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Al_2O_3/Y-TZP and Y-TZP materials fabricated by stacking layers obtained by aqueous tape casting

J. Gurauskis, A.J. Sánchez-Herencia, C. Baudín *

Instituto de Cerámica y Vidrio (CSIC), Campus de Cantoblanco, 28049 Madrid, Spain

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Abstract

A processing method for production of multilayer ceramics by laminating stacked green ceramic tapes with help of adhesive layer and pressing at room temperature was extended. Ceramic tapes with two compositions within the system alumina/yttria stabilized zirconia (Y-TZP) (alumina–40 vol.% Y-TZP and 100 vol.% Y-TZP), obtained from aqueous ceramic slurries by tape casting procedure were employed in this work. The optimum conditions to fabricate pieces made of six tapes with same composition, and to obtain defect free sintered materials, by varying the level of pressure applied, was investigated. For this purpose, previously selected composition of adhesive layer and type of pre-treatment of tapes were used.

The analysis of engineering stress–apparent strain curves recorded during the lamination process was done to control the formation of cracks within the pieces under pressure. Optimization of the pressure range was performed by the analysis of the green density change as a function of pressure. The cross section observation by optical and scanning electron microscopy of sintered ceramic pieces was carried out to assure the control of the process. Fabricated pieces were subjected to testing by Impulse Excitation Technique (IET) and Vickers indentation in order to evaluate the adequacy of the fabrication procedure and to analyse the possible effects of interfaces. Dense pieces with maximum sizes of the defects of the same level as those of the microstructural ones and without interface effects on the elastic and mechanical behaviour were obtained.

Keywords: Al_2O_3; ZrO_2; Tape casting; Cold pressing; Joining; Laminates

1. Introduction

The fabrication of multilayer ceramics by lamination of tape cast tapes to form a ceramic piece with desired architecture is an area of extensive research.[1–8] As summarized previously,[7,8] several routes are available to obtain ceramic multilayers by stacking individual tapes.

One of the principal issues is to optimize the fabrication process in order to employ minimum amounts of organics and to perform the joining process at low pressures and temperatures. A possibility to minimise problems related to organics is the use of tapes fabricated from water-based suspensions.[1–5,8] The employment of aqueous ceramic slurries in tape casting process permits to avoid the presence of volatile, toxic and expensive organic solvents. In this case, only small amounts of organics are required as binders and dispersants.

In a previous paper,[8] the fabrication of monolithic pieces consisting of six high density (≈59th.%) tapes obtained from aqueous ceramic slurries with the composition 95 vol.% Al_2O_3–5 vol.% Y-TZP and using an adhesive was addressed, as a first approach to the problem of stacking water-based tapes. Different piece shapes, pre-treatment routes of the constituent tapes, solid content in the adhesive and pressures were investigated. Relatively low pressures (<20 MPa) were required to obtain defect free and dense bodies, as compared with those reported previously[5] for lamination of water-based cast tapes (≅50–100 MPa). Moreover, pressing was performed at room temperature.

* Corresponding author. Tel.: +34 91 7355840; fax: +34 91 7355843.
E-mail address: cbaudin@icv.csic.es (C. Baudín).

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The engineering stress–apparent strain curves recorded during pressing were proposed to control the pressing procedure. Macroscopic failure of the pieces as well as the formation of microcracks perpendicular to the interfaces between layers were demonstrated to be related to distinct features of the stress–strain curves. Therefore, it was concluded that the analysis of the curves allows the establishment of maximum pressure levels to avoid such defects. Further analysis of the microstructure of the interfaces and green density changes as a function of the pressure applied, permitted to establish minimum pressure values to obtain defect free materials. As general conditions for the proposed joining process the use of round shaped tapes, immersed in water prior to pressing, and the application of an adhesive layer consisting of a low concentration water dispersion of the polymeric emulsion used to fabricate the tapes were established.

In this work, the optimised general conditions were used in order to fabricate monolithic pieces consisting of tapes with different characteristics. Two different systems have been addressed. Medium green density (≈55th.%) tapes of alumina + 40 vol.% Y-TZP and relatively low green density (≈51th.%) ones made of Y-TZP. The methodology to analyse the stress–strain curves and the microstructure, proposed in the previous paper, has been used to evaluate its adequacy to other systems and to establish the optimum conditions to fabricate alumina + 40 vol.% Y-TZP and Y-TZP monoliths.

The properties of the obtained materials have been analysed at two levels. The Impulse Excitation Technique (IET) was used to evaluate the bulk behaviour of the materials in terms of the elastic properties; additionally, this method is an indirect method to detect any possible delaminations in the pieces.[9] In order to detect possible local effects in the mechanical properties of the materials, the size of cracks developed from Vickers indentations at different points of the cross section of the pieces was analysed.

2. Experimental

2.1. Slurry formulation and tape casting

The cast tapes were produced from stable slurries of high purity α-Al_2O_3 and Y-TZP powders in deionised water as dispersing media. The starting powders of α-Al_2O_3 (Condea HPA 0.5, USA), with mean particle size of 0.35 μm and specific surface area of 9.5 m^2/g, and a t-ZrO_2 stabilised with 3 mol% Y_2O_3 (TZ_3Y_S, TOSOH, Japan), with a mean particle size of 0.4 μm and a specific surface area of 6.7 m^2/g, were used.

Depending on the final amount of drying cracks, the surface finish, and flexibility of the tapes, and the highest solid content of the ceramic slurry employed, the optimum conditions to fabricate tapes containing 60 vol.% of α-Al_2O_3 and 40 vol.% of Y-TZP (named A-40YTZP, slurry with 47 vol.% of solids content) and 100 vol.% of Y-TZP (named YTZP, slurry with 45 vol.% of solid content) were established. A polyelectrolyte (Dolapix CE 64, Zschimmer & Schwarz, Germany) was used for powder dispersion. Water-based polymeric emulsion Mowilith DM 765 E (Celanese, Spain), with a T_g of −6 °C and solid content 50 vol.%, particle size 0.05–0.15 μm was used as binder emulsion.

First, the stabilisation of the slurries with the dispersant (dispersant with 0.7 and 0.5 wt.% of solids for A-40YTZP and YTZP, respectively) was performed by ball milling with alumina balls for 4 h. Then 5 wt.% of binder, referred to solid content, was added to the deflocculated suspensions and further mixing with a blade mixer for 30 min was done. The tape casting was performed on stationary polypropylene film using a moving tape casting device with two doctor blades (laboratory developed device).[8] The final casting parameters were 10 mm/s of casting velocity and 500 μm of gap height between the blades and the carrier film. After drying in air at room conditions for 24 h, the green ceramic tapes were subjected for further drying at 60 °C for 48 h. The final thickness of the dry tapes obtained varied between 480 and 520 μm. Round shaped tapes were used to avoid heterogeneous stress distribution within the specimens during pressing;[8] for this purpose, the final shaping by punching out round shape (diameter $Ø_{green}$ 26 mm and $Ø_{green}$ 60 mm) pieces of tape was performed.

Green densities of the tapes were measured after the drying cycle described above, using the geometrical method. Reported values are the average of those obtained for four discs of 26 mm diameter processed under nominally identical conditions and error bars are the standard deviations.

2.2. Lamination by gluing and pressing

Each monolithic piece was made using six tapes with the same composition. Before applying the gluing agent, the tapes were subjected to a "wet" treatment. That consists of dipping the ceramic tape in distilled water during 1 min. The tapes were stacked sequentially to form the monolithic pieces and the application of the gluing agent was done with a paintbrush on the contact surfaces of consecutive tapes. Previously established optimum composition of the gluing agent, which is 5 wt.% dilution in distilled water of the binder Mowilith DM 765 E was used.[8]

The pressing experiments were carried out using a universal testing machine (Microtest SA, Spain) with steel compression plates. In order to avoid friction with the plates, the stacked pieces were placed between two sheets of polypropylene film. The pressure was applied using a load frame displacement rate of 0.05 mm/min. The load and the displacement of the load frame were recorded during the pressing process and engineering stress–apparent strain curves were calculated assuming uniaxial compression using the dimensions of the pieces. The stress–strain curves of two individual small ($Ø_{green}$ 26 mm) pieces of each composition pressed up to the maximum stress values (≈90 MPa) were first analysed to investigate macroscopic failure. The curves corresponding to five small ($Ø_{green}$ 26 mm) pieces of each composition

pressed up to pressures from 3 to 18 MPa were analysed to look for cracks perpendicular to the interfaces. In this case, the engineering stress–apparent strain curves were adjusted to polynomial fits and the average of the five different curves was used to represent the behaviour of the pieces, with error bars corresponding to the standard deviations. The selection of optimum pressure values for interface quality was done using the green densities and the microstructural characteristics of these pieces after sintering.

Green densities were determined by Archimedes method in mercury, using five dry pressed pieces fabricated under nominally identical conditions, and compared to those of the dry green tapes. Reported values are the average of the five values and errors are the standard deviations. Relative green densities were calculated as percentage of the calculated theoretical density for each composition, using 3.99 g/cm^3 for α-Al_2O_3 (ASTM 42-1468) and 6.10 g/cm^3 for Y-TZP (ASTM 83-113).

2.3. Thermal treatment and characterisation of the materials

Binder burn out and sintering were performed in single thermal treatment cycle. The binder burn out was carried out in air with heating at the rate of 1 °C/min up to 600 °C, with a dwell time of 30 min. The following sintering was carried out by increasing the temperature with heating rate of 5 °C/min up to 1550 °C with a dwell time of 2 h. Density values of sintered pieces were determined by the Archimedes method in distilled water and relative densities were calculated as described for green densities.

The polished cross sections of all sintered pieces were investigated for interface defects and delaminations by optical microscopy (Carl-Zeiss H-P1, Germany) and scanning electron microscopy (Zeiss DSM-950, Germany, thermally etched samples at 1400 °C–1 min).

Impulse Excitation Technique measurements were carried out with "GrindoSonic MK5" (J.W. Lemmens-Electronica N.V., Belgium) apparatus using large ($Ø_{green}$ 60 mm) pieces. Two samples of each composition were tested, without any previous machining, following ASTM[9] standard procedure. In order to record the torsional and flexural mode vibrations, the piece was placed on the support points allowing first and second natural vibrations of the disc. The Young's modulus, *E*, the shear modulus, *G*, and the Poisson's ratio were calculated from the values of the vibration frequencies and the dimensions and the densities of the samples.[9]

Vickers indentation ("Microtest", Spain) testing was performed on two cross sections of small ($Ø_{green}$ 26 mm) pieces of each composition fabricated with 18 MPa. The polished central parts (length ~ 20 mm) of the cross sections of two different pieces were indented in 17 points, selected in the way to obtain nine indentations at the interface areas and eight indentations in the centres of the constituent tapes. Fig. 1 shows as schema of half length (10 mm) of the cross sections investigated with the positioning of the indentation points.

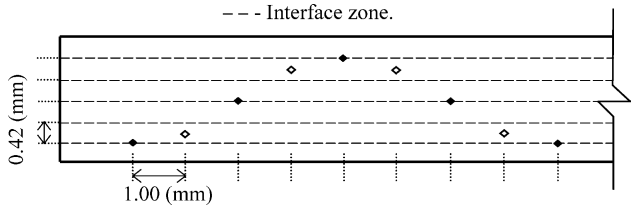

Fig. 1. Vickers indentation positions at the polished central part (20 mm) of the cross section of the sintered pieces ($Ø_{green}$ = 26 mm). Schema corresponding to the half length of the samples. Indentation performed in (♦) the interface zone and (◊) the centre of the constituent tapes.

Therefore, reported crack size values are the average of 18 and 16 determinations for interface and centre cracks, respectively, and errors are the corresponding standard deviations. The indentations were performed in controlled displacement mode at 0.01 mm/s up to maximum a load of 50 N and using 10 s as holding time. The indentation impressions produced by a Vickers indenter were measured within the time period of 15 min using optical microscopy with a graduated ocular (with a precision between grades of 4 μm).

3. Results and discussion

3.1. Optimisation of the fabrication procedure

The behaviour of A-40YTZP and YTZP pieces pressed up to 90 MPa is shown in Fig. 2. The stress–strain curves corresponding to different specimens of the same composition were practically coincident in the whole stress interval (Fig. 2). The curves were smooth, with monotonously increasing stress values across the whole strain interval, revealing increasing contact between particles within the interfaces. As shown by the accompanying photos (Fig. 2), macroscopic failure did not take place in any of the samples, in agreement with the lack of a rapid decrease of the slope values in the stress–strain curves and the coincidence of curves for different samples, as observed in the previous work[8] for pieces fabricated using wet tapes.

For both compositions the stress–strain curves were easily adjusted by third degree polynomials, as shown in Fig. 3, for the low pressure interval. At the initial part of the curves, the increase of stress with strain was more pronounced for A-40YTZP (Fig. 3a) than for YTZP (Fig. 3b) whereas this tendency changed from about 8 MPa, with a steep increase of stress with strain for YTZP specimens. These features show that the formation of a more significant number of interface contacts occurred from pressures of about 8 MPa for YTZP than for A-40YTZP. The aspect of the YTZP tapes, which were shiny white, indicated that surface finish was smoother than in A-40YTZP tapes. Therefore, at least for these levels of green density values (≈50–55% of theoretical), the stress response of the pieces to deformation is more related to surface characteristics of the tapes such as smoothness than to green density, which was higher for the A-40YTZP tapes.

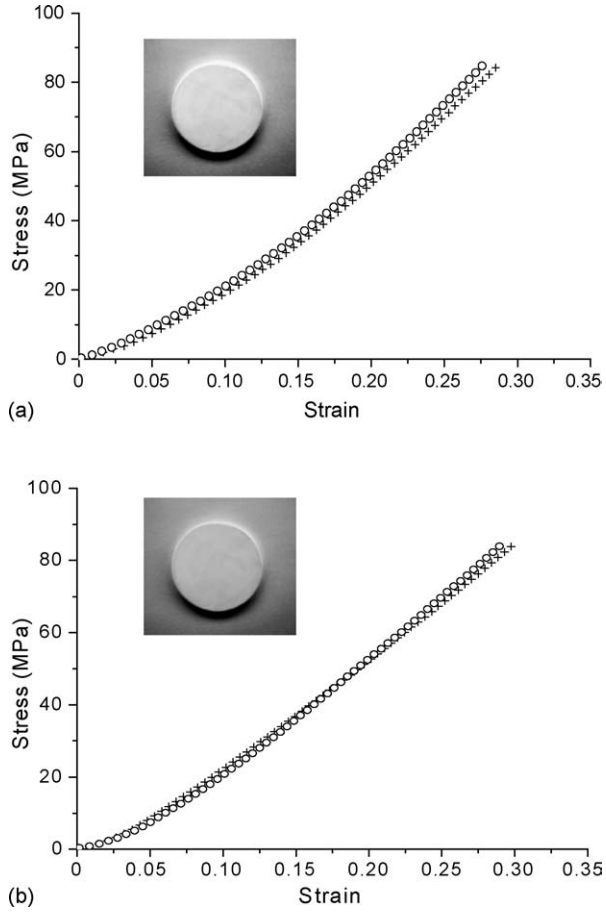

Fig. 2. Experimental apparent stress–strain curves, corresponding to pieces ($Ø_{green}$ = 26 mm) fabricated under nominally identical conditions. The aspect of the pieces after the pressing up to maximum pressure (90 MPa) is shown in the photos. (a) Two tests of A-40 YTZP and (b) two tests of YTZP.

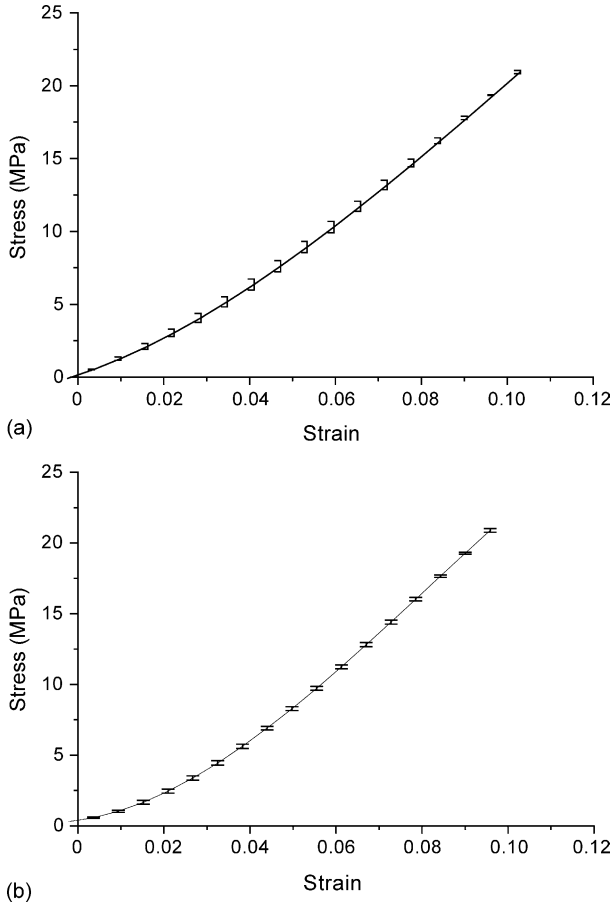

Fig. 3. Average polynomial stress–strain curves corresponding to pieces ($Ø_{green}$ = 26 mm) pressed up to 20 MPa. The variability range plotted corresponds to values obtained from five pieces fabricated under nominally identical conditions. (a) A-40YTZP and (b) YTZP.

The higher capability for accommodation of the rougher surface A-40YTZP tapes in the early stages of pressing ($P \cong 3$–15 MPa) could be the origin of the slightly larger differences between the pressing behaviour of different pieces, as revealed by the expanding deviation values in the stress–strain curve within the range of pressures of 3–12 MPa (Fig. 3a). In fact, variability in the response of different YTZP pieces is constant across the whole pressure interval (Fig. 3b).

In Fig. 4, the derivative plot of the average polynomial fit curves of Fig. 3 in the low (20 MPa) pressure range is represented. Accordingly, with previous results,[8] the formation of cracks perpendicular to the interfaces between layers is shown in these plots by a well-defined peak, indicating an inflexion point in the stress–strain curve. For both kinds of pieces, A-40YTZP and YTZP, the derivate values indicate continuous smooth behaviour in the strain range considered, which indicates that no cracks are formed, as confirmed by the analysis of the cross sections of these pieces shown in Fig. 5. This behaviour is similar to that of the previously studied composition when the tapes received identical "wet" treatment route before pressing as the one used here, that

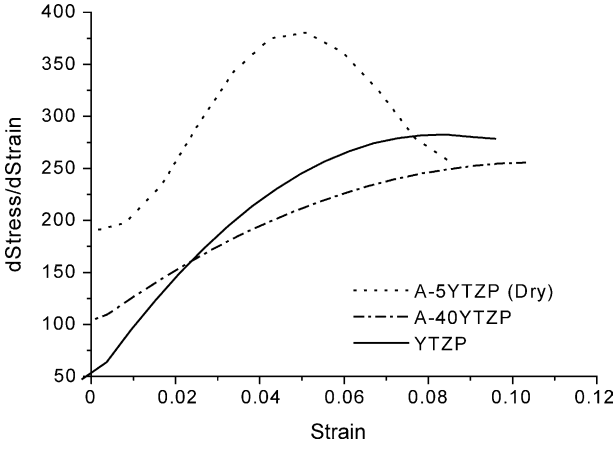

Fig. 4. Derivative of the average polynomial curves of Fig. 3. Behaviour during pressing of pieces ($Ø_{green}$ = 26 mm) fabricated using the A-40YTZP and YTZP wet tapes at low pressures. For comparison purposes, the curve for A-5YTZP (dry) pieces that developed cracks perpendicular to the interfaces at low pressures is represented [8].

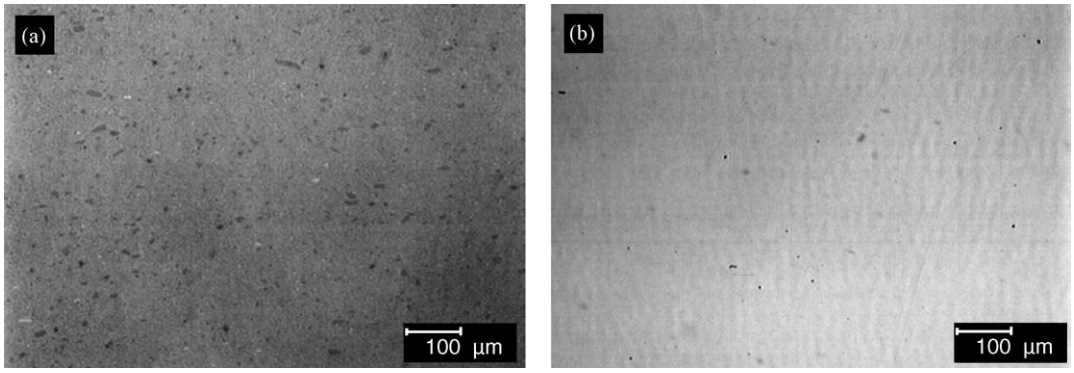

Fig. 5. Optical micrographs of polished cross sections of pieces ($Ø_{green}$ = 26 mm) pressed up to 20 MPa. The formation of cracks perpendicular to the interfaces is not observed. (a) A-40YTZP and (b) YTZP.

confirms the adequacy of this tape treatment to obtain pieces free from cracks perpendicular to the interfaces.

The above mentioned results indicate that pressures up to 20 MPa would be a safe working interval for fabrication of pieces of both kinds of tapes studied here. From the previous work done with A-5YTZP pieces,[8] the control parameter to select minimum pressure values for optimum interface quality was the green density change as a function of pressure. In this system, once the density of the pressed pieces reached that of the single tapes, the size of the interface defects was reduced to that of characteristic microstructural defects (≈5–10 μm), such as pores or agglomerates. Further pressing to reach higher densities would result not just in the formation of good and uniform adhesion between the tapes, but also, in the densification within the tapes. The results of green and sintered density corresponding to A-40YTZP and YTZP pieces pressed between 3 and 18 MPa are given in Table 1 and compared to those of the as dried single tapes of the same composition. The increase of green and sintered density with increasing pressure was detectable for both compositions. No differences between sintered densities of pieces pressed at pressures equal or higher than 10 and 5 MPa were detected for A-40YTZP and YTZP pieces, respectively. Conversely, even tough differences between green densities were very low, and even inside the variability limits, clear tendencies can be derived from data in Table 1. In this low pressure interval, A-40YTZP pieces show a monotonous increase of the green density values with pressure while for YTZP pieces a sharp increase occurs. These behaviours can be related with the differences between the stress–strain curves of these pieces (Fig. 3), smooth for A-40YTZP and rapidly increasing for YTZP.

Characteristic micrographs of the cross sections of A-40YTZP and YTZP pieces are shown in Figs. 6 and 7. For the A-40YTZP pieces pressed at pressures lower than or equal to 15 MPa, relatively large (40–100 μm) linear defects along the interface (Fig. 6a) were observed. The scale of these defects is in good agreement with the green density values, as for this interval of pressure values the densities of the pieces were lower than that of the single tapes (Table 1). Once the density of the single tape was reached, for pieces pressed under 18 MPa (Table 1), the frequency and the scale (5–8 μm) of the interface defects (Fig. 6b) were minimized down to the level of microstructural defects such as pores or agglomerates. In these pieces, mainly good and uniform adhesion between the tapes was observed (Fig. 6c). The YTZP pieces presented a similar behaviour. For pieces obtained using pressures up to 15 MPa, for which the density was lower than that of the single tapes, relatively large (>10 μm) interface longitudinal defects were observed (Fig. 7a). Once the density of

Table 1
Green and sintered density of pieces ($Ø_{green}$ = 26 mm) fabricated using the A-40YTZP and YTZP tapes, as a function of the applied pressure

Material	Stress (MPa)	ρ_{green}		$\rho_{sintered}$	
		g/cm^3	$\rho_{piece}/\rho_{single\ tape}$	g/cm^3	th.%
A-40YTZP	3	2.61 ± 0.02	98.5 ± 0.8	4.76 ± 0.01	98.5 ± 0.1
	5	2.61 ± 0.01	98.5 ± 0.4	4.76 ± 0.01	98.5 ± 0.1
	10	2.63 ± 0.01	99.2 ± 0.4	4.78 ± 0.01	99.0 ± 0.1
	15	2.64 ± 0.01	99.6 ± 0.4	4.79 ± 0.01	99.2 ± 0.2
	18	2.65 ± 0.01	100.0 ± 0.4	4.79 ± 0.01	99.2 ± 0.1
YTZP	3	3.09 ± 0.01	99.3 ± 0.4	6.03 ± 0.01	98.7 ± 0.1
	5	3.10 ± 0.01	99.6 ± 0.4	6.04 ± 0.01	99.0 ± 0.1
	10	3.10 ± 0.01	99.6 ± 0.4	6.05 ± 0.01	99.1 ± 0.2
	15	3.10 ± 0.01	99.6 ± 0.4	6.05 ± 0.01	99.1 ± 0.2
	18	3.11 ± 0.01	100.0 ± 0.3	6.05 ± 0.01	99.1 ± 0.1

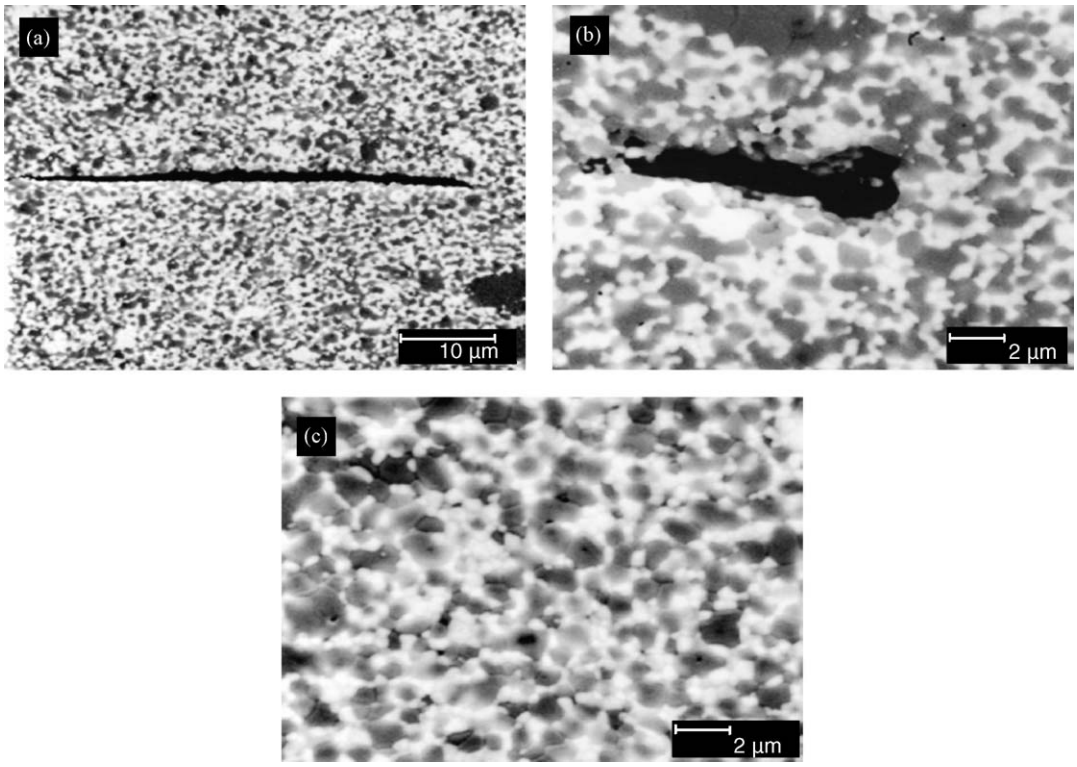

Fig. 6. Microstructures of A-40YTZP pieces ($Ø_{green}$ = 26 mm) pressed using various pressures. SEM micrographs of polished and thermally etched (1400 °C–1 min) cross sections. (a) Piece pressed under 15 MPa. Longitudinal defects along the interface, (b) piece pressed under 18 MPa. Less frequent and smaller scale longitudinal defects at the interface, (c) piece pressed under 18 MPa. Mainly good and uniform adhesion within the interface.

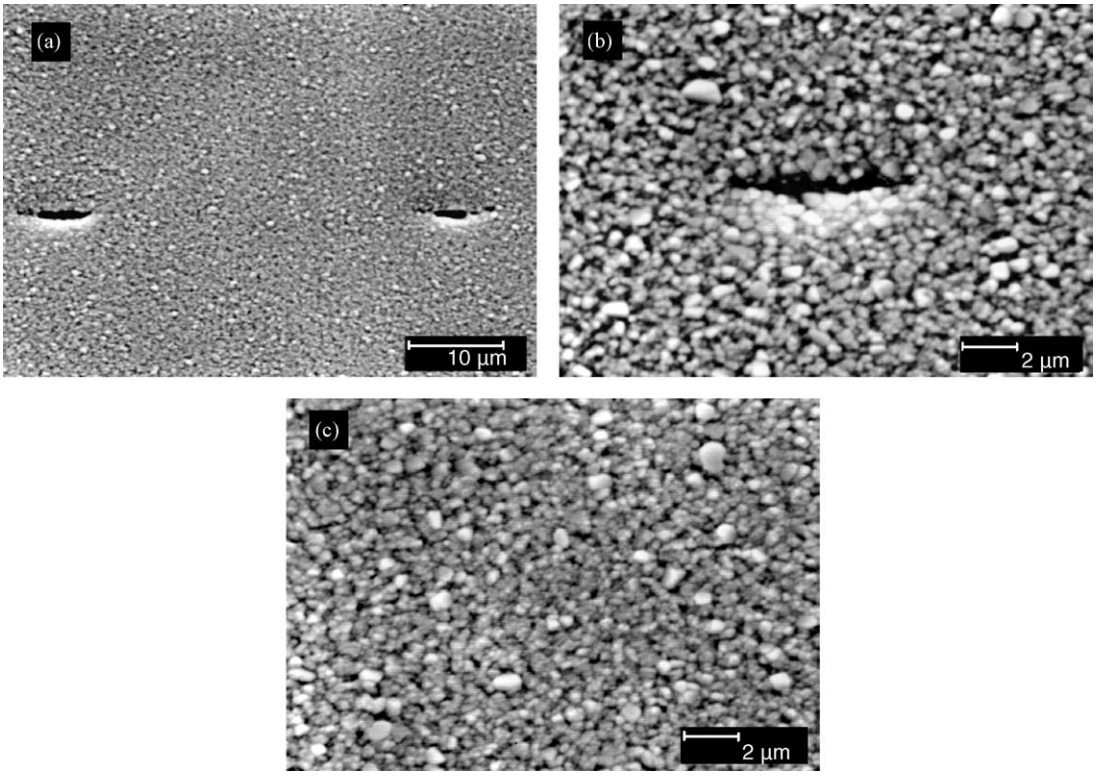

Fig. 7. Microstructures of YTZP pieces ($Ø_{green}$ = 26 mm) pressed using various pressures. SEM micrographs of polished and thermally etched (1400 °C–1 min) cross sections. (a) Piece pressed under 15 MPa. Longitudinal defects along the interface, (b) piece pressed under 18 MPa. Less frequent and smaller scale longitudinal defects at the interface, (c) piece pressed under 18 MPa. Mainly good and uniform adhesion within the interface.

the single tapes was reached, good quality interfaces were observed (Fig. 7c) and the size (down to 4–6 μm) and frequency of the defects decreased (Fig. 7b).

Even tough the size of the interface defects was smaller for the YTZP specimens than for the A-40YTZP pieces, the frequency was higher for all pressure levels investigated. Moreover, in many cases, these defects were arranged linearly (Fig. 7a). Probably, the rapid accommodation between the tapes, demonstrated by the steep stress–strain curves (Fig. 3b), leads to the trapping of small amounts of gluing agent that can not be removed for further pressure increase.

3.2. Results obtained from IET and Vickers indentation

As derived from Table 1 and the discussion above, high density sintered pieces (≥99% of theoretical) free of large defects were obtained for both compositions by pressing under 18 MPa. Therefore, this maximum pressure was selected to fabricate the samples for mechanical characterisation.

Pieces with the adequate geometry (diameter $\emptyset_{green}$ 60 mm) to carry out the determinations of the torsional and flexural vibration frequencies, by IET, of "as sintered" pieces were fabricated. These large pieces presented the same density values as those of the small ($\emptyset_{green}$ 26 mm) ones, demonstrating the validity of pressure as a processing control parameter, at least in the level of sizes considered here.

During testing, one single value for the vibration frequencies was easily obtained for the A-40YTZP specimens, revealing their homogeneity and lack of large defects, as described above. Conversely, in some YTZP specimens, several different frequency values were recorded, which revealed the higher heterogeneity of these pieces, as a result of the presence of the interface defects described above.

Table 2 summarises the values of Young's modulus, shear modulus and Poisson's ratio, calculated from the recorded frequencies. Values are similar to those (E = 210 GPa, G = 81 GPa, υ = 0.31)[10] reported for a YTZP material of similar density (99.3% of theoretical). Values for A-40YTZP are similar to those (E = 320 GPa, G = 130 GPa, υ = 0.26) calculated using the Voight limit,[11] from the values corresponding to a slip cast alumina material with 99% of theoretical density (E = 400 GPa, υ = 0.22) characterised using the same equipment, and those corresponding to YTZP. From these results, no differences between the bulk properties of the monoliths fabricated by stacking individual cast tapes and those of materials with the same porosity levels and processed differently can be derived.

Table 2
Elastic properties of A-40YTZP and YTZP pieces ($\emptyset_{green}$ = 60 mm) calculated from IET results

Material	Young's modulus (GPa)	Shear modulus (GPa)	Poisson's coefficient
A-40YTZP	309 ± 2	121 ± 2	0.26 ± 1
YTZP	230 ± 4	97 ± 4	0.29 ± 1

Table 3
Sizes of Vickers indentation cracks and imprints at the cross sections of sintered pieces ($\emptyset_{green}$ = 26 mm)

Material	Position	Crack size, 2C		Imprint size, 2a	
		$2C_1$ (μm)	$2C_2$ (μm)	$2a_1$ (μm)	$2a_2$ (μm)
A-40YTZP	Interface	157 ± 4	176 ± 4	75.9 ± 4	75.9 ± 4
	Centre	158 ± 4	173 ± 4	75.9 ± 4	75.9 ± 4
YTZP	Interface	156 ± 4	167 ± 4	82.5 ± 4	82.5 ± 4
	Centre	156 ± 8	167 ± 4	82.5 ± 4	82.5 ± 4

Localization is addressed in Fig. 1. Subscripts 1 and 2 refer to parameters parallel and perpendicular to the pressing direction, respectively.

The sizes of indentation cracks and imprints are given in Table 3. For both compositions considered, no significant difference was observed between the sizes of cracks as a function of location, which confirms further the good quality of interfaces between different tapes.

Even tough results summarised in Table 3 are not conclusive, because of the standard deviation values, it seems to be a slight tendency to form larger cracks in the direction perpendicular to the pressing direction ($2C_2$) than in the parallel one ($2C_1$). The extension and origin of this possible effect has to be further analysed.

4. Conclusions

The method to fabricate defect free ceramic materials by stacking green water-based tapes by employing an adhesive layer and pressing at room temperature has been proved to be efficient for tapes with different characteristics, i.e. green density and composition.

The response to pressing of the stacked green pieces using engineering stress–apparent strain curves serves to control the pressing process and avoid the formation of macroscopic failure and microscopic cracking perpendicular to the interfaces. Green density control accompanied with cross section analysis by SEM permitted to establish minimum pressure required to fabricate defect free monoliths.

In particular, using the optimum conditions of pretreatment of tapes and dilution level of gluing agent, monolithic pieces formed by six tapes with two compositions, alumina + 40 vol.% of YTZP and YTZP, were fabricated. The quality of the interface in these pieces is assured by the values of the dynamic elastic properties, determined by the Impulse Excitation Technique, and the lack of variation of the geometrical parameters of Vickers indentations as a function of location in the cross sections.

Acknowledgments

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