Journal of the European Ceramic Society 20 (2000) 2149–2152

Bending strength and microstructure of Al_2O_3 ceramics densified by spark plasma sintering

L. Gao [a,*], J.S. Hong [a], H. Miyamoto [b], S.D.D.L. Torre [b]

[a] *State Key Lab on High-Performance Ceramics and Superfine Microstructure, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200050, China*

[b] *Technology Research Institute of Osaka Prefecture, Osaka 594-1157, Japan*

Received 27 November 1999; received in revised form 8 March 2000; accepted 13 March 2000

Abstract

Al_2O_3 ceramics were superfast densified using spark plasma sintering (SPS) by heating to a sintering temperature between 1350 and 1700°C at a heating rate of 600°C/min, without holding time, and then fast cooling to 600°C within 3 min. High-density Al_2O_3 ceramics could be achieved at lower sintering temperatures by SPS, as compared with that by conventional pressureless sintering (PLS). The bending strength of Al_2O_3 superfast densified by SPS in the range of sintering temperature between 1400 and 1550°C reached values as high as 800 MPa, almost twice that obtained by the PLS. SEM observations indicated that intragranular fracture was the preponderant fracture mode in these samples, resulting in these excellent bending strength values.

Keywords: Al_2O_3; Mechanical properties; Microstructure-final; Sintering; Spark plasma sintering

1. Introduction

Spark plasma sintering is a new process which provides a means by which ceramic powder can be sintered very rapidly to full density. It is similar to hot-pressing to the extent that graphite dies are used, but the heating is accomplished by spark discharges in voids between the particles, generated by an instantaneous pulsed direct current which is applied through electrodes at the top and bottom punches of the graphite die. Due to these discharges, the particle surface is activated and purified, and a self-heating phenomenon is generated between the particles, as a result of which heat-transfer and mass-transfer can be completed instantaneously.[1–5] Because of these advantages, superfast densification can be accomplished for ceramic powders through the SPS process. In this work, Al_2O_3 ceramics are superfast sintered by the SPS process at different temperatures. It is shown that the sintering time is significantly shortened compared with PLS with a normal holding time of 2 h, but also the sintering temperature can be reduced and the sintered density can be increased. Al_2O_3 ceramics superfast sintered by the SPS technique at temperatures from 1400 to 1550°C yield flexural strength values of ≈ 800 MPa, more than twice that of Al_2O_3 ceramics sintered by the normal PLS process. In fact, the strength reached a maximum of 860 MPa when superfast sintered at 1450°C.

2. Experimental procedure

Commercially available pure α-Al_2O_3 (0.4 μm, >99.9%, UA5105, Showa Denko, Japan) was used in this investigation. It was poured into a 20-mm diameter graphite die and was sintered using an SPS system (Dr Sinter 1020, Sumitomo Coal Mining Co., Ltd.) by heating to the sintering temperature at a heating rate of 600°C/min without a holding time, and cooling in 3 min to temperatures below 600°C. The schematic presentation of the spark plasma sintering apparatus is shown in

* Corresponding author. Tel. +86-21-62512990; fax: +86-21-62513903.

E-mail address: liangaoc@online.sh.cn (L. Gao).

PII: S0955-2219(00)00086-8

Fig. 1. The temperature was measured by means of an optical pyrometer focused on to the sintered sample through a small hole in the die. The pressure used was 40 MPa and this was applied at the start and retained to the sintering temperature. The Al_2O_3 powder was processed by SPS at 1350, 1400, and at 50°C intervals up to 1700°C. Sintered disks obtained by SPS were 20 mm in diameter and 5 mm thick.

All the resulting disks were cut and ground into bars 18 by 4 by 3 mm for strength measurements. The bars were polished to a 600-grit SiC finish on one side and were levelled parallel to their lengths in order to eliminate edge flaws for strength testing. Density measurements were made using the Archimedes technique with an immersion medium of distilled water. The strength was measured with a mechanical machine (SHIMADZU AG-20KNG) using three-point bending test with a span length of 10 mm and a crosshead speed of 0.5 mm/min. Fracture surfaces were examined by scanning electron microscopy (SEM, Philips XL 20).

3. Results and discussion

Fig. 2 shows sintered density as a function of temperature for the Al_2O_3 samples superfast sintered by SPS (holding time: 0 h). The densification curve for the samples sintered by the PLS process (holding time: 2h) is also represented in Fig. 2. The comparison indicates that high-densities were achieved for Al_2O_3 superfast densified by SPS in runs of very short duration (< 3 min). Furthermore, at the same sintering temperature (< 1550°C), the density was higher than that of the Al_2O_3 samples densified by PLS. The densification curves were shifted downwards by about 100°C at the lower sintering temperatures, with the difference gradually decreasing as the temperature was increased, and at 1550°C, almost no difference was found. It is clear that SPS offers significant advantages over PLS for the densification of Al_2O_3. This arises from factors such as the self-heat generation by microscopic discharge between particles, activation of particle surfaces, and the high-speed mass and heat transfer effectively operating in the sintering process.[5]

Fig. 3 shows the bending strength of Al_2O_3 samples superfast sintered by SPS as a function of temperature. High strengths of 800 MPa were obtained by SPS over a broad temperature range from 1400 to 1550°C. At the sintering temperature of 1450°C, the sample reached an average strength of 860 MPa, which was at least twice

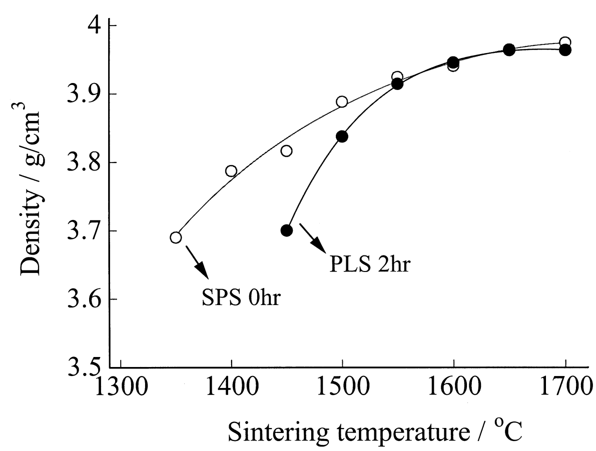

Fig. 2. Relative density as a function of sintering temperature for Al_2O_3 samples prepared by SPS and PLS techniques.

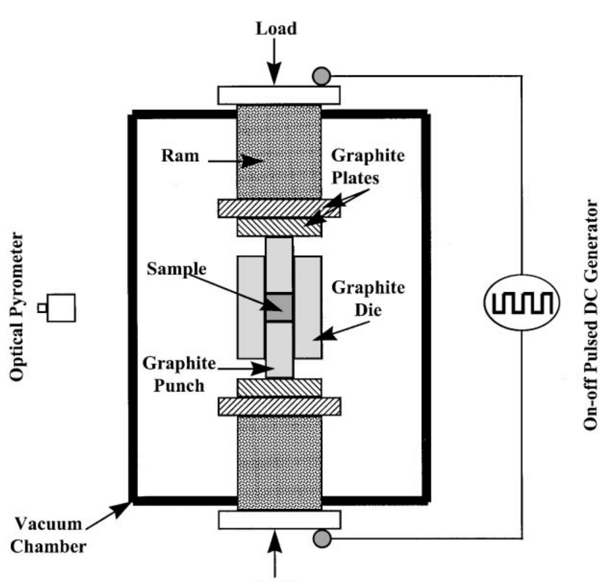

Fig. 1. Schematic presentation of the spark plasma sintering apparatus.

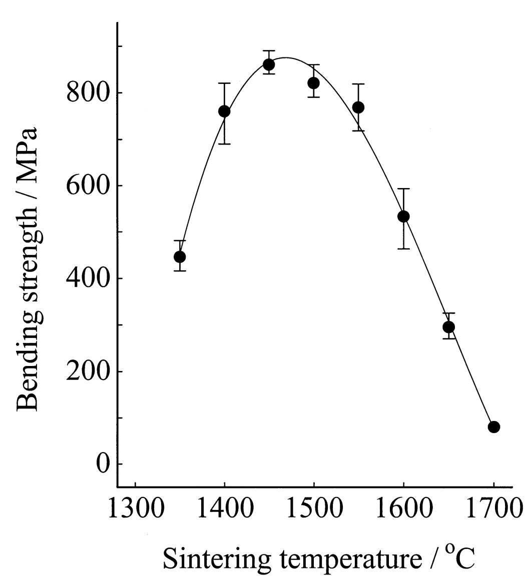

Fig. 3. Bending strength of Al_2O_3 samples superfast sintered by SPS as a function of sintering temperature.

that of the sample obtained by the PLS. This result demonstrates that the strength of Al_2O_3 ceramics can be significantly increased by means of superfast sintering using the SPS technique. For structural ceramics, bending strength is one of crucial mechanical properties.

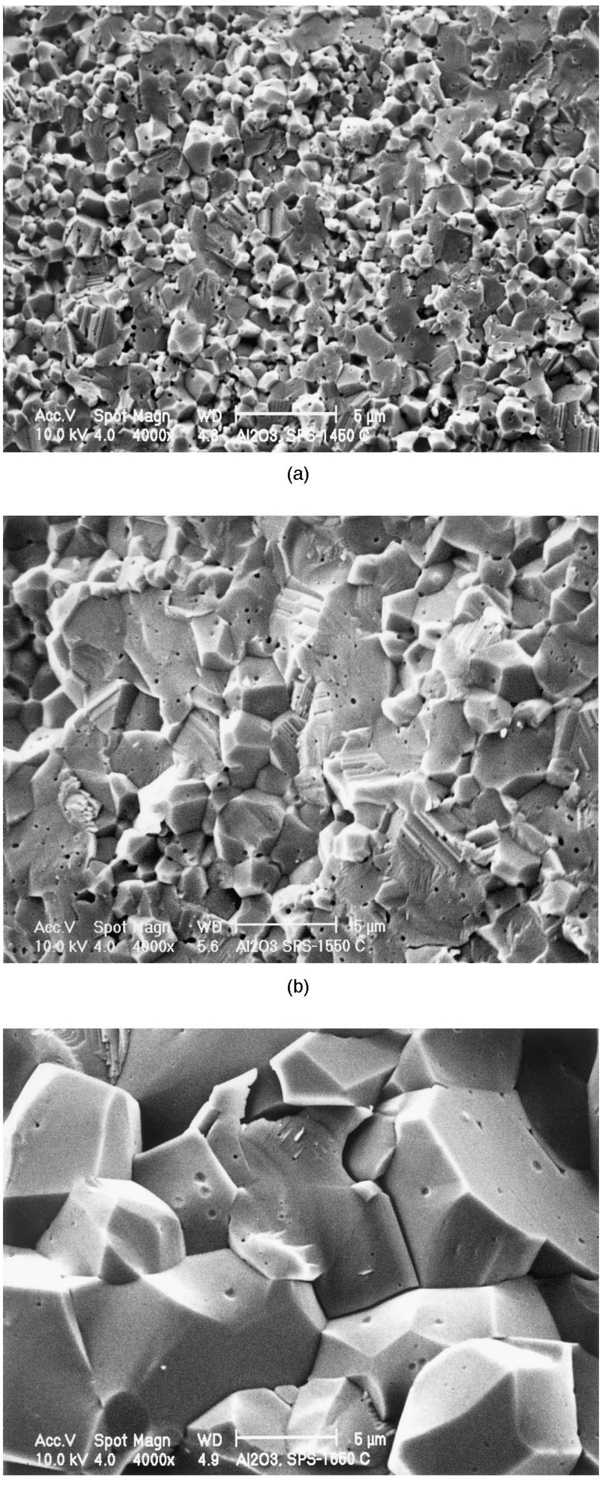

Fig. 4. SEM photographs of fracture surfaces of Al_2O_3 samples superfast sintered by SPS at (a) 1450°C, (b) 1550°C and (c) 1650°C.

Al_2O_3 is a low-priced structural material, but its relatively low strength (typically 400 MPa) limits its choice of applications. These results obtained in this study serve to emphasise that provided the microstructure is optimized by the use of appropriate processing techniques, for example, by retaining small grain size and enhancing grain boundary homogeneity, the strength of Al_2O_3 ceramics can be significantly raised above current values even if the conventional PLS process is used.

It is generally accepted that the denser the ceramic, the higher its strength. However, in this study, the density of the sample superfast sintered by SPS at 1450°C was lower than that of samples superfast sintered by SPS at much higher temperatures, but its strength exhibited the highest value, as shown in Figs. 2 and 3. This can be explained by the superior microstructural characteristics. Fig. 4 shows SEM micrographs of fractured surfaces of samples superfast sintered by SPS at 1450–1550°C. In the case of Fig. 4(a), there exist a number of closed pores, entrapped in Al_2O_3 grains, and the intragranular fracture mode was dominant during fracture, indicating the presence of strong grain-boundaries and leading to a high measured strength. This accounts for the highest strength of this sample, even though its density was relatively lower. In the case of Fig. 4(b), the sintering temperature of 1550°C provided better densification and less porosity in the sample, but the grain size was larger and the intergranular fracture mode became predominant. These microstructural discrepancies lead to a lower strength as compared with the sample sintered at 1450°C. When the sintering temperature was increased to 1650°C [shown in Fig. 4(c)], the density of the sample further increased, but the grains greatly enlarged in size, resulting in a reduced strength.

4. Conclusions

Superfast densification of ceramics can be accomplished using the SPS process with a special processing and sintering procedure. The bending strength of Al_2O_3 ceramics produced by this process was more than twice the value of equivalent samples attained by PLS methods. The samples obtained by SPS have advantages such as a more homogeneous microstructure, a higher density and excellent mechanical properties, showing that SPS is a novel sintering technique with a wide range of potential applications.

References

1. Tokita, M., Trends in advanced SPS spark plasma sintering systems and technology. *J. Soc. Powder Tech. Jpn*, 1993, **30**(11), 790–804.

2. Gao, L. and Miyamoto, H., Spark plasma sintering technology. *J. Inorg. Mater.*, 1997, **12**, 129–133.
3. Gao, L., Hong, J. S., Miyamoto, H. and Torre, S. D. D. L., Superfast densification of oxide ceramics by spark plasma sintering. *J. Inorg. Mater.*, 1998, **13**, 18–22.
4. Perera, D. S., Tokita, M. and Moricca, S., Comparative study of fabrication of Si_3N_4/SiC composites by spark plasma sintering and hot isostatic pressing. *J. Eur. Ceram. Soc.*, 1998, **18**, 401–404.
5. Tamari, N., Tanaka, T., Tanaka, K., Kondoh, I., Kawahara, M. and Tokita, M., Effect of spark plasma sintering on densification and mechanical properties of silicon carbide. *J. Ceram. Soc. Jpn*, 1995, **103**(7), 740–742.