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Microstructural response and grain refinement mechanism of commercially pure titanium subjected to multiple laser shock peening impacts

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ABSTRACT

The microstructural response and grain subdivision process in commercially pure (CP) titanium subjected to multiple laser shock peening (LSP) impacts were investigated by means of optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) observations. The micro-hardness curves as a function of the impact time were also determined. The deformation-induced grain refinement mechanism of the close-packed hexagonal (hcp) material by laser shock wave was subsequently analyzed. Experimental results showed that uniform equiaxed grains with an average size of less than 50 nm were generated due to the ultra-high plastic strain induced by multiple LSP impacts. Special attention was paid to four types of novel deformation-induced microstructural features, including a layered slip band in the tension deformation zone, and inverse-transformation martensite, micro-twin grating and micro-twin collision in the compression deformation zone. Furthermore, the grain refinement mechanism in the near-surface layer of CP titanium subjected to multiple LSP impacts contains two types of simultaneous subdivision modes: multi-directional mechanical twin (MT)-MT intersections at (sub)micrometer scale, and the intersection between longitudinal secondary MTs and transverse dislocation walls at nanometer scale. In addition, both grain refinement (nanocrystallization) and the existence of a small amount of inverse-transformation martensite induced by multiple LSP impacts contribute to an increase in the micro-hardness of the near-surface layer.

1. Introduction

Titanium and titanium alloys are widely used in aerospace, chemical and petrochemical industries, naval crafts, as well as medical and dental implants [1–4]. Among these alloys, commercially pure (CP) titanium is an especially attractive candidate material in chemical and biomedical engineering, due to high strength-to-weight ratio, good ductility and excellent exceptional biocompatibility [2,5]. However, relative low strength, micro-hardness, as well as poor wear resistance, of CP titanium also limit its extensive application.

Over the years, surface treatment techniques by severe plastic deformation with high strain rate, such as high energy shot peening [6], cold rolling [7], water cavitation peening [8], and surface mechanical attrition treatment (SMAT) [9], have been utilized to improve its mechanical properties by substantially refining coarse grains in the surface layer of CP titanium. Compared with these traditional surface treatment technologies, laser shock peening (LSP) is a novel surface treatment method to improve the fatigue durability, corrosion resistance and other mechanical properties of metallic materials and alloys, which is also applied to surface modification of CP titanium [10–13]. For instance, the effects of LSP on the micro-hardness, residual stress, and fatigue life of near α titanium (Ti834) were investigated, and the high-cycle fatigue (HCF) life of LSPed specimens has been increased by 54% due to the introduction of compressive residual stress delaying the initiation and growth of fatigue cracks [10]. At room temperature, LSP caused the formation of high-density dislocation and dislocation-cell of CP titanium, and therefore brought an obvious improvement in the

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ultimate tensile strength (UTS) [14]. HCF performance of a near α titanium (Ti–2.5Cu) was impressively improved, depending strongly on the greater amount and depth of compressive residual stresses in the surface layer after LSP without coating [15]. These investigations have mainly focused on the mechanical properties of CP titanium subjected to LSP with different parameters. In fact, decreasing grain size leads to increasing yield strength and micro-hardness of metallic materials and alloys. Previous investigations have demonstrated that the strength and micro-hardness of metals with ultra-fine grains (100 nm < grain size < 1 μm) or nano-crystalline grains (grain size < 100 nm) are significantly enhanced over those of their coarse-grained counterparts [16]. An almost six-fold improvement in the nano-hardness of CP titanium can also be found due to the refined grains in the surface layer by micro-twins and dislocation cells generated by laser shock wave [17].

Numerous reports are available in the literature of the occurrence of mechanical twins (MTs) and dislocation activities generated by severe plastic deformation. For example, a thin lath structure formed by parallel MTs at micrometer scale was found in the surface layer of cold-rolled CP titanium [18]. MT-MT intersections with two directions associated with high-density dislocations among the MT bands were also observed in the deformation layer treated by water cavitation peening [8]. Meanwhile, LSP lead to the formation of high-density dislocation cells produced from dislocation arrangement in the surface layer of CP titanium [14]. CP titanium has a close-packed hexagonal (hcp) structure, and its deformation response is generally guided by the axial (c/a) ratio [19]. Hence, the plastic deformation behavior of CP titanium is very complex due to the activation of different types of slips and twinning systems under different conditions.

Our previous work systematically presented the grain refinement mechanisms of face-centered cubic (fcc) metals generated under the mechanical effect of multiple LSP impacts, such as LY2 Al alloy [20] and AISI304 stainless steel [21]. Compared with fcc metals, the grain refinement mechanism of hcp materials subjected to multiple LSP impacts lags well behind, and the microstructural response in the surface layer during multiple LSP impacts is still pending. In addition, laser shock wave causes the formation of a compression deformation zone and a tension deformation zone in the surface layer of metallic materials and alloys, and there are different microstructural characteristics in both zones. However, up to now, much less attention has been paid to these topics. Hence, microstructural response and grain refinement mechanism of multiple LSP impacts on hcp material are worthy of investigation.

The aim of this paper is to investigate the effects of multiple LSP impacts on the microstructural response and grain subdivision process in the near-surface layer of CP titanium. Micro-hardness curves as a function of the impact time are also determined. Subsequently, four types of novel microstructural features induced by plastic deformation are identified. Special attention is paid to the plastic strain-induced grain refinement mechanism of hcp materials subjected to multiple LSP impacts. Finally, the underlying micro-hardness enhancement mechanism induced by multiple LSP impacts on CP titanium is systematically revealed.

2. Experimental procedures

2.1. Experimental material and LSP parameters

CP titanium with an average grain size of 10–15 μm, supplied by Xi'an Aerospace New Materials Co., Ltd, was used as the specimen material. The chemical composition of CP titanium was 0.15 wt% O, 0.05 wt% H, 0.05 wt% N, 0.10 wt% C, 0.30 wt% Fe, and balanced Ti. All specimens were cut into a rectangle shape with dimensions of 10 mm × 10 mm × 2 mm (width × length × thickness) from the same plate. Prior to LSP treatment, all specimen surfaces to be treated were mechanically gritted using SiC paper with different grades of roughness (from 800 to 2400) and polished to a mirror surface (Ra~0.03 μm), followed by cleaning in deionized water. Ultrasound in ethanol was used to degrease the specimen surface, followed by LSP treatment.

LSP experiments were carried out using a Q-switched Nd: YAG (GAIA-R, France Thales Co., Ltd) laser system operating at 1064 nm with a flat-top (Super-Gaussian distribution) pulse width of 10 ns and repetition–rate of 5 Hz. In the present work, the laser pulse energy was set to be 10 J, and the laser spot diameter is set to 3 mm. During LSP treatment, a water layer of ~1 mm in thickness was used as the transparent confining layer. A professional aluminum tape of 100 μm in thickness (Made in USA) was used as an absorbing layer to protect the specimen surface from the thermal effect. Furthermore, the laser beam was always kept perpendicular to the specimen surface. For multiple LSP impacts, the same location at the top surface was repetitively beaten by multiple laser pulses, and the absorbing layer was also replaced after each LSP impact.

2.2. Micro-hardness measurement

Micro-hardness tests of as-machined specimens and LSPed specimens were carried out using a HXD-1000TMSC/LCD Vickers indenter (Shanghai Taiming Optical Instrument Co. Ltd.) under different loads and a 10-s hold time. An average micro-hardness value has been determined on the basis of the measured data from 5 indentations at each point. For the measurement of micro-hardness in the depth direction, the electrolytic polishing material removal method was used, and the thickness of each removed layer was measured using a precise micrometer.

2.3. Microstructural observations

After LSP treatment, two types of LSPed specimens with different impact times used for metallographic investigation were cut as the sections perpendicular to the specimen surface. They were then subjected to several successive steps of grinding and polishing. Cross-sections of these specimens were etched using a professional reagent that consists of 10% HF, 5% HNO_3, and 85% H_2O and then characterized by scanning electron microscopy (SEM) and cross-sectional optical microscopy (OM) observations.

Microstructural evolutions at different depths of both LSPed specimens were characterized using a JEM-2100 TEM operated at a voltage of 200 kV. The thin foils at different depths of the deformation layer for TEM observations were prepared in the following steps: (i) polish the top surface to a given depth and then cut off one piece (1.5 mm × 1.5 mm × 10 mm in size) from the opposite side, (ii) put it into a 3-mm-in-diameter copper tube and bond them together, (iii) grind it carefully to approximately 30 μm in thickness via SiC paper with different grades of roughness (from 1800 to 3000), and (iv) dimple and ion-thin it to perforation at room temperature. Typical micro-structures at different depths of the LSPed specimens are observed.

3. Results and discussion

3.1. Cross-sectional grain arrangement and micro-structure after LSP treatment

According to the previous research [21–23], it is well known that with decreasing strain rate from the maximum (up to 10^6 s^{-1}) at the top surface to zero in the strain-free matrix after LSP treatment, the microstructural evolution process may be signed by the microstructural characteristics (with different strain rates and