Synthesis and Densification of Nanostructured Al_2O_3-(Zro_2+3%Mol Y_2O_3) Bioceramics by High-Frequency Induction Heat Sintering

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Abstract

Nanostructured Alumina – 20 vol% 3YSZ composites powder were synthesized by wet-milling technique. The starting materials were a mixture of Alumina micro-powder and 3YSZ nano-powders. Nano-crystalline grains were obtained after 24 h milling time. The nano-structured powder compacts were then processed to full density at different temperatures by high-frequency induction heat sintering (HFIHS). Effects of temperature on the mechanical and microstructure properties have been studied. Al_2O_3-3YSZ composites with higher mechanical properties and small grain size were successfully developed at relatively low temperatures through this technique.

Keywords: bioceramic, mechanical wet-milling, high frequency induction heating sintering

1. Introduction

One of the main impediments to the use of nanostarter powders is their higher cost and the retainment of the nanostructure in the final product after exposure to elevated temperatures during conventional methods of powder consolidation [1]. It is therefore essential to minimize grain growth through careful control of consolidation parameters, particularly sintering temperature and time. To this end, the novel technique of HFIHS has been shown to be an effective sintering method which can successfully consolidate ceramics and metallic powders to near theoretical density [2-5]. It is similar to hot pressing, which is carried out in a graphite die, but the heating is accomplished by a source of high frequency electricity to drive a large alternating current through a coil. The objective of this study was to synthesize and sinter Alumina–3YSZ powders with fine grain size and homogeneous constituent distribution using HFIHS.

2. Experimental and Results

Alumina powders (mean particle size of 2.91 μm) and 3YSZ (particle size 58-76 nm), were used in this study. The initial powder mixture was composed of 80 vol% alumina and 20 vol% 3YSZ. All powders were milled in a Universal Mill using zirconia balls in polyethylene bottles and was performed at a horizontal rotation velocity of 250 rpm for 24 hrs. After drying the mixed powders were placed in a graphite die and then introduced into the HFIH machine. Details of this apparatus were introduced elsewhere [4, 5]. Fracture toughness is given by the values of K_{IC} using the direct crack measurement method [6].

As shown in Fig. 1 the initial Al_2O_3 powder before milling shows multi-crystalline and agglomerations. Compared with the initial powder, the morphology of the milled powder shows more regular structures, similar to a spherical particle. Thus, milling results in a uniform distribution of Al_2O_3 and 3YSZ powders. It can be concluded that the synthesis of nanosized Al_2O_3–3YSZ has been prepared directly by using wet-milling technique. This technique is remarkable due to the easiness of application.

2.1. Effect of sintering temperature on shrinkage and densifications

In Fig. 2 (b) the specimen attained maximum shrinkage before reaching the maximum sintering temperature, particularly at about 1370 °C. Although, the maximum sintering temperature reached to 1400 °C, there was no more shrinkage after 1370 °C. It is clear from these results that, the maximum sintering temperature with respect to maximum shrinkage is 1370 °C. Fig. 3 shows the density and relative density of Al_2O_3–20vol% 3YSZ as a function of sintering temperature. The relative densities of the specimens increased with increasing the sintering temperature. It is clear that, despite the short dwelling time when the current was applied, the relative density of the sintered samples reached as high as 99 % at 1370 °C, which means that the sintering efficiency of this method is very high.

2.2. Effect of sintering temperature on hardness and fracture toughness

Fig. 4 shows the Vickers hardness and fracture toughness of the Al_2O_3–3YSZ composites as a function of sintering temperature. As was expected, the Vickers hardness increased with increasing the temperature, reached maximum in the samples of 1370 °C due to increasing the relative densities of the specimens with increasing the sintering temperature, while at 1400 °C, hardness slightly decreased. The fracture toughness did not almost vary with the sintering temperature, being on the same level of approximately 5.08 $MPa.m^{1/2}$.

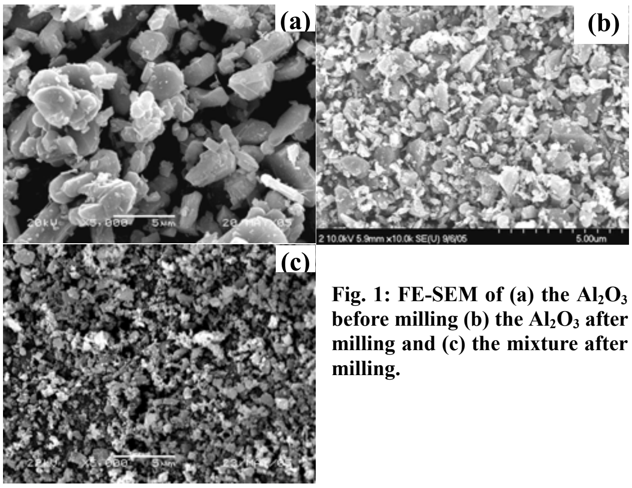

Fig. 1: FE-SEM of (a) the Al_2O_3 before milling (b) the Al_2O_3 after milling and (c) the mixture after milling.

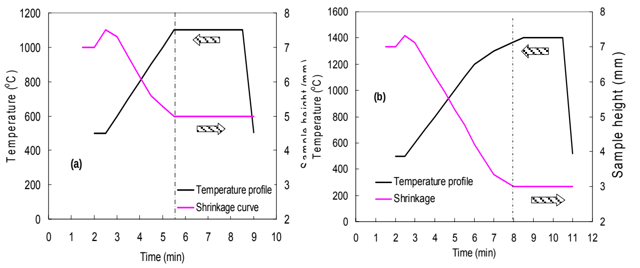

Fig. 2 Variation of temperatures and shrinkage displacements vs. heating time at (a) 1100 (b) 1400 °C maximum sintering temperature.

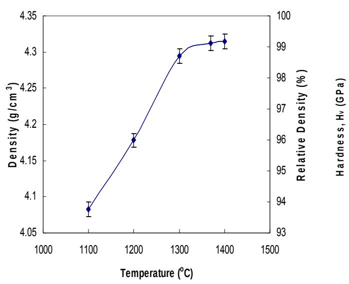

Fig. 3 Sintering temperatures vs. relative density.

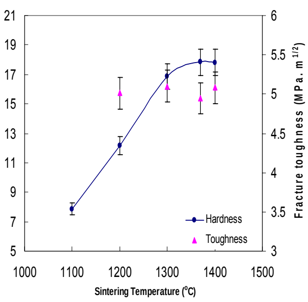

Fig. 4 Sintering temperatures vs. hardness and toughness.

2.3. Effect of sintering temperature on Microstructure

Fig. 6 (a) through (d) shows various SEM micrographs of the Al_2O_3 + 20vol% 3YSZ samples fracture surfaces. The microstructure seems to be like a green compact at 1100 °C because insufficient sintering temperature. Samples sintered at temperature of 1370 °C, showed highly homogeneous microstructures without agglomerates, provided better densification, less porosity and no abnormally grown grains. At temperature 1400 °C, the grain size lightly increased with increasing temperature. For comparison the SEM fracture surfaces of the initial Al_2O_3 powder compacts without milling sintered at 1400 °C is shown in Fig. 6 (f).

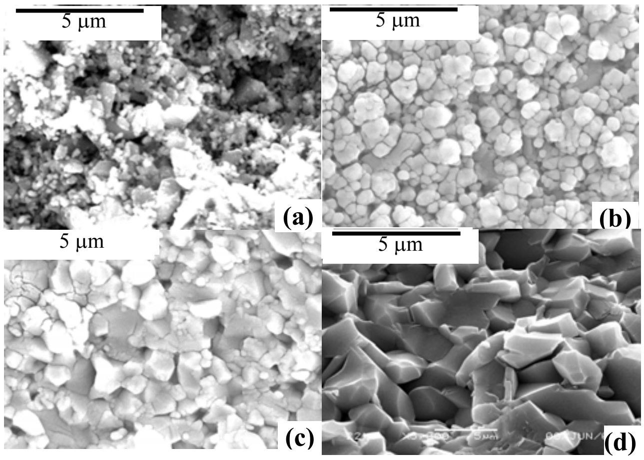

Fig. 6 Effect of sintering temperature on microstructure, (a) 1100, (b) 1370 (c) 1400 and (d) 1400 °C pure Alumina.

3. Summary

The Alumina micro-powder and 3YSZ nano-powders mixtures were synthesized and optimized by using low energy wet-milling technique. This technique is remarkable due to the easiness of application. The mean particle size was decreased by wet-milling after 24 hours of milling. After milling, the samples were superfast consolidated through the technique of high-frequency induction heated sintering (HFIHS). Al_2O_3-3YSZ composites with small grain size, homogeneous microstructure, higher density, good toughness and hardness were successfully developed at relatively low temperatures through this technique.

4. References

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