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Enhancing efficiency of field assisted sintering by advanced thermal insulation

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ABSTRACT

The influence of advanced thermal insulation on energy consumption and temperature distribution during electric field assisted sintering of conductive stainless steel powder and non-conductive zirconia powder was investigated. Four types of tool setup were considered: i) without insulation, ii) with die wall insulation, iii) with additional insulation of die faces and iv) with spacers manufactured from carbon fiber reinforced carbon composite (CFRC). The influence of thermal insulation on energy consumption was experimentally studied for samples with diameter of 17 mm. The temperature distribution in samples with diameters of 17 mm, 50 mm and 150 mm was modeled using the Finite Element Method. The power consumed during dwell was almost half the value when die wall insulation was used. The additional insulation of die faces and the application of CFRC spacers provide a threefold decrease in power during sintering of steel powder and a fivefold reduction during sintering of zirconia powder. The advanced thermal insulation significantly homogenizes the temperature distribution within samples of small and medium size. The advanced thermal insulation provides a strong decrease in the temperature gradient inside large conductive sample with a diameter of 150 mm. However, insulation apparently cannot ensure acceptable temperature homogeneity within non-conductive parts of such diameter. The reason for this is the specific current path and related heat concentration near the sample edge.

1. Introduction

Field Assisted Sintering Technique (FAST), also known as Spark Plasma Sintering (SPS), is a sintering technology based on resistive heating to consolidate powders in an electrically conductive die. Sintering during FAST/SPS is additionally supported by uniaxial pressure. This specific configuration allows densification of ceramic and metallic powders with a tailored microstructure at a high heating rate and with a short processing time. The direct contact between the FAST/SPS tool and water-cooled metallic electrodes provides rapid and/or controlled cooling of sintered samples. Easy sample preparation, flexibility in sintering parameters, short sintering cycle and the availability of highly sophisticated equipment explains the popularity of the FAST/SPS technique within the scientific community. Nowadays, this technique is successfully used in many laboratories worldwide to sinter metals, alloys, ceramics, composites, functionally graded and other advanced materials. Commonly, sintered samples are small in size (10–20 mm in diameter) and only a few sintering cycles a day are performed. Therefore, matters such as energy consumption, productivity and sintering homogeneity are hardly discussed in the literature. However, these issues become important when the industrial application of this technology is considered in line with its actual development trend. Recently, unique FAST/SPS facilities for sintering of components with diameters of up to 500 mm were manufactured and installed. Automatized lines were developed and put into operation. Further up-scaling and commercialization of the FAST/SPS process requires a critical analysis of energy consumption and the development of new measures for its reduction. In addition, sintering inhomogeneity associated with temperature distribution must be investigated for parts of different sizes, and approaches to reducing it must be developed.

A typical FAST/SPS tool setup consists of cylindrical die, two punches and spacers contacting with water-cooled metallic electrodes (Fig. 1).

All tool elements (die, punches and spacers) are usually manufactured from a high-strength, isostatically pressed graphite. The initial powder is separated from the die and punches by a graphite foil. This

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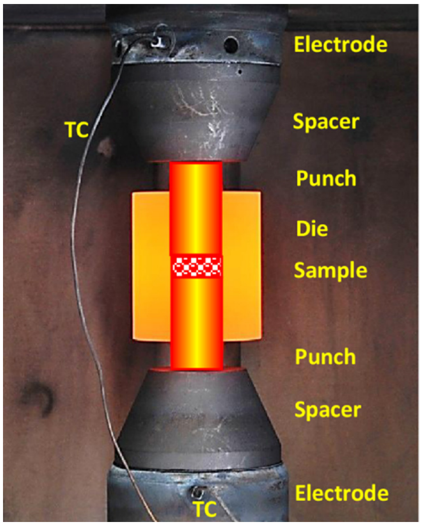

Fig. 1. Components of FAST/SPS tool setup and two thermocouples (TC) for measurement of electrodes temperature.

foil improves the thermo-electrical contact between the powder and the tool, and protects them from sticking to each other. The Joule's heat is generated as a consequence of current flow throughout all electrically conductive components. The heat is redistributed within the setup owing to thermal conduction. Some part of the heat is lost due to radiation from the external setup surface. Another portion of heat is lost because of the water cooling of electrodes. Such a setup can be considered an open thermal system with internal heat sources. The corresponding thermal balance can be formulated as $W_j = W_h + W_r + W_c$, where W_j is the Joule's heat generated due to current flow; W_h is the heat consumed during powder and tool heating-up; W_r is the heat loss due to radiation and W_c is the heat loss due to electrodes cooling. In an energetically effective system, the W_r and W_c components should be minimized. Obviously, thermal insulation of the tool from the electrodes as well as from the ambient reduces the energy consumption during sintering.

There are only a few publications discussing the thermal balance in the FAST/SPS setup. Zavaliangos et al. (2004) reported a radiation-related contribution to a total heat loss ranging from 20% at a temperature of 350 °C to 60% at a temperature of 1350 °C. Vanmeensel et al. (2007) were probably among the first to notice the importance of die thermal insulation to reduce radiation energy loss (W_r). Nowadays, thermal insulation of the die wall with graphite felt is widely employed in FAST/SPS practice. Nevertheless, the influence of insulation design on energy consumption remains hardly explored. Several solutions for decreasing the heat flow from the FAST/SPS tool towards the water-cooled electrodes (W_c) have been proposed. Giuntini et al. (2013) firstly suggested the use of several spacers aiming to reduce the temperature difference between the punch and the electrodes. Then, Giuntini et al. (2015) proposed a new design for punches with drilled cylindrical or machined ring-shaped holes. This design provides a certain reduction in the contact area between the punches and spacers and consequently some decrease in the heat flow towards the electrodes. However, the energy balance has been not discussed. Huang et al. (2017) also decreased the contact area between the punches and electrodes. They proposed a terraced punch design with the part contacting to the spacer having a smaller diameter than the part inserted in the die. Electric current and power input were reduced to 75–85% of their original values for sintering of alumina (1250 °C) and copper (850 °C). However, the reduced punch cross-section leads to overheating of its narrow part and to a decrease in the maximal admissive load. In the present paper, we discuss two solutions to reduce power and energy required for FAST/SPS. Firstly, we consider the use of thermal insulation not only of the cylindrical die surface but also of its top and bottom faces. Secondly, we propose replacing graphite spacers with spacers manufactured from carbon fiber reinforced carbon composite (CFRC). This composite combines acceptable electrical resistivity with low thermal conductivity. This part of the work was performed experimentally. Another part of our work was dedicated to the influence of thermal insulation on temperature homogeneity in FAST/SPS setups and in samples with different conductivity and with different size. The inhomogeneous temperature distribution can lead to poor densification and unacceptable mechanical and other properties in low temperature areas. At the same time, the overheating can result in undesirable grain growth and even in local melting of material. The tolerable value of temperature difference depends on particular material and requirements to product quality. In present paper we considered the acceptable temperature difference of 20 °C. This number can be revised depending on particular requirements an application. Anyway, the temperature gradients should be minimized as much as possible. The aim of our work was to give an inside into the possibilities and limitations of thermal insulation for reduction in temperature inhomogeneity during FAST/SPS sintering. This effect is known but has been scarcely discussed in the literature. Vanmeensel et al. (2007) studied the influence of thermal insulation on temperature distribution in the electrically conductive ZrO_2-TiN (60/40) sample with a diameter of 40 mm using the Finite Element Method (FEM). Insulating the die wall with graphite felt resulted in a decreased temperature difference between the center and edge from 147 °C to 32 °C during dwell at 1500 °C. This result was experimentally confirmed by a lower hardness gradient when the sample was sintered in the insulated die. In some cases, thermal insulation can lead to a certain increase in the radial thermal gradient during sintering of non-conductive materials. Achenani et al. (2017) reported this phenomenon for FAST/SPS sintering of alumina at 1300 °C in a die with a diameter of 40 mm.

Another possibility to reduce thermal gradients involves optimizing the setup design. Vanherck et al. (2015) used FEM to study the influence of tool geometry on temperature distribution in alumina samples with diameters of up to 60 mm. They found that the radial thermal gradient in non-conductive samples can be significantly reduced by optimizing die wall thickness. Giuntini et al. (2015) proposed the special punch design with drillings or ring-shaped holes, which can influence the current path and temperature distribution within samples. The punch geometry optimized by FEM provided acceptable temperature gradients in a Si_3N_4 sample with a diameter of 62 mm. However, drillings and holes led to a decrease in the mechanical strength of punches and to a reduced permissible load.

In the present paper, we systematically examined the influence of conventional and proposed methods of thermal insulation on the temperature distribution in electrically conductive steel (SS316L) samples and non-conductive zirconia (8YSZ) samples. The diameter of the samples was varied from 17 mm to 150 mm. Thus, the influence of sample conductivity and size on temperature distribution was investigated and explained in-depth. This part of the work was done mainly by Finite Element Analysis.

2. Experimental procedures

Two powders with different electrical conductivities were used. The conductive 316L steel powder with particle sizes of 10–36 µm was manufactured by gas atomization at Sandvik Osprey Ltd, UK. Particles of this powder are spherical in shape, and the melting structure can be seen on their surface (Fig. 2a,b). The non-conductive TZ-8Y (8YSZ) zirconia powder with a specific area of 16 ± 3 m^2/g was supplied by Tosoh Corp., Japan. Its nano-sized particles are agglomerated into spheres with diameters of several micrometers (Fig. 2c,d).

Sintering was performed in a HP D 5/2 facility manufactured at FCT Systeme GmbH, Germany. This machine is equipped with a 50 kN uniaxial hydraulic press and with a 37 kW power source. All