

# Mechanical and Lamination Properties of Alumina Green Tapes Obtained by Aqueous Tape-casting

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## Abstract

*Mechanical characterisation and lamination were carried out on alumina green tapes prepared by aqueous tape casting using two acrylic emulsions having different glass transition temperatures ( $T_g$ ) as binders. The tensile strength and strain were strongly dependent on the binder nature and content. Namely, the mechanical properties of the green tapes reflected those of the binders at room temperature: the green tapes obtained with the higher  $T_g$  binder showed a brittle behaviour, whereas those obtained with the lower  $T_g$  binder showed an elastoplastic behaviour. The mechanical properties of the green tapes prepared by mixing the two acrylic binders lies in between, giving the possibility of tailoring the flexibility and strength in the range of the values obtained for pure binders. Lamination gave rise to an increase of both green and sintered densities, compared with monolayer specimens, whatever the composition of the binder system. Such improvements significantly depended on lamination pressure, but were insensitive to lamination temperature for the two temperatures tested higher than the  $T_g$  of the two binders. © 1999 Elsevier Science Ltd. All rights reserved*

**Keywords:** lamination, tape casting, mechanical properties, strength,  $Al_2O_3$ .

## 1 Introduction

Tape casting is the most widely used technique for large-scale fabrication of green sheets for multilayer

ceramic packaging technology.<sup>1–10</sup> The tape cast green sheet forms the basic building block of these complex structures and must meet several requirements during fabrication to enable final high quality and reliable products. More specifically, the green tapes must possess dimensional stability and mechanical integrity during punching, thick film paste screening. Furthermore, good bonding characteristics between layers are required to ensure good adhesion in lamination and to avoid delamination during sintering.<sup>1</sup> These conditions are usually better achieved by using organic-based systems.<sup>11,12</sup> However, these organic solvents are volatile and irritating, and necessitate special precautions concerning inflammability and toxicity.<sup>9,10,12</sup> These drawbacks brought to an increasing pressure over the traditional tape casting process and have been encouraged the research towards alternative aqueous-based systems which are lower cost and environmentally more friendly.<sup>12–22</sup>

The major disadvantage that has been pointed out for tape-casting in water is the slower drying rate to respect organic solvent.<sup>12</sup> Further, slurry formulations for water-based tape casting often reveal a certain pH instability, high tendency to foaming and premature polymerisation.<sup>7</sup> Therefore, producing suitable green tapes strongly depends on a strict control of all these variables, making the slurries' formulation for water-based tape casting process much more problematic than when the solvent is an organic liquid.<sup>12</sup>

The organic binders for aqueous tape casting can be either dissolved or dispersed in water as an emulsion.<sup>8,13</sup> Among the various formulations proposed in literature to accomplish tape casting in water, those using acrylic emulsion binders seem to give the best results in terms of surface finishing,

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strength and uniformity of the green tapes. For instance, acrylic emulsion binders were recently and successfully used to produce aqueous tape-casting of alumina,<sup>15–19</sup> mullite,<sup>20</sup> silicon carbide,<sup>21</sup> and yttria-stabilised zirconia.<sup>22</sup> Further, the great sensibility towards suspension stability usually experienced in water-based slurries is somewhat reduced, since the only organic components included in such formulations are dispersant and acrylic emulsion binders. Proper combinations of acrylic emulsion binders with different  $T_g$ , indeed, allow obtaining green tapes with different degrees of flexibility without using plasticiser agents. Further, no antifoaming agent is usually needed, due to the high wettability of such polyacrylic binders onto the most part of ceramic powders.

Although the mechanical properties of green tapes prepared by both non-aqueous and aqueous systems have been discussed in the literature<sup>8,23–29</sup> only few reports are concerned with emulsion binder systems.<sup>28</sup> The present work aims to evaluate the mechanical properties and lamination behaviour of alumina green tapes obtained in water using two pure acrylic emulsion binders, and mixed in several proportions. The effects of the  $T_g$  of the binders, and their amount and composition have on (i) tensile strength/strain to rupture of green sheets, (ii) lamination behaviour and (iii) green/sintered densities are also investigated.

## 2 Experimental Procedure

### 2.1 Materials and slip preparation

A commercial alumina powder A16 SG (Alcoa, USA) with a mean particle size of  $0.4\ \mu\text{m}$  and a specific surface area of about  $10\ \text{m}^2\ \text{g}^{-1}$  was used. The dispersant used to stabilise the alumina suspensions was a low molecular weight ammonium polyacrylate salt solution (Duramax D-3005, Rohm and Haas, Philadelphia, USA), a surface active agent expected to disperse through an electrosteric mechanism. The binders were two stable acrylic emulsions dispersed in water, Duramax B-1035 and Duramax B-1050 (Rohm and Haas, Philadelphia, USA). The  $T_g$  and latex concentration of these binders are  $10^\circ\text{C}$ , 55 wt% for the B-1035, and  $-40^\circ\text{C}$ , 49 wt% for the B-1050.

A stock suspension containing 83 wt% solid loading, was prepared by adding the alumina powder to distilled water containing the optimal amount of dispersant (0.3 wt% based on dry weight alumina powder).<sup>15</sup> Then, mechanical stirring was performed for 30 min followed by a ball milling deagglomeration for 12 h. This composition corresponds to a solid loading of 55.2 vol%, which is close to the maximum obtainable with the A16

SG alumina, and the dispersant used. Maximisation of solid loading of the starting suspension is important to avoid particle segregation effects, to minimise the shrinkage during drying and to achieve a high green density. This is particularly crucial when the concentration of emulsion binders is lower than that of the slurry.

A first set of suspensions for tape casting was prepared by taking portions of the stock suspension, adding binders and stirring for at least 4 h. Three different concentrations (5, 10 and 15 wt%) of latex particles (active binder), based on the alumina powder were added in five B-1035/B-1050 weight ratios (100/0, 70/30, 50/50, 30/70, 0/100) (Table 1). Due to different solid concentration of the two emulsion binders, the total solid loading of the suspension (i.e. alumina powder + latex) depends on the amount of latex emulsions added, and on their ratio in the system. Since the solid loading is an important variable that should be maintained under strict control, a second set of suspensions was prepared with the pure binders (100% B-1035 and 100% B-1050) by adding the required amounts of water to achieve a total solid concentration of 76.13 wt%. This solid loading corresponds to that of the slip containing 15 wt% B-1050 without any extra-water added.

Rheological characterisation of slurries was already reported in a previous work.<sup>15</sup>

### 2.2 Tape casting and physical characterisation of the green tapes

Tape casting experiments were carried out on a laboratory tape caster (BC, Elmetherm, Oradour sur Vayres, France) on a polypropylene film (Western Wallis, USA). A gap of  $350\ \mu\text{m}$  under the blade and a casting rate of  $1.7\ \text{cm}\ \text{s}^{-1}$  were selected for all the casting tests. After drying, the thickness of the tapes was about  $220\ \mu\text{m}$ .

The influence of the composition of the binder system (B-1050/B-1035 ratio) and of the binder content on mechanical performance of dried green tapes was evaluated by tensile tests. Dog-bone shaped specimens with 12.7-mm tensile width and 80-mm gauge length were cut parallelly to the casting direction with a razor blade. Transversal specimens were also taken for the tapes containing pure binders (100% B-1035 and 100% B-1050) in order to study the effect of the casting direction on the mechanical properties. The tensile tests were carried out using an electromechanical universal testing machine (Schimazu 25AG, Japan) equipped with mechanical edge grips and a 1 kN load cell. The cell sensibility was 0.1 N and the traverse speed was set to  $0.5\ \text{mm}\ \text{min}^{-1}$ . The stress and strain values obtained correspond to the average of at least 10 measurements. Tests were rejected if the

**Table 1.** Compositions of the alumina suspensions prepared with different active binder (latex particles) concentrations and different B-1035/B-1050 weight ratios

B-1035/B-1050 (wt%)	Active binder content					
	5 wt%		10 wt%		15 wt%	
	Alumina	Total	Alumina	Total	Alumina	Total
100/0	76.85	81.00	71.91	79.32	67.49	77.82
70/30	76.76	80.83	71.58	78.95	67.06	77.30
50/50	76.62	80.68	71.35	78.70	67.49	76.96
30/70	76.49	80.55	71.12	78.46	66.46	76.63
0/100	76.30	80.34	70.79	78.08	66.02	76.13

failure did not occur in the middle part of the specimen gauge. No special precautions were taken concerning the humidity under which the tensile tests were performed since the binders used are characterised by a low moisture pickup and excellent humidity resistance, as claimed by the supplier.

Disc-shaped specimens were punched from the green tapes, stacked in a die up to six discs per sample, and laminated under different pressures and temperatures. In order to investigate the ability of green tapes to be laminated under different lamination conditions, three pressures (50, 75 and 100 MPa) were tested at two different temperatures (20 and 70°C). The lamination temperatures were chosen 60°C above the  $T_g$  of B-1035 binder (i.e. -40°C) and of the  $T_g$  of B-1050 (i.e. 10°C). These lamination conditions allow comparing the behaviour of both pure binders during this processing step.

Apparent density of the laminated and green tapes was calculated by measuring the thickness and weight of the disc-shaped specimens. The average density of the unlaminated green sheets corresponds to the arithmetic mean of the density of five discs punched along the dried tape.

### 2.3 Characterisation of the sintered tapes

Sintering was carried out in air at 1600°C using both: (i) monolayer specimens to study the effects of binder composition and, (ii) multilayer specimens to evaluate the effects of lamination conditions.

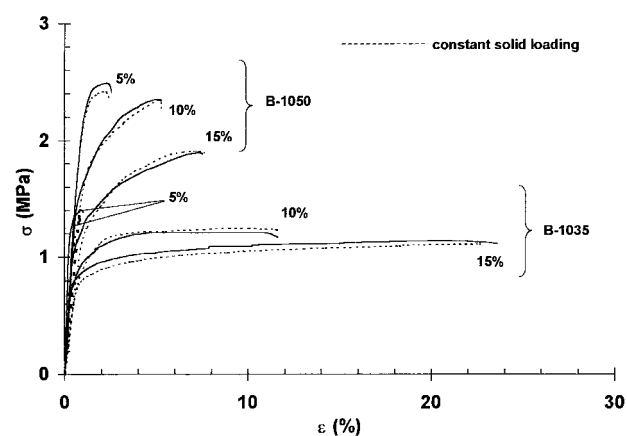
For both binders, thermogravimetric analysis (L81, LINSEIS, Germany) showed similar weight loss versus temperature behaviours. The most part of the weight loss began at about 200°C and was completed at around 500°C. According to these results, the adopted debinding heating cycle was 4°C min<sup>-1</sup> from 20 up to 250°C, 0.5°C min<sup>-1</sup> from 250 up to 500°C, and a dwell of 2 h at 500°C to allow complete binder removal. Then, the sintering was performed at 4°C min<sup>-1</sup> up to 1600°C with a holding of 3 h, and free cooling down.

Densities of the sintered bodies were measured by Archimedes' technique in distilled water. Polished and thermally etched surfaces of sintered samples were also observed (HR-SEM, Hitachi S-4100, Japan).

## 3 Results and Discussion

### 3.1 Tensile behaviour of the green tapes

Figure 1 shows typical tensile curves for the green tapes prepared with 5, 10 and 15 wt% of the pure binders (100% B-1035 and 100% B-1050). Two different groups of curves can be distinguished, corresponding to the two binders used. As a general trend, for both binders, the maximum stress decreases and the maximum strain increases with increasing binder content. The most significant stress and strain variations are observed for the higher  $T_g$  (B-1050) and lower  $T_g$  (B-1035) binders, respectively. For 15 wt% binder, the strain reached only ≈7% of the initial probe size in the case of the high  $T_g$  B-1050, compared with ≈22% in the case of the low  $T_g$  B-1035. As expected, the lower  $T_g$  B-1035 binder leads to green tapes with more elastoplastic behaviour, compared with the higher  $T_g$ , B-1050 binder. For tapes containing 5 wt% of



**Fig. 1.** Stress–strain curves for green tape specimens prepared with different amounts of pure binders, with (dotted lines) and without (continuous lines) solid loading adjustment.

binder, such a behaviour is not evidenced even with the lower  $T_g$  binder, indicating that this amount of binder was not enough to maintain the mechanical integrity of the alumina green sheets. For higher amounts of binders the stress/strain curves exhibited an elastic domain, and one or two plastic domains (hardening and/or flow domain), depending on the type and amount of binder. We can also observe a small elongation between the maximum and failure stresses (ductile behaviour) for binder contents of 10 and 15 wt%. Finally, no significant differences are detected between the tapes prepared from slurries with and without solid loading adjustments. This result is not surprising since the amount of water added to maintain the solid loading constant was very small and probably not enough to disturb the distribution of polymeric particles within the tape.

The maximum stress and the corresponding strain values reached for the different binder compositions, as a function of active binder content, are reported in Figs 2(a) and (b), respectively. Whatever the binder composition tested, the maximum stress linearly decreases with increasing binder contents, whereas the maximum strain exhibits an opposite trend. The more accentuated decrease of the maximum stress is observed for the pure B-1050 binder, varying from 2.5 to 1.9 MPa when the high  $T_g$  B-1050 binder content changes from 5 to 15 wt%. These tensile strength values are comparable to those obtained for green sheets prepared from aqueous<sup>4,13</sup> and non-aqueous<sup>24</sup> media. The more pronounced variation in the maximum strain (from  $\approx 0.9$  to  $\approx 22\%$ ) is observed for the pure low  $T_g$  B-1035 binder. The last strain value is higher than those most frequently reported in the literature<sup>26–29</sup> for tapes prepared from typical aqueous or non-aqueous formulations, but lower than that measured by Nagata<sup>18</sup> using aqueous soluble polymers as binders.

Figure 2 shows that the pure binders represent extreme cases in terms of maximum stress and strain

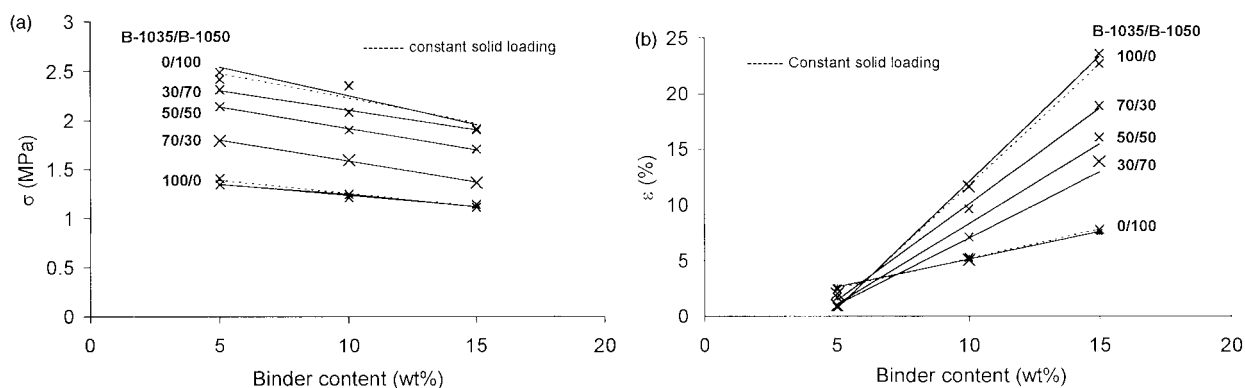
variations. All the intermediate compositions lie in between. Since there is a linear variation of these parameters for all compositions tested, the tailoring of the mechanical properties of green tapes, in the range of values obtained, seems relatively easy to achieve in this system. Furthermore, the mechanical behaviour of the green tapes corresponds to what would be expected from the mechanical behaviour of the binders at room temperature, i.e. brittle for high  $T_g$  B-1050 and elastoplastic for low  $T_g$  B-1035. Above  $T_g$ , polymeric molecules have high flexibility and mobility. Moreover, their average distance from tip to tip is far less than the length of the extended molecules. Hence, these chains will straighten and align when tensile stress is applied, enhancing elongation under load. Pure B-1035 binder leads to high flexibility green tapes, although relatively weak, while the pure B-1050 binder gives high strength and brittle green tapes. The two acrylic emulsion binders B-1035 and B-1050 are complementary in terms of strength and flexibility, and mixtures of these two binders lie in between individual behaviours.

Beyond the nature and amount of binder, the maximum values reached for stress [Fig. 3(a)] and for strain [Fig. 3(b)] also depend on the orientation of the sample with respect to the casting direction, (i.e. parallel or perpendicular to the casting direction). Specimens aligned in the casting direction exhibit higher values of stress and strain than those orientated perpendicularly. This might probably be due to some preferential orientation of the polymer chains, originated by the shear stress imposed under the blade during casting.<sup>12</sup>

## 3.2 Lamination

### 3.2.1 Effects on the green density

Preliminary lamination tests on green tapes have showed that a polymer content of 5 wt% was not enough to obtain sufficient adhesion between layers.



**Fig. 2.** Evolution of (a) maximum stress and (b) maximum strain for different binder contents and for different B1035/B1050 ratios. The dotted lines represent the values obtained for samples with solid loading adjustment.

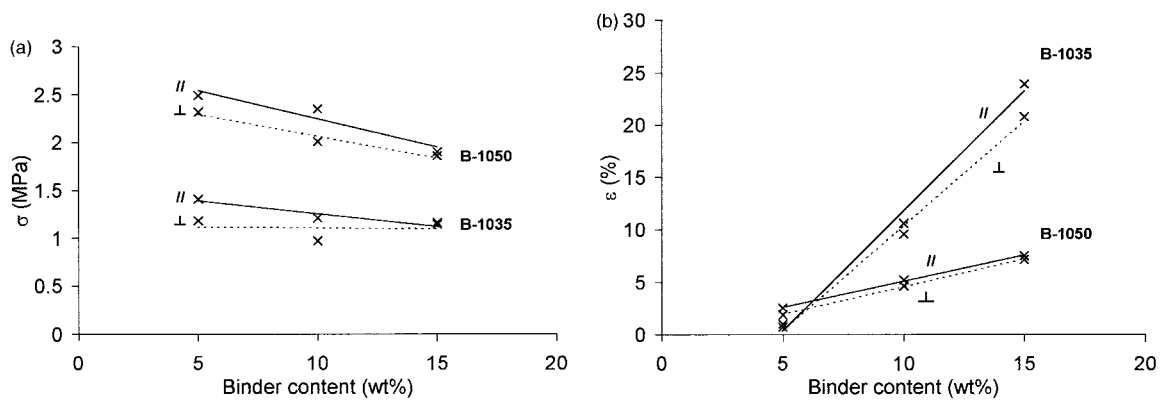
Then, bulk specimens were fabricated by laminating monolayers containing 10 and 15 wt% of latex active binder. Figure 4(a) and (b) show that under similar lamination conditions (pressure and temperature) both the binder composition and the binder concentration have no significant effect on the green density. Some fluctuations occur between the different compositions but they seem random and likely result from the measurement accuracy ( $\pm 1\%$  TD). We have to remind that the chosen lamination temperatures are both  $60^\circ\text{C}$  above the  $T_g$  of the two binders. Further, the green density of the layers appears to be more affected by the pressure than by the temperature of lamination, especially for tapes containing 15 wt% of binder. This trend observed for the green density is in good agreement with other lamination results reported for tapes prepared in organic media.<sup>17,20,22</sup> The increase of green density promoted by the thermo-compression stage can be attributed to a reduction, or even elimination, of the voids present in the as-cast tapes. This will reduce the maximum flaw size and will improve the uniformity of the microstructure and the mechanical performance of the

final products. This behaviour has been demonstrated in both  $\text{Al}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3/\text{ZrO}_2$  laminates.<sup>29</sup>

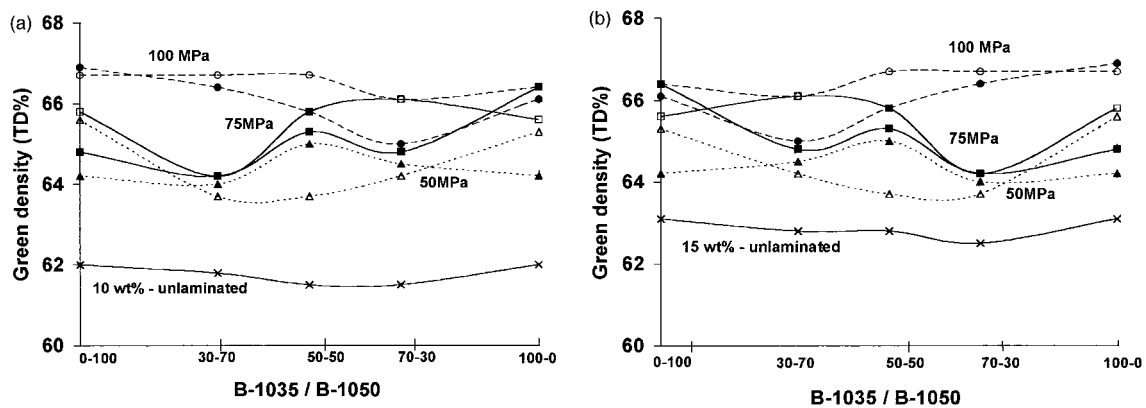
### 3.2.2 Effects on the sintered density

The effects of both temperature and pressure of lamination on sintered density of unlaminated and laminated samples containing 10 and 15 wt% of binder are reported in Figs 5(a) and (b), respectively.

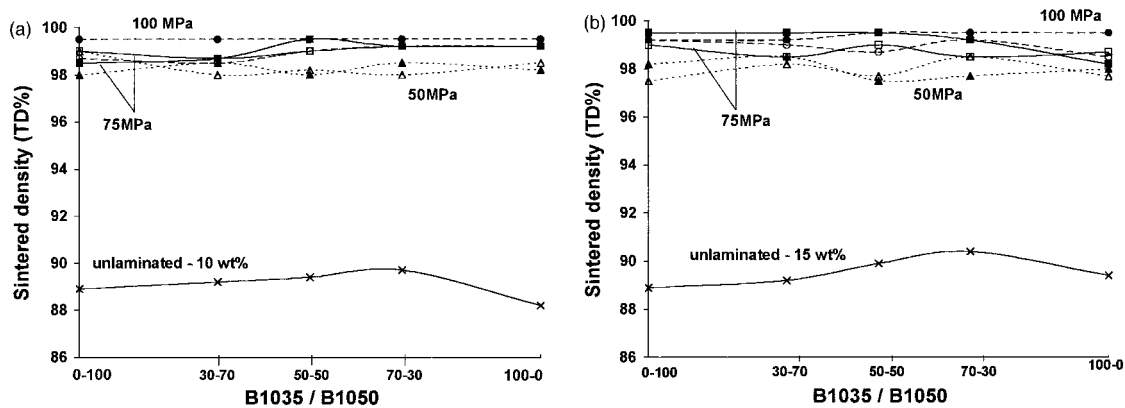
The final density seems to be more or less independent on binder composition. The observed fluctuations remain within the error range ( $\pm 1\%$  TD). For the unlaminated specimens, sintered density increases with the amount of binder added. The same trend was observed for the green sheets, attributed to a more favourable particle size distribution of the system when the amount of finer latex particles increases. This is in agreement with other literature reports.<sup>15,17,18,20</sup> As in the case of the green sheets; the final density of the laminated samples (98.5% TD) was significantly improved, compared with that of the non-laminated specimens. However, no significant differences in sintered density were encountered for different lamination conditions (temperature and/or pressure), binder



**Fig. 3.** Influence of the cutting direction, with respect to the casting direction, on (a) the maximum stress and (b) the maximum strain for the samples prepared with different amounts of pure binders (continuous lines: samples taken along the casting direction '//', dotted lines: samples taken normally to the casting direction ' $\perp$ ').



**Fig. 4.** Effect of binder composition and concentration on the green density for unlaminated (-x-) and laminated samples with (a) 10% and (b) 15% of binder. Pressure of lamination are: ( $\Delta$ ,  $\blacktriangle$ ) 50 MPa, ( $\square$ ,  $\blacksquare$ ) 75 MPa, and ( $\circ$ ,  $\bullet$ ) 100 MPa. Open and full symbols stand for lamination at  $20^\circ\text{C}$  and  $70^\circ\text{C}$ , respectively.



**Fig. 5.** Effect of binder composition and concentration on the sintered density for unlaminated (-x-) and laminated samples with (a) 10%, and (b) 15% of binder. Pressure of lamination: ( $\Delta$ ,  $\blacktriangle$ ) 50 MPa, ( $\square$ ,  $\blacksquare$ ) 75 MPa, and ( $\circ$ ,  $\bullet$ ) 100 MPa. Open and full symbols stand for lamination at 20 and 70 °C, respectively.

proportions and/or binder contents. This might probably derive from (i) the high degree of homogeneity of the green tapes, and (ii) the low heating rate and the high sintering temperature used that will tend to homogenise eventual small differences in sintering behaviour among the various laminated samples. As a matter of fact, SEM observations on top, bottom and cross-section surfaces of the sintered specimens did reveal similar microstructures.

#### 4 Summary and Conclusions

This work shows that good quality tapes could be obtained via water-based tape casting using acrylic emulsion binders. The tensile tests performed on the green sheets at room temperature and the lamination behaviour observed are very promising from the point of view of handling and subsequent process operations used in the microelectronics ceramic industry such as cutting, processing via holes, screen printing stacking and sintering.

The acrylic latex binder with a low  $T_g$  allows the production of highly deformable tapes with a typical elastoplastic behaviour and relatively small maximum stress. Conversely, a high  $T_g$  acrylic latex binder allows the production of stronger tapes but having a more brittle behaviour. Mixtures of the two binders confer to the tapes intermediate mechanical behaviours and values for the maximum strain and stress that lie in between.

Both green and sintered densities of monolayer and laminated specimens are independent on the binder composition, but depend on the amount of binder added. For the laminated specimens, the green and sintered density also depend on the lamination pressure, but is almost unaffected by the lamination temperature for temperatures higher than the glass transition temperature of the two binders.

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