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ARTICLE

Microstructure Evolution and Mechanical Properties of SLM Pure Titanium by ECAP

Luo Lei[1,2], Duan Ximing[1], Yang Xirong[1,2], Liu Xiaoyan[1,2], Wang Wen[1,3], Han Peng[1]

[1] School of Metallurgical Engineering, Xi'an University of Architecture and Technology, Xi'an 710055, China; [2] National Experimental Teaching Demonstration Center of Metallurgical Technology, Xi'an 710055, China; [3] National and Local Joint Engineering Research Center for Functional Material Processing, Xi'an 710055, China

Abstract: The pure titanium prepared by selective laser melting (SLM) was modified by equal channel angular pressing (ECAP). The pure titanium prepared by SLM was subjected to single-pass deformation modification at room temperature by a die with the angle of Φ=120° and ψ=20° between two channels, and its microstructure and mechanical properties were evaluated. The results show that the microstructure of SLM+ECAP pure Ti sample is refined, the grain size decreases from 13 μm to 7 μm, and the dislocation density increases. During the ECAP deformation process, twinning and continuous dynamic recrystallization (CDRX) occur simultaneously. The appearance of tensile and compression twin and the increase of dislocation density together promote the microhardness of SLM+ ECAP pure Ti samples, increased by 13%, and YS and UTS are increased by 18% and 20.4% respectively, while the elongation decreases slightly.

Key words: equal channel angular pressing; selective laser melting; microstructure; mechanical properties

Titanium and titanium alloys are widely used in aerospace, biomedicine, automotive and chemical fields because of their low density, high specific strength, high temperature resistance, corrosion resistance and other advantages[1-7]. The titanium structural parts prepared by the traditional casting process have the problems of shrinkage cavity, inner hole, rough and uneven surface, easy cracking, poor precision, etc, which deteriorate performances, and make it difficult to meet the application needs of the current industry. Furthermore, the casting process consumes a lot of energy, the production cycle is long, the material utilization rate is low, and it is difficult to form complex parts, which restricts the application and development of titanium alloys[8-11]. Therefore, it is necessary to adopt a new technology to prepare titanium and titanium alloys with more optimized properties.

Selective laser melting (SLM) is a new type of additive manufacturing technology, Which not only breaks the limitation of the traditional preparation process on the geometric shape of the workpiece, but also can easily and accurately obtain personalized, integrated, and functionalized complex three-dimensional structure[12-14]. However, in the SLM process, due to the rapid melting and cooling of the material, the component has solidification defects such as large deformation, easy cracking, and many pores, which damage the normal function and structural integrity of the component.

Equal channel angular extrusion (ECAP) does not change the cross-sectional shape and area of the sample, so as to obtain a large cumulative strain, thus realizing the refinement, homogenization and densification of the microstructure of the sample[15,16], and thereby improving the mechanical properties and corrosion properties of the sample. For example, Naseri et al[17] used room temperature tensile, three-point bending, Charpy impact and Vickers microhardness test methods to compare the mechanical properties of CP-Ti before and after ECAP modification. The results showed that after ECAP deformation for three times, the yield strength of the ECAP samples was increased from 174 MPa to 273 MPa, the tensile strength increased from 396 MPa to 715 MPa, and the microhardness increased from 1530 MPa to 2470 MPa. In

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Corresponding author: Luo Lei, Ph. D., Associate Professor, School of Metallurgical Engineering, Xi'an University of Architecture and Technology, Xi'an 710055, P. R. China, E-mail: luolei@xauat.edu.cn

addition, the ultimate bending strength of ECAP samples was increased from 664 MPa to 1275 MPa, and there was no obvious change in Charpy impact energy. Mishnaevsky et al[18] performed 6 passes of ECAP treatment for the titanium rod, and found that the ECAP samples showed equiaxial crystal grains with an average grain size of 150 nm, and the yield strength and tensile strength were 1190 and 1250 MPa, respectively.

Up to now, there are few reports on the modification of SLM pure Ti by ECAP. The difference in the microstructure of pure Ti obtained by SLM and traditional preparation technology will inevitably lead to differences in the microstructure after ECAP modification. Therefore, it is necessary to clarify the effect of ECAP on the microstructure and mechanical properties of SLM pure Ti. In this study, SLM pure Ti was modified by ECAP, and its microstructure and mechanical properties were characterized.

1 Experiment

Metal powder with particle size of 25~45 μm was prepared from industrial-grade pure titanium powder by gas atomization method, and the chemical composition was Ti-0.03Fe (wt%). In the SLM process, high-purity argon was used as the protective gas to prepare CP-Ti rods with a size of *Φ*20 mm. SLM pure Ti bar was cut into a bar sample with the size of *Φ*20 mm×200 mm. The single pass ECAP extrusion was carried out by the die with the channel angle of *Φ*=120° and the outer fillet *ψ*=20° at room temperature. The extrusion speed was 2.5 mm·s^{-1}, and MoS_2 and graphite were used as lubricants.

The sample with the size of 0.5 mm× *Φ*10 mm was cut along TD direction, as shown in Fig. 1a. After grinding with different specifications of sandpaper, the double jet thinning was carried out in the environment of methanol: n-butanol: perchloric acid=60: 30: 5. The microstructure and grain size, orientation difference angle and grain orientation of the center position of the SLM pure Ti and SLM + ECAP pure Ti samples was analyzed by TEM and EBSD technology. Low angle and high angle grain boundaries (LAGBs/ HAGBs) are defined as orientation angles of 2°~15° and >15°, respectively.

The longitudinal section (TD-ND plane) of SLM pure titanium and SLM+ECAP pure titanium specimens were cut on a 12 mm×12 mm plane for microhardness test; tensile test was conducted at room temperature with an initial strain rate of 1×10^{-3} s. Fig. 1b and Fig. 1c are the hardness point diagram and the tensile sample size diagram, respectively. Three parallel samples are selected for each group of tensile samples to ensure data reliability. After the tensile test, the fracture surface was observed by SEM technology.

2 Results and Discussion

2.1 Microstructural evolution

Fig. 2 shows the TEM microstructure of SLM pure Ti and SLM+ECAP pure Ti samples. It can be seen from Fig. 2a and 2b that the SLM pure Ti sample has clear grain boundaries, unevenly distributed dislocation lines in the grains and high dislocation density near the grain boundaries; while the SLM+ECAP pure Ti sample in Fig. 2c and 2d shows elongated lath structure; the lath boundary is straight, and there is a small amount of equiaxed cell structure. The dislocation density at the cell wall is higher, and there is almost no dislocation in the cell, as shown by the red and white arrows in Fig. 2d, respectively, indicating that SLM pure Ti undergoes dynamic recovery during the single-pass ECAP deformation process at room temperature, and there are needle-like contracture crystals of different sizes, as shown by the white arrow in Fig. 2c.

Fig. 3 shows the grain orientation of SLM pure Ti and SLM+ECAP pure Ti samples. It can be seen from Fig. 3a that the grain size of SLM pure Ti sample is different, and the average grain size is about 13 μm. The color changes in the crystal grains, indicating that a small misorientation has accumulated in the crystal grains. A small amount of tensile twins of 85°/ $\langle 11\bar{2}0 \rangle$ are distributed inside the crystal grains, as shown by the white arrow in Fig. 3a. In the metal with hcp structure, twinning is the main plastic deformation mechanism, coordinating the dislocation slip deformation, and jointly affecting the microstructure. The grain orientation of the SLM+ECAP pure Ti sample is shown in Fig. 3b, and the grain size is also different and unevenly distributed. The uneven microstructure is caused by the difference of original grain orientation, which is due to the difference of Schmidt factor

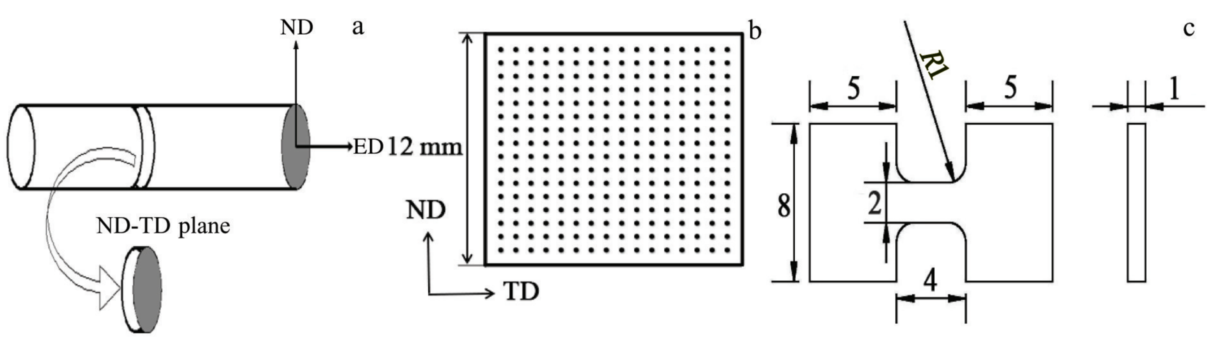

Fig. 1 Definition of sample axis (a); schematic diagram of hardness points for cross-section measurement (b) and tensile specimen at room temperature (c)

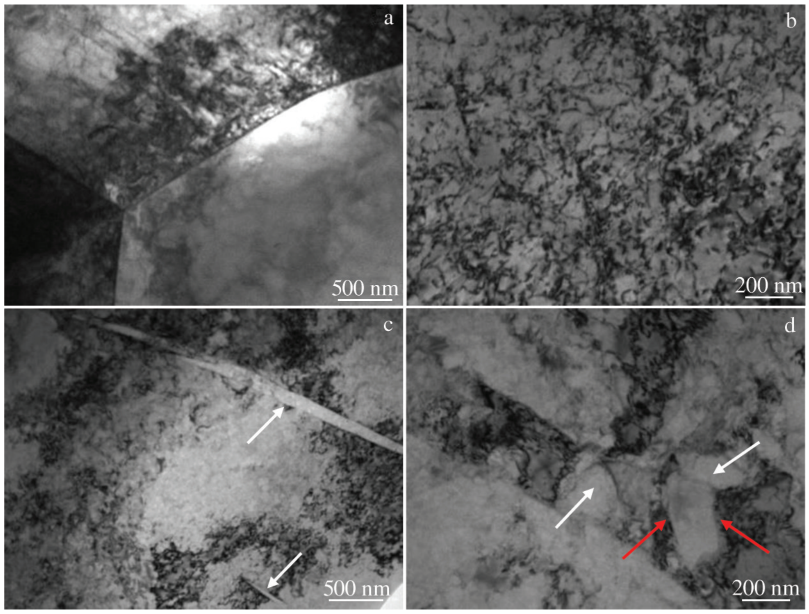

Fig.2 TEM images of SLM pure titanium (a, b) and SLM+ECAP pure titanium samples (c, d)

leading to different deformation resistance. After ECAP single-pass deformation, the microstructure has strong directionality; most of them are plate-like grains and ultrafine grains, which are typical structures after ECAP deformation. At the same time, there are some grains close to equiaxed morphology and some sharp grain boundaries at the large grain boundaries, as shown by the white arrow in Fig.3b, which indicates that the dislocation annihilation and dynamic recovery occur during the ECAP process, and the average grain size is 7 μm.

Fig.4 shows the grain boundary diagram of size and angle of SLM pure Ti and SLM+ECAP pure Ti samples. From

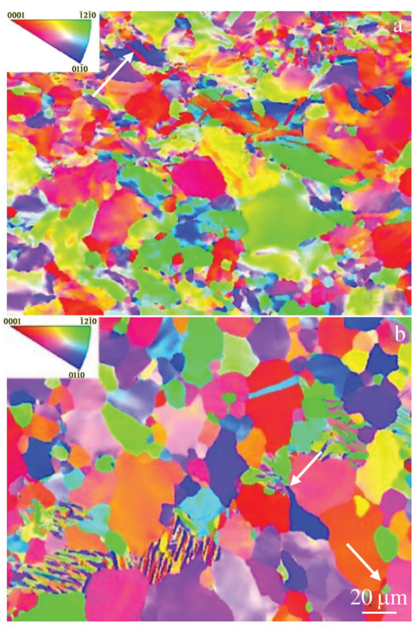

Fig.3 Grain orientation of SLM pure titanium (a) and SLM+ECAP pure titanium samples (b)

Fig. 4a and 4c, small angle grain boundaries are the main grains in SLM pure Ti samples, and the proportion of grain boundaries of big and small angles is 17% and 83%, respectively. The proportion fraction of small angle grain boundary of SLM+ECAP pure Ti sample shown in Fig.4b and 4d is reduced, while that of large angle grain boundary increases, and the proportion of grain boundaries of big and small angles is 40% and 60%, respectively. Fig.4b shows the incomplete high angle grain boundary (HAGB) fragments, that is, they do not form completely enclosed grains, combined with the large misorientation angles produced in the grains, etc, indicating that continuous dynamic recrystallization (CDRX) occurs after ECAP[19]. CDRX is easy to appear in titanium and titanium alloys, because it has a high interlaminar energy[20,21]. CDRX is the process of absorbing dislocation with low angle grain boundary (LAGB). With the continuous deformation, new and smaller grains with HAGBs are formed.

It can be observed from Fig. 4d that there are two main peaks that are relatively wide, located in the vicinity of 62°~65° and 86°~90°. This is because the axis angle pair is 57.2°/$\langle 11\bar{2}0\rangle$, that is, $\{10\bar{1}1\}\langle\bar{1}012\rangle$ compression twins and axis angle is 85°/$\langle 11\bar{2}0\rangle$, that is, $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twins are caused. They are shown by the blue arrow and black arrow in Fig.4b, which are consistent with the conclusions of Chen et al[22]. Studies[23] have shown that twinning and CDRX exist at the same time in single-pass deformation. More specifically, twinning is the main deformation mechanism in the initial stage of single pass deformation. However, with the continuous deformation, many of their orientations are conducive to dislocation activity. Therefore, in the later stage of single pass deformation, CDRX process plays a dominant role in twinning deformation, forming new fine grains and

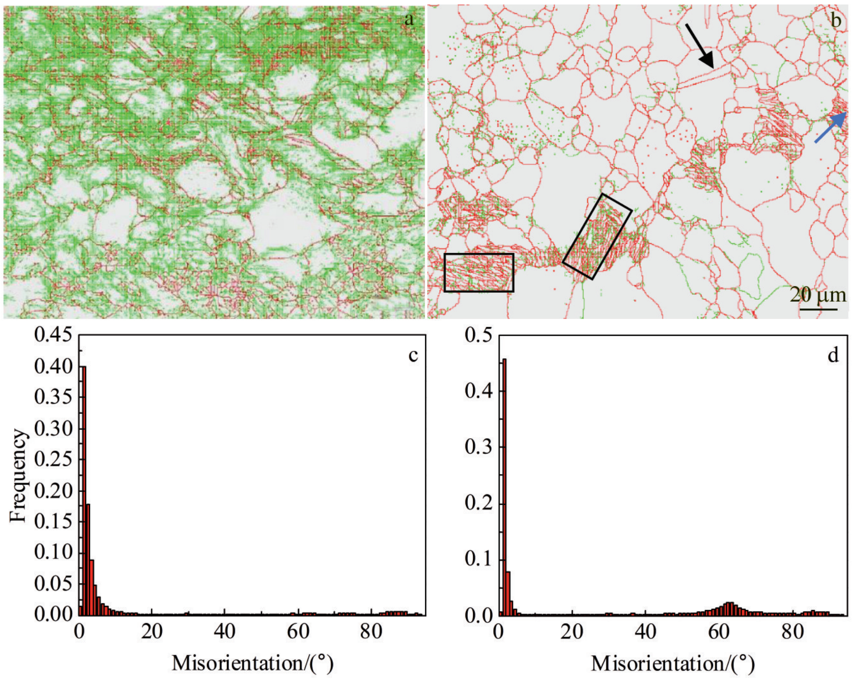

Fig.4 Grain boundary diagram (a, b) and misorientation distribution (c, d) of SLM pure titanium (a, c) and SLM+ECAP pure titanium (b, d)

promoting CDRX to become larger grains. Fig.4b shows these processes in the section circled by the rectangular square.

2.2 Mechanical properties

Fig.5 shows the microhardness distribution of SLM pure Ti and SLM+ECAP pure Ti samples. It can be seen that the hardness of the pure Ti sample is significantly improved after ECAP. The average hardness values of SLM pure Ti and SLM+ ECAP pure Ti samples are 2570 and 2900 MPa, respectively. Compared with SLM pure Ti samples, the hardness of SLM+ ECAP pure Ti samples is increased by ~13%. The main reason for hardness improvement is that shear strain occurs when the sample passes through the extrusion angle of the die, and the deformation is severe and the grains are broken obviously, which leads to an increase in dislocation density, dislocation clusters, dislocation strengthening inside the structure, and smaller grains. So that the microstructure is refined and the hardness is improved. Twins in the deformation of titanium and titanium alloys are usually $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twin formed under tension parallel to the c-axis direction and $\{10\bar{1}1\}\langle\bar{1}012\rangle$ compression twin formed under compression parallel to the c-axis direction[24], and the critical shear variables formed are 0.167 and 0.099, respectively[25,26]. Therefore, under the action of compressive stress, the material is more likely to form twin crystals and relax the internal stress. At the same time, the twin crystals become the nucleation center of the recrystallization stage. Compared with the part under tensile stress, the recrystallized grains in the part under compressive stress are more, so the average grain size is smaller, which is consistent with the results of EBSD. At the same time, the part with fine structure is more prone to grain boundary sliding, and the resistance of the material to deformation is small, so it is reflected as a lower hardness in the macroscopic view. This can also explain the phenomenon that the hardness of the lower surface of the sample after ECAP is slightly lower than that of the upper surface.

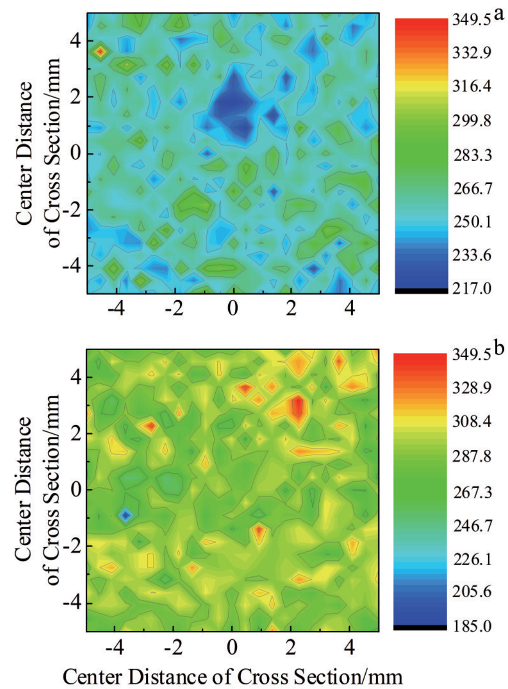

Fig.5 Micro-hardness diagram of SLM pure titanium (a) and SLM+ ECAP pure titanium samples (b)

Fig. 6a shows the engineering stress-strain curves of SLM pure Ti and SLM+ECAP pure Ti samples. It can be seen that the YS, UTS, and elongation of the SLM pure Ti sample are 621 MPa, 687 MPa, and 19.5%, respectively, while the YS,

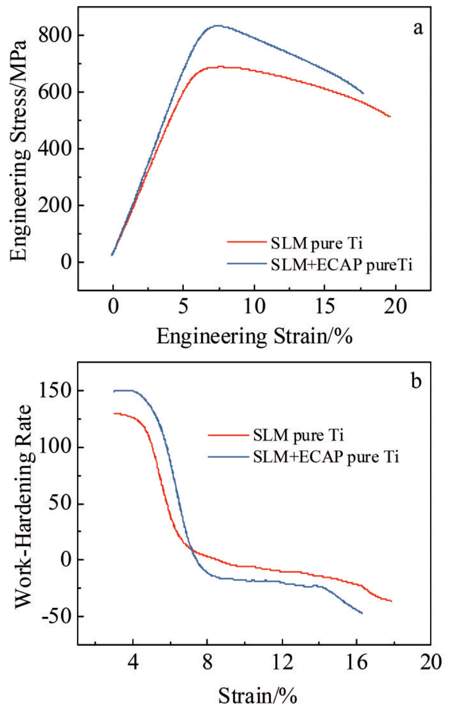

Fig.6 Mechanical properties of SLM pure titanium and SLM+ECAP pure titanium samples: (a) stress-strain curve and (b) work hardening rate

UTS, and elongation of the SLM+ECAP pure Ti sample are 733 MPa, 827 MPa, and 17.5%, respectively. Compared with those of SLM pure Ti, the YS and UTS of SLM+ECAP pure Ti samples are increased by 18% and 20.4%, respectively, while the elongation is decreased by 10.3%. At lower strain rates, as shown in Fig.6b, compared to SLM pure Ti samples, SLM+ECAP pure Ti samples have a higher work hardening rate, but the slope of the work hardening rate curve is almost the same. With the increase of strain rate, when the strain rate is greater than 8%, the work hardening rate of SLM+ECAP pure Ti sample is lower than that of SLM pure Ti sample. Generally speaking, the work hardening rate of both samples decreases, which is consistent with the research results of Becker et al[27]. Ref. [28] found that the reduction or disappearance of the strain hardening ability of severely plastically deformed materials is due to the easy dynamic recovery during the tensile deformation process, which makes the increases in dislocation density negligible. Park[29] believed that the decrease of strain hardening ability of materials after severe plastic deformation is due to the decrease of average free path of dislocations in the materials, which is because there are high internal stress non-equilibrium grain boundaries in the materials after severe plastic deformation, and the decomposition of dislocations at the non-equilibrium grain boundaries induces the recovery process.

As the grain size decreases after ECAP, the total elongation of the sample decreases slightly. This is because in the process of tensile deformation of fine-grained materials, as the grain size decreases, the ability of the grains to accommodate dislocations decreases, so the plastic elongation decreases, but the uniform elongation slightly increases.

Fig.7 shows the fracture morphologies of SLM pure Ti and SLM+ECAP pure Ti samples, both of which exhibit typical ductile fracture characteristics. SLM pure Ti samples show a large number of dimples and pores on the fracture surface, with mixed distribution of large and small dimples; the size and depth of dimples are not uniform, and the fracture surface is rough. In contrast, SLM+ECAP pure Ti samples show more stable small dimples and fewer pores, and the average sizes of dimples and pores are 1 and 8 μm, respectively.

3 Discussion

After single pass deformation, the dislocation density in the ECAP sample increases, the grains are refined, and different types of twin crystals appear. According to the theory of grain refinement, the strength of ECAP specimens is improved as the grains are refined, as shown in the Hall-Petch formula:

$$\sigma_s = \sigma_0 + k_y d^{-1/2} \quad (1)$$

where σ_s is the yield strength, and σ_0 is the yield strength of the single crystal; k_y is the influence coefficient of the grain boundary on the strength, and d is the grain size. Due to the close packed hexagonal structure and less slip system of titanium, k_y is larger than that of face centered cubic metal. It can be seen from the formula that the yield strength of polycrystalline materials is inversely proportional to the grain size, that is, the smaller the grain size, the higher the yield strength. It can be seen from the orientation diagram that after a single pass of ECAP deformation, the grain size decreases, and the grain boundaries increase, which increases the obstacle to the movement of dislocations, and it is difficult to

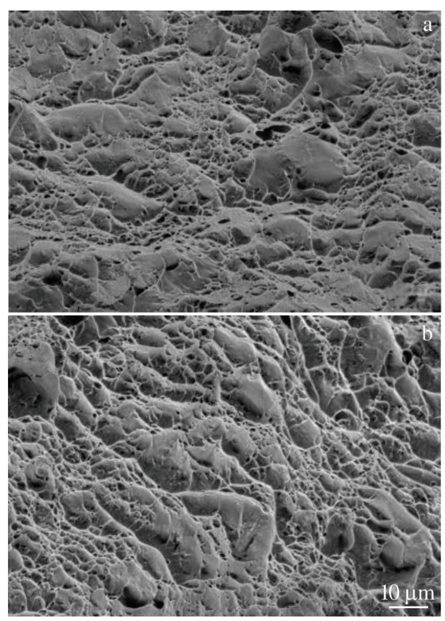

Fig.7 Fracture morphologies of SLM pure titanium (a) and SLM+ECAP pure titanium (b) samples

pass through the grain boundaries, so as to accumulate at the grain boundaries, causing stress concentration and leading to the increase of material strength.

Secondly, the appearance of twins will also affect the microstructure and mechanical behavior during deformation. At room temperature, pure Ti is a single closely packed hexagonal (hcp) α phase. Its crystal structure has low symmetry and few independent slip systems at room temperature, so the deformation is difficult and complex, which is mainly accomplished by dislocation slip and deformation twinning[24]. In the process of plastic deformation, slip and twinning are complementary and competitive with each other[30]. Twinning occurs in the process of slip. Twinning deformation makes the crystal orientation change, so it is easy to continue to slip. When crystal sliding encounters grain boundaries or obstacles, it will produce higher stress concentration, or make the crystal in an orientation unfavorable to slip, which will also induce twinning deformation[31]. Comparing Fig. 5a and Fig. 6a, it can be seen that after ECAP single pass deformation, $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twins and $\{10\bar{1}1\}\langle\bar{1}012\rangle$ compression twins appear, while the proportion of LAGB decreases and the proportion of HAGB increases, which is inconsistent with the conventional conclusion. It is well known that $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twins and $\{11\bar{2}2\}\langle11\bar{2}\bar{3}\rangle$ compression twins are the most commonly observed at room temperature[32-35]. Because $\{10\bar{1}1\}\langle\bar{1}012\rangle$ twin needs larger atomic movement than $\{11\bar{2}2\}\langle11\bar{2}\bar{3}\rangle$ compression twin, the interface energy of twin grain boundary is high, so it is not easy to produce at room temperature, and it is generally believed that it will occur at high temperature deformation. Shin et al[36] performed ECAP single-pass deformation on pure Ti at an extrusion temperature of 350 °C. The results show that there are a large number of fine $\{10\bar{1}1\}\langle\bar{1}012\rangle$ twin bands in both OM and TEM structures. A large number of $\{10\bar{1}1\}\langle\bar{1}012\rangle$ twin bands have also been observed in the ECAP single pass deformation of pure Ti at 400~450 °C by many scholars[37,38]. However, Zhao et al[39] found a large number of small $\{10\bar{1}1\}$ twin bands similar to those during high temperature deformation in the TEM microstructure of pure Ti under room temperature ECAP single-pass deformation (120° mold), but did not observe the common $\{10\bar{1}2\}$, $\{11\bar{2}1\}$ and $\{11\bar{2}2\}$ twin deformation of pure titanium during room temperature deformation. Chen et al[21] carried out single pass ECAP deformation on pure Ti at room temperature. The results show that there are many kinds of twins in the structure, which are $\{10\bar{1}1\}$, $\{10\bar{1}2\}$, $\{11\bar{2}1\}$ and $\{11\bar{2}2\}$, among which $\{10\bar{1}1\}$ and $\{11\bar{2}1\}$ are the main twins. The EBSD results of the SLM pure Ti and SLM+ECAP pure Ti samples show that the increase in the proportion of HAGB is also caused by $\{10\bar{1}1\}\langle\bar{1}012\rangle$ twin and continuous dynamic recrystallization (CDRX), which is also consistent with the conclusion of Chen[21].

On the one hand, the appearance of twins changes the orientation of the crystal lattice, more slip systems in the crystal are activated, and the resistance of dislocation slip is reduced; at the same time, the shear strain is directly provided to reduce the hardening rate; on the other hand, the appearance of twins changes the orientation of the crystal lattice and turns the original movable dislocations into immovable dislocations, which increases the strength of the material, that is, Basinski strengthening. In addition, twins have the effect of refining the crystal grains, reducing the distance of dislocation slippage, and playing a strengthening effect, that is, Hall-Petch strengthening. After ECAP, the hardness of the sample is improved by the change of microstructure and the appearance of more twins, which leads to the increase of twin interfaces, which can effectively hinder dislocation movement. This is similar to the effect of grain boundary on material strengthening. With the increase of shear stress, the more dislocations accumulated at the twin interfaces, the higher the stress concentration caused at the twin interfaces. In a word, the mechanism of twin strengthening is similar to that of fine grain strengthening. Both of them use more grain boundaries to hinder dislocation slip, thus increasing the hardness and strength of materials.

Table 1 shows the UTS and elongation of SLM pure Ti samples processed before and after different processing techniques[40,41], including ECAP, annealing, static magnetic field (SMF) and atmosphere assisted method (AA). It can be seen that after ECAP, annealing, SMF and AA treatments, the UTS of SLM CP-Ti is increased by 140, −10, 28, 274 MPa, while the elongation is increased by −2%, 2%, 7%, −11%, respectively. The different techniques change the UTS and elongation of SLM pure Ti, mainly due to the following facts: among them, annealing is a high temperature and long-term processing which causes grain coarsening; the addition of SMF can affect the temperature gradient field of the SLM process and improve the cooling rate of the molten pool; AA introduces N_2 to form nitrides and then to strengthen the second phase. As a severe plastic deformation technology, ECAP can not only eliminate some defects, but also refine and homogenize the microstructure, so that the UTS of SLM pure Ti can be significantly improved, while the elongation is only slightly reduced. Considering the comprehensive mechanical properties, the use of ECAP technology to improve SLM pure Ti samples is satisfactory. Therefore, it can be concluded that ECAP deformation is a good way to change the structure of SLM pure Ti specimens to obtain the best comprehensive mechanical properties.

Table 1 Mechanical properties of SLM pure Ti before and after different processing techniques

Property	ECAP		Annealing		SMF		AA	
	Before	After	Before	After	Before	After	Before	After
UTS/MPa	687	827	682	672	766	794	714	988
δ/%	19.5	17.5	22	24	28	35	17	6

4 Conclusions

1) ECAP process can improve the microstructure of SLM pure Ti sample, and realize the refinement and homogenization of microstructure. Twinning and continuous dynamic recrystallization occur.

2) At room temperature, the increase of high angle grain boundaries after single pass ECAP deformation is caused by continuous dynamic recrystallization, and $\{10\bar{1}1\}\langle\bar{1}012\rangle$ compression twins and $\{10\bar{1}2\}\langle\bar{1}011\rangle$ tensile twins appear.

3) After single pass of ECAP, the microhardness and strength of SLM pure Ti samples are improved. The strength and hardness of SLM pure Ti are affected by grain size, orientation and twinning. Compared with annealing, SMF and AA method, the mechanical properties of SLM pure Ti improved by ECAP are better.

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ECAP对SLM纯钛组织演变和力学性能影响

罗　雷[1,2]，段西明[1]，杨西荣[1,2]，刘晓燕[1,2]，王　文[1,3]，韩　鹏[1]

(1. 西安建筑科技大学 冶金工程学院，陕西 西安 710055)

(2. 冶金技术国家级实验教学示范中心，陕西 西安 710055)

(3. 功能材料加工国家地方联合工程研究中心，陕西 西安 710055)

摘　要：等通道转角挤压（equal channel angular pressing，ECAP）成功对选择激光熔化（selective laser melting， SLM）制备的纯钛进行了改性处理。采用两通道夹角Φ=120°，ψ=20°的模具，在室温下对SLM制备的纯钛进行单道次变形改性处理，并对其显微组织和力学性能进行了评价。结果表明：SLM+ECAP纯钛试样组织细化，晶粒尺寸由13 μm减小到7 μm，位错密度增加。ECAP变形过程中，孪生和连续动态再结晶同时存在，拉伸与压缩孪晶的出现和位错密度的增加共同促使SLM+ECAP纯钛试样显微硬度增加了13%，屈服强度和极限抗拉强度分别提高了18%和20.4%，而延伸率略有减小。

关键词：等通道转角挤压；选择性激光熔化；显微组织；力学性能

作者简介：罗　雷，男，1985年生，博士，副教授，西安建筑科技大学冶金工程学院，陕西 西安 710055，E-mail：luolei@xauat.edu.cn