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Mechanical properties of strengthened surface layer in Ti−6Al−4V alloy induced by wet peening treatment

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Abstract: A modified surface layer was formed on Ti−6Al−4V alloy by wet peening treatment. The variations of the residual stress, nano-hardness and microstructure of the modified layer with depth from surface were studied using X-ray diffraction analysis, nano-indentation analysis, scanning electron microscopy and transmission electron microscopy observations. The results show that both the compressive residual stress and hardness decrease with increasing depth, and the termination depths are 160 and 80 μm, respectively. The microstructure observation indicates that within 80 μm, the compressive residual stress and the hardness are enhanced by the co-action of the grain refinement strengthening and dislocation strengthening. Within 80–160 μm, the compressive residual stress mainly derives from the dislocation strengthening. The strengthened layer in Ti−6Al−4V alloy after wet peening treatment was quantitatively analyzed by a revised equation with respect to a relation between hardness and yield strength.

Key words: Ti−6Al−4V alloy; wet peening; nano-hardness; compressive residual stress; local yield strength

1 Introduction

Ti−6Al−4V alloy is applied widely in aerospace and biomedical industries because of its high specific strength and low density [1]. The fatigue resistance of Ti−6Al−4V alloy is a key factor in these applications. Shot peening is a normal surface impact treatment that is used to improve fatigue resistance [2,3]. Previous studies have proposed various benefits of shot peening (SP) method on fatigue performance, such as the compressive residual stress, hardness and grain refinement [4−6]. Among these benefits, the formation of compressive residual stress on the surface layer plays an important role on fatigue property improvement, which retards the nucleation of surface cracks and delays crack propagation [7,8]. Thus, numerous studies have focused on improving compressive residual stress of titanium alloy by many impact methods. AMANOV et al [9] applied the ultrasonic nanocrystalline surface modification (UNSM) technique to commercially pure titanium (CP Ti) (the yield stress, σ_y=275 MPa) and Ti−6Al−4V (σ_y=970 MPa) alloy materials to improve their compressive residual stress. In the research, the highest compressive residual stress of CP Ti and Ti−6Al−4V alloy reached up to −1279.4 and −1142.7 MPa, respectively. In addition, LEE et al [10] applied the cavitation shot peening (CSP) technique to Ti−6Al−4V alloy (σ_y=930 MPa), and obtained a compressive residual stress of −1000 MPa. In particular, there is a phenomenon that compressive residual stress is higher than yield stress, which has not been explained in these reports. This phenomenon must be related to the strengthening process of impact modified layer. However, the detailed impact strengthening mechanism of titanium alloy has been rarely discussed.

In the aspects of impact strengthening mechanism investigation, TAO et al [11] investigated the grain refinement mechanism of pure Fe by surface mechanical attrition treatment method. The experimental results indicated that the mechanism involved formation of dense dislocation walls (DDWs) and dislocation tangles (DTs), transformation of DDWs and DTs into sub-boundaries, and evolution of sub-boundaries to grain boundaries. Moreover, HOU et al [12] proposed a similar microstructural evolution process for AZ91D magnesium alloy induced by high-energy shot peening, and considered that the refinement process consisted of the

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simultaneous co-actions of two mechanisms, namely, dislocation slipping and dynamic recrystallization operation. These mechanisms indicate that the mechanical properties (e.g., yield stress) can be enhanced during the impact process. By now, the strengthening mechanisms have been put forward through numerous experimental observations on various materials qualitatively [13,14]. The quantitative analysis for the variation of the mechanical property with depth has not been conducted.

Compared with other SP methods, the wet peening (WP) technique is more economical and produces less amount of waste, and results in a lower surface roughness owing to the protection provided by the water film when processing the blade/disk components [6,15]. In the present paper, WP treatment, which is a more environmental impact technique, was employed to form a modified surface layer of Ti−6Al−4V alloy. X-ray diffraction (XRD) and nano-indentation were used to investigate the residual stress and nano-hardness varying with depth from the surface of Ti−6Al−4V alloy subjected to WP treatment. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to investigate the surface strengthening mechanism of WP. Based on these experimental results, an equation which can quantitatively describe the variation of local yield stress with depth was obtained to explain the phenomenon that compressive residual stress is higher than yield stress.

2 Experimental

In this study, a cold-rolled plate of Ti−6Al−4V (3 mm in thickness), with chemical compositions (in mass fraction) of 6% Al, 4% V, 0.3% Fe, 0.2% O, 0.1% C, 0.05% N, 0.015% H and rest of Ti, was firstly polished, and then treated with WP method. The yield and tensile stresses of the initial plate targeted for stress−strain test were approximately 820 and 900 MPa, respectively. All samples were peened with the mixture of ceramic beads (600 μm in mean diameter, 7 GPa in hardness) and water in the scale of 10% (mass fraction) to Almen target intensity of 0.28 mm(N), using 0.35 MPa of air pressure. The jet rate of the mixture was approximately 1100 mL/s. The residual stress and hardness responses of these samples subjected to WP were tested by XRD (XRD−6000, Shimadzu, Japan) and nano-indentation (nano-indenter XP with Berkovich diamond tip, HM2000S, Fischer, Germany), respectively. The XRD high voltage was adjusted for 40 kV, scan range from 20° to 120°, scan step of 0.016711°. Through calculating the peak shift, using the system function about residual stress calculation, the residual stress was calculated. Firstly, the surface was etched by a solution of with mass ratio $m(HF)$:$m(HNO_3)$:$m(H_2O)$ of 2:4:94 to remove the top layer, and the residual stress was tested. Secondly, the surface was polished to remove the etched layer, and the nano-hardness (H) was then tested. The circling test was repeated until the depth of the layer without influence of WP. Each delaminated thickness was measured with a micrometer during the etching and polishing processes. SEM (Zeiss, Supra 55) and TEM (Tecnai G2 20 s Twin) were employed to investigate the micro-structure of the peened layer with different depths.

3 Results and discussion

Figure 1 shows the evolution of residual stress and nano-hardness (H) as a function of depth from surface. Both compressive residual stress and H decreased gradually with the increase in depth. It is worthy to note that, within the depth range from surface to 80 μm, the compressive residual stress is higher than the yield stress of the original material. And in such a depth range, the nano-hardness decreases rapidly from 5.65 to 4.5 GPa; when the depth is beyond 80 μm, the decreasing trend becomes saturated. Thus, the depth of 80 μm is a particular site for both the residual stress and H.

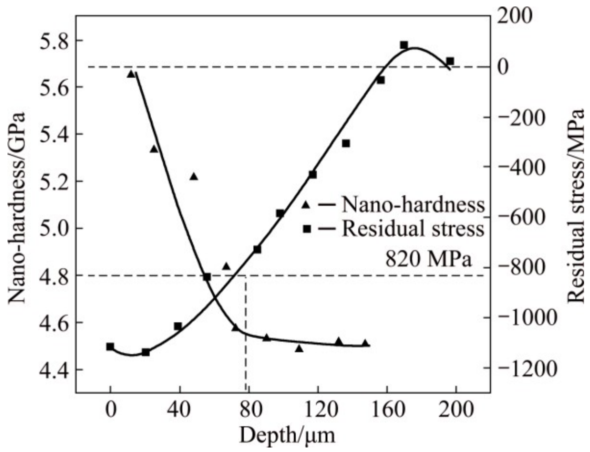

Fig. 1 Residual stress and nano-hardness variation of WP samples with distance from surface to substrate

3.1 Microstructure evolution

To further investigate the phenomena mentioned above, the microstructures with varying depths are given in Fig. 2. The signs of b, c, d and e in Fig. 2(a) are the observation locations of the cross-sectional TEM sample, and the distances of these locations to the interface (surface) are about 0, 20, 80, and 500 μm, respectively. As shown in Fig. 2, the domain size at the depth of 0 μm is about 20 nm (Fig. 2(b)), and increases with increasing depth. When the depth increases to 20 μm, a sub-grain, about 30 nm (Fig. 2(c), a misorientation angle of 2.57°), which can evolve into a fine grain, was found to form

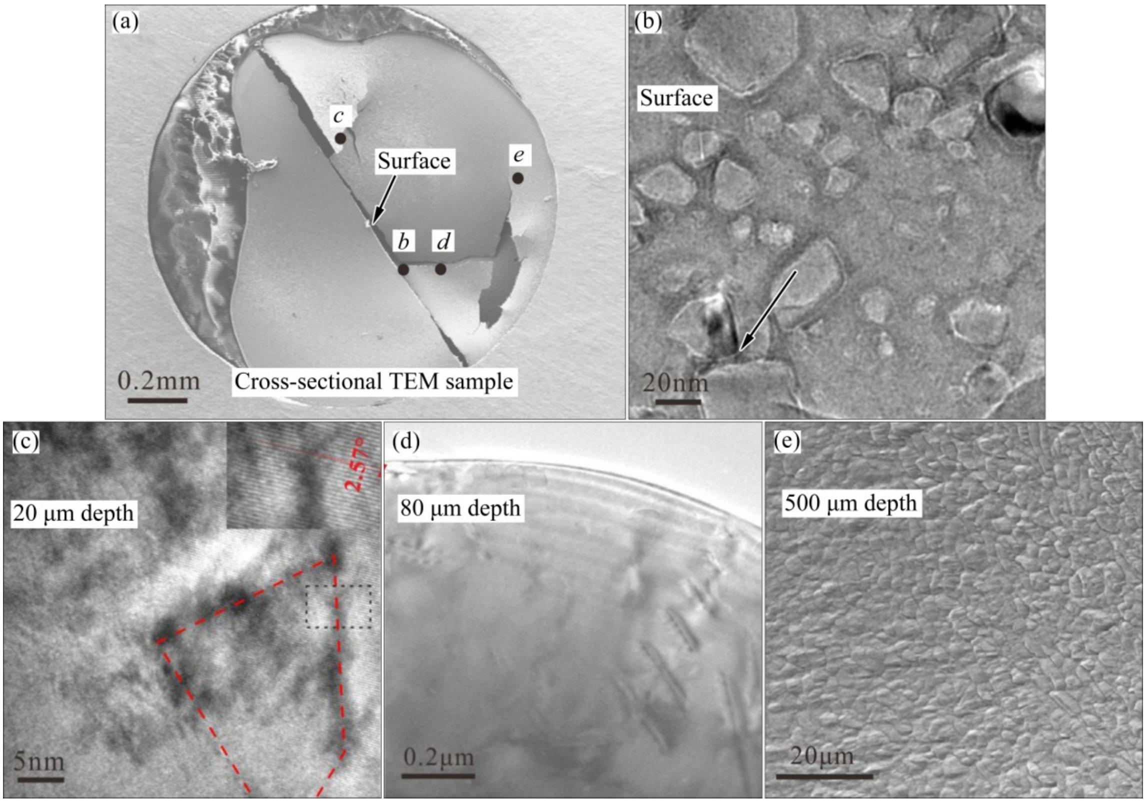

Fig. 2 Gradual change of microstructure in depth of WP sample: (a) SEM image of TEM sample; (b) TEM image of WP surface; (c) HRTEM image at depth of about 20 μm; (d) TEM image showing dislocation piling-up at depth of about 80 μm; (e) SEM image of location at depth of about 500 μm, which was not affected by WP

through dislocation sliding and pile-up. On the consideration that the location at a depth of 80 μm was an inflection point of the *H* profile in Fig. 1, thus, the microstructure at about 80 μm depth was detected, as shown in Fig. 2(d). Grain or sub-grain boundaries were not observed, except for some piling-up dislocations at this depth. The microstructure of the substrate was observed with SEM, and the domain size was about 3 μm, as illustrated in Fig. 2(e).

3.2 Strengthening mechanism

The residual stress and hardness test results, as well as the TEM results, showed that both the residual stress and hardness decrease, whereas the grain size increases, with the increase in depth. Grain refinement strengthening seems to dominate the enhancement on residual stress and hardness. However, no independent refined grains were observed in the depth range of 20−80 μm, except for some sub-grains and dislocations piling-up induced by dislocation activities. Meanwhile, some residual stresses still exist until the depth of 160 μm, suggesting that dislocation strengthening plays another strengthening role in the strengthening process.

Based on the surface strengthening mechanism proposed by TAO et al [11], the microstructural evolution process of Ti−6Al−4V alloy induced by WP treatment was deduced (shown in Fig. 3). Firstly, dislocation activities led to the formation of dislocation pile-up in original grains in the surface layer (Step I). Secondly, the dislocation pile-up transformed into dislocation cells (Step II). The accumulation of dislocations at the cell boundaries continuously increased through dislocation sliding with the increase of the plastic deformation. Sub-grain boundaries with low misorientations then emerged through dislocation accumulation to minimize the system energy [11] (Step III). Finally, the sub-boundaries evolved to high misorientation boundaries through the increase of the plastic deformation, and the refined grains with individual boundaries formed (Step IV). This kind of refinement process will repeat and result in nanostructure formation in the surface layer. Some clear evidences supporting this mechanism are proposed in Fig. 2, such as those final refinement grains (20 nm) in the surface layer, a few sub-grains at depth of about 20 μm, and the dislocation pile-up at depth of about 80 μm. In addition, activated dislocation occurred in matrix with plastic deformation (80−160 μm in depth) where no change in grain size was observed [11]. Consequently, the modified layer should be composed of the fine grain layer at depths of 0−20 μm and the dislocation activity layer at depths of 20−160 μm.

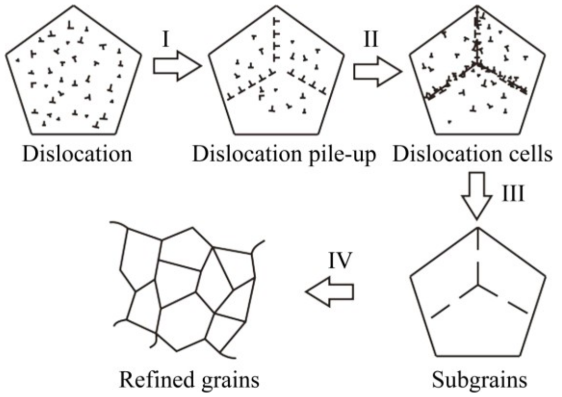

Fig. 3 Schematic illustration showing refinement mechanism of Ti−6Al−4V alloy induced by WP treatment

3.3 Quantitative analysis for strengthened layer

In view of the mechanism above, taking Fig. 1 into consideration, the dislocation pile-up occurred at depth of 80 μm, which is a transition site. High dislocation accumulation occurs before the site, whereas only dislocation activation occurs beyond the site. As shown in Fig. 1, the depth of 80 μm is also a transition site for the hardness, in which the value increased before the site and the decreasing trend became saturated beyond it. However, the residual stress still had effect even at approximately depth of 160 μm. Thus, hardness was highly influenced since the dislocation pile-up step and weakly influenced in the activation step. And the residual stress could more sensitively respond to the lattice deformation induced by the activation dislocations at the depth of 80−160 μm than the hardness.

Combining the above strengthening mechanism and that from a previous study [14], the yield stress can be enhanced locally in this strengthened layer. To quantitatively analyze the local yield stress, a model can be reasonably put forward, as seen in Fig. 4(a). The strengthened layer is regarded as many independent thin plates with different mechanical properties compositing in together. The thickness of each "plate" is equal to the mean cell size corresponding to the depth. Based on the model and the experimental results shown in Fig. 1, some calculations can be conducted. In the calculation process, each plate was assigned with corresponding values of residual stress and nano-hardness. Thus, the strengthened layer can be separated into individual ones to investigate the local yield stress. The hardness value decreases linearly from plate 1 to plate *n*.

Yield stress increases with the increase of hardness [16–21]. For the quantitative relationship between yield stress and depth, previous studies have liner relationships between yield stress σ_y and hardness H_v, which is given by [20]

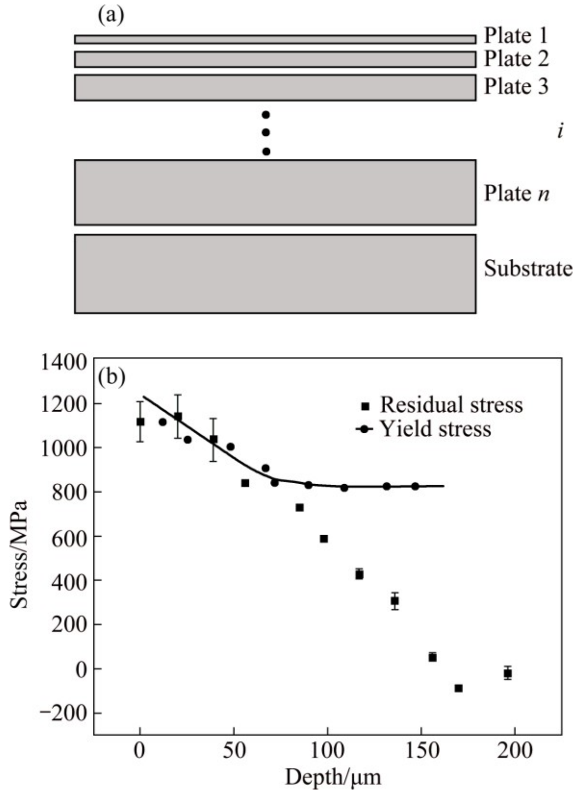

Fig. 4 Separated plate model of WP layer (a) and variation of residual stress and yield stress WP sample with depth (b)

$$\sigma_y = 2.5(H_v - 68) \quad (1)$$

where σ_y is in MPa and H_v is in kg/mm^2. In this paper, the relationship is fixed with minor revisions because of the variation of different research objectives according to the experimental results, and the current relationship can be expressed as follows:

$$\sigma_y = 2.5(H - 68) - 157.5 \quad (2)$$

The revised constant −157.5 was obtained by the substrate mechanical property (σ_y=820 MPa and H= 459 kg/mm^2 (4.5 GPa)). Substituting the corresponding value of H into Eq. (2) and fitting, the relationship between yield stress σ_y and depth D is displayed in Fig. 4(b). The σ_y of the corresponding plate decreases linearly from the surface to depth of 80 μm as follows:

$\sigma_{y1} > \sigma_{y2} > \sigma_{y3} > \cdots > \sigma_{yn} > \cdots \sigma_y$ (80 μm)=820 MPa.

Notably, the local yield stress decreases linearly with the increase of depth in the strengthened layer, and the decreasing trend becomes saturated at depth of approximately 80 μm. Moreover, through the comparison between the residual stress and local yield stress, the residual stress was found to be approximately equal to the local yield stress in the 0−80 μm strengthened layer; the difference between the σ_y and residual stress gradually increased with the increase of depth in the range of 80−160 μm. It is well known that the yield stress should be lager than the residual stress to avoid the material deformation or failure. In this work (Fig. 4(b)), the phenomenon that the residual stress is higher than 820 MPa is reasonable within the depth range from surface to 80 μm because the local yield stress is significantly improved by WP treatment. That is to say, the WP treatment could efficiently increase the "residual stress tolerance" in the first 80 μm depth of the strengthened layer. And the maximum value of the compressive residual stress which could benefit the fatigue resistance is increased to approximate saturation degree in this investigation.

4 Conclusions

1) The thickness of the modified layer was approximately 160 μm, and the seriously strengthened layer was above 80 μm in depth. The strengthened layer is quantitatively calculated by an equation which is revised and used in Ti−6Al−4V alloy.

2) At 80 μm<D<160 μm, activated easy-slip dislocations have weak influence on the mechanical properties (i.e., σ_y and H) of the material. However, these dislocations accelerate the lattice deformation, thereby inducing residual stress.

3) At 20 μm<D<80 μm, the effective dislocation strengthening exists from dislocation piling-up to sub-grain forming. Both the yield and residual stresses increase linearly in this process. The residual stress is approximately equal to the local yield stress of the corresponding depth.

4) In conditions of 0 μm<D<20 μm, grain refinement strengthening plays a dominant role. The microstructures transform from sub-grains to nano-grains. The value of σ_y and yield stress increase simultaneously until the final refinement.

References

[1] KUMAR S A, SUNDAR R, RAMAN S G S. Influence of laser peening on microstructure and fatigue lives of Ti−6Al−4V [J]. Transactions of Nonferrous Metals Society of China, 2014, 24: 3111−3117.

[2] SHENG Xiang-fei, XIA Qin-xiang, CHENG Xiu-quan, LIN Lie-shu. Residual stress field induced by shot peening based on random-shots for 7075 aluminum alloy [J]. Transactions of Nonferrous Metals Society of China, 2012, 22(S): s261−s267.

[3] ZHANG Bing, CAO Xue-gang, LIU De-lin, LIU Xin-ling. Surface recrystallization of single crystal nickel-based superalloy [J]. Transactions of Nonferrous Metals Society of China, 2013, 23: 1286−1292.

[4] HU Lan-qing, WANG Ke, LIU Gang, XU Bing-she. Microstructure characteristics of as-surface nanocrystallized 1420 aluminum alloy by high-energy shot peening [J]. Transactions of Nonferrous Metals Society of China, 2005, 15: 615−618.

[5] ZHANG Jin, OU Xin-bing. Thermal stability of nanocrystalline in surface layer of magnesium alloy AZ91D [J]. Transactions of Nonferrous Metals Society of China, 2010, 20: 1340−1344.

[6] CHEN Guo-qing, JIAO Yan, TIAN Tang-yong. Effect of wet shot peening on Ti−6Al−4V alloy treated by ceramic beads [J]. Transactions of Nonferrous Metals Society of China, 2014, 24: 690−696.

[7] WITHERS P J. Residual stress and its role in failure [J]. Reports on Progress in Physics, 2007, 70: 2211−2264.

[8] THOMAS M, JACKSON M. The role of temperature and alloy chemistry on subsurface deformation mechanisms during shot peening of titanium alloys [J]. Scripta Materialia, 2012, 66: 1065−1068.

[9] AMANOV A, CHO I S, KIN D E. Fretting wear and friction reduction of CP titanium and Ti−6Al−4V alloy by ultrasonic nanocrystalline surface modification [J]. Surface and Coatings Technology, 2012, 207: 135−142.

[10] LEE H, MALL S, SOYAMA H. Fretting fatigue behavior of cavitation shotless peened Ti−6Al−4V [J]. Tribology Letters, 2009, 36: 89−94.

[11] TAO N R, WANG Z B, TONG W P, SUI M L, LU J, LU K. An investigation of surface nanocrystallization mechanism in Fe induced by surface mechanical attrition treatment [J]. Acta Materialia, 2002, 50: 4603−4616.

[12] HOU Li-feng, WEI Ying-hui, LIU Bao-sheng, XU Bing-she. Microstructure evolution of AZ91D induced by high energy shot peening [J]. Transactions of Nonferrous Metals Society of China, 2008, 18: 1053−1057.

[13] LIU G, WANG S C, LOU X F, LU J, LU K. Low carbon steel with nanostructured surface layer induced by high-energy shot peening [J]. Scripta Materialia, 2001, 44: 1791−1795.

[14] CHEN X H, LU J, LU L, LU K. Tensile properties of a nanocrystalline 316L austenitic stainless steel [J]. Scripta Materialia, 2005, 52: 1039−1044.

[15] GREEN R G, TAYLOR R W. The instrumentation and control of a wet peening process [J]. Journal of Physics E (Scientific Instruments), 1986, 19: 110−115.

[16] LI H Q, EBRAHIMI F. Synthesis and characterization of electrodeposited nanocrystalline nickel-iron alloys [J]. Materials Science and Engineering A, 2003, 347: 93−101.

[17] LU L, SCHWAIGER R, SHAN Z W, DAO M, LU K, SURESH S. Nano-sized twins induce high rate sensitivity of flow stress in pure copper [J]. Acta Materialia, 2005, 53: 2169−2179.

[18] SANDERS P G, YOUNGDAHL C J, WEERTMAN J R. The strength of nanocrystalline metals with and without flaws [J]. Materials Science and Engineering A, 1997, 234: 77−82.

[19] HIGGY H R, HAMMAD F H. Effect of fast-neutron irradiation on mechanical-properties of stainless-steels—AISI types 304, 316 and 347 [J]. Journal of Nuclear Materials, 1975, 55: 177−186.

[20] SUZUKI S, TANAKA Y, KODAMA M, NISHIMURA S, FUKUYA K, SHIMA S, NAKATA K, KATO T. Mechanical properties and SSRT results of various kinds of irradiated stainless steels [C]//Proceedings of the 12th ICG-IASCC Meeting. Toronto, Canada, 1996.

[21] AN X H, WU S D, ZHANG Z F, FIGUEIREDO R B, GAO N, LANGDON T G. Evolution of microstructural homogeneity in copper processed by high-pressure torsion [J]. Scripta Materialia, 2010, 63: 560−563.

湿喷丸处理 Ti−6Al−4V 合金强化层的力学性能

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摘 要：采用湿喷丸处理方法在 Ti−6Al−4V 合金表面制备了改性层。利用 X 射线衍射技术、纳米压痕技术、扫描电镜以及透射电镜对合金改性层的残余应力、硬度以及显微组织随深度的变化进行研究。结果表明，改性层的残余压应力和硬度均随深度增加而减小，影响深度分别为 160 和 80 μm。通过显微组织观察发现，在 80 μm 深度范围以内，残余压应力和硬度的增加是由细晶强化和位错强化共同主导的；在 80~160 μm 深度范围内，残余压应力由位错强化主导。通过对硬度与屈服关系的公式进行修正，深入研究了 Ti−6Al−4V 合金湿喷丸处理后的强化层。

关键词：Ti−6Al−4V 合金；湿喷丸；纳米硬度；残余压应力；局部屈服强度

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