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Identification of Porous Materials Rheological Coefficient Using Experimental Determination of the Radial and Longitudinal Strain Rate Ratio

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Abstract. Traditional approach of identification of the f1 and f2 function of the Green plasticity criterion [1] for porous materials is based on the two experiments: isostatic pressing of samples in the interrupted cycles (determining density as a function of pressure providing the f2 values) and upsetting providing an equation for determining the f1 values responsible for the shear deformations. Treatment of HIP dilatometer data allows to get an independent f1 evaluation which shows that upsetting is not representative of stress state met during powder HIP densification. In the second part, a new approach is suggested allowing to determine these two functions using only the deviations from the isotropic shrinkage obtained in the interrupted cycles. This anisotropy is characterized by the ψ coefficient ratio between the radial and the axial strain rate.

Introduction

Traditional approach of identification of the f1 and f2 function of the Green plasticity criterion [1] for porous materials is based on the two experiments: isostatic pressing of samples in the interrupted cycles determining density as a function of pressure providing the f2 values and upsetting providing equations for determining the f1 values responsible for the shear deformations.

In [2], HIP dilatometer was presented (device and treatment of results). It was established an equation giving the energy equilibrium between effect of HIP pressure acting on external surface of capsule versus energy needed for strain of capsule and powder (densification and shear stress strain).

Modeling

Mechanical analysis

The basic Green equation is:

$$Y^2 = \frac{S_1^2}{9*f_2^2} + \frac{3}{2} * \frac{S_2^2}{f_1^2} \quad (1)$$

where Y is the flow stress of fully dense powder material and S1 and S2 are the two invariants of stress tensor with $S_1 = \frac{\sigma_r+\sigma_\varphi+\sigma_z}{3}$ and $S_2 = \sqrt{\frac{1}{3} * ((\sigma_r - \sigma_\varphi)^2 + (\sigma_z - \sigma_\varphi)^2 + (\sigma_z - \sigma_r)^2)}$.

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a. *HIP Dilatometer Experiments*

In HIP dilatometer experiments the equation for the energy equilibrium gives the following equation for the HIP pressure:

$$P=\frac{1}{(\varepsilon+2A)}\left\{\frac{Y}{3}\sqrt{\left(9f_2^2-2f_1^2\right)(2A+\varepsilon)^2+6f_1^2\left(2A^2+\varepsilon^2\right)}+ +2\frac{h}{R}T\sqrt{\frac{1}{3}}\sqrt{3\varepsilon^2+(2A+\varepsilon)^2}\right\} \quad (2)$$

Y is the flow stress of full dense powder material, T is the flow stress of capsule material, P the HIP pressure on the capsule, h is the capsule thickness, R is the radius of capsule, f1 and f2 are coefficients defined in Green equation, ε is the radial strain rate and A the axial strain rate.

It is interesting to introduce coefficient ψ such as $\psi=\frac{\left(\frac{\Delta b}{b}\right)}{\left(\frac{\Delta a}{a}\right)}$ where Δb is the relative momentary axial shrinkage, b- the current height of powder layer, Δa - is the relative (momentary) radial shrinkage of powder and a - the current radius of powder. Equation 2 can be re-written as:

$$P=\left\{\frac{Y}{3}\sqrt{\left(9f_2^2-2f_1^2\right)+6f_1^2\frac{\left(2+\psi^2\right)}{(\psi+2)^2}}+ +2\frac{h}{R}T\sqrt{\frac{1}{3}}\sqrt{3\frac{\psi^2}{(\psi+2)^2}+1}\right\}. \quad (3)$$

$$\frac{dP}{d\psi}=Y\frac{-4f_1^2\frac{(1-\psi)}{(\psi+2)^3}}{\sqrt{\left(9f_2^2-2f_1^2\right)+6f_1^2\frac{\left(2+\psi^2\right)}{(\psi+2)^2}}}+\frac{h}{R}T\sqrt{\frac{1}{3}}\frac{12\frac{\psi}{(\psi+2)^3}}{\sqrt{3\frac{\psi^2}{(\psi+2)^2}+1}}. \quad (4)$$

From the condition $\frac{dP}{d\psi}=0$, we get:

$$\frac{h}{R}T\sqrt{3}\frac{\psi}{\sqrt{3\frac{\psi^2}{(\psi+2)^2}+1}}=Y\frac{f_1^2(1-\psi)}{\sqrt{\left(9f_2^2-2f_1^2\right)+6f_1^2\frac{\left(2+\psi^2\right)}{(\psi+2)^2}}}. \quad (5)$$

Combining with equation (3), we get:

$$\frac{Y}{3}\sqrt{\left(9f_2^2-2f_1^2\right)+6f_1^2\frac{\left(2+\psi^2\right)}{(\psi+2)^2}}+2\frac{h}{R}T\sqrt{\frac{1}{3}}\sqrt{3\frac{\psi^2}{(\psi+2)^2}+1}=P \quad (6)$$

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Then $$\sqrt{\left(9f_2^2-2f_1^2\right)+6f_1^2\frac{\left(2+\psi^2\right)}{\left(\psi+2\right)^2}}=\frac{3\left[P-2\frac{h}{R}T\sqrt{\frac{1}{3}}\sqrt{3\frac{\psi^2}{\left(\psi+2\right)^2}+1}\right]}{Y} \quad (7)$$

Combining with equation (5) we get:

$$\frac{h}{R}T\sqrt{3}\frac{\psi}{\sqrt{3\frac{\psi^2}{\left(\psi+2\right)^2}+1}}=Y^2\frac{f_1^2\left(1-\psi\right)}{3\left[P-2\frac{h}{R}T\sqrt{\frac{1}{3}}\sqrt{3\frac{\psi^2}{\left(\psi+2\right)^2}+1}\right]} \quad (8)$$

Then:

$$f_1^2=\frac{h}{R}\frac{T}{Y^2}\left[P-2\frac{h}{R}T\sqrt{\frac{1}{3}}\sqrt{3\frac{\psi^2}{\left(\psi+2\right)^2}+1}\right]3\sqrt{3}\frac{\psi}{\left(1-\psi\right)\sqrt{3\frac{\psi^2}{\left(\psi+2\right)^2}+1}} \quad (9)$$

We can get f2 using equations (6) and (9):

$$f_2^2=\frac{1}{Y^2}\left[P-2\frac{h}{R}T\sqrt{\frac{1}{3}}\sqrt{3\frac{\psi^2}{\left(\psi+2\right)^2}+1}\right]^2-\frac{4}{9}f_1^2\frac{\left(1-\psi\right)^2}{\left(\psi+2\right)^2}. \quad (10)$$

b. *Evaluation of ψ coefficient*

Coefficient ψ was evaluated using HIP dilatometer results for TA6-4 powder HIPed in 304L capsule [2]. Theoretically ψ is only a function of powder relative density with following particularities:

1. For initial density, f1=0 then ψ=0
2. For full density, f1=1 then ψ has a given value depending on used rheology (0 for a purely viscous law and around 0.2 for an elasto-viscous law).

Figure 1 gives the values of ψ function of ρ for the bar trial based on the dilatometer measurements for:

1. a very small step (20s)
2. a 2% density step

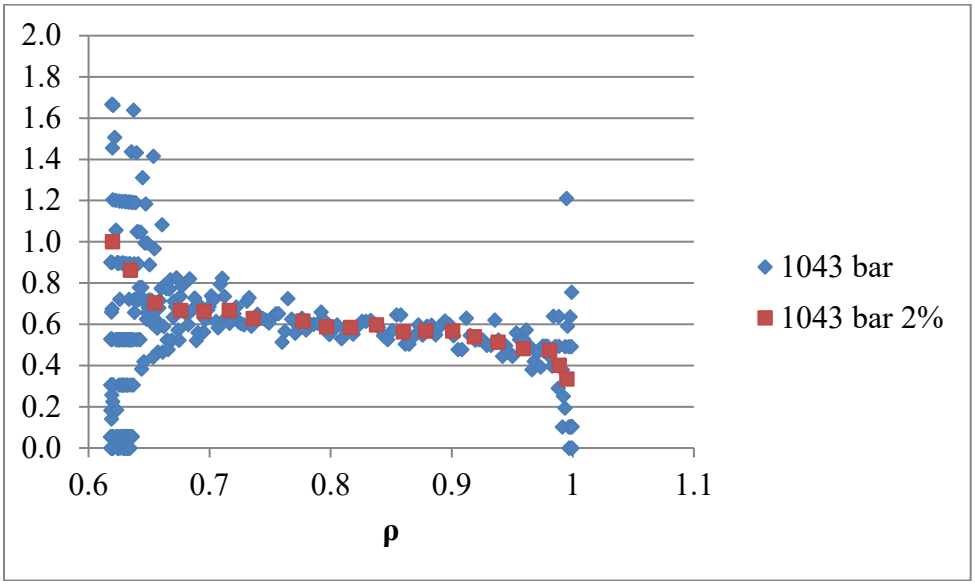

Figure 1: ψ function of ρ for the 1043 bar trial.

For medium density, evolution of ψ is rather even.

At low density, tendency is not compatible with what as expected. Even if a strong scatter is generated by low values of densification, it is necessary to involve the fact that used capsule was not really what generates instabilities of radial shrinkage due to the deformation of lids. Data are not valuable at low density. After some densification (around 0.7-0.75) flow pattern becomes stable, and data become valuable.

At high density, even if a strong scatter is generated by low densification rate, tendency seems compatible with expected evolution.

c. Identification of f1 and f2

Identification requires use of equations 8 and 9. However it is necessary to fix used rheologies for full dense powder material and capsule material. In fact, f1 and f2 cannot be identified by themselves, only a set of values (f1, f2, Y and T) can be identified. For example, in Fig. 2, are given flow stress of full dense powder material function of density with a viscous behavior [3] and for an elastic-viscous behavior [4]. Discrepancy is small but not negligible.

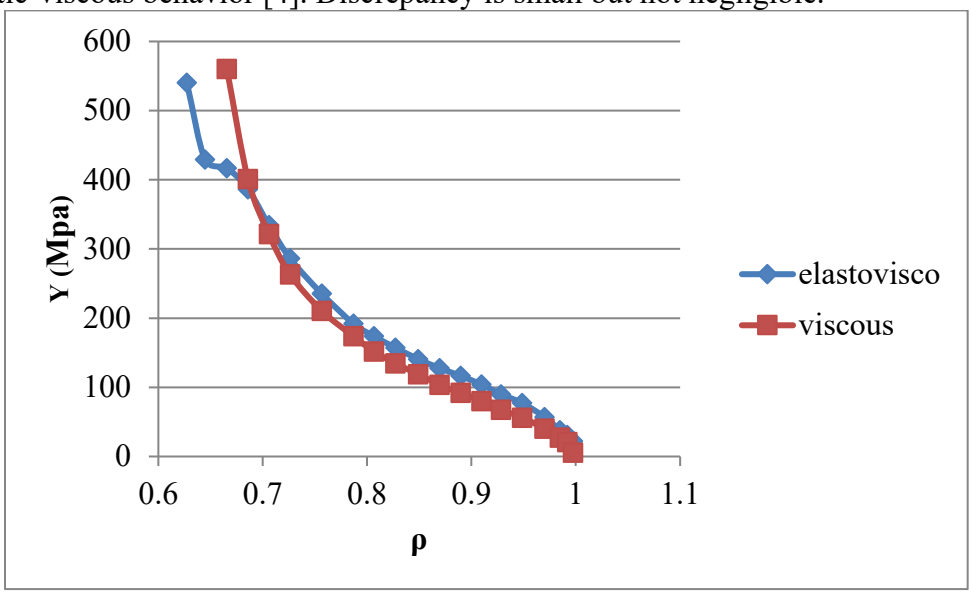

Figure 2: Flow stress of TA6-4 function of density along densification.

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For capsule material rheology, an elastic-viscous- plastic law is used [5].
Figure 3 shows estimation of coefficient f1 with an elastic-viscous rheology for powder material (TA6-4).

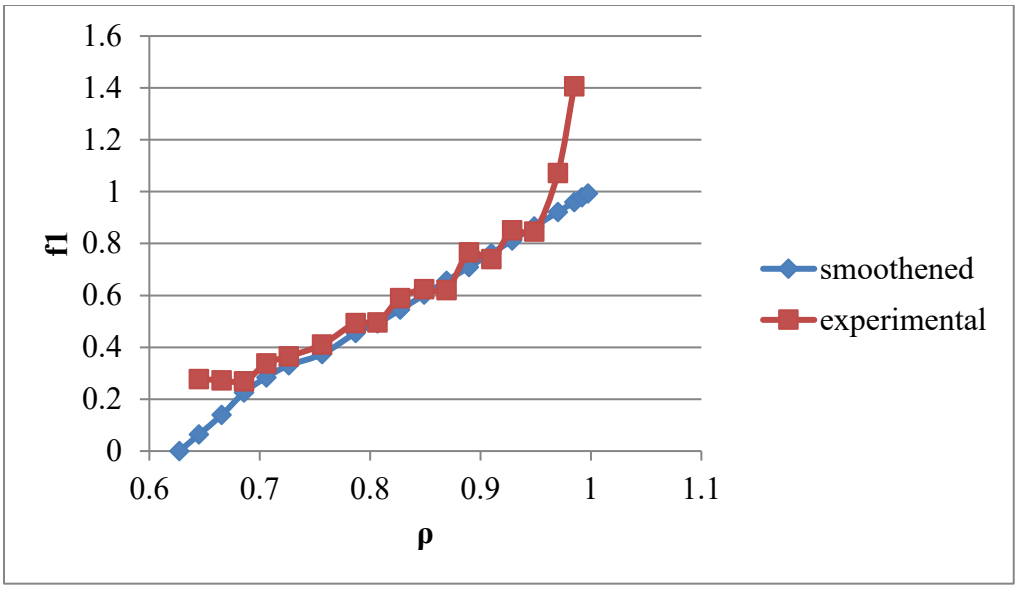

Figure 3: f1 function of density (experimental and after smoothening)

For medium density (between 0.7 and 0.95) variation is even and almost linear. At low density the measured value of f1 does not tend toward 0 and at high density does not tend toward 1.

At low density, it has been said that experimental data are not valuable. At high density, uncertainty due to scatter and very high sensitivity of f1 to ψ value explain this anomaly. An extrapolation of central part of the curve toward f1=0 for filling density and f1=1 for full density has been proposed. From proposed f1=f(ρ) curve, it is easy to calculate a new ψ=g(ρ) curve (see figure 4). The agreement with experimental points is satisfactory. It has been added ψ function of density with a purely viscous law for TA6-4.

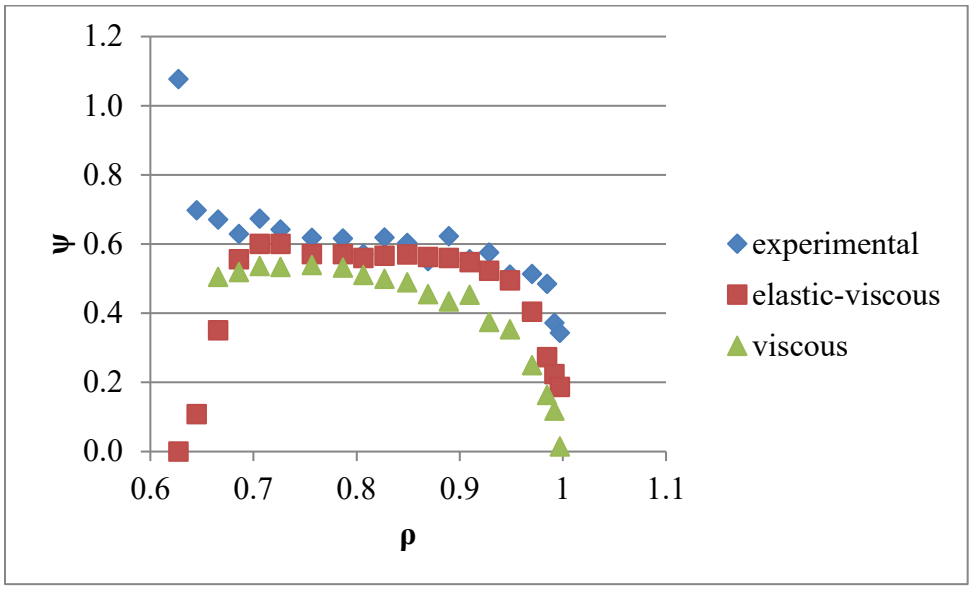

Figure 4: ψ function of density calculated using smoothened f1=f(ρ) curve for two rheologies.

Figure 5 gives f2 values as a function of density. It can be noticed that these values are almost of those obtained in isotropic conditions. It means that $f_2 = \frac{P_{int}}{Y}$ where P_{int} is the pressure in the

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capsule with correction of pressure drop due to the wall of capsule. This result is very good, because it means that identification of f2 with the method of interrupted HIP cycle in a cylindrical capsule is valuable.

Figure 6 gives f2 values as a function of density for an elastic-viscous rheology and for a viscous law. The discrepancy expresses flow stress discrepancy.

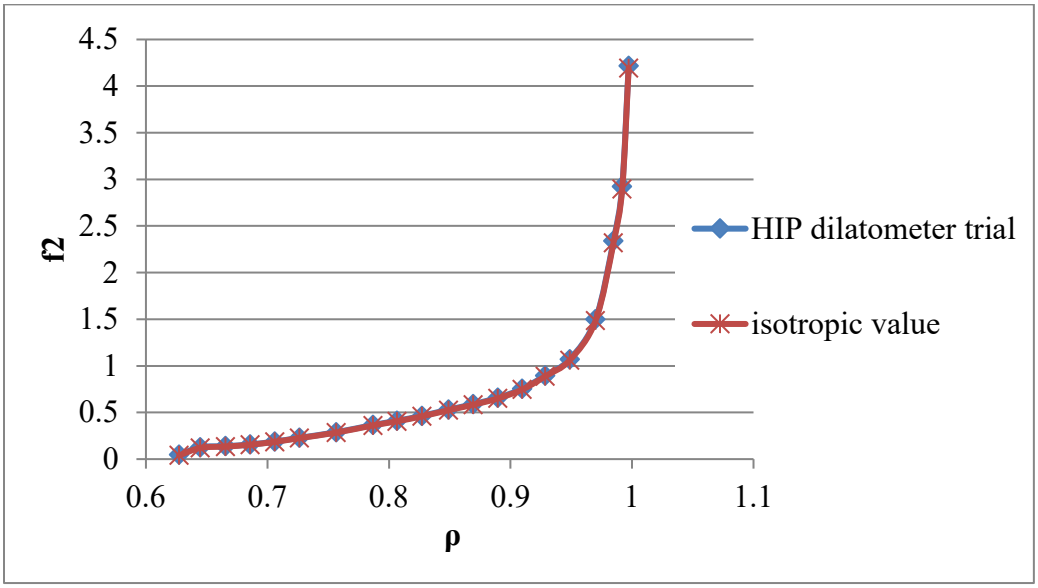

Figure 5: f2 function of density (HIP dilatometer data treatment and isotropic calculation).

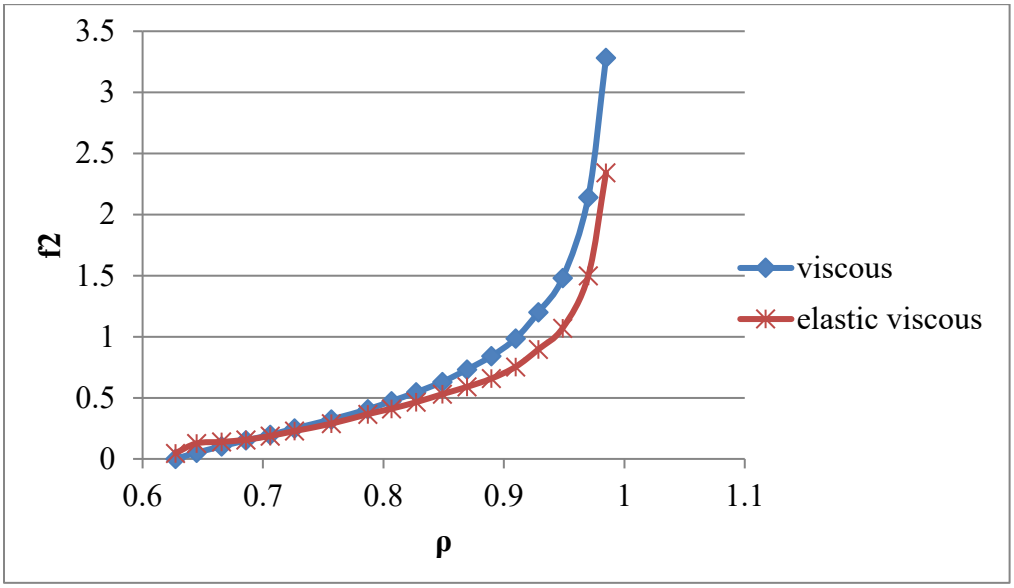

Figure 6: f2 function of density for viscous and elastic viscous law for TA6-4

d. Comparison with other identification processes

It is interesting to compare results of identification by exploitation of HIP dilatometer data with other existing techniques. Two are available:

1. Upsetting of partially dense blanks: for this process $f_1^2 = \frac{\sigma_z^2}{Y^2 - \frac{\sigma_z^2}{9*f_2^2}}$ where σ_z is the pressure applied to the sample with a controlled strain rate. Two set of data have been used [3] and [6]

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2. Unidirectional pressing of powder in a rigid die at a controlled strain rate with a measure of axial and axial stress: for this process [5], it was observed a ratio K between radial and axial stresses depending only on density.

$$f_1^2 = \frac{2*(1-K)^2*\sigma_z^2}{\sigma_{\bar{e}q}^2 - \frac{1}{9*f_2^2}*(1-2*K)^2*\sigma_z^2} \text{ with } K = \frac{\sigma_r}{\sigma_z} = 1 - 1.6 * (1-\rho)^{0.45} \text{ for } \rho > 0.75.$$

As said previously it is important to remember that f1 is not an independent parameter. Actually, it is an established set of data (full dense powder rheology, capsule material rheology, f1 and f2). It is necessary to homogenize data and sometimes it is not trivial.

For upsetting we have used [3] at 800°C and [4] at 925. For unidirectional pressing, we have used [3] at 800°C.

For TA6-4, we have used elastic-viscous rheology of [4]

For 304L (capsule material) we have used elastic-viscous-plastic rheology of CEA [5]

For f2, we have used Fig. 5 relationship.

Figure 7 gives f1 curves as a function of density for treated data.

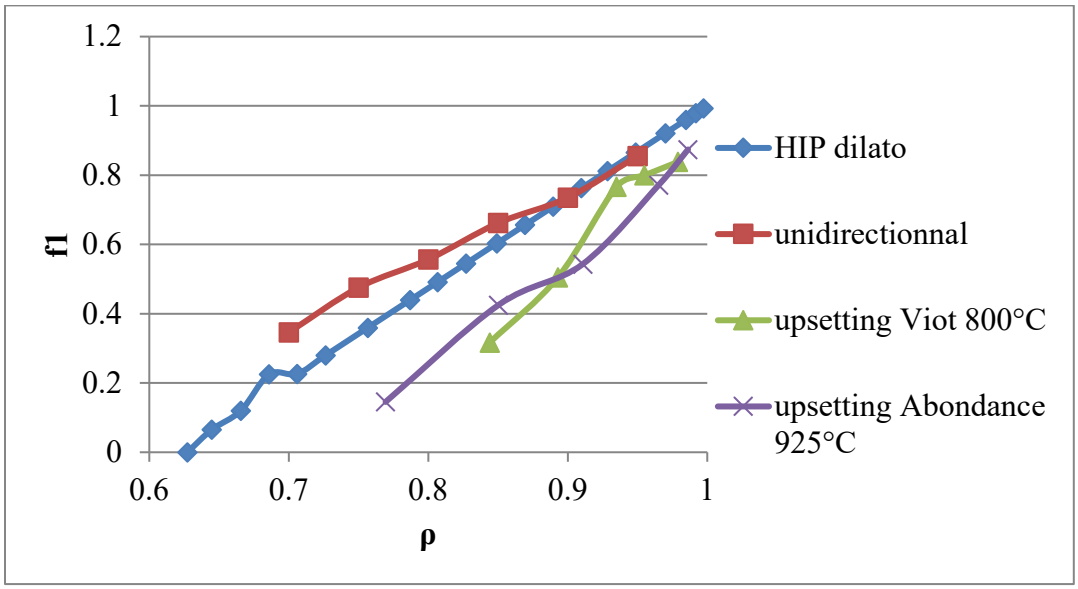

Figure 7: f1 function of density according to different identification processes.

If we place stress conditions of experimental points on the plasticity ellipse with $\frac{S_1}{\sqrt{3}}/Y$ and S_2/Y reduced coordinates for density levels calculated with HIP dilatometer data (TA6-4, f1 and f2) it can be noticed (Fig. 8 and 9) that:

Unidirectional pressing points are close of plasticity ellipse knowing that precision of data is limited particularly for HIP dilatometer.

In opposite upsetting points are definitively apart.

It is interesting to look at angular position of plasticity points: $\theta = Arctg(\frac{S_2}{S_1*\sqrt{3}})$ (Fig. 9)

For upsetting, positioning angle is constant: $tg(\theta) = \sqrt{2}$

For unidirectional pressing positioning angle varies in a large range.

For HIP dilatometer, angles are small which means that stress conditions are close of isotropic. An evaluation of angle range function of ψ range from 0 (flat densification) to 1 (isotropic densification) is between 18 and 0°. This angle has been put in Fig. 9.

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In HIP densification in a capsule, stress conditions are located between isotropic and flat densification HIP dilatometer according to the capsule design. Upsetting conditions are usually not met in regular HIP of capsules with powder.

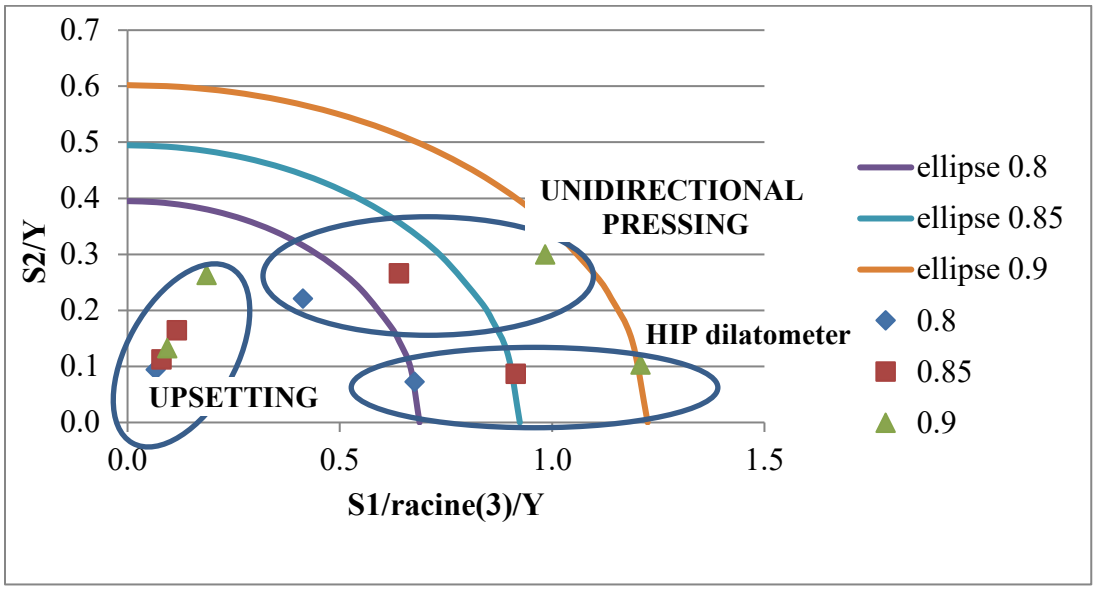

Figure 8: Reduced plasticity ellipse for 3 density levels and corresponding experimental points obtained with 3 identification processes.

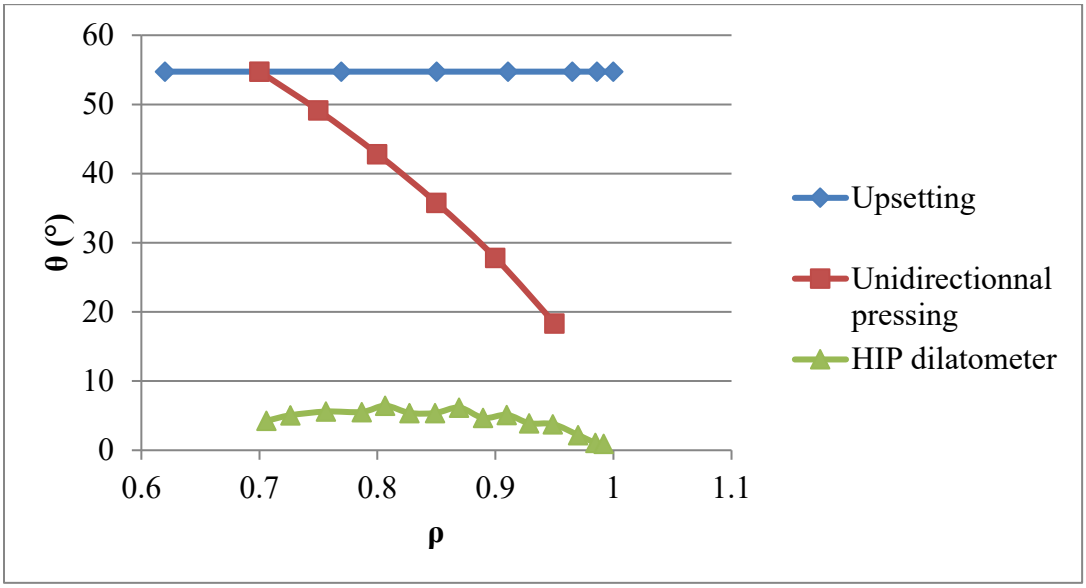

Figure 9: Angular positioning of experimental points in function of density and identification process.

Intermediate conclusions

Identification process of coefficients of Green's law is an important issue. Knowing that real stress conditions during HIP densification are located between 0 (isotropic) and 18° angles on the plasticity ellipse and so, it is possible to claim that upsetting is not representative of met conditions. HIP dilatometer and unidirectional pressing gives closer results. It means that other efficient identification processes are existing and that it would not be useful to modify Green's law in order to introduce a coupling of isotropic and shear stresses as several authors have proposed [6, 7, 8].

Optimizations of determination of powder radial and longitudinal strain rates

Experimental conditions of HIP dilatometer are not optimal particularly at low density. There are two possibilities to improve the situation:

To design a new experimental HIP dilatometer device to use long thin wall capsules. It means investment and time.

To use classical interrupted cycles with optimized capsule geometry to generate a non-negligible shear stress and to get uniform axial strain in the middle of capsule where deformation pattern is uniform on a section.

The evenness of f1 function of density shows that identification between 0.75 and 0.9 density and a step of 5% (in order to limit temperature gap) which means 4 or 5 capsules would be efficient. Using available experimental data used previously, it has been carried out a simulation of a 5% density step in comparison of 2% step used has been done (figure 10). It shows that results are very similar and extrapolation toward tap density and full density is easy.

Measuring radial strain rate is easy after cutting and measuring of powder radius. Measuring axial strain rate either by a precise measurement of powder radius and capsule thickness after HIP using mass conservation equations or by a direct measurement of grooves drawn at the external surface of capsules.

A critical issue is to use the same temperature and pressure ramps in order to avoid scattering. It will be necessary to optimize capsule design in order to get a sufficient shear stress level in the capsule.

For identification of f2, the new proposed method must be more thoroughly analyzed for low density in order to account for thermal conductivity, which is a fundamental issue in the beginning of HIP cycle for thick parts.

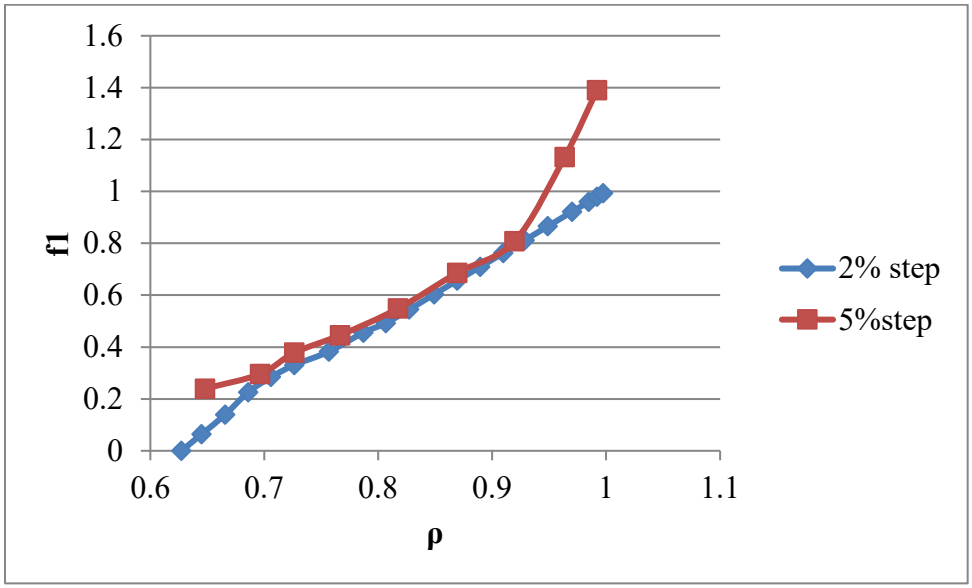

Figure 10: f1 function of density with 2 and 5% density step.

Conclusion

Comparison of experimental data of 3 identification processes shows that unidirectional pressing and HIP dilatometer gives consistent results for f1 coefficient. In opposite, upsetting conditions are not representative of met conditions during real powder densification.

It is possible to identify f1 coefficient with a better precision through a simplified process consisting in 5 or 6 HIP interrupted HIP cycles of cylindrical capsule with a precise dimensional

measurement before HIP and after HIP to reveal the difference between the axial and radial strain rates.

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