

Evaluation of sintering stress from 3-D visualization of microstructure: Case study of glass films sintered by viscous flow and imaged by X-ray microtomography

F. Wakai^{a,*}, O. Guillon^b

^a Secure Materials Center, Materials and Structures Laboratory, Tokyo Institute of Technology, R3-23 4259 Nagatsuta, Midori, Yokohama 226-8503, Japan

^b Otto Schott Institute for Materials Research, Friedrich Schiller University of Jena, Loebdergraben 32, D-07743 Jena, Germany

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Abstract

The sintering stress is a driving force for morphological evolution of pores in sintering, and can be determined from knowledge of the microstructure. The sintering stress of non-equilibrium, non-uniform and non-isotropic porous glass films cast and sintered on rigid substrates was computed from synchrotron X-ray microtomography data. This method was able to show how the inhomogeneous distribution of local density at the particle scale led to a difference in local sintering stress. The anisotropic microstructure in the film was correlated to the deviatoric components of the sintering stress, which were defined by surface energy tensor. The topological evolution of pore structure was characterized by genus and Euler characteristic.

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1. Introduction

Advances in X-ray microtomography allow the observation of complex pore structures in three dimensions. It reveals real microstructural evolution during sintering, and provides an opportunity to analyze the local particle arrangements and non-uniformity at the particle scale [1–5]. Consider, for example, the pore space evolution in viscous sintering of spherical glass particles, as shown in Fig. 1. In the initial stage (Fig. 1a, stage 1), the pore structure is a continuous network with numerous circular holes resulting from contacts between particles. As holes expand with the neck growth, pore channels are pinched off, breaking the continuous network into fragments: closed pores are formed one by one in the intermediate stage (Fig. 1b stage 2 and Fig. 1c stage 3). Complicated shaped pores become spherical in the final stage of sintering (Fig. 1d stage 4).

The macroscopic shrinkage in sintering is a result of the microstructural evolution that is driven by capillarity. The shape and form of pore structures, either open or closed, generate the sintering stress, which in turn is a driving force for shrinkage and change in pore morphology. Both phenomena lead to a decrease in the total surface energy. The sintering stress in a volume element can be, in principle, calculated from the knowledge of pore structures. It is determined rigorously from energy [6], curvature [7] and force balance [8,9] for the equilibrium pore structures where the curvature is constant at any position (constant mean curvature (CMC) surface). The sintering stress is either isotropic [10] or anisotropic [11]. However, real microstructures in the intermediate stage of sintering (Fig. 1b and c) are quite different from such CMC surface models or simple geometric models [12,13]. The shrinkage and sintering stress of non-equilibrium structures has been estimated from sintering simulations using the discrete element method [14] and the Potts kinetic Monte Carlo method [15]. These simulations and theoretical models

* Corresponding author. Tel.: +81 45 924 5361; fax: +81 45 924 5390.
E-mail address: wakai.f.aa@m.titech.ac.jp (F. Wakai).

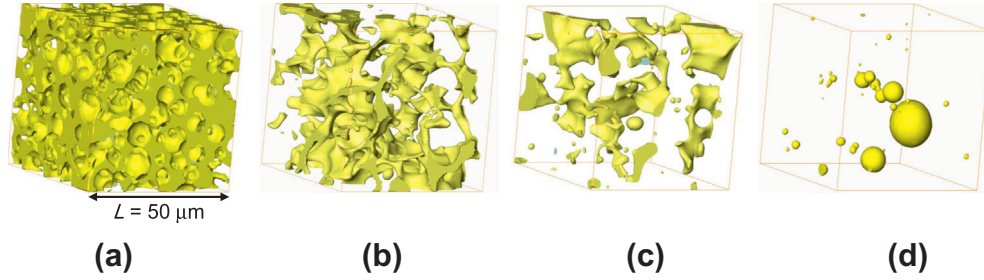


Fig. 1. Evolution of pore space in sintering: (a) stage 1 (relative density, $\rho = 63.5\%$); (b) stage 2 ($\rho = 87.8\%$); (c) stage 3 ($\rho = 94.1\%$); (d) stage 4 ($\rho = 98.4\%$).

[16,17] have been compared with the sintering stress determined experimentally by sinter forging tests [18]. The sintering of crystalline particles is made more complicated by several diffusion mechanisms that contribute concurrently to mass transport [19]. The grain growth further complicates sintering in the later stage. However, the microstructural evolution in viscous sintering of glass is simply described by fluid mechanics [13,20–22]. The sintering stress depends not only on the relative density, but also on the shape and structure of pores [23] and the distribution function of pore size [24,25]. In non-equilibrium porous structure, the sintering stress is expressed by the surface energy tensor, which is defined only by pore geometry [26].

The purpose of the present paper is to analyze the sintering stress of non-equilibrium, non-uniform and non-isotropic pore structures characterized by synchrotron X-ray microtomography, as illustrated in Fig. 1. The continuum theory of sintering is normally concerned with the behavior of matter on a macroscopic scale that is large compared with particles [27–31]. However, real porous sintered bodies are non-uniform when they are viewed on such a small scale as to reveal the individual particles. The consideration of local inhomogeneity in relative density and sintering stress at the particle scale is important for understanding defect formation during sintering.

The initial particle packing is slightly anisotropic, usually owing to powder processing such as uniaxial pressing, injection molding, extrusion and tape casting [32]. The microstructures of sintering bodies become anisotropic also in constrained sintering of a thin film on rigid substrate [27,33–35], as well as in stress-assisted densification such as hot pressing, spark plasma sintering or sinter-forging [14]. Bernard et al. [4] used X-ray computed microtomography to study the constrained sintering of glass films, which were cast on rigid substrates. Anisotropy was analyzed using the autocorrelation function and pore orientation distribution function of the three-dimensional (3-D) image. Here, their tomography data are re-examined from the point of view of sintering mechanics. It is shown that the sintering stress tensors, especially the deviatoric components, are useful to reveal the anisotropic microstructure and its effects on sintering. The aim has been to improve

the understanding of complex pore structures, and to provide a new description of microstructural features and the forces behind their formation/evolution.

2. Mechanics of viscous sintering

The continuum mechanical modeling of sintering is expressed by a set of linear relations between the macroscopic strain rate \dot{E}_{ij} and the macroscopic stress Σ_{ij} [17,35]

$$\dot{E}_{ij} = S_{ijkl}(\Sigma_{kl} - \Sigma_{kl}^s) \quad (1)$$

where S_{ijkl} is the viscous compliance tensor, and Σ_{ij}^s is the sintering stress tensor. The summation convention for repeated indices is applied throughout this paper. The volumetric shrinkage rate, which is the trace of the strain rate tensor, is driven by the hydrostatic component of macroscopic stress $\Sigma_m = \Sigma_{ii}/3$, and the hydrostatic component of the sintering stress $\Sigma^s = \Sigma_{ii}^s/3$. The deviatoric components of the sintering stress induce anisotropic shrinkage. When the microstructure is isotropic, the constitutive equation is simply expressed as [27,29]

$$\dot{E}_{ij} = \frac{\Sigma'_{ij}}{2G} + \delta_{ij} \frac{\Sigma_m - \Sigma^s}{3K} \quad (2)$$

where Σ'_{ij} is the deviatoric stress, G and K are the shear viscosity and the bulk viscosity, respectively.

In viscous sintering, the constitutive Eqs. (1) and (2) are derived directly from the principles of fluid mechanics [26]. The pore deformation is driven by capillary traction $\gamma_s \kappa n_i$ on the pore surface, where γ_s is the surface energy, κ is curvature, and n_i is the unit (outward) normal to the pore surface. Using the virial method [36,37], the total traction on pore surface is related to the surface energy tensor

$$\int_A x_i \gamma_s \kappa n_j dA = \int_A \gamma_s (\delta_{ij} - n_i n_j) dA \quad (3)$$

where x_i is the i th coordinate. The sintering stress of a single pore of arbitrary shape is defined as

$$\sigma_{ij}^s = \frac{1}{V_n} \int_A \gamma_s (\delta_{ij} - n_i n_j) dA \quad (4)$$

where V_n is the pore volume. The macroscopic sintering stress in a volume element V is defined as the volume average of local sintering stress of pores [26]

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