3D printing of zirconia-alumina composites using DLP Stereolithography

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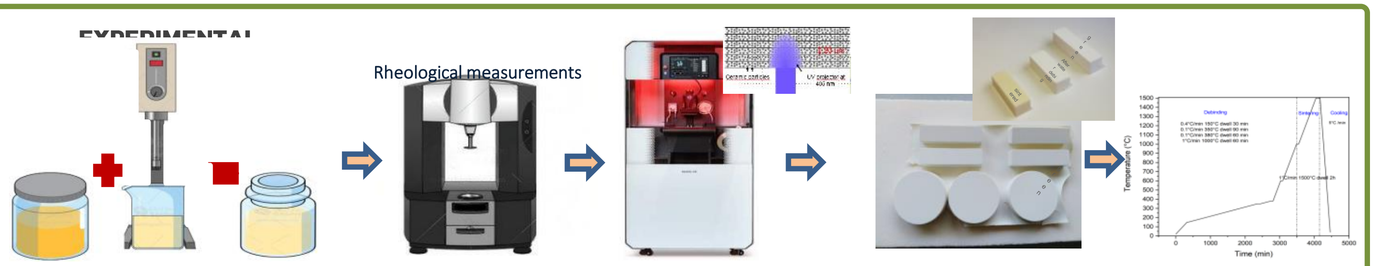
INTRODUCTION

Additive manufacturing (AM) of high-performance ceramics is of great interest because it allows most issues regarding conventional ceramic shaping techniques to be overcome. Digital light processing (DLP) based stereolithography (SLA), is a promising AM technique for the production of parts with complex geometries, very high spatial resolution, and fine surface finishing.

The printability is strongly correlated with the rheological properties of ceramic ink. In this study, the printability of the slurry and the rheological and mechanical properties of ZrO_2-16 vol% Al_2O_3 composites through the SLA process were studied. A microstructural evaluation of sintered specimens was performed to observe their morphology and to measure the grain size. The Bending strength was measured by a three-point bending test. These ceramics present excellent mechanical properties, combining outstanding fracture strength and toughness with high aging resistance, thus making them suitable for mechanical and biomedical applications.

EXPERIMENTAL



RESULTS

1) Particle size distribution of the powders

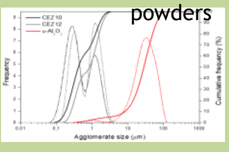


Fig 1. Particle size distribution of *AS-RECEIVED CEZ 10, CEZ 12 and α-alumina*

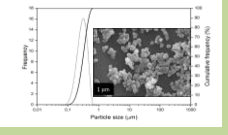


Fig 2. Particle size distribution of the composite powders *AFTER 18 h OF DISPERSION IN BALL MILLER*

Ceria-stabilized zirconia (10 and 12 mol% CeO_2 into CEZ10 and CEZ 12 respectively) and α-alumina were mixed to develop the composite: 11Ce-ZrO_2/16vol% α-Al_2O_3. The particle size distributions of the as-received powders show a certain level of agglomeration (Fig 1) but, in spite of this, after ball-milling, the mixture achieved a monomodal and fine distribution with mean particle size values of about 0.3 µm (Fig 2). For preparing the slurry, the dried powders were added to the acrylic resins, by changing the solid loading from 50 to 78 wt%, with the addition of a dispersant Disperbyk 103 to the liquid monomer.

2)Rheological properties

Two categories of slurries were prepared: the first with only the biphasic powder and the commercial resin, the second with the addition of the dispersant, Disperbyk 103. All the slurries show a shear-thinning behaviour (viscosity decreased with increasing shear rate). The addition of the dispersant permitted a high solid loading to be reached, while maintaining a low viscosity.

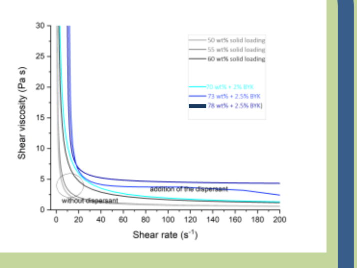
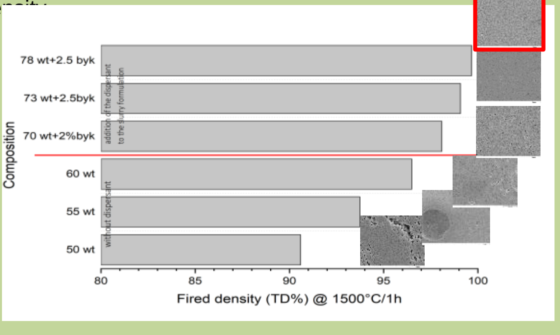


Fig.3 Shear viscosity of the slurry without (three grEy curves) and with the dispersant Disperbyk 103 added to the formulation

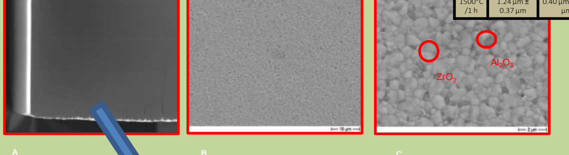
3) Effect of the solid loading and dispersant on the fired density



The histogram shows the slurry formulations at increasing solid loadings. Slurries without (50-60 wt%) and with (70 wt%+2% byk, 73 wt%+2,5 byk and 78 wt%+2.5% byk) the dispersant are compared. While the fired density increases with the solid loading, the presence of defects decreases in the microstructure. The addition of the dispersant allows very high densification with a defects-free microstructure to be achieved.

4)Microstructure and mechanical properties

GRAIN SIZE		
	Mean grain Size ZrO2	Mean grain Size Al_2O_3
1500°C /1 h	1.24 µm ± 0.37 µm	0.40 µm ± 0.15 µm



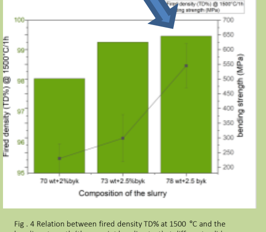


Fig . 4 Relation between fired density TD% at 1500 °C and the bending strength (three-point bending test) at different solid loadings

The cross-section micrographs (A) of the 78 wt% + 2.5% byk printed sample sintered at 1500° C/1h show good compactness. The microstructure is free from large defects and there is no delamination. At higher magnification (C), a good distribution of the two phases can be observed. A TD% > 99% was reached. Fig. 4 shows that the increase of the solid loading decreases microstructural defects and improves the bending strength of the sintered bars (45x5.6x4.2 mm). 545 MPa was reached for the 78wt%+2.5 byk.

CONCLUSION

Dense zirconia-alumina composites were successfully fabricated by DLP-stereolithography 3D printing technology. The optimal formulations achieved >99% theoretical density. Results revealed that the ceramic ink has an appropriate viscosity behaviour, which decreased with increasing shear rate. The microstructural observations carried out on the sections and surfaces showed highly homogeneous structures, free from the typical 3D printing flaws, such as delamination between layers and cracks due to organic matter decomposition. In addition, a fine microstructure with a good second-phase particle distribution was observed, demonstrating that SLA is a promising technique for fabricating dense ceramic components. The printed specimens with the formulation 78 wt% + 2.5% byk exhibit promising mechanical properties, however, the bending strength value obtained, 545 MPa, has a margin of improvement. Further research will be carried out to minimize the residual defects will be further reduced by working on the printing parameters and the whole process, with a resulting enhancement of the mechanical properties. The results achieved demonstrate that it is possible to avoid long machining post-processing, in future, more complex structures, with even more tailored microstructures, will be fabricated with the aim of fully exploiting this challenging technology.

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