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Powder/die friction in the spark plasma sintering process: Modelling and experimental identification

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ABSTRACT

The powder/die friction phenomenon is known to generate densification inhomogeneities in the spark plasma sintered sample. The measurement of a powder/solid friction coefficient at high temperature is very difficult if not impossible by classical means. Then, an experimental/simulation method of identification of the friction coefficient based on the sample displacement field is introduced. This reveals that the friction of contact type powder/wall is low and about 0.1 and the friction type powder/graphite-foil/wall is close to zero. The relative density inhomogeneities are limited to a maximum difference of 3%.

Spark plasma sintering (SPS) also known as Field-Assisted Sintering is a breakthrough technique in the field of powder consolidation technology [1–3]. This manufacturing approach involves the combination of external pressure and an electric pulsed current applied simultaneously to the powder (Fig.1). The process presents many benefits such as a strong reduction of the sintering temperature and/or time, minimizing granular growth and allowing nanostructured materials [4–7] to be sintered. The sintering of a wide range of materials (ceramics, metals and polymers) takes only a few minutes with the SPS technique compared to several hours or days with other techniques like Hot Pressing (HP) or free sintering.

However, the SPS tools itself remain a black box for which only the surface die temperature and the external applied pressure can be directly controlled. Most of the time, the sintering of complex shaped parts requires a better control and understanding of the thermal and pressure gradients present in the sample during the process. In order to predict the internal thermal gradients, Joule heating models of the SPS process are used. Several authors like [8–14] have emphasized the strong potential effect of the electric and thermal contacts present in every inner interface of the device. The die/sample temperature difference is generally quite high, ranging from some tens to hundreds Kelvin mainly due to contact resistances. The presence of hot spots at the edges of samples was experimentally reported in the PhD work of A. Pavia [15] and can be the object of strong local inhomogeneities in microstructure. In die configurations with non-common geometry, the thermal gradient can be exacerbated. Using such configurations, Functionally Graded Materials (FGM) [16–18] are elaborated with microstructural gradients.

Apart from the thermal gradients, microstructural differences may appear because of the presence of pressure inhomogeneities. A lot of experimental and theoretical studies [19–22] have shown the importance of height differences in the parts that cause high porosity gradients in complex shaped samples. To solve this problem of porosity gradients, sintering simulations are then performed. The sintering simulations can be used to predict the densification field at every stage of a sintering cycle. The main interest of these simulations is to verify the validity of the tooling configurations for the sintering of complex shaped parts without having recourse to expensive experimental “trial and error” adjustment testing. Powder compaction modelling in processes like SPS or HP is most often built on porous creep behaviour laws. The Abouaf [23], Olevsky [24,25] or Camclay [26] sintering models are the most widely cited in the literature. In order to describe either the thermal or mechanical aspect of the SPS process, fully coupled electro-thermal–mechanical–microstructural simulations can then be used to model the relative density/thermal/grain size gradients in a more relevant way.

Complex shapes are not the only source of pressure inhomogeneities. Even in simple shapes such as a cylindrical sample, pressure inhomogeneities can occur because of the powder/mould friction that creates a shear stress on the sliding wall. This effect and the related microstructural changes were studied by P. Mondalek [27]. In classical models, the friction is determined using the Coulomb law (1)

$$\tau = \mu \tau_n \tag{1}$$

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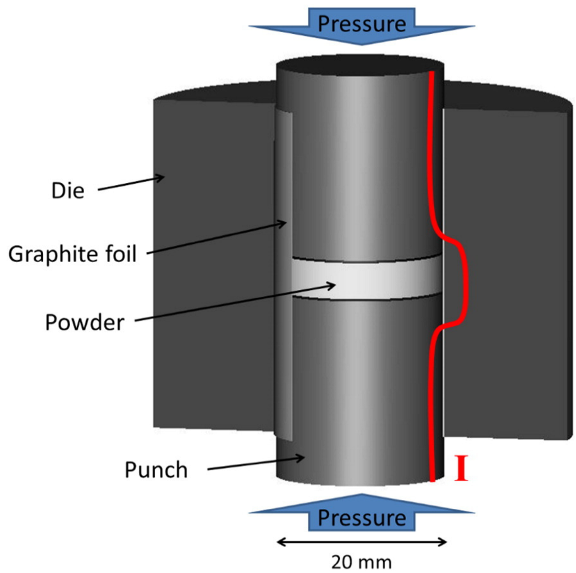

Fig. 1. Scheme of the spark plasma sintering process (I is the pulsed current).

where the tangential stress τ is a defined function of the friction coefficient μ (in displacement regime) and the normal pressure τ_n.

For viscoplastic materials, a Norton–Hoff friction law (2) can also be used.

$$\tau = -\mu K |\boldsymbol{V}_g|^{p-1} \boldsymbol{V}_g. \tag{2}$$

This Norton–Hoff creep (3) based friction law is a function of the material consistency K (3), the sliding relative velocity $\boldsymbol{V}_g$ and a constant p that can be assimilated to the power law creep strain rate sensitivity m.

$$\sigma_{\text{eq}} = K\dot{\varepsilon}_{\text{eq}}^{m}. \tag{3}$$

The viscous friction law is particularly useful to start gradually with the increase of the relative velocity. Indeed, in displacement regime, the Coulomb law starts directly by the nominal shear stress, and this may create divergences leading to computing problems. In her work, P. Mondalek [27] introduced friction into the Norton–Green compaction model by a viscoplastic approach. Two implementation routes were developed:

i) by a lateral boundary condition on the sample/wall
ii) by an equivalent thin layer at the lateral powder/die interface.

The form of the tangential stress (4) is a function of c, a term that depends on the porosity of the material in the Norton–Green model.

$$\tau = -\mu K c^{-\frac{m+1}{2}} |\boldsymbol{V}_g|^{m-1} \boldsymbol{V}_g \tag{4}$$

The tangential stress generated by the boundary layer (5) has a form similar to that of Eq. (4) and depends on the layer thickness e.

$$\tau = -\frac{1}{e^m} K c^{-\frac{m+1}{2}} |\boldsymbol{V}_g|^{m-1} \boldsymbol{V}_g \tag{5}$$

The friction coefficient is then the inverse of the thickness to the power m (6).

$$\mu = \frac{1}{e^m} \tag{6}$$

It is then possible to vary the layer thickness or the consistency to create an equivalent friction coefficient. The two ways provided comparable results and point out how the powder/die friction influences the relative density gradient. To summarize, the relative density is higher at the points of high relative sliding velocity. On the other hand the relative density is lower at half the height of the pellet, where there is no powder/die sliding due to compaction displacement.

The determination of a powder/solid friction coefficient at high temperature is very difficult and cannot be performed by classical solid/solid techniques. An interesting method was used by P. Mondalek in her PhD work. The friction coefficient was identified using the observable effect of friction on the relative density field. The simulated and experimental relative density fields obtained by SPS experiments for a TiAl powder are compared for various values of the friction coefficient subsequently introduced into the simulation. Then, the value giving the best simulated/experimental concordance is considered as the friction coefficient. Apart from this study, the modelling of the powder/die friction in the SPS or similar processes is sparse. We can cite the work of J.R. Cho et al. [28] and the work of Ashoka G.K. Jinka et al. [29] on an Abouaf viscoplastic friction model and the work of K.V. Ranjit et al. [26] with the Camclay model.

The aim of the present work is to identify the powder/die friction properties and its effect on the SPS process with various types of contact. The experiments were performed on the SPS machine (Dr. Sinter 2080, SPS Syntex Inc., Japan) of the "Plateforme Nationale CNRS de Frittage Flash" located at University Toulouse III-Paul Sabatier. The SPS column (spacers, punches and mould) is made of graphite (Mersen ref. [23]2333). The geometrical configuration of the punches, mould and powder is reported Fig. 1 with a 20 mm punch diameter. The powder sintered is a fine-grained alumina of 0.14 µm average grain size (alumina 99.99%, reference TM-DAR, Taimei Chemicals Co. Ltd). To ensure good contact between punches and sample/die and for easy removal of the sample once densified, three types of interfacial materials are generally used: graphite foil (papyex from Mersen), a sprayed layer of graphite or boron nitride powders. Thus, these three powder/die contacts were studied in the present work (Fig. 2). To simulate the densification of the power considered, we used the Olevsky sintering model. Then, the equivalent strain rate $\dot{\varepsilon}_{eq}$ is defined by:

$$\dot{\varepsilon}_{eq} = \frac{1}{\sqrt{1-\theta}} \sqrt{\varphi\dot{\gamma}^2 + \psi\dot{e}^2} \tag{7}$$

where, θ, $\dot{e}$ and $\dot{\gamma}$ correspond to the porosity, the shrinkage rate and shape change rates respectively. The latter are given by:

$$\dot{e} = \dot{\varepsilon}_x + \dot{\varepsilon}_y + \dot{\varepsilon}_z \tag{8}$$

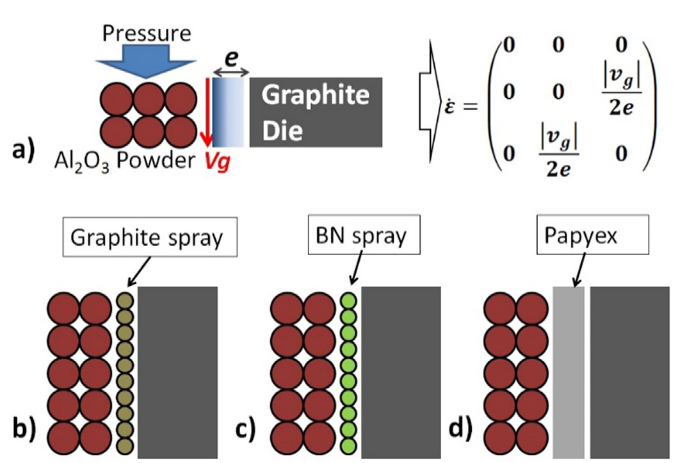

Fig. 2. a) Powder/die interface and the three configurations studied: b) alumina/graphite spray/die c) alumina/boron nitride/die d) alumina/carbon sheet/die.