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Effect of a novel sintering process on mechanical properties of hydroxyapatite ceramics

Mehdi Mazaheri*, M. Haghighatzadeh, A.M. Zahedi, S.K. Sadrnezhaad

Materials and Energy Research Center, P.O. Box 14155-4777, Tehran, Iran

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ABSTRACT

Two-step sintering (TSS) has been applied, in the present work, to suppress the accelerated grain growth of hydroxyapatite (HA) nanopowder compacts in the last sintering stage. The average grain size of near full dense (>98%) specimens made via conventional sintering (CS) was around 1.7 μm. By using novel two-step sintering, however, the final grain size (∼190 nm) dropped 9 times less than that for the specimens produced by the CS method. Regarding the reduction of the grain size from 1.7 μm (CS method) to 190 nm (TSS method), the fracture toughness of samples increased 95% from 0.98 ± 0.12 to $1.92 \pm 0.20\,\mathrm{MPa\,m^{1/2}}$. Due to the lower second step sintering temperature, the phase analysis shows no HA phase decomposition in TSS method.

1. Introduction

Hydroxyapatite (HA) is one of the most attractive bioceramic materials for human hard tissue implants because of its closed resemblance to bones and teeth [1]. Despite its excellent biocompatibility and efficacious biological fixation to bony tissues, the poor mechanical properties of HA with regards to its brittleness and low fracture toughness restrict its use in load bearing applications [1–3].

A well-known method of improving the mechanical properties of HA specimens is based on the synthesis of composites made of HA and other reinforcing phases. For instance, Chang et al. [4] have shown that zirconia as a second phase develops excellent fatigue resistance against high pressure. Miao et al. [5] have also, reported that HA–zirconia composites show significantly higher mechanical properties (bending strength, microhardness and Young's modules) than those of pure HA. In spite of a better mechanical behavior, such composites cannot be sintered at temperatures same as the sintering of pure HA. Khalil et al. [6] have, for instance, shown that while the sintering temperature for pure HA is around 1000 °C, the specimens made of HA–20 vol.% zirconia must be necessarily heated up to 1100 °C to derive near full dense bodies. On the other hand, the higher sintering temperature of composite-HA, often, causes an extreme grain coarsening and HA phase decomposition leading to a considerable deterioration of mechanical properties [3,6,7].

Another parameter to control the mechanical properties of HA is the final grain size of near full dense samples [8]. Nanostructured HA is expected to have superior mechanical performance in comparison with conventional HA as used for multifunctional implant systems. In addition, the nanometer-sized grains and high volume fraction of grain boundaries in nanostructured HA have been found to increase osteoblast adhesion, proliferation, and mineralization [9,10]. However, fabrication of full dense nanostructured ceramics is difficult [11,12]. The reason lies in the detrimental grain growth after the density of 90% of theoretical density (TD), when the continuous network of pores (in second stage of sintering) disintegrates to the closed ones (in final stage of sintering) and leads to an accelerated grain growth in conventionally sintered specimens [11,13]. The conventional sintering (CS) method is generally incapable of preparing full dense ultra-fine grained ceramics. The fact is that the grain growth and densification are both driven by diffusive mechanisms which result to the simultaneous activation of densification and grain growth in the later stage of sintering [11]. Spark plasma sintering (SPS) [14,15] and hot pressing [16] are also, two promising techniques for production of nanostructured ceramics. Guo et al. [15], have, for instance, spark plasma sintered synthesized HA nanopowders at a low temperature (825 °C) and short time (3 min), while the final grain size became <130 ± 44 nm. Using SPS equipments, Kumar et al. [3] managed to improve the fracture toughness of HA specimens from 0.77 (conventionally sintered) to $1.17\,\mathrm{MPa\,m^{1/2}}$ (spark plasma sintered). In spite of the advantageous consequences of SPS, sophisticated and expensive equipments required, cast a shadow on the technological application of this method.

* Corresponding author. Tel.: +98 912 169 1309; fax: +98 261 4412303.
E-mail addresses: mmazaheri@gmail.com, mazaheri@merc.ac.ir (M. Mazaheri).

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