Materials Letters 164 (2016) 144–147

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

A novel *in situ* deposition of hydroxyapatite nanoplates using anodization/hydrothermal process onto magnesium alloy surface towards third generation biomaterials

Hamouda M. Mousa [a,b], Arjun Prasad Tiwari [a], Jinwoo Kim [a], Surya Prasad Adhikari [a,c], Chan Hee Park [a,d,*], Cheol Sang Kim [a,d,*]

[a] Department of Bionanosystem Engineering, Graduate school, Chonbuk National University, Jeonju, Jeonbuk 561-756, Republic of Korea
[b] Department of Engineering Materials and Mechanical Design, Faculty of Engineering, South Valley University, Qena 83523, Egypt
[c] Institute of Engineering, Tribhuvan University, Kathmandu, Nepal
[d] Division of Mechanical Design Engineering, Chonbuk National University, Jeonju, Jeonbuk 561-756, Republic of Korea

ARTICLE INFO

Article history:
Received 3 September 2015
Received in revised form
20 October 2015
Accepted 28 October 2015
Available online 30 October 2015

Keywords:
Magnesium alloy
Biomaterials
Hydrothermal deposition
Bone tissue regeneration
Biomimetic

ABSTRACT

Third-generation biomaterials aim to stimulate specific cellular responses at the molecular level, these materials characterized with a resorbable and bioactivity that help body heal once they have been implanted. Here, a biomimetic method was used to generate hydroxyapatite (HA) nanoplates on the surface of AZ31B Mg alloy via anodization in simulated body fluid (SBF), followed by a hydrothermal (HT) process. The resulting nanoplates were characterized using FE-SEM, XRD, and FT-IR, surface hydrophobicity, in addition, corrosion was assessed electrochemically. The excellent bioactivity of the treated samples compared with naked ones were confirmed *in vitro* with MC3T3-E1 osteoblastic cells with significant growth and proliferation.

1. Introduction

Bone reconstruction defects resulting from osteoporosis are a challenge for orthopedic surgery, and one of the current difficulties is to engineer materials that can match both the mechanical and biological characteristics of real bone tissue matrix (BTM) [1]. Biodegradable metals are second generation of biomaterials, and these have been a longstanding area of interest for medical applications [2,3]. Mg is a promising material for stent and orthopedic applications since it has many appealing properties, such as a low density and good mechanical properties [4]. The degradation products of Mg are not toxic to human physiology, moreover, these aid the growth and healing of damaged tissue. Unfortunately, Mg alloys undergo corrosion in human body fluid, which results in a loss of their mechanical integrity and an increase in the pH of the surrounding tissues [5]. However, proper bio-coating materials can improve the interface of the implant surfaces with the connected tissues [6,7], and HA is one such bioactive ceramic that has been proven to be effective for use as a protective coating for Mg alloys to control the biodegradability and improve the biocompatibility [8].

Natural bone consists of HA nanocrystrals in a plate-like shape with a length of 30–200 nm and a thickness of 2–7 nm [9]. These nanoplates can promote the porosity of the interface, and can thus avoid a mechanical mismatch between the implant interface and the host since the stress shield effect can be eliminated by altering the porosity [10]. Next-generation biomaterials combine these biodegradable and bioactive properties without the need for another surgery [11]. The main objectives of this work is to improve the bioactivity and to control the degradation of the Mg alloy by mimicking HA-nanoplates in natural bone to contribute in next-generation biomaterials.

2. Materials and methods

A commercial alloy (3% Al, 1% Zn and 96% Mg; Alfa Aesar, South Korea) was used in this study with samples size (12 mm × 12 mm × 6.35 mm). Samples were polished with SiC paper up to 2000 grit. The anodization process was then performed in a stainless-steel container (cathode) containing SBF solution with and without adding 10% of hydroxyapatite (fast flow type, particle size 75–180 μm), and these were termed as (SBF-HA/HT) and (SBF/

* Corresponding authors. Division of Mechanical Design Engineering, Department of Bionanosystem Engineering, Graduate School, Chonbuk National University, Jeonju, Jeonbuk 561-756, Republic of Korea. Tel.: +82 63 270 4284; fax: +82 63 270 2460.
E-mail addresses: biochan@jbnu.ac.kr (C.H. Park), chskim@jbnu.ac.kr (C.S. Kim).

http://dx.doi.org/10.1016/j.matlet.2015.10.145
0167-577X/

HT), respectively. The SBF solution was prepared according to our previous work [7], and the anodization conditions were set to 50 V at 30 mA with a process time of 10 min. Thereafter, the samples were subjected to two HT process. First, the samples were rinsed at 5 M NaOH at 60 °C for 2 h, secondly, the samples were immersed in SBF solution for 2-days at 60 °C, after each process samples rinsed in distillated water.

The surfaces of the coated samples were characterized using FE-SEM (SU-70 SCHOTY type, HITACHI), X-ray diffractometer (XRD, Rigaku, Japan) with Cu Ka (k = 1.540 Å), and FT-IR (Spectrum GX). The surface hydrophobicity was measured by using a DPRO-image standard device to measure the water contact angle for the different surfaces. The electrochemical corrosion behavior was investigated in SBF at 37 ± 1 °C, according to Ref. [12]. The *in vitro* cell culture procedure was performed according to our group [13] for 1, 5, and 8-days. Samples were immersed in culture media (alpha-mem supplemented with 10% FBS and 1% penicillin-streptomycin). Thereafter, the samples were shacked for 4-days at 120 rpm and 37 °C, and then the extraction of the samples was used for indirect cell culture. The MC3T3-E1 cells were seeded at 4×10^5 cells/well and were incubated at 37 °C in a 5% CO_2 atmosphere. A 200 mL culture media followed by 10% of CCK-8 solution added to 96-well plate then incubated for 4 h. The cell viability was determined by measuring the absorbance at 450 nm. Cells

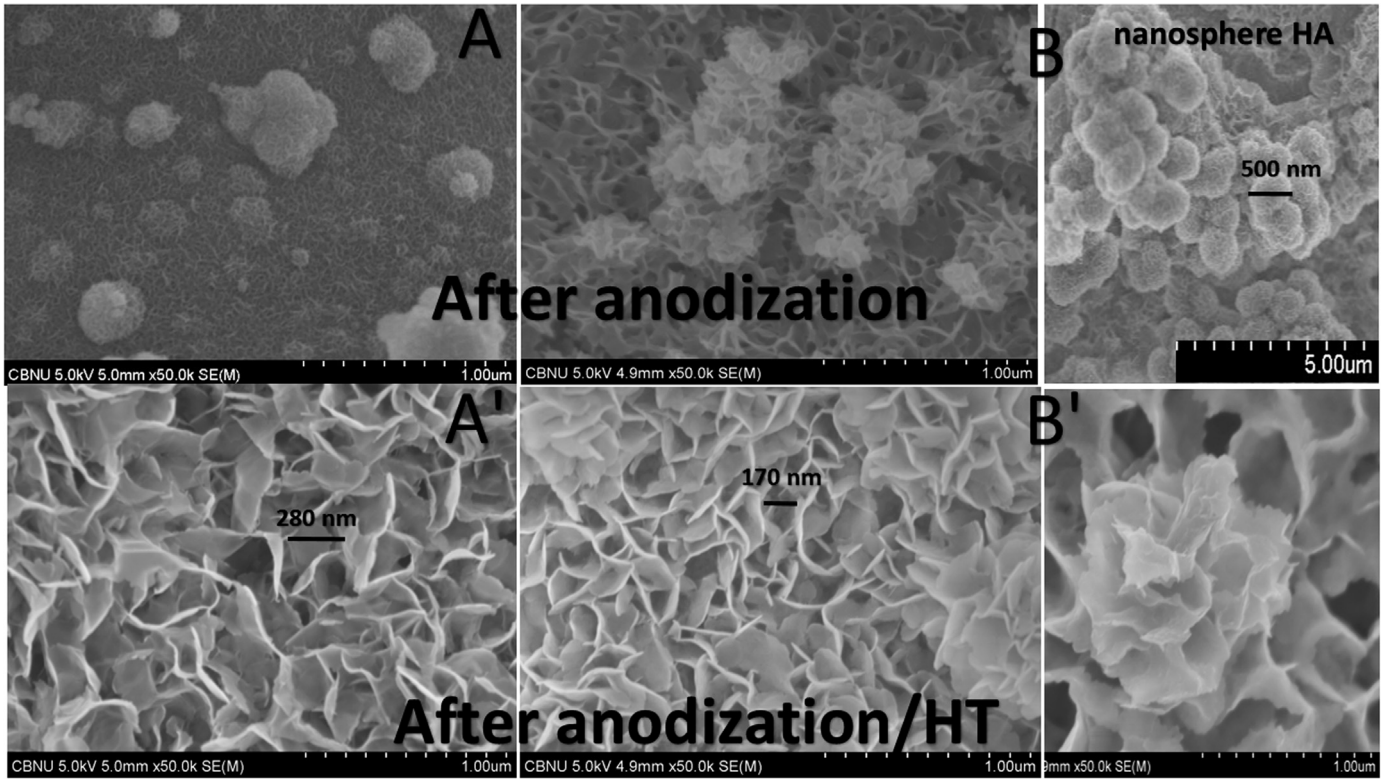

Fig. 1. FE-SEM images of the different samples: (A) SBF/HT and (B) (SBF-HA)/HT after anodization. (A′) and (B′) indicate the samples after the HT process.

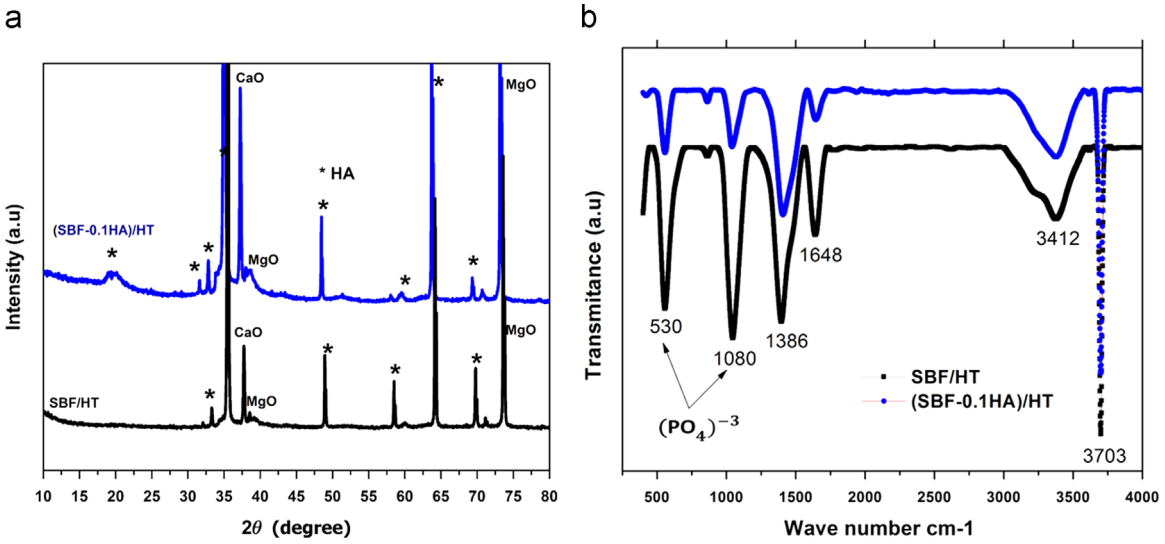

Fig. 2. XRD patterns of the treated samples (a) and FTIR analysis of two different kinds of sample (b).