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Field assisted sintering of electro-conductive ZrO_2-based composites

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Abstract

In order to reveal the fundamentals of the field assisted sintering technique (FAST), also known as spark plasma sintering (SPS), the evolution of the current density and temperature distribution in the punch-die-sample set-up during FAST of ZrO_2–TiN powder mixtures was modeled by finite element calculations supported by in situ measured electrical and thermal input data. The thermal and electrical properties of partially sintered composite powder compacts were estimated using theoretical mixture rules, allowing to calculate the current density and temperature distribution inside the tool and the specimen during the FAST sintering process. The electrical properties of the sintering composite powder compact, and hence the thermal distribution in the sinter set-up, changed drastically during densification once percolation occurred. Based on the calculated thermal distribution inside the composite powder compact, an optimal tool-powder compact design was determined in order to process electrically conductive ZrO_2–TiN composites from electrical insulating powder compacts within minutes with high reproducibility.
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Keywords: Spark Plasma Sintering (SPS); Electrical properties; Thermal properties; ZrO_2; Composites

1. Introduction

The field assisted sintering technique (FAST), also known as spark plasma sintering (SPS) or pulsed electric current sintering (PECS), belongs to a class of sintering techniques that employ a pulsed DC current to intensify sintering.[1] Some general advantages of field assisted sintering, compared to traditional hot pressing or hot isostatic pressing, are technological advantages such as short processing time, the use of high heating rates hereby minimizing grain growth often leading to improved mechanical,[2] physical[3] or optical[4] properties and elimination of the need of sintering aids.

Lately, a lot of research is focused on a more fundamental understanding of the sintering process. Recent studies focused on the effect of (a) the electrical current and (b) the DC pulse pattern on the solid state reactivity of Mo and Si,[5,6] on the effect of contact resistances on the temperature distribution during the FAST process in case of monolithic fully densified materials[7,8] and on the enhanced sintering kinetics and diffusion mechanisms during the sintering process.[9]

Furthermore, high heating rates, especially in combination with short dwell times, can cause temperature gradients and subsequently sintering inhomogeneity leading to non-uniform microstructural and mechanical properties of the sintered parts.[10–12] Therefore, the temperature field within the sintering powder compact during FAST sintering should be understood and controlled as well as possible. Up till now, most researchers controlled the temperature during a FAST sintering cycle by focusing a pyrometer on the outer die wall surface, leading to an underestimation of the sintering temperature. In order to correlate this temperature with the temperature of the sintering powder compact inside the die, the temperature distributions within the whole tool-specimen system should be known. The most practical way to find this out is theoretical modelling.

Previous research performed at our institute pointed out the importance of both the contact resistances induced by the graphite papers and of the electrical properties of the specimen. When a fully dense TiN compact was placed inside the FAST equipment, the radial temperature gradient at high temperatures (>1500 °C) was much higher compared to the gradient in a fully dense 3Y–ZrO_2 compact.[8] Based on these observations, the thermal cycle controlling pyrometer was positioned more strategically i.e. focussing on the bottom of a borehole inside the upper punch of a graphite tool set-up (Fig. 1). In this way, the temperature at the specimen's centre differed less than 5 °C from the temperature measured by the central pyrometer, independent on the sample's electrical properties. Furthermore, it

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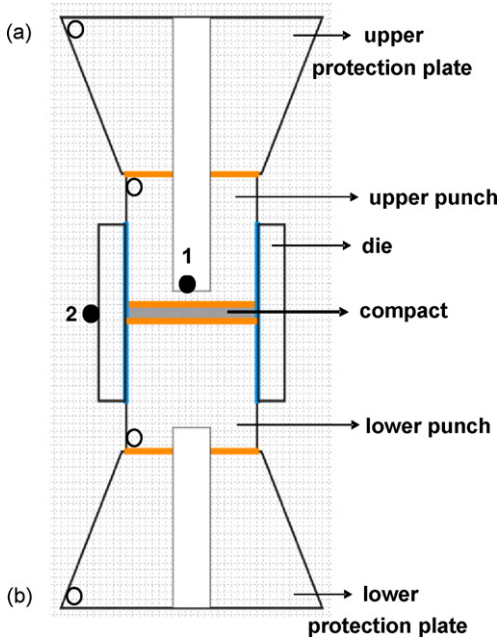

Fig. 1. Cross-section of the tool used during FAST cycles. The position of the 1 mm diameter boreholes (O) used for voltage measurements or for the positioning of thermocouples: (a) upper protection plate; (b) lower protection plate. The vertical and four horizontal graphite papers are highlighted. Points 1 and 2 are the focus points of the two pyrometers used. The white areas indicate the position of the boreholes.

was suggested that the radial temperature gradient in electrically conductive samples can be reduced by reducing the radiation heat losses from the outer die wall surface. Therefore, the die can be surrounded by porous carbon felt insulation.

In the present work, the developed finite element method and ANSYS code were used to predict the thermal gradients inside a sintering ZrO_2–TiN (60/40) composite during the FAST process. The simulated data were in very good agreement with the experimentally measured temperature and resistance values. Interrupted sintering cycles were performed in order to correlate the shrinkage of the powder compact with its changing thermal and electrical properties.

2. Experimental procedures

ZrO_2–TiN (60/40) powder compacts were prepared by ball milling 3Y–ZrO_2 (Daiichi HSY-3U, $d_{50} = 20$ nm), TiN (H.C. Starck, grade C, $d_{50} = 1.4$ μm) and a small amount (0.75 wt.% with respect to the Y–ZrO_2 content) of Al_2O_3 (Taimicron, TM-DAR, $d_{50} = 100$ nm) in a multidirectional Turbula T2C mixer.

Experiments were performed on a FCT FAST device (Type HP D 25/1, FCT Systeme, Rauenstein, Germany). More details about the used FAST device can be found elsewhere.[8] During the presented experiments, a pulse-pause combination of 10–5 ms is used throughout all the experiments. Controlling the power, which is done by controlling the voltage difference over the electrodes, generates a preset time–temperature profile. In this way, the current flowing through the specimen-punch-die set-up is controlled. During the experiments, the temperature is measured by a central pyrometer with a focus point at the bottom

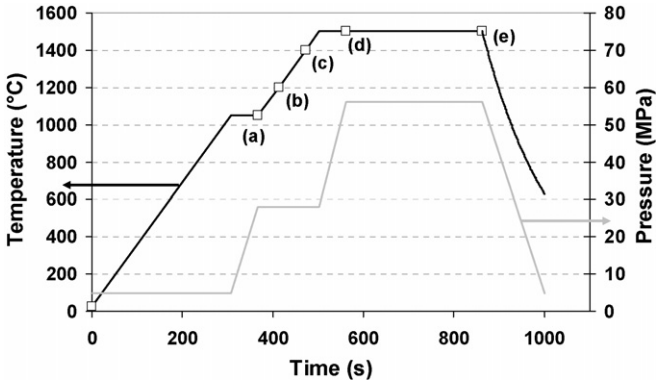

Fig. 2. Pressure–temperature profile used during processing of the ZrO_2–TiN (60/40) composites. In case interrupted sintering cycles were performed, the current was switched off at the points indicated by the open symbols. In any case, the total processing time, including cooling, was less than 25 min.

of the central borehole of the graphite set-up, 2.88 mm from the bottom of the upper punch and 5.125 mm from the centre of a 4.25 mm thick sample inside the die (Fig. 1). The temperature of the die is measured by a second two-colour pyrometer, focussed on the outer die wall surface at the same height as the centre of the compact (Fig. 1).

A standard temperature and pressure cycle is shown in Fig. 2 and consists of six segments: (1) applying a constant current until the central pyrometer reached a temperature of 450 °C, the onset temperature of the pyrometer, (2) applying a linearly increasing current resulting in a heating rate of 200 °C/min in the temperature region between 450 and 1050 °C, (3) applying a constant current during the dwell period at 1050 °C (during this dwell period the pressure was increased from 5 to 28 MPa), (4) applying a linearly increasing current resulting in a heating rate of 200 °C/min in the temperature region between 1050 and 1500 °C, (5) applying a constant current during the dwell period at 1500 °C (during the first minute of this dwell period with a total length of 6 min, the pressure is increased from 28 to 56 MPa, and (6) applying no current during cooling from 1500 °C down to room temperature and lowering the pressure at the same time. The interrupted sintering cycles were performed at different temperature and pressure combinations as indicated by the open symbols in Fig. 2.

The electrical resistivities of fully dense, hot pressed ZrO_2–TiN composites with a TiN content varying between 35 and 95 vol.% TiN were measured at room temperature using a four point contact technique.

3. Results and discussion

3.1. Interrupted sintering cycles using a ZrO_2–TiN (60/40) powder compact

Typical microstructures of FAST sintered ZrO_2–TiN (60/40) composite powder compacts obtained at 1050 °C (a), 1200 °C (b), 1400 °C (c), 1500 °C for 1 min (d) and 1500 °C for 6 min (e) are shown in Fig. 3. Table 1 describes the evolution of the density of the sintering compacts as a function of temperature and applied pressure. The major part of the densification takes place

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