Research Article

Relationship between Rheological Behaviour and Final Structure of Al_2O_3 and YSZ Foams Produced by Replica

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Using rheological parameters of ceramic suspensions, it is possible to taylor the structure of the ceramic foams produced by replica. This method consists in the impregnation of a polymeric flexible template (polyurethane foam) with a ceramic suspension (slurry) containing the appropriate additives, followed by burning out organic compounds and additives and sintering the ceramic structure. In this work, ceramic foams were produced by the replica method from Al_2O_3 and 3% Y_2O_3-ZrO_2. Rheological parameters of the ceramic suspensions were investigated to improve the mechanical performance of final structures. Different types and quantities of raw materials were combined in order to select the formulations for ceramic foams. The parameters that have a significant influence on the process are the binder type and the amount of solids. Significant changes on the hysteresis area of the suspensions resulted in a lower density of macrodefects in the material. Likewise, when the shear rate viscosity is enhanced, the thickness of the struts increased proportionally. Lastly, when the hysteresis area magnitude and the ceramic thickness increased, the material with higher uniformity was internally densified, and the stress concentration of the internal defects was smoothed

1. Introduction

Ceramic foams (CFs) are a particular subset of ceramic materials with cellular structure (interconnected net of solid struts), which have properties depending both on material and topology [1].

The importance of the CF resides in the properties acquired from the synergy of the porous structure and the ceramic material. Properties such as high surface area, high permeability, and low density, due to the high porosity (usually higher than 70%), in addition to the characteristics that are inherent to ceramic materials, allow applications such as catalytic supports [2], filters for metal smelting [3], filters for hot gases [4], porous burners [5], and bone implants [6].

Ceramic foams may be manufactured by the replication method or by different types of foaming techniques

[7]. These methods have in common the use of ceramic suspensions, differing in how the structure is shaped. Replica, which is the most common manufacturing method, consists in impregnating a polymeric foam with a ceramic suspension followed by a thermal treatment in which the organic substances are decomposed and the remaining ceramic structure is sintered.

Ceramic suspensions are generally required to present shear-thinning behaviour to be suitable for the process [8]. In agreement with data related to ceramic foams manufactured by replica (Table 1 [9–17]), it is possible to conclude that there is a relationship between the characteristics of the suspension and the structure of the ceramic foams. Particularly, the time-depending parameters, which are reflected by hysteresis area (HA) on a rheogram, play an important role. However, a direct relationship between

TABLE 1: Powder, rheological, and process parameters for ceramic foams manufactured by replica.

| Ceramic powders | Rheological parameters | | Process parameters | | Reference |
	Shear depending	Time depending	Solid concentration (wt%)	Sponge ppi	
Al_2O_3	Pseudoplastic	Thixotropic	70	10	[9]
Mullite	Pseudoplastic	Thixotropic	55	50 and 65	[10]
	Pseudoplastic	Thixotropic	60	25 and 50	
SiC/Al_2O_3	Pseudoplastic	Thixotropic	80	5 and 9	[11]
$SiC/Al_2O_3/bentonite$	Pseudoplastic	Thixotropic	76	13	[12, 13]
	Pseudoplastic	Thixotropic	80	13	
$SiC/Al_2O_3/SiO_2$	Pseudoplastic	Thixotropic	80	11	
Si_3N_4/Al_2O_3	Pseudoplastic	Thixotropic	70	7 and 9	[14]
LZSA/bentonite	Pseudoplastic	—	65	50 and 60	[15]
Cordierite	Pseudoplastic	Thixotropic	52	10	[16]
YSZ	Pseudoplastic	Thixotropic	80	10 and 20	[17]

TABLE 2: Formulations of YSZ (Z) and Al_2O_3 (A) suspensions, basis related to total composition (additives + solids = 100 wt%).

System	Defoamer (wt%)	Solids (wt%)	Solvent type	Dispersant type	Dispersant (wt%)	Binder type	Binder (wt%)	pH modifier (wt%)
Z1	0.0	80	Ethanol	DS009	0.8	WB4101	3.5	0.8
Z2	0.0	80	IPA	DS001	0.4	WB40B-53	3.5	0.0
Z2b	0.0	75	IPA	DS001	0.4	WB40B-53	3.5	0.0
Z2c	0.0	70	IPA	DS001	0.4	WB40B-53	3.5	0.0
Z3	0.5	80	Ethanol	Darvan C	0.6	WB40B-53	5.0	0.0
A1	0.0	60	Ethanol	Darvan C	1	WB4101	8.0	0.0
A2	0.0	65	Ethanol	Darvan C	1	WB40B-53	5.0	0.0
A3	0.0	65	Water	Darvan C	1	WB40B-53	5.0	0.0
A4	0.0	65	IPA	DS001	0.4	WB40B-53	3.5	0.0

rheological behaviour, manufacturing process, and the final structure of ceramic foams was not established up to now.

Additives play an important role in defining the rheological behaviour of suspensions and the mechanical properties of green and sintered bodies. A higher adsorption of a dispersant improves the efficiency of powder packing, resulting in a green part with higher density [18]. The dispersant also hinders the sedimentation of ceramic suspensions [19]. Binders are used for enhancing the green strength and slowing down the setting time when in solution [20, 21]. Water or an organic liquid is used as solvent to provide a viscous medium with a required consistency. Either a base or an acid may be added to a suspension to prevent it reaching the isoelectric point, thus avoiding coagulation or phase separation [22].

Ceramic foams should present mechanical reliability. A critical point in manufacturing them by the replication method is to obtain a uniform coating on the polymeric template, in particular on the triangular shape corners that become a sharp hole in the ceramic foam struts, causing stress concentration and low strength [23, 24].

This paper investigates the effect of suspension characteristics (rheological behaviour) on the structure of alumina and yttria-stabilized zirconia (YSZ) foams produced by replica.

2. Experimental

2.1. Materials. In order to determine a suitable suspension composition, a study was undertaken according to an orthogonal array L_{18} [25] for a total of 108 tests, including as factors ceramic powder type (YSZ and Al_2O_3), defoamer amount, solids content, solvent type, dispersant type and amount, binder type and amount, and ammonium hydroxide amount, as described in Table 2.

Ceramic Powders: yttria-stabilized zirconia (YSZ, 3 mol% Y_2O_3, D_{50} = 2.5 μm, Zircoa, USA), alumina (Al_2O_3, D_{50} = 2.3 μm, Alcoa, Brazil).

Foams: polyurethane foams (PUF, 20 and 65 ppi, Foam Partner, Switzerland).

Dispersants: three alternative ammonium polymethacrylates (Darvan C, R.T. Vanderbilt, USA; DS001 or DS009, Polymer Innovations, USA).

Binders: water-based acrylic polymers (WB4053 or WB4101, Polymer Innovations, USA).

Solvents: distilled water, ethanol (97 vol%, Campota, Colombia), or isopropyl alcohol (IPA, Campota, Colombia).

Defoamer: acrylic polymer with partially neutralized acid groups (DF002, Polymer Innovations, USA).

pH Modifier: ammonium hydroxide (30 wt%, Campota, Colombia).

Three formulations for each powder (YSZ: Z1, Z2, and Z3; Al_2O_3: A1, A2, and A3) were used to obtain ceramic foams (Table 2).

2.2. Processing

Mixture. The powder was mixed with the additives into a ceramic suspension for 5 min at 1800 rpm using a high shear stirrer (BYK, Dispermat, Germany) with a tooth profile geometry.

Impregnation. The polyurethane foams ($25 \times 25 \times 25 \pm 1$ mm) were immersed under compression into the ceramic suspension. Afterwards, each impregnated PUF was manually compressed for removing the excess of suspension and promoting an open cell structure.

Drying. The samples were dried in air at room temperature for 24 h.

Thermal Treatment. The dried foams were brought to a high temperature oven (MHI, USA). The treatment consisted in increasing the temperature at a heating rate of 0.5°C/min until 500°C, followed by a heating rate of 10°C/min until 1600°C, and a holding time of 2 h at the maximum temperature.

2.3. Characterization

Ceramic Powders. The particle size distribution of the powders in an aqueous suspension was determined by laser scattering (1064, CILAS, USA). Figure 1 shows a narrow monomodal distribution for YSZ with a maximum at 3 μm, and a wide bimodal distribution for Al_2O_3 with maxima at 2 and 20 μm.

Ceramic Suspensions. The rheological measurements were carried out in a rotational rheometer (AR-G2, TA Instruments, USA) with a parallel-disc geometry (diameter 20 mm) with a gap of 1000 μm. Tests were performed at 25°C with the shear rate ($\dot{\gamma}$) varying from 0.1 to 300 s^{-1}, using a solvent trap to avoid evaporation. The samples were submitted to a preshearing at 1 s^{-1} for 60 s.

Ceramic Foams. A sample of each foam was sectioned with a diamond disc saw (Isomet 1000, Buehler, USA). The microstructure of the foams was evaluated by scanning electron microscopy (SEM, JSM-6490LV, Jeol, Japan). The thickness of the struts of the foams was measured randomly in 5 different regions, with an average of 30 measurements in each region.
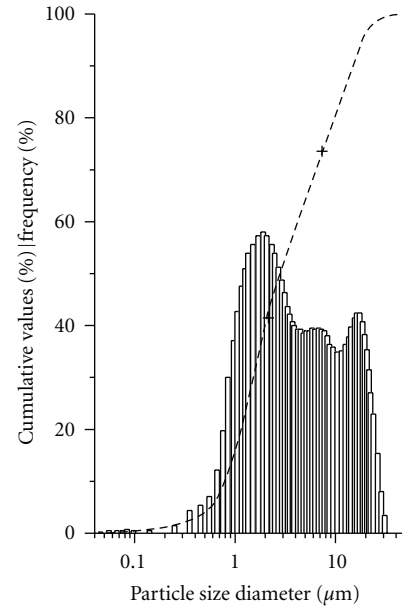
(a)
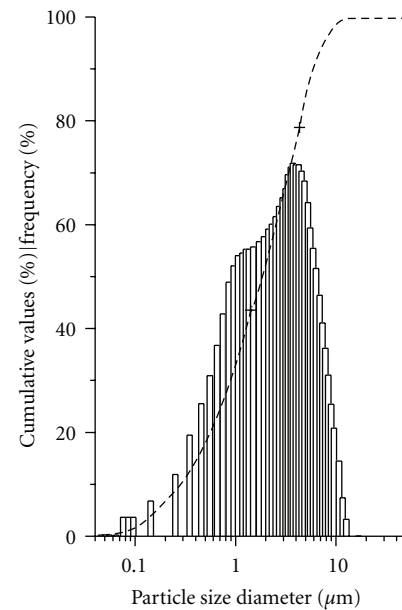
(b)

FIGURE 1: Particle size distribution for ceramic powders, (a) Al_2O_3; (b) YSZ.

3. Results and Discussion

3.1. Formulation

Solids. The amount of ceramic powder in the suspension was a highly determinant factor in the process. All foams with <80 wt% YSZ fractured during the thermal cycles. In the case of Al_2O_3 foams, the suspensions with <65 wt% solids also ruptured during the thermal cycles, with the exception of A1, which was prepared with a high amount of binder (8 wt%). These materials were used as received.
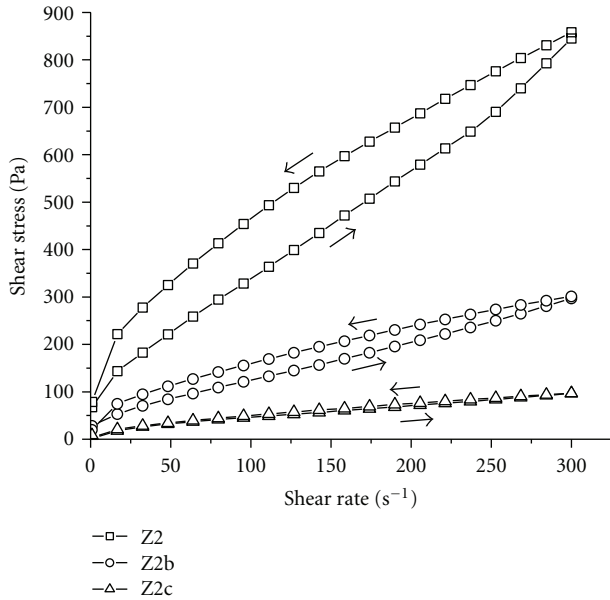
FIGURE 2: Time-dependent rheological behaviour of YSZ suspensions with different solid contents (Z2c, 70 wt%; Z2b, 75 wt%; Z2, 80 wt%).
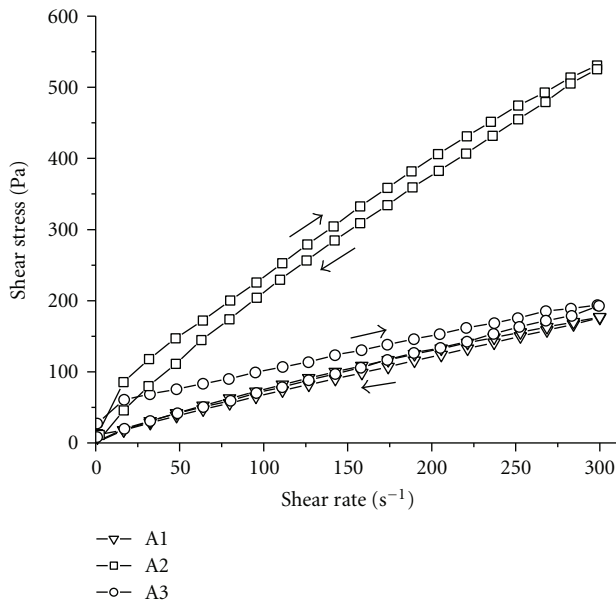

FIGURE 3: Time-dependent rheological behaviour of alumina suspensions.

Defoamer. This additive presented a low influence on the process. In most cases, a defoamer was not required (Table 2).

Solvent. Most of the samples that resisted to the process were those formulated with organic solvents (ethanol and IPA) due to the molecular structure. The hydrophilic part of the molecule is adsorbed on the oxides, because they are highly
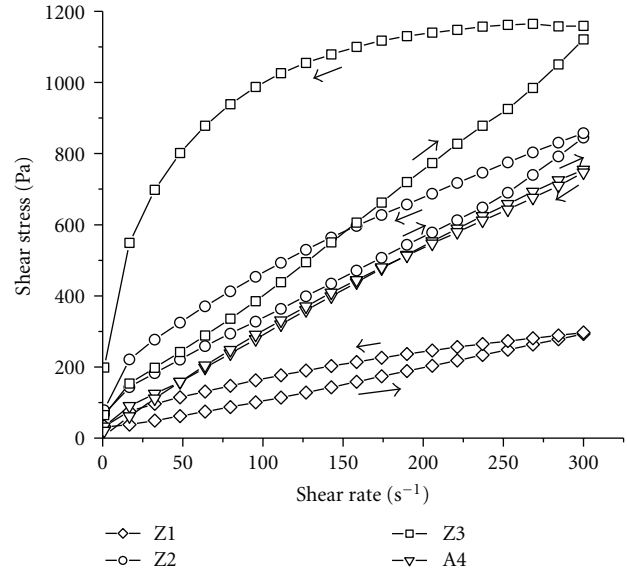

FIGURE 4: Shear stress (τ) versus shear rate ($\dot{\gamma}$), where $\tau = \eta \dot{\gamma}$, formulations Z1, Z2, Z3, A4.

polar, and the hydrophobic part is compatible with the apolar struts of the polyurethane foam [26].

Dispersant. Suitable ceramic foams were obtained regardless of the quantities and types of dispersants used. Therefore, it is implied that the manufacture is not sensible to variations of this additive.

Binder. For binder content <3.5 wt%, the manufacturing process was not successfully completed, due to the higher amount of binder that improved the ceramic material densification [12].

pH Modifier. This additive presents a low influence on the processm and, in most cases, the foams could be obtained without it (Table 2), probably due to the decrease of the isoelectric point caused by the dispersant addition [27].

The main adjustment of the formulation should be related to binder and solids content, since variations in these factors affect notably the manufacturing process. This effect is attributed to the retention of the suspension on the polyurethane surface, which is in turn influenced mainly by solids content and binder type and amount [28].

3.2. Rheology of Ceramic Suspensions. The suspensions were characterized rheologically, including parameters such as the hysteresis area (HA) and the viscosity at high shear rates (η_∞). These effects were determined, respectively, from the area between the curves of the rheogram (Figure 2) and from an adjustment of the high shear area based on the model of Cross [22]. All suspensions measured were pseudoplastic with variable degrees of thixotropy. These results are in accordance with the rheological data found for silicon carbide [12] and 8 YSZ [17] suspensions.
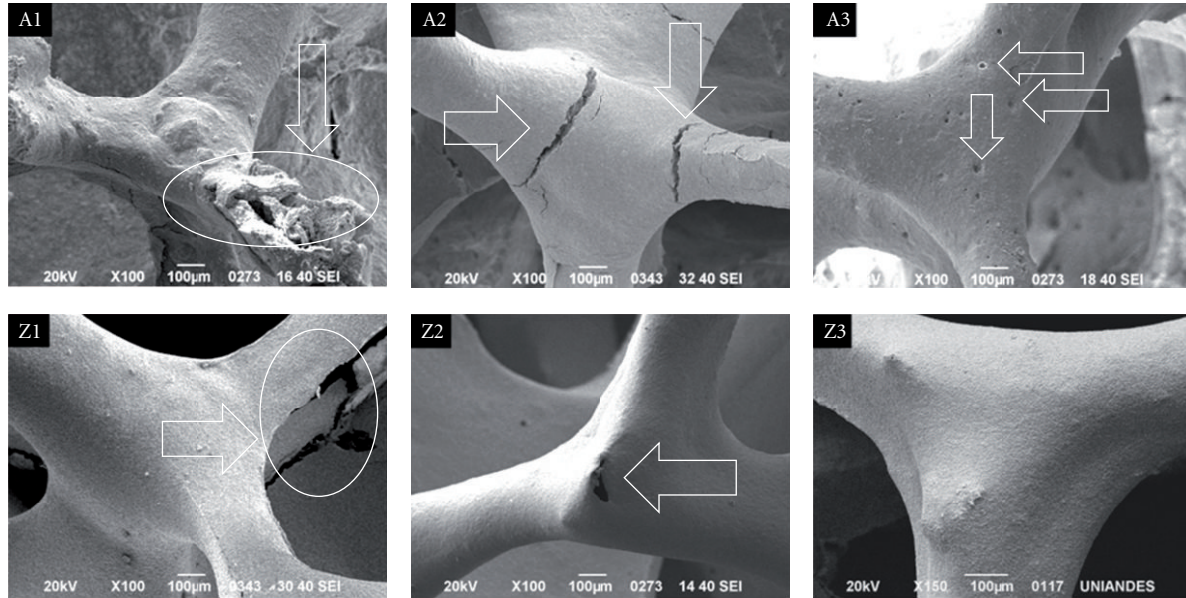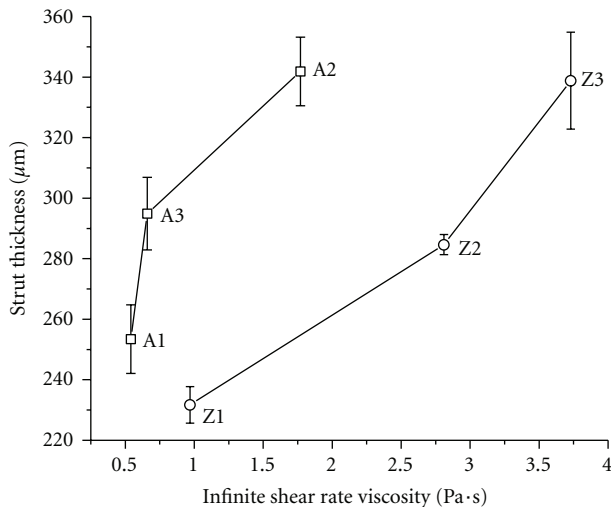
Figure 5: Vertexes of the manufactured ceramic foams.

Figure 6: Mean thickness of ceramic struts (t) versus viscosity at infinite shear rate (η_∞), 95% confidence interval.

Table 3: Rheological parameters measured for YSZ (Z) and Al_2O_3 (A) suspensions.

System	Solids (wt%)	Binder (wt%)	HA (Pa/s)	η_∞ (Pa·s)
Z1	80	3.5	12970	0.97
Z2	80	3.5	29790	2.81
Z2b	75	3.5	8401	0.99
Z2c	70	3.5	1041	0.32
Z3	80	5.0	123700	3.73
A1	60	8.0	4801	0.54
A2	65	5.0	6994	1.77
A3	65	5.0	7302	0.66
A4	65	3.5	1063	1.48

Solids Content. Figure 2 shows the rheograms of YSZ suspensions with different solid contents (Table 3). The magnitude of the hysteresis area (HA) increases as the solids content increases, and this effect is attributed to increases in particle interaction. Higher apparent viscosity is obviously observed for suspensions with higher solid concentrations.

For alumina suspensions, as in Figure 3, the amount of binder and/or the solids seems to be dominant in increasing the apparent viscosity for the same solvent (A1; 60 wt%, A2; 65 wt%).

In the systems Z2, Z2b, and Z2c, an increase of approximately three times for η_∞ with the addition of 5 wt% solids can be observed, due to the increase of solids fraction (φ). The suspensions start to pack, and the thickness of the hydrated interparticle layers is reduced, diminishing the sliding among the powder particles.

Binder. The rheograms in Figure 4 (systems Z1, Z2, Z3, and A4) show the effects on the rheological behaviour caused by changes in binder amount and/or type (Table 3). The hysteresis area (HA) increases when the content of binder is increased (Z2, A4; 3.5 wt%, Z3, A3; 5 wt%), possibly due to steric repulsion caused by the higher concentration of polymers in solution. Moreover, the Al_2O_3 suspension (A4) presents neglectable hysteresis, at least for low binder content (3.5 wt%). Regarding the binder type, WB40B-53 causes a higher increase in HA and viscosity when compared to WB4101 (Z1, Z2). This probably corresponds to an increase on fluidity of the medium diminishing the particles interaction; both binders have similar composition with exception of 7% of plasticizer on the formulation of WB4101.

The additives introduced in the suspensions (binder and dispersant) stabilize the particles system due steric and/or
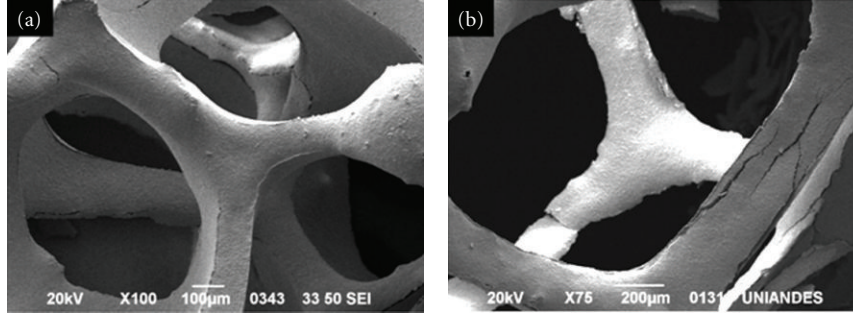
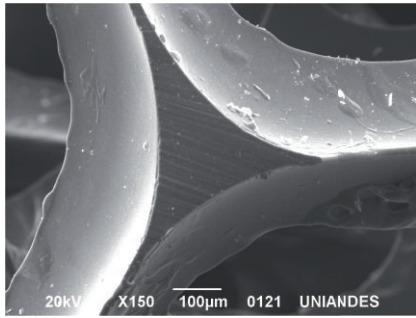FIGURE 7: YSZ foams (Z2) manufactured from PUF: (a) 65 ppi, (b) 20 ppi.

FIGURE 8: Typical form of the PUF (20 ppi).

electronic repulsion, this adsorbing part of them on the particle surface [21]. When a polymeric additive is adsorbed by the particles, the solids fraction (φ) is replaced by a higher effective fraction (φ_{ef}). In this way, the addition of polymers (present in the binder and dispersant) has a similar effect to the solids increase. The effective fraction can be estimated as follows [22, 29]:

$$\varphi_{ef} = \left(1 + \frac{2\delta}{r}\right)^3 = \frac{\left(V_{solids} + V_{polymer}\right)}{V_{total}}, \qquad (1)$$

where r is the particle radius; δ is the thickness of the polymer layer; φ is the solids volume concentration; φ_{ef} is the volume effective fraction; V_{solids}, $V_{polymer}$, and V_{total} are the volume of solids, polymer, and total of the suspension, respectively. For the calculations it was assumed that no agglomeration occurs due to the mixing step. Thus, the fraction of solids as example for Z2, Z3, which was initially $\varphi = 40.0\%$ for both systems, increases to $\varphi_{ef} = 43.5\%$ and $\varphi_{ef} = 44.6\%$, respectively. The results support the idea that the increase of binder amount has a similar effect of solid increase on viscosity.

Type of Solvent. For the systems A3, A2, the infinite shear rate viscosity is approximately three times higher for ethanol as solvent. This is attributed to probable formation of microbubbles in the ceramic suspension that diminish the interaction between particles; this is reflected on the foam macrostructure (Figure 5-(A3)). However, the magnitude of HA is slightly higher for water as solvent; this result suggests that the organic solvent diminishes the effect of

particle interactions and the formation of pseudostable configurations on the suspension.

Type of Ceramic Powder. The difference in HA due to the use of alumina and zirconia powders is shown in Figures 3 and 4. The smaller HA for Al_2O_3 ceramic suspensions may be attributed to the wider distribution of particle sizes, which provides for a better particle packing and decreases the suspension viscosity. This effect of the viscosity in ceramic suspensions has been reported elsewhere [30].

3.3. Ceramic Foams. The differences in the structure of the manufactured ceramic foams (Z1, Z2, Z3, A1, A2, and A3) were evaluated at macro- ($>50\,\mu$m) and microscale ($\leq 50\,\mu$m). The inherent defects of the replica method might be observed. After drying cracks were not observed in any sample, This suggests that macrodefects were developed after removing the PU template.

Macrostructure. Figure 5 shows SEM micrographs of the different manufactured foams.

Ceramic foams A1 and Z1 exhibit cracks along the structure. A2 presents cracks in regions where the struts connect with the vertex. Holes and cracks may be observed in Z2. A3 foams present a lower relative amount of cracks compared to the other Al_2O_3 systems. In this case, small round-shaped imperfections are observed; these are attributed to microbubbles on the precursor suspension. For Z3, small holes in the vertexes were detected.

The mismatch in thermal expansion between PUF and ceramic is considered the main source of crack formation [9]. However, the results of Figure 5 and Table 3 disclose that the macrostructure defects decrease with the increase of HA (A1 to A3; Z1 to Z3). Probably higher HA enhances the packing fraction with time, inherently helping to increase the green strength.

In Figure 6, it is observed that the thickness of the foam struts correlates well with the viscosity at high shear rates of the suspension. A similar behaviour was reported for SiC suspensions, in which the viscosity η_∞ of $1.9\,Pa\cdot s$ corresponded to a strut thickness of $300\,\mu$m [14].

Previous investigations showed that the smaller the size of the PUF cell, the smaller the thickness of the vertexes [31]. The same effect is expected for lower solids load in the
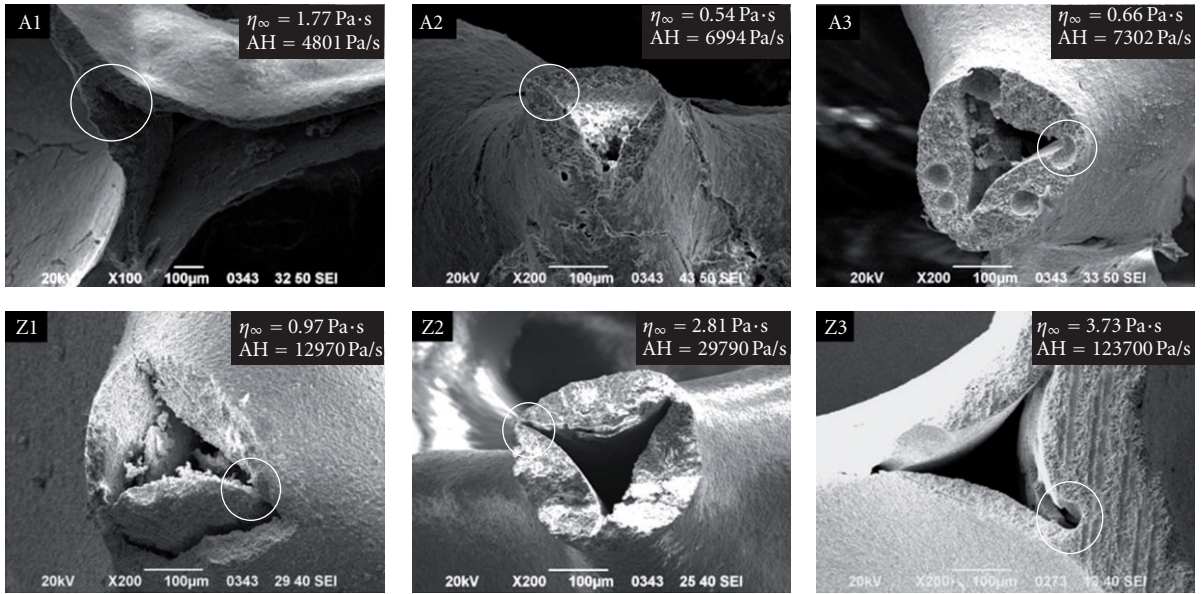
FIGURE 9: Internal defects of the ceramic foam made with PUF of 20 ppi, system of Table 3.
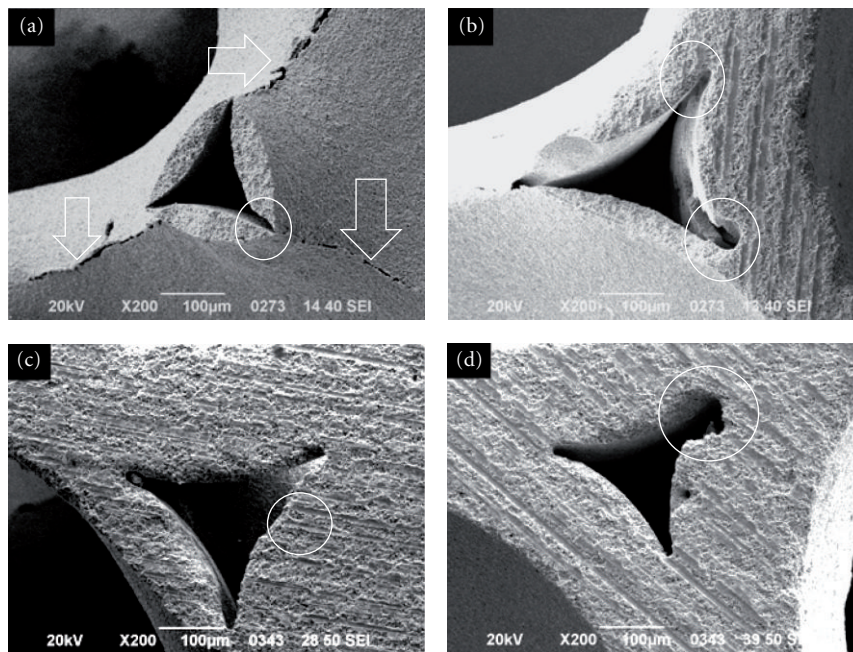

FIGURE 10: Internal defects for different struts of manufactured foams with the system Z3.

ceramic suspension, which corresponds to a low viscosity of the suspension and consequently to thinner ceramic struts.

The SEM micrographs of foams with different cell sizes showed that a smaller density of defects was found for 65 ppi foams when compared to 20 ppi foams (Figure 7).

The strength of the ceramic foams depends on the defects, regardless of the struts thickness [31, 32]. However, for thicker struts, the probability of finding defects is higher [33].

Furthermore, cracks appear mainly in the regions where the polymeric coating is thinner, as observed in the literature [9].

The mechanical strength of the ceramic foams is governed by the amount of defects. An internal void or pore is formed for instance, after polyurethane is burned out; Figure 8 shows a typical vertex of a 20 ppi PUF.

Moreover, the angled shape of the PUF originates a high stress concentration in the ceramic foam, and this is the main
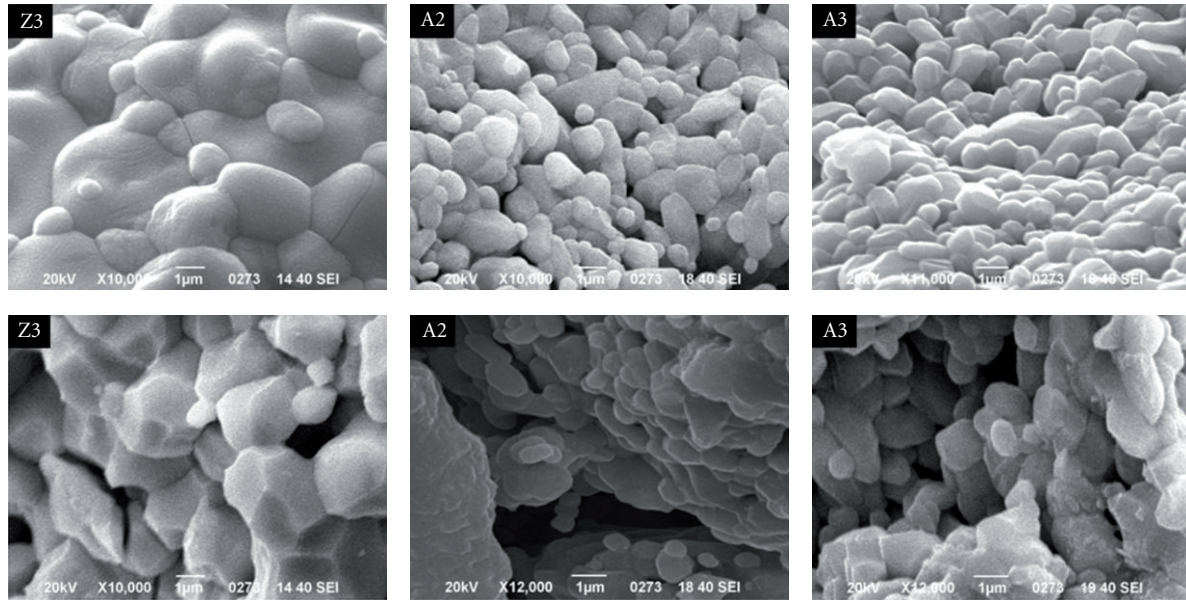
FIGURE 11: Microstructures: external (top) and internal in the spaces left by the PUF (bottom), foams A2, A3, and Z3.

reason of the low mechanical strength of the material [9, 23, 24].

Figure 9 shows different types of defects in the manufactured foams. It can be observed that the increasing of the HA and/or viscosity of the ceramic suspension results in smoothing the corners of the internal void.

In Figure 10, different wall thicknesses are shown for the YSZ system (Z3). Thicker coatings lead to smoother corners of the internal defect. In addition, as shown in Figure 7, the ceramic thickness is proportional to η_∞.

Microstructure. The surface of sintered foams presented some different features (Figure 11). A higher HA corresponded to a better packing and a higher strength of the ceramic foam.

4. Conclusions

It is possible to taylor the structure of the ceramic foams using rheological parameters of ceramic suspensions, such as the hysteresis area (HA) and viscosity at high shear rates (η_∞).

The factors that influence most of the formulation in the replica process are the solid content and the binder (amount and type). These components (solids and binders) affect HA and η_∞ of the ceramic suspensions. Higher HA indicates a higher packing fraction, helping to increase the green strength. In this case, the densification inside of the voids is more homogenous, decreasing the cracks nucleation and allowing the smoothing of the stress concentrations. On the other hand, the increasing of η_∞ in the ceramic suspensions causes an increase on the thickness of the ceramic coating, which facilitates the smoothing of the corners of the internal defects increasing the strength of the ceramic foams.

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