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Thermal-electrical-mechanical simulation of the nickel densification by Spark Plasma Sintering. Comparison with experiments

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ABSTRACT

Spark Plasma Sintering is a non-conventional process of the powder metallurgy field which uses a high electrical current to rapidly produce fully dense materials. In the present paper, a thermal-electrical-mechanical model developed on ABAQUS Software is proposed to simulate the densification of a nickel disk. A compaction model, studied in [Wolff, C., Mercier, S., Couque, H., Molinari, A., 2012. Modeling of conventional hot compaction and spark plasma sintering based on modified micromechanical models of porous materials. Mechanics of Materials 49 (0), 72–91. URL http://www.sciencedirect.com/science/article/pii/S0167663611002195], has been used to reproduce the densification of the sample. Two SPS experiments have been necessary to identify the parameters of the densification law. In order to evaluate the robustness of the present model, two other SPS experiments have been performed. The whole results of the simulation show a good agreement with the experimental data confirming the validity of the compaction model developed in [Wolff, C., Mercier, S., Couque, H., Molinari, A., 2012. Modeling of conventional hot compaction and spark plasma sintering based on modified micromechanical models of porous materials. Mechanics of Materials 49 (0), 72–91. URL http://www.sciencedirect.com/science/article/pii/S0167663611002195].

1. Introduction

Spark Plasma Sintering (SPS) process has been extensively studied for recent decades to densify powders in a relatively short time period. This process combines a high pulsed electrical current and a mechanical loading to manufacture fully dense materials (ceramics, metals, functionally graded materials,...). The working of a SPS process is the following : the sample is surrounded by a die and punches. All components are placed in a vacuum chamber. The electrical current flows through the conductive part of the device and generates large heating rates mainly due to the Joule effect (Anselmi-Tamburini et al., 2005; Pavia et al., 2013) decreasing therefore the consolidation time of the powder. This short sintering time limits considerably the grain growth and preserves consequently the microstructure. This aspect of moderate modification of the grain size is an essential feature for metals in order to create dense materials with large yield stress (nanomaterials). For instance, without being exhaustive, one can cite the work of Naimi et al. (2013) on a dense nanostructured nickel produced by SPS and the densification of a multimodal nickel (mixture of nano-grains and micro-grains) performed by Farbaniec et al. (2014). The manufacturing of these materials requires a good synthesis of the powder size distribution but also a precise knowledge about how the SPS system works.

In the recent years, numerous investigations have modeled the SPS device to understand the densification process. The first numerical simulations of the process were focusing on the electrical and temperature fields within the system. Yoneya and Ikeshoji (2001) and Matsugi et al. (2004) have observed that heating of an electrical isolator sample is performed via the punches and the die since the electrical current is deviated in the toolings. On the other hand, in the case of a conductor sample, the current flows through the punches and the specimen. Wang et al. (2006) noted that the temperature in the centre of a conductor sample is

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always higher than on its lateral side during a SPS cycle. By contrast, the lateral side of an insulator sample, in contact with the die, is the hottest part. When radiative losses at the outer surface of the die become important, temperature gradients increase in the electrical conductor sample while the temperature field tends to be homogeneous, in its insulator counterpart, (Wang et al., 2003). Such large thermal gradients in the sample are highly detrimental, so (McWilliams et al., 2006) and (Vanmeensel et al., 2007) have observed that these thermal losses can be limited by covering the die by a felt. The same authors studied the influence of the electrical and thermal contact resistances on the thermal and electrical fields in order to obtain a more realistic temperature field. Nevertheless, (Anselmi-Tamburini et al., 2005) suggest that for large applied mechanical loading ($> 50MPa$), contact resistances can be neglected. Some of the current works focused on the temperature and electrical fields but consider that the sample is fully dense. In fact, the temperature and porosity dependence of the material must be taken into account to capture accurately the temperature field in the sample, especially during densification where the porosity evolves. To overcome this difficulty (coupling the porous behaviour with thermal electrical simulations), some investigations prescribed the experimental punch displacement to reproduce the porosity evolution. This porosity evolution is computed from the shrinkage and the mass conservation of the sample, (Wang et al., 2010). Nevertheless, this strategy assumes that the porosity is homogeneous in the sample which is not so realistic. McWilliams et al. (2006) and McWilliams and Zavaliangos (2008) are the first authors to establish a thermal-electrical-mechanical model with ABAQUS software to reproduce the densification kinetics. In these simulations, the behaviour of the porous material is described by a law which was developed initially for free sintering, (without taking into account the mechanical loading effect). Recently, (McWilliams et al., 2015) have improved the previous work by taking into consideration the mechanical loading effect in the densification of the sample . The authors demonstrate that temperature gradients in the sample are larger than those computed from a thermal-electrical model. Since the coupling with the mechanical aspect is necessary, thermal-electrical-mechanical simulations that include a densification model based on continuum mechanics, have been developed recently and are available in the literature (Mondalek et al., 2011; Garcia and Olevsky, 2011; Song et al., 2011; Wolff et al., 2012). The mechanical behaviour of the porous medium subject to high temperature is viscoplastic. The viscoplastic potential to define the creep behaviour depends on stress and porosity. The porosity dependencies can be either derived from micromechanical approaches or empirical expressions. Mondalek et al. (2011) performed a numerical inverse analysis to propose empirical porosity relations based on a combination of SPS tests and creep response of *TiAl* powders. They observe that the proposed parameters are not affected by the heating rate during SPS. For the inverse analysis, parameters are calibrated so that the simulated temperature history on the die coincides with the one measured by the pyrometer. Wolff et al. (2012) firstly propose to modify existing micromechanical models by comparing their predictions with results of hot compaction tests. Thereafter, these models have been applied to simulate the densification of lead during the SPS process. However, no comparisons with experimental results were achieved. Recently, (Maniere et al., 2016) used the model of Skorohod et al. (1993) to determinate the creep response of submicronic alpha-alumina. Material parameters are obtained by comparing finite element calculations and SPS tests. Note that contrary to Mondalek et al. (2011), the creep parameters are observed to vary when the heating rate is changed. So clearly from the literature, this aspect is still debated.

In the present paper, four experimental SPS tests have been performed on nickel for different sintering temperatures to validate the modified micromechanical model suggested in Wolff et al. (2012). In our contribution, a constant applied stress is considered in all experimental SPS tests. The experimental conditions as well as the theory, based on a micromechanical approach for porous material, are presented in the following. The thermal-electrical-mechanical simulations, are precisely described with a precise definition of boundary conditions and material data. A procedure is also introduced by using the sample temperature to identify the material parameters of the micromechanical model. Finally, a comparison between the predictions of the model and experimental results is presented and discussed. A close agreement between numerical results and experimental data is observed.

2. Experimental procedure

2.1. Description of the SPS device

SPS experiments on nickel are performed on a FCT System GmbH HP D 125 technology device (MANAPI, Université de Bourgogne). All tools, located in a vacuum chamber, are made of graphite grade 2333 Mersen. Their corresponding dimensions are given in Figure 1. The sample is made of nickel powder (250g Alfa Aesar powder) with a particle size ranging from 3 to 7 μm and a purity of 99, 9%. The sample is subject to a constant mechanical loading of 70 MPa . In this specific device, the lower punch is moving upward so as to apply the prescribed load while the upper punch is fixed. In addition, a pulsed electrical current is delivered to generate a high heating rate due to Joule effect. The signal pattern is made up of 12 pulses “ on” and 2 pulses “ off”. The duration of each pulse is 3 ms, see Figure 2. Figure 2 shows a sequence of two patterns of the signal really delivered to the apparatus. Nevertheless, only the RMS (root mean square) value of the current will be adopted in the simulation. Indeed, according to Anselmi-Tamburini et al. (2005), the RMS current is an accurate approximation of the pulsed current. To improve the electrical and thermal conductivities of the tooling, graphite foils have been inserted between each part of the device. Furthermore, to measure the sintering temperature, a thermocouple is located at 3 mm from the lateral surface of the sample, named *RTC* in Figure 1.

2.2. Test conditions

In order to validate the robustness of the numerical model, four tests $T-413$, $T-750$, $T-760$ and $T-900$ have been performed under different imposed electrical energy histories to reach precise sintering temperatures of 413°, 750°, 760° and 900°, respectively. All tests are conducted under the same applied stress (70MPa). For instance, in test $T-900$, an initial voltage of 6V (see Figure 3) has been applied to generate an initial heating rate of $750^{\circ}\text{min}^{-1}$, as shown in Figure 4. After a 35s transient regime, the voltage decreases up to 4.3V (stabilized regime) modifying the heating rate towards a stabilized value of $95^{\circ}\text{min}^{-1}$. After 254s, the sintering temperature achieves the selected value of 900° and the cooling step starts.

The thermal conditions of the four experiments are summarized in Table 1. Note that these specific SPS experiments have been selected in this work to test the capability of our model with different initial conditions changing the heating rate. To obtain a good densification of nickel, one may refer to the work of Minier et al. (2010). During sintering, the shrinkage of the sample is measured by a displacement sensor located on the bottom cooled electrode pushing on the lower spacer-punch. At the end of each cycle, the overall porosity of all samples is defined by using the Archimedes' principle. The overall porosity and the final dimensions of each sample are reported in Table 2. For instance, with the electrical