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Sintering effects of mullite-doping on mechanical properties of bovine hydroxyapatite

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ABSTRACT

In this study, sintering effects on microstructural behavior of bovine derived hydroxyapatite doped with powder mullite are considered in the temperature range between 1000 °C and 1300 °C. Results show that maximum values of both compressive strength and microhardness are achieved in the samples sintered at 1200 °C for all mullite additions of 5, 7.5, 10 and 12.5 wt%. Moreover, above 1000 °C, decomposition of HA and new phase formations such as whitlockite and gehlenite play a major role in both compressive strength and microhardness properties which increase up to 10 wt% mullite reinforcement.

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1. Introduction

Calcium phosphate ceramics (CPCs) are the most popular bioceramics because of promoting bone growth around the implant material [1]. Hydroxyapatite (HA, $Ca_5(PO_4)_3(OH)_2$) is one of the most popular CPCs, which is widely accepted for nearly five decades, and clinically used as a preferred biomaterial for skeletal, dental, cosmetic restorations and their treatments [2]. HA materials are produced synthetically or naturally. For the natural productions, biological HA is obtained from biological sources, such as sheep bone [3], fish waste [4] and marine shells [5–10]. Biological HA accommodates several substitutional trace elements at the Ca^{2+}, PO_4^{3-}, and OH^- sites of its lattice with high importance in biological performance of HA after implantation [2]. Especially, bovine derived HA (BHA) is one of the most preferred HA types. BHA is usually produced by using diluted HCl acid which enables demineralization and freeze-drying of the tissues. Even strict regulations are applied, some deadly high priority diseases (bovine spongiform encephalopathy) can survive even after all controlled processes. And, high temperature calcination performed at 850 °C, prevents those diseases to occur. Actually, this production method is very economic when compared with traditional HA production methods [11–13].

In mechanical point of view, the applications of HA bioceramics are limited to non-load bearing implants because of its poor mechanical properties and poor reliability [14]. It has been investigated that load resistant HA bioceramics reinforced with other second-phase ceramic materials with moderate tolerable compressive strengths powders (for instance, glass, zirconia, titania, magnesium, alumina or titanium) [14–16].

Mullite is a rare silicate mineral of post-clay genesis. It can form two stoichiometric forms $3Al_2O_32SiO_2$ or $2Al_2O_3SiO_2$ [17]. It is a solid solution phase of alumina and silica commonly found in ceramics. Being the only stable intermediate phase in the $Al_2O_3 - SiO_2$ system at atmospheric pressure, mullite is one of the most important ceramic materials. Mullite's temperature stability and refractory nature are superior to corundum's in certain high-temperature structural applications [18]. There are some limited HA-mullite composite applications in recent studies. Nath et al. consider synthetically derived HA with 10-20-30 wt% mullite with respect to nanoindentation technique. Their results reveal lower hardness of 3–4 GPa than expected for HA–mullite composites not conforming with mullite content; whereas pure HA and mullite exhibit higher hardness values of ~4.5 GPa and ~9 GPa, respectively. Considerable decrease in modulus of elasticity (E) with mullite addition is also noticed. The composites have E of ~80 GPa, whereas higher values of ~125 GPa and 230 GPa are recorded for pure HA and pure mullite, respectively [19]. Nath et al. also conduct some in-vivo studies with 20 wt% mullite added and then sintered HA-mullite

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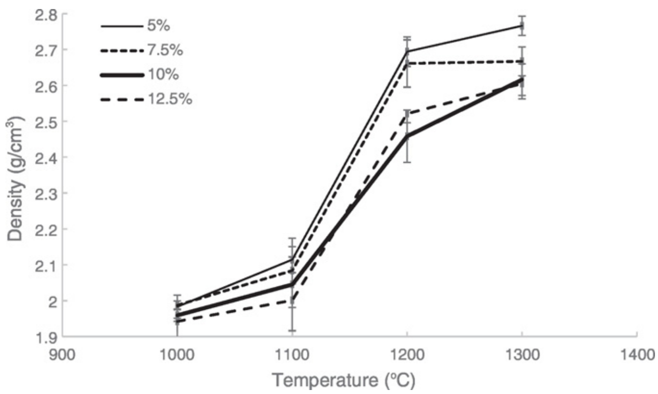

Fig. 1. Density results of mullite reinforced composites for 5, 7.5, 10 and 12.5 wt%.

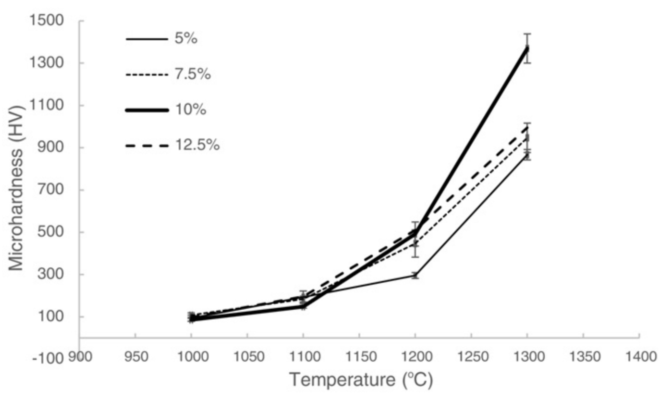

Fig. 2. Microhardness results of mullite reinforced composites for 5, 7.5, 10 and 12.5 wt%.

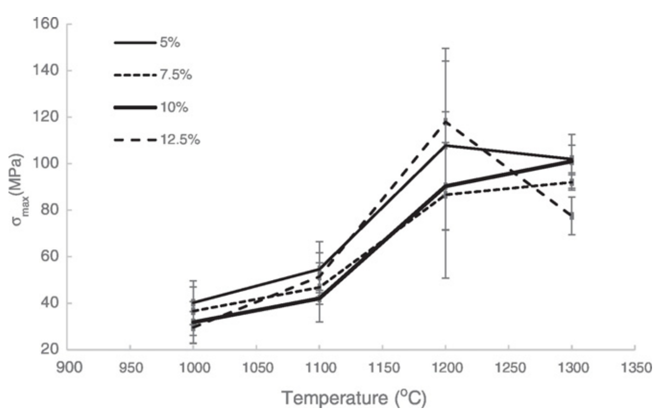

Fig. 3. Compressive strength of mullite reinforced composites for 5, 7.5, 10 and 12.5 wt%.

samples. They observe presence of new bone formation without any noticeable inflammation and minimal collagen at the interface, confirming the suitability of the HA-mullite [20]. In more recent study, Nath et al. reach maximum compressive strength of ~400 MPa (30 wt% mullite mixed in HA and sintered at 1350 °C for 2 h without providing any details on the compressive strength measurements) [21]. It is also reported that the best blend of HA and mullite is 10 wt% [22]. Nevertheless, recent studies say that there may be very few information about the mechanical properties of BHA-mullite composites for orthopaedic and dental graft applications.

The aim of this study is to investigate the microstructural and mechanical properties of BHA-mullite composites by adding 5, 7.5, 10 and 12.5 wt% mullite into the BHA structure respectively.

2. Materials and methods

BHA is prepared from bovine bones calcined at 850 °C [23]. The calcinated BHA powder is grinded and sieved to a scale of 100 μm particles size. After sieving, the BHA fine powder is mixed with 5, 7.5, 10 and 12.5 wt% mullite powder and ground using conventional ball milling for ~4 h. Then, the mixture is dried and pressed forming pellets with a uniaxial cold press at 350 MPa. The dimensions of composite pellets are 6 mm in diameter and 12 mm in height according to the British Standard of BS7253 [24]. Finally, the samples are sintered at temperatures 1000, 1100, 1200 and 1300 °C for 4 h with a ramp rate 5 °C/min [13]. Regarding to testing procedures of density, microhardness and compression, six samples of each group are taken for measurements. Density of the sintered samples is determined by the Archimedes method. A universal tensile testing machine (Devotrans FU 50kN, Turkey) is used for compression tests under a loading rate of 3 mm/min. Vickers microhardness measurements (HV) are conducted under 200 g load and 20 s of dwell time (Shimadzu HMV-2, Japan). Ten HV measurements are obtained from each of the samples. X-ray diffraction studies are performed using a vertical diffractometer (Bruker Instruments, Darmstadt, Germany). Micro-image analysis is carried out using a scanning electron microscope (JSM 7000F, JEOL Ltd., Japan).

3. Results

3.1. Mechanical analysis

Fig. 1 indicates that (i) the highest density value is determined as 2.766 g/cm^3 at 1300 °C for 5 wt% mullite addition into BHA, (ii) the lowest value is 1.942 g/cm^3 with 12.5 wt% mullite addition for 1000 °C sintering. The Vickers microhardness results are given in Fig. 2. Maximum value for HV is obtained as 1369.4 HV for pellets containing 10 wt% mullite sintered at 1300 °C whereas minimum

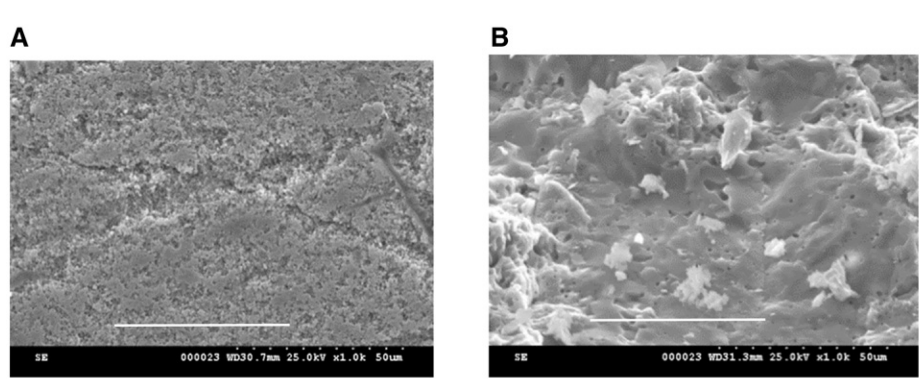

Fig. 4. SEM images of BHA-mullite 5 wt% sintered at A: 1000 °C and B: 1300 °C.

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