

Short communication

Preparation of porous titanium by direct in-situ reduction of titanium sesquioxide



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ARTICLE INFO

Keywords:

Titanium sesquioxide
Calciothermic reduction
Porous titanium
Pore size
Porosity

ABSTRACT

A new process to fabricate porous titanium by calcium vapor in-situ reduction of titanium sesquioxide was presented in this paper. The mixture of Ti_2O_3 and $CaCl_2$ powders was preformed and reduced by calcium vapor. The porous titanium structure was obtained after leaching reduction products with hydrochloric acid and deionized water. The pore structure, morphology, composition of reduction products were investigated by SEM-EDS and XRD. The influence of different contents of anhydrous calcium chloride on porosity was discussed. The results show metallic titanium products with two types of pore structures and vacuum sintering treatment strengthened effectively connection between titanium particles. The porosity of porous titanium samples was 62%–82% and more than 90% of pores are open-pores.

Because of its excellent mechanical properties, superior corrosion resistance, low density, high specific strength and remarkable biocompatibility, porous titanium has already become an increasingly attractive material, especially for bone implants. Many technologies for preparing porous titanium have been reported in recent years, such as vacuum plasma sintering technology [1–3], combustion synthesis technology [4], 3D printing technology [5–7], powder sintering technique [8–10], laser sintering technology [11,12], microwave assisted in-situ synthesis technology [13], and anodic oxidation technology [14]. Almost all industrialized production processes of porous titanium are using titanium powders as the raw material produced by hydrogen dehydrogenation or the Kroll method.

This work demonstrates a new and short approach combining the extraction metallurgical process of titanium by calcium vapor reduction of its sesquioxide oxides with porous structure fabrication to prepare porous titanium. $CaCl_2$ was used as additive in this method. Consequently, the product body, a mixture block of titanium and calcium oxide, would have tunnel structure by the evaporation of calcium chloride at reduction temperatures. Besides, porous structure would be generated after removal of reduction by-product CaO particles as well. And pore size would be inherited from CaO particle size, basically. Moreover, releasing reaction heat of calciothermic reduction of titanium suboxides should contribute to sintering of titanium particles.

Hopefully, this concept will be helpful to develop the direct method to built porous titanium structure from titanium oxides.

Fig. 1 shows this concept of preparing porous titanium structure through the above procedures. The mixed powders of commercial titanium sesquioxide powders (100 mesh, 99.9%, Aladdin Industrial Co., Ltd., China) and anhydrous calcium chloride (Analytical Reagent, Sinopharm Chemical Reagent Co., Ltd, China) with certain theoretical mass ratios was milled for 3 min and pressed to be a cylindrical preform. Subsequently, these perform feeds were transferred to the stainless-steel screen plate, and calcium metal calcium metal ($Ca > 98\%$, mass fraction) as the reductant was placed in the bottom of a stainless steel crucible. The crucible and screen plate were placed in a stainless steel cylindrical container, which was placed in a well-type resistance furnace. The calciothermic reduction was conducted below 0.1 Pa and heated constantly at 1273 K for different holding time. The volatilization of $CaCl_2$ happening made the porous precursor of titanium oxides, then these pores still kept settled companying with the in-suit reduction of titanium oxides by calcium vapor when temperature ramped up. After that, the reduction products were leached with dilute hydrochloric acid and washed by deionized water and absolute alcohol for three times. As a result, more new porous structures were generated with the removal of by-product CaO particles of reduction reaction. Finally, the dry samples after vacuum dring for 24 h was sintered at

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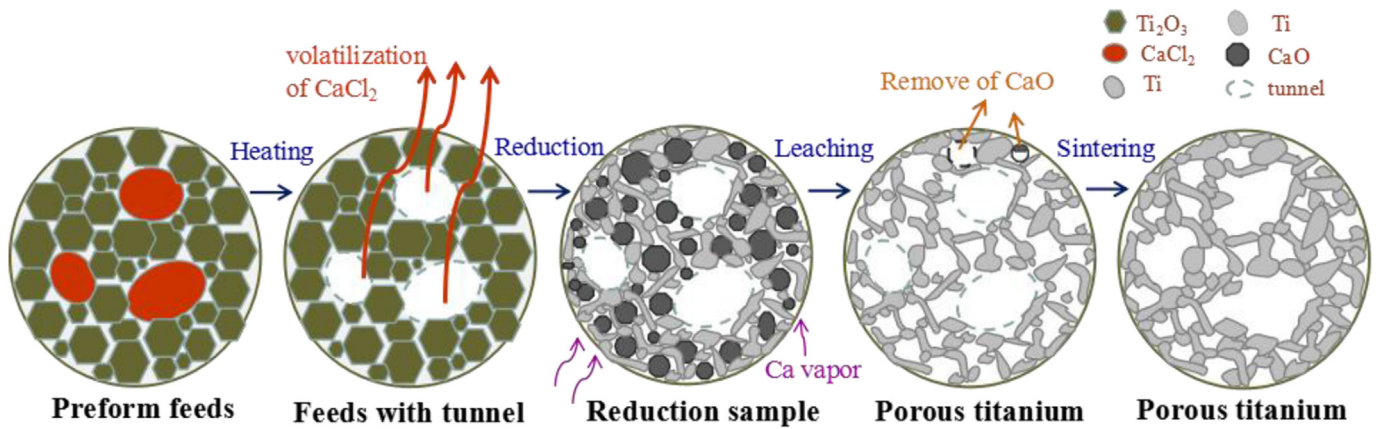


Fig. 1. The concept to prepare porous titanium by calciothermic in-situ reduction of Ti_2O_3 .

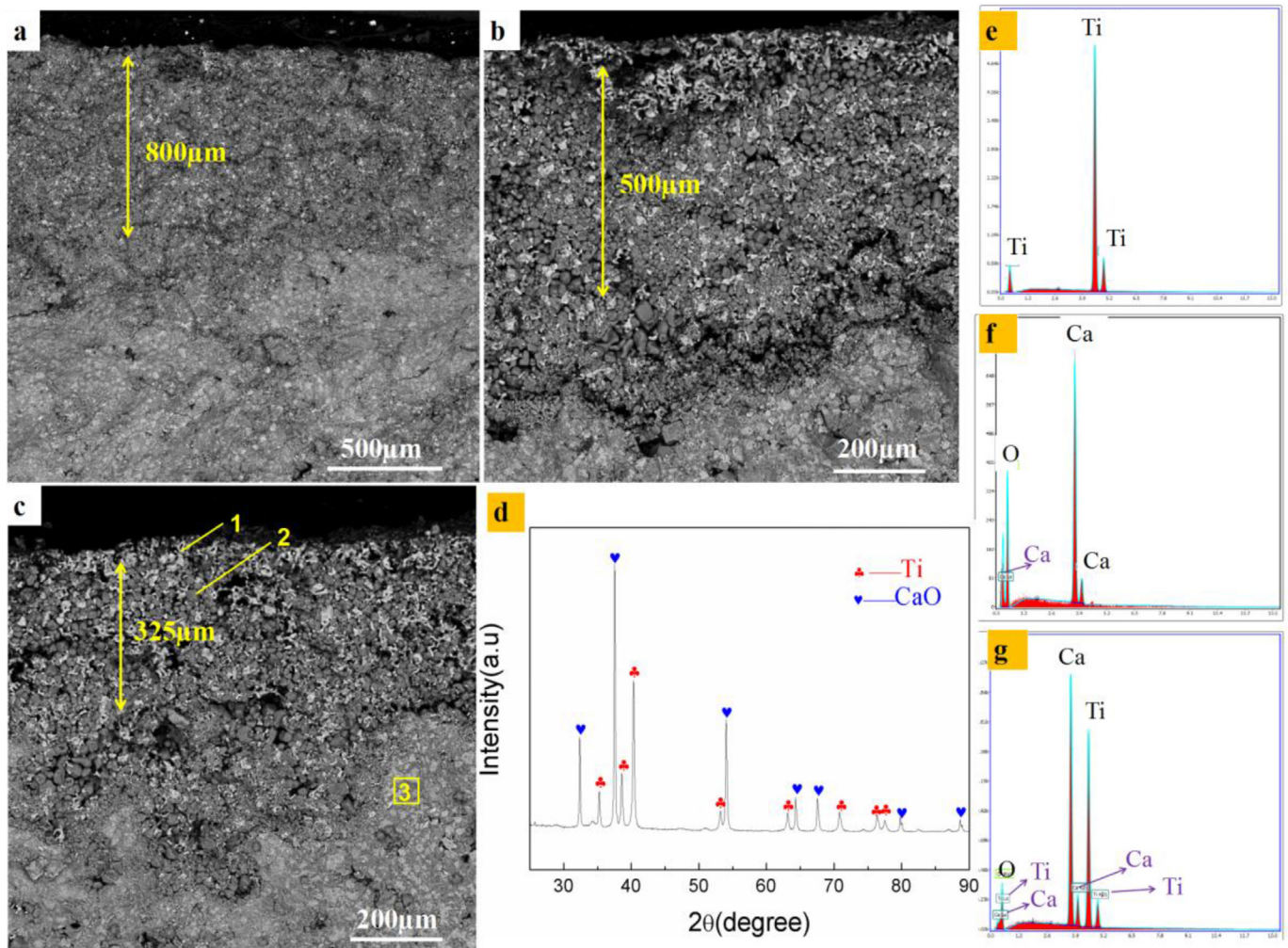


Fig. 2. The microstructural images and composition of the reduction samples obtained at different holding time: (a) sample at 4 h; (b) sample at 6 h; (c) sample at 8 h; (d) XRD patterns of sample at 8 h; (e)–(g) the composition of different points by EDS in (c)1–3.

1573 K in a vacuum for 2 h to strengthen the porous structure in metallic titanium pieces.

SEM-EDS was employed to capture the morphology and detected the composition of reduction samples of porous titanium. XRD was used to identify the phase of samples. The porosity of samples was calculated by measuring the dimensions and weight according to the following equation [15]: $P = 1 - \rho_a / \rho_m$, where ρ_a is determined by dividing the weight by the volume of the porous titanium and ρ_m is the density of

titanium (4.54 g/cm^3). The porosity of the open-pore structure was calculated with the Archimedes method [16].

Two distinct areas in the reduction samples are presented in Fig. 2a–c. The clear antler-shaped titanium particles and polyhedral CaO particles are distributed on the porous surface area about 0.5 mm–1 mm, while there is a mixed zone of Ti and CaO particles in the compact interior of samples by detected EDS and XRD shown as Fig. 2d–g. No any definite chloride composites in reduction samples

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