Stress-enhanced grain growth during high pressure spark plasma sintering (HPSPS) of nanocrystalline alumina.

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Highlights

- Final grain size of alumina increased with the applied pressure during HPSPS.
- Grain growth is stress-enhanced and controlled by creep at relatively low temperatures where diffusion is negligible.
- Strain rate during final stage of sintering controls grain growth.

1. Introduction

The SPS process enables rapid densification of ceramic powder at relatively lower temperatures, which limits grain growth considerably and allows fabrication of fully dense nanocrystalline ceramics. High-pressure spark plasma sintering (HPSPS) allows to reduce the sintering temperature even further and makes it possible to obtain fine nanostructured ceramics. It is well established that the major grain growth takes place during the final stage of sintering (relative density >90%) and typically involves a diffusional process governed by the grain boundary curvature. In on our earlier contribution [1], we have observed an increase in final grain size with applied pressure during HPSPS of nanocrystalline magnesium aluminate spinel. The present work focuses HPSPS of nanocrystalline alumina and on elucidating this phenomenon.

2. Methods

Commercial alumina nano-powder with an initial particle size of about 75 nm was used in this study. SPS was carried out using a hybrid SiC-Carbon tool setting. Sintering was conducted at temperatures of 1000-1050°C under various applied pressures of 500-800 MPa at a heating rate of 12.5 °C/min with dwell times of 0-15 min. Several partially sintered samples were prepared to observe where the grain growth takes place during sintering as well as pressureless sintered samples to measure static grain growth. Samples were ground and mirror polished, then chemically etched in boiling phosphoric acid. Microstructure was characterized by a high-resolution scanning electron microscope. The grain size was meticulously determined by Thixomet® software for image analysis. Hardness of the samples was measured (Vickers microhardness under 2kg load).

3. Results and discussion

We suggest that there is a dynamic grain growth during HPSPS at a relatively low temperature, where conventional coarsening mechanisms are negligible. This grain growth is stress-enhanced and depends on the creep strain rate during final stage of sintering, similar to grain growth during deformation of superplastic ceramics.

Results are still in preliminary stage, however the final grain sizes (~150-350nm) of the fully sintered transparent alumina are in accordance with the theoretical strain rates calculated according to creep parameters for fine-grained alumina (grain size at 90-95% was determined) and the applied pressure and temperature as in Eq. 1:

$$\dot{\varepsilon} = A\sigma^{n} d^{-p} \exp\left(\frac{-Q}{RT}\right) \tag{1}$$

Where A is a constant, σ - applied stress, n - stress exponent, d - grain size, p - grain size exponent, Q - apparent activation energy, R - gas constant and T - temperature. Dynamic grain growth is given by Eq. 2:

$$\dot{d}_{\varepsilon} = \frac{d_f - d_a}{t} \tag{2}$$

Where $\dot{d}_{\varepsilon}$ is the stress enhanced grain growth, d_f is the final grain size, d_a is the static annealed grain size and t is the time.

A strong correlation was found between the calculated creep strain rates for the final stage of sintering and the final grain size of the sintered ceramics. In addition, the hardness measurements are in agreement with hall-petch relation for nanocrystalline alumina (Fig. 1).

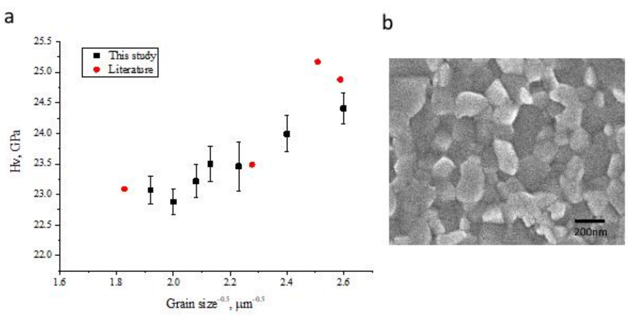

Figure 1. (a) Hardness values for nanocrystalline alumina, red dots are data from literature [2], (b) example of microstructure for alumina sintered at 1000°C under 650 MPa.

4. Conclusions

The grain growth during high-pressure SPS at relatively low temperature is dynamic stress-enhanced and controlled by the creep rate at the final stage of sintering. The nanocrystalline alumina abides strongly to the hall-petch relation.

References

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Keywords
High-pressure spark plasma sintering; alumina; creep rate; stress-enhanced grain growth.