MICROSTRUCTURAL EVOLUTION OF 3YSZ FLASH SINTERED WITH CURRENT RAMP CONTROL

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Ceramics sintered by Flash Sintering (FS), sometimes have heterogeneity in their microstructure. Among the possible causes of this problem, the formation of hotspots is probably the principal issue observed in flash sintering. The hotspots are formed due to the heterogeneity of the resistance of current passing in the green sample. In this way, in the flash phenomenon, the current finds preferential paths, carrying on non-uniform thermal runaway. In this work, we observed that one way of reversing this problem was gradually increasing the electric current density at FS. For this, 3YSZ was shaped in cylindrical form (5 mm height and 6 mm diameter) and flash sintered, at a tubular setup proposed before[1], under the application of an AC electric field of 120 V/cm (RMS basis) applied from the beginning of the furnace heating. Three different electric current density ramps were studied: 0.012, 0.024 and 0.048 $A.s^{-1}$, named as Z1, Z2 and Z3 respectively, until they reached the maximum value of 100 $mA.mm^{-2}$ (the moment the power supply was turned off). For comparison purposes, conventional FS (Z0) was performed using the same electrical parameters. The electric source, in this case, remained on after reaching 100 $mA.mm^{-2}$ for 142 s (time calculated to reach the same total energy supplied by the electrical source of samples Z1). After sintering, the apparent densities of the samples were measured according to the Archimedes principle. For analysis of the microstructure, the samples were cut radially and three regions of each sample were observed in SEM: center, right and left surfaces. The grain size distribution was made for each region using ImageJ software. The apparent density of samples sintered by FS was 94 % (Z0) and samples with electrical current ramp were 93 %, 92 % and 87 % for Z1, Z2 and Z3, respectively. The apparent density is proportional to the total energy supplied to the sample (energy provided by the power supply and thermal energy provided by the furnace). Thus, the density of Z0 was expected to be close to the density of Z1. As samples Z2 and Z3 had more abrupt ramps, the energy supplied was lower, which resulted in lower apparent density. The grain size distribution indicates that the mean grain size between the three different regions did not present statistically significant differences (ANOVA test) for the same sample.

Figure 1 shows the microstructure of sample Z1 in three different regions. It was suggested that the gradual increase of the electric current during the FS prevented the formation of hotspots and consequently resulted in homogeneous microstructures. The Z0 sample presented a statistically significant difference in grain sizes between the surfaces and the center. In FS the heating is very fast, which causes heterogeneity in the microstructure due to thermal retention in the center of the sample, while in the surfaces the temperature is lower, due to energy losses by radiation. When the electric current increases gradually the heating ramp will be smoother, so there is no significant heat retention in the center of the sample[2]. Thus, the gradual rise of the current during flash sintering provides a better homogeneity of grain size along the center of the same sample. However, for the experiments studied here, the densification of sintered samples with the current ramp was lower than that of the sintered sample in FS. Therefore, for better densification results, an even longer ramp could be the solution.

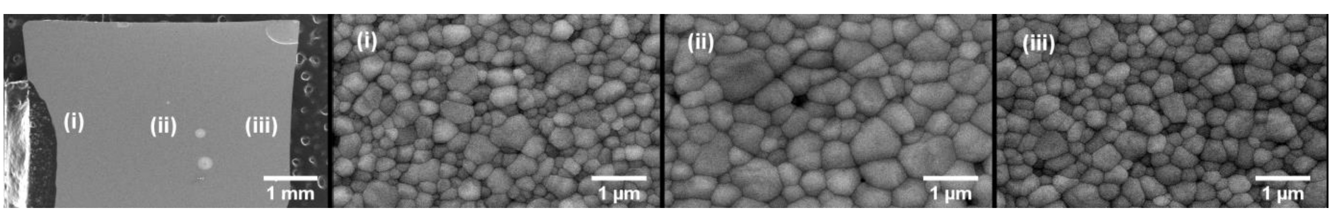

Figure 1 – Overview of the Z1 sample and micrographs of (i) left surface, (ii) center, and (iii) right surface.

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