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Journal of the European Ceramic Society 31 (2011) 167–173

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Role of organic additives on non-aqueous tape casting of SiAlON ceramics

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Received 2 June 2009; received in revised form 19 July 2010; accepted 24 July 2010
Available online 15 September 2010

Abstract

Suspensions consisting of precursor α/β SiAlON forming powders, azeotropic solvent mixture of 60 MEK/40E, dispersant, binder, and plasticizer were optimized for tape casting by rheological measurements and tape properties. Sodium tripolyphosphate (STPP) was introduced as a dispersant for low temperature applications of α/β-SiAlONs. Optimum STPP amount was determined as 0.012 g/m^2 (of the particle surface) for stable α/β-SiAlON suspensions. Different amounts of binder/plasticizer mixtures were added to the slurries and the effects on rheological and green tape properties were investigated. Green tapes with dibutyl phthalate (DBP), and plasticizer mixture, poly(ethylene glycol) (PEG) and DBP, exhibited centered cracks with high plasticity, on the other hand, polyvinyl butral (PVB) and PEG showed no crack but low plasticity. Therefore, many different parameters were found to be effective on final tape properties. In addition, tapes were prepared with 6 vol% PVB + PEG, sintered at 1800 °C for 2 h and exhibited almost 97%TD in room temperature applications of α/β SiAlONs.

Keywords: α/β SiAlON; Suspension; Tape casting; Plasticity

1. Introduction

Silicon nitride (Si_3N_4) is one of the structural ceramics for high temperature structural and tribological applications. Si_3N_4 exhibits excellent oxidation resistance as a result of the presence of a protective oxide layer.[1,2] SiAlON ceramics are structural group formed by adding AlN, MgO, BeO, Y_2O_3 and other metal oxides such as Yb_2O_3, Nd_2O_3 and Sm_2O_3 to a Si_3N_4 matrix. The best known SiAlON phases, α and β have structures based on α- and β-Si_3N_4 with aluminum and oxygen partially replacing silicon and nitrogen, respectively.[3,4] α-SiAlON has a general formula of $M_xSi_{12-(m+n)}Al_{(m+n)}O_nN_{16-n}$, where M is a metal ion typically Li, Mg, Ca, Y and Ln.[5] On the other hand, β-SiAlON, does not incorporate with other cations. The composition of β-SiAlON is given by the formula as $Si_{6-z}Al_zO_zN_{8-z}$, with z varying from 0 (i.e., Si_3N_4) to a maximum value of about 4.2 depending on sintering conditions.[6]

Conventionally equiaxed grains of α-SiAlON give a high hardness while elongated β-SiAlON grains can provide a high toughness to the structure. In addition recently, some progress on elongated α-SiAlON grains have opened up a new possibility to obtain SiAlON ceramics with both high hardness and toughness.[7,8] Chen and Rosenflanz[8] have successfully produced α-SiAlON with whisker-like microstructures by GPS (gas pressure sintering) under 1950 °C in rare earth SiAlON system by using β-Si_3N_4, instead of the more conventional α-Si_3N_4 as starting powder. Therefore, the final properties can be determined by tailoring the microstructure and α/β ratios in a SiAlON-based ceramic system.[9] The mixing of these two phases enables one to reach novel mechanical properties. Furthermore, α/β-SiAlON ceramics can be densified easily according to their α and β-SiAlON ratios.[10,11] The temperature properties of α/β-SiAlON ceramics are better than those of the individual α and β-SiAlON ceramics. On the other hand, the properties of α/β-SiAlON ceramics change over 1400 °C and they exhibit some problems due to grain boundary phases and α to β phase transformation as reported by Mandal et al.[12,13] To date, SiAlON-based ceramics have been fabricated by several methods including dry pressing, hot isostatic pressing,[12–14] and lately tape casting.[15] Among these fabrication methods, tape casting is a relatively new method for SiAlON ceramics.

Tape casting is a low cost and a useful process for preparing thin ceramic sheets which are utilized to fabricate multilayer capacitors, solid electrolytes, and solid oxide fuel cells, etc. For

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doi:10.1016/j.jeurceramsoc.2010.07.034

structural materials, tape casting has already been used to design laminated and multilayered composites to improve mechanical properties. In addition, recently Acikbas et al reported utilization of tape casting to produce functionally graded SiAlON ceramics.[16] Ferreira and co-workers used tape casting to produce α-SiAlON tapes.[17] Our group also used tape casting to produce functionally graded SiAlON ceramics.[18] For this understanding to develop a formation for tape casting of SiAlON-based ceramics is very important. To exploit the utilization of tape casting for production of SiAlON-based ceramics with a controlled and tailored microstructure, the effects of polymeric additions (binder and plasticizer) on rheological properties of non-aqueous SiAlON suspensions should be well understood. In addition, a new and cheap class dispersant, STPP was used in this study as a dispersing agent. Even though, STPP is harmful for high temperature mechanical properties of SiAlON ceramics due to its Na content by lowering glass melting temperature, it is not harmful for low temperature applications of SiAlONs. This study is mainly concentrated on the additive effects of SiAlON slurries and tape structures for low temperature applications of SiAlONs such as cutting purposes, cutting textile threads, cutting blades for winding machine and as well as for thread-guiding, forming and modifying yarns, seal and regulator disc for sanitary fittings and diverters related with hardness, toughness, wear, and thermal shock properties.[19] In this study, functionally graded α/βSiAlON structures containing both high hardness and toughness were also aimed to produce by tape casting for the low temperature applications of SiAlONs.

2. Experimental procedure

In this study, an azeotropic mixture of 60 vol% methyl ethyl ketone (MEK, Carlo Erba, Italy) and 40 vol% ethanol (E, Merck, Germany) was chosen as solvent. This azeotropic mixture is commonly used in non-aqueous systems because medium polar solvents like the azeotropic mixture are compatible with many commonly used dispersant and binders.[20]

Si_3N_4, AlN and Y_2O_3 were used to form α/β-SiAlON. The characteristics of these oxides and other components of the tape casting slurry are presented in Table 1. Sodium tripolyphosphate, $Na_5P_3O_{10}$, with molecular weight 367.86 g by Esan, Turkey was tested as a new and cheap class dispersant for non-aqueous solvents. Polyvinyl butral (PVB, Butvar B98, Solutia, USA), with molecular weight of 40,000–70,000 g was utilized as a binder. Poly ethylene glycol 400 (PEG 400, Merck, Germany) with molecular weight of 380–420 g, dibutyl phthalate (DBP, Merck, Germany), with molecular weight of 278.34 g, and mixtures of DBP and PEG were employed as plasticizers.

The starting powders were mixed according to a batch formula of 84.5 wt% Si_3N_4, 8.7 wt% AlN, and 6.8 wt% Y_2O_3 to form a final composition of 85 wt% α and 15 wt% β-SiAlON. The powder mixtures were planetary milled for 2 h in isopropyl alcohol, using SiAlON jars and SiAlON media with solid loading of 30 vol%. The milled powder mixtures were dried in an oven at 70 °C for 2 h. The powders were crushed and sieved through 150 μm sieve.

For sedimentation tests, 15 ml glass tubes covered with lids were used to test sedimentation behavior of 3 vol% α/β-SiAlON suspensions with different amounts of STPP between 0 and 0.036 g/m^2. The aim of this sedimentation experiment was to determine the adequate amount of dispersant for α/β-SiAlON forming powders. The position of the sedimentation height was monitored and recorded at regular time intervals.

Once the optimum dispersant amount was determined, the powder mixtures were milled with the addition of 0.012 g/m^2 STPP. This chemically aided milling operation with the mixtures of the powders, solvent (i.e., ethanol and MEK) and dispersant is known as dispersion milling.[20] During dispersion milling, the dispersant was expected to be adsorbed on to powder surfaces so that reagglomeration of the particles can be prevented. Three SiAlON powders (Si_3N_4, AlN, and Y_2O_3) were dispersion milled up to 5 h, and every hour particle size was measured. After dispersion milling, plasticizer was added to slurries and ball milled for 3 h, finally, the binder was added and the suspension was mixed for an additional 15 h. Binder and plasticizer were added equally into the suspensions in amounts of 3 and 15 vol%. Suspensions were prepared at solid loadings of 25–35–50 and 59 vol% max.

The rheological behavior of the slurries was investigated by using a rheometer (Haake K20, Germany), capable of operating at both controlled shear rate and controlled shear stress conditions. The measurements were performed by using a concentric cylindrical system, with a rotor diameter of 18 mm. Viscosity and shear stress changes were measured by applying the shear rate intervals between 50 and 550 s^{-1} with shear rate ramp of 5 s^{-1} in 5 min.

Tape casting of the slurries was performed by using a homemade tape casting machine containing a stationary reservoir with a tempered glass substrate that moves at a controlled speed. Blade height was determined to be 400 μm with casting speed as 4 cm/s ($\dot{\gamma} = 100\,s^{-1}$). Tapes were sintered at 1800 °C for 12 h with pressure sintering. The microstructures of green and sintered bodies were examined by Zeiss Supra 50 VP scanning electron microscope (SEM).

3. Results and discussions

3.1. Sedimentation

Slurry stability was evaluated by measuring sedimentation behavior of powder for different amounts of the dispersant (STPP, 0–0.036 g/m^2) in glass tubes. Fig. 1(a) and (b) shows the results of the sedimentation tests and sedimentation velocities, respectively, for α/β-SiAlON suspensions after 1 month. Some of the separation interfaces between sediment and supernatant liquid were sharp and moved downward in time. This sedimentation behavior was typical for flocculated suspensions. At the moderate amount of STPP level (0.012 g/m^2), particles were settled very slowly and were packed very closely. A cloudy supernatant was observed at 0.012 g/m^2 STPP. It preserved its cloudy state for 3 weeks. On the other hand, at the different STPP additions which were lower and higher than this STPP value (0.012 g/m^2), very clear supernatants were observed. The sedi-

Table 1
The properties of used materials.

Powder	Purity	Supplier	D_{50} (μm)	Density (g/cm^3)
Si_3N_4	1.4 wt% surface oxide	UBE (E-10)	0.98	3.29
AlN	1.6 wt% surface oxide	H-Type, Tokuyama, Japan	1.2	3.3
Y_2O_3	99.999% pure	H.C, Starck, Germany	0.39	5.01
STPP	98% pure	Esan, Turkey	–	0.4
PVB	40,000–70,000 g (molecular weight)	Butvar B98, Solutia, USA	–	1.07
PEG	380–420 g (molecular weight)	PEG400, Merck, Germany	–	1.2
DBP	278.34 g (molecular weight)	Merck, Germany	–	1.04

mentation behavior of the 0.012 g/m^2 STPP containing sediment suggested well dispersion. Furthermore, the cloudy supernatant was also a sign of balanced forces of small particles due to Brownian motion. At amounts higher than the optimum value (i.e., 0.012 g/cm^3), the dispersant showed adverse effect such as formation of agglomerates, high gravitational forces and quicker sedimentation. At lower dispersant level lower than the 0.012 g/m^2, the dispersant was not enough to fully cover all the particle surfaces and provide enough repulsive forces resulting once in quicker sedimentation and agglomeration.[21]

3.2. Rheology

The composition of the suspensions with 59 vol% solid loading and 6 vol% polymer is given as an example in Table 2. The rheological behavior of the slurries should meet the needs for tape casting process.[23] The slurry viscosity decreases due to shear forces, and immediately behind the blade the viscosity rapidly increases again. This suppresses uncontrolled flow and prevents sedimentation of ceramic particles.[20–22] When glass substrate contacts with ceramic slurry, the slurry must be fluid enough to flow uniformly and subsequently regain enough viscosity under static conditions to keep its shape for tapecasting process. It is suggested that the slurry should have little or non-thixotropy with shear thinning or pseudoplastic behavior.[20,23] The characteristic of shear thinning or pseudoplastic behavior is such that as shear rate, increases viscosity gradually decreases. This principle is applied under the blade or easy flow of slurry with applied shear stress.[20–22]

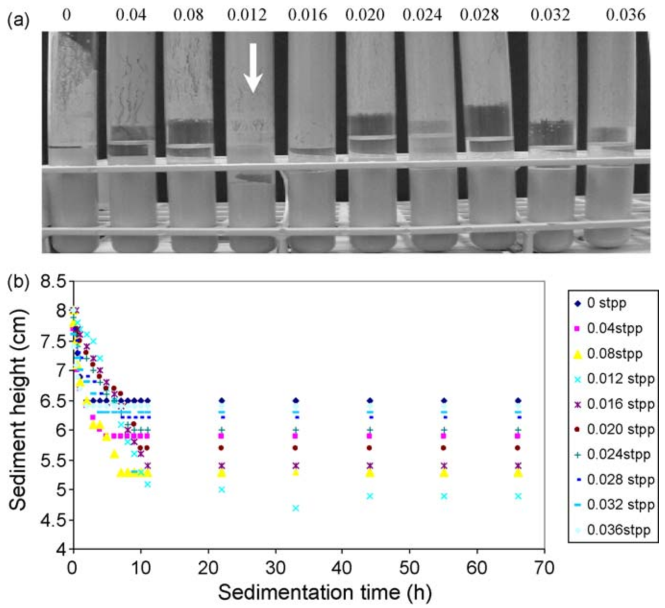

Fig. 1. (a) Sedimentation tubes at different dispersant (STPP) additions from 0 to 0.036 g/m^2 and (b) sedimentation velocity behavior at different dispersant additions.

Fig. 2(a) and (b) shows the viscosity vs. shear rate and the shear stress vs. shear rate curves of suspensions with solid loading at 25 vol% as a function of PVB + PEG for 3–15 vol% in equal amount. Increasing PVB + PEG, resulted any increase in viscosity and shear stress. At shear rates of 100 and 500 1/s, viscosities at 15 vol% polymer content were found to be 20 and 15 mPa s, respectively, and shear stress values were determined as 1 and 8 mPa, respectively. At this solid loading, suspensions showed shear thinning behavior with thixotropy. By increasing polymer ratio, shear thinning behavior became more consistent with thixotropy. However, the slurries at this solid loading (25 vol%) were tape cast and all tapes from these slurries were failed due to very low solid loading and hence loosely packing of powder with very low green density. Even the maximum added polymer could not manage to bind the individual grains.

Fig. 3(a) and (b) shows the viscosity vs. shear rate and shear stress vs. shear rate curves for different amounts of PVB + PEG (3–15 vol%) at 35 vol% solid loading, respectively. The suspensions at this solid loading could be characterized by shear thinning behavior at low shear rates followed by a trend to constant viscosity at high shear rates with little thixotropy. At shear rates, 100 and 500 1/s, viscosities at 15 vol% (PVB + PEG) were found to be 40 and 28 mPa s, respectively, and shear stress values were determined as 2 and 9 mPa, respectively. The tapes of these slurries were observed to be better than the tapes from the 25 vol% solid containing slurry but both tapes were not strong enough for handling. In tape casting slurries, solid loading is fairly important.[20]

Table 2
One of the composition for used suspensions for 59 vol% solid loading.

Composition	Function	vol%
Si_3N_4, AlN, Y_2O_3	Powders	59
STPP	Dispersant	1
PVB	Binder	3
PEG, DBP and mixture of both	Plasticizer	3
Azeotropic mixture of 60MEK/40E	Solvent	34

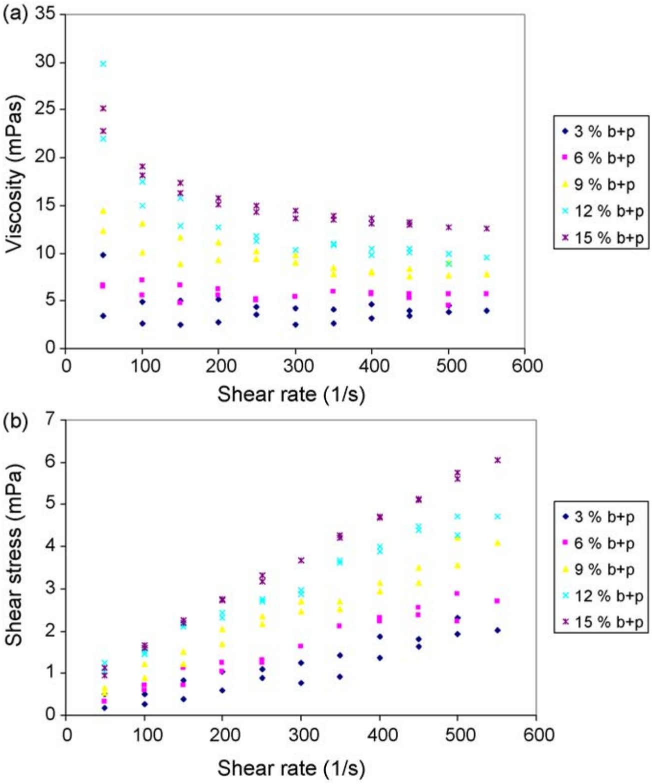

Fig. 2. (a) Viscosity and (b) shear stress changes according to shear rate at solid loading 25 vol% with different amounts of binder (PVB) + plasticizer (PEG) from 3 to 15 vol%.

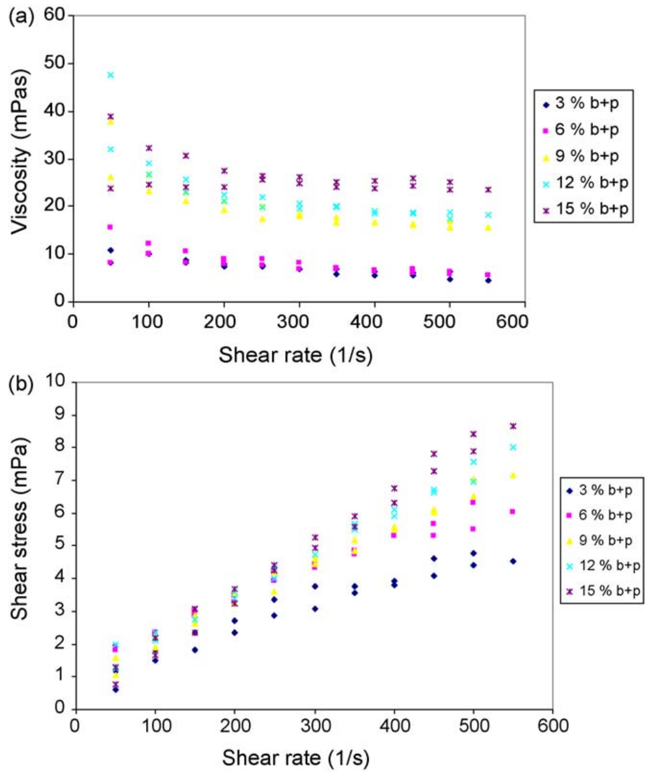

Fig. 3. (a) Viscosity and (b) shear stress changes according to shear rate at solid loading 35 vol% with different amounts of binder (PVB) + plasticizer (PEG) from 3 to 15 vol%.

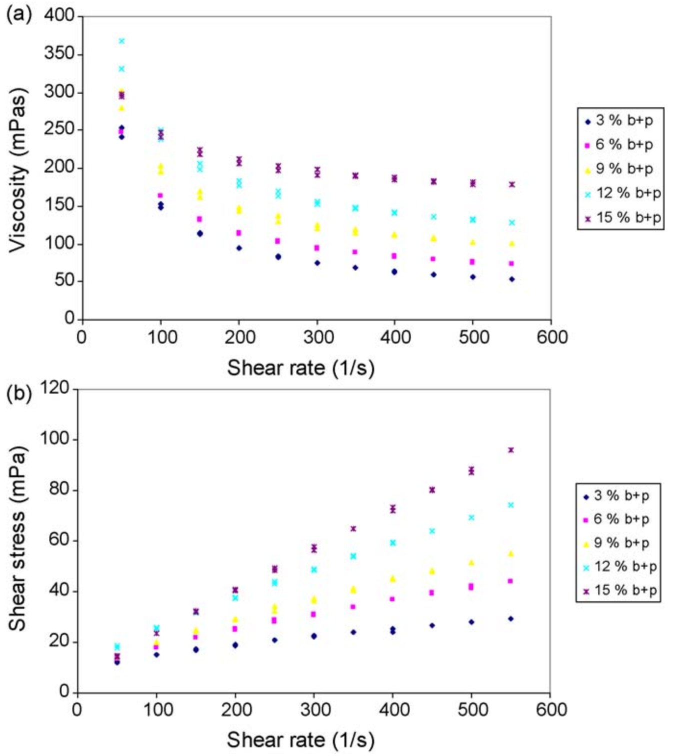

Fig. 4. (a) Viscosity and (b) shear stress changes according to shear rate at solid loading 50 vol% with different amounts of binder (PVB) + plasticizer (PEG) from 3 to 15 vol%.

Fig. 4(a) and (b) shows the viscosity vs. shear rate and the shear stress vs. shear rate curves for the same amounts of PVB + PEG (3–15 vol%) with solid loading as 50 vol%. These suspensions also showed shear thinning and little thixotropy. At shear rates, 100 and 500 1/s, viscosities for the highest polymer content were found to be 280 and 200 mPa s, respectively and shear stress values were determined to be 10 and 95 mPa, respectively. At high polymer content, viscosity and shear stress increased probably due to bridging flocculation of polymers. After 9 vol% polymer, all tapes were found to be in good conditions with respect to samples containing less amount of solid.

Since higher solid loading results in less sintering shrinkage,[20] solid loading was increased to 59 vol% in the current α/β-SiAlON suspensions. Fig. 5(a) and (b) shows the viscosity vs. shear rate and shear stress vs. shear curves for this solid loading max. These suspensions showed two characteristic properties of tape casting slurries as well as shear thinning and little thixotropy. At shear rates, 100 and 500 1/s, viscosities at 15 vol% polymer were found to be 350 and 250 mPa s respectively and shear stress values were determined as 20 and 110 mPa, respectively. After addition of 6 vol% polymer (binder + plasticizer), good tapes were obtained. Although, strong and uniform tapes were obtained, plasticity of tapes was not adequate for further processing steps (e.g., lamination). Low plasticity effect of PEG could not manage to loose the chain structure of PVB.

There are many studies showing that dibutyl phthalate, DBP has more effects on loosing the chain structure of PVB.[24] Therefore, a new plasticizer system, DBP and a combination of dibutyl

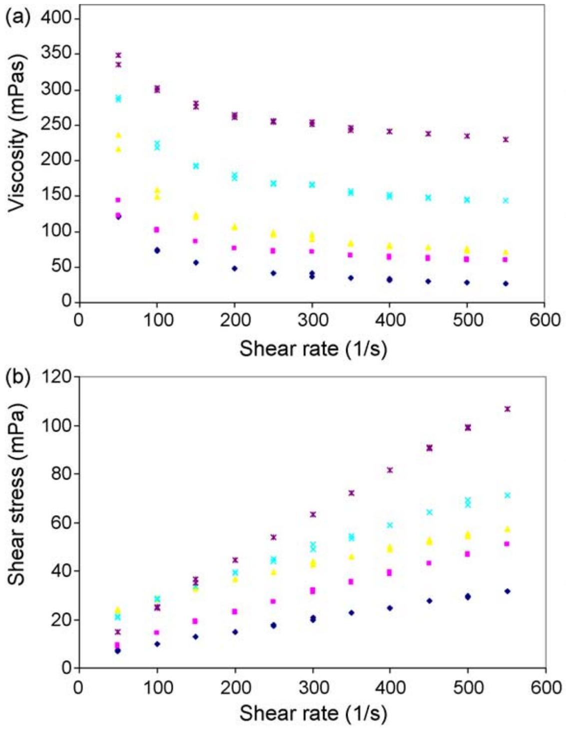

Fig. 5. (a) Viscosity and (b) shear stress changes according to shear rate at solid loading 59 vol% with different amounts of binder (PVB) + plasticizer (PEG) from 3 to 15 vol%.

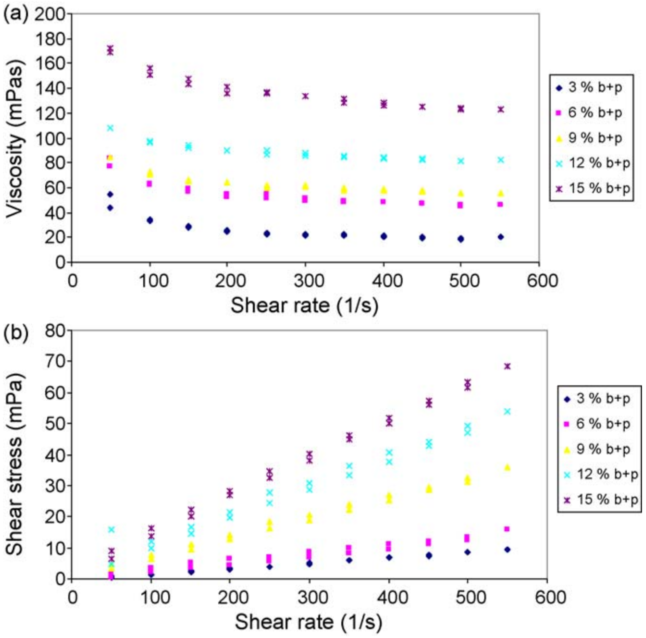

Fig. 6. (a) Viscosity and (b) shear stress changes according to shear rate at solid loading 59 vol% with different amounts of binder (PVB) + plasticizer (DBP) from 3 to 15 vol%.

phthalate, DBP and poly(ethylene glycol), PEG were used with binder (PVB). There are some studies about the usage of the combination of PVB and DBP.[20,24] Phthalates have an aromatic group and around this there are two aliphatic chains. The plasticity of the tapes was expected to increase prior to using DBP increased. This behavior is mainly due to the aromatic structure of DBP against aliphatic structure of PEG.[20] Fig. 6(a) and (b) shows the changes of viscosity and shear stress by shear rate at different PVB + DBP from 3 to 15 vol% with solid loading at 59 vol% max. According to the viscosity curves, suspensions behaved as shear thinning with viscosity reduction by increasing shear rate. At shear rates, 100 and 500 1/s, viscosities at the highest polymer content were found to be 180 and 160 mPa s, respectively and shear stress values were determined as 15 and 70 mPa, respectively. The viscosity decreased significantly in the PVB/DBP system due to the presence of DBP. It was thought that the addition of DBP caused a decrease in the molecular weight of the polymeric system by loosing PVB chains. DBP used suspensions showed shear thinning with Newtonian dominant flow type behavior. All tapes from these suspensions came out in bad conditions. It was observed that many centered cracks were placed on the tape surfaces and tapes were stacked on the glass carrier. However, these DBP containing tape-like structures were provided better plasticity than the PEG containing system by many cycling bending. In the DBP containing system, we observed that tape-like structures showed better plasticity than the PEG system.

The combinations of plasticizer (DBP, PEG) systems were also used for good plasticity tapes at highest solid loading (59 vol%). Mixtures of PEG and DBP were added in equal amounts from 3 to 15 vol%. Fig. 7(a) and (b) shows the behavior of viscosity and shear stress according to shear rate. These suspensions also showed shear thinning with Newtonian dominant region and little thixotropy. At shear rates, 100 and 500 1/s,

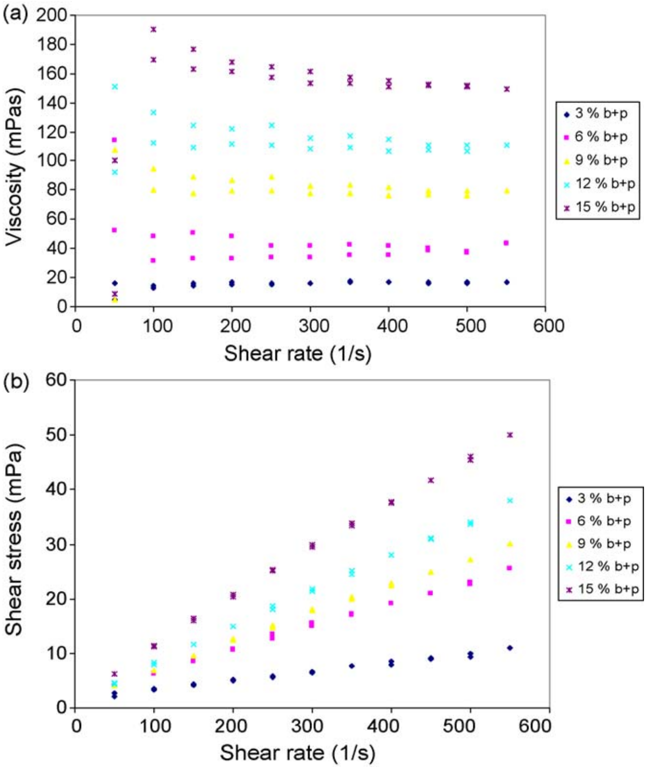

Fig. 7. (a) Viscosity and (b) shear stress changes according to shear rate at solid loading 59 wt% with different amounts of binder (PVB) + plasticizer mixtures (PEG + DBP) from 3 to 15 wt%.

Table 3
The properties of slips and green tapes for 59 vol% solid loading.

Binder + plasticizer ratio (vol%)	Green tape density (g/cm^3)	Green tape thickness (μm)
3	1.61	290
6	1.58	288
9	1.59	300
12	1.61	297
15	1.61	293

viscosities at the highest polymer content were found to be 200 and 160 mPa s, respectively and shear stress values were determined as 8 and 50 mPa, respectively. The tapes from these suspensions showed centered cracks on the tape surfaces as the DBP added tapes and these tapes were also stacked on the glass carrier. The consistency of these cracks was found to be less than the DBP added tapes. Furthermore, the plasticity of these tape-like structures was also found to be better than that of PEG system. However, we had only small pieces of tape-like structures.

3.3. Green and sintered tape characteristics

The total added polymer provides a further characterization of green, binder-burn-out and sintered tapes.[18] Tapes were cast at 4 cm/s ($\dot{\gamma} = 100\,\mathrm{s}^{-1}$) with blade height of 400 μm. The tape thickness was obtained by observation with optical microscope. Densities of tapes were measured via geometrical method by weighing 1 × 1 cm square samples. Table 3 shows the characteristics of the produced tapes for 59 vol% solid loading.

The differences between green density and thickness were found due to the drying behavior of tapes. Drying process is consisting of two stages.[20] The first is controlled by capillary migration of the solvent and the second is controlled by solvent diffusion through the solidified part of the film with a decreasing drying rate. According to some authors, the first stage is very short.[18] In the early periods of the first drying stage, particles approach each other, the viscosity of fluid phase is low enough to allow particles rearrangement, avoiding internal stresses and the shrinkage occurs perpendicularly to casting direction with balancing solvent loss. Evaporation proceeds, the viscosity increases. In the second stage, the internal stresses increase due to much evaporation rate and finally the cracks are observed. At low polymer ratios, internal stresses cannot be balanced and more shrinkage takes place in the tape structure.[20] At the same time, the green strength of the tapes is not enough for easy handling. All these factors are affecting the final properties of the produced tapes.[18,20] Fig. 8(a)–(c) show scanning electron microscopy (SEM) structures of green, binder-burn-out and sintered tapes, respectively. These microstructures were found to show different amount of packing. One could clearly see the effects of binder on packing. After binder-burn-out, structure was obtained to contain more porous structure. Fig. 8(c), exhibited equiaxial and elongated α-SiAlON and elongated β-SiAlON grains. Density of the sintered tape shown in Fig. 8(c), was found to be 3.11 g/cm^3 (97% theoretical).

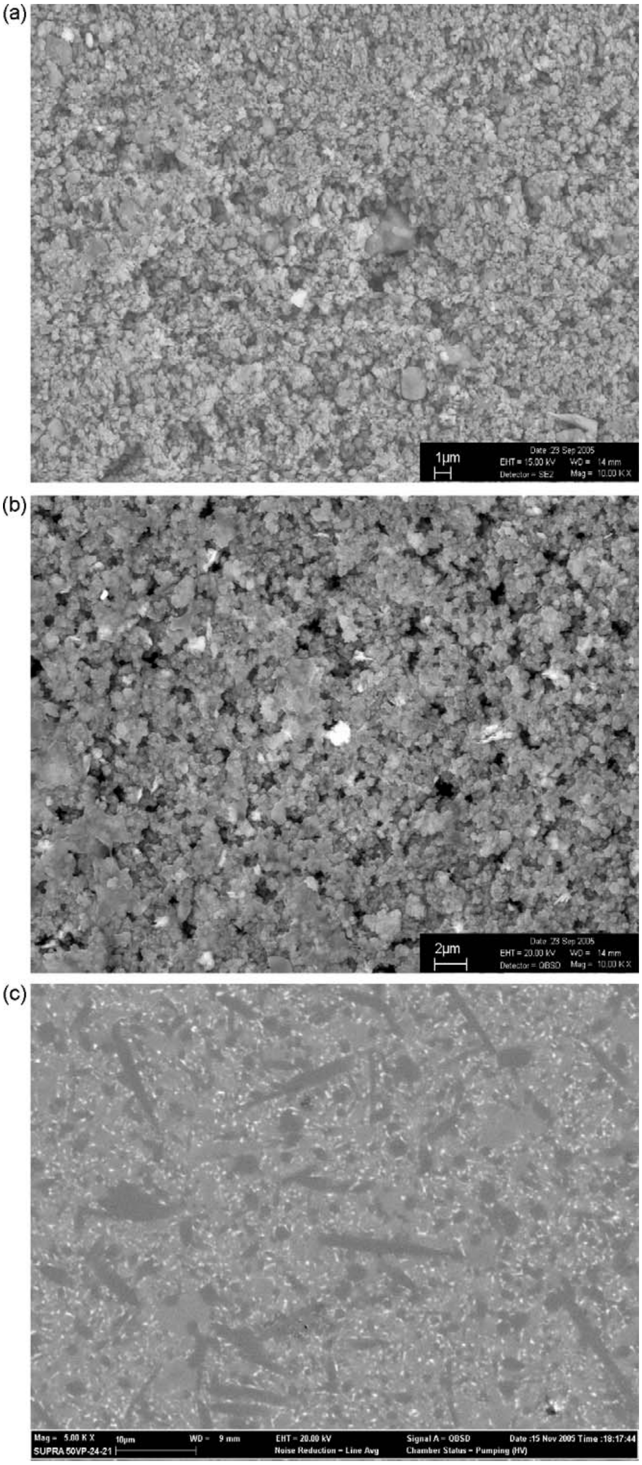

Fig. 8. SEM micrographs of (a) green tape with binder ratio as 6 vol% (b) microstructure of binder-burn-out tape, and (c) the surface of sintered tape.

4. Conclusions

In this work, the effects of the organic additives on rheological and green tape properties have been investigated. In the composition of α/β-SiAlON, the powder mixture as Si_3N_4, AlN and Y_2O_3 was dispersed in 60 MEK/40E azeotropic mixture. A new and cheap class of dispersant (STPP) in non-aqueous system was used. In addition, α/β-SiAlON forming powders were managed to be dispersed well in non-aqueous solution. Optimum content of STPP for a stable suspension was determined as 0.012 g/m^2 and was found to be one of the choices for α/β-SiAlON suspensions in good dispersion; even though, STPP is harmful for high temperature mechanical properties of SiAlONs by lowering glass melting temperature, this study was mainly concentrated on low temperature applications of SiAlONs.

Suspensions with PVB/PEG systems showed shear thinning and little thixotropy or no thixotropy. The tapes from these combinations were found to be good had limited plasticity. In addition, the best binder + plasticizer combination for α/β-SiAlON suspension was found to be PVB and PEG system. The PVB/DBP and PVB/PEG-DBP systems showed shear thinning with Newtonian dominant region and little thixotropy. The tapes from these suspensions showed good plasticity but it was difficult to remove these tape-like structures from the substrate. Furthermore, DBP and PEG + DBP mixture plasticizer systems with PVB can be studied by changing some parameters such as tape casting, carrier properties and drying characteristics to get better tapes.

The green density was found to be higher than 1.6 g/cm^3 (50%TD) with casting speed as 4 cm/s ($\dot{\gamma} = 100\,\mathrm{s}^{-1}$) with blade gap 400 μm. Sintered tape was found to have aquiaxial and elongated α-SiAlON and elongated β-SiAlON. Almost 97%TD was achieved after sintering of the tapes (3.11 g/cm^3).

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