The Effect of Pre-compaction on Density and Mechanical Properties of Magnetic Pulsed and Sintered Al_2O_3 Bulk

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Abstract

This research reports for the successful consolidation of Al_2O_3 powder with retained ultra-fine structure using MPC and sintering. Measurements in the consolidated Al_2O_3 bulk indicated that hardness, fracture toughenss, and breakdown voltage have been much improved relative to the conventional polycrystalline materials. Finally, optimization of the compaction parameters and sintering conditions will lead to the consolidation of Al_2O_3 nanopowder with higher density and even further enhanced mechanical properties.

Keywords : Al_2O_3 nanopowder, magnetic pulse compaction, sintering

1. Introduction

The nano-powder that hase to be consolidated at high temperature exposure could cause the nanograins to growth resulting in the loss of the superior properties. Unfortunately, however, processing these nanopowders into fully dense, bulk products that retain the original nanoscale grain size has proven to be difficult, owing to a unique combination of problems [1-2] such as high surface area, severe interparticle friction, and high level of chemisorbed gases. Therefore, the consolidation of nanopowder without grain growth is scientifically and technologically important. The most important aspect in the compaction of nanopowders is hot to achieve full density while simultaneously retaining a nanoscale microstructure. Therefore, the process to compact the nanopowder while conserving the nanostructure is strongly required.

Many researchers have discussed several times throughout the research the difficulty of compacting nano-size powders into green bodies that can be easily examined without crumbling. Nanosize α-Al_2O_3 powder is also very difficult to compact, and high pressures are typically required to obtain structurally sound green bodies. The aim of this research is to consolidate nanoalumina powder by magnetic pulsed compaction (MPC) and sintering process, and subsequently characterized them for their microstructure, mechanical properties, and electrical properties. Finally, we are going to apply this result to high power micro-wave window.

2. Experimental and Results

The starting powder was α-Al_2O_3 (purity of 99.8%) with an average powder particle size of 50~200 nm. 1.5 grams of raw Al_2O_3 powder was loaded into a die and punch unit whose outer and inner diameters were 50 and 15 mm, respectively. The Al_2O_3 powder was consolidated with the shape of disc by magnetic pulsed compaction (MPC). The pressure of magnetic pulsed compaction (MPC) varied from 0.5 to 2.1 GPa in the room temperature. In order to improve the density and properties, the starting powder was pre-compacted in a die under 110 MPa, 220 MPa, and 330 MPa, respectively and then each precompacted sample was MPCed at room temperature. The MPCed bulks were sintered at 1,450 ^oC for 3 hrs in an air atmosphere.

The sintered bodies were used for Vickers hardness test and break down voltage testing after polishing. The MPCed and sintered bulk was polished using diamond paste and thermally etched at a temperature 100 ^oC. The apparent density of the bulk was measured by the Archimedes method using water and the values averaged. The relative density was calculated assuming a true density of 3.987 g/cm^3 for α-Al_2O_3.

Fracture surface of sintered bulks was observed with a scanning electron microscope (SEM). Grain size analysis was performed on the digitized SEM photographs using image analysis. Vickers hardness measurements were performed on a Vickers hardness tester using a Vickers indenter with a load of 19.6 N applied for 10 s.

The Al_2O_3 nanopowder particles have a size of approximately 50~200 nm, a smooth surface and elliptical shape. Further, several powder particles seem to consist of

the large particles appeared to be formed by agglomeration of smaller particles. The defects such as cracks and dimple on the surface of MPCed and sintered bulks were not observed.

Fig. 1 shows the density variation of pre-compacted, MPCed and sintered bulks as a function of pre-compaction pressure. It is clear from plot that the density increases with increasing pre-compaction pressure from 0 MPa to 220 MPa and then saturated at 94 % on pre-compaction pressure byond 330 MPa. The highest density of 94 % is achieved in the pre-compacted sample at 220 MPa, while the density of the sample compacted without pre-compaction is 92 %. It means that pre-compaction of powder before MPC improves the final density of sintered bulk.

Fig. 2 shows the SEM micrographs of the pre-compacted, MPCed, and sintered bulk. The relative density of the 220

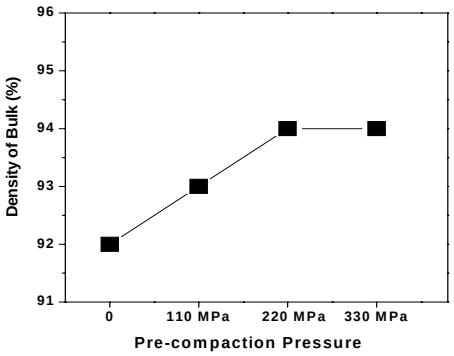

Fig. 1 Variation of density of MPCed and sintered bulks with pre-compaction pressure.

MPa sample was 94 % and most of the resolved porosity appeared to be isolated at grain interstices. The authors noticed that, as the pre-compaction pressure increased, the average grain size decreased and resulting micorstructure was more homogeneous after sintering. The average grain size was determined to be 0.49 μm. Vickers hardness also measured as a function of pre-compaction pressure. With increasing pre-compaction pressure, the hardness (Comercial:Hv 1500, Without precompaction: Hv1567, Precompaction of 110 MPa: Hv1590, Precompaction of 330MPa: Hv 1600) of bulk is increased. This suggests that particle rearrangement during pre-compaction is occurring at lower pressures. The improved hardness with increasing pre-compaction pressure might be associated with the improved density and grain size of bulk. These results clearly indicate that pre-compaction for MPC and sintering is an efficient process to improve the density and Vickers hardness. It is ambiguous to distinguish the contribution to the property enhancement from the grain size effect or from other effects such as pores or strain with the consolidated samples. The indentation fracture method is often employed to characterize the relative fracture toughness and hardness

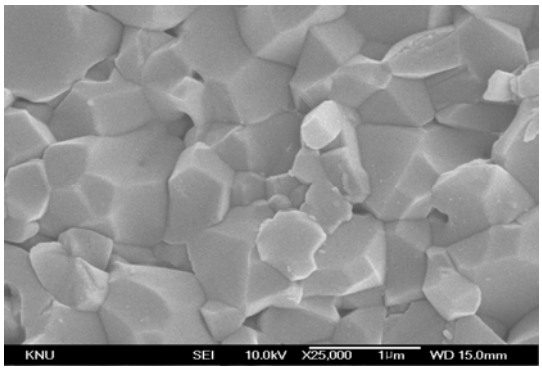

Fig. 2 SEM fracture surface of the MPCed(1.2 GPa) and sintered bulk with pre-compaction at 220 MPa.

of materials. Under a load of 19.6 N, many large cracks caused by an indentation were observed in the MPCed and sintered bulk without pre-compaction, whereas few small cracks were presented in the MPCed and sintered bulk with pre-compaction. This result suggests that the sintered Al_2O_3 bulk with pre-compaction possess higher apparent fracture toughness (Precompaction of 330MPa: 6 MPa $m^{1/2}$) relative to that of the sample without pre-compaction. The breakdown voltage of sintered bulks increased with increasing pre-compaction pressure. The highest breakdown voltage (53 kV/cm) is achieved in pre-compacted specimens at 220 MPa and 330 MPa.

3. Summary

The magnetic pulsed compaction method permits making compacts of Al_2O_3 nanopowders with densities up to 94 %, whereas the density of stationary compaction is 90 %. A further increase in the density of specimens compacted by the magnetic pulsed compaction is not only determined by the amplitude of the pushing pressure but also other important process parameters, such as density prior to compaction and sintering temperature.

The highest density and breakdown voltage in this research is 94 % and 53 kV/cm, respectively. The higher hardness, fracture toughness, and breakdown voltage could be attributed to the crack deflection by a homogeneous distribution and the retention of nanostructure, regardless of the presence of porosities.

4. References

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