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Tape casting of strontium and cobalt doped lanthanum chromite suspensions

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Abstract

Lanthanum chromite ($LaCrO_3$) is currently the most widely studied material as interconnector layers for solid oxide fuel cells (SOFC). The complexity of microstructures and geometries of SOFC devices, which are usually built-up by lamination of the different constitutive layers, make it necessary a precise control of processing parameters to achieve the desired combination of properties. Much effort has been devoted to the processing of electrodes and electrolytes but the other layers, such as that of interconnecting material, have received scarce attention. This work deals with the preparation and optimisation of the rheological behaviour of concentrated suspensions of Sr- and Co-doped $LaCrO_3$ and the subsequent tape casting to produce homogeneous thin sheets to be used in the SOFC stack.

The starting powder was produced by combustion synthesis from the corresponding nitrates and urea as a fuel, and had a final composition of $La_{0.80}Sr_{0.20}Cr_{0.92}Co_{0.08}O_3$. These powders were dispersed in ethanol with commercial copolymers (Hypermer, KD6) to solids loading of up to 58 wt%. The binding system (BS) consisted of a mixture of a binder, polyvinyl butyral-co-vinyl alcohol-co-vinyl acetate (PVA-PVAc), and two plasticizers, polyethyleneglycol (PEG400) and benzylbutylphthalate (BBP). The effect of the binding system content and the binder-to-plasticizer ratio on the tape casting performance and the characteristics of the green and the sintered tapes, were studied, as well as the influence of the casting parameters (casting speed and blades height).

Keywords: Lanthanum chromite; Rheology; Tape casting; SOFC

1. Introduction

Lanthanum chromite ($LaCrO_3$) is a refractory synthetic ceramic material, which shows good electrical conductivity at high temperatures (>800 °C), high melting temperature (2500 °C), physical and chemical stability in both oxidizing and reducing atmospheres and significant catalytic activity. Because of these properties, this material is used as solid oxide fuel cell interconnector (SOFC),[1–3] magneto hydrodynamic generators (MHD),[4,5] catalysts for soot combustion,[6,7] high temperature NO_x sensors[8] and electric heaters.[9] In power generation field, doped lanthanum chromite is also used as anode for intermediate temperature solid oxide fuel cells (ITSOFC).[10–12] The preparation of thin tapes of lanthanum chromite is required for many of these applications.

Whilst dry forming routes often lead to agglomeration due to the poor powder flow capability, colloidal processing allows the production of tapes, coatings and complex-shaped parts with a decreased number of flaws and pores and hence, higher reliability.[13] There is a wide range of wet forming routes such as tape casting, slip casting, direct coagulation or gel casting, and all of them require stable slurries with high solids loading and low viscosity.[14] Unfortunately, the maximum solids loading attainable for stable suspensions of nanopowders is significantly lower than those of particles with larger sizes.[15] Tape casting is a well-established technique for large-scale fabrication of ceramic products with large areas and small thicknesses, ranging from 50 to 500 μm, such as substrates,[16] electronic components,[17] solid oxide fuel cell components[18] and multilayered structures.[19] The use of appropriate additives is essential to achieve homogeneous, dense and flexible green tapes with high strength.

Although there has been a wide variety of materials produced by tape casting, the information available in the literature about tape casting of chromites[18,20] is very restricted in spite of its importance in the fabrication of the stack.

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This work deals with the manufacture of doped lanthanum chromite sheets by non-aqueous tape casting. The influence of suspension parameters, such as the binder system content and the binder/plasticizer ratio, and casting parameters, such as the casting speed and the blades height, on the rheological behaviour of the suspensions, the tape casting performance and the characteristics of the green and the sintered tapes, were studied.

2. Experimental procedure

A powder with the composition of $La_{0.80}Sr_{0.20}Cr_{0.92}Co_{0.08}O_3$ was firstly obtained by combustion synthesis.[21,22] For that, the following precursors were used: chromium (III) nitrate nonahydrated, $Cr(NO_3)_3{\cdot}9H_2O$ (99%, Aldrich, Germany), lanthanum (III) nitrate hexahydrated, $La(NO_3)_3{\cdot}6H_2O$ (99.99%, Aldrich, Germany), strontium nitrate, $Sr(NO_3)_2$ (99%, Aldrich, Germany) and cobalt (II) nitrate hexahydrated, $Co(NO_3)_2{\cdot}6H_2O$ (98%, Vetec, Brazil). Urea ($CO(NH_2)_2$, Nuclear, Brazil) was used as a fuel. The nitrates were stoichiometrically mixed in deionised water until complete solubility to obtain the desired composition. The details of the synthesis, the physico-chemical characterization of the obtained powder and its crystallographic analysis were reported in previous works.[21,22] The synthesized powders were attrition milled in ethanol (absolute ethanol, Casa Americana, Brazil) for 2 h. The powder was then dried in an oven at 60 °C. The specific surface area of the strontium and cobalt doped $LaCrO_3$ powder after milling, as measured by one point nitrogen adsorption (Monosorb, Quantachrome, Florida, USA), was 10.2 $m^2\,g^{-1}$. The particle size distribution of the powder was measured by laser diffraction (Mastersizer S, Malvern, UK) and revealed an average particle diameter of ∼2 μm, although a primary particle size of ∼50 nm was obtained by field emission gun scanning electron microscope observations, FEG-SEM (Hitachi S-44700, Japan).

The zeta potential of the milled powders with different deflocculant contents, 0–1.5 wt% referred to dry solids (Hypermer KD6, Uniquema, UK, which is an acrylic graft copolymer added to polyethylene glycol), was studied by laser Doppler velocimetry (Zetasizer Nano-ZS, Malvern Instruments, UK) on diluted suspensions (0.065 g l^{-1}) prepared in absolute ethanol (Panreac Química S.A., Spain).

Doped $LaCrO_3$ concentrated suspensions (40, 50, 55 and 57 wt% which correspond to 9.3, 13.3, 15.8 and 16.9 vol%, respectively) were prepared in absolute ethanol by the addition of 1 wt% dispersant (referred to dry solids) prior to ball milling for 24 h using alumina balls. The influence of the volume fraction of solids on the suspension rheology was studied. Values of viscosity extrapolated to infinite shear rates were calculated using the Cross model on viscosity curves measured using the controlled rate (CR) mode. The maximum packing fraction of the suspensions was estimated by using the Krieger–Dougherty model with the calculated limit viscosities.[23] The rheological behaviour of the suspensions was studied using a rheometer (Model RS50, Thermo Haake, Karlsruhe, Germany) with a double-cone and plate system (60 mm in diameter, 2° cone angle) provided with a solvent trap to reduce evaporation. A controlled rate experiment consisted in a three-stage measuring program with a linear increase in the shear rate from 0 to 1000 s^{-1} in 300 s, a plateau at 1000 s^{-1} for 120 s, and a further decrease to zero shear rate in 300 s. Controlled stress (CS) measurements were also performed with a two stage measuring program with a logarithmic increase of shear stress from 0 to 15 Pa in 450 s. The yield point was calculated as the cross point between shear stress and deformation from the CS flow curves plotted in log–log scales. Temperature was kept constant at 25 °C during all the measurements.

Tape casting suspensions were prepared following a two-steps mixing procedure in order to avoid competitive adsorption among additives. In a first step, the powder, the dispersing medium, and the deflocculant were mixed together and ball milled for 24 h; the binder system (binder plus plasticizers) was added in a second step.[24] Different additions of binder system (10 and 15 wt% referred to suspension weight) and binder/plasticizer (B/P) weight ratios (1/1, 2/1 and 1/2) were studied. The binder system (BS) consisted of a mixture of a binder (polyvinyl butyral-co-vinyl alcohol-co-vinyl acetate, PVA-PVAc, Aldrich-Chemie, Steinheim, Germany) and two plasticizers: polyethyleneglycol (molecular weight 400 g/mol; PEG400, Panreac Química S.A., Spain) and benzylbutylphthalate (BBP, molecular weight 312.4 g/mol; Merck, Germany). According to most experimental compositions reported in the literature for tape casting technology[25] PEG and BBP are widely used plasticizers offering a good slip performance when they are added in a 1:1 weight ratio.

Tape casting experiments were performed in a self-made machine with a stationary carrier and a container that moves at controlled speed. The slips pass through a double blade system that provides a uniform thickness of the tape, whose height was adjusted by micrometer screws. Slips were cast onto a fixed Mylar substrate (Dupont Teijin Films, Wilmington, USA), the blade height was varied from 200 to 1000 μm and the casting speeds were 5 and 10 cm s^{-1}. All the tapes were longer than 40 cm and the width was 10 cm. The tapes were dried at room conditions for 48 h, and the density was measured geometrically using punched squares with 2.0 cm in side.

Thermogravimetric analysis of the tapes (TGA) were carried out in air with a SETARAM-Labsys Thermoanalyzer (France), using alumina as the reference material, in the temperature range of 20–700 °C at a heating rate of 10 °C min^{-1}.

The tapes were sintered at 1600 °C/4 h, according to sintering studies described in a previous work[26] and the density values were determined by using the Archimedes' method and reported as percentage of theoretical density of $La_{0.80}Sr_{0.20}Cr_{0.92}Co_{0.08}O_3$ (6.50 g cm^{-3}).

Microstructural observations (SEM) were performed on top and bottom surfaces of the green tapes and on cross-sections of the sintered ones. The thickness of the sintered tapes was measured by direct observation of their cross-section by scanning electronic microscopy, SEM, (Philips XL30, Philips, Eindhoven, Netherlands).

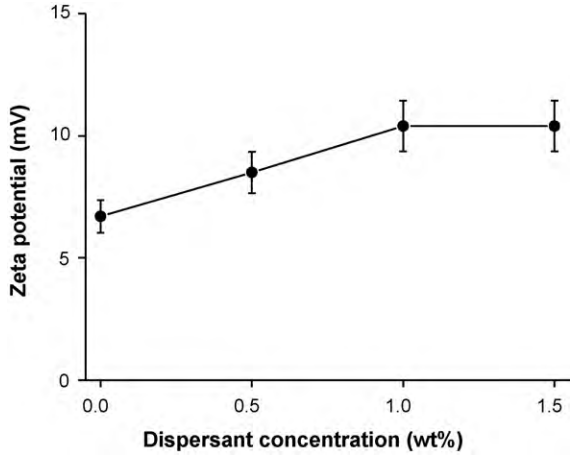

Fig. 1. Variation of zeta potential of doped lanthanum chromite suspensions in ethanol with deflocculant concentration.

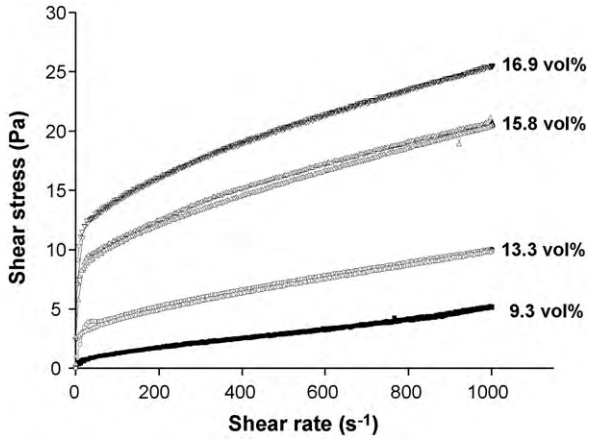

Fig. 2. Flow curves of doped lanthanum chromite suspensions in ethanol with different solids loadings.

3. Results and discussion

Since well-dispersed concentrated suspensions are required for the tape casting process, the effect of deflocculant content, solids loading and binder system was previously optimised for the suspensions in ethanol. Hypermer KD6 was selected as deflocculant due to its efficiency at low concentrations in ethanol.[17,27]

Fig. 1 shows the effect of the deflocculant content on the zeta potential of dilute suspensions, where a maximum value of 10.5 mV was achieved after the addition of 1.0 wt% deflocculant. For larger amount of deflocculant, the zeta potential remains constant in the range of study. Although this value is apparently low, it is worthy to note that particles dispersed in ethanol show lower zeta potential values than in water[28] due to the lower electrical permittivity of ethanol. Fig. 2 shows the flow curves of the suspensions with 1.0 wt% deflocculant prepared to different solids loadings and milled for 24 h. All the suspensions show a shear thinning behaviour with no thixotropy where both the viscosity and the yield stress increase with the solids loading, thus suggesting the formation of a connecting attractive network.[29] Table 1 shows the main rheological properties of those suspensions (viscosity values taken at 100 s^{-1}, thixotropy and yield point).

Fig. 3 shows the variation of relative viscosity with the volume fraction of solids (η_r–ϕ curves) of the milled suspensions calculated in the high shear rate region by fitting the experimental η_∞ data (symbols) to the Krieger–Dougherty model (continuous line), where ϕ_{max} values up to 0.20 were found, very near the maximum solids loading used in this work.

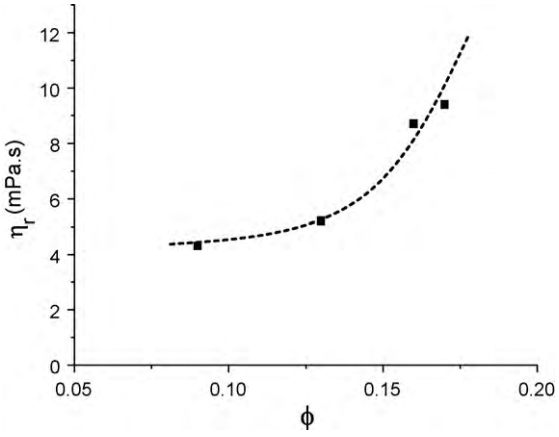

Fig. 3. Variation of viscosity of doped lanthanum chromite suspensions in ethanol with volume fraction of solids.

Once the suspensions were optimised, the effect of the binder system at different B/P ratios was studied on the rheological behaviour of the suspensions. The suspension with the highest solids loading, 57 wt%, led to high viscosity after the binder addition, thus making it necessary to reduce the solids loading of the starting suspension to 55 wt%. Fig. 4 shows the flow curves of 55 wt% slips with binder system (B + P) contents of 0, 10 and 15 wt% using B/P ratio of 1. Fig. 5 shows the corresponding CR curves of 55 wt% suspensions with 15 wt% (B + P) for B/P ratios of 0.5, 1.0 and 2.0. The viscosity of the suspension increases with the binder system content, and for a fixed content, with increasing B/P ratio. Table 2 shows the effect of the binder system on the rheological behaviour of the suspensions (yield point, viscosity and thixotropy). In general, all the tape casting

Table 1
Rheological properties of doped lanthanum chromite slips prepared in ethanol at different solid loadings.

Solids loading (wt%)	Yield stress (Pa)	Thixotropy (Pa s^{-1})	Viscosity at 100 s^{-1} (mPa s)
40	–	136	12.7
50	1.0	348	43.3
55	2.0	665	109.0
57	1.8	372	139.0

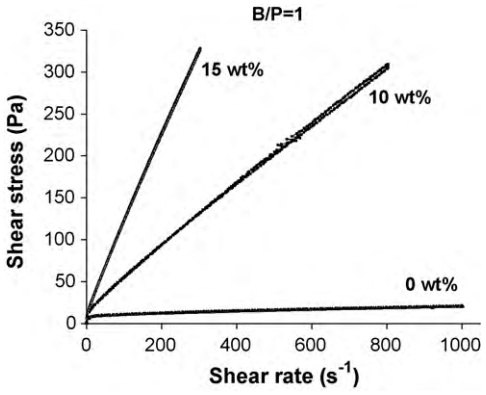

Fig. 4. Flow curves of doped lanthanum chromite suspensions in ethanol without BS and with BS contents of 10 and 15 wt%.

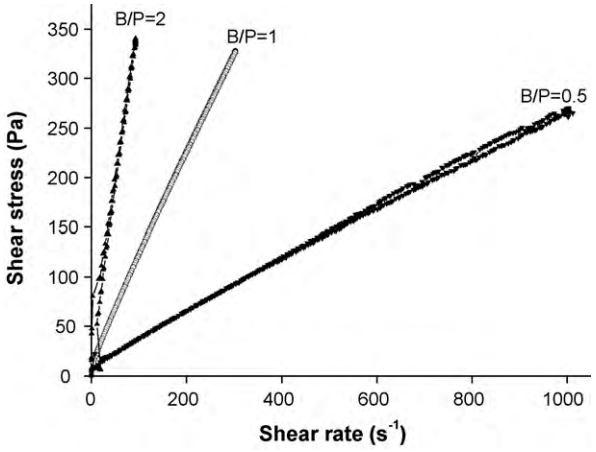

Fig. 5. Effect of binder-to-plasticizer (B/P) ratio on the flow curves of doped lanthanum chromite suspensions in ethanol with 55 wt% solids and 15 wt% BS.

suspensions showed a slightly shear thinning behaviour, with no relevant thixotropy; this means that these suspensions will show a high stability in the range of study with small differences in viscosity with shear rate, so that no variation of the properties of the samples can be expected when changing the blades height. The effect of casting conditions on the properties of the green tapes is shown in Table 3, which correlates the rheological properties of the suspensions with the casting parameters (casting speed and height blades) so that the flow behaviour during casting can be estimated.

The casting speed has a small effect on the green tape thickness. The thickness decreases with the casting speed, this being probably related to the lower viscosity of the slips passing through the blades at those strong shear conditions.

The influence of the blades height at constant casting speed ($5\,cm\,s^{-1}$) and BS content (10 wt%) was also evaluated. The gap was varied from 200 to 1000 μm. The thickness obviously increases with the gap, being 100, 130, 240, 250, and 330 μm for blade heights of 200, 400, 600, 800, and 1000 μm. When considering the ratio between the resulting thickness and the imposed gap height, a value of 0.37 ± 0.06 is obtained in all cases. This means that the thickness ratio keeps constant independently of the imposed gap. However, for lower heights the

Table 2
Rheological properties of doped lanthanum chromite slips prepared in ethanol with different BS contents.

BS (wt%)	B/P ratio	Yield stress (Pa)	Thixotropy ($Pa\,s^{-1}$)	Viscosity at $100\,s^{-1}$ (mPa s)
10	0.5	1.0	3130	380
	1	1.8	3223	583
15	1	2.2	1090	1248
	2	2.4	4406	3676

Table 3
Effect of casting parameters and rheological properties of casting slips on green tape properties.

BS (wt%)	B/P ratio	Blade height (μm)	Casting rate ($cm\,s^{-1}$)	Shear rate (s^{-1})	Viscosity at previous shear rate (mPa s)	Green tape thickness (μm)	Tapes aspect
10	1	200	5	250	451	100	Homogeneous
			10	500	403	80	Homogeneous
		400	5	125	516	130	Homogeneous
			10	250	451	120	Homogeneous
		600	5	83	573	240	Homogeneous
		800		62	625	250	Cracked
		1000	5	50	678	330	Cracked
			10	100	545	320	Cracked
15	0.5	600	5	83	392	240	Cracked
		800		62	425	240	Cracked
	1	600		83	1265	160	Cracked
		800		62	1319	230	Homogeneous
	2	800		62	3884	230	Homogeneous

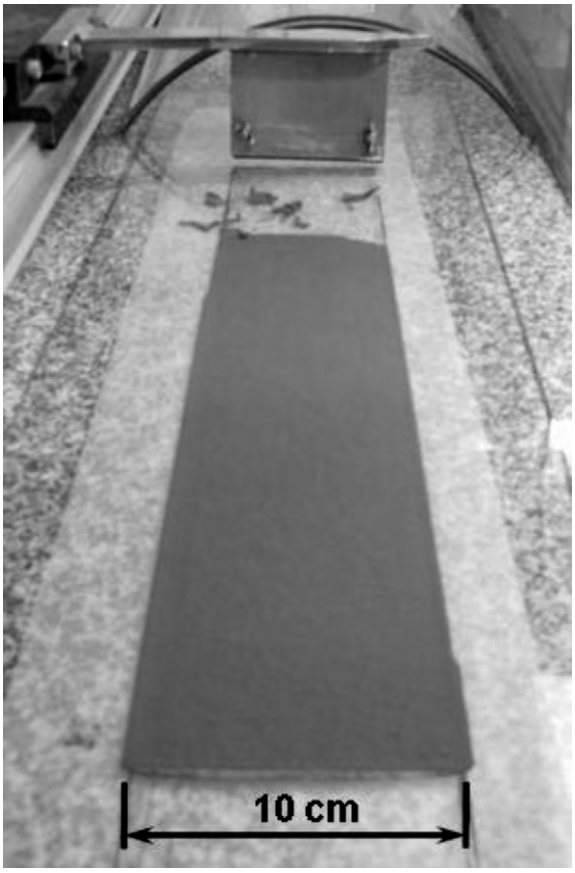

Fig. 6. As-cast tape of doped lanthanum chromite suspensions in ethanol with 55 wt% solids and 15 wt% BS.

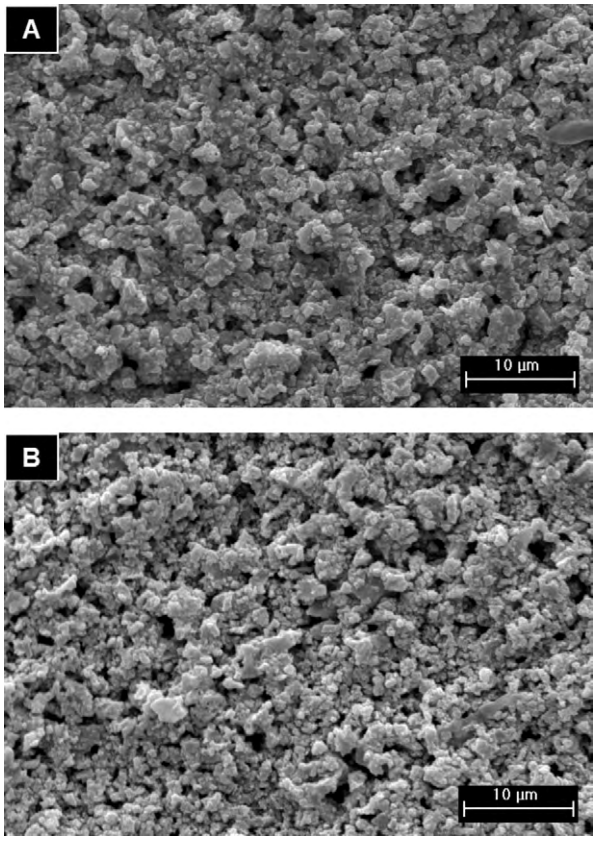

Fig. 7. SEM microstructure of top surfaces of green tapes prepared with 15 wt% BS content and B/P ratios of 1.0 (A) and 2.0 (B).

tape shows higher tendency to adhere to the substrate. Higher gaps tend to promote crack formation as the slip formulation was made considering high volatility of the solvent in order to produce thin tapes. Based on the above results, defect-free surfaces were obtained and thus, were considered adequate for further studies. Green tape thickness decreases when B/P ratio increases and when BS content increases, from 10 to 15 wt%. The best conditions to obtain a homogeneous tape were a blades height of up to 600 μm and a casting speed of up to 10 cm s^{-1} for a total content of binding system (B + P) of 10 wt%. Good results were also obtained for thick tapes obtained with a BS content of 15 wt% and B/P weight ratios of 1 and 2 and blade height of 800 μm. In these conditions, the tapes obtained with B/P ratios of 1.0 and 2.0 showed similar characteristics.

The thickness of tapes cast at 5 cm s^{-1} with a blade height of 800 μm was determined by direct observation at the optical microscope. Density was evaluated by weighting squares with 2.0 cm in side obtained by punching the green tapes. The green densities of the tapes obtained by using slips with a BS content of 15 wt%, and B/P ratios of 1.0 and 2.0, were 2.0 and 2.1 g cm^{-3}, respectively. Fig. 6 shows a view of an as-cast tape.

Fig. 7 shows the SEM microstructure performed on the top tape surfaces. The particle size distribution is uniform along the tape thickness, for both tapes. The corresponding SEM pictures of the bottom surfaces are shown in Fig. 8. There are significant differences in the microstructures as compared with the top tape surfaces. Dark regions that can be observed in both bottom surfaces can be attributed to the drying process. As it is well-known, drying process consists of two stages.[30] The first one is controlled by capillary migration of the solvent, and the second one is controlled by solvent diffusion through the solidified part of the film with a decreasing drying rate. In the case of organic media, the first drying stage is very short.[31] At the beginning 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 the casting plane and hence balancing the solvent loss. However, as the evaporation proceeds, the liquid phase becomes enriched in polymeric binder, increasing viscosity and changing fluid motion within particle–particle pores, modifying evaporation rate. During second stage of drying, internal stresses occur that could lead to cracking of the green tapes. The generated stresses are owed to two opposite phenomena: the tendency to increase the stress level due to constrained volume shrinkage and the tendency to decrease the stress level due to the relaxation of the polymeric phase. The presence of these stresses is owed to adhesion force of green tape to the substrate and shrinkage could only occur perpendicularly, along the tape thickness.

The temperature to remove the organic additives was determined by thermogravimetric analysis for all slip compositions. As shown in Fig. 9, the organic material is removed at roughly 400 °C and thus, a step at this temperature was considered in the sintering schedule.

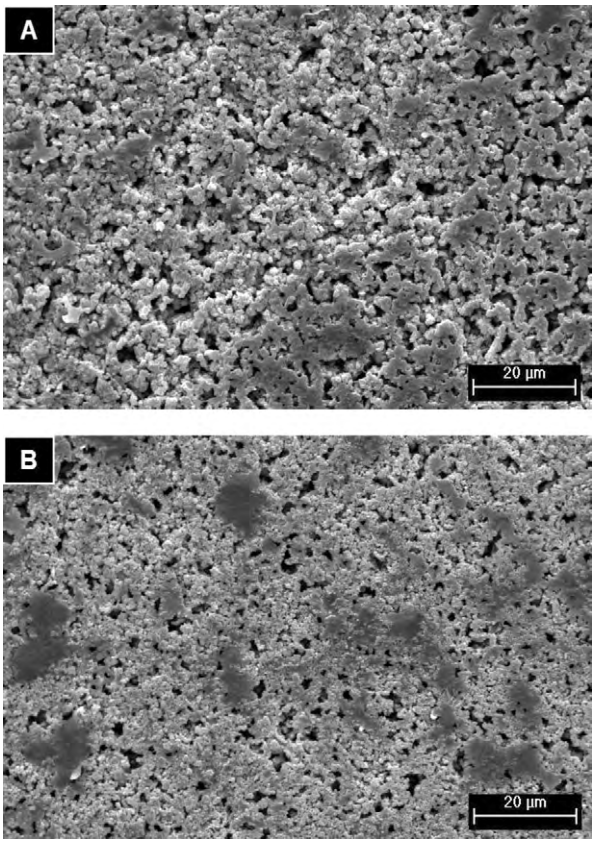

Fig. 8. SEM microstructure of bottom surfaces of green tapes prepared with 15 wt% BS content and B/P ratios of 1.0 (A) and 2.0 (B).

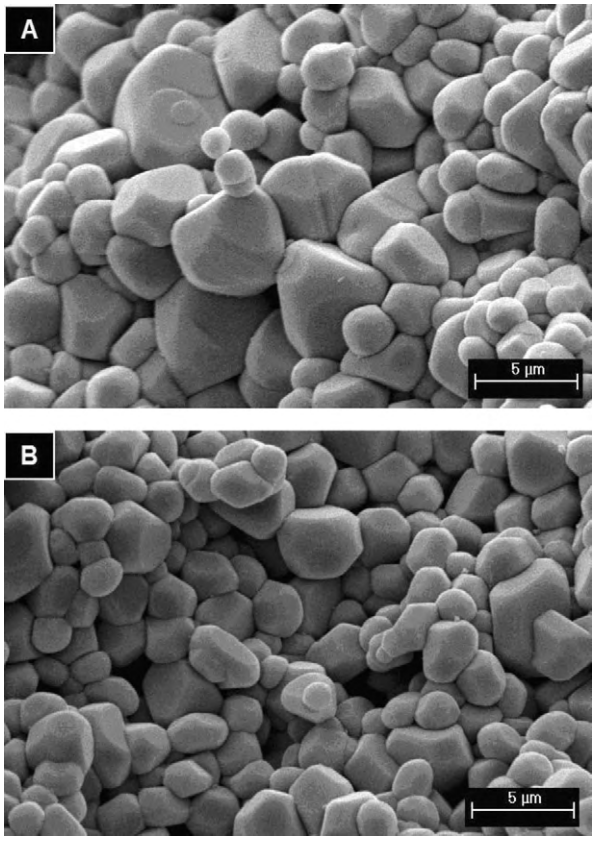

Fig. 10. Fracture surfaces of sintered tape with 15 wt% BS content and B/P ratios of 1.0 (A) and 2.0 (B).

The best tapes attained were sintered at 1600 °C/4 h in accordance with previous works.[22] The fracture surfaces for both samples are presented in Fig. 10. The microstructures show homogeneous grains distribution with intergranular fracture mode and some remaining porosity, in agreement with the density values of about 94% of theoretical, as determined by Archimedes' method for both samples. This density values are suitable for SOFC interconnector applications.

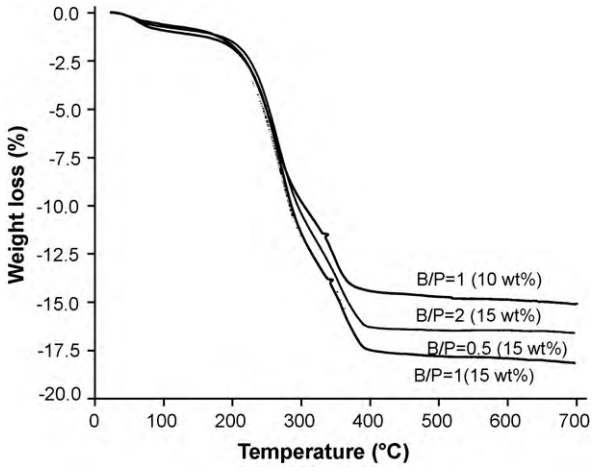

Fig. 9. Thermogravimetric analysis for the studied slip compositions.

4. Conclusions

Stable suspensions of Sr-, Co-doped lanthanum chromite were prepared in ethanol to solids loading of up to 57 wt% (16.9 vol%). Good flowability and tape casting performance was achieved for suspensions with a total content of binder and plasticizers of 15 wt%, with a relative ratio of binder-to-plasticizer of 1:1. Homogeneous tapes with controlled thickness ranging from 80 to 230 μm were obtained. Tapes prepared with 10 wt% of binding system and blades height above 600 μm cracked, as well as those prepared with 15 wt% of binding system and B/P ratio of 0.5, but increasing binder content to B/P ratio of 1 allowed to obtain crack-free tapes when increasing the blades height.

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