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Efficient Modeling of PM HIP for Very Large NNS Parts (up to 2.5 Meter Diameter) and Key Physical, Material and Technological Parameters to Control Dimensional Scattering in a 15 mm Range

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Abstract: To get via PM HIP a 2.5 meter diameter or even larger part in a Near Net Shape (NNS) configuration at the first attempt with a 15 mm max over thickness is a very challenging task. However, its solution will enable production of such parts for various critical applications. To achieve this goal, it is necessary to increase precision of HIP modeling. Our analysis has shown that rather than to work on constitutive equations, it is more efficient to improve the consistency of the data base of rheological properties for the powder and capsule materials and more particularly at the first step of HIP cycle which controls heat conductivity. Also it is necessary to account additional effects of the strain rate hardening for the capsule material and of the initial packing density for the powder.

Independently of modeling, it is necessary to control all parameters generating the dimensional scattering (HIP cycle trajectory, temperature homogeneity, filling and handling of capsules). Modeling helps to define what the most critical parameters are and what dimensional tolerances can be respected.

Introduction

Production of very large parts by HIPing of thin wall capsules filled with pre-alloyed powder is one of the major fields of development of powder metallurgy where this technology can be advantageous. For large parts, economically speaking over thickness is an important issue because of the cost of scrapped materials and subsequent machining costs. Limitation of over thickness to 15 mm means a control of distortion of +- 7.5mm. For a 2.5 m diameter part if we consider isotropic conditions, the shrinkage will be around 15%, i.e. 375 mm. So, 7.5 mm precision requirement represents 2% of total distortion.

To reach this very ambitious target, modeling is an important tool. The paper shows what are the key points and difficulties with modeling. Besides modeling, a lot of parameters (process or mechanical) can be a cause of scattering. For example, control of filling density and its uniformity is not at all trivial for thin wall large parts. Modeling can also be very useful to evaluate sensitivity of final geometry to these technological parameters.

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

Modeling

Mechanical analysis

Development of efficient modeling programs has been carried out in the beginning of 1980s [1] and [2]. Generally speaking, they use Green analysis [3] using stresses equilibrium with:

- Full dense material flow stress
- 2 coefficients depending of relative density giving effect of density on densification related to pressure and on shear distortion related to shear stress.

Some other authors [4], [5] have proposed to introduce other coefficients to take in count crossed effect of pressure and shear stress.

Later on, we will use the Green formulation:

$$\frac{\sigma^2}{f_2^2} + \frac{T^2}{f_1^2} = Y$$

Where T is the shear stress, σ the mean stress (or pressure), Y full dense material flow stress and f_1 and f_2 two coefficients (plasticity functions) depending on the relative density of powder during densification.

Parameters identification

In order to identify these functions and coefficients mechanical trials are performed:

- Full dense material flow stress (Y in equation 1) is measured by classical upsetting at several temperatures with a controlled strain rate.
- Pressure coefficient (f_2 function) can be obtained either through interrupted HIP cycles. In any case a calculation is needed to know the actual pressure in the powder that is shielded by a HIP can.
- Shear stress coefficient (f_1 function) can be obtained by upsetting trials with several density levels.
- In the frame of this presentation, it has been used HIP dilatometer technology [4] and [5]. The principle of device is given in figure 1. An axial and a radial recording have been carried out with the same HIP cycle. The main advantage of this technology is to allow a continuous recording of data. Shielding effect of capsule has to be calculated to get actual pressure and shear stress in powder. In adjusting thickness and material of capsule, it is possible to control shear stress and this parameter is closer of conditions met in an actual industrial capsule in comparison of conventional upsetting. Shielding effect of capsule is relatively easy to calculate by an analytical way for a long thin walled capsule.
- In this paper it will be presented mainly f_2 function of density results. Presentation of f_1 function will be the subject of a future publication.

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

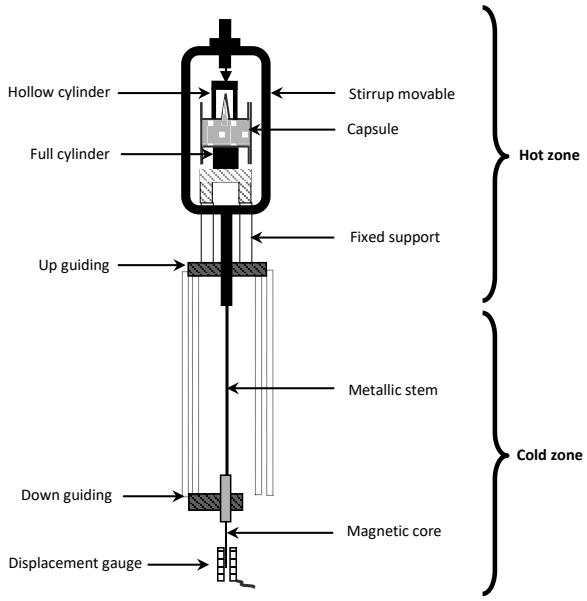

Figure 1: Principle of Villetaneuse HIP dilatometer [6] and [7].

A first comment, at this level, is that the principle of full dense material rheology is not actually known and, some way:

- Microstructure is evolving all along HIP cycle with associated effect on flow stress.
- Strain rate at the scale of particle is inhomogeneous particularly in the first stage of densification.

Actually, it means that it is not realistic to identify independently full dense material flow stress and coefficients. In fact, only a set (Y, f_1, f_2) is identified and valid physically speaking. For example, for isotropic trial, if full dense material flow stress is given, identification gives a set (Y, f_2) in order that P= f_2*Y where P is the (calculated) actual pressure in the powder. If there is a Y misestimating, f_2 value will compensate it if we give the sets of values. It means that it is necessary for identification trials (interrupted cycles or HIP dilatometer) to stay close to the HIP cycle that is used for making a part and to carry out upsetting trials at the same temperature of cycle interruptions. We'll see in the next paragraph what means:"to stay close to the HIP cycle".

Three capsules of Ti6Al4V PREP powder have been densified according to 3 different pressure ramps when heating rate is kept constant (figure2, [6], [7]). Pressure in powder is also indicated. Figure 3 gives corresponding densification curves.

Using the same Y function of temperature law for the full dense material, f_2 function of relative density is obtained and presented in figure 4. It can be seen that in a certain validity domain, a simple rheology of "full dense material" can be used. The same result has been obtained on 316L and nickel base grades.

On figure 5, it is shown densification rate in function of relative density for 316L with several pressure levels with the heating rate kept constant[7]. Densification is over before the temperature dwell is reached and the curves are identical taking in account effect of initial density. Same trials were carried out for TI6AL4V and nickel base grade with the same results. It

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

means that the deformation pattern during densification is similar, what could justify proposed approach. It is also evident that a change in heating rate will change these curves and the sets (**Y,** f_2) should be adjusted.

The important question is: what precision of parameters involved in mechanical analysis (rather sets of parameters) is needed? For massive parts, shear stress level in powder is low as the influence of capsule is small. Therefore, some incertitude on f_1 coefficient could be acceptable.

In opposite, particularly due to the heterogeneity of temperature (as it will be seen later), the densification curve (the f_2 coefficient and the Y function of temperature) has to be as precise as possible.

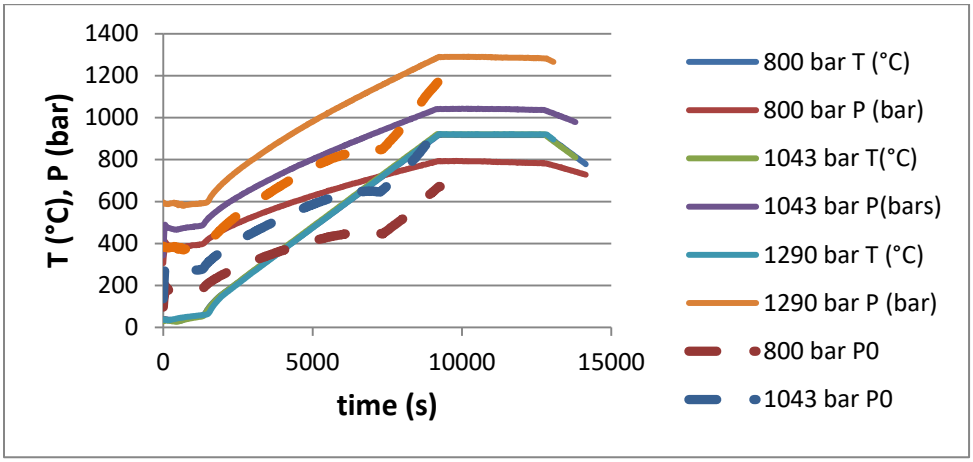

Figure 2: TI6AL4V HIP parameters with calculated powder pressure.

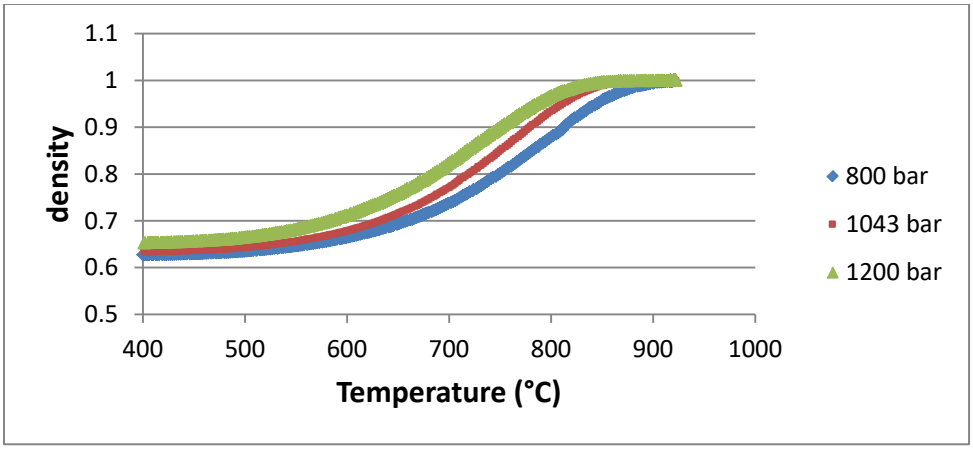

Figure 3: TI6AL4V densification curves.

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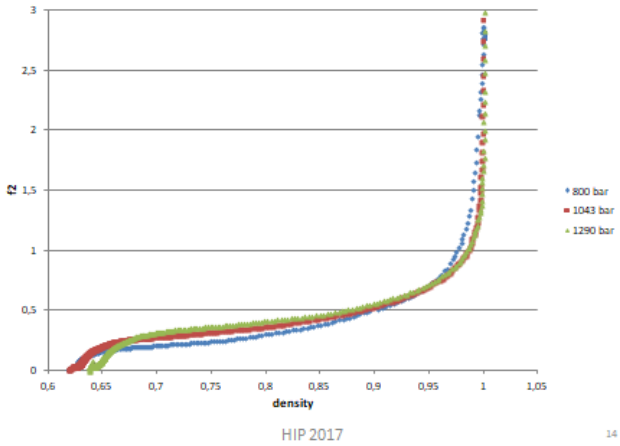

Figure 4: f_2 function of density.

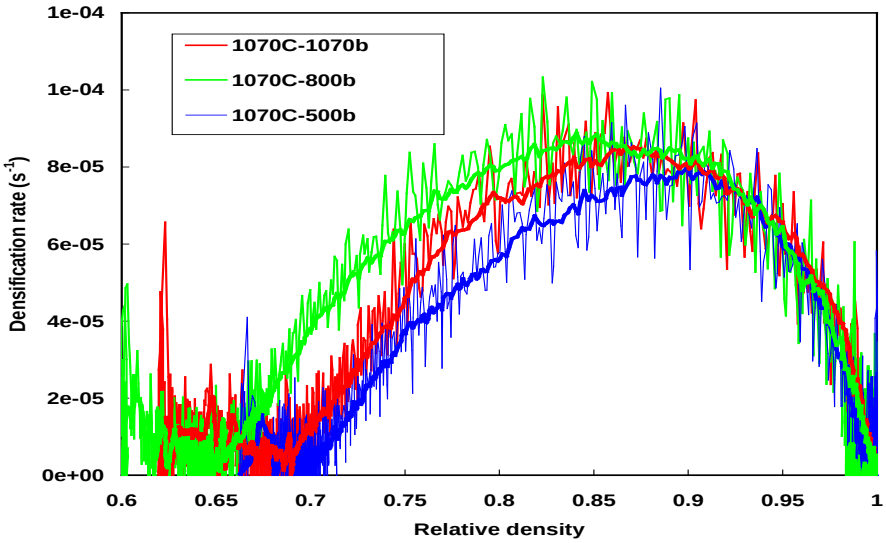

Figure 5: 316L densification rate function of relative density for three pressure levels [7].

Also the strain rate sensitivity of the capsule material can influence its stiffness and the resulting deformation pattern in the HIP cycle. Therefore we have undertaken the following Strain Rate study.

To study the influence of the strain hardening of the capsule material on the HIP deformation pattern and the final geometry we consider the deformation of a long cylinder (the length much exceeding the radius).

For such a HIP can we can neglect the influence of the lids.

With Z being the axis of symmetry, let the area $0 < r < R, 0 < z < H$ be the area of powder and $R < r < R + h, 0 < z < H$ - of the capsule material

As before, the Green's criterion was used to describe the "Plasticity Condition" for the powder material:

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

$$\frac{\sigma^2}{f_2^2}+\frac{s^2}{f_1^2}=Y^2 \tag{1}$$

Here: σ - the average stress; s^2- intensity of the deviatoric stress tensor; f_1, f_2 - experimental values of the plasticity functions of the current density ρ: Y - the yield strength of the full dense powder material.
Here:

$$\varepsilon_{ij}=\omega\frac{\partial\Phi}{\partial\sigma_{ij}} \tag{2}$$

where: ε_{ij} - the components of the strain rate tensor,

The capsule material is non-compressible and is described by the ideal plasticity law:

$$s^2=T^2 \tag{3}$$

Where T - is the yield strength.

Let U_r, U_z be the radial and axial rates. We consider the strain rate on the symmetry axis constant. Then in the powder material:

$$U_z=-\varepsilon z,\ U_r=-Ar. \tag{4}$$

Due to the non-compressibility of the capsule material and continual radial rate at $r=R$ we have:

$$U_z=-\varepsilon z,\ U_r=\frac{1}{2}\varepsilon r-\frac{1}{2}\varepsilon\frac{R^2}{r}-A\frac{R^2}{r} \tag{5}$$

Using the equilibrium equation of the cylindrical system of coordinates:

$$\frac{\partial\sigma_r}{\partial r}+\frac{\sigma_r-\sigma_\varphi}{r}=0, \tag{6}$$

and an integral equilibrium equation relative to the Z axis, we get:

$$2\pi\int_0^{R_1}\sigma_z r dr=-P\pi R_1^2 \tag{7}$$

(where $R_1=R+h$, P - external pressure), assuming that $h<<R$ (relatively thin capsule), we obtain the following relations for ε, A

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

$$Y \cdot \frac{f_1^2(3\varepsilon - 1)}{\sqrt{\left(9 f_2^2 - 2 f_1^2\right) + 3 f_1^2\left((1-\varepsilon)^2 + 2\varepsilon^2\right)}} + 2\frac{h}{R} T \frac{\sqrt{3}\varepsilon}{\sqrt{3\varepsilon^2 + 1}} = 0 \tag{8}$$

$$A = \frac{1}{2}(1 - \varepsilon) \tag{9}$$

Notice that $\varepsilon \in \left[0; \frac{1}{3}\right]$:

at $f_1 \to 0$ the value of $\varepsilon \to 0$ - flat deformation

at $f_2 \to \infty$ the value of $\varepsilon \to 0$ - flat deformation.

at $h \to 0$ the value of $\varepsilon \to \frac{1}{3}$ - uniform compression

This partially explains the difficulties of predicting the initial stages of densification. One the one hand, the capsule is thin, on another -at the low densities the value of f_1 is small. As far as $\varepsilon \in \left[0; \frac{1}{3}\right]$, i.e. relatively small, in accordance with (8), we have an approximate equation to determine the value of ε

$$\varepsilon = \frac{Y}{\left\{ Y \cdot \frac{3 f_1^2}{\sqrt{\left(9 f_2^2 - 2 f_1^2\right)}} + 2\sqrt{3}\frac{h}{R} T \right\}} \cdot \frac{f_1^2}{\sqrt{\left(9 f_2^2 - 2 f_1^2\right)}} \tag{10}$$

The values of H and R as functions of the density ρ are determined by (11) and (12) :

$$H(\rho) = H_0 \exp\left\{ -\int_{\rho_0}^{\rho} \frac{Y}{Y \cdot \frac{3 f_1^2}{\sqrt{\left(9 f_2^2 - 2 f_1^2\right)}} + 2\sqrt{3}\frac{h}{R} T} \cdot \frac{f_1^2}{\sqrt{\left(9 f_2^2 - 2 f_1^2\right)}} \frac{d\rho}{\rho} \right\} \tag{11}$$

$$R^2(\rho) = R_0^2 \frac{\rho_0}{\rho} \cdot \frac{H_0}{H} \tag{12}$$

Where R_0, H_0 are the initial values.

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

If the capsule yield strength as a function of the strain rate is presented as: $T = B\left(e_u\right)^{\alpha}$ (13)

Where $e_u = \frac{3}{2}\sqrt{\left(\varepsilon_r^2 + \varepsilon_\varphi^2 + \varepsilon_z^2\right)}$

Let the external pressure as a function of times is:

$$P = P_0 t \tag{14}$$

Then, introducing the following dimensional values:

Dimensional time τ as $t = \frac{Y}{P_0}\tau$; dimensional A as $\overline{A}$ in the form of

$A = \frac{P_0}{Y}\overline{A}$ (the over score further omitted), get the following relations:

$$\frac{2 f_1^2\left[1-x\right]}{\sqrt{\left(9 f_2^2 - 2 f_1^2\right)\left(2+x\right)^2 + 6 f_1^2\left(2+x^2\right)}} - \gamma A^{\alpha}\left(x^2+x+1\right)^{\frac{\alpha}{2}} \frac{\sqrt{3}x}{\sqrt{\left(1+x+x^2\right)}}\frac{h}{R} = 0 \tag{15}$$

$$\frac{1}{3}\frac{\left[9 f_2^2(2+x) + 2 f_1^2(1-x)\right]}{\sqrt{9 f_2^2\left(2+x\right)^2 + 2 f_1^2\left(1-x\right)^2}} + \gamma A^{\alpha}\left(x^2+x+1\right)^{\frac{\alpha}{2}}\sqrt{\frac{1}{3}}\frac{\left(x+2\right)}{\sqrt{\left(1+x+x^2\right)}}\frac{h}{R} = \tau \tag{16}$$

Where $\gamma = \frac{B}{Y}\left(\frac{3\sqrt{2}}{2}\frac{P_0}{Y}\right)^{\alpha}$, $x = \frac{\varepsilon}{A}$

The density is described by the equation:

$$\frac{d\rho}{d\tau} = \rho A\left(2+x\right) \tag{17}$$

The solution for a specific material is defined by the two parameters:

$\gamma = \frac{B}{Y}\left(\frac{3\sqrt{2}}{2}\frac{P_0}{Y}\right)^{\alpha}$, characterizing the rate of loading and

α - that describes the yield strength dependence of the strain rate

Calculations were done for the f_1 and f_2 coefficients derived from the dilatometric studies and show a serious influence of the strain rate dependence of the capsule flow stress on the deformation pattern measured by anisotropy of deformations (figure 6).

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

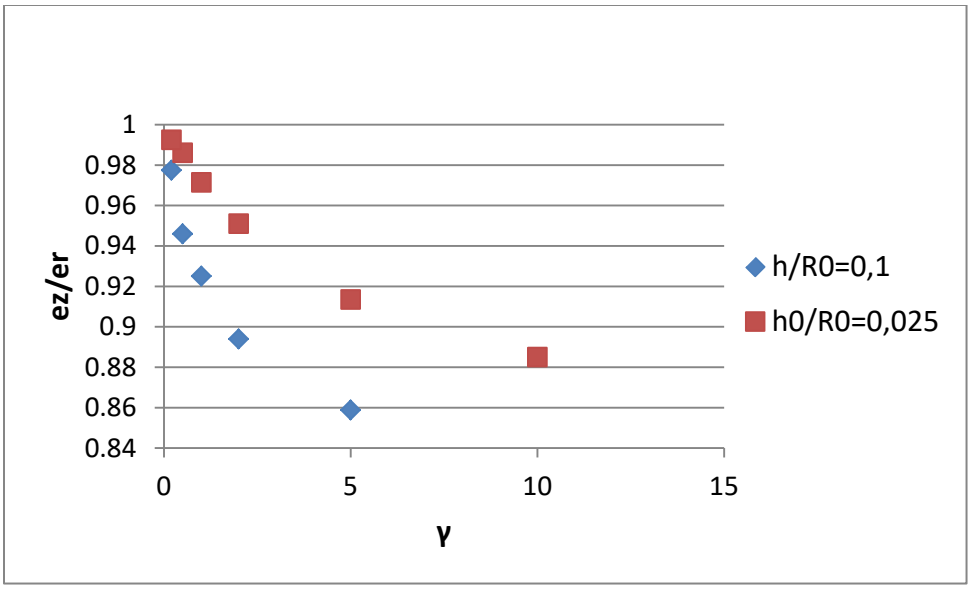

Figure 6: Anisotropy of densification in function of γ parameters (α 0.2).

This parametric study shows that actual strain rate dependence of capsule flow stress has to be accounted as it can strongly influence the deformation pattern:

- Densification rate varies along HIP cycle
- α and γ vary along HIP cycle
- anisotropic effect grows when temperature is growing, strain-rate is lowering and capsule thickness-diameter ratio is growing.

Thermal conductivity:

For massive parts, it is mandatory to introduce thermal conductivity because of non negligible temperature gradients in the part.

Thermal conductivity has been introduced in LNT and Abouaf modeling [1], [9]. An important point is to know thermal conductivity as a function of relative density. Using work of Argento [8] , the relationship is:

$$\lambda(\rho) = \lambda_0 * \left(\frac{\rho - \rho_0}{1 - \rho_0} \right)^{1,46*(1-\rho_0)}$$

where λ0 is thermal conductivity of full dense material, ρ0 initial density of powder, λ(ρ) thermal conductivity at density ρ. Figure 7 shows the curves λ f(ρ) for several initial densities function. The difference in the values of the thermal conductivity in the same points of the HIP trajectory is significant and must be properly accounted. During the very beginning of densification is due to initial cold pressurization of HIP unit, so, strain of powder is purely elasto-plastic. Relationship between pressure and density can be obtained through Ashby or Raisson analysis [10], [11]. These very close results are given in figure 8.

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

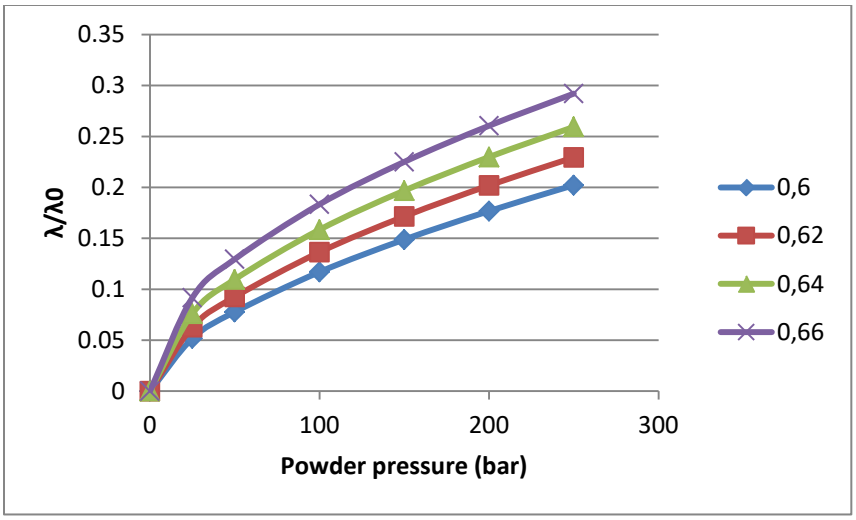

Figure 7: Thermal conductivity of TI6AL4V in function of pressure and initial density.

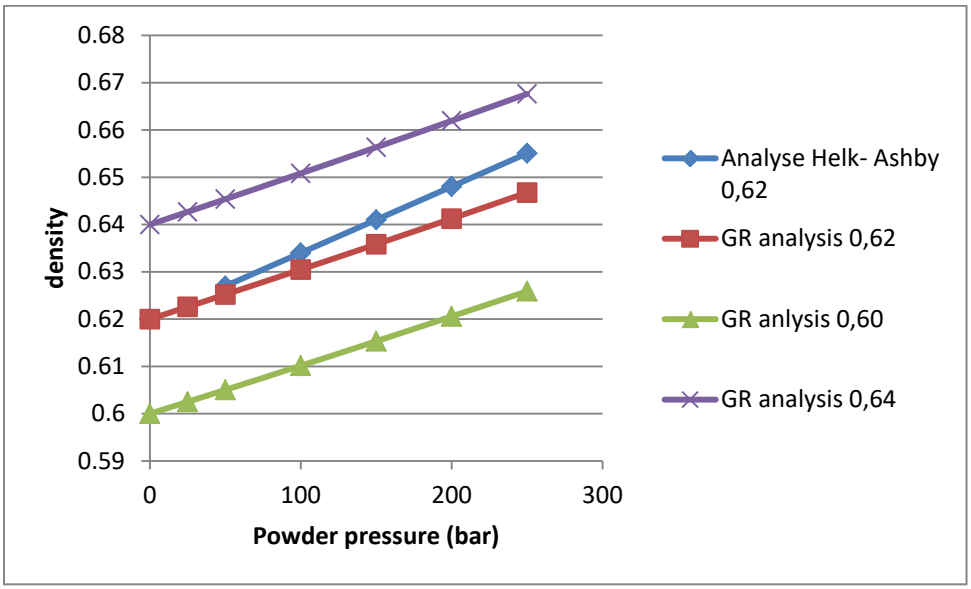

Figure 8: Density of TI6AL4V powder in function of pressure.

It is evident that there is a strong effect of initial density and pressure on thermal conductivity during the first stage of HIP cycle. At higher density, the effect becomes negligible.

Critical parameters for dimensional scattering:

As far as the massive parts are concerned, two parameters are critical for precision of modeling:

a. Filling density and its possible heterogeneity in a large (high) can where the weight effects become significant:

i. Mean filling density knowing that 1% difference of density generates 0.3% geometry discrepancy and mainly

ii. Heterogeneity of filling density which generates local discrepancy, the worst being 3D effect (bending) when heterogeneity is asymmetric [10]. For large thin walled capsule it is difficult to get the same density in any place. It needs a specific know-how to be developed.

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

iii. Additional complement of tapping during the transportation between filling and HIP shops.

b. Taking into account thermal conductivity and consequently again filling density and HIP cycle and particularly the pressure and temperature ramp and in order to estimate the relative effect of parameters, two modeling have been carried out:

i. For a low alloyed steel ring (internal diameter 183 mm, external diameter 1100 mm, height 100 mm) shrinkage ratio of internal diameter has been calculated in function of initial thermal conductivity (figure 9). The effect is major and at low initial thermal conductivity, the deformation is even negative (outward)

ii. For a nickel base massive ring (internal radius 500 mm, external diameter 1000 mm, height 500 mm) change of external diameter and height has been calculated in function of filling density with two hypothesis:

1. Simple law giving initial thermal conductivity function of filling density.
2. Calculation of density and thermal conductivity function of filling density and pressure after pressurization according to **b-c** paragraph.

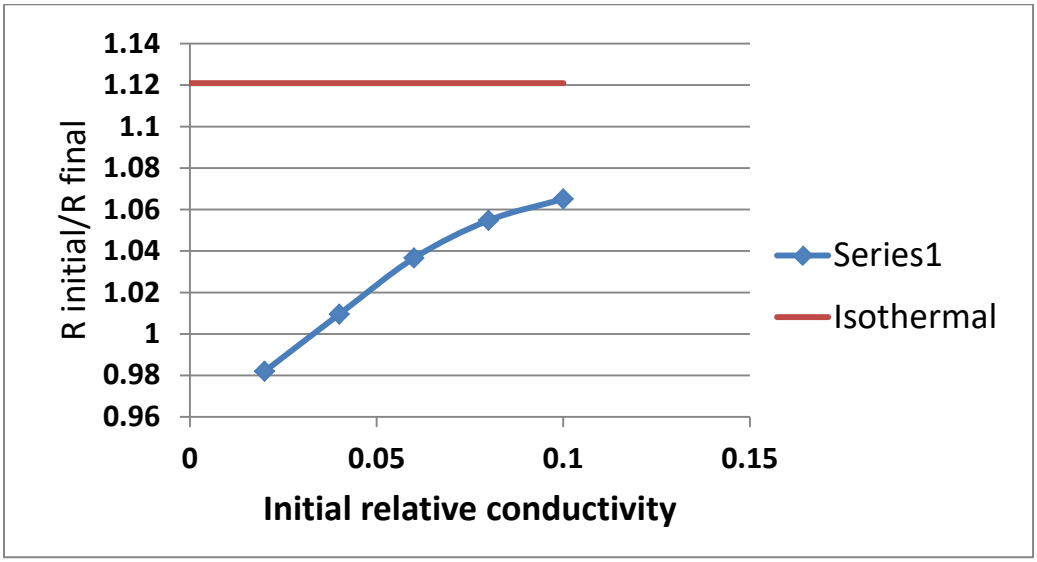

Figure 9: Internal diameter shrinkage ratio of low alloyed steel ring function of initial thermal conductivity.

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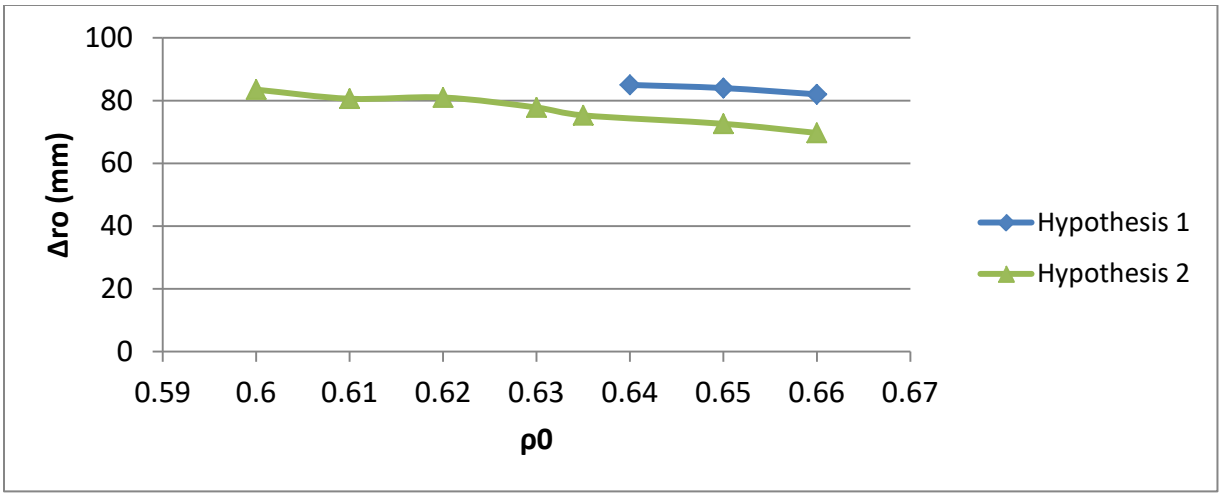

Figure 10: Variation of external radius of a nickel base ring function of filling density and hypothesis for treatment of density and thermal conductivity during pressurization step.

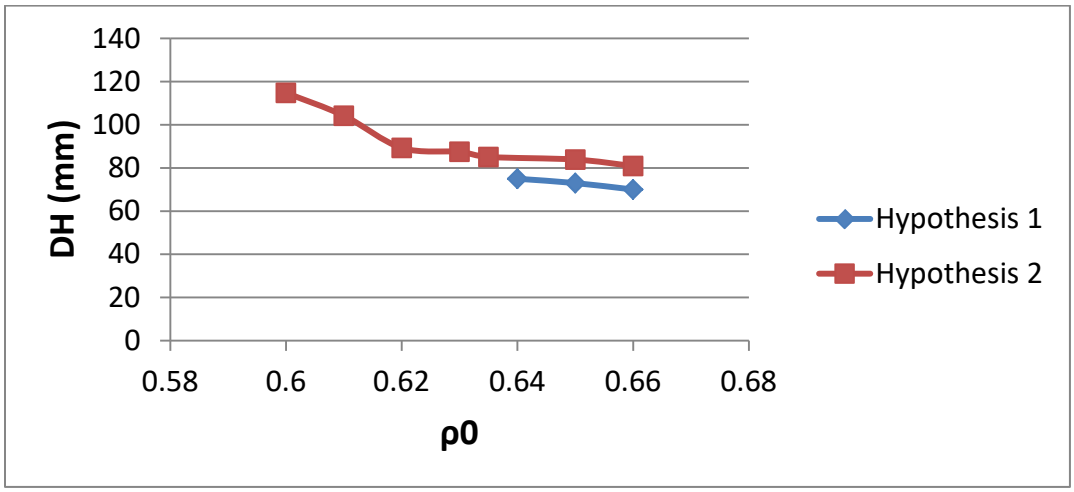

Figure 11: Variation of height of a nickel base ring function of filling density and hypothesis for treatment of density and thermal conductivity during pressurization step.

The effect (figures 10 and 11) is minor but not negligible (around 10 mm) and should be accounted for large parts

Others scattering parameters:

a. Geometry of the capsule:
   i. At the level of making of large size welded capsule
   ii. During filling due to stresses generated by the weight of powder. Some additional supporting tooling needs to be developed.
   iii. During handling of a several tons part. If handling points are not well designed, there is a risk of irreversible plastification of capsule.
   iv. During handling again due to shocks giving depressions. Experience shows that it is not a negligible risk.

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

b. Cooling step: particularly for low alloyed steel (when massive parts of the HIP capsule are made from this steel), γ->α transformation corresponds to up to 0,7 % expansion as a function of cooling rate. Due to non-uniform temperature distribution, it generates some anisotropic plastic deformation of the HIP capsule reaching several millimeters.
c. Powder batch: the knowledge of powder characteristics is an evident issue. It means that filling density and rheology are perfectly reproducible. On figure 12, it is shown densification curve for Astroloy powder produced by Rotating Electrode Pulverization and Gas Atomization with the same HIP cycle and particle size distribution [11]. Effect of process which controls microstructure is strong. But the most important item is actual filling density.

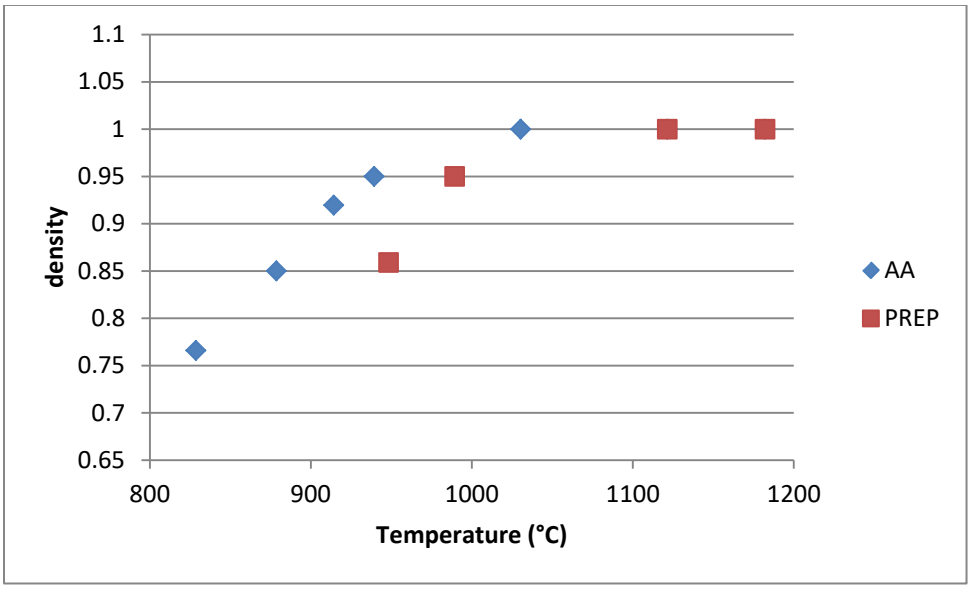

Figure 12: Densification curves for Astroloy PREP and AA powders. [13].

Conclusion

To get the given target to have at disposal a modeling allowing at the first attempt to produce a 2.5 m diameter massive part with a precision better than +-7.5 mm seems realistic:

- It is not necessary to use a sophisticated law for full dense material rheology as far as heating rate is rigorously controlled in production HIP cycles and pressure ramp kept in a validity domain.
- Initial conditions are fundamental issues to control thermal conductivity. Some progress is still necessary to have a good physical description of the first step of densification (pressurization).
- Modeling is not enough to guaranty to reach the target. A lot of other parameters have to be taken in account. Modeling can help to determine the order of magnitude of their effect.

Materials Research Proceedings **10** (2019) 210-223 doi: http://dx.doi.org/10.21741/9781644900031-28

References

[1] Abouaf, Chenot, Raisson: Finite Elements Simulation of Hot Isostatic Pressing of Metal Powder. J.Numerical Methods in Engineering 25, 191-212, 1988. https://doi.org/10.1002/nme.1620250116

[2] Samarov et al: Application of HIP for Near Net Shape critical parts and components. Hot Isostatic Pressing 1993 L.Delaey and H.Tas (editors) 1994 Elsevier Science B.V. pp 171-185. https://doi.org/10.1016/B978-0-444-89959-0.50022-0

[3] R.J.Green: Int.Mech.Sci., 1972, 14, (4), 215-224. https://doi.org/10.1016/0020-7403(72)90063-X

[4] Sanchez, Ouedraogo, Stutz, Dellis: Integration of a new viscoplastic constitutive equation in a finite element code. Powder Metallurgy World Congress, 98, Vol 2, pp 531-536, Granada, Spain.

[5] P.Viot: Modelisation du comportement viscoplastique des poudres métalliques. Developpement d'un nouveau dispositif experimental de Compression Oedometrique à Chaud. *« Modeling of viscoplastic behavior of metallic powder. Development of a new Hot Oedometric Pressing device »*. PHD thesis 2000 december 18 Grenoble 1.

[6] Fondere, Federzoni, Raisson: Identification Models of Powder Densification using in-situ HIP Dilatometer. 2004 PM World Congress. Vienna October 2004.

[7] Burlet, Gillia : Model identification for powder densification. HIP 2005 Paris may 2005

[8] C.Argento and D.Bouvard: Modeling the effective thermal conductivity of random packing of spheres through densification. *Int. J. Heat Mass Transfer* vol.**39**-7, 1343-1350 (1996)

[9] Goloveshkin, Raisson, Ponomarev, Bochkov: Accounting of non-stationary Temperature Field while modeling of HIP for large size Components. HIP 2011 Kobe April 2011.

[10] Samarov, Raisson, Seliverstov, Goloveshkin: Principles of shape and densification Control during HIP. HIP 2011 Kobe April 2011.

[11] Ashby: The Modelling of Hot Isostatic Pressing. Proceedings of th international Conference on Hot Isostatic Pressing. Lulea, 1987.

[12] Van Nuguyen, Bezold, Broeckmann: Anisotropic Shrinkage of Hot Isostatically Pressed Components. HIP 2014 Stockholm June 2014.

[13] Than-Chi Lu: Structure superficielle des poudres de superalliages base nickel et mécanismes intervenant au cours de la densification. *Nickel base Superalloys Surface Structure and involved mechanism during densification"* PHD thesis Ecole des Mines de Paris 1987.