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Biomimetic apatite coating on Mg-PSZ/Al_2O_3 composites. Effect of the immersion method

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Abstract

A bioactive zirconia/alumina composite for orthopedic applications has been developed. The composite was obtained by uniaxial pressing of powder mixtures. To bioactivate the composite a biomimetic method, consisting in the immersion of samples in SBF and 1.4 SBF, was used. The immersion procedure was performed by two different routes: (i) single immersion (21 days in SBF) and (ii) re-immersion method (7 days in SBF+14 days in 1.4 SBF). No change was observed on the surface of the samples immersed for 21 days in SBF. However, a dense and homogeneous bonelike apatite layer was formed on the ceramic composite by using the re-immersion method. The average value for the thickness of the apatite layer was 20 μm.

Keywords: Bioceramics; Biomimetic process; Simulated body fluid (SBF); Bioactive composites; Zirconia–alumina composites; Apatite coating

1. Introduction

Zirconia has been used as a biomaterial for orthopedic applications due to its excellent biocompatibility and high mechanical properties [1]. Currently, zirconia ceramics are used in the manufacture of femoral heads of total hip replacement prostheses [2]. Despite its advantages, zirconia ceramics present the major problem of undergoing a phase transformation when exposed to an aqueous environment similar to that of an autoclave sterilization process, leading to the degradation of mechanical properties.

It has been reported that zirconia–alumina composites show high resistance to low-temperature degradation [3]. Furthermore, the addition of 20 vol.% Al_2O_3 produces composites with improved flexural strength and fracture toughness [4]. Zirconia/alumina composites are bioinert materials that do not bond chemically with living tissue. Therefore, they must be bioactivated in order to be used in load-bearing applications for long periods of time. It has been found that bioactive materials, like hydroxyapatite and Bioglass®, bond chemically with osseous tissue by the formation of a bonelike apatite layer on their surface [2]. The bioactivity assessment can be performed by the immersion of samples in a simulated body fluid (SBF) with an ionic concentration nearly equal to that of human blood plasma [5].

Biomimetic processes have been widely studied for growing a bonelike apatite layer on different substrates by using simulated body fluids with an ionic concentration higher than that of SBF [6]. The formation of this layer depends partially on the calcium and phosphorous ionic concentration in the vicinity of the immersed substrate [7]. Thus, when the SBF is replaced during the biomimetic process or a more concentrated solution is used (1.4, 1.5, 2.0 or 5 SBF) the ionic concentrations will remain high, resulting in the formation of a hydroxyapatite layer by consuming calcium and phosphorous from the solution [6–8]. In the biomimetic coating on cobalt base alloys, the immersion of chemically treated samples in SBF for 7 days followed by the immersion in a more concentrated simulated body fluid has shown to induce

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Table 1
Ion concentration of simulated body fluids and human blood plasma

	Na^+	K^+	Ca^{2+}	Mg^{2+}	Cl^-	HCO_3^-	HPO_4^-	SO_4^-
SBF	142	5	2.5	1.5	148.8	4.2	1	0.5
1.4 SBF	198.9	7	3.5	2.1	208.32	5.88	1.4	0.7
Blood plasma	142	5	2.5	1.5	103.0	27.0	1	0.5

the formation of a bonelike apatite layer on these materials [8]. In the present work, the effect of this particular immersion method on the formation of a bioactive apatite layer on Mg-PSZ/Al_2O_3 composites was investigated. For comparison purposes, the in vitro bioactivity assessment of the ceramic materials by using SBF was also performed.

2. Experimental

2.1. Specimen preparation

The Mg-PSZ/Al_2O_3 composites were prepared by using reagent-grade magnesium oxide stabilized zirconia (Stanford Materials, USA) and alumina (Sasol, USA) with a particle mean size of 0.5 and 0.4 μm, respectively. Powder mixtures of 80 vol.% of PSZ and 20 vol.% of Al_2O_3 were ball-milled in a polyethylene jar with alumina balls for 1 h in acetone. The slurries were dried and disk-shaped by uniaxial pressing at 100 MPa for 15 s. The zirconia composites were sintered in air at 1550 °C for 2 h.

2.2. Preparation of SBF and 1.4 SBF

Two simulated body fluids, one with an ion concentration nearly equal to that of human blood plasma (SBF) and other 40% more concentrated (1.4 SBF), were used (Table 1). The solutions were prepared dissolving reagent-grade NaCl, $NaHCO_3$, KCl, $K_2HPO_4 \cdot 3H_2O$, $MgCl_2 \cdot 6H_2O$, $CaCl_2 \cdot 2H_2O$ and Na_2SO_4 in deionized water and buffered to pH 7.4 with $C_4H_{11}NO_3$ and 1N HCl at 36.5 °C [5].

2.3. Immersion of the materials in simulated body fluids

Each composite was immersed in 150 ml of solution and kept at 36.5 °C in an incubator. The immersion was performed following two different routes. The first one consisted in the immersion of the substrates in SBF for 7 days. Then, the SBF was replaced for 1.4 SBF and, 1 week later, the solution was replaced for fresh 1.4 SBF (re-immersion method). The second route was a single immersion method where the substrates were placed for 21 days in SBF without the replacement of the solution at any time.

At the end of the immersion periods, the ceramic substrates were removed from the flasks, gently washed with deionized water and dried at room temperature.

2.4. Characterization methods

The surface of the biomimetically treated substrates was analyzed by using scanning electron microscopy (SEM) (JSM 6300, Jeol, Japan), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD) (X'Pert, Philips, Holland).

3. Results and discussion

Fig. 1 shows the surface of samples after 7 days of immersion in SBF (Fig. 1A), after 14 days (7 days in SBF+7 days in 1.4 SBF, Fig. 1B) and after 21 days (7 days in SBF+14 days in 1.4 SBF, Fig. 1C). No significant change

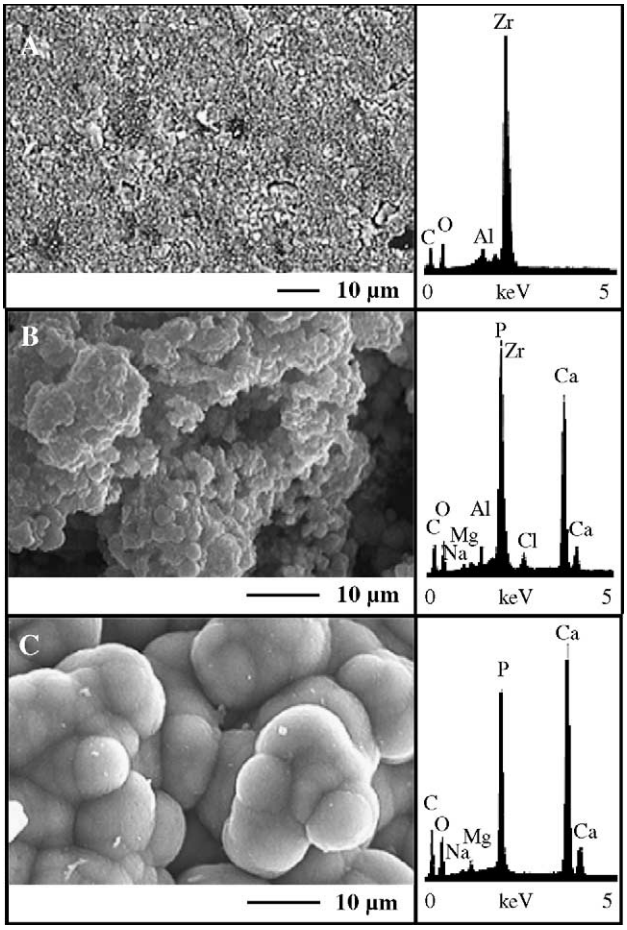

Fig. 1. SEM photographs and corresponding EDX spectra of the composite surface after 7 days of immersion in SBF (A), after 14 days: 7 days in SBF+7 days in 1.4 SBF (B) and after 21 days: 7 days in SBF+14 days in 1.4 SBF (C).