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► **To cite this version:**

Jiupeng Song, Ghassane Ayad, Thierry Barrière, Jean-Claude Gelin, Baosheng Liu, et al.. Modeling, simulation and identification of the solid state sintering after ceramic injection moulding. 7e colloque national en calcul des structures, CSMA, May 2005, Giens, France. hal-01813010

HAL Id: hal-01813010

<https://hal.science/hal-01813010>

Submitted on 12 Jun 2018

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Modelling, simulation and identification of the solid state sintering after ceramic injection moulding

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RÉSUMÉ. Un modèle continu basé sur l'évolution d'une loi de comportement visco-plastique est utilisée pour décrire le procédé de densification par diffusion en phase solide pour des composants fabriqués par le procédé de moulage par injection de poudres céramiques CIM. Le modèle a été implémenté dans un code éléments finis développé sur la plateforme Matlab[®]. Une méthode d'optimisation est proposée afin d'identifier les paramètres du modèle de la loi de comportement. La simulation d'une prothèse de hanche est réalisée par ce procédé CIM est pris comme exemple du point de vue numérique et expérimentale. Les résultats numériques sont validés par des essais expérimentaux.

ABSTRACT. To describe the sintering process of solid state diffusion for the components of ceramic powders, obtained by the powder injection moulding and debinding, a continuum model based on the constitutive law in viscoplastic type is introduced. This physical model has been implemented in a Finite Element Software developed on the platform Matlab[®]. Then an optimization method has been developed for the identification of parameters in the proposed sintering model. The identification by optimization software has been applied on a powder moulded component of the hip implant, consisted of the alumina ceramic powders. Based on the comparisons between experiments and modelling by FE simulations, the identification procedures give accurate results.

MOTS-CLÉS : densifications, moulage par injection de poudres céramiques, simulation numérique, identification.

KEYWORDS: sintering, ceramic injection moulding, alumina ceramic, numerical simulation, identification.

1. Introduction

Ceramic injection moulding (CIM) is a combination of technologies in particulate materials, injection molding and sintering processes. It is currently used in ceramic industries to manufacture in large quantities the small, complex and near-net-shaped components with high performance. In CIM, the sintering stage is one of the most important steps, in which the debinded green components are treated at the temperature below the melting point of the main constituent to obtain the desired final properties by bonding the powder particles together. The shrinkage between the green component and the sintered one is generally in the range 10-20% and the density of the final component is in the range 90-100% (German, 1997).

Because of the large shrinkage in sintering process, determination of the dimensional changes and distortions is necessary in industrial production to produce the components in near-net-shape and high quality. Besides the conventional trial and error method, numerical simulation of the sintering stage may be a fast and cost-effective alternative to solve this problem. Several constitutive models based on the continuum mechanics have been developed to describe the sintering process. A purely phenomenological model due to Olevsky seems easy to be implemented in numerical simulations, but is restricted for the possibilities to get accurate results (Olevsky, 1998). In the experimental model due to Bouvard (Bouvard, 1996), data are directly obtained from real materials but the investigation on loading conditions is limited at high temperature, and the other stress conditions are calculated by extrapolation. The micromechanical model due to Riedel and Kraft (Riedel *et al.*, 1994) seems comprehensive for solid sintering because it considers majority of the mechanisms and factors in the entire sintering process, but it is necessary to adjust the model parameters for various materials.

In this paper, a viscoplastic constitutive model and the associated finite element method (FEM) are used to simulate the sintering process. The numerical identification methods, incorporated with dilatometer experiments, are used to determine the parameters in constitutive model. More accurate simulation can be carried out based on this model, because the constitutive parameters are determined with the same software that will be used for simulation. The simulations are applied on the components of alumina ceramic powders obtained by CIM and debinding. Compared with the experimental data, the simulation results are reasonable.

2. Sintering model

2.1. Constitutive sintering model

To set up the relationship between the macroscopic strain rate tensor and the stress tensor in sintering, a linear viscoplastic (creep) law is used (Riedel *et al.*, 1994). Such a constitutive law is expressed:

$$\dot{\boldsymbol{\varepsilon}}^{vp} = \frac{\boldsymbol{\sigma}'}{2G} + \frac{\text{tr}(\boldsymbol{\sigma}) - 3\sigma_s}{9K} \mathbf{I} \quad [1]$$

where $\boldsymbol{\sigma}'$ is the deviatoric stress tensor, $\text{tr}(\boldsymbol{\sigma})$ is the trace of stress tensor, \mathbf{I} is the identity tensor of second order, G is the shear viscosity modulus, K is the bulk viscosity modulus, and σ_s is the sintering stress.

To identify the parameters in the constitutive law for sintering process, different models have been developed. In the present study, following expressions are used (McMeeking *et al.* 1992):

$$G = \left(\frac{d_0}{d}\right)^{\frac{2}{3}} \left(\frac{d-d_0}{1-d_0}\right)^2 \frac{d r_0^3}{90D_{\text{iff}}(T)} \quad [2]$$

$$K = \left(\frac{d_0}{d}\right)^{\frac{2}{3}} \left(\frac{d-d_0}{1-d_0}\right)^2 \frac{d r_0^3}{54D_{\text{iff}}(T)} \quad [3]$$

$$\sigma_s = \frac{8\gamma_s}{r_0} \left(\frac{d_0}{d}\right)^{\frac{1}{3}} d \quad [4]$$

where r_0 is the initial radius of spherical powder particles, d_0 the initial relative density, d is relative density, γ_s is the surface energy of the particles. D_{iff} is a diffusion term generally written in the following form:

$$D_{\text{iff}}(T) = \frac{\Omega \delta D_{\text{b0}} e^{-\frac{Q_b}{RT}}}{kT} \quad [5]$$

where k is the Boltzmann constant, Ω the atomic volume, δD_{b0} is a diffusion coefficient at grain interface, R the specific gas constant and Q_b is an activation energy. The main limitations of relationships in [2], [3] and [4] are related to their validity from the initial density of the porous media to the final dense state

2.2. Governing equations

The momentum conservation equations and mass conservation equation describe the mechanical behavior of the powder aggregation during the sintering stage (Renault *et al.*, 2002). The moment conservation equation is written as :

$$\nabla \cdot \boldsymbol{\sigma} + \mathbf{f} = \rho \frac{\partial^2 \mathbf{u}}{\partial t^2} \quad [6]$$

where $\boldsymbol{\sigma}$ is the Cauchy stress tensor, \mathbf{f} is external applied force, \mathbf{u} is the displacement field and t is the current processing time. Because the sintering process is very slow, the inertia forces in the right hand of the equation [6] are often neglected. The calculation of relative density is related to the mass conservation equation expressed as:

$$\dot{d} + d \cdot \text{tr}(\dot{\boldsymbol{\epsilon}}^{\text{vp}}) = 0 \quad [7]$$

2.3. FEM solution procedures

The coupled equations [1, 6, 7] are solved by FEM in the following forms. The velocity and relative density are chosen as the main nodal variables:

$$\begin{aligned} \mathbf{u} &= \mathbf{N}\mathbf{u}_n \\ \dot{\boldsymbol{\epsilon}} &= \mathbf{B}\dot{\mathbf{u}}_n \\ d &= \mathbf{N}d_n \end{aligned} \quad [8]$$

where \mathbf{N} are the matrix of interpolation functions, \mathbf{B} is strain rate interpolation matrix. Galerkin method is applied to build the resulting FE equations expressed as:

$$\begin{aligned} \mathbf{M}_d(\mathbf{u}_n)\dot{d}_n + \mathbf{C}_d(\mathbf{u}_n, \dot{\mathbf{u}}_n)d_n &= 0 \\ \mathbf{K}_u(\mathbf{u}_n, d_n)\dot{\mathbf{u}}_n + \mathbf{F}_u(d_n) &= 0 \end{aligned} \quad [9]$$

The matrixes \mathbf{M}_d , \mathbf{C}_d , \mathbf{K}_u and \mathbf{F}_u are expressed by the following relationships:

$$\begin{aligned} \mathbf{M}_d(\mathbf{u}) &= \sum_e \int_{\Omega_e(\mathbf{u})} \mathbf{N}^T \mathbf{N} dV \\ \mathbf{C}_d(\mathbf{u}, \dot{\mathbf{u}}) &= \sum_e \int_{\Omega_e(\mathbf{u})} \mathbf{N}^T \text{tr}(\mathbf{B}\dot{\mathbf{u}}_n) \mathbf{N} dV \\ \mathbf{K}_u(\mathbf{u}, d, T) &= \sum_e \int_{\Omega_e(\mathbf{u})} \mathbf{B}^T \mathbf{A}(d, T) \mathbf{B} dV \\ \mathbf{F}_u(d) &= \sum_e \int_{\Omega_e(\mathbf{u})} (-\mathbf{N}^T f(d) + \mathbf{B}^T \sigma_s(d) \mathbf{I}) dV \end{aligned} \quad [10]$$

where \sum_e stands for the assembly operator over the elements. $\mathbf{A}(d, T)$ is a tensor describing the viscoplastic constitutive behavior:

$$\begin{aligned} \boldsymbol{\sigma} &= \mathbf{A}(d, T) : \dot{\boldsymbol{\epsilon}}^{\text{vp}} + \sigma_s \mathbf{I} \\ \mathbf{A}(d, T) &= 2G(d, T) \mathbf{I}_4 + \left(K(d, T) - \frac{2}{3} G(d, T) \right) \mathbf{I} \otimes \mathbf{I} \end{aligned} \quad [11]$$

where \otimes is the tensorial product operator.

For free sintering, \mathbf{f} includes only gravity effects expressed as:

$$f(d) = -\rho_{th}(Nd)\mathbf{g}\begin{bmatrix} 0 & 0 & 1 \end{bmatrix}^T \quad [12]$$

where ρ_{th} is the theoretical density and \mathbf{g} is gravity acceleration.

Galerkin method is used to build the FEM procedures for solution of the governing equations and constitutive equations. An explicit high order Runge-Kutta scheme is employed for the time integration, developed on the platform of Matlab®.

3. Numerical identification

3.1. Identification algorithm for sintering physical parameters

The identification algorithm is designed for proper determination of the material parameters in the constitutive models, which is to be used in subsequent numerical simulations. The dilatometer experiments are used to study the densification behavior of the specified material, in which the shrinkage curve is obtained for 1D case. On the other hand, the numerical simulations based on the above sintering model are carried out for 1D part with the given initial value for the material parameters. A numerical response curve is obtained by means of the FE simulations. The optimization strategy is proposed to minimize the differences between both the shrinkage curves obtained by dilatometer experiments and numerical simulations, respectively. The material parameters are the variables to be optimized. So the optimization is formulated as a minimization problem expressed as

$$\begin{cases} \min F(x) \\ F(x) = \sum_{i=1}^n |f_e(T_i, x) - f_s(T_i, x)|^2 \\ x = [\delta D_{b0}, Q_b, \gamma_s, r_0] \end{cases} \quad [13]$$

where $f_e(T, x)$ is the shrinkage curve resulting from the dilatometer experiments, $f_s(T, x)$ is the shrinkage curve of numerical simulations, $F(x)$ is the mean residual squares between both the two curves where $i=1, \dots, n$ are sintering temperatures, x is the set of material parameters.

3.2. Identification for alumina ceramic powders

The identification method is applied to get the material parameters of injection moulded alumina ceramic powders. The data given in (Opfermann, *et al.* 1998) are used as reference for dilatometer experiments. The shrinkage curve obtained by using the initial material parameters is optimized in order to approach the curve obtained from dilatometer experiments, as shown in Fig. 1. The initial values are chosen from the references.

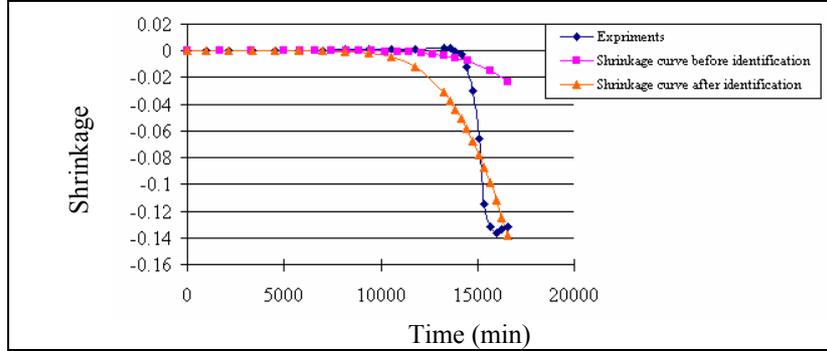


Fig. 1. Comparison between shrinkage curves obtained by experiments and optimization

The initial material parameters of alumina ceramic powders obtained from the references and the ones obtained by identification are listed in Table 1.

Table1. Identified material parameters

Parameter	Initial	References	Identified
Q_b (KJ/mol)	384	He <i>et al</i> 2005	327.5
δD_{b0} (m^3/s)	8.6×10^{-10}	Cannon <i>et al</i> 1980	7.367×10^{-10}
γ_s (J/m^2)	0.71	Rhee S.K 1972	0.8431
r_0 (m)	7×10^{-6}	He <i>et al</i> 2005	5.128×10^{-6}
Ω (m^3)	4.2×10^{-29}	Chevalier <i>et al</i> 1997	5.004×10^{-29}

4. Validation example

The injection moulded hip implant parts of alumina ceramic powder are chosen as an example to validate the sintering model and proposed identification procedure.

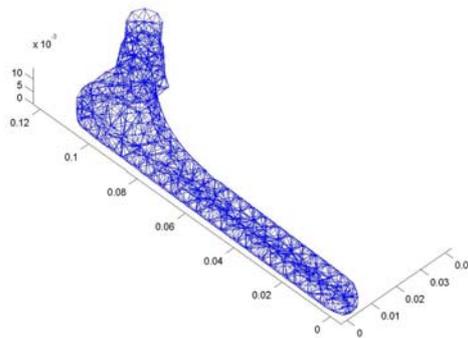


Fig. 2. Mesh of the hip implant component

The mesh of the part is given in Fig. 2. The temperature cycle for FE simulations is heating to 1400°C with the rate 5°C/min. The identified material parameters given in Table.1 are used for FE simulation. With the identified parameters, the final relative density obtained by simulation in the part is given in Fig. 3.

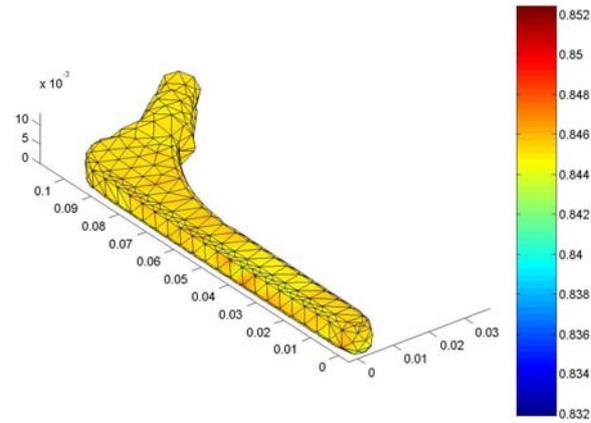


Fig. 3. The final relative density in hip implant component

The averaged shrinkage ratio is 11.85%. The final relative density is about 84.5%. Such a result is in good agreement with the dilatometer experiments.

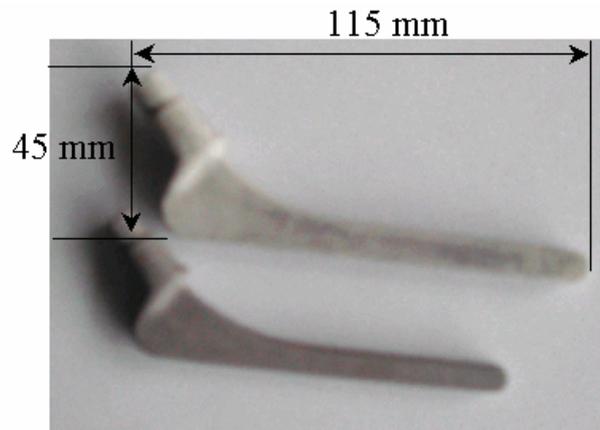


Fig. 4 The photos of the sintered hip implant

For sintering experiments, the green part of hip implant after debinding is sintered in a furnace. The temperature cycle is the same as prescribed in simulation. The

photographs on the parts of hip implant, after moulding and sintering respectively, are shown in Figure 3.

In experiments, the shrinkage is about 13% and the final relative density is about 95%. In general, the simulation results agree with the experiments. But the final relative density obtained by simulation is less than the experimental ones. This is due to the fact that the sintering model expressed by equations [2-5] is not totally suitable for the final sintering stage.

4. Conclusion

A phenomenological model for the sintering by solid state diffusion has been proposed, based on the physical considerations and the determination of main driving parameters. The experiments using dilatometer have been carried out on alumina ceramic powder with different temperature cycles. These experiments provide the necessary data for identification of the parameters in the constitutive model employed in simulation. This identification is surely combined with the use of simulation software and developed optimization tool. This method is justified by the comparison of experimental results and the ones issued by simulation, on an example of the sintering of a hip implant component. Such a component is obtained by the injection moulding and debinding of the alumina ceramic powder. The results are in good agreement, which validates the modelling, simulation and the identification procedure.

Acknowledgement

The authors present their thanks to the support of French Embassy in China for the alternant doctoral education project (20041859) and the Education Ministry of China for the doctoral education fund (20020613005).

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