# Ceramic Sintering And Properties Characterization Based On Solid Mechanics

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## ABSTRACT

Solid mechanics offers a considerable potential to describe densification and deformation during sintering as well as the polycrystal structure-properties relationship. Densification and deformation are described during sintering by means of appropriate creep constitutive laws. These laws connect macroscopic behavior with structural parameters such as grain growth and porosity. FEM simulation allows to obtain maps to describe density, stress, and deformation distribution in order to have a better characterization of macroscopic deformation during sintering. Effective properties estimation for polycrystal is developed based on a homogenization technique applied to a representative volume element (RVE) with the help of the Finite Element Method (FEM). Implementing this model, the effect of the crystallographic texture on the ceramic properties can be estimated by considering the interaction at grain boundaries, which provides a more realistic analysis of the material properties.

***Keywords***: solid mechanic, ceramic sintering, effective properties estimation, homogenization, EBSD.

## 1 INTRODUCTION

Solid mechanics studies body deformations under the action of forces [1]. The systematic descriptions of body mechanical behaviors provide a formalism that constitutes a useful tool to study a wider variety of phenomena that goes beyond traditional mechanical behaviors, like constrained sintering [2] and magneto-electro-elastic behaviors [3].

Sintering is known as a process where a powder compact transforms into a useful solid [4]. At a particle size level, necks between particles grow resulting in a better mechanical performance. Experimental manipulation of ceramic firing process is somehow tedious. Several techniques have been developed to face these complex processing. One of the most popular techniques relies on different approaches for the design of experiments and/or statistical process control.

In this line, solid mechanics provides a useful description where constrained sintering is taking place [2], which can be a result of several factors such as non-uniform particle size distribution, multi-materials systems, rigid substrates, or rigid inclusions [5]. For these cases, it is established to work with constitutive laws to study the effect of stress distribution [6]. Microstructural heterogeneities along the powder compact have a macroscopic effect on the sintering body [7]. The viscous parameter that describes sintering can also be obtained from a sinter forging unit[8], [9] and considered for the simulation process. This could be a more direct way to characterize sintering for a constrained system, for example, a bilaminar layer where high stresses can be developed at the interphase. These examples show how helpful can be solid mechanics to characterize ceramics materials processing.
Properties manipulation for composite materials is a topic of great interest. Several micromechanical theories have been developed to estimate final properties. One of them is the Asymptotic homogenization method (AHM) [10] which is based on a carefully revised mathematical background. Recently, a hybrid method based on AHM and the Finite Element Method (FEM) has allowed an easier way to implement the effective properties estimation [11], [12]. Another useful option for effective properties estimation was developed by H. Berger *et al.* [13] and it is based on FEM and appropriate boundary conditions. This type of models opens the doors to connect properties estimation with structural characterization techniques. As an example, it is

shown a Representative Volume Element (RVE) built from Electro Backscattering Diffraction (EBSD) and the overall properties are connected with the pole figure.

## 2 FUNDAMENTALS

In Solid Mechanics, there are three major moments to check up when a static problem is stated. They are:

i) the stress equilibrium:

$$\sigma_{ij,j} = 0\,, \tag{1}$$

where $\sigma_{ij}$ are the components of the stress tensor and the comma notation is the partial derivate relative to the $x_j$ spacial coordinates.

ii) The constitutive relations:

$$\sigma_{ij}(x_m) = C_{ijkl}(x_m)\varepsilon_{kl}(x_m)\,, \tag{2}$$

where $C_{ijkl}$ and $\varepsilon_{kl}$ are the components of the stifness and the strain tensors [1]. Eq. (2) is known as the generalized Hooke law. It describes a linear relation between strain and stress by means of stiffness, i.e., a cause and its effect are connected by the material properties.

iii) The body surfaces are divided into several sections, and they are classified into two types: surfaces where stress values are imposed and surfaces where displacement values are imposed. No intersection is allowed between these surface sections.

### 2.1 Ceramic Sintering

The generalized Hooke law can take a reduced form for isotropic materials [14], where only two elastic properties are involved. They are the Young's modulus $(E_0)$ and the Poisson coefficient $(\nu)$. The thermal dilation effect can be included adding the term $\varepsilon_f$ where $\alpha$ and $\Delta T$ are the thermal dilatation coefficient and the temperature difference with respect to a reference temperature. Then, volumetric strain components for thermomechanical problems can be written as:

$$\begin{aligned}
\varepsilon_x &= \varepsilon_f + E_0^{-1}\left[\sigma_x - \nu_o\left(\sigma_y + \sigma_z\right)\right],\\
\varepsilon_y &= \varepsilon_f + E_0^{-1}\left[\sigma_y - \nu_o\left(\sigma_z + \sigma_x\right)\right],\\
\varepsilon_z &= \varepsilon_f + E_0^{-1}\left[\sigma_z - \nu_o\left(\sigma_x + \sigma_y\right)\right],\\
\varepsilon_f &= \alpha\,\Delta T.
\end{aligned} \tag{3}$$

The visco – elastic analogy has been stated to characterize constrained sintering, where a linear viscous behavior is capable of describing mechanical deformations during sintering [2]. As shown in Eq. (4), the strain components are replaced by the strain rate components $(\dot{\varepsilon} = d\varepsilon / dt)$; the Young's modulus by uniaxial viscosity; Poison coefficient by viscous Poisson coefficient and thermal strain by free sintering strain. Uniaxial viscosity and the viscous Poisson coefficient have received considerable attention in Refs. [8], [15]–[17] and, as expected, are directly related to the structural evolution during sintering. The free sintering rate is related to the free sintering processes where no constrain or external stresses are involved.

$$\varepsilon_{ij} \Leftrightarrow \dot{\varepsilon}_{ij}\,,\quad E_0 \Leftrightarrow E_p\,,\quad \nu_0 \Leftrightarrow \nu_p\,,\quad \varepsilon_f \Leftrightarrow \dot{\varepsilon}_f \tag{4}$$

Eq. (3) is rewritten with the definitions of Eq. (4) and a useful tool to characterize and control sintering creep is available. Nowadays, works are going on in order to improve this description including anisotropic effects [18].

### 2.2 Effective properties

The structure-properties relationship is a key issue in materials science and engineering. The established formalism of solid mechanics considering a variety of constitutive relations provides a way to study material system performances. Specifically, homogenization techniques establish a connection between heterogeneous and an equivalent homogeneous media [10]. Figure 1 illustrates the two-scale asymptotic homogenization method (AHM) considered. In material science, this tool provides a connection between material structure (Fig. 1 (b)) and properties at a macrolevel (Fig. 1(a)).

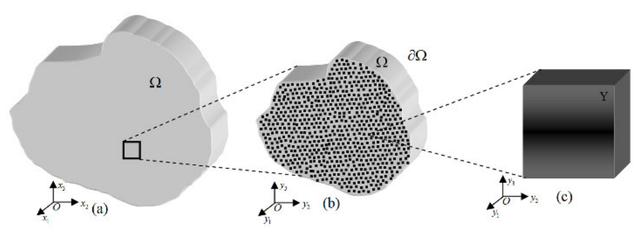


*Figure 1. A two-scale homogenization approach. (a) Homogenized media or material at macroscopic level. (b) Heterogeneous media representing a periodic microstructure. (c) The representative volume element (RVE).*

Most of the homogenization techniques rely on defining the RVE which consists of identifying a small section of the microstructure capable of describing the whole material. Of course, this assumption can be questionable, and some treatments have been developed to accomplish more realistic descriptions [19]–[22].

Substituting Eq. (2) into Eq. (1), the following equation is obtained:

$$\left(C_{ijkl}(x_m)\,\varepsilon_{kl}(x_m)\right)_{,j} = 0\,, \tag{5}$$


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then, when the AHM is applied, Eq. (5) is transformed into Eq. (6):

$$\bar{C}_{ijkl}\,\varepsilon_{kl,j} = 0\,. \tag{6}$$

Eq. (5) can be connected to Fig. 1(b) and Eq. (6) to Fig. 1(a). $\bar{C}_{ijkl}$ are known as the effective properties and from the AHM, the following relation is obtained:

$$\bar{C}_{ijpq} = \left\langle C_{ijpq} + C_{ijkl}\,{}_{pq}L_{k,l} \right\rangle, \tag{7}$$

where $L_k$ is a function of the interactions at the interphaces, for example, grain boundaries. The symbol $\langle\cdot\rangle$ represents the volume average that is usually considered over the REV. More information about this procedure can be found in the literature [12], [23].

## 3 CASE STUDIES

### 3.1 Ceramic Sintering


*Figure 2. Ceramic bilaminar where the thinner layer has higher viscosity. From lest to right: an example of a ceramic bilaminar, von Mises stress and density maps.*

Figure 2 illustrates the result of the numerical implementation using Eq. (3) and considering the visco-elastic analogy (Eq. (4)). Available FEM software can be used. Fig. 2 shows from left to right, an example of a ceramic bilaminar, the von Mises and density maps. von Mises maps are especially useful because they provide stress distribution and a stress value estimation. It is very difficult, not to say impossible, to experimentally obtain these data. For the case of bilaminar shown in Fig. 2, it can be observed that the maximum von Mises stress values are obtained at the interphase. This is an expected results considering that the two materials have different viscosity values. Then, the two layers will shrink at different velocities generating stress at the interface. The numerical value of this stress is of interest to avoid, for example, a detachment. In general, the stress distribution in the whole sample is of interest because it is related to shape deformation and densification. The density map reported in Fig. 2 shows a non-uniform distribution. As expected, the higher densities are reached in the layer with lower viscosity. Anyway, density distribution is affected by the stress distribution. It is possible to directly measure density distribution, but sometimes may be tedious. At least measuring densities in some parts of the sample may be a way to validate the numerical implementation in addition to shape deformation.

### 3.2 Effective properties

As mentioned in the previous section, the effective properties estimation by AHM has as one of the starting points of the RVE. A number of simplified geometries have been reported in the literature and useful results have been obtained. A large number of numerical implementations and comparisons between different homogenization techniques is a proof of the high level of accuracy of these techniques, which offer an important potential to better understand and control properties of polycrystals materials.

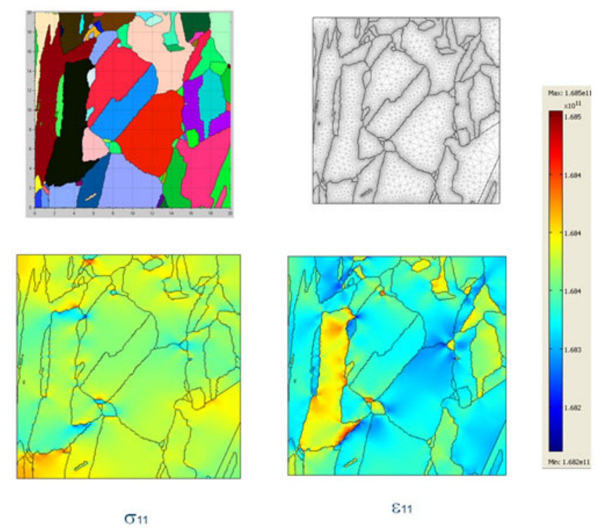


*Figure 3. Numerical implementation starting from an Electron Backscattering Diffraction (EBSD).*

Fig. 3 illustrates an Electron Backscattering Diffraction (EBSD) map (top left) where each color represents a different crystal orientation. Each colored area is a grain domain. This map is transformed into a FEM model (top right), where each grain has different property components because of its crystal orientation. Then, the local problem for calculating $L_k$ is solved as requested by Eq. (7). Then, a map of stress distribution (down left) and strain distribution (down right) is calculated. It is important to note that stress and strain distribution is not uniform inside of each grain, and the cause of this fact can be attributed to the interaction at the grain boundaries. The reported EBSD can be taken as the RVE and Eq. (7) should be used to calculate the effective properties. This way, a more realistic property estimation can be performed.

## 4 CONCLUSIONS

Solid mechanics is known as a subject that deals with solid bodies behaviors under different types of loads. The fundamental formalism of this subject has reached a level that allows detailed descriptions of materials behaviors such as mechanical, electrical, magnetic, and thermal ones, including coupling effects between them. Creep during sintering of powder compact is another useful application of the mentioned formalism. In conclusion, solid mechanics provides useful tools for advanced materials characterization.

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