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SPARK PLASMA SINTERING OF ALUMINA: EFFECT OF TEMPERATURE ON MICROHARDNESS

Key words: spark plasma sintering, electrochemical synthesis, supercritical fluid, alumina, microhardness.

The spark plasma sintering (SPS) of two types of α-Al_2O_3 powders (600 nm, 50 nm), synthesized by supercritical fluid and electrochemical methods, was investigated. The evolution of microhardness was studied after sintering the specimens at temperatures ranging from 1300 to 1650 ^{0}C. The microhardness (HV) of obtained bulk samples at room temperature was evaluated by the Vickers indentation technique.

Ключевые слова: искровое плазменное спекание, электрохимический синтез, сверхкритические флюиды, алюминия оксид, микротвердость.

В условиях искрового плазменного спекания исследовалось поведение образцов оксида алюминия, полученных электрохимическим и сверхкритическим флюидным методами. Микротвердость исследуемых объемных образцов, полученных путем спекания при температуре 1300-1650^{o}C, определялась по Виккерсу.

Introduction

Nowadays, Spark Plasma Sintering (SPS) is a modern technique used for characterizing different materials. The SPS process is a pressure-assisted pulsed current sintering process where the densification is highly promoted at lower temperatures compared with the usual processes. This process generally leads to highly dense materials with control of grain structure. Spark plasma sintering (SPS) makes it possible to sinter powders with of density and little grain growth (<1 μm). This technique is able to work at heating rates of hundreds degrees per minute, getting the high temperatures in a short time In particular, SPS was used for the densification of Al_2O_3 specimens [1-5].

Alumina (Al_2O_3) has been extensively considered for its multifunctional applications in electronics, biomedical, chemical, optical, refractory properties [6-8].

SPS powder of alumina is reported to have mechanical properties that differ from those of materials sintered by other methods. D. Pravarthana et al. [9 ,10] found that SPS alumina showed better microhardness than samples obtained by the other sintering methods.

In this research, the effect of temperature on microhardness was determined. The Vickers indentation test is a general method used to distinguish the microhardness of materials. These experiments are easy to perform, need a small quantity of material which is not destructed and can be analyzed many times.

Materials and Methods

Two samples of α-Al_2O_3 powders, obtained by supercritical fluid and electrochemical methods, were used. The first powder synthesized by supercritical method (99.99%, RIKOM Co, Saint Petersburg) had an average particle size of 0.6 μm [11] and the second powder synthesized by electrochemical method had an average particle size of 50 nm [12,13].

The powder was sintered by SPS (Thermal Technology LLA., USA). A graphitic sheet was placed between the punches and the powder, and between the die and the powder for trouble-free removal. Sintering was performed in vacuum (residual cell pressure <0.03 torr). An optical pyrometer, focused on a small hole at the surface of the die, was used to measure the temperature.

For all sintering, heating rate of 200 ^{0}C min^{-1} was used from room temperature to the desired temperature. The cooling rate was fixed at 100 ^{0}C min^{-1} for all samples. The uniaxial pressure was released during cooling for all samples. Samples were sintered in the temperature interval 1300°–1650 °C by step 50 ^{0}C. The holding time at dwell temperature was set at 5 min. Also bulk samples were obtained at applied pressure of 60 MPa and temperature of pressure application of 20 ^{0}C. Fig. 1 and Fig. 2 present the curve of sintering by SPS technique.

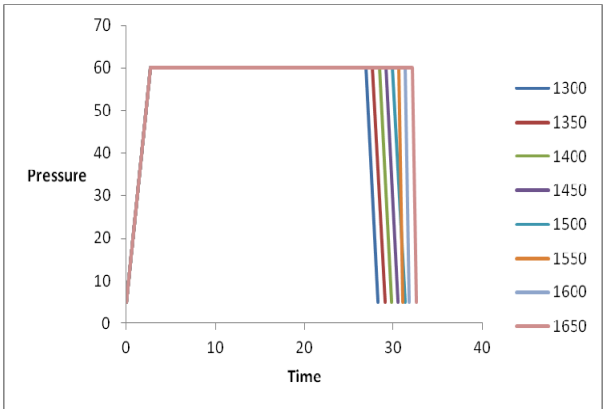

Fig. 1 - Pressure cycles during SPS sintering

These curves show the cycle of the sintering temperature and pressure depending on sintering time. After sintering, the materials were subjected to a study of hardness. Before the hardness measurements, the specimens were carefully polished, by standard diamond polishing techniques, down to a diamond particle size of 1 μm by Buehler machine. The hardness (HV) at room temperature were evaluated by the Vickers indentation technique at a load of 4.903 N and time of 10s according to Jean-Marc Schneider et al [14].

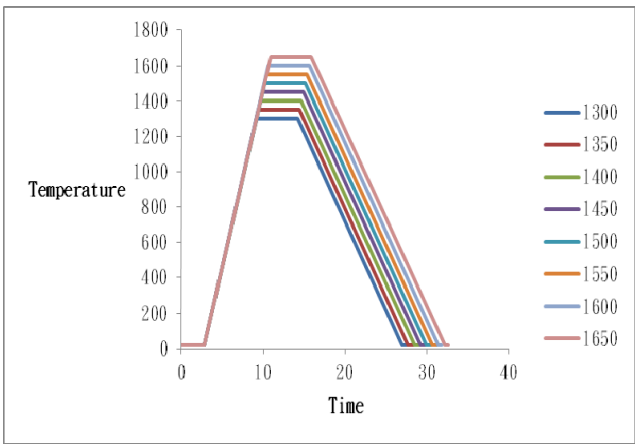

Fig. 2 - Temperature cycles during SPS sintering

Result and Discussion

Fig. 3 shows the effect of sintering temperature on the Vickers hardness (HV) of different powders.

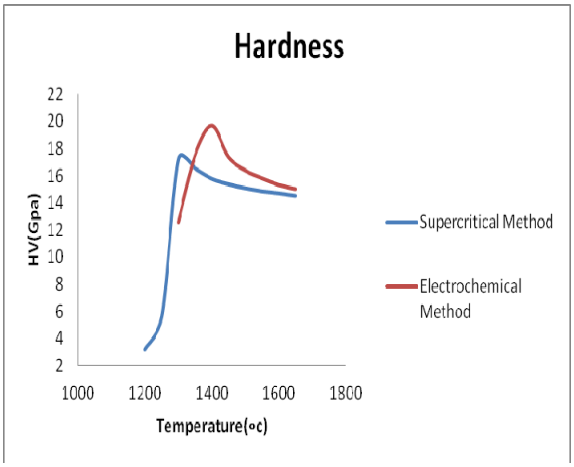

Fig. 3 - Effect of sintering temperature on microhardness

In supercritical method, from 1300 to 1650 ^{0}C, the density of specimens was almost equal to theoretical value and then the Vickers hardness of specimens slightly decreased from 17.3 to 14.5 GPa with increasing sintering temperature from 1300 to 1650 ^{0}C according to the Hall–Petch relation

$$\sigma = \sigma_0 + kd^{-0.5}$$

where σ is the yield stress which mainly determines the bending strength of materials , σ_0 is a material constant that is the starting stress for dislocation movement, or the resistance of the lattice to dislocation motion. k is the strengthening coefficient showing the resistance of dislocation motion, and d is the average grain size.

Based on the Hall–Petch relation, it was easy to learn that the σ of specimen would decrease with the increase of sintering temperature, resulting from the increase of grain size with the increase of sintering temperature. The higher the sintering temperature is, the bigger the size of grains is, the less the interface of grains is, and the less the resistance of dislocation motion is. The hardness of the specimen from powder obtained by electrochemical method, firstly increased in the range of 1300–1400 ^{0}C because of increasing density of specimen and then decreased in the range of 1400–1650 ^{0}C because of grain growth. There are two main factors that effect on the hardness of the specimen, that is, density of specimen and the grain size.

Conclusion

The spark plasma sintering of two types of α-Al_2O_3 powder (600 nm, 50 nm) obtained by supercritical fluid and electrochemical methods was investigated. In the first case, from 1300 to 1650 ^{0}C, Vickers microhardness of the samples slightly decreased from 17.3 to 14.5 GPa. In the second case, Vickers microhardness of the SPS samples firstly increased then decreased with the increase of sintering temperature, providing an optimal value of sintering temperature for maximum of Vickers microhardness close to 19.7 GPa.

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