

Fabrication of nano-porous γ -Al₂O₃ layers on porous Ti-48Al-6Nb alloys



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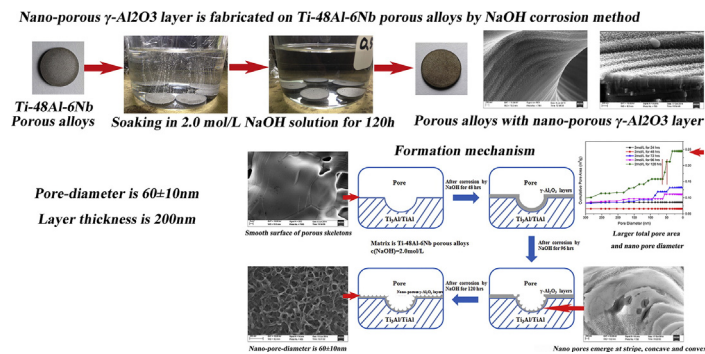
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HIGHLIGHTS

- Nano-porous γ -Al₂O₃ layers were fabricated by soaking in 2.0 mol/L NaOH solution for 120 h.
- Nano-pore-diameter was 60 ± 10 nm and the thickness of layers was 200 nm.
- Total pore area increased nearly to 0.25m²/g due to nano-porous layer.
- The porous alloys transformed to gradient porous materials with nano and micron range pores.

GRAPHICAL ABSTRACT



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ABSTRACT

Nano-porous materials are spreading widely in various fields, such as electrochemistry, biochemistry, adsorption, and catalysis. In the present work, nano-porous γ -Al₂O₃ layers with 200 nm thickness have been fabricated on porous Ti-48Al-6Nb alloys by soaking in NaOH solution. Corrosion products of γ -Al₂O₃ with 200 nm thickness form after 48 h and begin to transform to nano-porous structures at 72 h in the areas with a smaller radius of curvature. Then the nano-pores shrink and reach the final size of 60 ± 10 nm within 120 h. Increasing the concentration of NaOH solution or soaking time accelerates the formation of nano-porous layers and the best situation is soaking for 120 h in a 2.0 mol/L NaOH solution. Meanwhile, the nano-porous γ -Al₂O₃ layers improve the electrochemical corrosion resistance of the porous alloys due to the more positive self-corrosion potential/current and the right-moved passive region. The NaOH corrosion method to fabricate nano-porous γ -Al₂O₃ layers is much easier than other methods and the porous alloys possess the better corrosion resistance and absorbability thanks to the nano-porous γ -Al₂O₃ layers and the micron-porous Ti-48Al-6Nb matrix.

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1. Introduction

Porous intermetallics attract a worldwide attention in the last decades due to their good performances, such as the high specific stiffness,

high energy absorption capacity, and good oxidation resistance and corrosion resistance among foam metals. In our group, more attention has been paid for the high Nb-containing TiAl alloys because of the attractive properties of high melting point, good oxidation resistance, high specific strength and modulus due to the high Nb contents [1–6].

Porous Ti-48Al-6Nb (at.%) alloys were synthesized by powder metallurgy (PM) method using elemental powders [7]. The Kirkendall effect and phase transformation contribute to the pore-formation and the

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pores have mainly micron scale [8]. Although the porous Ti-48Al-6Nb coatings were fabricated by cold gas spraying and reactive sintering by Yang et al. [9], the pore size is still in the micron range of 1.8 μm due to the difficulty to control the pore size in nanometer range. However, nano-porous or nano-coatings have been fabricated by various methods for other materials. Nano Cu coatings were prepared on porous Si by acid etching the Al-Si alloy powder by Li et al. [10] and nano-porous bioactive glass film was prepared by the sol-gel method by Ma et al. [11]. The nano-porous structures have implications for bioactivity [12], electrochemical properties [13] and batteries efficiency [14]. In addition, the nano-porous $\gamma\text{-Al}_2\text{O}_3$ has special properties. The $\gamma\text{-Al}_2\text{O}_3$ nano-structured hollow microspheres, used to treat dye wastewater, were synthesized by Fang et al. [15] in terms of methylene blue as structure directing agent. The nano- Al_2O_3 with porous structure, used to separate iridium ions from aqueous solutions due to its better adsorption properties, was synthesized by Zhang et al. [16]. Also the nano- Al_2O_3 can be used as the excellent catalyst carrier. The electrochemical performance of $\text{LiNi}_{0.6}\text{Co}_{0.2}\text{Mn}_{0.2}\text{O}_2$ powder has been improved greatly by coating with nano- Al_2O_3 particles via ultrasonic coating [17]. However, little is known about the nano-porous structure of TiAl based alloys. In the present work, the nano-porous $\gamma\text{-Al}_2\text{O}_3$ layers are fabricated on porous Ti-48Al-6Nb alloys by soaking in NaOH solution. According to the aforementioned nano- Al_2O_3 , the present porous Ti-48Al-6Nb alloys with nano-porous $\gamma\text{-Al}_2\text{O}_3$ layers have significant contributions in many fields.

2. Experimental procedures

Porous Ti-48Al-6Nb (at.%) alloys were fabricated by the powder metallurgy (PM) under a pressure of 200 MPa [18]. Purity and particle sizes of elemental powders are shown in Table 1. The schematic procedure of heat-treatment is shown in Fig. 1. Air and vapor escaped from compacts were at the temperature of 120 °C and the initial temperature of Ti-Al reaction was 600 °C [19]. Samples were heat-treated at 900 and 1350 °C for 3 h, where Ti-Al and Nb-Al phase transformations occurred with much pores formed [20]. Finally, the porous Ti-48Al-6Nb alloys were achieved when the temperature decreased to room temperature in vacuum.

The nano-porous $\gamma\text{-Al}_2\text{O}_3$ layers were synthesized on the skeleton surface of porous Ti-48Al-6Nb alloys by soaking in NaOH solution for different times. The NaOH solutions used in our research were 0.5, 2.0 and 3.0 mol/L and the soaking times were 24, 48, 72, 96, and 120 h. X-ray diffraction (XRD, Multipurpose X-ray Diffractometer TTR III) was used to analyze the phases on the skeleton surface using an incident angle of 1.5°. The pore structure was examined by scanning electron microscopy (SEM, ZEISS SUPRA 55) and the pore parameters were measured by mercury intrusion porosimetry (MIP, Quantachrome AUTOSCAN-33) [21]. Polarization curves were achieved by the three electrodes method [22,23].

3. Results and discussion

3.1. Morphology of nano-porous $\gamma\text{-Al}_2\text{O}_3$ layers on porous Ti-48Al-6Nb alloys

The morphology of porous Ti-48Al-6Nb alloys after sintering is shown in Fig. 2. There exist a lot of pores with different pore diameters among the Ti-Al skeletons. The inner skeletons present a sleek polygon,

Table 1
Purity and particle size of the elemental powders.

Element	Purity (%)	Particle size (μm)
Nb	>99.9	48–75
Ti	>99.9	48–75
Al	>99.9	48–75

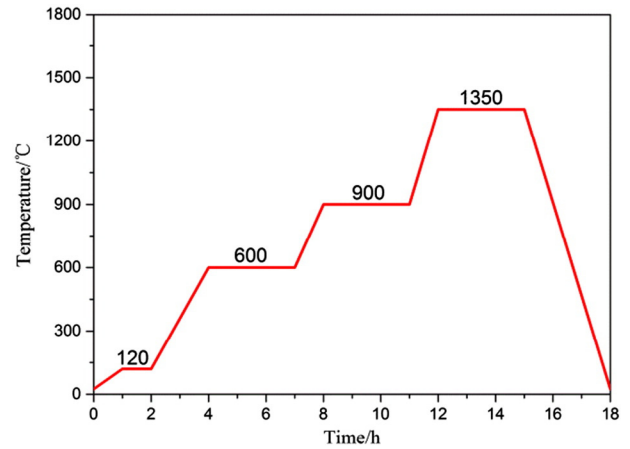


Fig. 1. Schematic procedure of heat treatment.

some areas are relatively flat and some are bent. After soaking in NaOH solution, the nano-porous layers emerged on the surface of these skeletons.

The surface of porous skeletons present rough and stripy in the 0.5 mol/L NaOH solution for 120 h (see Fig. 3a). A representative area magnified 50,000 times is shown in Fig. 3b. The nano-porous structures are obvious wherever the porous skeletons are flat or bent. Some nano-pores are deeper and some shallower, which will be analyzed in next Section. The morphology of porous Ti-48Al-6Nb alloys in the 3.0 mol/L NaOH solution is shown in Fig. 3c and d. Compared to the 0.5 mol/L solution case, lots of nano-threads emerge on the nano-porous layers with different orientations. Some nano-threads are grown from one root and some are grown out of order. In order to observe the nano-porous structure more clearly, the higher magnification and fracture photos are presented in Fig. 4. As is shown in Fig. 4a, the nano-pores are covered and connected by each other and most of them are open pores. The pore sizes distributed at 60 ± 10 nm are measured roughly by SEM and the pores present an irregular shape unlike the micron-sized ones with smooth edges as shown in Fig. 3a. Due to the long soaking time (>120 h) in a higher concentration of NaOH solution (e.g. 3.0 mol/L), more nano-threads emerge on nano-porous layers and they are measured roughly the same as the nano-pores size indicating that the nano-threads are grown from inside of the nano-pores (see Fig. 4b). The accurate pore size will be measured by the MIP method later. From the fracture photos shown in Fig. 4c and d, the growth orientation of the nano-porous layers is nearly perpendicular to the Ti-Al matrix with fully lamellar phases and there exist obvious boundaries between them. The nano-porous layers have a uniform thickness of 200 nm,

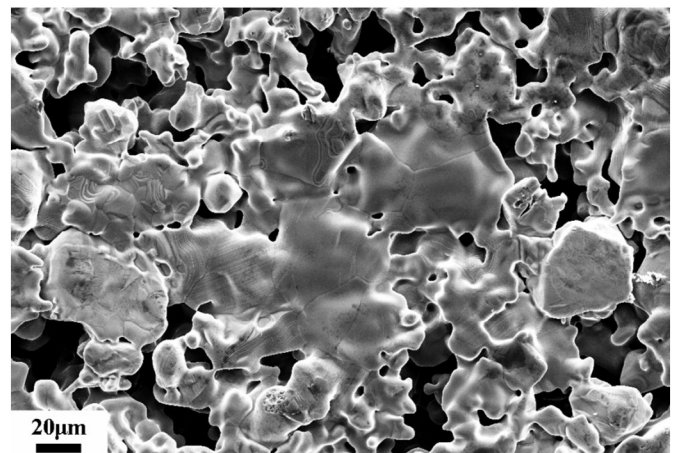


Fig. 2. SEM-SE image of porous Ti-48Al-6Nb alloys.

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