Available online at www.sciencedirect.com
ScienceDirect
Trans. Nonferrous Met. Soc. China 26(2016) 1019−1024
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Preparation of micro-nanostructure on titanium implants and its bioactivity

Rui ZHANG, Yi WAN , Xing AI, Teng WANG, Bo MEN

Key Laboratory of High Efficiency and Clean Mechanical Manufacture, Ministry of Education, School of Mechanical Engineering, Shandong University, Jinan 250061, China

Received 23 April 2015; accepted 25 February 2016

Abstract: Surface modification of medical implants was considered as an effective method to improve the cellular behaviors and the integration of tissue onto materials. The micro-nanostructured surface on the titanium alloy was prepared by laser treatment and multiple acid etching. The surface morphologies of different titanium alloy substrates were characterized by scanning electron microscopy (SEM). The effects of micro-nanostructured surfaces on the cellular responses were investigated in vitro by observing hydroxyapatite formation, cell morphology and cell adhesion. The results indicate that the micro-sized structure promoted the adhesion and proliferation of cultured osteoblasts. Furthermore, the micro-nanostructured surface was more conducive to cell adhension stretching compared with the micro-structured surface. All results suggest that the micro-nanostructured surface improved the biocompatibility and integration of tissue onto titanium alloy implants.

Key words: titanium alloy; micro-nanostructure; laser treatment; multiple acid etching; bioactivity

1 Introduction

Biomedical titanium alloys show good physical and mechanical performance, and are therefore widely used as surgical implant materials [1−3]. However, the bone could not be reconstructed efficiently on the surface of titanium and titanium alloys implants, which leads to poor integration with the surrounding bony tissue [4−6]. Therefore, to further improve the biocompatibility of titanium and titanium alloys implants, it is quite important to modify their surface with biotechnology, which will accelerate the integration between implanted materials and the interface of human tissue.

The current surface modification methods for titanium alloys include hydroxyapatite (HA)-coating, acid etching, sand blasting and anodizing. Among the various technologies for surface biomodification, the biomimetic topography of implant surface has attracted special attention of researchers [7−11]. A microstructure promotes secretion and mineralization of extracellular matrix [12,13], and promotes fast and steady bone integration [14]. A nanostructure also shows a positive effect on the osteoblast behavior [15], can accurately mimic the cellular growth environment [16], and promotes adhesion, proliferation and differentiation [17,18], which in turn promotes bone mineralization [19,20]. However, most of former methods focus on developing single scale structures on the implant surface, microstructure or nanostructure. There are few studies on the micro-nanostructured surface.

This work proposed a novel method to construct micro-nanostructure on bio-titanium alloy surface with laser treatment and multiple acid etching. The bioactivity of the samples was investigated in vitro by observing hydroxyapatite formation, cell morphology and cell adhesion. The new method for surface modification was expected to be widely applied in the biomedical field.

2 Experimental

2.1 Sample preparation

The sample material used was a titanium alloy (TC4), which is widely used in the field of biomedicine. Titanium alloy (TC4) samples with dimensions of 10 mm × 10 mm × 1.5 mm were used. The samples were polished by a polishing machine and further cleaned with acetone and water.

Foundation item: Projects (51175306, 51575320) supported by the National Natural Science Foundation of China; Project (TS20130922) supported by the Taishan Scholar Foundation, China; Project (2014JC020) supported by the Fundamental Research Funds for the Central Universities of China

Corresponding author: Yi WAN; Tel: +86-531-88392539; E-mail: wanyi@sdu.edu.cn

DOI: 10.1016/S1003-6326(16)64217-6

The polished samples were manufactured by a YAG−T80C laser-marking machine (Shenzhen Han's Laser Technology Limited Company, China), which has a wavelength of 1064 nm and a spot diameter of 30 μm. The current and frequency of the laser machine were 13 A and 3 kHz, respectively, which were optimized in advance. The microstructure samples contained micro-pits with a diameter of 140 μm and a depth of 35 μm. Surface topography was characterized by scanning electron microscopy (SEM), as shown in Fig. 1. The clinkers produced by the laser on the inner surface and micro-pits edge can easily fall off, and were found to be harmful to the biocompatibility of the implant. Therefore, the clinkers were cleared in advance.

Multiple acid etching was used to clean off the laser clinkers and generate the micro-nanostructure, as shown in Fig. 2. Firstly, the samples were treated with a mixed solution containing 0.09 mol/L HNO_3 and 0.11 mol/L HF at room temperature for 10 min. Secondly, the samples were treated with a mixed solution containing 4.5 mol/L H_2SO_4 and 2.9 mol/L HCl at 80 °C for 25 min and then cleaned with water quickly. Thirdly, the samples were treated with a mixed solution of 98% H_2SO_4 and 30% H_2O_2 with volume ratio of 1:1 at room temperature for 70 min. Finally, the samples were washed with distilled water.

The laser clinkers were cleaned by multiple acid etching, as shown in Fig. 2. At the same time, the micro-nanostructure was obtained. After multiple acid etching, orderly ridge structures with widths of 0.5−2 μm were observed in the inner micro-pits containing a cluster of nanopores with diameters of 20−100 nm.

The samples were divided into three groups: polished group, microstructure group and micro-nanostructure group.

2.2 Cell culture

MC3T3 osteoblasts (Oral Medicine Institute of

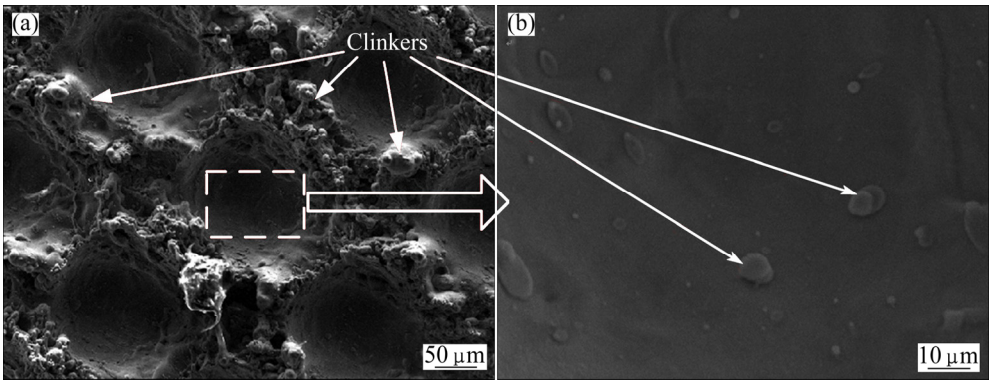

Fig. 1 SEM images of titanium alloy (TC4) surfaces after laser treatment

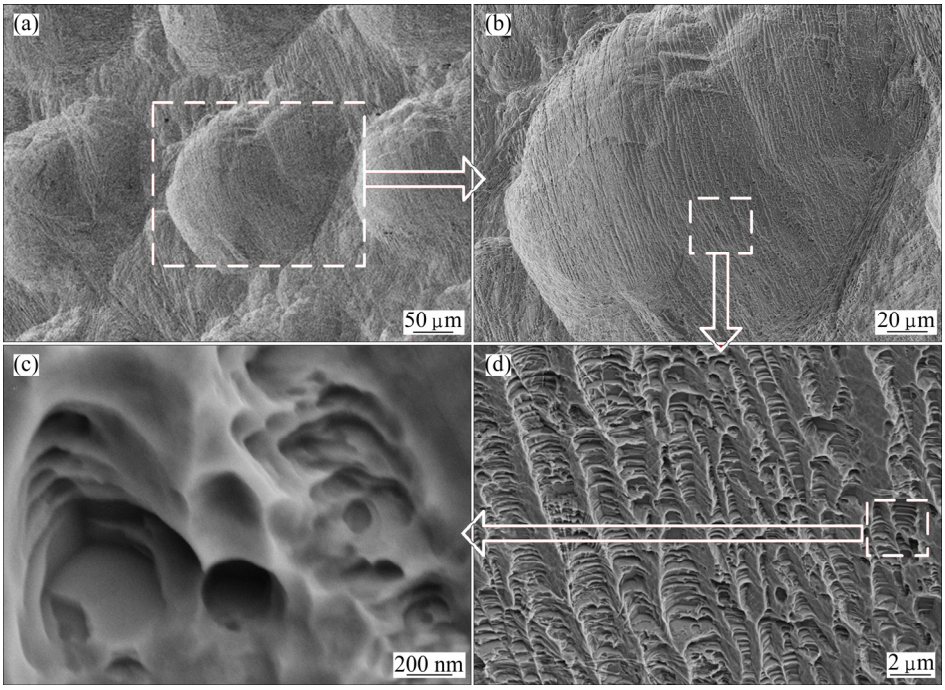

Fig. 2 SEM images of titanium alloy (TC4) surfaces after multiple acid etching