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Influence of yttria-alumina surface coating on the colloidal processing of silicon nitride slips

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Abstract

Silicon nitride (Si_3N_4) powders coated with 6 wt.% Y_2O_3 and 4 wt.% Al_2O_3 were prepared by coprecipitation. For comparison, the Si_3N_4 powder containing the same amount of additives was also prepared by mechanical mixing. The resulting powders were dispersed in water with ammonium polyacrylate (NH_4PA) to produce 32 vol.% slips. The influence of pH on the dispersion properties of 32 vol.% coated Si_3N_4 slips with NH_4PA were investigated and compared with those of the mixed Si_3N_4 slips. The microstructure and density of green cast samples were determined and related to the degree of slip dispersion. In addition, the influence of the surface coating of Si_3N_4 on the sintering behavior was investigated. Lower viscosity values of the coated Si_3N_4 slips were found in the pH range studied. The better slip dispersion of the coated Si_3N_4 slips resulted in higher green density values. A linear correlation between the sintered density and the green density was found. The densification and the α- to -β phase transformation of Si_3N_4 were not affected and enhanced, respectively, with the surface coating of Si_3N_4 particles with sintering aids.

Keywords: A. Sintering; D. Si_3N_4; D. Y_2O_3; D. Al_2O_3; Dispersion properties; Green density

1. Introduction

Silicon nitride is an important structural material, because of its favorable mechanical performance at both ambient and elevated temperatures. The densification of a covalent-bonded compound such as Si_3N_4 can be enhanced by using oxide additives (Y_2O_3 and Al_2O_3) which promote sintering via a transient liquid phase.

Silicon nitride ceramics are conventionally prepared by mechanical mixing of Si_3N_4 powder and sintering additives [1]. However, this processing route does not guarantee a homogeneous distribution of the additives. An alternative method involves the coating of the Si_3N_4 particles with yttrium and aluminium hydroxides. The coated hydroxides layer can be transformed to yttria and alumina, respectively, by heating. In this work, Si_3N_4 powders coated with 6 wt.% Y_2O_3 and 4 wt.% Al_2O_3 were prepared by coprecipitation. For comparison, the Si_3N_4 powder containing the same amount of additives was also prepared by mechanical mixing.

The application of Si_3N_4 materials is still limited, mainly because of its low reliability and high machining costs [2]. An approach to overcoming these problems is to prepare homogeneous and near-net-shape green bodies through colloidal forming processes, such as slip casting. One of the key steps of this process is the preparation of a concentrated suspension with a high degree of stability. The Al_2O_3 powder is nearly insoluble between pH 4 and 10.5. However, the preparation of stable suspensions of the powders is strongly limited by the solubility of Y_2O_3, which is rather high [3]. Only between pH 9 and 10.5 is the solubility low enough for the preparation of stable suspensions [3]. The addition of base to achieve those pH values does not allow to reach a high solid concentration without affecting significantly the colloidal stability of the suspensions. For this reason a polyelectrolyte additive such as NH_4PA was chosen as a dispersant. This additive will adsorb at the particle surface and generates repulsive interaction caused by electrostatic and steric effects [4].

The study of the metal hydroxide precipitation reactions which provides a homogeneous distribution of sintering additives around Si_3N_4 particles has been reported [2,5,6]. However, the potential improvement in

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the dispersion properties and sintering behavior by using a coated Si_3N_4 powder instead of a mixed Si_3N_4 powder in the slip casting process has not been clearly demonstrated. Therefore, in this study, the influence of pH on the dispersion properties of 32 vol.% coated Si_3N_4 slips with NH_4PA were investigated and compared with those of the mixed Si_3N_4 slips. The microstructure and density of green cast samples prepared from coated and mixed Si_3N_4 slips were studied and related to the degree of slip dispersion. Finally, the influence of the surface coating of Si_3N_4 on the sintering behavior was investigated.

2. Experimental procedure

2.1. Materials

A commercial α-Si_3N_4 powder (SN-E10, UBE Industries, Japan) was used in this study. The mean particle diameter and the specific surface area were 0.6 μm and 10 m^2/g, respectively. Y_2O_3 (Molycorp, USA) and Al_2O_3 (A-16 SG, Alcoa Chemicals, USA) powders were used as sintering aids.

The $Y(OH)_3$ and $Al(OH)_3$ precursor solutions were 0.1 M $Y(NO_3)_3.5H_2O$ solution and Al_{13} polymer solution (0.02 M Al concentration), respectively. These solutions were mixed under magnetic stirring in a N_2 atmosphere. The resulting coating solution was used to coat the Si_3N_4 particles.

2.2. Powder preparation

The Si_3N_4 powder was dispersed in deionized water at about pH 10 to form a 9.4 vol.% (25 wt.%) slurry and ultrasonicated for 10 min to break down agglomerates. Then, 25 ml of the coating solution was added while the slurry was stirred, and the pH adjusted at 9 by additions of NH_3 (25%). This process was repeated several times to produce Si_3N_4 particles coated with 6 wt.% Y_2O_3 and 4 wt.% Al_2O_3 (equivalent concentration determined by assuming complete exhaustion of the Y^{+3} and Al^{+3} contents of the coating solution). The pH 9 was maintained during the additions. The suspension of the coated particles was centrifuged. The solid was then recovered, washed with distilled water and dried at 100 °C. The dried powder was finally calcined at 500 °C for 2 h in a N_2 atmosphere.

For comparison, the Si_3N_4 powder containing the same amount of additives was also prepared by mechanical mixing.

2.3. Powder characterization

The specific surface area of the coated and mixed Si_3N_4 powders was measured by single-point BET (Micromeritics Accusorb).

The $Y(OH)_3$ precipitated from the $Y(NO_3)_3.5H_2O$ solution and then calcined at 500 °C yielded an Y_2O_3 which was characterized by electrokinetic sonic amplitude (ESA) measurements. Zeta potential values against pH of Y_2O_3, Al_2O_3 (Al-160SG-1), as-received and coated Si_3N_4 suspensions with 1 vol.% solids loading were obtained with ESA measurements using an electroacustic equipment (Model ESA-8000, Matec Applied Science, Hopkinton, MA). For coated Si_3N_4 slips, the ESA measurements were conducted from pH 7.5 to 11.

2.4. Slip preparation

Slips with a solid loading of 32 vol.% (60 wt.%) were prepared. A commercial ammonium polyacrylate solution (Dolapix CE 64, Zschimmer & Schwarz) was used as the deflocculant. The pH of the suspension was adjusted with ammonia (25%).

We have previously determined the optimum concentration of NH_4PA for the mixed and coated powders by viscosity measurement for different amounts of NH_4PA solution added at pH 9.2. The minimum viscosity occurred at 0.12 and 2.3 wt.% NH_4PA for the mixed and coated powders, respectively.

Aqueous slips were prepared by deagglomeration of the mixed and coated powders in an attrition mill using 1.6 mm diameter alumina balls at different pH values in a range of 9.2–10.5 with 0.12 and 2.3 wt.% NH_4PA, respectively. First distilled water with the pH adjusted at a desired value was added to the attrition mill, then the dispersant and finally the powder were added. The powder was added in portions to the suspensions. The milling time was 40 min; by this time the whole amount of powder was present in the suspension.

The pH was manually adjusted to be maintained at a desired value and the suspension was allowed to equilibrate for 30 s before the pH was measured.

2.5. Rheological measurements

Steady state flow curves of coated and mixed Si_3N_4 slips were performed by measuring the steady shear stress value as a function of shear rate in the range of 0.5–541 s^{-1} using a concentric cylinder viscometer (Haake VT550, Germany) at 25 °C. As soon as stationary conditions were reached at each shear rate, the shear rate increased in steps up to the maximum value and then decreased. The majority of the curves did not show hysteresis area.

2.6. Slip casting and characterization of green samples

Slips were cast in plaster molds into disks of diameter 2.5 cm. The consolidated disks were dried slowly in air for 24 h at room temperature and 24 h at 100 °C.

The density of the green compacts was determined by the Archimedes method using mercury displacement. The pore size distribution of green samples was measured using mercury porosimetry (Porosimeter 2000 Carlo Erba, Italy).

2.7. Sintering and characterization of sintered samples

Green samples were embedded in a powder bed, consisting of 50 wt.% Si_3N_4, 45 wt.% BN, and 5 wt.% sintering additives, and were sintered in a N_2 atmosphere of 0.1 MPa at 1650 °C for 4 h using heating rates of 2 °C/min up to 800 °C and 5 °C/min up to 1650 °C. The N_2 flow rate was 0.5 l/min.

The bulk density of the sintered samples was determined by water immersion (Standard Method ASTM C20). The crystalline phases present in the sintered specimens were determined by X-ray diffraction (XRD) using a diffractometer Philips PW3710. The microstructures were observed by scanning electron microscopy (SEM) on polished sections after etching in hydrofluoric acid (40%) at room temperature for about 15 min.

3. Results and discussion

3.1. Powder characterization

The specific surface area of the coated powder was 15 m^2/g and that of the mixed powder was 11 m^2/g.

Fig. 1 shows the zeta potential versus pH curves of the Al_2O_3, Y_2O_3, as-received and coated Si_3N_4 powders. The isoelectric point (IEP) of the Al_2O_3 and Y_2O_3 powders was found to be 8 and 10.7, respectively (b and d curves). The pH_{IEP} values of these oxides were in agreement with those reported in the literature [3,7].

The Si_3N_4 powder surface was characterized by an IEP at pH 6.4 (a curve). This IEP value was in agreement with that previously reported for this Si_3N_4 powder [8].

The coated powder (c curve) showed an IEP at pH 9.7, which corresponded to an intermediate value between the pH_{IEP} of Y_2O_3 and Al_2O_3. Thus, the coating shifted the pH_{IEP} of the as-received Si_3N_4 powder from 6.4 to 9.7. The surface charge properties of the coated powder particles changed from Si_3N_4-like to Y_2O_3 and Al_2O_3-like.

3.2. Rheological properties

Fig. 2 shows the flow curves of viscosity versus shear rate as a function of pH for the mixed Si_3N_4 slips with 0.12 wt.% NH_4PA. The suspensions exhibited a pseudoplastic behavior followed by either a near Newtonian plateau at pH 9.2–10.3 or a shear-thickening effect at pH 10.5. The viscosity decreased with increasing pH from 9.2 to 10.3 throughout the whole range of shear rates. The viscosity values at pH 10.3–10.5 were nearly the same up to 100 s^{-1}. At shear rates higher than 100 s^{-1} the slips at pH 10.5 showed a shear-thickening behavior with the corresponding increase in the viscosity values. As the pH increased from 9.2 to 10.3 the minimum viscosity values decreased from 497.4 to 143.5 mPa.s, respectively. Then the minimum viscosity increased to 155.2 with further increasing of pH (pH = 10.5).

The ammonium polyacrylate dissociation according to the reaction

$$RCOONH_4 = RCOO^- + NH_4^+$$

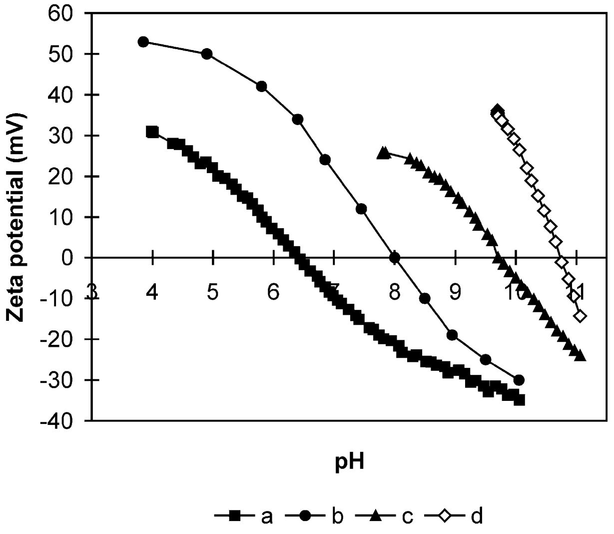

Fig. 1. Zeta potential versus pH curves of different powders: (a) as-received Si_3N_4, (b) Al_2O_3, (c) coated Si_3N_4, (d) Y_2O_3.

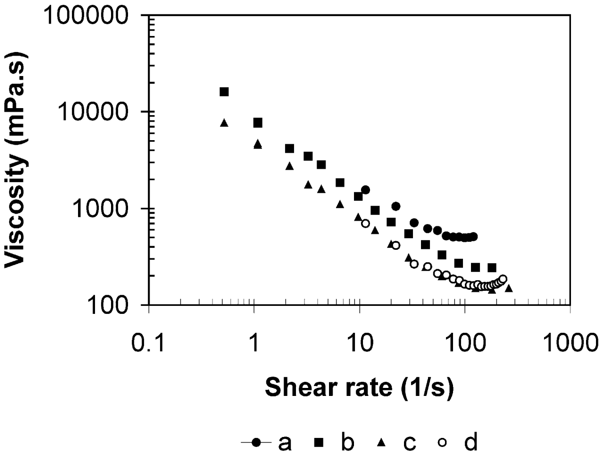

Fig. 2. Viscosity versus shear rate curves of mixed Si_3N_4 slips with 0.12 wt.% NH_4PA solution added at different pH values: (a) 9.4, (b) 9.8, (c) 10.3. (d) 10.5.

begins at pH > 3.5; at pH values ⩾8.5 the polymer charge is negative with the degree of ionization (α) approaching 1 [4].

The amount of NH_4PA adsorbed on the mixed powder at alkaline pH was very low and the polymer adsorbed mainly on the sintering aid particles [9]. The positive Y_2O_3 zeta potential at pH 9–10.7 changed to negative by the adsorption of NH_4PA. Consequently, the NH_4PA did not contribute significantly to the interfacial charge properties of the Si_3N_4 powder. Thus, the dispersion properties were basically controlled by the Si_3N_4 surface charge. As the pH increased from 9.2 to 10.3 the negative zeta potential value of the Si_3N_4 powder increased accordingly (Fig. 1a) and consequently the electrostatic repulsion between particles, thereby decreasing the slip viscosity. High amounts of NH_4OH (0.2 M) should be added to obtain pH values higher than 10.3. The increase in the ionic strength of the solution decreased the zeta potential of the Si_3N_4 powder due to the large compression of the double layer [10]. Therefore, a decrease in the electrostatic repulsion between particles at pH 10.5 could be expected which produced the increase in the slip viscosity.

The optimum pH value (10.3) seemed to be an intermediate situation between a low electrostatic repulsion (pH < 10.3) and a high ionic strength (pH 10.5).

Fig. 3 shows the flow curves of viscosity versus shear rate as a function of pH for the coated Si_3N_4 slips with 2.3 wt.% NH_4PA. The suspensions had a pseudoplastic behavior at low shear rates followed by a shear thickening behavior at higher shear rates. The degree of shear thinning and shear thickening at the different pH values were nearly the same. The viscosity increased with increasing pH throughout the whole range of shear rates. As the pH increased from 9.2 to 10.2 the minimum viscosity values increased from 35 to 70 mPa.s, respectively.

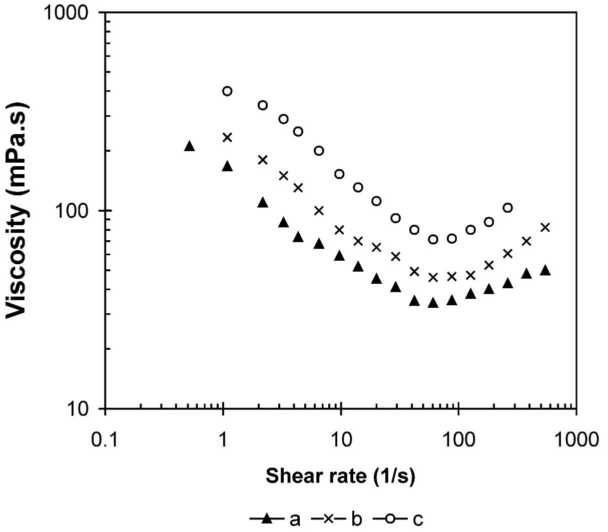

Fig. 3. Viscosity versus shear rate curves of coated Si_3N_4 slips with 2.3 wt.% NH_4PA solution added at different pH values: (a) 9.2, (b) 9.7, (c) 10.2.

As was previously mentioned the optimum concentration of NH_4PA at pH 9.2 was 2.3 wt.%. At pH 9.2, which was near the pH_{IEP}, the zeta potential value of the coated powder was + 11.3 mV (Fig. 1c), therefore a favorable electrostatic interaction between the $RCOO^-$ groups of the deflocculant and the coated Si_3N_4 particles was expected. The maximum adsorption of NH_4PA on Al_2O_3 at a pH value scarcely more acidic than its pH_{IEP} was about 0.4 mg/m^2 which corresponded to 0.6 wt.% NH_4PA [4]. Therefore, the adsorption values on the coated powder were expected to be higher than those observed for Al_2O_3. Another additional effect than simple electrostatic considerations and specific-surface-site-NH_4PA segment interactions, enhanced the adsorption on the coated powder. Since the solubility of Y_2O_3 was ten orders-of-magnitude higher than that of Al_2O_3, different positive yttrium ions such as $Y(OH)_2^+$ and $Y(OH)^{++}$ existed in the solution at alkaline pH [3]. These cations decreased the negative charge of the polymer and greatly affected the polymer conformation in solution. The polymer chains had a coiled bulk conformation due to the presence of the positive yttrium ions in the solution [11]. The coiled conformation of NH_4PA permitted a denser packing on the particle surface, thereby increasing the NH_4PA adsorption. A decrease in the amount of NH_4PA adsorbed with increasing pH was expected due to the electrostatic repulsive forces between the NH_4PA-adsorbed coated Si_3N_4 particles and the anionic polymer remaining in solution.

The pH dependence of the coated Si_3N_4 slip viscosity was markedly different from that observed for the mixed Si_3N_4 slips, indicating that the dispersion of the coated powder was governed by the adsorbed polyelectrolyte. This was a consequence of the greater adsorption of the coated powder with respect to the mixed one.As the adsorption decreased with increasing pH, the electrosteric repulsion between particles decreased accordingly, and an appreciable amount of NH_4PA remained in solution. Thus, the increase in the slip viscosity with increasing pH was attributed to a combination of two effects: less NH_4PA on particle surface and high ionic strength of the solution.

The viscosity values of the coated Si_3N_4 slips were lower than those of the mixed Si_3N_4 slips at approximately the same pH values. The electrosteric contribution from the greater adsorption of NH_4PA at the pH values studied was responsible for the lower viscosity values of the coated Si_3N_4 slips.

3.3. Characterization of green samples

Fig. 4 shows the green density of cast samples prepared from the coated and mixed Si_3N_4 slips versus the

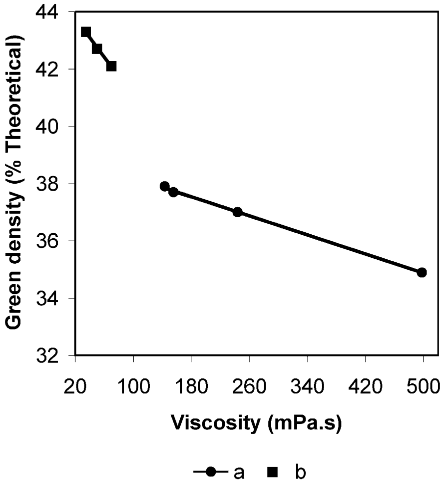

Fig. 4. Green density versus the minimum slip viscosity of cast samples prepared from different slips: (a) mixed Si_3N_4 slips, (b) coated Si_3N_4 slips.

minimum slip viscosity. A linear correlation between the green density and the minimum viscosity was found. As the slip viscosity decreased a more dense packing of the samples could be obtained. The better slip dispersion of the coated Si_3N_4 slips resulted in higher green density values. For the coated Si_3N_4 slips, green density values between 42 and 43.3% of theoretical density were found; while for the mixed Si_3N_4 slips, the green density had values between 34.9 and 37.9% of theoretical density. Thus, the surface coating of Si_3N_4 particles with sintering aids improved their dispersion with NH_4PA resulting in a more dense packing of cast samples.

Fig. 5 a shows the cumulative micropore volume by mass unit versus pore radius curve of green samples produced from the mixed and coated Si_3N_4 slips. Fig. 5b is a representation of the micropore size distribution as dV/dlog r (differential pore volume) versus pore radius. The total micropore volume of the green bodies prepared from the coated Si_3N_4 slips was lower than that of the mixed Si_3N_4 slips. This reduction of the total micropore volume produced the increase in the green density of cast samples.

Both samples had a narrow micropore size distribution. The green sample prepared from the mixed Si_3N_4 slip had pore radius between 0.037 and 0.18 μm and the most frequent pore radius was 0.12 μm. The pore size distribution of the sample obtained from the coated Si_3N_4 slip was shifted to smaller pore radius. Thus, it had pore radius between 0.0061 and 0.12 μm and the most frequent pore radius was 0.081 μm.

3.4. Sintering behavior

Fig. 6 shows the sintered density versus the green density of cast samples. A linear correlation between the sintered density and the green density was found. The better sintering results were obtained with the samples prepared from the coated Si_3N_4 slips which have higher green density values. For the green samples obtained from the coated Si_3N_4 slips, sintered density values between 94 and 96% of theoretical density were found; while for the mixed Si_3N_4 slips, the sintered density had values between 86 and 89% of theoretical density.

The correlation between the sintered density and the green density indicated that the densification was not enhanced by the surface coating.

Fig. 7 shows the XRD spectra of sintered samples prepared from the mixed and coated Si_3N_4 slips. β-Si_3N_4 was the predominant crystalline phase with much smaller amounts of α-Si_3N_4 and yttrium nitrogen melilite ($Y_2Si_3O_3N_4$). α-Si_3N_4 dissolves and precipitates as β-Si_3N_4 when its solubility limit in the liquid is exceeded [12]. The X-ray data indicated an initial reaction

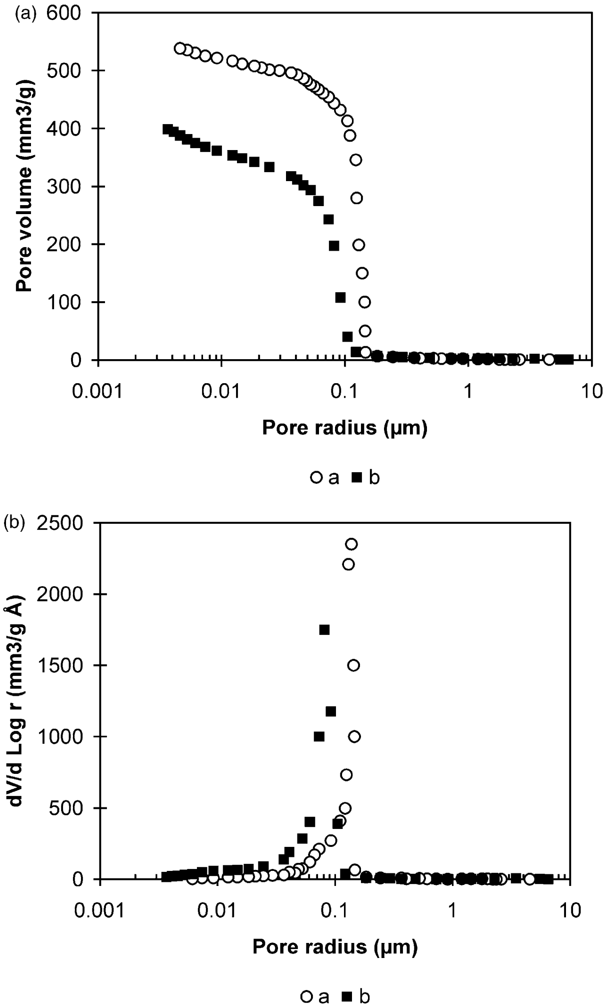

Fig. 5. (a) Cumulative micropore volume by mass unit versus pore radius curves of green samples produced from different slips: (a) mixed Si_3N_4 slips, (b) coated Si_3N_4 slips. (b) Micropore size distribution curves of green samples produced from different slips: (a) mixed Si_3N_4 slips, (b) coated Si_3N_4 slips.

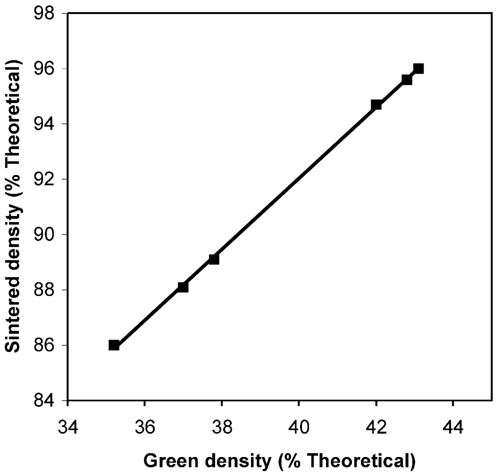

Fig. 6. Sintered density as a function of green density of cast samples.

between Y_2O_3 and Si_3N_4 to form $Y_2Si_3O_3N_4$ which slowly disappears with increased heating time by dissolution in the liquid distributed through the sample [12].

The α-Si_3N_4+$Y_2Si_3O_3N_4$/β-Si_3N_4 intensity ratio calculated with the 31.96° (α-Si_3N_4+$Y_2Si_3O_3N_4$) and 27° (β-Si_3N_4) peaks was 0.08 and 0.03 for the sintered samples prepared from the mixed and coated Si_3N_4 slips, respectively. These intensity ratios indicated that lesser amounts of α-Si_3N_4 and $Y_2Si_3O_3N_4$ and greater amounts of β-Si_3N_4 were present in the sintered sample obtained from the coated Si_3N_4 slips. The reduction in the area of the 20.62° (α- Si_3N_4) and 31.96° (α-Si_3N_4+$Y_2Si_3O_3N_4$) peaks by a factor of about 3 (Figs. 8 a and b) confirmed the smaller content of α- Si_3N_4 and $Y_2Si_3O_3N_4$ phases in the sintered samples prepared from the coated Si_3N_4 slips. Thus, the α- to-β phase transformation and the dissolution of $Y_2Si_3O_3N_4$ in the liquid were enhanced by the surface coating of the Si_3N_4 powder.

Solution-reprecipitation for Si_3N_4 with Y_2O_3 and Al_2O_3 is controlled by interfacial reaction, as explained in other works [13,14]. The interfacial reaction increases with increasing the solid-liquid interfacial surface. An increase in the interfacial surface was expected in the samples obtained from the coated Si_3N_4 slips due to the homogeneous distribution of sintering aids within the green bodies. Consequently, the phase transformation was enhanced by the surface coating. The dissolution of $Y_2Si_3O_3N_4$ in the liquid phase was also enhanced by increasing the solid-liquid interfacial surface. Thereby, the surface coating of Si_3N_4 promoted the phase transformation and the dissolution of crystalline phases such as $Y_2Si_3O_3N_4$. In this work, densification and phase transformation behavior were not affected and enhanced, respectively, with the surface coating of Si_3N_4 particles with sintering aids.

4. Conclusions

The dispersion of the mixed Si_3N_4 powder with NH_4PA showed that the viscosity of the mixed Si_3N_4

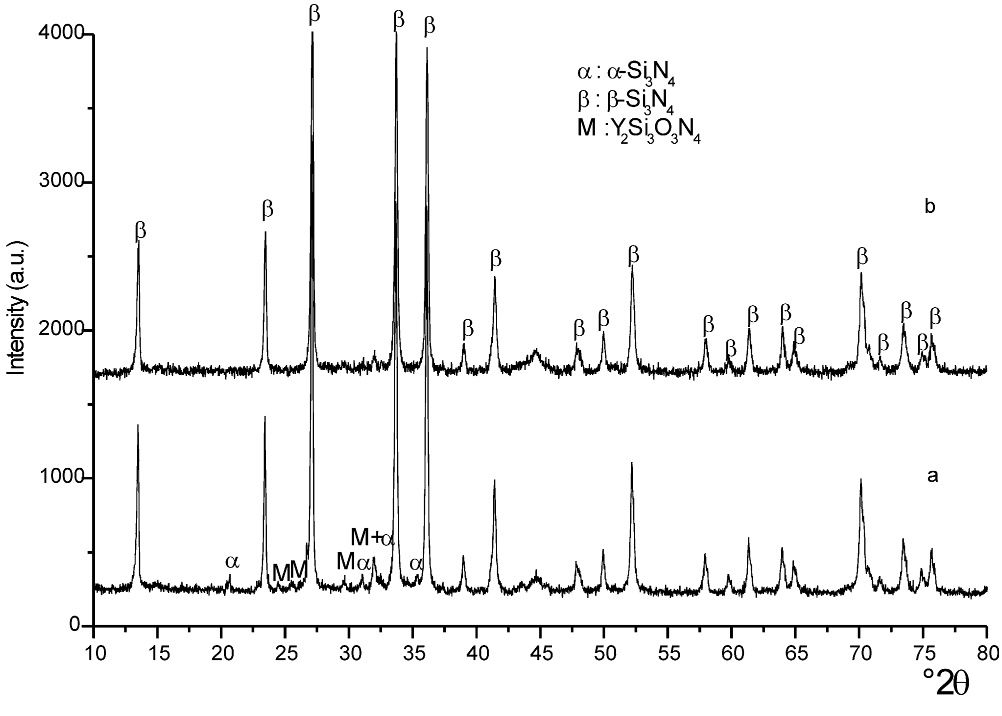

Fig. 7. XRD spectra of sintered samples prepared from different slips: (a) mixed Si_3N_4 slips, (b) coated Si_3N_4 slips.

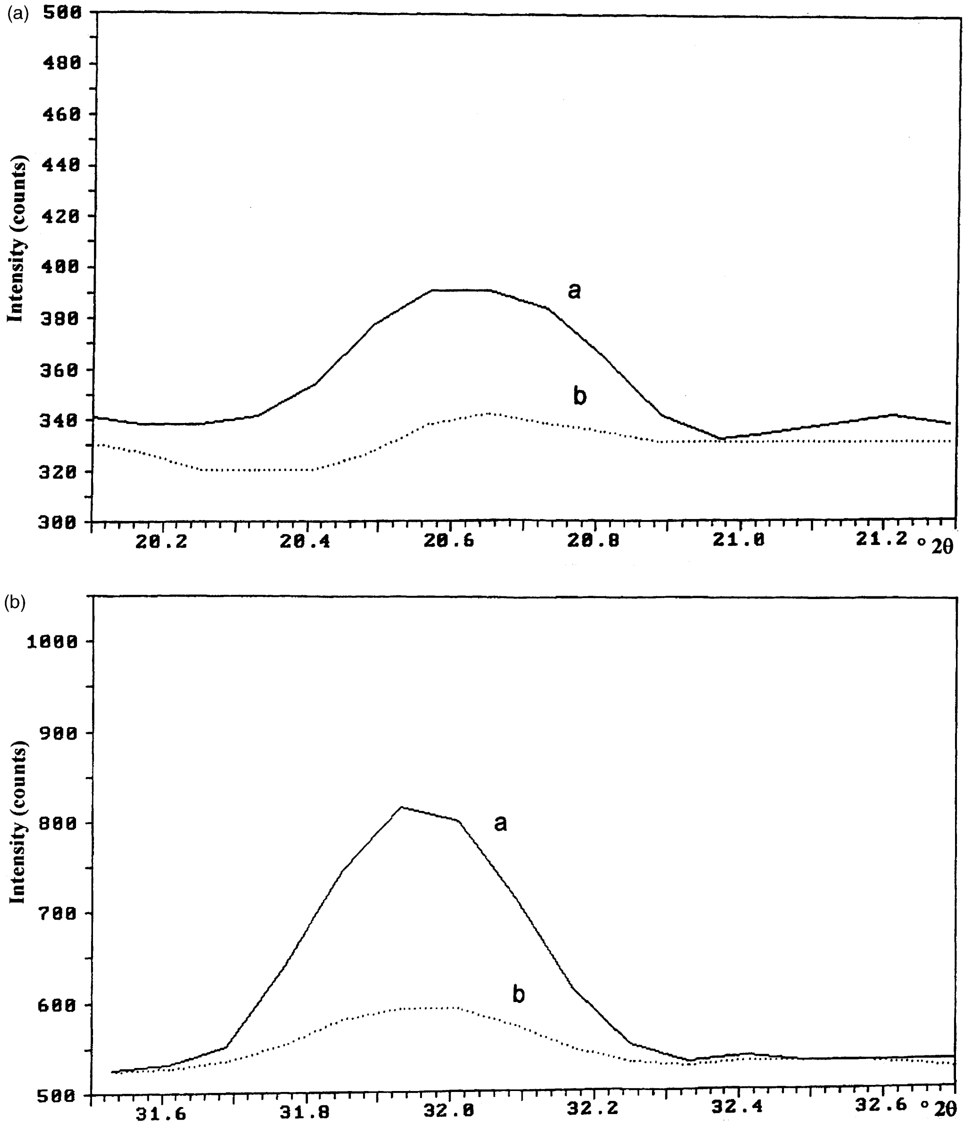

Fig. 8. (a) XRD 20.62° peak of sintered samples produced from different slips: (a) mixed Si_3N_4 slips, (b) coated Si_3N_4 slips. (b) XRD 31.96° peak of sintered samples produced from different slips: (a) mixed Si_3N_4 slips, (b) coated Si_3N_4 slips.

slips decreased with increasing pH from 9.2 to 10.3 and then increased with further increasing of pH up to 10.5. On the contrary, the viscosity of the coated Si_3N_4 slips increased with increasing pH from 9.2 to 10.2. The dispersion of the mixed Si_3N_4 powder was basically controlled by the Si_3N_4 surface charge, while the dispersion of the coated powder was governed by the adsorbed polyelectrolyte.

Lower viscosity values of the coated Si_3N_4 slips were found in the pH range studied. The better slip dispersion of the coated Si_3N_4 slips resulted in a reduction of the total micropore volume of cast samples and consequently in higher green density values.

A linear correlation between the sintered density and the green density was found. Since the mixed and coated powders had the same sintering-additive content the densification process was not enhanced by the surface coating of the Si_3N_4 powder. Nevertheless, the surface coating promoted the α- to- β phase transformation of Si_3N_4 and the dissolution of crystalline phases such as $Y_2Si_3O_3N_4$ in the liquid phase. Thus, in this work, densification and α- to- β transformation behavior were not affected and enhanced with the surface coating of Si_3N_4 particles with sintering aids.

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