



Dynamic grain growth during low-temperature spark plasma sintering of alumina

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An existing defect model for dynamic grain growth is modified in order to incorporate the role of grain-boundary sliding on the generation of defects during sintering. On the basis of the concept of enhanced defect generation, the unusual grain growth of alumina during spark plasma sintering is discussed in correlation with the densification caused by grain-boundary sliding. The modified model explains well the experimentally observed dependence of grain size on pressure, heating rate and loading schedule. © 2014 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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Sintering is macroscopically a process of densification, and microscopically a process of pore shrinkage and annihilation. During sintering, most of the densification process proceeds in the intermediate stage, where channel-like open pores and large isolated pores shrink by the movement of particles or grains towards the pores. The grain rearrangement without significant shape change, one of the characteristics of sintering, is a result of the grain-boundary sliding, which is the most important mechanism for high-temperature deformation such as superplastic deformation. Grain-boundary sliding is an essential process during densification, and has been incorporated in some theoretical models of sintering [1–3]. In those models, however, grain-boundary sliding was treated as a factor only affecting the densification rate. The present study focuses on the role of grain-boundary sliding to explain the unexpected grain growth behavior observed during spark plasma sintering (SPS) of transparent alumina. Discussion is made on three experimental results of grain growth: two already reported and one newly obtained.

The grain growth during high-temperature deformation, called dynamic grain growth, generally occurs faster

than growth caused by annealing alone, called static grain growth. As a mechanism of dynamic grain growth, several theoretical models have been proposed, such as defect generation [4–5] or damage-zone formation [6] by grain-boundary sliding, grain coalescence [7] and biased grain-boundary diffusion [8]. All the models were experimentally consistent with grain growth during high-temperature deformation. Then, the dynamic effect of grain growth should also appear during sintering, because most densification deeply relates to the deformation or grain-boundary sliding in the porous polycrystalline matrix.

The dynamic effect during sintering was reported first by Besson and Abouaf [5]. They sintered alumina powder directly by hot isostatic pressing (HIP), and observed that the final grain size is linearly dependent on the pressure. The pressure-dependence of the grain size was explained by the increased concentration of defects, such as vacancies. According to the defect model of Clark and Alden [4], an excess concentration of defects is generated as a result of grain-boundary sliding and they are concurrently annealed towards a thermal equilibrium. The equation governing the defect concentration n is represented by

$$\dot{n} = K_1 \dot{\epsilon} - K_2 n \quad (1)$$

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where $\dot{\epsilon}$ is the strain rate, K_1 is an experimental constant, and K_2 is the constant dependent on the thermal nature of defect annihilation. The excess defect concentration increases the grain-boundary mobility and hence the rate of grain growth. Thus, high deformation rates enhance the defect concentration and accelerate the grain growth. Besson and Abouaf [5] modified the defect model by replacing the strain rate in Eq. (1) with the external viscoplastic deformation energy, and obtained a linear relationship between the final grain size and the applied pressure, which is consistent with their experimental observations during HIP of alumina powder. On the other hand, under hydrostatic compressive pressure on dense polycrystals where no deformation occurs, the free volume and the transfer of atoms between grain boundaries are reduced, resulting in a corresponding decrease in the rate of grain growth [9,10].

The increasing grain size with increasing pressure was also observed recently during SPS of transparent alumina. Kim et al. [11] applied high pressures (50–400 MPa) at the beginning of SPS, and observed two types of grain growth behavior: non-dependence and positive dependence of the grain size on pressure. The non-dependence was observed at 1100 °C, and the positive dependence at 1200 °C, as shown in Figure 1. On the pressure effect during SPS, both non-dependence and positive dependence have been reported for zirconia [12] and TaC [13], respectively. For the positive dependence of grain growth, one can consider that the pressure effect is significant at temperatures where the grain-boundary mobility is sufficiently high [11].

However, the observation of the densification behavior during the high-pressure SPS suggests that the densification temperature should also relate to the dynamic grain growth. In general, higher pressures enable densification to occur at lower temperatures. During the high-pressure SPS, remarkable densification occurred in the temperature range 1000–1110 °C at 50 MPa and 940–1040 °C at 300 MPa. The densification at low temperatures tends to increase the stress required for the deformation of grain-boundary sliding and would enhance the defect generation. In addition, since the original defect model [4] is based on the concept of defect

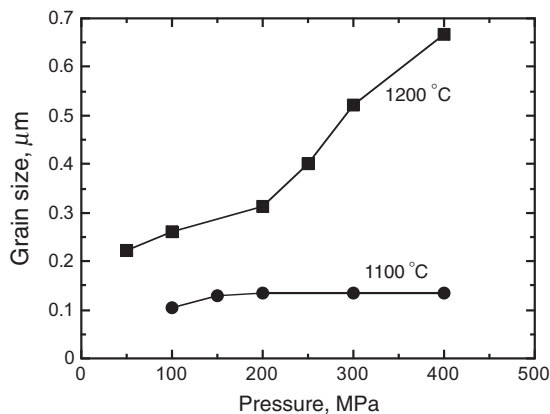


Figure 1. Pressure-dependence of grain size for alumina. After a soaking time of 20 min, the sintered body was obtained with diameter 10 mm and thickness 2 mm.

generation near sliding boundaries, it is more rational to replace the strain rate $\dot{\epsilon}$ in Eq. (1) with a term relating to the grain-boundary sliding. Here, Eq. (1) is modified as

$$\dot{n} = K_1 \eta \dot{\gamma}^2 - K_2 n \quad (2)$$

where η is the viscosity for grain-boundary sliding, and $\dot{\gamma}$ is the shear strain rate on grain boundaries. During sintering, $\dot{\gamma}$ is proportional to the densification rate. In Eq. (2), it is assumed that part of the energy ($\eta \dot{\gamma}^2$) required for viscous grain-boundary sliding is consumed for defect generation. Since η depends on 1/temperature, following the Arrhenius law, the deformation at low temperatures under the application of high pressures enhances defect generation, leading to accelerated grain growth. Thus, the mechanism of the positive dependence of the grain size on the pressure observed during SPS of alumina [11] can be explained physically using Eq. (2).

The modified defect model can also explain the unexpected behavior of grain growth dependent on the heating rate. Kim et al. [14,15] conducted SPS of alumina under fixed sintering conditions, but at various heating rates (2–100 °C min⁻¹). After sintering at 1150 °C for 20 min, the final grain size was observed to increase from 0.29 μm to 0.55 μm with an increase in the heating rate. Similar dependence of the grain size on the heating rate has also been reported during SPS of alumina [16,17] and yttria [18] at low temperatures (1050–1250 °C) without sufficient explanation. The large grain size for rapid heating is contradictory to general understanding, because a higher heating rate indicates a shorter heating time.

In order to understand the unexpected grain growth, the deformation rate during sintering was re-examined. Figure 2 shows the shrinkage rate of the alumina powder compacts during heating. Most shrinkage occurs above 850 °C, and the shrinkage rate and, accordingly, the shear strain rate $\dot{\gamma}$ increase with increasing heating rate. According to the modified defect model in Eq. (2), the defect generation was enhanced by the

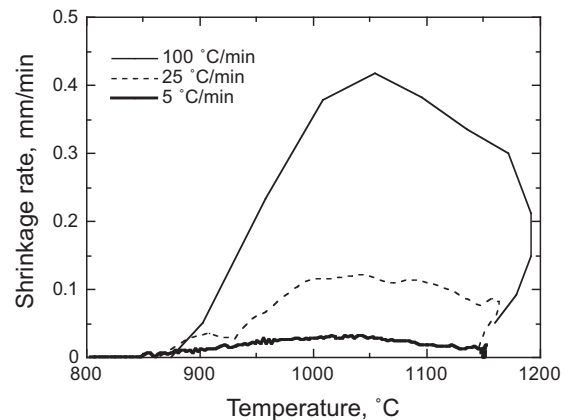


Figure 2. Shrinkage rate of alumina powder compacts at various heating rates during heating to the sintering temperature (1150 °C) at a pressure of 80 MPa. At high heating rates, the specimen temperature temporarily overshoots the sintering temperature. After a soaking time of 20 min, the sintered body was obtained with diameter 30 mm and thickness 3 mm.

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