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Effect of a biomimetic titania mesoporous coating doped with Sr on the osteogenic activity

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ABSTRACT

Fabrication of titanium (Ti)-based biomedical implants with appropriate topography as well as capacity for drug delivery is highly pursued in the field of orthopedic and dental implants. In this study, a biomimetic mesoporous coating imbedded with strontium (MPs-Sr) is prepared by the high current anodization (HCA) and hydrothermal treatment (HT). This coating provides a more stable mechanical performance than the conventional nanotube arrays. The Sr loading is regulated by the HT reaction time and the Sr is released in a controllable manner from the MPs-Sr surface. The hydrophilic performance of MPs-Sr are significantly improved. Furthermore, it is showed that the attachment and spreading of preosteoblast MC3T3-E1 cells are significantly up-regulated by the nanoscale topology of MPs and the doped Sr. The improved collagen secretion and matrix mineralization levels of cells are closely related with the Sr release. The excellent osteogenic properties of MPs-Sr samples highlight their promising potential for use in clinical application.

1. Introduction

At present, the clinical use of Ti-based implants still faces a challenge: lack of native tissue integration, which often leads to the loosening or failure of the implantation [1,2]. It has been widely recognized that the surface characteristics of hard implants play an important role in the osseointegration [3–5]. The physicochemical properties of implant surface are mainly determined by the surface topographies and substance decorated on the implant [6,7]. From the biomimetic point, a hierarchical structure composed of micro- and nanoscale components may provide a suitable surface topography for bone-to-implant contact as it can well mimic the structure of the natural bone tissues [8–10]. In addition, the implant surface can be treated with drugs, bioactive inorganic elements, biomacromolecules, etc., to impart osteogenesis and enhance osteointegration [11–16]. Therefore, a simple strategy for biomedical Ti surface engineering that imparts both biomimetic bone surface and sustained delivery of bioactive factors, would be promising in bone implant applications.

Different strategies have been attempted to fabricate micro/nanostructures doped with some trace elements on the Ti-based implants [8,17–19]. The TiO_2 nanotubes (NTs) have been demonstrated to be an excellent delivery platform for the release of the therapeutic agents, especially inorganic bioactive elements. For instance, Gao et al., embedded Ag_2O nanoparticles into titania nanotubes by anodizing TiAg surface [15]. Chen et al., doped Sr into the TiO_2 nanotubular structures by treating the NTs successively with Sr^{2+} solution in a hydrothermal manner [20]. Especially, it has been widely accepted that Sr can stimulate bone formation and inhibits osteoclastic activity. However, the main issue of Ti-based implant is that the NTs are prone to peeling off from the substrate due to the poor interfacial adhesion, which significantly compromises their potential for clinical applications.

Recently, we have fabricated a micro/nano-textured layer with TiO_2 mesoporous (MPs) arrays by a high current anodization (HCA). Compared to the NTs, the interfacial strength of MPs is significantly improved, indicating their potential application value in biomedical implants. Furthermore, it is very necessary to test whether MPs can be used as a delivery platform to dope and release the inorganic bioactive elements. The development of a more stable and efficient delivery platform for inorganic elements would be clinically useful. Here, a nanopore structure loaded with Sr was fabricated on Ti implants by treating the MPs in a hydrothermal manner. In order to determine the optimal quantity of Sr incorporated into the MPs, a set of MPs-Sr implants with different Sr loadings were prepared and the effect of the Sr doped surface on the osteoblast behaviors was performed. This biomimetic and controlled releasing MPs-Sr coatings would be a promising platform, used to be expedite osteointegration and new bone formation.

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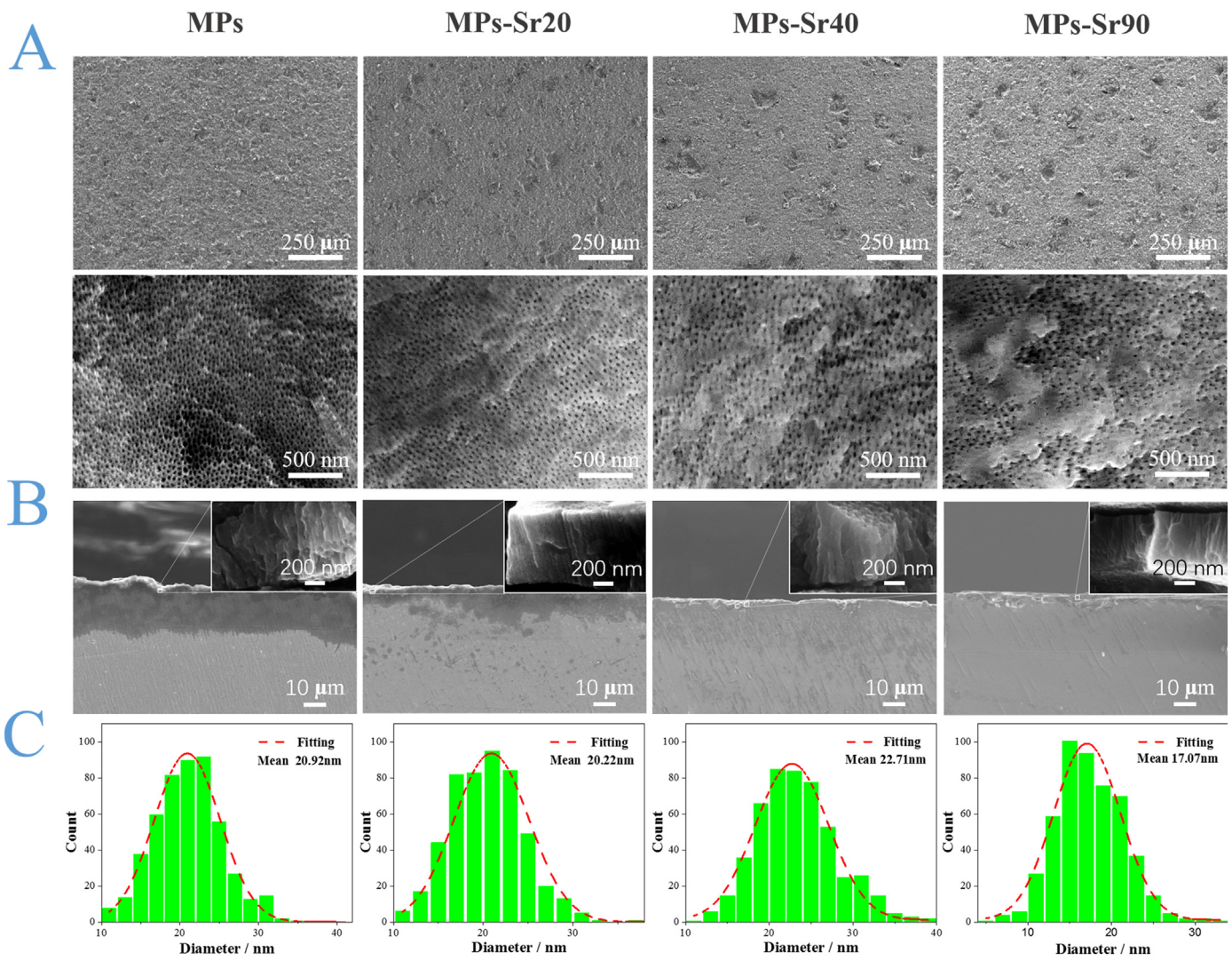

Fig. 1. Surface morphology and cross-sectional SEM images of MPs and MPs-Sr samples. (A) Images of the anodized and hydrothermal treated samples. (B) Cross-sectional images of the anodized and hydrothermal treated samples. (C) Histogram of the MPs and MPs-Sr diameter, the red dashed lines were the fitting results according to Gaussian distribution. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

2. Experimental section

2.1. Materials and methods

Pure titanium plates (99.6% purity) with dimensions of Φ 14 mm × 2 mm were polished to 1200 grit by SiC abrasive paper and then ultrasonically rinsed with acetone, ethanol and deionized water, respectively. Briefly, the rinsed Ti plate was used as anode on a power supply with constant current density of 0.26 A/cm^2 in 4.0 g/L $Cu(NO_3)_2$ electrolyte, with the frequency of 800 Hz, duty cycle of 30%, the duration of 240 s in 1 L electrolyte. The stainless-steel groove was used as cathode. The prepared samples were washed to remove the residual electrolyte. The MPs-Sr were fabricated by hydrothermal treatment in 33 mL of 0.01 M $Sr(OH)_2$ solution in a 50 mL teflon-lined autoclave and heated at 150 °C for 20, 40 and 90 min, which were labelled as MPs-Sr20, MPs-Sr40 and MPs-Sr90, respectively. Then the MPs-Sr samples were ultrasonically washed with 0.1 M HCl for 5 min to remove residual $Sr(OH)_2$, rinsed with distilled water, and dried in oven at 60 °C for 1 h.

2.2. Sample characterization

Field-emission scanning electron microscopy (FE-SEM, JSM-7001F, JEOL) at an accelerating voltage of 10 kV was performed to examine the surface and cross-sectional morphology of the as-prepared MPs and MPs-Sr. The diameter distribution of MPs were measured by Nano Measurer1.2.0 software, 500 pores were measured at each picture and the porous diameter distribution was fitted by Orign 8.6. Sr contents in the MPs-Sr coating were determined by energy-dispersive X-ray spectroscopy (EDS, QX200, Bruker). To evaluate the hydrophilicity of the MPs and MPs-Sr, static contact angle measurements were performed using a pendant drop method on a contact angle analyzer (SL200B, Solon) in an ambient atmosphere. The interfacial strength between the oxide layer and its substrate was evaluated by a scratch tester with an initial load of 1 N and final load of 16 N. The load rate is at 5 N/min during the test and the scratched samples was observed by FE-SEM. The surface elemental composition and chemical states of elements in the MPs-Sr were determined by an X-ray photoelectron spectroscopy (XPS, K-Alpha, Thermo) with monochromatic Al Ka radiation (6 mA, 12 kV and 1486.68 eV) after 20 s Ar etching. The results were processed by the Thermo Avantage 4.51 software based on the Gaussian-Lorentzian mix product (L/G = 0.3) and Powell algorithms, all binding energies were referenced to the C 1s peak centered at 284.8 eV. The crystal structure was evaluated by X-ray diffraction (XRD, DX-2700, Haoyuan).