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# First stage in flash sintering of zirconia based ceramics

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Flash sintering ability to densify ceramic material in less than 5 seconds, at much lower furnace temperatures than conventional sintering has been reported and studied for a large set of conductive ceramics and recently of composite ceramic materials. Since no die is used, the current mandatorily flows through the sample and the flash process is characterized by an abrupt increase in the sample conductivity accompanied by shrinkage and densification. In the literature two types of experiments are reported, referring to the furnace temperature: **Constant heating rate (CHR)** and **isothermal** experiments. The flash sintering elementary mechanisms are not yet fully understood. It has been recognized that, in isothermal experiments, the flash densification stage ("flash stage") is observed after an incubation period during which no macroscopic densification is observed ("pre-flash stage", or first stage); and in the CHR experiments, the densification temperature varies according to the intensity of the applied electric field. According to the literature and to our previous works, the effective electrical conductivity of the sample is a key parameter of the flash effect. In the present work, this parameter was varied using conducting/insulating ceramic composites with different amounts of insulating phase. This presentation reports and analyses the result of a study on the effect of a non-conductive second phase, alumina, on the first stage of the flash sintering of zirconia based ceramics in both CHR and isothermal furnace experiments. Spray dried 8 mol% $Y_2O_3$ zirconia (from Tosoh) and alumina - 3 mol% $Y_2O_3$ zirconia powder blends provided by Baikowski Company were used, with alumina volume fraction ranging from 0 to 60%, enabling a wide range of effective conductivity values.

In the **CHR experiments** samples were heated from 20 to 1500°C at 10°C/min and the electric field was applied from 500°C. In this case, the **flash temperature** is the experimental macroscopic analyzed result. For **the isothermal experiments**, the samples were preheated at different temperatures $T_0$ (900 to 1400°C) before applying the electric field. In this case the **delay time (or incubation time)** to observe the flash event is the experimental macroscopic analyzed observation. All experiments were performed under constant AC (1kHz) electric field $E_0$, with $E_0$ of 100 and 200V/cm. The sample electrical conductivity was measured by impedance spectroscopy and recorded during all the sintering cycle.

Regarding the pre-flash period, our results show that the flash event is related to sample conductivity (which is determined by the amount of non-conductive phase) in both CHR and isothermal experiments. The data analysis for both types of experiments shows that the flash conditions are determined by a balance between the electric power supplied to the sample and the heat losses. Experimental data are in agreement with a thermal process in which the internal Joule dissipation heating competes with external heat losses. A simple model is proposed on this basis, which predicts the experimentally observed scaling laws.

The observation of alumina-zirconia eutectic microstructures in flash sintered composite samples and microstructure evolution of 8YSZ powder confirms that internal sample temperatures reached during the flash are in agreement with the temperature estimations obtained from the recorded conductivity evolution.