High-frequency Induction Heating Sintering of Hydroxyapatite-(ZrO_2+3%Mol Y_2O_3) Bioceramics

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Abstract

In this study, hydroxyapatite (HAp) and hydroxyapatite-yttria stabilized zirconia (HAp-3YSZ) with 20 vol. %– (ZrO_2+3 %mol Y_2O_3) nanopowders were consolidated very rapidly to full density by High-frequency induction heat sintering (HFIHS). Effects of temperature and the addition of 3YSZ on the toughness, hardness and microstructure properties have been studied. 3YSZ second phase toughening HAp composites with higher toughness were successfully developed at relatively low temperatures through this technique. Compared with hardness and toughness obtained for pure HAp, the hardness and toughness for HAp-20vol. % 3YSZ were much higher.

Keywords: bioceramics, hydroxyapatite, yttria stabilized zirconia, high frequency induction heating sintering

1. Introduction

Calcium hydroxyapatite (HAp, $Ca_{10}(PO_4)_6(OH)_2$) is one of the most important biocompatible materials with human bones and teeth [1]. However, HAp possesses low mechanical strength and fracture toughness, which is an obstacle to its applications. A suitable method for improving the mechanical properties is based on the synthesis of composites made of HAp and other second phases [2–6]. On the other hand, the use of conventional methods of powder consolidation often results in grain growth in the powder compact. It is therefore essential to minimize grain growth through careful control of consolidation parameters. To this end, the technique of HFIHS has been shown to be an effective sintering method which can successfully consolidate ceramics and metallic powders to near theoretical density [7, 8]. In this study, HAp and HAp–20 vol% 3YSZ composites were sintered at different temperatures by HFIHS. The microstructure, toughness and hardness were investigated.

2. Experimental and Results

HAp powders and 3YSZ (purity 99.9%) were used in this investigation. The initial powder mixture was composed of 80 vol% HAp and 20 vol% 3YSZ. The average particle sizes were 200 nm and 58 nm for the HAp and 3YSZ, respectively. All powders were milled in a Universal Mill using zirconia balls for 24 hrs. The dried mixed powders were placed in a graphite die and then introduced into the HFIHS. Details of this apparatus are described elsewhere [8]. Vickers hardness and toughness were measured by performing indentations at a load of 500 g and a dwell time of 15 s. Fracture toughness is given by the values of K_{IC}. The factor K_{IC} was determined using the direct crack measurement method [9].

Effect of sintering temperature on shrinkage and density. As shown in Fig. 1, the specimen attained minimum height before reaching the maximum sintering temperature, particularly at a temperature of about 1000°C for HAp and 1100°C for HAp-20vol. % 3YSZ. During the holding time, the specimen started to expand again due to decomposition of HAp into other phase TCP [9]. It is clear from these figures that, the maximum sintering temperature with respect to maximum shrinkage is about 1000°C for HAp and 1100°C for HAp-20vol. % 3YSZ.

Fig. 2 shows the XRD patterns of HAp and, HAp-20vol. %3YSZ. There is no phase change for the samples of HAp and HAp-20vol. %3YSZ sintered at 1000 and 1100°C. HAp phase and Tetragonal zirconia phase were the main constituent phases in these two compacts. However, when sintered at 1200°C, HA starts to decompose and a small peak corresponding to TCP is observed.

As shown in Figs. 3 (a) and (b), the relative densities of the specimens increases with increasing sintering temperature, consequantly reaching the maximum at 1000°C for HAp and 1100 for HAp-20vol. % 3YSZ followed by a decrease in

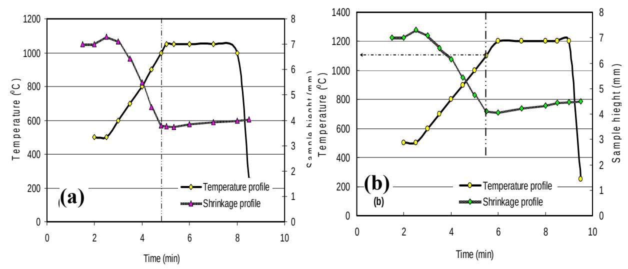

Fig. 1. Variation of temperatures and shrinkage displacements Vs. heating time for, (a) HAp and (b) HAp-3YSZ

density with further increase in sintering temperature. This is can be attributed to the decomposition of HAp into TCP which has low density. It is clear that, despite the short sintering time, the relative density of the sintered samples reached as high as 97.3% at 1000°C for pure HAp and 98.6 % at 1100°C for HAp-20vol.%3YSZ which means that

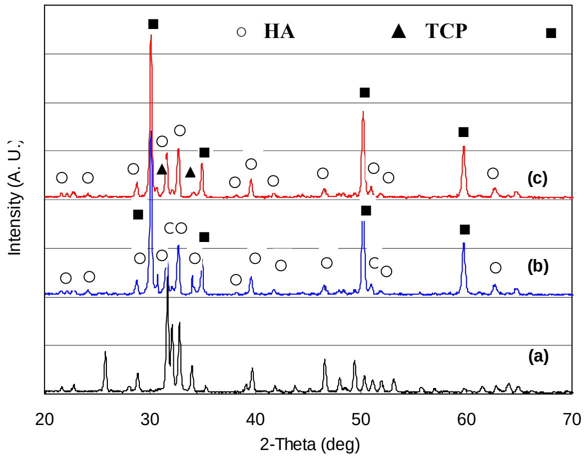

Fig. 2. XRD patterns pure HAp and HAp-20vol. %3YSZ (a) Pure HAp sintered at 1000°C (b) HAp-20vol. %3YSZ sintered at 1100 °C (c) HAp-20vol. %3YSZ sintered at 1200 °C

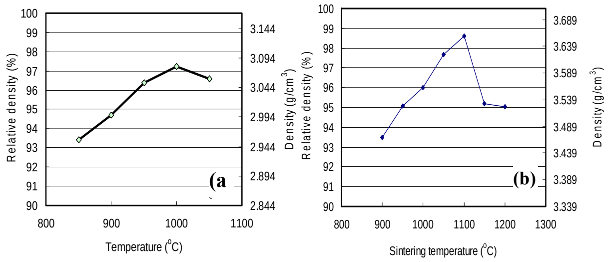

Fig. 3. Effect of sintering temperatures on density and relative density for, (a) HAp and (b) HAp-3YSZ

the sintering efficiency of this method is very high.

Effect of sintering temperature on hardness and toughness. Figs. 4 (a) and (b) show the Vickers hardness and fracture toughness of HAp and HAp–20vol% 3YSZ. The Vickers hardness increases with increasing temperature, consequently reaching a maximum in the dense samples of about 1000°C for HAp and 1100 to 1150°C for HAp-20vol. % 3YSZ, followed by a slight decrease in toughness and hardness with increasing temperature.

Effect of sintering temperature on the microstructure. In the microstructure of the sample sintered at 950°C, there exist a number of closed pores, entrapped in sample grains. Samples sintered at 1100 °C, showed highly homogeneous microstructures without agglomerates, the structure is well densified and the grains are very small. The sintered density value for the sintered compacts was determined to be 98.6 %. At the temperature range from 1150 °C to 1200°C, the grain size increases with increasing temperature and many voids appeare due to thermal decomposition and expansion of the HAp, as shown in Fig. 5 (a) through (c).

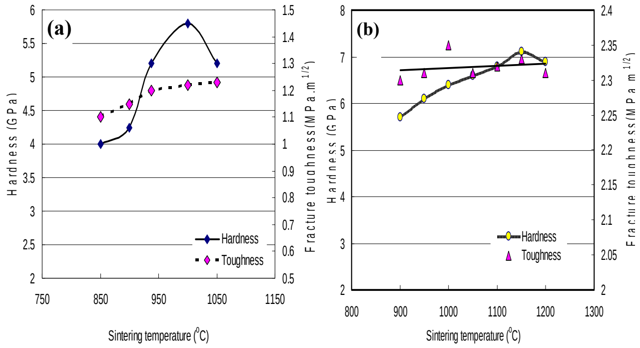

Fig. 4. Effect of sintering temperatures on hardness and fracture toughness for, (a) HAp and (b) HAp-3YSZ

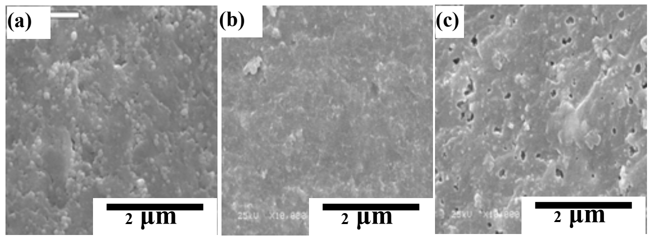

Fig. 5. Effect of sintering temperature on microstructure of HAp-20vol. % 3YSZ (a) 950, (b) 1100, and (c) 1200 °C

Summary

HAp and HAp–20vol% 3YSZ composites were sintered by HFIHS technique at a temperature ranging from 900 to 1200°C. Compared with hardness and toughness obtained for pure HAp, the hardness and toughness for HAp-20vol. % 3YSZ are much higher. HAp-20vol. % 3YSZ composites with small grain size, homogeneous microstructure, higher density, hardness and toughness were successfully developed at relatively low temperatures through HFIHS.

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