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Porosity dependence of powder compaction constitutive parameters: Determination based on spark plasma sintering tests

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ABSTRACT

The modeling of powder compaction process, such as spark plasma sintering (SPS), requires the determination of the visco-plastic deformation behavior of the particle material including the viscosity moduli. The establishment of these parameters usually entails a long and difficult experimental campaign which in particular involves several hot isostatic pressing tests. A more straightforward method based on the coupled sinter-forging and die compaction tests, which can be easily carried out in a regular SPS device, is presented. Compared to classical creep mechanism studies, this comprehensive experimental approach can reveal the in situ porous structure morphology influence on the sintering process.

The simulation of the powder compaction in advanced sintering techniques, such as spark plasma sintering, is a helpful research tool enabling the prediction and optimization of: the densification nonuniformity [1], the tooling resistance to the stress solicitation [2], and the elaboration of complex shapes [3]. For the SPS technology, these mechanical simulations are often coupled with the Joule heating modeling [4–10] in a multiphysics approach rendering a comprehensive prediction of this process' electro-thermal-mechanical phenomena [11–13]. As described by numerous authors [5,14–17], the main challenge related to the Joule heating modeling is to identify the non-ideal electric and thermal contacts in the SPS tooling-specimen setup as the dominant parameters controlling the temperature field distribution. Concerning the aspects of the mechanical modeling, the great challenge is to identify all the model constitutive parameters. The powder compaction model for both pressure and pressureless sintering techniques can be described by the general continuum theory of sintering [18]. For pressure assisted techniques such as SPS this approach can be reduced to the description of a visco-plastic porous body (a continuum made of a dense phase and porosity) behavior [3,19,20]. The dense phase nonlinear viscous behavior is often modeled via a power law creep. The stress/strain behavior of the porous medium is also described by the porosity-dependent shear and bulk moduli. The experimental determination of these moduli is usually rather cumbersome, therefore, as a rule, the values of these parameters are approximated theoretically to the detriment of the overall modeling accuracy [18].

Numerous theoretical derivations of the shear and bulk moduli consider linear viscous [21,22] or power law creep [23–25] materials with an idealized porous body “skeleton”. As reported by Wolff et al. [26] these theoretical moduli are often characterized by a good functional trend, but the difference of a considerable magnitude is observed between the theoretical and experimentally determined moduli values [19,27–29]. The consequence of this discrepancy is then a possible theoretical overestimation of the equivalent creep parameters. Failure in the identification of the creep mechanism has been reported [30] when using traditional isothermal linear regression methods [3,31–33]. The isothermal regime is very sensible and the somewhat inaccurate estimation of the shear and bulk moduli can generate a significant error in the sintering mechanism evaluation [30]. A more precise experimental determination of the shear and bulk moduli is therefore of high interest for the sintering modeling. However, the traditional methods of the determination of these moduli are very time consuming and require, inter alia, several instrumented hot isostatic pressing (HIP) tests [19,29], a rather expensive instrumentation to setup. An alternative solution using coupled sinter-forging and die compaction tests is described in this paper. This combination, inspired from refs. [27,28], can be easily adapted to a regular SPS machine such as the one utilized in the studies [34,35] of the consolidation of Ti-6Al-4V and TiAl

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powders, respectively. It should be noted that for small grain size ceramic powders the in situ creep tests [34–40] are more accurate since the SPS processing is able to better preserve fine microstructures [41–45].

To apply this method, the continuum theory of sintering needs to be reduced to its general analytical form for both the sinter-forging and die compaction tests. Considering the minimum operational pressure of 40 MPa and the 5 μm average particle size, the sintering stress can be neglected and the general formulation of the continuum theory of sintering gives the stress tensor σ the following expression [18]:

$$\underset{=}{\sigma} = \frac{\sigma_{eq}}{\dot{\varepsilon}_{eq}}\left(\varphi\underset{=}{\dot{\varepsilon}} + \left(\psi - \frac{1}{3}\varphi\right)\dot{e}\mathbb{1}\right) \tag{1}$$

where $\dot{\varepsilon}$ is the strain rate tensor, $\mathbb{1}$ is the identity tensor, φ and ψ the shear and bulk moduli to be determined, $\dot{\varepsilon}_{eq}$ and σ_{eq} the equivalent strain rate and stress defined by [46]:

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

$$\sigma_{eq} = \frac{\sqrt{\frac{\tau^2}{\varphi} + \frac{P^2}{\psi}}}{\sqrt{1-\theta}} \tag{3}$$

with θ being the porosity and the strain rate and stress tensor invariants given by:

$$\dot{\gamma} = \sqrt{2\left(\dot{\varepsilon}_{xy}^2 + \dot{\varepsilon}_{xz}^2 + \dot{\varepsilon}_{yz}^2\right) + \frac{2}{3}\left(\dot{\varepsilon}_x^2 + \dot{\varepsilon}_y^2 + \dot{\varepsilon}_z^2\right) - \frac{2}{3}\left(\dot{\varepsilon}_x\dot{\varepsilon}_y + \dot{\varepsilon}_x\dot{\varepsilon}_z + \dot{\varepsilon}_y\dot{\varepsilon}_z\right)} \tag{4}$$

$$\tau = \sqrt{(\sigma_x - \sigma_y)^2 + (\sigma_y - \sigma_z)^2 + (\sigma_z - \sigma_x)^2 + 6\left(\sigma_{xy}^2 + \sigma_{yz}^2 + \sigma_{xz}^2\right)}/\sqrt{3} \tag{5}$$

$$\dot{e} = \dot{\varepsilon}_x + \dot{\varepsilon}_y + \dot{\varepsilon}_z \text{ and } P = (\sigma_x + \sigma_y + \sigma_z)/3 = I_1/3. \tag{6}$$

The porosity is determined locally by the mass conservation equation:

$$\frac{\dot{\theta}}{1-\theta} = \dot{\varepsilon}_x + \dot{\varepsilon}_y + \dot{\varepsilon}_z. \tag{7}$$

The equivalent strain rate and stress of the dense phase are related to each other via a creep power law:

$$\dot{\varepsilon}_{eq} = A\sigma_{eq}^n = A_0 \exp\left(\frac{-Q}{RT}\right)\sigma_{eq}^n \tag{8}$$

where for pure nickel [47,48]: $A_0 = 2.06E-8\ MPa^{-n}s^{-1}$, $Q = 171.1$ kJ mol^{-1} and $n = 7$.

The die compaction case (such as in traditional SPS configuration) is characterized by a unique displacement along z-axis (assuming compaction direction along z-axis) which gives the external strain rate tensor the following analytical approximation:

$$\underset{=}{\dot{\varepsilon}} \equiv \begin{pmatrix} 0 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & \dot{\varepsilon}_z \end{pmatrix}. \tag{9}$$

Replacing (9) in (2,4,6), one obtains the simplifications:

$$\dot{e} = \dot{\varepsilon}_z; \dot{\gamma} = |\dot{\varepsilon}_z|\sqrt{\frac{2}{3}}; W = |\dot{\varepsilon}_z|\sqrt{\frac{\psi + \frac{2}{3}\varphi}{1-\theta}} \tag{10}$$

Which, using (1) and (8), renders the general analytical form of the die compaction loading mode:

$$|\dot{\varepsilon}_z| = A\left(\psi + \frac{2}{3}\varphi\right)^{\frac{-n-1}{2}}(1-\theta)^{\frac{1-n}{2}}|\sigma_z|^n. \tag{11}$$

The sinter-forging case is characterized by a unique loading along z-axis (assuming loading direction along z-axis) which gives the external stress tensor the following analytical approximation:

$$\underset{=}{\sigma} \equiv \begin{pmatrix} 0 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & \sigma_z \end{pmatrix}. \tag{12}$$

To determine the sinter-forging constitutive equation we need to determine first the strain rate tensor expression that depends on the stress tensor components.

Starting from (1) and considering the relationship $P\dot{\varepsilon}_{eq} = \sigma_{eq}\psi tr(\dot{\varepsilon})$ for the stress and strain rate tensor invariants [46] one can determine:

$$\underset{=}{\dot{\varepsilon}} = \frac{\dot{\varepsilon}_{eq}}{\sigma_{eq}}\left(\frac{\underset{=}{\sigma}}{\varphi - \left(\frac{1}{3\varphi} + \frac{1}{9\psi}\right)I_1\mathbb{1}}\right). \tag{13}$$

Then, considering the stress tensor deviator expression $s = \sigma - I_1\mathbb{1}/3$, we finally obtain the general form:

$$\underset{=}{\dot{\varepsilon}} = \frac{\dot{\varepsilon}_{eq}}{\sigma_{eq}}\left(\frac{\underset{=}{s}}{\varphi + \frac{I_1}{9\psi}\mathbb{1}}\right). \tag{14}$$

If we consider the simplification of (12) in (3,5,6) we obtain for sinter-forging:

$$\tau = \sqrt{\frac{2}{3}}|\sigma_z|; P = -\frac{|\sigma_z|}{3}; \sigma_{eq} = |\sigma_z|\frac{\sqrt{\frac{2}{3\varphi} + \frac{1}{9\psi}}}{\sqrt{1-\theta}}; s_z = -\frac{2}{3}|\sigma_z|. \tag{15}$$

Using (14), (8) and (15), the final constitutive equation for sinter-forging is then:

$$|\dot{\varepsilon}_z| = A(1-\theta)^{\frac{1-n}{2}}\left(\frac{2}{3\varphi} + \frac{1}{9\psi}\right)^{\frac{n+1}{2}}|\sigma_z|^n. \tag{16}$$

Combining (11) and (16) it is possible to experimentally determine parameters φ and ψ at fixed porosity and temperature values. This can be achieved by solving the system of the two equations below where the first member is unknown (φ and ψ), and the second member can be accessed experimentally by sinter-forging and die compaction tests ($\theta|\dot{\varepsilon}_z| \cdot |\sigma_z|$ are experimentally determined; A, n are known by creep tests.)

$$\begin{cases} \frac{2}{3\varphi} + \frac{1}{9\psi} = \left(|\dot{\varepsilon}_z|A^{-1}(1-\theta)^{\frac{n-1}{2}}|\sigma_z|^{-n}\right)^{\frac{2}{n+1}} \text{ sinter-forging} \\ \psi + \frac{2}{3}\varphi = \left(|\dot{\varepsilon}_z|^{-\frac{1}{n}}(1-\theta)^{\frac{1-n}{2n}}A^{\frac{1}{n}}|\sigma_z|\right)^{\frac{2n}{n+1}} \text{ Die compaction} \end{cases} \tag{17}$$

Considering the sinter-forging case, it is obvious that the behavior of a loose powder specimen at a constant applied stress $|\sigma_z|$ can provoke the specimen's collapse and, in turn, a very high strain rate $|\dot{\varepsilon}_z|$. Consequently, in Eq. (16), the summation $\frac{2}{3\varphi} + \frac{1}{9\psi}$ tends to infinity, and both φ and ψ tend to zero at a critical porosity θ_c close to the porosity of a loose powder. Another fact is that the equivalent stress (3) tends to the von Mises stress expression at full specimen's density when φ and