Vacuum 125 (2016) 215–221

Contents lists available at ScienceDirect

Vacuum

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

Microstructure evolution and strengthening mechanisms of pure titanium with nano-structured surface obtained by high energy shot peening

Shijuan Dai [a, *], Yuntian Zhu [b], Zhaowen Huang [a]

[a] Nano Structural Materials Center, Nanjing University of Science and Technology, Nanjing 210094, China
[b] College of Engineering, North Carolina State University, NC 27695, USA

ARTICLE INFO

Article history:
Received 27 November 2015
Received in revised form
31 December 2015
Accepted 3 January 2016
Available online 6 January 2016

Keywords:
Pure titanium
Gradient nanocrystallization
High energy shot peening
Microstructure
Tensile properties

ABSTRACT

In this study, the microstructure evolution and strengthening mechanisms of pure titanium processed by high energy shot peening (HESP) have been studied. The results show that the deformation layer is formed on the surface and the microstructure exhibits with the equiaxed 20–40 μm grains in the matrix after HESP. A nanocrystal surface layer is produced by means of HESP on pure titanium. The formation of nano-grains on the surface can be separated into four steps: (1) the formation of the dislocations tangles; (2) the occurrence of the intersection of twins; (3) the appearance of slip band and subgrains; (4) the formation of uniformly distributed nanometer-scale grains. With increasing the holding time, the strength increases and the elongation decreases due to the work hardening effect and the formation of the nanocrystals on the surface.

1. Introduction

Titanium and its alloys have become excellent metal biomaterials due to their lower modulus, superior biocompatibility and enhanced corrosion resistance compared with stainless steels and cobalt-based alloys [1,2]. So far, the most widely used alloy is pure titanium and Ti–6Al–4V alloy for clinical application, but the latter contains V and Al elements, which exhibit high cytotoxicity [3]. Pure titanium without toxic elements is considered as the one of the best implanted materials [4,5]. However, the strength of pure titanium is lower than that of Ti–6Al–4V, leading the narrower application scope.

The surface nanocrystallization (SNC) is an effective and economical route to improve the mechanical properties of metal materials. A variety of severe plastic deformation processes have been proposed to produce SNC. Shot peening treatment, which can produce nano-grains in the surface layer of the metals, is an effective surface strengthening technology. During the process, the surface of the workpiece is continuously impacted by a number of shot, which causes the severe plastic deformation. Unal et al. [6] studied the microstructure and hardness of AISI 304 austenitic stainless steel performed by different types of shot peening, including air blast conventional shot peening, severe shot peening and repeening. The results showed that the deformation layer had nano-grain size distributions with much higher hardness. Bagherifard et al. [7] studied the microstructure and roughness of cast iron specimens treated by severe shot peening and the results indicated the presence of a highly deformed near surface layer and the surface roughness increases with increasing the impact energy of shot peening process. The above literatures have focused on the microstructure evolution of metal materials after shot peening, but few investigations about the influences of the microstructure evolution on mechanical properties after shot peening have been founded.

In this study, a nano-structured grade surface layer was prepared on the surface of pure titanium by means of HESP technique in order to enhance the strength. The mechanism of grain refinement was discussed and the effect of the formation of the nano-grains on the mechanical properties was also studied.

2. Experimental

The material used in this study was a 4 mm-thickness plate

* Corresponding author. Nano Structural Materials Center, Nanjing University of Science and Technology, 200 Xiao Ling Wei, Nanjing 210094, China.
E-mail address: daishijuan_1982@163.com (S. Dai).

http://dx.doi.org/10.1016/j.vacuum.2016.01.001
0042-207X/

made of pure titanium with the chemical composition in weight percent: 0.10Fe, 0.14O, 0.25Al and balance Ti. The plate was annealed in argon atmosphere at 1023 K for 2 h and then air-cooled, resulting in equiaxed grains averaging 30 μm in size. The shot velocity was 40 m/s and the stainless steel balls with the 3 mm diameter were used as the shots. Both sides of the annealed samples were high energy shot peened at room temperature and the holding time was 15, 30, 45 and 60min respectively.

The cross-section of each sample was mechanically polished using silicon carbide paper and a polishing cloth, and finally etched at room temperature in a solution of 1 ml HF, 3 ml HNO_3 and 7 ml distilled water. Then, the microstructure close to the treated surface was examined by optical microscope (OM). The phase structure of the surface layer was performed using an X-ray diffractometer (XRD, D8 Discover) with Cu Kα radiation and graphite monochromator operated at 40 KV and 40 mA. The micro-strains and average grain sizes of all the samples were calculated according to the XRD pattern using Jade5.0 software. Transmission electron microscopy (TEM) investigations were carried out on FEI Tecnai 20 microscopes operating at 200 KV. The preparation of the TEM foils of the cross-sections was separated into four steps: (1) polishing the cross-section of the sample mechanically until it was about 50 μm thick (Fig. 1(a)); (2) cutting along the dotted line (Fig. 1(b)); (4) sticking on a copper ring with the butt joint of the two treated surface (Fig. 1(c) and (d)); (4) electro-polishing the foils using a twin-jet technique in a solution of 6 ml $HClO_4$, 60 ml CH_3OH and 36 ml C_4H_9OH at a voltage of 30 V and a temperature of 243 K. The mechanical properties of the alloys were obtained by an electronic universal test machine (CMT 5105). The size of the sample was shown in Fig. 2 and the tensile specimens were prepared by using electro-discharging.

3. Results and discussions

3.1. Microstructures

The cross-sectional microstructures of the treated specimens under different conditions are presented together in Fig. 3. It is seen that the grain boundaries have not been indentified and it is called the severe deformation layer which is marked in dotted line. Also, it is observed that the interior is not affected by the deformation and

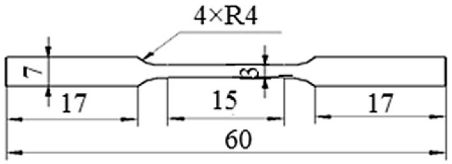

Fig. 2. Size of tensile test specimen.

the microstructure exhibits with the equiaxed 20–40 μm grains. From Fig. 3, it is known that the thickness of the severe deformation layer increases obviously with the increase of the holding time. When the holding time increases, the accumulation of severe deformation causes by the repeated impact of the shots enhances, leading to the increase of the thickness of the severe deformation layer [8].

3.2. XRD investigations

Fig. 4 shows XRD patterns of the samples treated for different holding time. No new peaks appear after HESP, which shows that there is no obvious phase transition after severe plastic deformation. Compared with the specimen before HESP, the diffraction peaks broaden after HESP and the widening of diffraction line profile intensifies with the increase of the holding time, which infers the occurrence of grain refinement and the increasing micro-strain.

The grain size and micro-strain of the samples are calculated from line broadening of Bragg diffraction peaks by using equations (1) and (2) respectively [9,10].

$$\text{Size} = \frac{K\lambda}{\beta}\cos\theta \tag{1}$$

$$\varepsilon = \frac{\beta}{4}\tan\theta \tag{2}$$

Where Size means grain size; K means constant, usually K = 1; λ means X ray wavelength; β means the full width at half maximum of the peak; θ is the Bragg angle of the [h k l] reflection; ε means micro-strain. Fig. 5 shows the average grain size and the micro-

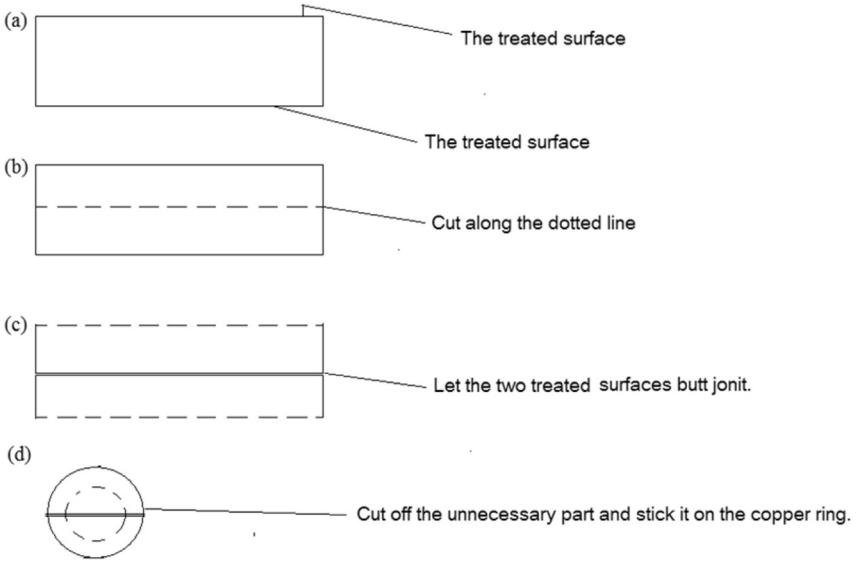

Fig. 1. Preparation process of TEM foils.