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Mechanical and Microstructural Properties of Sheep Hydroxyapatite (SHA)–Niobium Oxide Composites

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The aim of this study is to produce and to investigate the mechanical and microstructural properties of composite materials made of hydroxyapatite, obtained from natural sheep bone and niobium oxide (5 and 10 wt%). Sheep hydroxyapatite (SHA) matrix was reinforced with 5 and 10 wt% of niobium (Nb) oxide powder. The calcinated SHA was ball milled separately with 5% and 10% niobium oxide for 4 h. The samples were subjected to sintering at different temperatures between 1000 °C and 1300 °C. The mechanical properties were determined by measuring compression strength and Vickers microhardness (HV). X-ray diffraction and scanning electron microscopy studies were carried out to analyze the microstructure. With increasing sintering temperature, mechanical properties of composites increased. The SHA-composites with 10 wt% niobium oxide addition had better mechanical properties at all sintering temperatures. The highest mechanical properties were obtained in SHA–10 wt% niobium oxide composite sintered at 1300 °C. Adding of niobium oxide to SHA could be a valuable method to produce rigid and high load carrying ability HA composite which is suitable for orthopedic applications.

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1. Introduction

Hydroxyapatite (HA, $Ca_{10}(PO_4)_6(OH)_2$) is one of the most widely used biomaterials for reconstruction of the skeleton and dental tissues. HA is a nontoxic and biocompatible material which could be used with bone tissues. It is usually used as an implant material both in its bulk form at grafting areas and as a thin biocompatible coating on metal implant surfaces to promote the osseointegration process [1]. But it has poor mechanical properties especially in wet environments. To improve mechanical reliability of HA-ceramics, i.e. to increase their fracture toughness, incorporation of metallic materials, ceramic oxides, whiskers, or fibers, have been strongly suggested [2, 3].

HA bioceramics can be obtained synthetically or naturally. Synthetic HA are reliable but their production is usually complicate, time consuming and expensive. Natural bioceramics can be obtained on economic production, but they can potentially bear some unwanted fatal diseases, such as human immunodeficiency virus (HIV), or bovine spongiform encephalopathy (BSE) [4, 5]. Safety issues about different infection sources from different HA sources were discussed in some recent papers [1, 5]. Ozyegin et al. had reported that high temperature calcination (850 °C) of HA structures could prevent disease transmission [1].

Niobium was first discovered in 1801 [6]. So far, in the literature there were not so much papers about addition of Nb oxide to HA structure or use as a biocompatible material. Tamai et al. and Fathi et al. had reported that Nb oxide addition to HA could be used as an effective bone repair material [7, 8]. Da Silva et al. had used also Nb oxide for other various biomedical applications [8, 9]. Fathi et al. and Tamai et al. had reported that Nb oxides were promoting the calcification process of human osteoblasts. Salman et al.'s cell culture study of Nb oxide addition to bovine derived HA (BHA) was supporting this event as Fathi and Tamai had reported before [10].

The applications of pure HA are limited to non-load bearing implants due to poor mechanical properties of HA [5]. This study was carried out in order to increase the mechanical properties of SHA via the incorporation of Nb oxide (with 5 and 10 wt% addition) in the form of SHA–Nb oxide composite materials.

2. Materials and methods

The SHA used in this study was prepared from calcinated sheep bones. Firstly, fresh cut femurs were deproteinized with NaOH and after reirrigation the samples were subjected to heat treatment (750 °C). Then calcinated sheep bones were wet ball milled for 24 h and they were dried at the drying oven. Sheep hydroxyapatite powder was mixed with (separately) 5 and 10 wt% niobium oxide powder for 4 h. The samples were prepared according to a British Standard for compression tests (BS 7253) [11]. The powder portions were pressed at 350 MPa between hardened steel dies. Pressed samples were subjected to sintering at different temperatures between 1000 °C and 1300 °C (with the heating rate of $+5\,^{\circ}\mathrm{C}\,\mathrm{min}^{-1}$) for 4 h. Compression strength, the Vickers microhardness, as well as density were measured. SEM and X-ray diffraction studies were also conducted. The compression tests were done with a universal test apparatus, at the crosshead speed of 3 mm/min. Microhardness values were determined under 200 g load. Scanning electron microscopy (SEM) images were taken with scan-

ning electron microscope (HITACHI TM-1000 Tabletop Microscope).

3. Results

Table summarizes the experimental results of density, compression strength and the Vickers microhardness of the samples sintered at different temperatures. The mean density, compression strength, and the Vickers microhardness values of SHA–niobium oxide composites increase with increasing sintering temperature, as seen in Table.

TABLE

Influence of niobium oxide content and sintering temperature on density, compression strength and Vickers microhardness of composites made of niobium oxide and sheep hydroxyapatite (SHA).

Temperature [°C]	Density [g/cm^3]		Compression strength [MPa]		Vickers microhardness [HV]	
	5 wt%	10 wt%	5 wt%	10 wt%	5 wt%	10 wt%
1000	2.13	2.19	31	58	52	89
1100	2.21	2.26	39	62	84	107
1200	2.44	2.55	59	72	163	183
1300	2.64	2.66	80	88	214	298

Figure 1 presents the Vickers microhardness and compression strengths of SHA–niobium oxide composites at the different sintering temperatures. The mechanical properties of composites increased with increasing sintering temperature and increasing Nb oxide content. The highest mechanical properties and the highest density were obtained in SHA–10 wt% niobium oxide composite sintered at 1300 °C.

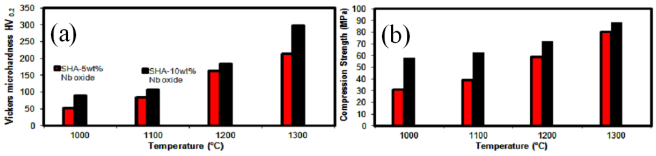

Fig. 1. (a) Vickers microhardness, (b) compression strength of SHA–niobium oxide composites at different sintering temperatures.

The microstructures of SHA–niobium oxide composites sintered at different temperatures are given in Fig. 2. It is clearly seen that similar microstructures were observed in composites sintered at 1000 °C (Fig. 2a,b) and at 1300 °C (Fig. 2c,d), but there were more white coloured phases which were determined as calcium niobium oxide at 1300 °C.

XRD patterns of the studied SHA–Nb oxide composites, sintered at 1300 °C, given in Fig. 3 showed that the present phases in both 5 and 10 wt% Nb oxide containing HA composites are: hydroxyapatite, calcium phosphate, niobium oxide, calcium niobium oxide, and whitlockite. Although both Nb oxide containing HA composites showed the same phases, there were sharper and more evident calcium niobium oxides and hydroxyapatite peaks in the XRD pattern of SHA–10 wt% Nb oxide composite. The best mechanical properties obtained in SHA–10 wt% Nb oxide composite sintered at 1300 °C, may be the result of its highest density and the present phases.

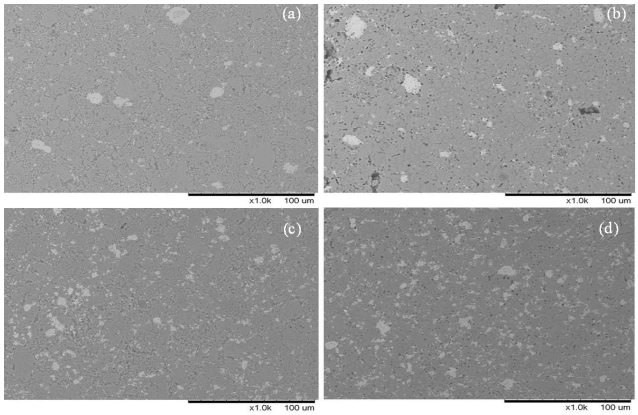

Fig. 2. Microstructures of SHA–niobium oxide (5 and 10 wt%) composites sintered at different temperatures. (a) SHA–5 wt% niobium oxide at 1000 °C, (b) SHA–10 wt% niobium oxide at 1000 °C, (c) SHA–5 wt% niobium oxide at 1300 °C, (d) SHA–10 wt% niobium oxide at 1300 °C.

4. Conclusions

The mechanical properties and density of SHA–Nb oxide (5 and 10 wt%) composites increased with increasing sintering temperature and increasing Nb oxide content. The highest mechanical properties and the highest den-

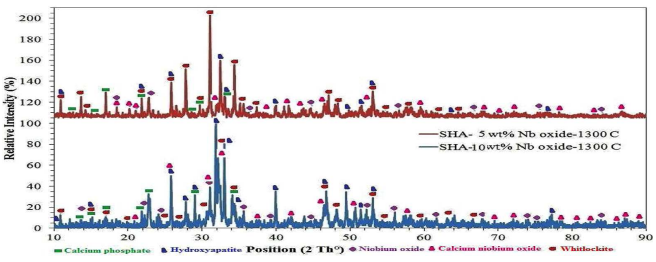

Fig. 3. X-ray diffraction studies of 5 and 10 wt% Nb oxide added SHA–Nb oxide composites, sintered at 1300 °C.

sity were obtained in SHA–10 wt% niobium oxide composite sintered at 1300 °C. According to the results of this study, SHA–Nb oxide composites could be regarded as a good bone substitute for partially load bearing areas in orthopedic applications. Biocompatibility tests of these composites are in progress.

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