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Effect of the addition ZrO_2–Al_2O_3 on nanocrystalline hydroxyapatite bending strength and fracture toughness

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Abstract

Nanocrystalline hydroxyapatite powder has been synthesized from a $Ca(NO_3)_2{\cdot}4H_2O$ and $(NH_4)_2HPO_4$ solution by the precipitation method. In the next step we prepared ZrO_2–Al_2O_3 powder. After preparation, the powder was dried at 80 °C and calcined at 1200 °C for 1 h. Various amounts (HAP–15 wt% ZA, HAP–30 wt% ZA) of powder were mixed with the hydroxyapatite by ball milling. The powder mixtures were pressed and sintered at 1000 °C, 1100 °C and 1200 °C for 1 h. In order to study the structural evolution, X-ray diffraction (XRD) was used. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) were used to estimate the particle size of the powder and observe fracture surfaces. Results show that the bending strength of pressed nanocrystalline HAP was improved significantly by the addition 15 wt% of ZrO_2–Al_2O_3 powders at 1200 °C, but the fracture toughness was not changed, however when 30 wt% of ZA powders were added to nanocrystalline HAP, the bending strength and fracture toughness of the specimens decreased at all sintering temperature.

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Keywords: Nanocrystalline hydroxyapatite; Zirconia–Alumina (ZA); Bending strength; Fracture toughness

1. Introduction

Hydroxyapatite (HAP: $Ca_{10}(PO_4)_6(OH)_2$) has attracted much attention as a substitute material for damaged teeth or bones over the past several decades because of its crystallographical and chemical similarity with various calcified tissues of vertebrates [1–3]. The principal limitation of this material is that it is brittle and weak, which restricts the clinical orthopedic and dental applications [4,5].

There are several approaches for the improvement in mechanical properties of the HAP. One is to fabricate HAP reinforced with other ceramics. This approach has attracted much attention since the successful development of ceramic–matrix composite materials [6].

To be effective as a reinforcing agent for a ceramic–matrix composite material, the following conditions should be satisfied. First, the strength of the second phase must be higher than that of the matrix. Second, the interfacial strength between the matrix and the second phase should be neither too weak nor too strong. For an appropriate interfacial strength, no excessive reaction should occur between the matrix and second phase. In addition, the coefficient of thermal expansion (CTE) of the second phase should not differ too much from that of the matrix [7]. In the case of biomaterials, the biocompatibility of the reinforcing agent is another important factor that should be considered [8].

Zirconia has been commonly used as reinforcement for many ceramics because of its high strength and fracture toughness [9,10]. Bioinertness is another merit of the ZrO_2 [11]. However, extensive reaction between the HAP and the ZrO_2 to from TCP and fully stabilized ZrO_2 is a serious disadvantage of this approach [12,13].

Alumina, which is also classified as a bioinert material, has been widely investigated as a reinforcing agent for HAP [14]. Therefore, it is desirable to combine the advantages of both materials as reinforcements for the HAP: the excellent mechanical properties of ZrO_2 and the chemical inertness of Al_2O_3 with respect to HAP. ZrO_2–Al_2O_3 is one possible approach.

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In this work, the precipitation method has been adapted to synthesize nanocrystalline HAP and we investigated the effects calcined of powder of ZrO_2–Al_2O_3 on the mechanical properties of nanocrystalline HAP powder. The powder was done by a colloidal process utilizing electrostatic force between the ZrO_2-sol and the Al_2O_3 precursor, AlOOH. The morphology of nanocrystalline HAP powder and final powder of ZrO_2–Al_2O_3 after being calcined were observed by TEM. After densification, the mechanical properties, the relative proportion of TCP formed, the microstructures of the composite, and the fracture surface were observed.

2. Experimental

Nanocrystalline hydroxyapatite compounds were prepared by a solution-precipitation method using $Ca(NO_3)_2 \cdot 4H_2O$ (Analar No. 10305) and $(NH_4)_2HPO_4$ (Merck No. 1205) as starting materials and ammonia solution as agents for pH adjustment. A suspension of 0.24 M $Ca(NO_3)_2 \cdot 4H_2O$ was vigorously stirred and its temperature was maintained at 25 °C. A solution of 0.29 M $(NH_4)_2HPO_4$ was slowly added dropwise to the $Ca(NO_3)_2 \cdot 4H_2O$ solution. In all experiments the pH of $Ca(NO_3)_2 \cdot 4H_2O$ solution by ammonia solution was 11. The precipitin HAP was removed from solution by the centrifuge method at a rotation speed of 3000 rpm. The resulting powder was dried at 100 °C.

In the next step, $AlCl_3 \cdot 6H_2O$ (Merck No. 1084) and $ZrOCl_2 \cdot 8H_2O$ (Merck No. 8917) were used as the starting materials for preparation of ZrO_2–Al_2O_3. The pH of the suspensions was controlled by using 0.01N HCl solution as an acid. First, the optimal pH required for change of aluminum chloride to a gel containing bohmite (AlOOH) was estimated because bohmite is generated by hydrolyzing aluminum chloride in an acidic medium. The sol containing zirconium hydroxide from zirconium chloride (which was prepared in an acid medium by hydrolyzing) was added to alumina base gel. The resulting mixture was heated for 12 h in a drier at the temperature of 80 °C and then calcined at 1200 °C for 1 h. The name of the resulting powder is ZA powder. ZrO_2:Al_2O_3 ratio is 1 in ZA powder. To obtain this ratio, starting materials including $AlCl_3 \cdot 6H_2O$ and $ZrOCl_2 \cdot 8H_2O$ were weighted according to their ratios in Eqs. (1) and (2).

Various amounts of ZA powder were mixed with the nanocrystalline HAP by ball milling in distilled water for 24 h with Al_2O_3 balls as media to break the HAP agglomerates. The powder mixtures were pressed in a steel mold at room temperature with an applied pressure of 500 kg. The dimensions of the pressed sample were 4 mm × 6 mm × 40 mm. Sintering conditions for pure hydroxyapatite specimens was identical to sintering condition for composites specimens that is, sintering in 1000 °C, 1100 °C and 1200 °C for 1 h.

The phase transformation of the ZA powder and pressed bodies were determined with an X-ray diffractometer (Siemens, 30 kV and 25 mA) with Cu Kα radiation (λ = 1.5405 Å).

Transmission electron microscopy (TEM) was used to characterize the particles of HAP and ZrO_2–Al_2O_3. For this purpose, particles were deposited onto Cu grids, which support a ''holey'' carbon film. The particles were deposited onto the support grids by deposition from a dilute suspension in acetone or ethanol. The particle shapes and sizes were characterized by diffraction (amplitude) contrast and, for crystalline materials, by high resolution (phase contrast) imaging.

Bending strengths were measured at room temperature in air with a crosshead speed of 0.5 mm/min by three-point bending test. Test specimens with dimensions of 3.5 mm × 5 mm × 35 mm were cut and machined for bending strength and fracture toughness tests. The entire specimen surface was ground with an 800-grit diamond wheel and the tensile surface was polished with diamond slurries. The fracture toughness was determined by the indentation-strength method using an applied load of 500 N. For each set of conditions, at least five specimens were tested for both the strength and the fracture toughness measurements.

The fracture surfaces were observed with a Cambridge scanning electron microscope (SEM) operating at 25 kV.

3. Results and discussion

3.1. Synthesis nanocrystalline hydroxyapatite powder

The XRD peaks for hydroxyapatite powder after drying are illustrated in Fig. 1(a). The crystal phase of precipitated HAP powder before calcinations and after drying at 100 °C was all HAP. It contains no other crystalline phase other than HAP. The broad patterns around at (2 1 1) and (0 0 2) indicate that the crystallites are very tiny in nature with much atomic oscillations. TEM micrographs of the HAP powders before sintering and after drying are seen in Fig. 2. The microstructure of the HAP particles after drying is observed be almost like needle shape, with particle size in the range 8–20 nm. Results show that HAP nanocrystalline can successfully be produced by precipitation technique from raw materials.

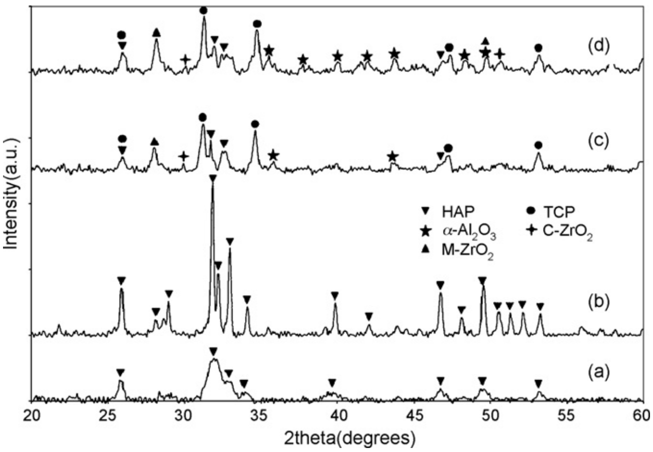

Fig. 1. XRD patterns of (a) nanocrystalline hydroxyapatite powders after drying at 100 °C, (b) nanocrystalline hydroxyapatite powders after calcinated at 1200 °C and (c), (d) HAP with 15 wt% ZA and HAP with 30 wt% ZA, respectively, after pressing and sintering.

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