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The onset of the flash transition in single crystals of cubic zirconia as a function of electric field and temperature

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ABSTRACT

We have measured the onset of flash in cubic zirconia single crystals, in experiments similar to flash sintering. This behavior is compared with results on sintering of porous specimens of 8–10 mol% cubic zirconia. The single crystals flash at lower temperatures and lower electric fields, which does not support an unalienable role for particle-particle interfaces in the flash mechanism. The flash onset in single crystals occurs at similar values of the power density as the powder samples, which, furthermore, lies in the same range as the onset of the flash transition in several oxides.

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1. Introduction

Flash sintering has been shown to possess three essential characteristics, a highly non-linear rise in conductivity [1], electroluminescence [2] and a colossal rate of diffusional mass transport which produces ultrafast rates of sintering [1]. The rates of sintering have been shown to be three to four orders of magnitude faster than in conventional sintering [3]. For example, yttria stabilized zirconia which nominally requires several hours to densify at 1400–1500 °C, can be sintered in mere seconds at a furnace temperature of ~800 °C [1], and specimen temperature of 1100–1200 °C [4,5]. Flash experiments can be carried out at isothermal furnace temperatures [6], where an electric field is applied as a step function, or by applying an electric field and then heating the furnace at a constant rate [1]. Once the flash initiates it is necessary to switch the power supply to current control.

The role of particle-particle interfaces in porous specimens remains of interest in the physics of flash sintering. Local heating at such interfaces [7,8], even melting [9,10] have been proposed as the underlying cause. Therefore, we ask whether or not single crystals, which are devoid of such features, may also exhibit the flash effect. Rather surprisingly, the single crystals flash at lower temperatures, and at lower electrical fields, than powder specimens, which is contrary to the proposal that particle-particle interfaces are the source of flash sintering.

The electrical power density dissipated in the specimen increases abruptly at the onset of the flash. Indeed the value of the power density at the flash transition has become of interest since it has been shown to fall within a narrow range for flash sintering of many different ceramic materials [11]. We examine how far the onset of the flash in single crystals of yttria stabilized cubic zirconia agrees with this behavior.

2. Experimental

Single crystals of cubic zirconia (8 mol% yttria stabilized zirconia) cut into parallelepiped shaped specimens, with a rectangular cross-section, were used. Thin platinum wires, wrapped around the ends of the sample, served as the electrodes, leaving an effective gage length of 4 mm with a cross section of 2 mm × 0.7 mm. The gage length was aligned along the [100] axis of single crystal. Platinum paste was applied to improve electrical contact between the wire and the sample. The samples were hung with these platinum wires within a furnace (Applied Test System, PA, USA). Electrical power was provided from a 600 W DC power source (Sorensen 300-2, Sorensen, San Diego CA). The current was measured with a digital multi-meter (Keithley 199) connected to a National Instruments GPIB-USB interface. Data was acquired through a graphical-user-interface developed in-house, with MATLAB.

Electric field was applied at room temperature. It ranged from 50 V cm^{-1} to 490 V cm^{-1}. The current limit was set to 25 mA mm^{-2} for all experiments. The furnace was heated at a constant rate until the onset of the flash, which was signaled by an abrupt increase in the conductivity of the specimen. This critical value of the temperature depended on the strength of the applied field. The power supply was switched from voltage to current control upon reaching the current limit. The samples were held in a constant state of flash, briefly, under current control before turning off the power supply to the specimen. The power dissipated was calculated from the product of voltage and current and then normalized with respect to the volume of the sample, and expressed in units of mW mm^{-3}.

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3. Results

The specimen was suspended within a conventional furnace, the field was applied and the furnace was heated as a rate of 10 °C min^{-1}, starting from room temperature. When the specimen flashed the power supply was switched to current control, which was held for ~10 s before the power to the specimen was turned off. A current limit of 25 mA mm^{-2} was used for all experiments. The principal variable in the experiments was the electric field, which was increased from 50 V cm^{-1} up to 490 cm^{-1}, in steps of 20 V cm^{-1}.

The voltage and current were measured continuously through the experiment, and their product was used to calculate the power expended in the specimen. These results, showing the change in the power density, and the current density, are given in Fig. 1. The curves show the characteristic flash transition, most clearly seen when the current reaches its preset limit and the experiment is changed from voltage control to current control. At higher fields the transition occurred sooner, that is at lower temperatures and is, apparently, sharper. Indeed, at these low temperatures the transition is so sharp that the power supply, which is stated to switch from voltage to current control in 50 ms, is unable to comply and current overshoots the prescribed limit, albeit only very briefly.

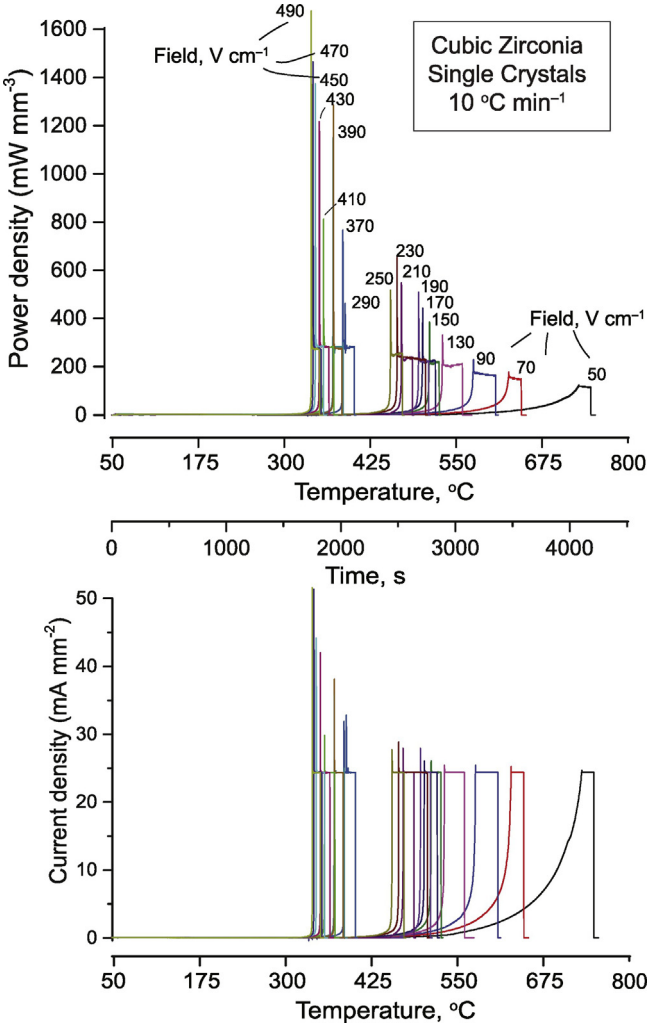

Fig. 1. (upper) Flash transitions in power density, and (lower) in current density in constant heating rate experiments. Note the current limit plateau when the power supply switches from voltage to current control. The overshoot at high electric fields arises from the delay in changing to current control, which is 50 ms according to specifications for the power supply.

The field and temperature combinations for the transition are plotted in Fig. 2(a). Data for powder specimens from the literature are included in Fig. 2(b). They are remarkably different. While 2500 V cm^{-1} is required to flash the powder samples near 300 °C, the single crystals flash at a similar temperature at a field of only 490 V cm^{-1}. These differences are highlighted in Fig. 2(c). While both sets of data show the same trend, the single crystals are far easier to flash than the powder samples. These results contradict the idea that local heating at particle-particle interfaces are the genesis of the flash phenomenon. It is more likely that the event occurs within the crystal matrix.

In constant heating rate experiments the flash transition is most clearly evident in the Arrhenius plots of the power dissipation. Below the transition the conductivity is expected to be thermally activated. Near the transition the power dissipation in the specimen rises quickly.

Such Arrhenius plots are shown in Fig. 3. Note that despite the wide range of fields and temperature for the onset of the flash, the transition occurs within a narrow band of the power density. Indeed, such behavior has been seen in experiments with various oxides as well [11]. In the present work the transition bifurcates into a low temperature, and a high temperature regime. The low temperature regime persists to a field of about 230 V cm^{-1} and up to ~400 °C with the data falling within a band of 4–8 mW mm^{-3}. The power dissipation falls into a higher band of 20–40 mW mm^{-3} above 500 °C and at fields from 210 V cm^{-1} down to 50 V cm^{-1}. The implication of these results is discussed in the next section.

The self-similarity of the shape of the power transition was explored further. By inspection we note that the transitions become more gradual, that is they are slower, as the field is reduced and the transition temperature rises. We wondered if this could be a result of an internal clock associated with the mechanism of the flash. This hypothesis was explored by compressing the time scale for the data at different temperatures to see if the results could be made to overlap. The result of this exercise is given in Fig. 4. The upper graph in Fig. 4(a) shows the transition for different fields over a time span of 20 s. The time is counted from the instant that the power dissipation reaches a value of 50 mW mm^{-3}. A period of 20 s from this reference point envelops all the transitions from 230 V cm^{-1} to 490 V cm^{-1}. The transition, from this starting point, occurs earlier at higher fields than at lower fields. The curves in Fig. 4(b) show the same data, but where the scale for the slower transitions is compressed until they match the data at the highest field. For example 20 s time period at 490 V cm^{-1}, where the flash occurs at 315 °C, is equivalent to 7 s for the same "real time" period at 230 V cm^{-1} which occurs at 415 °C. In other words, 20 s at 315 °C is equivalent to 7 s at 415 °C. Thus it would appear that increasing the temperature from 315 °C to 415 °C accelerates the intrinsic time clock by a factor of about three, and so on. There may be a role for the electric field in the time-temperature relationship, as well.

The results in Figs 3 and 4 suggest that the flash is instigated at a specific value of the power density. Since the specimen is under voltage control during the transition, the power density is directly related to the specific conductivity by

$$P_W^* = \Gamma^2 \sigma^* \tag{1}$$

where P_W^* is the power density at the transition, Γ is the electric field (under voltage control), and σ^* is the specific conductivity at the transition temperature, T^*, which are related by $\sigma^* = \sigma_o \exp(-\frac{Q}{RT^*})$, where σ_o is the pre-exponential, Q is the activation energy, and T^* is the transition temperature. Since P_W^* is found to have an approximately constant value, we obtain the following relationship between the applied field and the transition temperature

$$\Gamma^* = \sqrt{\frac{P_W^*}{\sigma_o}} \exp\left(\frac{Q}{2RT^*}\right) \tag{2}$$

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