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Self-joining of zirconia/hydroxyapatite composites using plastic deformation process

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Abstract

A plastic deformation process was demonstrated to self-join 3 mol.% yttria partially stabilized zirconia (3Y-TZP)/hydroxyapatite (HA) composites. The 3Y-TZP/40 vol.% HA composites were fabricated by conventional ceramic processing by cold pressing premixed 3Y-TZP and HA powders into pellets. Densification (≈90%) of composites was achieved by sintering composite powder compacts at 1450 °C for 5 h. Optimum self-joining of 3Y-TZP/40 vol.% HA composites was obtained at 1300 °C for a strain rate of 5×10^{-5}/s. The flow stress during joining was 40 MPa. Microstructural and mechanical characterizations of the joint interface demonstrated that there were no discernible differences between the joint and the composite material away from the interface.

Keywords: Hydroxyapatite; Zirconia; Composite; Joining; Deformation

1. Introduction

Hydroxyapatite (HA) ceramic has been established as one of the most appropriate ceramic materials for biomedical applications [1–3] because of its compatibility with human tissue and long-term chemical durability [1–4]. HA possesses excellent osteconductive properties that help it to bond to human tissue. However, HA has poor mechanical properties (strength and fracture toughness) which limit its use for biomedical applications such as implants [5]. Several approaches including reinforcing HA matrix with second phase materials such as zirconia (ZrO_2) ceramics, polymers, or metal particles have been explored [6,7]. Zirconia is particularly attractive as a reinforcing phase since it is bio-inert and HA/ZrO_2 composites give significantly improved mechanical properties [8]. Furthermore, by creative processing techniques, HA/ZrO_2 composites can be fabricated with controlled porosity and pore structure that promotes implant–tissue bonding by intergrowth.

One of the major challenges is producing complex shapes of HA and its composites that replicate human bone structures. Typical ceramic processing routes allow fabrication of HA/ZrO_2 composites with simple geometric shapes. One approach being considered is to join multiple simple-shaped parts to produce a complex part. In this regard, joining by plastic deformation, as discussed in this paper, presents a novel approach to producing complex-shaped parts from HA and its composites.

Joining of various structural ceramics by a high-temperature plastic deformation process has been demonstrated [9–12]. The joining process requires heating the

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components to elevated temperatures and deforming them under a constant displacement rate. The total strains in the joined parts are typically <10%. Dense joints are formed by interpenetration of grains at the interface as a result of a grain boundary sliding (GBS) mechanism [9–12].

The high-temperature plastic deformation joining process has several advantages in that it (a) requires no surface preparation or interlayer material, (b) has minimal permanent deformation, and (c) occurs at lower temperatures than those required for conventional diffusional bonding [13]. The interface between the bonds formed by plastic deformation is indistinguishable from the bulk material and possesses strengths as high as the unjoined material [12]. Moreover, dissimilar materials can be joined by the plastic deformation process, provided the post-processing residual stresses are small.

Limited studies of the high-temperature deformation behavior of HA have been reported in the literature because the applications for HA are generally at low temperatures. However, high-temperature deformation behavior becomes important if the plastic deformation process is used for fabrication of complex-shaped HA parts. Wakai et al. [14] demonstrated superplastic behavior in fine-grained HA prepared by hot isostatic pressing. They were able to deform HA as much as 150% at 1000 °C in the tensile mode.

Recently Singh et al. [15] conducted a detailed high-temperature deformation study of HA in the temperature range 1175–1275 °C with strain rates ranging from 5×10^{-6}/s to 10^{-4}/s. The grain size of HA studied ranged from 1 to 5 μm [15]. The authors showed that a diffusion-assisted GBS mechanism controlled the high-temperature deformation behavior of HA under the test conditions studied. Furthermore, they demonstrated self-joining of HA by plastic deformation at 1275 °C at a strain rate of 10^{-5}/s, yielding joints with minimal porosity and interfacial microstructures that were identical to the microstructures far from the joint.

High-temperature tensile tests of 3 mol.% Y_2O_3-stabilized tetragonal zirconia (3Y-TZP) have shown that it can superplastically (>100%) deform at 1450 °C [16]. Grain boundary sliding is the primary deformation mode that facilitates the superplastic behavior in 3Y-TZP and is dependent on the impurity levels. Furthermore, 3Y-TZP and its composites with alumina deform by a GBS mechanism that can be exploited to join 3Y-TZP with alumina/zirconia composites [11].

Based on the high-temperature deformation behaviors of monolithic HA and 3Y-TZP, it is reasonable to assume that under appropriate test conditions, 3Y-TZP/HA composites will plastically deform, and thus can be potentially self-joined. The main objectives of this paper are to (a) fabricate 3Y-TZP/HA composites, (b) demonstrate self-joining of 3Y-TZP/HA composites by plastic deformation, and (c) establish the efficacy of the joints by microstructural and mechanical characterizations.

2. Experimental details

2.1. 3Y-TZP/HA composite fabrication

Composite samples of 3Y-TZP/HA were fabricated by premixing HA powder (Cerac Inc., Speciality Inorganics, Milwaukee, WI) with commercial zirconia powder (3 mol.% yttria tetragonal zirconia polycrystals from Tosoh Corp., Japan), such that the ratio of zirconia to HA powders was 60:40 by volume or 74:26 by weight. This powder mixture was ball-milled for 24 h in alcohol with 1–2 ml of Witco Emphos PS-21A as a dispersant. Subsequently, the powder mixture was dried, milled with a mortar and pestle, and sieved through a 325 mesh sieve. The composite powder was cold-pressed in a cylindrical hardened steel die at a pressure of 115 MPa. The green composite bodies were then sintered at 1450 °C for 5 h in air. Furthermore, monolithic HA samples were also sintered with the 3Y-TZP/HA composites to discern any phase changes in HA caused by the higher sintering temperature of the composite samples as compared to the normal sintering temperatures for monolithic HA.

2.2. Specimen characterization

The density of sintered 3Y-TZP/40 vol.% HA samples was determined by the Archimedes method. Sample preparation for microstructural examination involved polishing one of the sample surfaces down to a 0.05-μm finish using an alumina slurry. To reveal grain boundaries, samples were thermally etched at 1200–1250 °C for 0.5 h in air. Subsequently, the microstructure was examined in a Hitachi Model S-4700-II (Tokyo, Japan) field emission scanning electron microscope (FE-SEM). Grain size was measured by the standard linear intercept method [17].

The phases present in the 3Y-TZP/40 vol.% HA composites after sintering were determined by room-temperature X-ray diffraction (XRD) measurements using a Cu $K\alpha_1$ radiation. These measurements were carried out in a Phillips X-ray diffractometer (Model X'PERT, Almelo, The Netherlands) using a step-scan program run at 100 s/° with a step size of 0.01°. The composite specimens were scanned over a 2θ range of 20–75°.

Monolithic HA samples sintered with the 3Y-TZP/HA composites were characterized by XRD to determine the stability of HA phases in the composite after sintering at 1450 °C for 5 h. The specimens were scanned over a 2θ range of 27–37° at similar scan rates as above. This particular range of 2θ was selected since it includes the principal XRD peaks for HA and HA's thermal decomposition products.

2.3. Joining

For joining experiments, right parallelepiped specimens ($\approx 3 \times 3 \times 5$ mm) were cut from as-sintered 3Y-TZP/40 vol.% HA samples using a low speed diamond saw.

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