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# Experimental based full process simulation of alumina selective laser processed parts densified by cold isostatic pressing and solid state sintering

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#### Abstract

The compound process of cold isostatic pressing (CIP) of alumina selective laser processed (SLP) parts and solid state sintering (SSS) and its full process simulation were realized in this paper, focusing on studying the overall deformation, relative density distribution, grain growth and sintering stress variation during the process. Especially, correlation was established between the macroscopic deformation and microscopic evolution. Model parameters for alumina are presented, which were optimized in accordance with the experimental results. CIPed part still exhibited density inhomogeneity, of which SSS tended to increase the overall density and homogenize density distribution. The sintering behavior was studied with the employment of dilatometer experiments. Furthermore, compared with conventional heating strategy, fast firing turned out to decrease sintering production time as well as drive the matter diffusion and densification process. The master sintering curve (MSC) moves upward a little under the condition of fast firing.

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#### 1. Introduction

Structural ceramics like alumina have been increasingly applied in modern industry due to their excellent mechanical and physical properties such as heat and corrosion resistance. Although the conventional technologies such as gelcasting can process high density ceramic components with complex structures, the difficulties in manufacturing the molds and discharging the organics remain to be settled. Selective laser processing (SLP) can manufacture complex components without any molds, reducing the mold production cost. Furthermore, when combined with cold isostatic pressing (CIP), the porous SLPed parts can be densified with homogeneous properties. Solid state sintering (SSS) is a common post-processing technology for ceramic densification. This SLP/CIP/SSS compound process proves to be a promising way to manufacture complex high density ceramic parts.

Currently, a small number of scholars have studied the SLP/CIP/SSS compound process. Deckers et al.<sup>3</sup> produced alumina parts with a density of 88% by the compound process, while they did not numerically predict part deformation and densification along the overall process from CIP to SSS. Proper prediction analysis helps to increase the forming precision of the compound process and optimize the SLPed part structure and size design.

Ceramic components undergo obvious contraction and deformation in both processes of CIP and SSS, which challenges the initial structure design, initial size and processing parameter determination. Traditionally, trial and error method or empirical formula was always applied to control the part shape and dimensional accuracy, however, with a higher experimental cost and limited control precision. Finite element simulation can be an effective alternative in predicting part deformation and directing the practical production of the compound process. So far, a few scholars have investigated the numerical simulation of CIP<sup>5,6</sup> and SSS<sup>7,8</sup> processes, respectively. While for the compound process of SLP/CIP/SSS, numerically analyzing the individual process such as CIP cannot meet the demand of controlling the overall forming accuracy of the compound process. This paper

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combined the simulation of CIP and SSS processes of alumina SLPed components, thus realizing the full process simulation of the compound process, which has not been reported in any literature up to now.

With regard to the modeling of SSS process, three major approaches are available, namely microscopic model (physically based), mesoscopic model (stereological)<sup>9</sup> and macroscopic model (phenomenological). 10 Phenomenological models were established to describe densification and viscous behavior of alumina powder during sintering, which yield many practical uses. 11–16 While these models cannot depict the microstructural evolution such as grain growth, which affects the macroscopic densification process in turn. The stereological models rebuild the particle system, used to analyze boundary evolution between particles during sintering in meso level. 17,18 Scientific work performed in physically based model can fully illustrate the thermal dynamic process of ceramic powders during sintering. However, difficulties are presented in obtaining all the parameters required in the physically based model, restraining its application. The model applied in this paper takes alumina boundary flow, sintering stress, grain size and other sintering parameters into consideration, effectively associating the microstructural evolution with macroscopic deformation during sintering.

This paper extends and consolidates the work described in He et al.<sup>6</sup> by fulfilling the full process simulation of the SLP/CIP/SSS compound process. Meanwhile, the microstructural evolution was associated with macroscopic deformation, embedded into the sintering constitutive equations. Furthermore, the influence of initial relative density and heating strategies on part deformation as well as microstructural evolution was systematically studied.

#### 2. Constitutive model of CIP and SSS

Drucker/Prager/Cap model, the typical clay plasticity model, was applied to analyze the CIP process in this work. It has been illustrated in detail in our previous study.<sup>6</sup>

With regard to the modeling of SSS, modified SOVS model<sup>14,15</sup> was used in our work, establishing the links between microscopic grain growth and macroscopic densification.

In this model, the stress–strain rate function is written as

$$\sigma_{ij} = p\delta_{ij} + \tilde{q} \tag{1}$$

where p and  $\tilde{q}$  are, respectively, the equivalent hydrostatic pressure and the Mises equivalent deviatoric stress, p and  $\tilde{q}$  are defined, respectively, as

$$p = \frac{1}{3}tr\sigma = 2\eta\psi\dot{\varepsilon}_{ii} + \sigma_s \tag{2}$$

$$\tilde{q} = \sqrt{\sigma'_{ij}\sigma'_{ij}} = 2\eta\varphi\sqrt{\dot{\varepsilon}'_{ij}\dot{\varepsilon}'_{ij}}$$
(3)

where  $\sigma'_{ij}$  is the deviatoric stress tensor,  $\eta$  is the apparent viscosity of the porous structure, which is a function of temperature T

only,  $\psi$  and  $\varphi$  are the coefficients of the effective shear and bulk moduli,  $\sigma_s$  denotes the sintering stress:

$$\eta = \eta_0 T \exp\left(\frac{Qv}{RT}\right) \tag{4}$$

$$\sigma_s = \frac{6\gamma_s}{G}(1-\theta)^2 \tag{5}$$

$$\varphi = (1 - \theta)^2 \tag{6}$$

$$\psi = \frac{2(1-\theta)^3}{3\theta} \tag{7}$$

where  $\eta_0$  is adjustable,  $Q_v$ ,  $\gamma_s$ , G, respectively, are the effective activation energy for material flow, the surface tension energy and the mean grain size. R is the gas constant, and porosity  $\theta$  is expressed as the ratio of the volume of pores  $V_{pores}$  to the total volume  $V_{total}$ :

$$\theta = \frac{V_{pores}}{V_{total}} \tag{8}$$

For sintering process characterized by creep and time dependent volumetric swelling, the creep and swelling laws are defined in the user subroutine (viscoelastic behavior) as  $\Delta \bar{\epsilon}^{cr}$  and  $\Delta \bar{\epsilon}^{sw}$ :

$$\Delta \bar{\varepsilon}^{cr} = \frac{\tilde{q}}{3\psi\varphi} \Delta t \tag{9}$$

$$\Delta \bar{\varepsilon}^{sw} = -\frac{p + \sigma_s}{2\psi\varphi} \tag{10}$$

As for solid state sintering of alumina powders, the densification is controlled by grain boundary diffusion and grain growth is controlled by the grain boundaries. The relationship between grain size and relative density named sintering path curve was gained by Guillaume Bernard-Granger<sup>19,20</sup> as follows:

$$\frac{1}{G^2} = -K_1 \rho + K_2$$

$$K_1 = \frac{\gamma_b D_b^{\dagger}}{100 \gamma_{sv} \delta_b^2 D_b}$$

$$K_2 = \frac{\gamma_b D_b^{\dagger}}{100 \gamma_{sv} \delta_b^2 D_b} \rho_0 + \frac{1}{G_0^2} = K_1 \rho_0 + \frac{1}{G_0^2}$$
(11)

where  $\delta_b$  is the grain boundary thickness,  $\gamma_b$  is the surface tension of grain boundary,  $D_b$  is the matter diffusion coefficient along the grain boundary,  $\gamma_{sv}$  is the solid–vapor surface tension,  $D_b^{\dagger}$  is the matter diffusion coefficient through the grain boundary,  $\rho_0$  is the initial relative density,  $G_0$  is the initial grain size. The  $K_1$  and  $K_2$  are constant whatever the temperature, which seems to be reasonable.

In order to determine temperature fields of alumina parts during sintering, the mathematic model of heating needs to be established. The model can be written as follows, ignoring deformation in the process:

$$\frac{\partial(\rho c_p T)}{\partial t} + \frac{\partial(q''_{diff,i})}{\partial x_i} + \frac{\partial(q''_{conv,i})}{\partial x_i} + \frac{S_{Rad}}{V} = 0$$
 (12)

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