Ray Diffraction Analysis

The relative density results presented in the preceding sections indicate that densification of a sample in the temperature range 1350° to 1700° C (2460° to 3090° F) possibly involves one or more time- and temperature-dependent (thermally activated) phenomena, such as diffusion, solutioning, and phase transformation. One effective method to reveal this information is X-ray diffraction. Experimental limitations prohibited direct X-ray examination of the sample during the pressure sintering experiments. In this study, all X-ray diffraction examinations were made on samples at room temperature after the pressure sintering run was completed. Helium gas was used to quench the sample at the end of pressure sintering to prevent further reaction taking place in the sample. Relative X-ray peak intensities were used to indicate the change in the relative amount of the phases present in the sample. The expansion in lattice parameters of a phase is interpreted as solid-solution formation.

X-ray results from samples of 40Si₃N₄-60Al₂O₃ that had been pressure sintered at different holding temperatures (2-hr hold) are summarized in figure 9. Since we are interested only in the trend of the change in the relative amount of each phase present in the sample, a semiquantitative approach was used in presenting the relative density data. The 40Si₃N₄-60Al₂O₃ composition is nearly 50 wt % Al₂O₃ and 50 wt % Si₃N₄ (table I). It is therefore a reasonable approximation to assign 0.5 as the reference relative X-ray peak intensity for Al₂O₃ and also for Si₃N₄ in the starting powder mixture before any reaction between Al₂O₃ and Si₃N₄ occurs. According to the suppliers,
Al₂O₃ powders are predominantly γ-Al₂O₃, while Si₃N₄ powders are composed of 94-percent α-Si₃N₄ and 6-percent β-Si₃N₄. Thus, the 0.5 relative X-ray peak intensity value assigned for Si₃N₄ is divided into 0.47 for α-Si₃N₄ and 0.03 for β-Si₃N₄.

In figure 9 the relative X-ray peak intensity of each phase present in the sample at the end of a pressure sintering experiment is plotted against the corresponding holding temperature. Reference relative X-ray peak intensity values of 0.5, 0.47, and 0.03 were used for γ-Al₂O₃, α-Si₃N₄, and β-Si₃N₄, respectively. Data from the two lowest-temperature pressure sintering runs (1200° and 1300° C; 2190° and 2370° F) showed no interaction between Si₃N₄ and Al₂O₃. The relative amounts of α-Si₃N₄ and β-Si₃N₄ remained unchanged, but almost all γ-Al₂O₃ had transformed to α-Al₂O₃ at 1200° C (2190° F) and no γ-Al₂O₃ was detectable at 1300° C (2370° F). The plotted variation in the relative X-ray peak intensities for γ-Al₂O₃ and α-Al₂O₃ between 750° and 1200° C (1380° and 2190° F) shown in figure 9 is based on the reported temperature range for γ-Al₂O₃ to α-Al₂O₃ transformation (ref. 17). Between 1300° and 1450° C (2370° and 2640° F), where α-Al₂O₃ and α-Si₃N₄ are the major phases and β-Si₃N₄ is present first as a trace amount, mutual solid solutioning between Si₃N₄ and Al₂O₃ is taking place. This is indicated by the double lines in figure 9. The β-Si₃N₄ phase begins to increase above 1300° C (2370° F) with the double lines denoting solutioning of Al₂O₃ in Si₃N₄ and formation of an expanded β-Si₃N₄ crystal structure termed β' (beta prime) by Jack and Wilson (ref. 9). The increase in the β' phase starting above approximately 1300° C (2370° F) corresponds to a resumption of densification, as was noted for 40Si₃N₄-60Al₂O₃ in figure 6.

As shown in figure 9, at 1550° C (2820° F) a trace amount of a phase hereinafter referred to as X-phase was found. This phase was first found by Oyama and Kamigaito in hot-pressed Si₃N₄-Al₂O₃ powder compacts and is characterized as to interplanar spacings and relative intensities in reference 21. In subsequent work on Si₃N₄-Al₂O₃-Ga₂O₃ solid solutions (refs. 22 and 23), Oyama found this phase again and referred to it as X-phase. In the work reported herein, the X-phase increased in relative amount as the pressure sintering temperature increased to 1700° C (3090° F), the maximum temperature used in this study. X-phase may enhance the densification process by liquid-phase sintering. Drew and Lewis (ref. 11) present evidence for X-phase having been a liquid in sintering and hot-pressing studies to 1800° C (3270° F). And, indeed, our metallographic results (discussed in the next section) also give evidence for the presence of a liquid phase. Drew and Lewis (ref. 11) found that qualitatively the amount of surface SiO₂ associated with the α-Si₃N₄ powder determines the amount of X-phase formed. Previously, Oyama and Kamigaito (ref. 21) had found that additions of SiO₂ to a Si₃N₄-Al₂O₃ powder mix which was subsequently hot pressed resulted in an increased quantity of X-phase. In our studies, X-ray examinations were also made on 80Si₃N₄-20Al₂O₃ and 60Si₃N₄-40Al₂O₃ samples which had been pressure sintered at 1700° C.
(3090° F) for 2 hours. The results, in comparison with the X-ray results from the 40Si3N4-60Al2O3 sample already discussed, show that the relative amount of X-phase increases with increasing Al2O3 content. Such a relation between Al2O3 and X-phase in hot-pressed Si3N4-Al2O3 compositions is reported by Oyama and Kamigaito (ref. 21).

We did not detect α-Si3N4 or α-Al2O3 at 1650° or 1700° C (3000° or 3090° F), with only β1, the major phase, and X-phase being found in the samples.

Metallographic Examination

Light microscope examination of samples of Si3N4-Al2O3 compositions (in mol %) of 80-20, 60-40, and 40-60 which had been pressure sintered at 1700° C (3090° F) and 27.6 MN/m² (4000 psi) for 2 hours showed pore-free structures for all three. Figure 10 shows the 40Si3N4-60Al2O3 microstructure, which is made up of a gray-matrix major phase, a darker uniformly dispersed minor phase, and a small amount of a bright, fine particle phase. The bright phase was identified as iron by scanning electron microscope (SEM) energy dispersion X-ray analysis. As mentioned previously, a small amount of iron, 0.4 to 0.8 wt %, was picked up from the steel grinding medium during the stirred-ball-mill blending of the Si3N4-Al2O3 powder mixtures. The darker minor phase in figure 10 was most prevalent for this highest-Al2O3-content sample and is believed to be the X-phase. The quantity of the darker minor phase was least in the 80Si3N4-20Al2O3 microstructure and somewhat greater in the 60Si3N4-40Al2O3 sample. These observations correlate with the X-ray results that indicated higher X-phase contents for higher Al2O3 levels. The gray-matrix major phase in figure 10 is the β1 matrix.

More detailed microstructures of the three pressure-sintered compositions of Si3N4-Al2O3 (80-20, 60-40, and 40-60 mol %) were revealed by the transmission electron microscope (TEM) technique, as shown in figure 11. The three compositions at the same magnification (27 000) are shown in figure 11(a). Some of the rod-shaped Si3N4 grains which made up part of the Si3N4 powder morphology can be seen in figures 11(a-1) and (b-1) for the lowest Al2O3 concentration. At the higher Al2O3 levels (figs. 11(a-2) and (a-3)) no such grains are found. The grain size increased with Al2O3 content, and the overall grain morphology is equiaxed with predominantly faceted grain boundaries. Even in the sample with the highest Al2O3 content, there is no indication of retention of the original spherical particle morphology of the Al2O3. A striking feature in figure 11 is the striations in some grains (figs. 11(a-3) and (b-2), in particular). These grains generally have a rounded boundary, suggesting the presence of liquid at one time. The number of striated grains increases with increasing Al2O3 content. Grains with striations have been identified as X-phase by Drew and Lewis.
through transmission electron diffraction. The striations were interpreted by Drew and Lewis as twin and antiphase boundaries "believed to form as growth accidents either on solidification of the residual liquid of X-phase composition or via an order-disorder transformation during post solidification cooling" (ref. 11).

The drastic difference in particle morphology and size between the as-mixed powders and the pressure-sintered samples would suggest that active recrystallization and growth occurred during pressure sintering, probably resulting from a combination of solid-state diffusion and solid/liquid interaction (solution reprecipitation) phenomena. Jack and Wilson (ref. 9) proposed that solid-state diffusion in the $\text{Si}_3\text{N}_4$–$\text{Al}_2\text{O}_3$ system was enhanced by the increased vacancy concentration that resulted from the solutioning of $\text{Al}_2\text{O}_3$ (Al and O ions) into the $\text{Si}_3\text{N}_4$ lattice in $\beta$' solid-solution formation. Thus, the diffusion rate in the material increases with the $\text{Al}_2\text{O}_3$ concentration. This solid-solutioning phenomenon occurs at temperatures above 1300°C (2370°F), as shown in the X-ray results in the section X-Ray Diffraction Analysis. The solid/liquid interaction was postulated by several workers and has recently been more convincingly demonstrated by Drew and Lewis (ref. 11). As pointed out earlier, the amount of X-phase, the liquid phase at high temperature, increases with the $\text{Al}_2\text{O}_3$ content in the sample. Therefore, in accordance with these two proposed phenomena (models), it is easy to understand why the average grain size in samples (fig. 11) that have been pressure sintered under identical conditions increases with the $\text{Al}_2\text{O}_3$ content in the sample.

The Question of Oxygen and $\text{SiO}_2$ Contents

In our discussion of X-ray results the effect of $\text{SiO}_2$ on the quantity of X-phase formed was acknowledged by reference to the work of Drew and Lewis (ref. 11) and Oyama and Kamigaito (ref. 21). And Lange (ref. 13) has indicated that the effect of $\text{SiO}_2$ on the solutioning of $\text{Al}_2\text{O}_3$ in $\text{Si}_3\text{N}_4$ warrants further investigation. Since it is generally felt that the most desirable SiAlON material would be one free of second phases such as X-phase, the following information regarding the $\text{SiO}_2$ content of our materials is provided.

The oxygen content of the as-received Plessey $\alpha$-$\text{Si}_3\text{N}_4$ was found by inert gas fusion to be 0.92 wt %. It has been postulated that oxygen stabilizes the $\alpha$-$\text{Si}_3\text{N}_4$ structure when there is a minimum oxygen level of 0.90 wt %. A more likely oxygen level is 1.48 wt % (ref. 24). However, more recently, $\alpha$-$\text{Si}_3\text{N}_4$ prepared by chemical vapor deposition was found to have an oxygen content of only 0.30 wt % (ref. 25). It has not been established just what minimum oxygen content in $\alpha$-$\text{Si}_3\text{N}_4$ can be achieved through atmosphere control during production of $\text{Si}_3\text{N}_4$. Assuming the Plessey $\alpha$-$\text{Si}_3\text{N}_4$ to have at least 0.30-wt % oxygen in solution would leave 0.62-wt % oxygen present in $\text{SiO}_2$. This
amount of SiO\textsubscript{2} was calculated to be 1.16 wt \%, or 1.68 vol \%. Oxygen pickup due to the 2-hour, stirred-ball-mill blending of the Si\textsubscript{3}N\textsubscript{4}-Al\textsubscript{2}O\textsubscript{3} compositions was estimated at 2.49, 2.10, and 1.65 wt \%, respectively, for the 80-20, 60-40, and 40-60 mol \% Si\textsubscript{3}N\textsubscript{4}-Al\textsubscript{2}O\textsubscript{3} compositions. These estimates were based on an oxygen analysis by inert gas fusion on a 100-percent-Si\textsubscript{3}N\textsubscript{4} powder batch that was mixed in the same manner as the Si\textsubscript{3}N\textsubscript{4}-Al\textsubscript{2}O\textsubscript{3} compositions. This oxygen pickup is believed to reflect oxidation of Si\textsubscript{3}N\textsubscript{4} and the formation of an SiO\textsubscript{2} skin on the fine Si\textsubscript{3}N\textsubscript{4} particles. The oxygen pickups correspond to SiO\textsubscript{2} volume percentages of 6.64, 5.59, and 4.42. Adding these values to the calculated amount of SiO\textsubscript{2} in the starting Si\textsubscript{3}N\textsubscript{4} powder (assuming partitioning of the oxygen as discussed) then gives SiO\textsubscript{2} volume percentages of 8.32, 7.27, and 6.10, respectively, for the 80-20, 60-40, and 40-60 mol \% Si\textsubscript{3}N\textsubscript{4}-Al\textsubscript{2}O\textsubscript{3} compositions. Based on these percentages, it might be reasoned that the highest-SiO\textsubscript{2}-content mixture, 80Si\textsubscript{3}N\textsubscript{4}-20Al\textsubscript{2}O\textsubscript{3} would have the most X-phase after pressure sintering. However, this was not the case; this pressure-sintered composition had the least X-phase. This indicates an Al\textsubscript{2}O\textsubscript{3} concentration dependence, as we noted and which was formerly found by Oyama and Kamigaito (ref. 21). It is apparent that the control, or hopefully the elimination of X-phase in SiAlON, requires further work.

CONCLUDING REMARKS

In this experimental study of the Si\textsubscript{3}N\textsubscript{4}-Al\textsubscript{2}O\textsubscript{3} (SiAlON) powder compacts under pressure-sintering conditions, a better understanding of the densification phenomenon was obtained by characterizing the solutioning and phase transformation phenomena in the material system and the microstructure of the pressure-sintered specimens. The new information generated can be used as a guide to better processing procedures for fabricating dense SiAlON bodies with controlled microstructures. The important findings from this study can be summarized as follows:

1. The densification behavior of Al\textsubscript{2}O\textsubscript{3}-Si\textsubscript{3}N\textsubscript{4} powder compacts is dependent upon the solutioning and phase transformation phenomena in the material system. Solutioning and phase transformation were found to be very active in the temperature range 1400\textdegree C to 1700\textdegree C (2550\textdegree to 3090\textdegree F), with a maximum at 1500\textdegree C (2730\textdegree F).

2. Fully dense bodies can be obtained at temperatures as low as 1500\textdegree C (2730\textdegree F) at 27.6 MN/m\textsuperscript{2} (4000 psi) for 2 hours with no X-ray indication of X-phase. The lower hot-pressing temperature is more economical and the resulting microstructure (with a minimum amount of second phase) is potentially a better material.

3. A 100-percent-dense body can be obtained by heating to 1700\textdegree C (3090\textdegree F) at 27.6 MN/m\textsuperscript{2} (4000 psi) without a holding time. The grain size of the dense body so produced should be much finer and therefore have higher mechanical strength than one pro-
duced by holding at 1700° C (3090° F) under pressure for 1 to 2 hours.

4. Because of the high mobility of the material system in the temperature range ~1350° to 1700° C (~2460° to 3090° F) (high rate of solutioning and phase transformation), it may be possible in this temperature range to mechanically form (similar to forging; extrusion, etc.) a dense SiAlON body into a desired shape.

5. New pressureless sintering procedures could also be based on the results of this study.

SUMMARY OF RESULTS

In this investigation, a pressure-sintering technique was employed to investigate solution formation and densification mechanisms of blends of silicon nitride (Si₃N₄) and aluminum oxide (Al₂O₃) powders. Four aspects of the problem were examined: (1) the effect of Si₃N₄:Al₂O₃ mole ratio on densification behavior, (2) the variation of the relative density of the powder compact with temperature and time during pressure sintering, (3) X-ray phase analysis of bodies that had been pressure sintered at 27.6 MN/m² (4000 psi) and various temperatures for 2 hours, and (4) metallographic analysis of the pressure-sintered bodies.

It is well to qualify this summary by restating that our Si₃N₄-Al₂O₃ mixtures that were converted to SiAlON bodies by pressure sintering did contain considerable amounts of silicon dioxide (SiO₂) (calculated based on oxygen analyses). For example, 6.10-mol % SiO₂ was found in the 40-mol %-Si₃N₄ - 60-mol % Al₂O₃ mixture. Silicon dioxide is thought to influence the densification behavior of SiAlON.

The results from these experimental studies are summarized as follows:

1. Essentially pore-free SiAlON bodies were obtained for all three blends (mole ratios of 4:1, 3:2, and 2:3) of Si₃N₄ and Al₂O₃ powders after pressure sintering at 27.6 MN/m² (4000 psi) and 1700° C (3090° F) for 2 hours. These dense bodies consist of mainly β' (SiAlON) solid solution with a minor amount of a particular second phase, termed X-phase, first found by Oyama and Kamigaito. Under the same pressure-sintering conditions, Si₃N₄ powder virtually did not compact, and Al₂O₃ powder densified to only 88 percent of theoretical density.

2. The higher the Al₂O₃ content in a SiAlON in the range 20 to 50 mol %, the higher was the densification rate and the more nearly it reflected the characteristic densification behavior of Al₂O₃.

3. Metallographic results showed that
   a. No spherical grains typical of the Al₂O₃ starting powder remained after any SiAlON formation, regardless of Al₂O₃ content, thus indicating complete Al₂O₃ solutioning.
b. Rod-shaped Si₃N₄ grains, which made up part of the starting Si₃N₄ powder morphology, could still be seen in the 20-mol % Al₂O₃ SiAlON. But at higher Al₂O₃ contents, these rod-shaped Si₃N₄ grains had recrystallized by solutioning and phase transformation.

c. As the Al₂O₃ content of the SiAlON was increased, the equiaxed grain size increased and the amount of X-phase (apparently once liquid) increased. It is suggested that grain growth resulted from a solution-precipitation model (as proposed by Drew and Lewis) and from vacancy promoted diffusion (as proposed by Jack).

4. For the 60-mol % Al₂O₃ SiAlON, which was selected for more extensive study, it was found

a. That densification to 1300⁰C (2370⁰F) was due to the characteristic compaction behavior of Al₂O₃, involving particle sliding

b. That densification above 1400⁰C (2550⁰F) also reflected Al₂O₃ compaction behavior but was hastened and sustained by mutual solutioning between Si₃N₄ and Al₂O₃ and the formation of β'

c. That densification above 1500⁰C (2730⁰F) was aided by the formation of a liquid phase (X-phase) with solid/liquid interaction

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, July 9, 1975,
505-01.

REFERENCES


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<th>( \text{Si}_3\text{N}_4: \text{Al}_2\text{O}_3 ) mole ratio</th>
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\( a \) Bulk density.
Figure 1. - Transmission electron micrographs showing the as-received Si$_3$N$_4$ and Al$_2$O$_3$ powder morphologies and typical particle sizes.
Figure 2. - Transmission electron micrograph showing $40\text{Si}_3\text{N}_4$-$60\text{Al}_2\text{O}_3$ powder mixture after stirred-ball-mill blending.
Figure 3. - Schematic of vacuum induction furnace used for pressure sintering.

Figure 4. - Heating schedule of a typical pressure sintering run.
Figure 5. - Relative densities of 100-percent-Si$_3$N$_4$ and 100-percent-Al$_2$O$_3$ powder compacts as a function of transient temperature during heating period of pressure sintering run.

Figure 6. - Relative densities of 100-percent-Si$_3$N$_4$, 100-percent-Al$_2$O$_3$, and Si$_3$N$_4$-Al$_2$O$_3$ powder compacts as a function of transient temperature during heating period of pressure sintering run.
Figure 7. - Relative densities of Si$_3$N$_4$-Al$_2$O$_3$ powder compacts as a function of time held at 1700°C (3090°F) and 27.6 MN/m$^2$ (4000 psi).

Figure 8. - Net change in relative densities of 40-mol % Si$_3$N$_4$ - 60-mol % Al$_2$O$_3$ powder compacts during 2-hour holding periods at temperatures to 1700°C (3090°F).

Figure 9. - Relative X-ray peak intensities for phases present in 40-mol % Si$_3$N$_4$ - 60-mol % Al$_2$O$_3$ powder compacts after pressure sintering followed by 2-hour holding period at temperature to 1700°C (3090°F).
Figure 10. - Microstructure of 40Si$_3$N$_4$-60Al$_2$O$_3$ powder compact after pressure sintering at 1700°F (3090°F) and 27.6 MN/m$^2$ (4000 psi) for 2 hours.
Figure 11. Transmission electron micrographs of Si$_3$N$_4$-Al$_2$O$_3$ powder compacts (in mol%) after pressure sintering at 1700° C (3090° F) and 27.6 MN/m$^2$ (4000 psi) for 2 hours.
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—National Aeronautics and Space Act of 1958

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