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Pulse analysis and electric contact measurements in spark plasma sintering

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ABSTRACT

In order to model the current density distribution and the temperature changes of the tools used during a spark-plasma-sintering (SPS) cycle, the variation of the power delivered by an SPS machine and the graphite-Papyex®-graphite electrical contacts were studied experimentally. The electric device was also characterized; in particular current pulse characteristics and their behavior with time were studied in various conditions of temperature, pulses sequences, materials and total electric power dissipated. It is well known that the performance of an electric contact is dependent on the applied pressure and the temperature. First, by varying the pressure during the SPS cycle the effect of the electric contacts is clearly seen. Secondly, in order to determine the behavior of such contacts experimentally over a pressure range of 10–50 MPa and temperatures of 50–800 °C, a Dœhlert experimental design was used.

1. Introduction

The spark-plasma-sintering (SPS) process is used to manufacture complex shaped solid materials from powder. The interesting part of the process is its ability to sinter in a few minutes while maintaining a fine microstructure in the refractory materials compared to hours with high pressure sintering and several tens of hours with natural sintering. This performance is due to the simultaneous application of high uniaxial pressure and temperature by Joule heating via pulsed current passing through the tools and also the material to be sintered if it conducts electricity. Moreover, the SPS method heats the part being made very quickly compared to the high-pressure method.

The aim of electro-thermal modeling this process was to predict the thermal gradients in the sample and to explain their effects on the final microstructure [1–3]. In the literature, most modeling is performed using the finite elements method (FEM). In FEM modeling authors often consider that all the contacts are perfect. However, recent works, in particular that of Pavia [4], where a rapid infrared camera was used to observe an open die during a whole SPS cycle, revealed that heat transfer at the Punch/Die interface has a predominant effect on the thermal-gradient particularly when insulating materials are sintered [4]. Usually, in SPS tools, a graphite sheet (Papyex® from Mersen Co., Gennevilliers, France) is introduced between the punch, the inner die wall and the sample to ensure easy sample removal, with a low friction coefficient and good thermal contact between the parts.

The contact phenomena and electric and thermal effects are typically dependent not only on the applied pressure but also on the temperature [5–8]. They are explained by the non-ideal surface due to the roughness of the materials involved in the contact [9].

The challenge of our present study was to evaluate these electrical contact resistances (ECR) essential in FEM modeling of the SPS process. There are already some works published on the determination of the electric contact between the parts of the SPS tools. Anselmi-Tamburini et al. [10] determined, by ambient electric resistance measurements, the pressure dependence of the electric contact in alumina and copper samples and concluded that above a uniaxial applied pressure of 50 MPa, it is useless to consider any contact phenomena in the vicinity of the sample. But they do draw our attention to the contact between the punches and the die which

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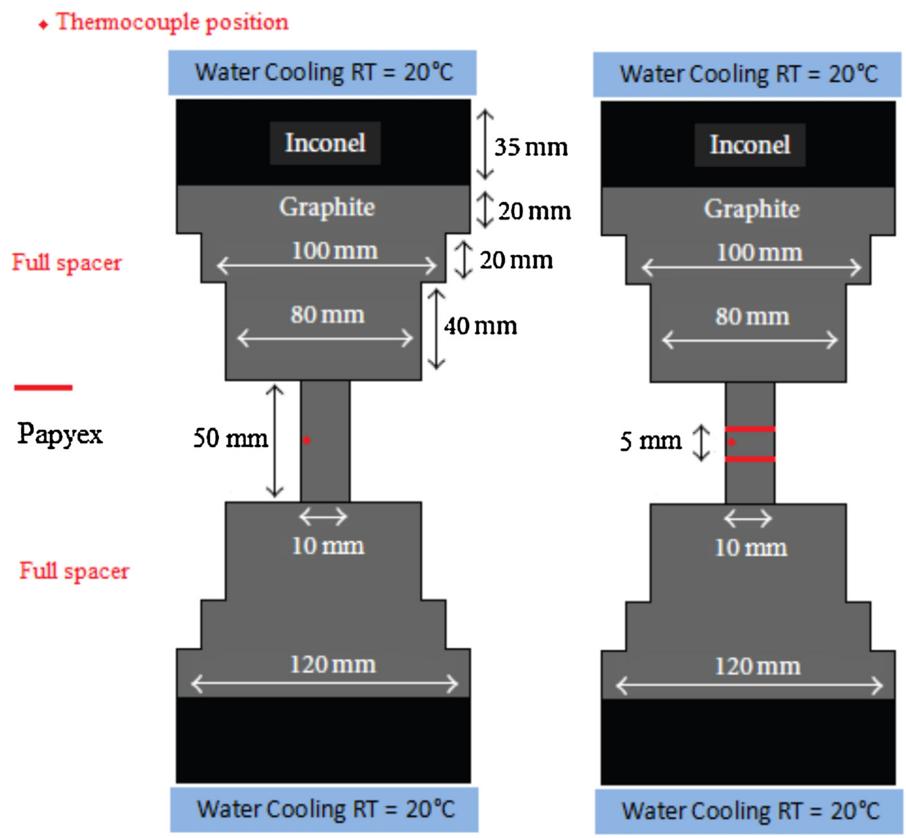

Fig. 1. Representation of the two set-ups: with and without contacts [1, 2]. (For interpretation of the references to color in this figure citation in text, the reader is referred to the web version of this article.)

is assumed to play a stronger role than the global resistance of the tool. Vanmeensel et al. and McWilliam et al. [11,12] made similar studies of the electric contact. They measured the electric resistances of different SPS configurations with and without contacts. The non-contact configuration is used to subtract all resistances except the electrical contact resistance in the contact configuration. This process is explained below in Eq. (IV) used to measure contact resistance.

In the present study, we chose the same strategy asking them to determine the electric resistances of the graphite/Papyex®/graphite contact to follow the behavior of the contact with temperature and pressure. Indeed previous studies mainly considered the pressure dependence of the ECR. Here, a Dœhlert experimental design was used.

2. Experimental

The experiment was carried out on the SPS machine (Dr. Sinter 2080, SPS Syntex Inc, Japan) at the "Plateforme Nationale CNRS de Frittage Flash" located at University Toulouse III-Paul Sabatier.

The first part of this paper is devoted to the analysis of the DC pulsed current given by the device used considering two types of materials to be sintered respectively more conducting (manganese) and insulating (alumina) compared to the graphite used for the tools (Ref. 2333 from Mersen Co., Gennevilliers, France). The molds used have either 8 or 36 mm inner diameters. Sensors were selected for sampling the signals ($u(t)$ across the column and $i(t)$ passing through) with a sufficiently high frequency (up to 10,000 Hz) to describe each pulse correctly. For the instantaneous current a wide band Rogowski coil sensor (Power Electronic Measurements, CWT60) was used. To measure the voltage across the SPS column, the potential was considered uniform over the entire contact surface. From the synchronized voltage and intensity measurements, average and RMS values (U_{ave}, U_{rms}, I_{ave} and I_{rms}) were calculated using a Labview routine (National Instrument software). The signal was also calibrated using an oscilloscope to verify the correspondence between measured and calculated mean values.

The second part of this paper is devoted to highlighting the importance of electric contacts in SPS tools. In particular, the electric contacts graphite/Papyex/graphite present mainly at the inner interfaces of the mold are determined using the principle described by Vanmeensel et al. [11]. Two SPS central punch configurations were studied. One with two graphite/Papyex®/graphite contacts (in red in Fig. 1) and one without contacts. The height of this part of the columns is the same in both configurations. The electric resistance was obtained for each of the two set-ups using the current and voltage values given by the SPS machine.

3. Results and discussions

3.1. Study of machine current

To know what kind of current value is given by the display of the SPS machine, the Rogowski coil was placed below the SPS chamber at the output of the current. The measurements reported in Fig. 2 show that the value of current given by the machine (I_{sps}) roughly corresponds to the average current calculated by the Rogowski coil (I_{ave}).

For thermal effect studies, it would be better to use the rms rather than the average values. The coefficient usually used to convert the average into rms values for theoretical rectified pulsed current, either U or I, is simple and near 1.11 [13]. In any case, as the electrical resistance is the ratio of the voltage to the current intensity, we should use either the average or the rms values.