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A simple flash sintering setup under applied mechanical stress and controlled atmosphere

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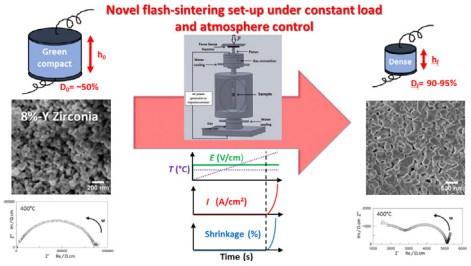
GRAPHICAL ABSTRACT



ABSTRACT

Most flash sintering experimental set-ups use dog bone-shaped specimen and DC current, which results in heterogeneously distributed densification and grain growth throughout the sample. This is the reason why only the sample's core characteristics after flash are taken into account. On the other hand, some recent procedures suggest the use of cylindrical pellets, which have some advantages compared to the traditional mode as the use of easily conformed samples and its final uniformity.

Our new experimental set-up offers the possibility of atmosphere control and pressure application. Also the electrodes material change can be easily made when necessary. Shrinkage measurements and impedance spectroscopy are realized in situ and experimental parameters, as oven heating for example, can be varied to control microstructure changes.

Advantages:

- Sample can be entirely recovered at the end of the experiment and can be analyzed throughout its entire extension, including regions in contact with the electrodes that may present some differences from pellets inner part.

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- The use of AC current enables the study of different frequencies effects.
- Experimental set-up can be adapted to different kinds of electrolytes (samples), easily changing electrode's material and atmosphere.

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Methods details

Flash sintering is a current assisted sintering technique able to densify samples in short periods of time (as fast as 5 s) and most importantly, at furnace temperatures significantly lower than in conventional sintering [1,2]. It was firstly reported for yttrium-stabilized zirconia and later it has been proved successful for a large range of ceramic oxide materials [3–8].

Traditional flash sintering experiments use a dog-bone shaped sample hanged in the furnace center by two metallic (platinum) wires which act simultaneously as electrodes and sample's support. Some new procedures, including one of ours [2,9], use easily pre-conformed pellet-shaped samples placed between two platinum discs electrodes. The sample's only contacts are the electrodes, so is mandatorily for the current to pass through the ceramic, which is the flash sintering technique principle.

The flash sintering is characterized by an abrupt increase in the current density through the sample, accompanied of shrinkage and densification. Samples are required to be slightly conductive in order to allow current flow and since each type of electrolyte has a different conduction mechanism, platinum electrode and oxygen-rich atmosphere may not be suitable for all kinds of electrolytes.

We developed a new experimental set-up that allows atmosphere change and control, the appliance of a mechanical pressure to improve densification, and also the electrode material change when necessary.

Despite any SPS (spark plasma sintering) similarities, our set-up do not involve the use of dies, forcing current to pass through the sample; uses much smaller pressures; uses pre-conformed samples; allows the use of the desired atmosphere; and the most important characteristic: flash sintering allows densification to happen in just a few seconds.

Sample preparation

Samples are uniaxial and isostatic cold pressed under 60 and 250 MPa, respectively, resulting in green relative densities of about 50–55% TD. Pellets form and dimensions are optional as long as there are flat surfaces with enough area to insure a good contact with the electrodes.

Set-up electrodes discs and wires are made of platinum. Sample is recovered with a layer of a metallic ink in both flat surfaces to insure intimate contact. Platinum grids can also be used to improve contact between set-up electrodes and electrode layer in the sample.

An interesting aspect of using a pellet sample instead of a dog-bone shaped sample is that the electrode material can be changed (changing the metallic ink layer between the ceramic and the platinum electrode) to ensure the electrical and electrochemistry compatibility between the electrode and sample material.

Set-up description

Flash sintering set-up consists in a vertical furnace where the sample is located in the center of an alumina tube, only in contact with both electrodes (grids optional). The set-up is divided in three main