

Extrusion characteristics of alumina–aluminium titanate composite using boehmite as a reactive binder

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Abstract

Alumina–aluminium titanate composite was fabricated through paste extrusion using boehmite gel as a binder. The rheological characteristics of alumina paste containing various amounts of boehmite binder were studied at different shear rates. The results of investigations on the development of torque, yield stress, viscosity and fluidity are presented. In general, Bingham type plastic behavior was observed. Alumina pastes containing 18.5 vol.% of boehmite exhibits adequate plasticity and fluidity for extrusion. The torque value for this composition is 68 kPa at shear rate 28.5 s^{-1} . The viscosity curves of alumina pastes showed “shear thinning” flow behavior. The mixture of 54.15 vol.% alumina and 18.5 vol.% boehmite resulted in 61% of theoretical green density on extrusion. These conditions were extended to synthesis of alumina–aluminium titanate composites, and the extrusion was carried out using 18.5 vol.% boehmite. Aluminium titanate was synthesized through sol–gel method and dispersed uniformly in alumina matrix through colloidal process. The composites could be sintered at 1400°C to 98% of its theoretical value. The SEM fractograph of sintered composite showed reasonably dense, uniformly distributed grains in the order of 2–3 μm . The present study shows that boehmite binder phase yields good workability for extrusion and contributes high green and sintered densities to the composites. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Alumina is a well recognized structural ceramic material, but under severe thermal environments, catastrophic failure occurs due to large stresses, developed during thermal changes.^{1,2} When the reasonably high mechanical strength and hardness of alumina show advantage as structural material, its high thermal expansion ($\alpha_{20-1000^\circ\text{C}} - 8 \times 10^{-6} \text{ K}^{-1}$) and thermal conductivity, restricts its use for high temperature structural applications.³ Suitable addition of a second phase particulate can improve the thermal properties of alumina. Aluminium titanate is one such additive, which is already reported for mullite and zirconia ceramics, to impart the thermal stability and mechanical strength.⁴ Aluminium titanate is chosen because of the following

reasons. The thermal expansion co-efficient of aluminium titanate is anisotropic and shows large mismatch with matrix alumina. Such unique nature of aluminium titanate causes extensive microcracking, results in large magnitude of internal stresses and influenced the intensity of bridge-clamping stress and ultimately increases the fracture toughness through crack tip shielding mechanism.⁵ In addition, aluminium titanate and alumina can co exist as two distinct phases with limited solubility at the firing temperature. The composite exhibits functional as well as structural properties, for possible applications as thermal barrier, insulating components for diesel engines, thermocouple sheath and high temperature substrates.⁶ Many processing methods have been adopted for the synthesis of such composites and more commonly, reaction sintering of alumina and aluminium titanate mixture at high temperatures is employed.⁷ Infiltration of precursor sols,⁸ sequential slip casting of alumina–aluminium titanate,⁹ gel casting of mullite–aluminium titanate¹⁰ are some of the advanced techniques reported for the preparation of

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aluminium titanate based composites. Alumina/titania, aluminium titanate–mullite and aluminium titanate–ZTA are some of the composites attempted earlier.

The extrusion process has been well recognized for engineering objects having extended length with uniform cross section. Conventional extrusion process requires the use of organic vehicle as binders to impart fluidity for assembling of ceramic powder into a shape.¹¹ Evans and Edrissinghe have reported the key problems in conventional extrusion and injection molding of ceramics.^{12,13} The polymeric binders could be removed only through capillary migration at very slow heating rate and extended heating schedule is required for binder burnout which finally increases the production cost. This also leads to the formation of pores and cracks/ channels in the green product, makes further densification very difficult. Recently, water soluble binder systems are seriously looked for plastic forming and available literature showed that ammonium alginate, agar based binders and carbohydrates such as potato starch d-glucose have been used in lieu of polymers.^{14–17} On the other hand, a group of inorganic binder systems such as sodium silicate, hydrated aluminosilicates and clay minerals like montmorillonite are also employed for the extrusion process.¹⁸

Sol–gel oxides and hydroxides have certain characteristics suitable for use as colloidal binders. In particular, a strong analogy between clay gels and sol–gel derived aluminium monohydroxide (Boehmite), due to its layered structures has been noted.¹⁹ Bruno Kindl used boehmite sol as dispersant for alumina ceramics.²⁰ Colloidal compaction of alumina slurries using boehmite as a dispersing medium was reported by Warriar et al.²¹ Messing et al. reported use of boehmite as a strengthening agent for macroporous alumina.²² Chen and Cawley reported use of colloidal boehmite along with 0.5% PVA in alumina.²³ Boehmite in presence of water under controlled conditions becomes a gelatinous mass and exhibits an adhesive nature.²⁴ Since the role of binders is to provide green strength and plasticity, gelatinous boehmite has been successfully used in place of conventional binders. Sunil Kumar et al. used seeded boehmite gel as an extrusion aid for alumina ceramics and found that the γ -alumina seeded alumina–boehmite mixture has good sintering properties and high mechanical strength.²⁵

The present paper reports the use of boehmite gel as a reactive binder for extrusion of alumina–aluminium titanate composites. Sol–gel aluminium titanate precursor was first dispersed with alumina powder followed by addition of boehmite. The paste extrusion under low pressure was employed for fabricating the composite body. Sintering characteristics and microstructural features of the composite are reported. The rheological characteristics of alumina containing boehmite have also been studied and reported.

2. Experimental

A16 SG alumina (ACC-ALCOA Chemicals, India) having 99.8% purity, BET surface area 8.0–11 m²/g and average particle size 0.3–0.6 μ m was used in this study. Boehmite (Pural SB1, Condea Chemicals, Germany) having crystallite size 48–60 Å and BET surface area 230 m²/g and alumina content 70% was used. Titanium iso propoxide (Fluka Chemicals, USA) was used as a precursor for titania. Acetic acid (BDH Chemicals, India) was used to stabilize the titanium iso propoxide. Distilled water was used as medium.

2.1. Rheological measurements

Flow properties of alumina pastes with and without boehmite was studied by using Brabender Plasticorder equipment (Model PLE 651) at room temperature. Alumina paste containing boehmite in the range 6.5–24.5 vol.% (5–20 wt.%) was prepared by colloidal method as per the flow sheet presented in Fig. 1. The development of torque was observed by changing the rotor speed in the range 10–40 rpm using a measuring mixer W50. The apparent viscosity (η) was determined by dividing torque (τ) with rotor speed. The particle size modification of alumina with the addition of different amounts of boehmite was analyzed by particle size analyzer (Sedigraph, Micromeritics Model: 5100, USA).

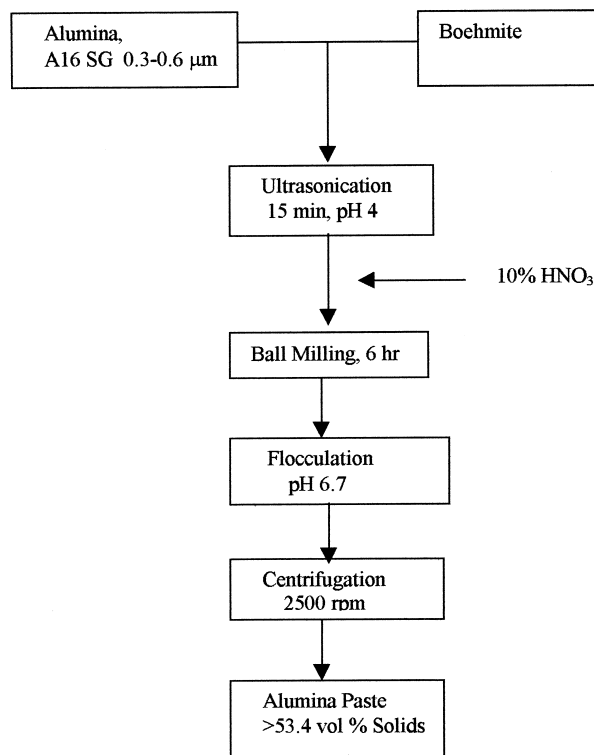


Fig. 1. Flow sheet for fabricating alumina ceramics through paste extrusion.

2.2. Sol–gel synthesis of aluminium titanate

Boehmite sol was prepared by dispersing 50 g boehmite powder in 1 l of water. The sol was maintained at pH 3.0 using dilute HNO_3 and the mixture was kept stirred for 4 h. Titanium iso propoxide (125.01 g equivalent to 35.14 g TiO_2) was stabilized by adding excess acetic acid, and stirred for 2 h at temperature below 5°C . The boehmite sol (equivalent to 44.86 g of alumina) was added slowly to the titania sol under constant stirring. The resultant mixture was further stirred for 5 h and gelled by controlled evaporation. Differential Thermal Analysis (DTA 50 H, Shimadzu, Japan) was taken on a gel sample and mixture was finally calcined at 600°C .²⁶

2.3. Extrusion of boehmite–alumina

The laboratory model vertically mounted plunger type extruder was used in this study and schematically shown in Fig. 2. Extruder assembly is made of stainless steel and has anti-corrosive coating inside. The feed chamber was 2.54 cm diameter and 22 cm length. The barrel and die were lubricated with stearic acid. The conical die arrangement has an entrance angle of 45° and inner diameter 6 mm was used as output.

Alumina–18.5 vol.% boehmite paste having aluminium titanate 5–20% with respect to the weight of alumina was obtained by colloidal method. The details of the process flow sheet are presented in Fig. 3. The composite mass was fed into the extruder chamber and pressure was carefully applied through the plunger at a rate of 2 mm/min and alumina rods having 6 mm diameter and length 150 mm were fabricated. The green samples were dried in humidity controlled oven (REMI Environment Chamber, India) at 65% RH and 45°C for 24 h. Sintering characteristics were observed in a Dilatometer (TMA 50 H, Shimadzu, Japan) at a heating

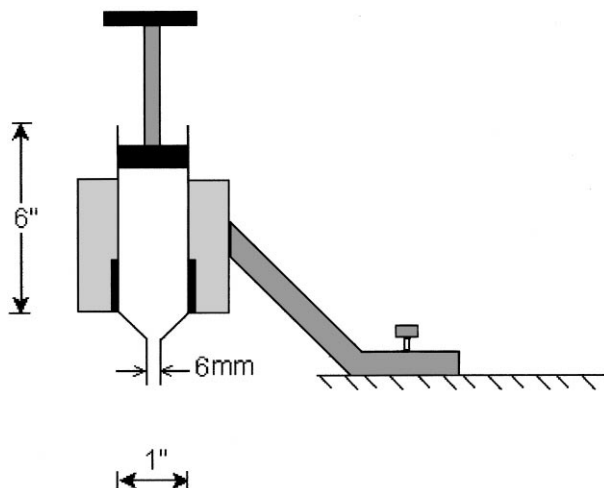


Fig. 2. A laboratory model extruder.

rate of $10^\circ\text{C}/\text{min}$. Sintering was done in the range $1350\text{--}1500^\circ\text{C}/2\text{ h}$ using programmable furnace (Nabertherm, Germany). The sintering cycle was as follows:

RT^{3°C/min}300^{5°C/min}1000^{8°C/min}1250^{5°C/min}
1350 to 1500°C

The sintered density was measured by Archimedes' principle. The formation of crystalline phases were analyzed by X-ray diffraction (Philips, Model 1740 PW, USA). Sintered microstructure of the composites was observed on fractured surface (SEM Hitachi, 2420, Japan).

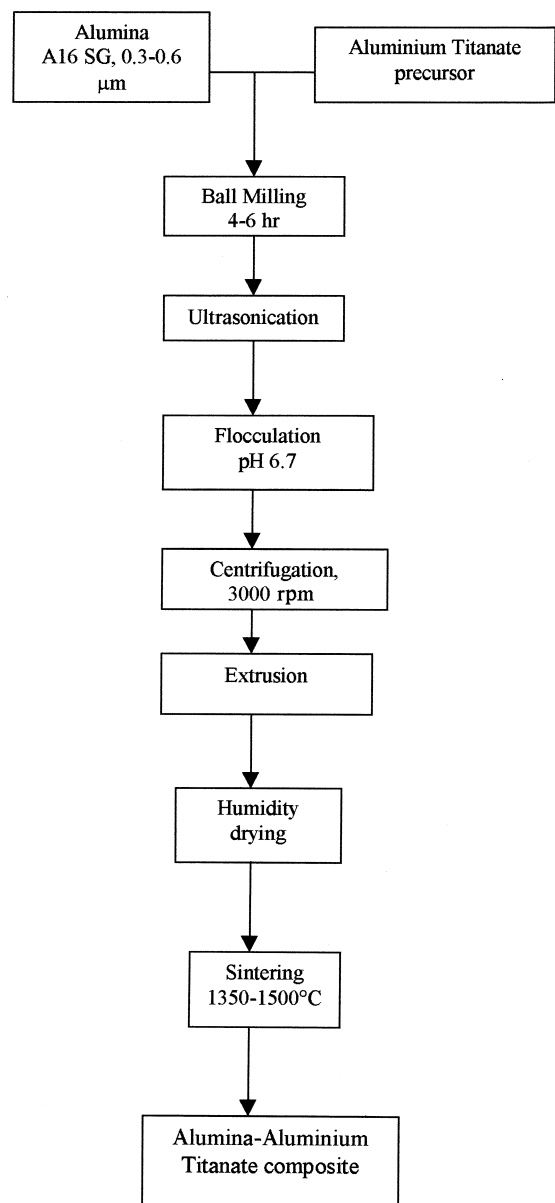


Fig. 3. Process flow sheet for preparing alumina–aluminium titanate composite.

3. Results and discussions

3.1. Effect of boehmite on paste rheology

Fig. 4 shows the development of torque for alumina pastes with and without boehmite binder. The alumina with out boehmite showed reasonably high torque value of about 69.8 kPa even at low shear rate in the range 9.5 s^{-1} and gradually increased to 102.97 kPa at shear rate 38 s^{-1} . The addition of boehmite binder showed considerable effect on torque depending upon the amount of addition. Alumina containing only 6.5 vol.% boehmite resulted in higher torque value of about 93.43 kPa at 9.5 s^{-1} shear rate and 133.48 kPa at shear rate 28.5 s^{-1} . It remains without much change until shear rate 38 s^{-1} . However, further addition of boehmite, drastically reduce the torque and resulted in reasonably low torque range. The alumina containing 18.5 vol.% of boehmite has the total torque in the range 38.15 kPa at shear rate 9.5 s^{-1} and 68 kPa at 28.5 s^{-1} . The torque was not appreciably changed even at high shear rates. However, it was noticed that once the addition of boehmite exceeds 18.5 vol.%, there is a tendency for developing high torque. Since high torque will have negative impact on mass flow and particle packing, alumina having 18.5 vol.% of boehmite was fixed for paste extrusion. Also,

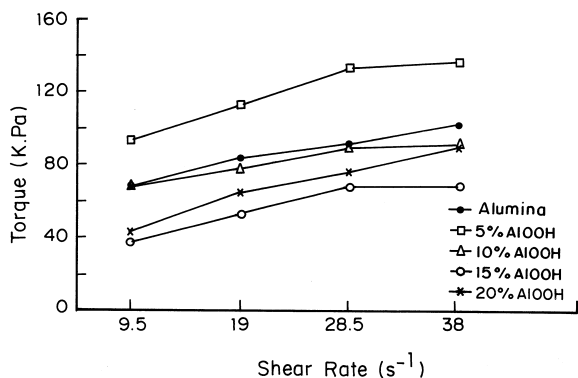


Fig. 4. Development of torque with varying amounts of boehmite binder.

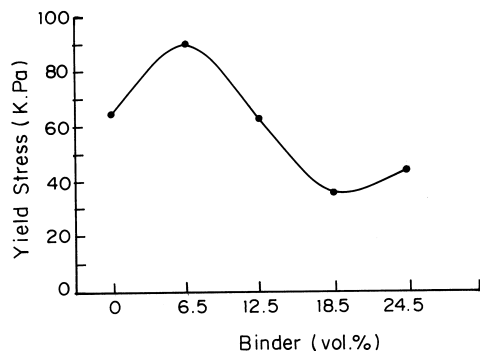


Fig. 5. Yield stress curve of alumina paste at different amounts of boehmite binder.

at this level of binder phase, the torque is maintained for the given extrusion velocity. In all the cases, it was generally observed that the alumina–boehmite paste exhibits Bingham type plastic behaviour and the relationship between $\log \tau$ and $\log \dot{\gamma}$ is a straight line with the slope $n < 1$. It is well known that the boehmite is hydrophilic in nature, a hydrated layer develops on the surface of the boehmite, which gets adsorbed over alumina surface. In an aqueous medium, at controlled pH conditions, boehmite gets converted to a gel, and a gel network is produced surrounding alumina particles. Due to this, a flocculated state of alumina exists, where a long range attractive inter particle van der Waals forces and a short range repulsive hydration forces are present, which ultimately decreases the frictional forces of coarse alumina and increase the degree of plasticity and finally reduced the torque.^{27–29} However, with the increase in boehmite, phase separation associated with agglomeration takes place, resulting in increased torque. Fig. 5 is the yield stress value of alumina containing various amounts of boehmite calculated from the square roots of shear rate ($\dot{\gamma}$) and torque (τ) are plot. The yield stress was calculated by extrapolating the straight-line towards the torque axis. The alumina without boehmite has very high yield stress, i.e. 65 kPa and the addition of boehmite, decreases the yield stress value considerably. Alumina–18.5 vol.% boehmite showed a yield stress value of 36 kPa. Fig. 6 is the apparent viscosity $[\eta]$ of alumina paste at different levels of boehmite binder. Shear dependent flow profiles, characterized as ‘shear thinning’ flow behavior, has been observed.

3.2. Influence of boehmite on alumina solids loading

Fig. 7(a) shows the torque rheology of alumina with and without boehmite at different solids loading. The alumina without boehmite showed a measurable torque range at 49.67 vol.% and gradually increases with increased solids loading. The increase is considerably beyond, when the solids loading is about 51.45 vol.%.

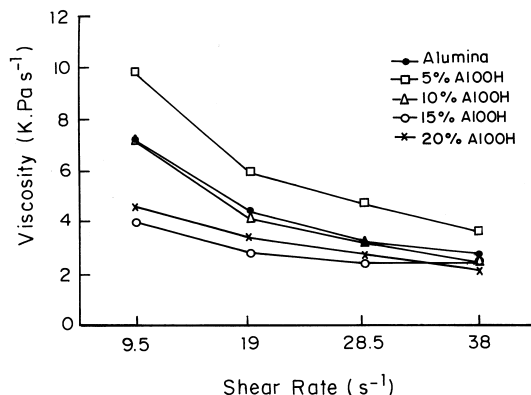


Fig. 6. Viscosity of alumina–boehmite paste.

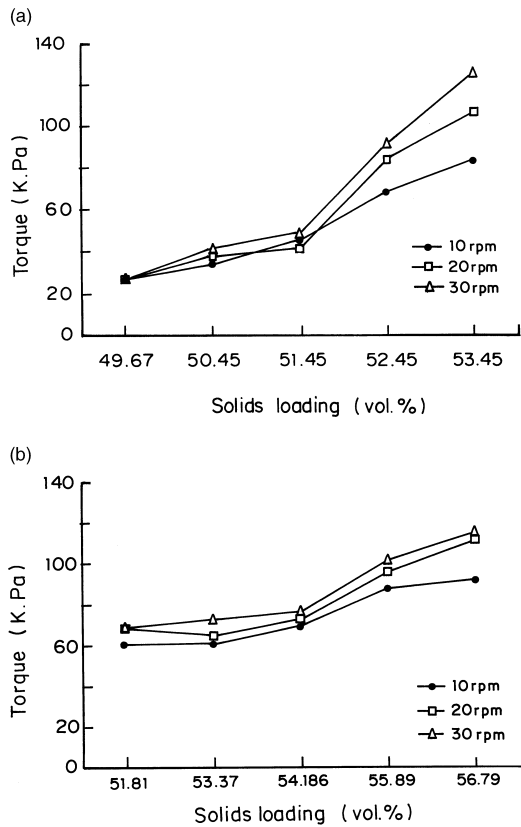


Fig. 7. (a) Torque vs solids loading of alumina paste without boehmite paste; (b) Torque vs solids loading of alumina with boehmite paste.

The addition of 18.5 vol.% of boehmite decreases the torque range and increases the effective solids loading. Fig. 7(b) shows the influence of boehmite on alumina solids loading. Reasonably low torque in the range 70 kPa was observed up to 54.19 vol.% of solids. The adsorption of boehmite on the alumina particles is expected to modify the particle size of the alumina. The particle size distribution of alumina at various levels of addition of boehmite is presented in Fig. 8. The cumulative fine fraction of alumina–boehmite mixture is increased with increase in boehmite content. Alumina containing only 6.5 vol.% has the fine fraction less than $0.346 \mu\text{m}$ is only 48%. On the other hand, alumina containing 18.5 vol.% of boehmite has 63.4% of fine fraction of less than $0.346 \mu\text{m}$. The average particle size of alumina containing boehmite binder above 18.5 vol.% is as high as 87%. It is already reported that the bimodal particle size distribution of alumina slurry results in higher solids loading and it is highly dependent on the critical volume fraction of fine particles.³⁰ In this case, alumina–18.5 vol.% boehmite is found favorable for higher solids loading and particle packing. The fluidity of alumina paste with and without boehmite in Fig. 9 indicates that alumina containing boehmite phase maintains the fluidity reasonably well at higher solids loading compared to alumina without boehmite.

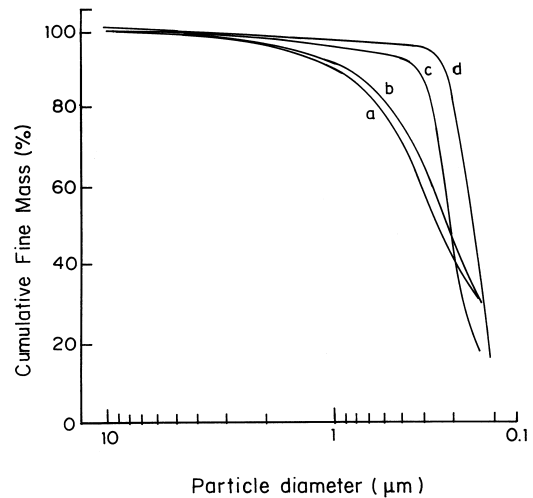


Fig. 8. Particle size analysis of alumina with varying boehmite content: (a) 6.5 vol.% boehmite; (b) 12.5 vol.% boehmite; (c) 18.5 vol.% boehmite; (d) 24.5 vol.% boehmite.

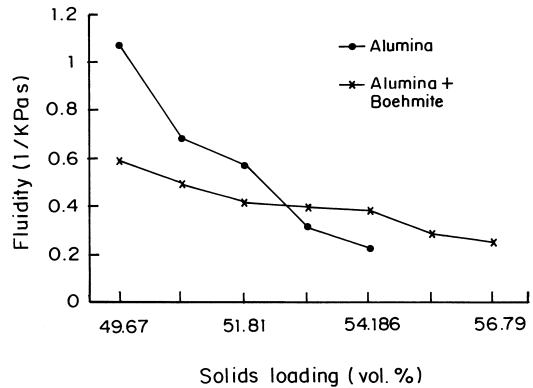


Fig. 9. Dependence of fluidity with and without boehmite at various solids loading.

3.3. Packing characteristics of alumina at different boehmite content

The green density values of extruded alumina with boehmite binder are presented in Fig. 10. Alumina without binder has only 51.6% of theoretical green density and is increased with increasing boehmite content. The maximum theoretical green density of about 61% was observed for samples having 18.5 vol.% boehmite. There is a decreasing trend when the amount of boehmite is increased further. It is known that during extrusion process, ceramic paste is subjected to various types of deformations and complex shear occurs during mass flow of concentrated suspensions.³¹ Normally, it is the binder which controls the extent of shear and maintain the consistency of powder mass throughout the cross section of the die. However, the presence of excess binder, resulted in high shear that limits the flow characteristics of powder mass and lowers the packing

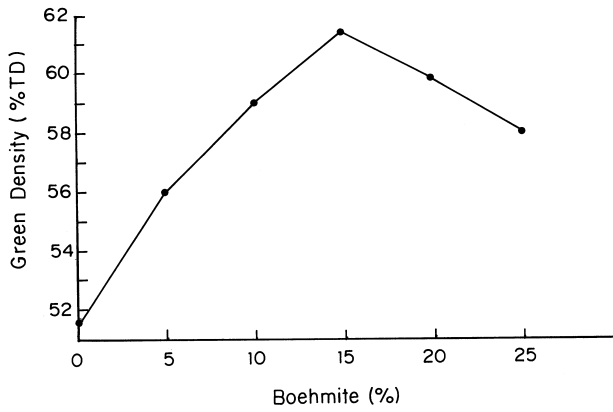


Fig. 10. Green density of extruded alumina shapes.

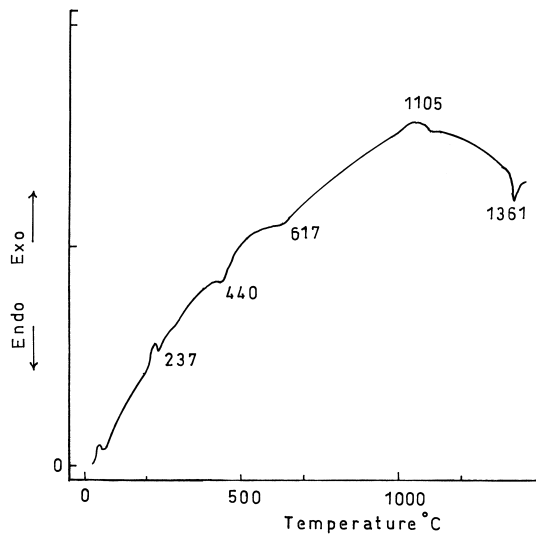


Fig. 11. DTA pattern of alumina–aluminium titanate precursor.

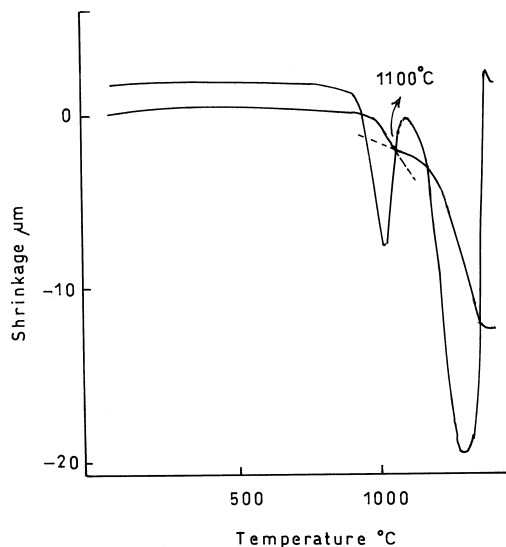


Fig. 12. Shrinkage curve of extruded composite.

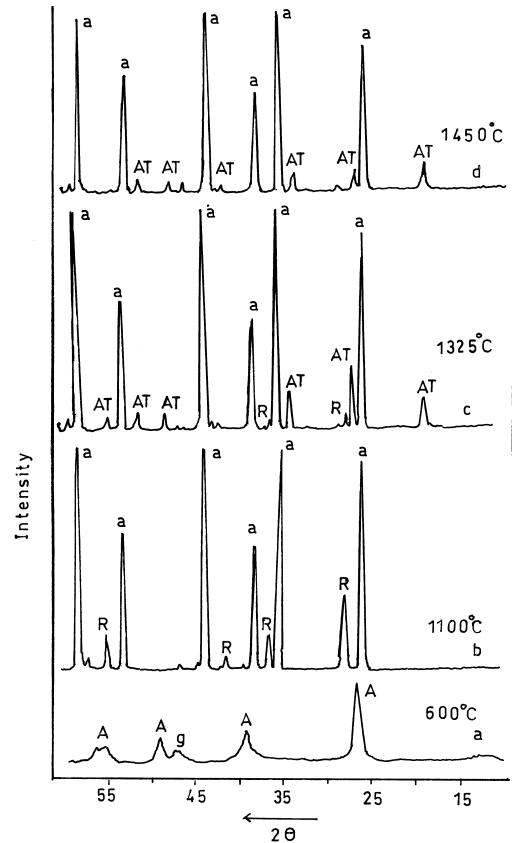


Fig. 13. Evolution of high temperature phases in alumina–aluminium titanate precursor.

efficiency. As was found earlier that the concentration of boehmite up to 18.5 vol.% is adequate to maintain the flow property and to yield optimum fluidity for alumina pastes. At this range, possibly, a long range adhesive force of boehmite exists which is responsible for higher packing and green density. The linear shrinkage of extruded alumina after drying at 45°C, under RH 65% is only <3% indicating the advantage for use as binders for shaping of ceramics.

3.4. Thermal decomposition of composite precursor gel and sintering characteristics

Fig. 11 is the DTA curve of alumina–aluminium titanate precursor up to 1400°C at heating rate 10°C/min. The endotherm present below 200°C is due to evolution of solvated molecules. The one at 440°C is due to the conversion of boehmite to γ -alumina. The endothermic peak at 617°C represents the formation of anatase titania. The exothermic peak at 1105°C is due to conversion of anatase to rutile titania as well as the formation of α -alumina. The transformation of anatase to rutile is known to occur at slightly high temperatures in presence of alumina. The endothermic peak at 1361°C corresponds to the formation of aluminium titanate phase. The shrinkage curve of alumina–aluminium

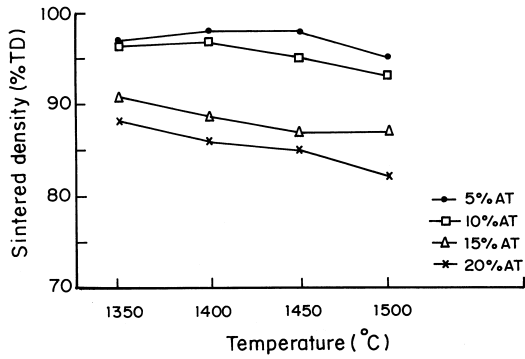
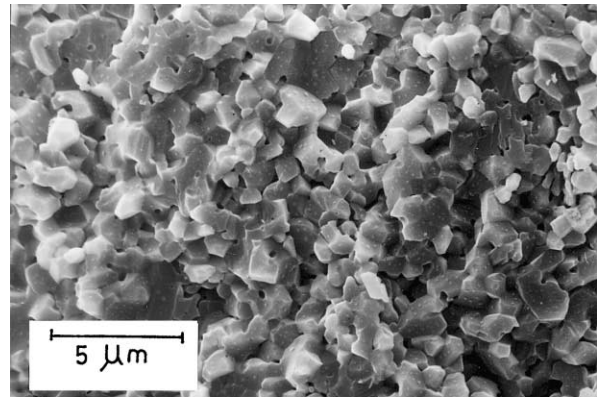
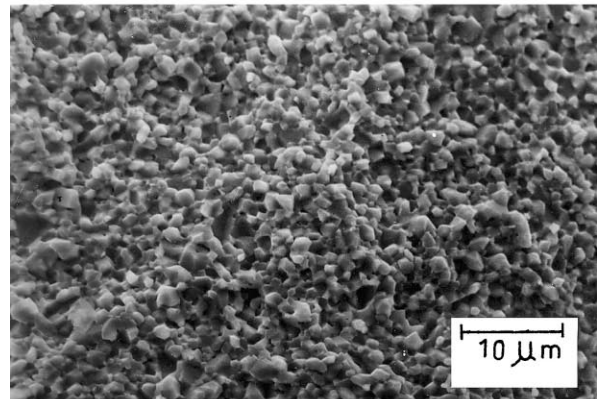


Fig. 14. Sintered density of extruded composite.

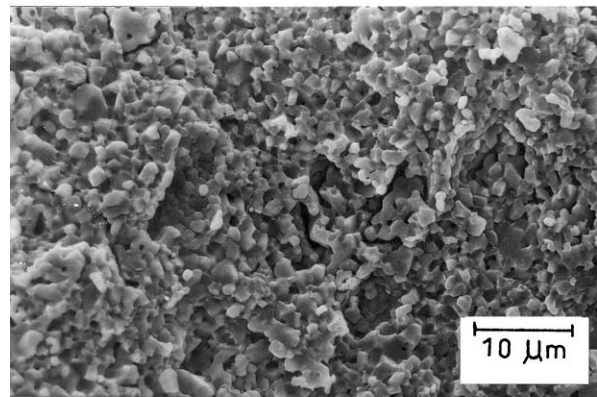
titanate composite rod is provided in Fig. 12. The heating rate was kept at 10°C/min. The densification process occurred in two stages. Shrinkage at 979°C indicates the initiation of densification and proceeds further to ~1100°C. This is due to nucleation of rutile phase as evidenced from the DTA curve. Sintering proceeds up to temperature ~1360°C. Where the reaction between alumina and titania takes place to produce aluminium titanate phase.³² This is further clear from the powder X-ray diffraction patterns presented in Fig. 13. A mixture of alumina and rutile phases is formed from the sol-gel precursor at 1100°C. On increasing the temperature, the matrix becomes rich in alumina, aluminium titanate and traces of rutile. During final sintering phase pure alumina and aluminium titanate phases are observed. Fig. 14 presents the theoretical sintered density of extruded alumina–aluminium titanate composite consisting various percentages of aluminium titanate. The sintered density gradually decreases with increase in aluminium titanate. The sintered density of about 97% TD was obtained for alumina–5 wt.% aluminium titanate sintered at 1350°C which increased to 98.5% on increasing the sintering temperature to 1400°C. On the other hand on increasing aluminium titanate content to 20 wt.%, the sintered density is reduced to 85%. Aluminium titanate is well known to have exaggerated grain growth above 1400°C and undergoes microcracking while cooling. The enhanced sintering kinetics for all the samples is due to nano size precursors as well as thermodynamically unstable intermediate phases. The grain growth appears to be inhibited in presence of boehmite derived alumina. However, as the aluminium titanate increases, the grain growth does not get controlled, as a result of which the aluminium titanate undergoes grain growth and later microcracking. Fig. 15(a) and (b) are the SEM fractographs of alumina and alumina–aluminium titanate composite containing 18.5 vol.% boehmite gel as binder. The fractograph of alumina–boehmite extruded samples sintered at 1450°C



(a)



(b)



(c)

Fig. 15. SEM fractograph of composite: (a) Alumina–boehmite sintered at 1450°C; (b) Alumina–5% aluminium titanate sintered at 1350°C; (c) Alumina–20% aluminium titanate sintered at 1350°C.

indicates varying grain sizes representing alumina and boehmite derived alumina. The boehmite derived grains are as small as 0.5 to 1.0 μm. The smaller grains are uniformly distributed in the alumina matrix. The microstructure also showed intergranular and closed pores. The average grain size of about 2–3 μm was observed for the composites sintered at 1350°C.

4. Conclusions

Alumina–aluminium titanate composite was fabricated using paste extrusion technique. Use of boehmite facilitates elimination of organic vehicle desired for extrusion. The boehmite functions as dispersing medium and when partially flocculated at pH 6.7 could be used as binder for extrusion. Incorporation of about 18.5 vol.% boehmite resulted in low torque and imparted fluidity for extrusion, which provides higher packing and green density of about 61%. On the other hand, the dispersion of sol–gel aluminium titanate precursor as second phase resulted in highly dense composite body at low temperature. Alumina upto 10 wt.% of aluminium titanate showed the theoretical sintered density of 97% at 1350°C. The presence of transition alumina and partially stable titania phases increase the densification behaviour of alumina–aluminium titanate composites. The boehmite gel not only provides higher green strength but also acts as sintering aid for alumina matrix composites. The sintered microstructure shows uniformly distributed extremely fine grains of the size 2–3 µm.

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