Forming of the Alumina Powder - Silicon Nitride Whisker System with Aqueous Suspension

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Abstract

Rheological characteristic of the aqueous colloidal suspensions in the Al_2O_3 powder - Si_3N_4 whisker system and the packing nature of the mixed solid particles were studied to control the microstructure and density of the composite materials. High purity α-Al_2O_3 powder (average diameter ~0.2 μm) of an isoelectric point of pH 7.0 and β-Si_3N_4 whisker (average size 3.7 μm x 0.4 μm) of an isoelectric point of pH 3.0 were mixed in water at pH 3.0 and 10.5, and consolidated by filtration through gypsum molds. Low viscosity Newtonian suspensions were prepared at pH 3.0 for the Al_2O_3 powder-rich composition and pH 10.5 for the whisker-rich composition. The density of powder compacts consolidated by filtration depends upon the surface characteristic of matrix phase solid and whisker fraction. Green compacts with high packing densities were obtained by filtration of the Newtonian suspensions in which highly charged matrix phase solid was well dispersed due to the strong repulsion force. Increase of whisker fraction up to ~40 vol% in the mixed solid particles increased

the packing density, but the density decreased at higher fraction of whisker. Whisker fraction dependence of packing density was analyzed by the packing models by Starr. Degree of orientation of whiskers in the green compacts was also investigated in relation to the coexistence effect of alumina powder or the concentration effect of whisker in the aqueous suspensions.

1. Introduction

Whisker-reinforced ceramics have high potential for structural materials application due to their high fracture toughness and high mechanical strength[1),2)]. The degree of whisker or fiber reinforcement of ceramics depends greatly upon the microstructure of the composite materials and the physicochemical difference between whisker or fiber and ceramic matrix. According to the crack deflection theory by Faber and Evans[3)], the degree of fracture toughness increase is closely related to the volume fraction and the aspect ratio of whiskers in the ceramic composite materials. In the powder processing of whisker reinforced ceramics, the first key step is to disperse uniformly a lot of whisker of high aspect ratio in the matrix powder. Once a raw powder and a raw whisker are given, controlling the density and microstructure of a green compact is very important in designing the microstructure and resulting properties of the final fired composite materials. The next step is to densify the green compact to full density at low temperatures to prevent the grain growth of matrix powder or undesired reaction between matrix powder and whisker. For the composite material processing of submicron size powder and whisker, colloidal dispersion and consolidation techniques have high potential in achieving uniform microstructure and high green density[4)-8)]. The stability and rheological properties of colloidal suspensions are reflected in the properties of green compacts which in turn control the properties of the sintered composite materials.

This paper clarifies the rheological properties of aqueous suspensions and the structures of consolidated powder compacts in the two-component system consisting of end members of submicron size Al_2O_3 powder and Si_3N_4 whisker. Experimental results and theoretical analysis suggest that the important parameter affecting the rheology of suspensions and the structures of powder compacts is the difference in surface characteristic and size between the two components.

2. Experimental procedures

Colloidal processing was used to mix high purity α-alumina powder (>99.99%, Sumitomo Chemical Co. Ltd.) of a specific surface area 10 m^2/g (equivalent spherical diameter ~0.15 μm) and β-silicon nitride whisker (Ube Industries Ltd., Chemical composition : N 40%, O 1%, Y 0.28%, Ca 10 ppm, Al 10 ppm, Fe 700 ppm) of a specific surface area 2.5 m^2/g. Figure 1 shows the transmission electron micrographs (H-700H type, Hitachi Co., Tokyo, Japan) of Al_2O_3 particles and Si_3N_4 whiskers. The as-received Al_2O_3 powders consisted of equiaxised particles with the particle size distribution of 0.03 to 0.40 μm. The Si_3N_4 whiskers were straight one with the lengths of 1.7 to 20 μm (average 3.7 μm) and the diameters of 0.1 to 1.3 μm (average 0.4 μm). The electrophoretic mobility of α-Al_2O_3 particles and Si_3N_4 whiskers in the dilute suspensions (0.02 wt%) were measured at room temperature to calculate the zeta potential (Zeta-Meter Inc., U.S.A.). HCl or NH_4OH solution was used for pH adjustment. The mixtures of Al_2O_3 powder and Si_3N_4 whisker were dispersed in water of pH 3.0 and 10.5 at solid concentrations of 12.5 to 51 vol% to investigate the rheological properties and the structures of consolidated powder compacts. After the aqueous suspensions were stirred for 24 hrs, ultrasonic vibration (Ultrasonic Homogenizer US-150, 20kHz, Nihon Seiki Seisakusho Co., Tokyo, Japan) was applied for 5 min to facilitate the dispersion of powder and whisker agglomerates. Air bubbles in the suspensions were eliminated in a bell jar which was connected to a vacuum pump (BSW-100, Satō Shinkū Kikai Kōgyō Co., Saitama, Japan). Shear rate - shear stress relation of the colloidal suspensions was measured by a cone and plate viscometer at room temperature (Visconic EMD type, Tokyo Keiki Co., Tokyo, Japan). The colloidal solids in the aqueous supensions were consolidated to form green compacts by filtration through gypsum molds. Green compacts were dried in air at 110°C overnight. Densities of the green compacts were measured by Archimedes method using kerosene. The degree of orientation of whiskers in the green compacts was evaluated by comparing X-ray diffraction intensity (Model No.2013, Rigaku Co., Tokyo, Japan) from the free upper surface of the compact with data of the JCPDS card[9].

3. Results and discussion

3.1 Rheological properties of the colloidal suspensions

Figure 2 shows the zeta potential of the dilute suspension of as-received α-Al_2O_3 powder and β-Si_3N_4 whisker as a function of pH. The isoelectric points for Al_2O_3 powder and Si_3N_4 whisker were pH 7.0 and 3.0, respectively. The pH dependence of zeta potential for Si_3N_4 whisker resembles that for SiO_2 powder[10], and suggests that SiO_2 film coated the surface of Si_3N_4 whisker. From these data and the previous experimental results[10)-12], Al_2O_3 powder and Si_3N_4 whisker were mixed at pH 3.0 and 10.5 where positively charged alumina particles and negatively charged whiskers were well dispersed due to the strong electrostatical repulsion force, respectively.

Figure 3 shows the shear rate - shear stress relation of suspensions for the Al_2O_3 powder (80 vol%) - Si_3N_4 whisker (20 vol%) system. A linear relation passing the origin of shear rate and shear stress for the suspensions with solid concentration below 31 vol% is indicative of Newtonian flow. At higher solid concentrations, rheological properties changed to Pseudoplastic or Bingham flow with yield stress at $\dot{\gamma}=0$. As seen in Fig.3, the difference in rheological properties between the Al_2O_3 powder - Si_3N_4 whisker suspension and the Al_2O_3 powder suspension was small at solid concentration below 41 vol%. This result indicates the small interaction between Si_3N_4 whiskers and Al_2O_3 particles in the suspension. One interpretation about the above experimental results is that the Si_3N_4 whisker has low zeta potential at around pH 3.0 (Effect 1). Another possible explanation is the size difference of alumina particles and whiskers. The surface area of the Al_2O_3 powder (80%) - Si_3N_4 whisker (20%) system is 84% of that of the Al_2O_3 powder at a similar solid concentration because the size of whisker is bigger than that of alumina particle. The contribution of whisker to the surface area of the Al_2O_3 powder (80%) - Si_3N_4 whisker (20%) system was calculated to be 5%. Since the interaction between the solids in suspensions would be proportional to the surface area, above calculation suggests that whisker addition decreases the interaction between solids in the suspension (Effect 2). In addition, whisker addition means the decrease of solid particle number in the suspension at a similar solid concentration. This decrease of solid particle number in the suspension indicates the increase of average distance between solid particles, implying decrease of the interaction between them (Effect 3). Effects 2 and 3 would be connected with the decrease of viscosity of the suspension. In our previous experiment[13) on the rheology of the Al_2O_3 powder - Si_3N_4 whisker suspension of the solid concentration about 30 vol% at pH 3.0, it was recognized that the viscosity of 95% Al_2O_3 powder - 5% Si_3N_4 whisker suspension was lower than that of Al_2O_3 powder suspension. Increase of the viscosity and appearance of the yield stress at higher solid content in the 80% Al_2O_3

- 20% Si_3N_4 whisker suspension in Fig.3 reflect the decrease of surface area effect (Effect 2) and solid particle distance effect (Effect 3) with the increase of solid concentration. Increase of whisker concentration accompanied by the increase of volume fraction of whisker or solid content will also cause the contact between whiskers and the formation of network structure by whisker. This change in solid structure of the suspension would increase the viscosity and yield stress (Effect 4).

Figures 4 and 5 summarize the viscosity and yield stress of the Al_2O_3 powder - Si_3N_4 whisker suspensions at pH 3.0 and 10.5 whose rheological behavior was approximated by Bingham flow. In the previous paper[10], we pointed out the physical similarity of Bingham flow to the Voight model representing a viscoelastic characteristic. The basic equation for the Voight model is expressed by Eq.(1),

$$S = E\gamma + \eta \frac{d\gamma}{dt} \qquad (1)$$

where S, E, γ, η , and t mean shear stress, Young's modulus, strain, viscosity, and time, respectively. That is, the slope and extrapolated intercept (S_0) at $\dot{\gamma} = 0$ of the curves in Fig.3 correspond to η (viscosity of a Newtonian liquid) and $E\gamma$ (elastic deformation stress of solid element), respectively. Increase in the yield stress (S_0) suggests the formation of rigid network structure of colloidal solids in the suspension, which is destroyed at a corresponding shear stress S_0. Once the yield stress is applied to the suspension, the property of suspension changes from an elastic solid of Young's modulus E to an ideal Newtonian liquid of viscosity η.

As mentioned before, the change from Newtonian flow to Bingham flow with increasing whisker fraction in Fig.4, which leads to the increase of viscosity and yield stress, is mainly explained by the network structure effect (Effect 4) of whisker. The low viscosity and low yield stress measured at the volume fraction of whisker below 40% for the suspensions of 20 vol% solid suggest that the surface area effect (Effect 2) and the solid particle distance effect (Effect 3) were bigger than the network structure effect (Effect 4). Change in the rheology at higher solid concentration in Fig.4 suggests the decrease of Effects 2 and 3, and the increase of Effect 4.

In contrast with Fig.4, the data in Fig.5 for the colloidal suspensions at pH 10.5 represent the opposite effect of whisker addition on the rheology. In this case, Effect 4 interacting among highly charged negative whiskers is relatively low due to the strong repulsion force. Increase of the viscosity and yield stress at low whisker fraction is due to the less Effects 2 and 3, and big Effect 4 by poorly charged alumina particles. According to the theoretical calculation[14)], van der Waals' attraction energy for the submicron size particles with the surface potential below about 25 mV is higher than the repulsion energy, suggesting a poor dispersion state. Although the surface potential is different from the zeta potential, the low zeta potential below 20 mV for the alumina particles at pH 10.5 (Fig.2) is associated with the strong network structure by alumina particles.

3.2 Consolidation of the Al_2O_3 powder - Si_3N_4 whisker system

The Al_2O_3 powder and Si_3N_4 whisker in the aqueous suspensions at pH 3.0 and 10.5 were consolidated by filtration through gypsum molds. Figure 6 illustrates the influence of solid concentration of the Al_2O_3 powder - Si_3N_4 whisker suspensions on the green density of consolidated powder compacts. The green density showed a tendency toward decrease with increasing solid concentration. This result is explanied by the rheology of colloidal suspensions. Experimental results on the rheology and consolidation of the ultrafine SiO_2-Al_2O_3 powder (~80 nm)[15)] indicate that (i) Newtonian suspensions form the green compacts with high densities and narrow pore size distributions, (ii) increase in the solid concentration of Newtonian suspension leads to an increase of green density[8),16)-18)] (this concentration effect is explained by the concept of long range particle segregation during consolidation), and (iii) the colloidal suspensions showing Bingham flow provide the porous green compacts. The relation between the green densities (Fig.6) and the rheological properties (Figs.4 and 5) was in accordance with the above conclusions. That is, the low flexibility of the network structure of powder or whisker during consolidation, which is expressed by the yield stress, would be the main reason for the lower green density.

Figure 7 shows the relative densities of the consolidated Al_2O_3 powder - Si_3N_4 whisker system as a function of volume fraction of whisker. Most of the consolidation experiment were done with the suspensions of 28-40 vol% solid at pH 3.0 and 20-30 vol% solid at pH 10.5. Comparison of green densities between the two suspensions at pH 3.0 and 10.5 indicates that high dispersion state (high zeta potential) of the matrix phase in the suspensions leads to high packing density. The suspensions of whisker fraction of 60 and 80 vol% at pH 10.5 provided green compacts of a two-layer structure, indicating long range particle segregation. Upper and lower layers of the compacts were richer in Si_3N_4 whisker and in Al_2O_3 powder respectively than the mixing composition. The relative densities in Fig.7 show the whole densities of layered green compacts. Dependence of green density on the volume fraction of whisker shows an important packing characteristic of the Al_2O_3 powder - Si_3N_4 whisker system. The lower density for the monolithic whisker compact than the monolithic Al_2O_3 compact represents bigger network structure effect for the solid particles with higher aspect ratio. However, addition of whisker (<40%) to the alumina powder increased the packing density. Above packing characteristic was quantitatively analyzed by Starr[19)]. Before analyzing the results shown in Fig.7 along the Starr's models, we clarify the concept of partial molar volume for the composite of powder - whisker system. The bulk volume (V) of the Al_2O_3 powder - Si_3N_4 whisker system per mole is given by Eq.(2),

$$V = \overline{V}_p X_p + \overline{V}_f X_f \qquad (2)$$

where $\overline{V}_p$, $\overline{V}_f$, X_p, and X_f are partial molar volumes of powder and whisker, and molar fraction of powder and fiber (X_p+X_f=1), respectively. The molar volume of powder (V_{pt}) and whisker (V_{ft}) are related to the molecular weight (M) and true density (ρ_t) by Eqs.(3) and (4),

$$V_{pt} = \frac{M_p}{\rho_{pt}} \qquad (3)$$

$$V_{ft} = \frac{M_f}{\rho_{ft}} \qquad (4)$$

Equation (5) expresses the density of fully dense composite.

$$\rho_c = \frac{M_p X_p + M_f X_f}{V_{pt} X_p + V_{ft} X_f} \equiv \frac{M_c}{V_c} \qquad (5)$$

On the other hand, the bulk density of the composite (ρ) is defined by Eq.(6).

$$\rho = \frac{M_c}{V} \qquad (6)$$

From Eqs.(5) and (6), we obtain the relation expressed by Eq.(7),

$$\frac{V}{V_c} = \frac{\rho_c}{\rho} = \frac{1}{\rho_{rel}} \qquad (7)$$

where ρ_{rel} is the relative density of composite.

The molar fraction of powder and whisker are transformed to the volume fraction (Y) by Eqs.(8) and (9).

$$Y_p = \frac{V_{pt}}{V_c} X_p \qquad (8)$$

$$Y_f = \frac{V_{ft}}{V_c} X_f \qquad (9)$$

From Eqs.(2),(7),(8), and (9), we obtain Eq.(10),

$$\left(\frac{V}{V_c}\right) = \frac{1}{\rho_{rel}} = \left(\frac{\overline{V}_p}{V_{pt}}\right) Y_p + \left(\frac{\overline{V}_f}{V_{ft}}\right) Y_f \qquad (10)$$

where (V/V_c), $(\overline{V}_p/V_{pt})$ and $(\overline{V}_f/V_{ft})$ represent relative specific volumes for composite (Z_c), powder (Z_p), and whisker (Z_f), respectively.

Starr[19)] proposed the two models, sphere model and fiber model, to the structure of composite.

(A) Sphere model

In this model, Z_p and Z_f are given by the following Equations,

(i) For $Y_f < Y_f^*$

$$Z_p = Z_p^o \quad (\rightarrow \overline{V}_p = V_{pt} Z_p^o) \qquad (11)$$

$$Z_f = 1 \quad (\rightarrow \overline{V}_f = V_{ft}) \qquad (12)$$

(ii) For $Y_f > Y_f^*$

$$Z_p = 0 \quad (\rightarrow \overline{V}_p = 0) \qquad (13)$$

$$Z_f = Z_f^o \quad (\rightarrow \overline{V}_f = V_{ft} Z_f^o) \qquad (14)$$

where Z_p^o and Z_f^o are the relative specific volumes of monolithic powder and monolithic whisker compacts, respectively. The critical composition Y_f^* represents the intersection composition of the two Z_c curves calculated from the conditions (i) and (ii).

$$Y_f^* = \frac{Z_p^o}{Z_p^o + Z_f^o - 1} \qquad (15)$$

(B) Fiber model

In this model, Z_p and Z_f are given by Eqs.(16) and (17), respectively.

$$Z_p = (1 - Y_f)Z_p^o \quad (\rightarrow \overline{V}_p = V_{pt} Z_p^o(1-Y_f)) \qquad (16)$$

$$Z_f = 1 + Y_f(Z_f^o - 1) \quad (\rightarrow \overline{V}_f = V_{ft} [1 + Y_f(Z_f^o - 1)]) \qquad (17)$$

Equations (18) and (19) express the relative specific volume for composite and the composition corresponding to minimum volume (maximum density).

$$Z_c = Z_p^o + (1-2Z_p^o)Y_f + (Z_p^o + Z_f^o - 1)Y_f^2 \qquad (18)$$

$$Y_f^* = \frac{Z_p^o - 0.5}{Z_p^o + Z_f^o - 1} \qquad (19)$$

Figure 8 shows the relative specific volumes as a function of whisker fraction for the composites in Al_2O_3 powder - Si_3N_4 whisker system. As shown in Fig.8, the fiber model provides denser compact than the sphere model but both models can explain the density increase with addition of whisker up to 20-40%. Comparison between the experimental results and the theory suggests that (i) the structure of composite consolidated from the suspension at pH 10.5 is closer to the sphere model than the fiber model, and (ii) the structure of composite with whisker fraction above 40 vol%, consolidated from the suspension at pH 3.0, is believed to be the intermediate structure between the two models.

3.3 Orientation of Si_3N_4 whisker in the green compacts

From the SEM observation and X-ray diffraction analysis of the monolithic whisker green compacts[10)], it was found that the green density of whisker compact was closely correlated to the orientation of whiskers in the compacts. Whiskers in the low viscosity suspensions at pH 10.5 were consolidated with orientation where the deirection in length of whiskers is perpendicular to the direction of filtration. On the other hand, whiskers in the viscous suspensions at pH 3.0 were packed randomly during consolidation. Thus, the increase in the degree of orientation of whiskers was accompanied by the increase of green density (Fig.6) and the reduction of pore size in the compact[10)]. Figure 9 shows the whisker concentration in the Al_2O_3 powder - Si_3N_4 whisker system with whisker fraction of 20-100 vol% and the degree of orientation of whiskers, illustrated by the ratio of relative X-ray diffraction intensity between the free upper surface of the green compacts and the JCPDS card[9)] of Si_3N_4 regarding (220) plane. Increase in the whisker concentration reduced the flexibility of whiskers in the suspensions and decreased the degree of orientation of whiskers during consolidation. Another interesting phenomenon is the coexistence effect of Al_2O_3 powder on the orientation of Si_3N_4 whisker. As seen in Fig.9, addition of the well-dispersed Al_2O_3 powder in the suspension at pH 3.0 seems to not affect the orientation of whiskers in the consolidated compacts. However, in the suspensions at pH 10.5, degree of the orientation of whiskers was decreased by coexistence of the cohesive alumina particles with low zeta potential. These results suggest that dispersion state of Al_2O_3 particles affects greatly the degree of orientation of whiskers in the powder compacts, and poorly charged particles inhibit the phase transformation of whiskers from disordered (random packing in the suspension) to ordered structure (oriented packing in the green compact).

4. Summary and conclusions

Rheological properties and consolidation of the aqueous suspensions in the Al_2O_3 powder - Si_3N_4 whisker system were studied with high purity α-alumina powder (average diameter ~0.2 μm) of an isoelectric point of pH 7.0 and β-Si_3N_4 whisker (average size 3.7 μm x 0.4 μm) of an isoelectric point of pH 3.0. In the suspensions of whisker fraction below 20% at pH 3.0, little interaction between Al_2O_3 particles and Si_3N_4 whiskers was measured in the rheological behavior. The suspensions of whisker fraction of 0 to 40% at pH 3.0 behaved as Newtonian liquid at the solid concentration below 20 vol%. At higher whisker fraction the rheological properties changed to Pseudoplastic or Bingham flow. On the other hand, the suspensions of whisker fraction of 20-100% at pH 10.5 behaved as Newtonian liquid at the solid concentration below 20%. These results were explained based on the surface potential effect (Effect 1), surface area effect (Effect 2), particle distance effect (Effect 3), and network structure effect (Effect 4) by mixing the solid particles with different sizes and surface characteristic.

The density of powder compacts consolidated by filtration through gypsum molds depends upon the surface characteristic of matrix phase solid and whisker fraction. High dispersion state (high zeta potential) of matrix phase in the suspensions leads to high packing density. Addition of whisker to the alumina powder up to ~40 vol% increased the packing density, but higher whisker fraction decreased the density. Structure and density of the Al_2O_3 powder - Si_3N_4 whisker system were analyzed by the concept of partial molar volume and packing models by Starr. This theoretical calculation could explain well the experimental results on the packing characteristic of the mixed powders.

In the powder compacts, whiskers showed a tendency to be arrayed with orientation where the direction of in length of whiskers was perpendicular to the direction of filtration. Increase in the whisker concentration reduced the flexibility of whiskers in the suspensions and decreased the degree of orientation of whiskers during consolidation. Coexistence of the well-dispersed alumina particles in the suspensions seems to not affect the orientation of whiskers, but addition of the cohesive alumina particles of low zeta potential decreased the orientation of whiskers.

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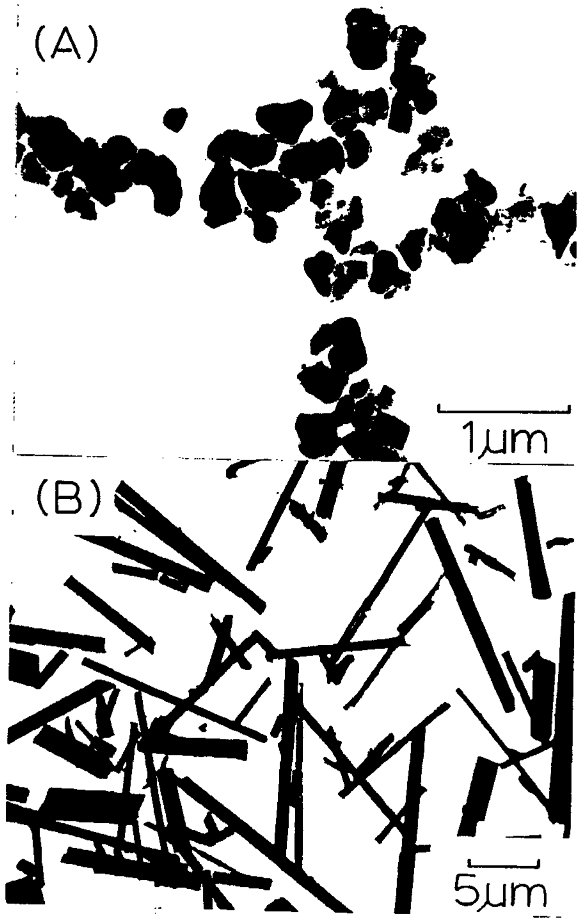

Fig. 1. Transmission electron micrographs of (A) α-Al_2O_3 powder with an average diameter of 0.2 μm and (B) β-Si_3N_4 whisker with an average size of 3.7 μm x 0.4 μm.

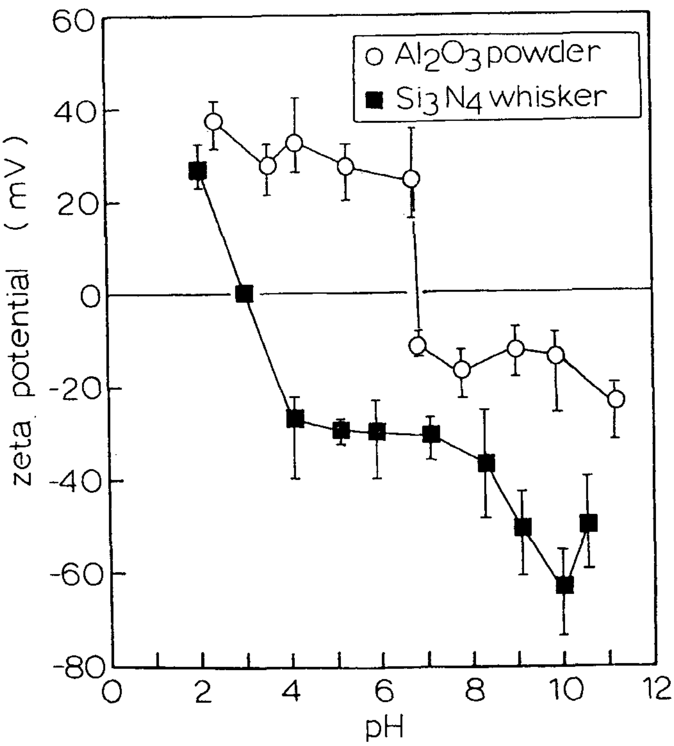

Fig. 2. Zeta potential of the colloidal α-Al_2O_3 particles (O) and β-Si_3N_4 whiskers (■) as a function of pH.

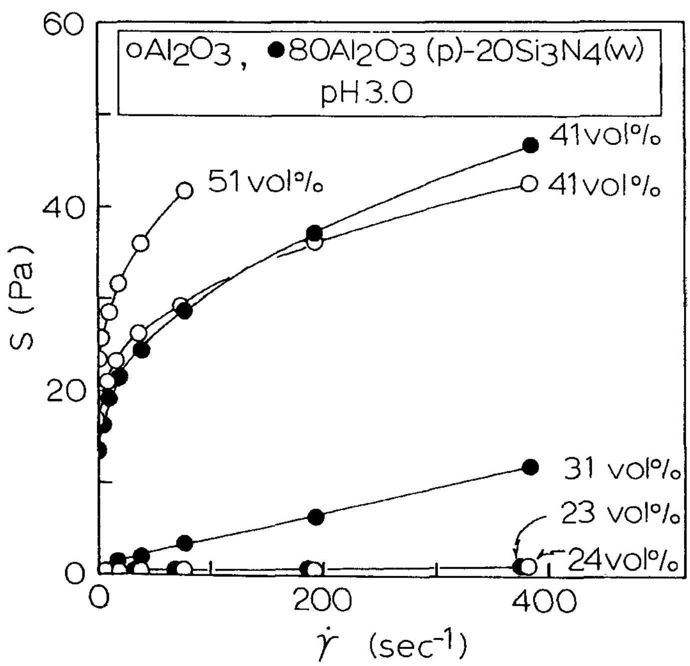

Fig. 3. The shear rate - shear stress relation for the alumina suspension (O) and the 80% Al_2O_3 powder - 20% Si_3N_4 whisker suspension (●).

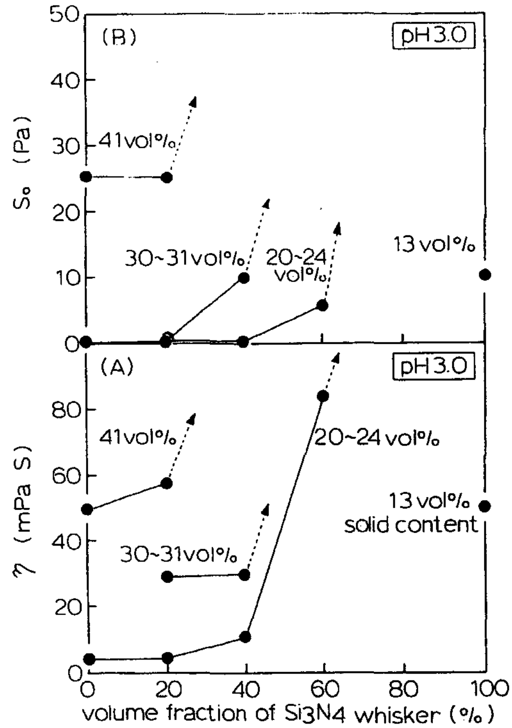

Fig. 4. Viscosity (A) and yield stress (B) of the Al_2O_3 powder - Si_3N_4 whisker suspension at pH 3.0 as a function of whisker fraction.

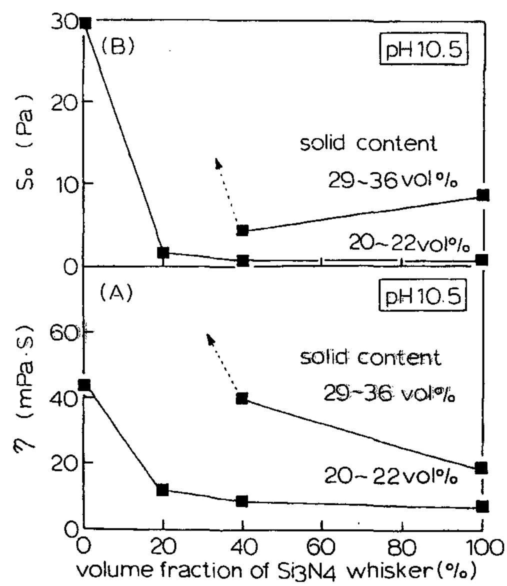

Fig. 5. Viscosity (A) and yield stress (B) of the Al_2O_3 powder - Si_3N_4 whisker suspension at pH 10.5 as a function of whisker fraction.

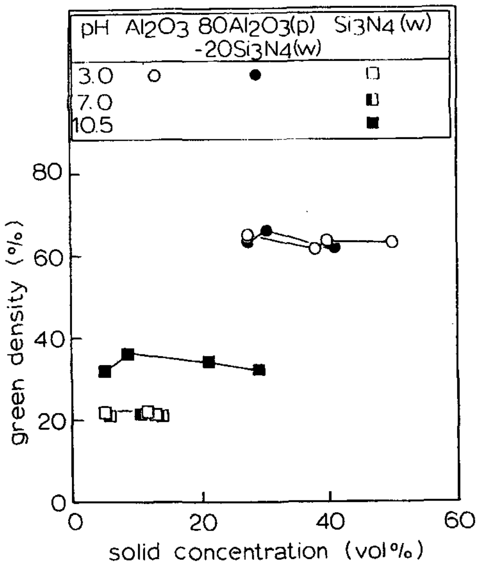

Fig. 6. Solid concentration in the aqueous suspension and green density of the powder compact consolidated by filtration.

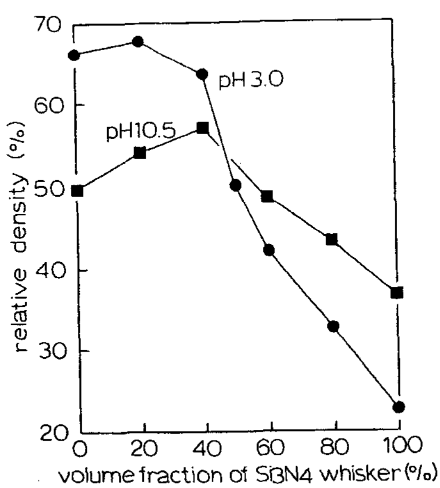

Fig. 7. Green densities of the compacts in the Al_2O_3 powder - Si_3N_4 whisker system consolidated by filtration of the aqueous suspensions at pH 3.0 and 10.5. The colloidal suspensions of whisker fraction of 60 and 80% at pH 10.5 provided the green compacts of a two-layer structure, indicating long range particle segregation during consolidation.

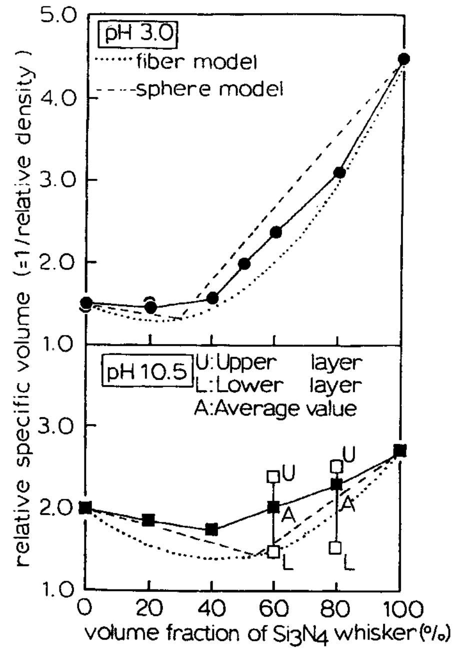

Fig. 8. Relative specific volume of the Al_2O_3 powder - Si_3N_4 whisker system as a function of whisker fraction. The two dotted curves show the calculated relative specific volume based on the fiber and sphere models by Starr[19].

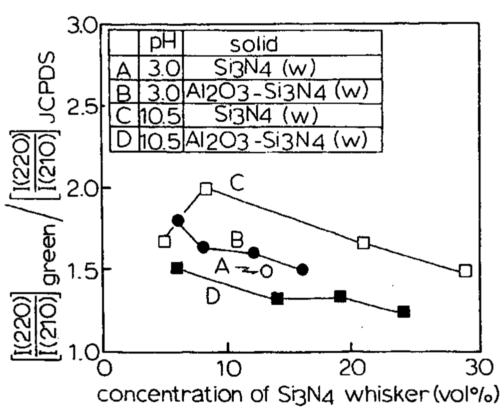

Fig. 9. Ratio of relative X-ray diffraction intensity on (220) plane of Si_3N_4 between free surface of the compacts in the Al_2O_3 powder - Si_3N_4 whisker system and the JCPDS card (No.9-259) as a function of Si_3N_4 whisker concentration. Relative X-ray diffraction intensity of (210) plane is 100 in the JCPDS card.