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Short communication

C-fibre reinforced hydroxyapatite bioceramics

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Abstract

A novel hydroxyapatite bioceramic composite with C-fibre as reinforcement was fabricated by hot pressing. With the increase of C-fibre content, the bending strength and fracture toughness of composites increased, attaining a peak value of 119.9 and 1.22 MPa.m$^{1/2}$ at 0.3 wt.% and 0.1 wt.% C-fibre addition respectively. After reaching the peak values, both bending strength and fracture toughness decreased.

Keywords: C-fibre; Ceramic-matrix composites; Fracture toughness; Bending strength

1. Introduction

Hydroxyapatite [HAp, chemical formula $Ca_{10}(PO_4)_6(OH)_2$] is a material of interest for artificial teeth or bones due to excellent biocompatibility and bioactivity. Unfortunately, the mechanical reliability and slow crack growth resistance of the pure HAp ceramic is low, and its medical applications are limited to small unloaded implants, powders, coatings and low-loaded porous implants. In order to improve the mechanical properties of HAp ceramics, many reinforcements, including ceramic particles, intermetallic particles, metal long fibers, partially stabilized zirconia, whiskers, and nano-particles etc. [1–4], have been used. The addition of reinforcements increased the toughness and strength of HAp materials. The introduction of bioinert materials into the HAp matrix, however, may generally lead to a decrease in biocompatibility and bioactivity.

Taking into account the bioactivity and biocompatibility of composites, we have chosen C-fibre as reinforcement to reinforce HAp bioceramics because the carbon materials are highly biocompatible [5].

2. Experimental procedure

HAp powder was prepared with wet-chemical precipitate method [6], the precipitate was washed by distilled water, dried and calcined at 1200 °C for 2 h. The XRD pattern of the powder is shown in Fig. 1 (A). It can be seen that the major crystal phase is HAp. C-fibre was supplied by Toray Co. LTD (Japan). The tensile strength of the filament was 3920 MPa, the density 1.77 g/cm^3, elongation rate 1.6% and the elastic modulus 235 GPa. Continuous high-strength fibre was cut with scissors to about 10 mm. The chopped C-fibre was soaked in water solution of 33% phosphoric acid for 10 h, then washed with distilled water and dried at 80 °C in a dryer.

The HAp powder and chopped C-fibre were weighted with the addition ratios of C-fibre: 0.0, 0.1, 0.3, 0.6, 0.9 and 1.2 wt.% respectively, then fully mixed with a high-speed blender (5000–8000 rpm). In the final mixture powders, the length of C-fibre was between 0.2 and 0.6 mm. The sintering of all samples was performed by hot pressing at 1080 °C using a graphite die. Nitrogen was used as protective gas. On reaching the hot pressing temperature, an uniaxial pressure of 15 MPa was applied. Both pressure and temperature were held for 30 min.

The sintering specimens were machined with a grit resin bonded diamond wheel and then cut into rectangular bars with a diamond saw. The dimensions of these bars were 4×3×36 mm for measuring the bending strength, and 4×2×36 mm for fracture toughness, with notch width 0.25mm. The strength of the specimens was determined by the three-point bending technique at ambient conditions. The span was 30 mm. The rate of loading was 0.5 mm/min. The fracture toughness was determined by the single-edge-notched-beam (SENB) technique. For each set of both strength and fracture

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toughness measurements, at least six specimens were tested. The phase identification was performed by X-ray powder diffractometry (XRD, D/max-rB, Japan) with Cu Kα radiation. The microstructure of fractured surfaces was observed with scanning electron microscopy (SEM, JXA-840, Japan).

3. Results and discussion

The colors of specimens changed gradually to dark gray with the increase in C-fibre content in the HAp composites.

3.1. Phase compositions

XRD patterns of the pure HAp ceramic and the composite with 0.6 wt.% C-fibre after hot pressing in the same conditions are shown in Fig. 1(B) and (C), respectively. Compared with these profiles, no noticed differences were observed except the intensity of diffraction peaks and no obvious diffraction peaks of carbon were detected, indicating that no new crystalline phase was formed during the sintering by hot pressing.

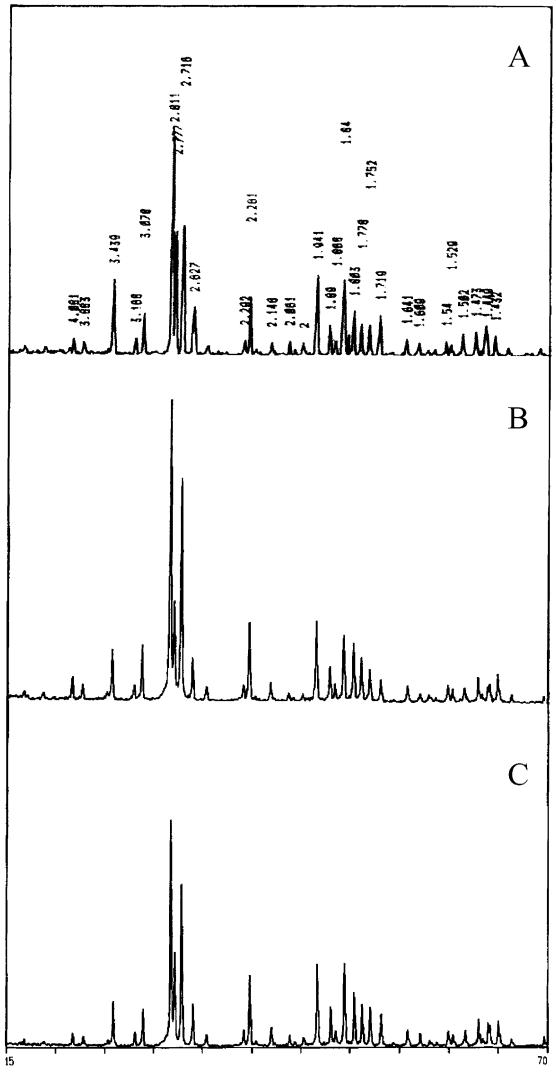

Fig. 1. The XRD patterns of HAp powder (A), pure HAp ceramic (B) and HAp composite with 0.6 wt.% C-fibre (C).

2.2. Mechanical properties

The values of the bending strength and fracture toughness for every composition are the average values of specimens tested. The experimental errors for the bending strength and fracture toughness were calculated to be less than 7 and 6.5%, respectively.

The strength of HAp was improved by the addition of C-fibre, as shown in Fig. 2. The strength of pure HAp was 89.07 MPa in our experimental conditions. When the content of C-fibre was 0.3 wt.%, the strength increased to 119.9 MPa. With further addition of C-fibre, however, the level of increase in strength was reduced, even lower than the strength value of pure HAp, presumably because of the insufficient densification of specimens.

The fracture toughness of pure HAp was less than 1 MPa.m$^{1/2}$, which is well known to be one of the biggest obstacles for wider application of this material [7]. In the present research, the value of fracture toughness of pure HAp was 0.9 MPa.m$^{1/2}$. When the addition

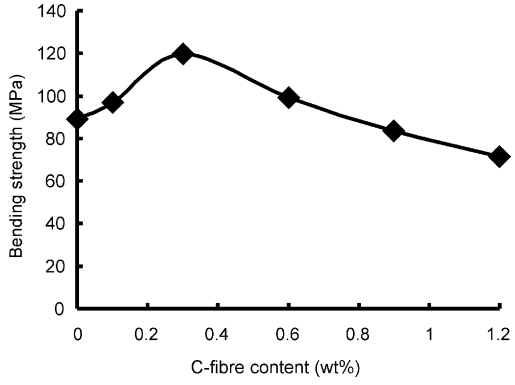

Fig. 2. Bending strength of composites as a function of C-fibre content.

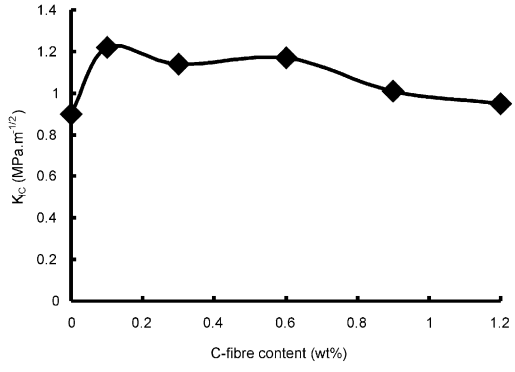

Fig. 3. Fracture toughness of composites as a function of C-fibre content.

amount of C-fibre was 0.1 wt.%, the fracture toughness was 1.22 MPa.m$^{1/2}$. With further addition of C-fibre, the level of increase was reduced, but the fracture toughness values of all composites are higher than those of pure HAp, as shown in Fig. 3.

3.3. Microstructure of the composites

SEM micrographs of the fracture surfaces of samples containing different C-fibres are shown in Fig. 4 (A–F).

A high density of small pores was observed within grains of pure HAp and in the matrix of the composites, as shown in Fig. 4. Moreover, the pores were bigger and more plentiful in the composites, in the samples with more C-fibre, in particular, than those in the pure HAp. The statistical analysis of the SEM showed that the size of the pores in the pure HAp sinter was in the range of 0.1–5 μm, and for the composites it was in the range of 0.1–20 μm. The pores exhibited irregular shapes. More pores within the matrix, however, will degrade the mechanical properties of the composites. The addition of C-fibre led to the formation of more small pores that are presumably produced by the gas phase generated by the burning of C-fibre in very small amount.

The length of C-fibre in the composites was between 200 and 600 μm, the diameter of C-fibre about 8–10 μm.

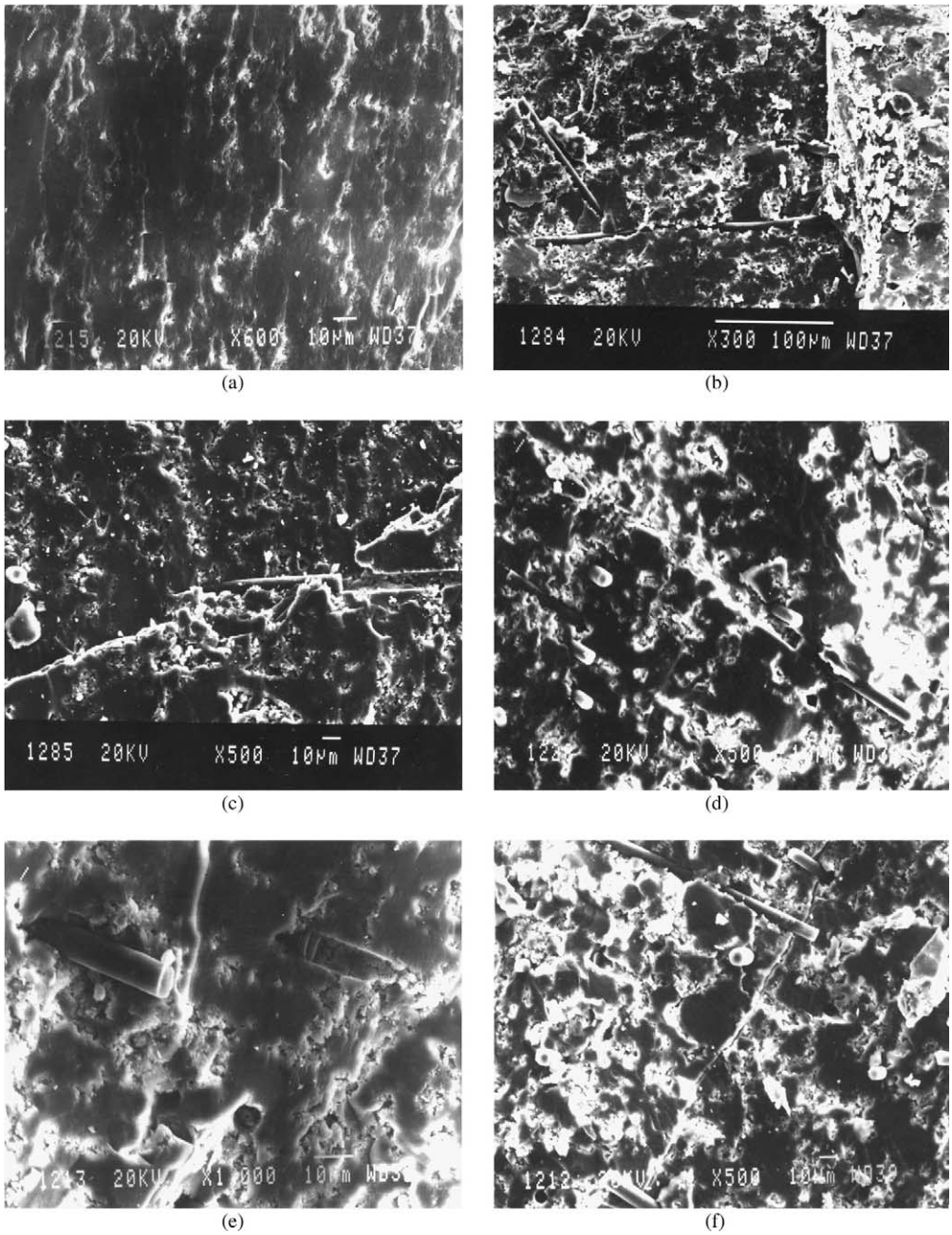

Fig. 4. SEM micrographs of the fracture surfaces. (a) Pure HAp; (b,c). HAp with 0.6 wt.% C-fiber; (d–f). HAp with 0.9 wt.% C-fiber.

Generally speaking, only when the ratio of length/diameter of fibre used as reinforcement is between 40 and 80, the strength of ceramic matrix would be improved [8]. In the present work, the ratio of length/diameter of C-fibre in the composites met this condition. As shown in Fig. 4(b–f), the orientation of C-fibre is random in the composites. We have also observed that the C-fibre distribution in the composites is not very homogeneous. This would be one of the reasons that the mechanical properties of HAp ceramics were limitedly improved in our experiments.

Fig. 4(f) shows the crack of the composite containing 0.9 wt.% C-fibre, the crack propagated nonlinearly by crack deflection and crack bridging also occurred.

In Fig. 4c–f, exposed fractured ends of C-fibre could be clearly observed and holes left behind through extracting of C-fibre were also observed.

Fig. 4(c) and (e) show marks left behind through extracting of C-fibre. They suggest that the bonding between HAp and C-fibre was not strong so that the C-fibre could easily pull out. Extracting process of C-fibre enhanced the mechanical properties of HAp.

We also can observe that there are gaps around some C-fibre filaments from Fig. 4(f), and that the matrixes of composites were not dense enough under the present experimental conditions. The addition of C-fibre increased the porosity level and hindered the densification of HAp composites. This is similar to the investigation result of SiC fibre reinforced Mo_2FeB_2 layered cermet composites by Dhaval Rao et al. [9]. It is possible to further improve the mechanical properties of HAp bioceramics via a suitable treatment method of C-fibre surface and a sintering under higher pressure or in a vacuum.

The increase in the fracture toughness and bending strength are attributed to the synergistic effects of crack deflection, interlocking of the fibers, pullout and crack bridging.

The addition of C-fibre is effective for improving the strength and fracture toughness of HAp at the same time.

4. Conclusion

C-fibre is an effective reinforcement that could both improve the mechanical properties and maintain the biocompatibility and bioactivity of HAp bioceramics. The increased toughness and strength are mainly attributed to crack deflection, interlocking of the fibers, pullout and crack bridging.

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