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ARTICLE

# Effects of Initial Grain Size on Microstructure and Properties of Pure Ti Processed by ECAP

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**Abstract:** The pure Ti with different initial grain sizes which was annealed at 923 and 1023 K for 1 h was processed by ECAP at room temperature. The influence of initial grain size on microstructure and properties of ECAPed pure Ti was investigated by TEM, EBSD, tensile tests and microhardness tests at room temperature. The twinning behavior and deformation mechanism of pure Ti were also discussed during the ECAP. The results show that the initial grain size of pure Ti increases with increasing the annealing temperature. The grain refinement effect of pure Ti annealed at 1023 K is more significant than that of pure Ti annealed at 923 K after 1 pass of ECAP, while the microstructure of pure Ti annealed at 923 K is finer and more uniform than that of pure Ti annealed at 1023 K after 4 passes of ECAP. With the increase of ECAP pass, the yield stress increases, especially after 1 pass with more than 100% implication. The larger the initial grain size, the greater the stress increase. The deformation mechanisms of pure Ti during the ECAP include dislocation slips and deformation twinning, and with the increase of the initial grain size, the number of twins increases.

**Key words:** pure Ti; ECAP; grain size; twin; microstructure; mechanical properties

With excellent biocompatibility and outstanding mechanical properties such as decent ductility, fatigue and corrosion resistance, Ti has great potential in aerospace, automotive and medical implantation applications. In addition, limited strength and poor workability restrict its widely application because of its hexagonal close-packed (hcp) crystal structure possessing low crystallographic symmetry. Equal channel angular pressing (ECAP) is a kind of severe plastic deformation (SPD) method which can refine grain size of metallic materials effectively and obtain bulk ultrafine grained (UFG) materials by approximate pure shear deformation<sup>[1,2]</sup>. After ECAP process, the strength, creep, fatigue, corrosion properties and microforming properties of pure Ti can be greatly improved while maintaining reasonable plasticity, which is conducive to the wider application of pure Ti<sup>[3-8]</sup>. The microstructure evolution and mechanical properties change of pure Ti during ECAP process have also been comprehensively studied<sup>[9-14]</sup>. The deformation mechanism of pure Ti during ECAP includes dislocation slips and deformation twinning, in which the twinning behavior is affected by deformation temperature, ram speed, die parameter, deformation route and

initial texture<sup>[15-20]</sup>.

It is well known that the grain size plays a predominant role in the mechanical properties of metal materials, which is fitted by the Hall-Petch equation.

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

where  $\sigma_y$  is the yield stress,  $\sigma_0$  is a lattice friction stress,  $k_y$  is the strengthening coefficient and  $d$  is the average grain size. However, a recent report on the ultrafine-grained pure Mg showed negative deviations from the Hall-Petch equation when the grain size is smaller than 4  $\mu\text{m}$ , and inverse Hall-Petch effect when the grain size is smaller than 1  $\mu\text{m}$ <sup>[21]</sup>. The dominant deformation mechanism is transition from deformation twinning to grain boundary sliding (GBS) with grain refinement down to sub-micrometer, and the critical grain size was found. Their results suggested that the Hall-Petch relationship is only applicable when the grain size exceeds a certain critical value. The grain size has an important effect on deformation twinning, which is also reported for Ti-5at%Al<sup>[22]</sup>. The deformation mechanism during ECAP is complex under different deformation conditions. Chen has reported that the pure Ti during 3 and 4 passes of

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ECAP at 723 K via route A showed a low fraction of  $\{10\bar{1}2\}$  twins<sup>[23]</sup>. The pure Ti after 1 pass of ECAP at room temperature via route Bc has been investigated by EBSD, and two types of deformation twins were found which were  $\{10\bar{1}2\}$  and  $\{11\bar{2}2\}$  twins<sup>[24]</sup>. However, the pure Ti samples which were unannealed, 1033 K annealed for 2 h, and 1173 K annealed for 4 h were processed by 1 pass of ECAP at room temperature. No deformation twinning was found in this case<sup>[25]</sup>.

The aim of the present work is to study the effect of the initial grain size on the microstructure evolution and mechanical properties of pure Ti processed by multi-pass ECAP. The pure Ti with different initial grain sizes was obtained through two different annealing temperatures, then comprehensive microstructure observation and mechanical property tests were carried out, and the twinning behavior and deformation mechanism in this process were discussed.

## 1 Experiment

A hot rolled pure Ti (TA1) bar with a composition (wt%) of O 0.18, H 0.015, N 0.03, C 0.08 and Fe 0.2, and the balance Ti, was used in this study. To obtain pure Ti with different initial grain sizes, the cylindrical samples with 25 mm in diameter and 200 mm in length were annealed at 923 and 1023 K for 1 h. The annealed samples with different initial grain sizes were processed by 4 passes of ECAP at room temperature. The ECAP was performed at the ram speed of 2.5 mm/s via route C, which requires the sample to rotate 180° around its longitudinal axis between adjacent passes. The internal channel angle and outer arc curvature angle are 135° and 20° correspondingly, which gives an equivalent strain of about 0.46 for each pass. To reduce friction between samples and die, the samples were coated with a mixture of MoS<sub>2</sub> and solid graphite powder, and then wrapped by a graphite foil.

Microstructures including average grain size, dislocation structures and deformation twinning of the longitudinal section were observed using JEM-200CX transmission electron microscope (TEM) and S-3400N scanning electron microscope (SEM) equipped with a Nordlys Nano electron backscattering diffraction (EBSD).

Mechanical properties of annealed and ECAPed samples were evaluated by tensile tests and Vickers microhardness tests at room temperature. Dog-bone tensile samples were cut from the central regions of the pressed billets parallel to the longitudinal axes with the gauge length, width and thickness of 12, 3 and 1.5 mm, respectively. Tensile tests were carried out using INSTRON 8801 universal testing machine with a quasi-static strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$ . The Vickers micro-hardness of the longitudinal section was measured using HX-1000TM Vickers hardness tester under a load of 1.96 N and a dwell time of 10 s.

## 2 Results and Discussion

### 2.1 Effects of initial grain size on microstructure evolution during ECAP

The grain orientation images and TEM micrographs of pure Ti annealed at 923 and 1023 K after 4 passes of ECAP are

shown in Fig.1 and Fig.2, respectively. The microstructures of two kinds of samples annealed at 923 and 1023 K are characterized by nearly equiaxed grains with the average grain size of about 64 and 97  $\mu\text{m}$ , respectively. With the increase of ECAP pass, the equiaxed grains disappear, a large number of twins appear, and the microstructure is severely deformed. Based on EBSD analysis, the number fractions of deformed structures after 4 passes are 72% in 923 K annealed sample and 85% in the 1023 K annealed one, indicating that these samples are fully deformed.

It can be seen in TEM micrographs that, equiaxed grains of coarse-grained (CG) Ti are changed to much refined lamellar structures, including parallel shear bands with clear and straight boundaries (Fig.2a) and intersected shear bands with different orientations (Fig.2b) after 1 pass of ECAP. There are high densities of dislocations in the shear bands. Compared with 923 K annealed sample, the 1023 K annealed one exhibits a smaller band width and higher dislocation density, indicating a better refinement effect, which agrees with the grain refinement ratio shown in Table 1. The refined lamellar structures along with smaller equiaxed grains are obtained after 2 passes of ECAP. And there are some twins inside bands. With the increase of ECAP pass, the band structures are gradually broken down to a refined one, and parallel with a certain direction. The microstructure of 923 K annealed sample is more refined and uniform than that of the 1023 K annealed one after 4 passes of ECAP. The difference in the microstructure of pure Ti with different initial grain sizes during ECAP is mainly focused on the dislocation density, grain size and the number fraction of deformation twins.

Based on EBSD analysis, grain size distribution of pure Ti with different initial grain sizes, which was annealed at 923 and 1023 K after different passes of ECAP, is shown in Fig.3 and Fig. 4, respectively. The grain size distribution, average grain size and grain refinement ratio are tabulated in Table 1 where the top row relates to the unprocessed condition (0 pass), the remaining rows cover different ECAP passes from 1 to 4.

The microstructure is significantly refined both in samples with different initial grain sizes after 4 passes of ECAP. The largest grain refinement ratio appears after 1 pass of ECAP. The average grain sizes of pure Ti annealed at 923 and 1023 K after 4 passes are 2.81  $\mu\text{m}$  and 3.71  $\mu\text{m}$ , and the proportion of grains smaller than 5  $\mu\text{m}$  is 93.37% and 85.83%, respectively. Generally, the grain size will be refined with increasing the ECAP passes, but there is saturation grain size in a certain deformation condition. Zhao has found that pure Ti processed by 6 passes and 8 passes of ECAP has the same average grain size of about 200 nm<sup>[26]</sup>. It is noteworthy that after 1 pass of ECAP, the average grain size of pure Ti annealed at 1023 K with a bigger initial grain size is smaller than that annealed at 923 K. After 4 passes of ECAP, the average grain size of pure Ti annealed at 1023 K is slightly larger, but the total grain refinement ratio is more significant.

Fig. 5 shows the deformation twinning types and number fractions of pure Ti annealed at 923 and 1023 K and processed

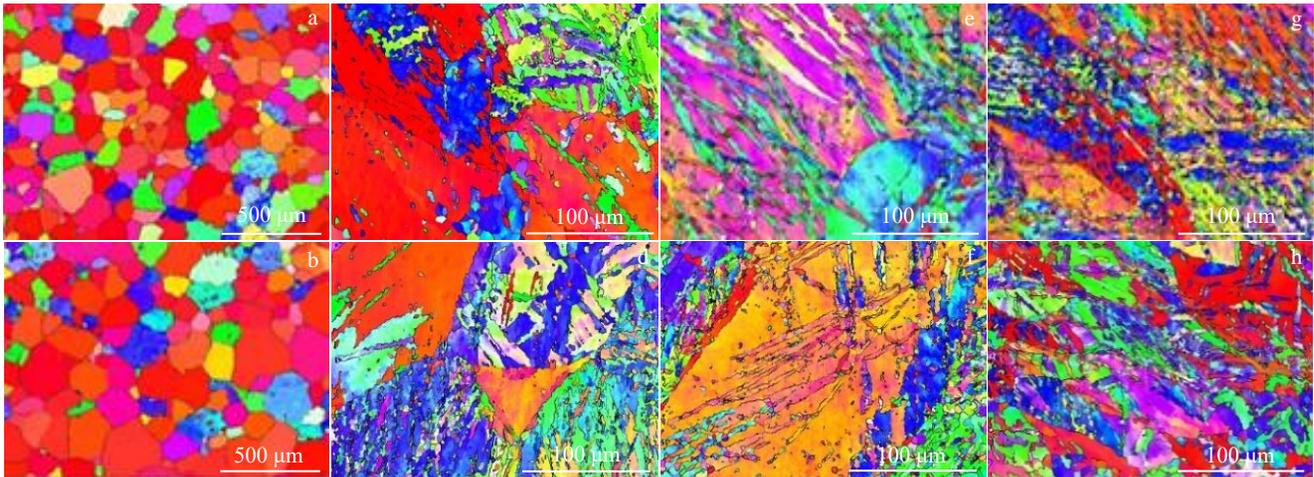


Fig.1 Grain orientation images of pure Ti annealed at different temperatures after different ECAP passes: (a) 923 K after 0 pass, (b) 1023 K after 0 pass, (c) 923 K after 1 pass, (d) 1023 K after 1 pass, (e) 923 K after 2 passes, (f) 1023 K after 2 passes, (g) 923 K after 4 passes, and (h) 1023 K after 4 passes

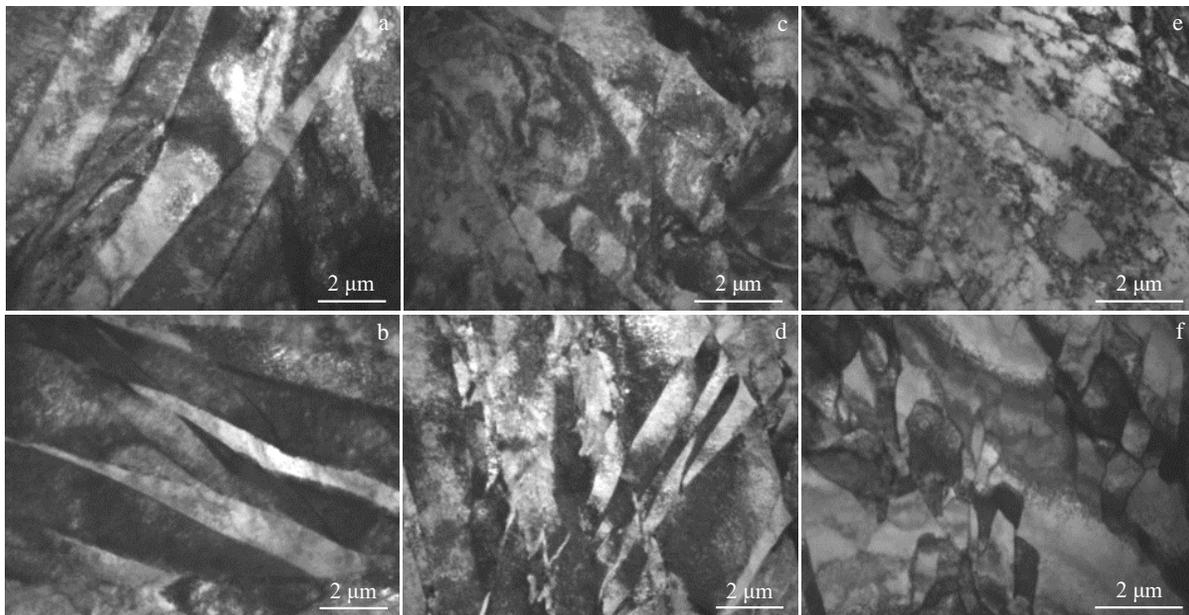


Fig.2 TEM micrographs of pure Ti annealed at different temperatures after different ECAP passes: (a) 923 K after 1 pass, (b) 1023 K after 1 pass, (c) 923 K after 2 passes, (d) 1023 K after 2 passes, (e) 923 K after 4 passes, and (f) 1023 K after 4 passes

by different ECAP passes. Three types of deformation twins including  $\{10\bar{1}2\}$  tensile twins as well as  $\{11\bar{2}2\}$  and  $\{10\bar{1}1\}$  compression twins are found in pure Ti during the ECAP, suggesting that twinning is one of the major deformation mechanisms of pure Ti during the ECAP process, which is also reported in several earlier reports<sup>[27,28]</sup>. With increasing the ECAP pass, the number fraction of twins is first increased and then decreased in 923 K annealed samples, which is 0.53%, 9.84%, 12.44% and 8.68% from 0 to 4 passes, while 0.73%, 11.14%, 10.50% and 10.58% in 1023 K annealed samples, respectively, which agrees with the grain refinement ratio. There are tiny amounts of annealing twins in annealed

samples, and the number fraction of 1023 K annealed sample is slightly higher.

Deformation twinning is highly sensitive to grain size, and with increasing the grain size, the activity of deformation twinning increases<sup>[29]</sup>. As the grain size increases, the slip distance of dislocations increases, dislocations tend to accumulate at grain boundaries, and the stress concentration generated by the large dislocation accumulated at the grain boundaries induces the activation of deformation twinning<sup>[30]</sup>. With the increase of the ECAP pass, further twinning becomes more and more difficult due to the continuous refinement of twins, and the number of twins decreases slightly. It is obvious

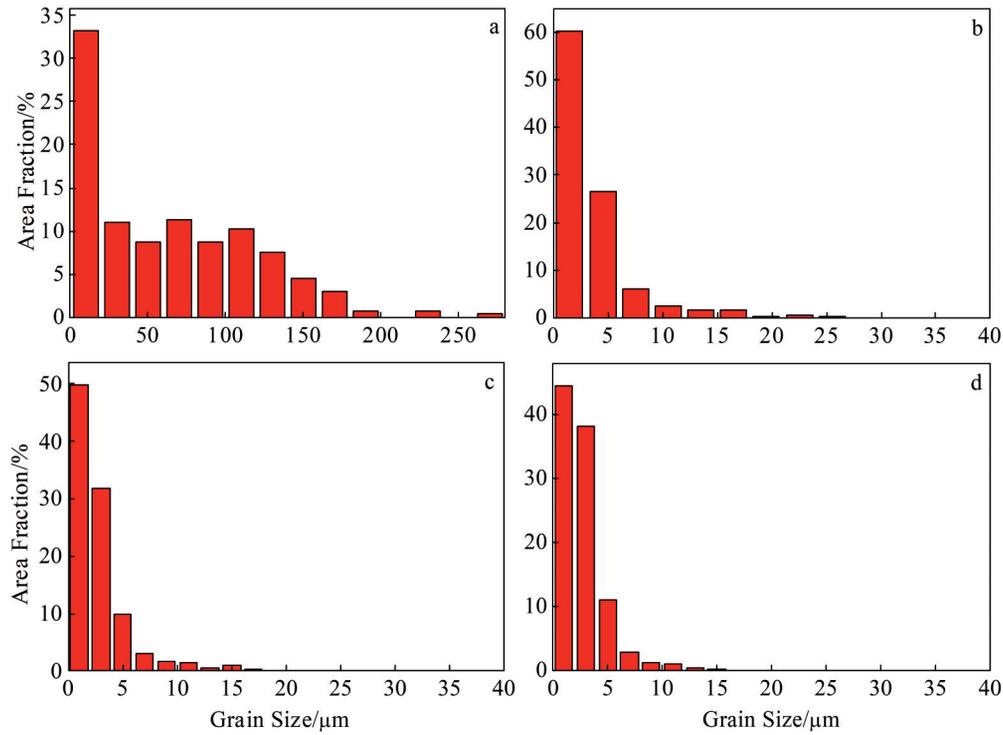


Fig.3 Grain size distribution of pure Ti annealed at 923 K after different ECAP passes: (a) 0 pass, (b) 1 pass, (c) 2 passes, and (d) 4 passes

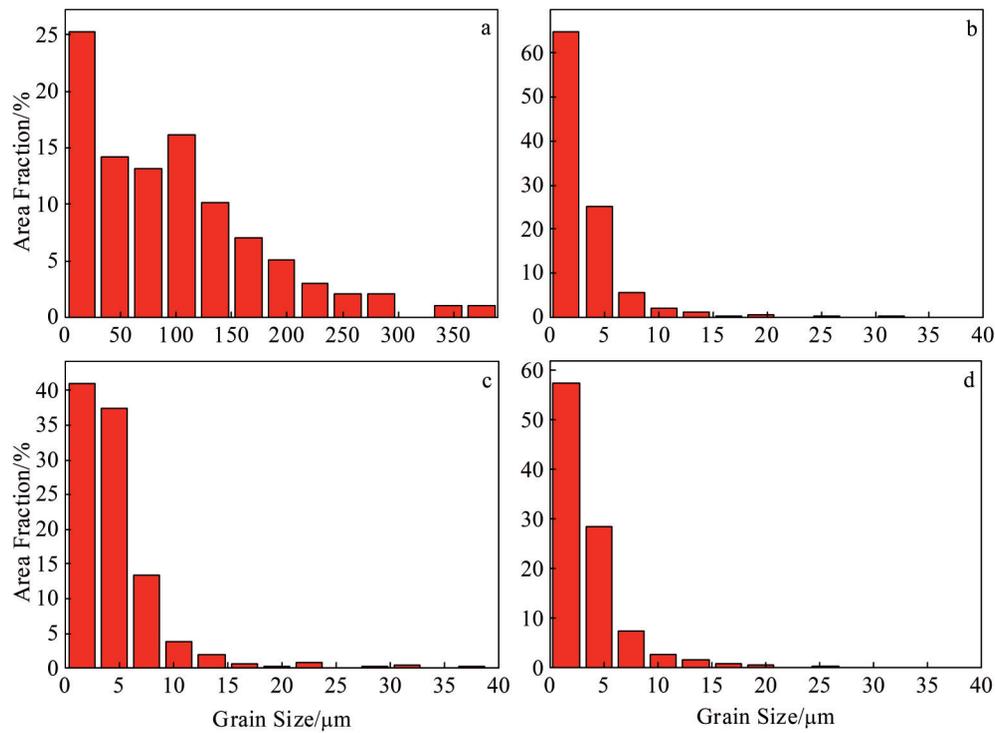


Fig.4 Grain size distribution of pure Ti annealed at 1023 K after different ECAP passes: (a) 0 pass, (b) 1 pass, (c) 2 passes, and (d) 4 passes

that pure Ti with different initial grain sizes has different twinning activities during the ECAP. Therefore, pure Ti annealed at 1023 K with a larger initial grain size has more twins activated to assist plastic deformation, which results in a better grain refinement effect during 1 pass of ECAP, but after 2 passes, the number fraction of twins decreases, and the grain

size increases slightly which is attributed to the dynamic recovery process.

## 2.2 Effects of initial grain size on mechanical properties during ECAP

The engineering stress-strain curves for pure Ti with different initial grain sizes after different passes of ECAP are

**Table 1** Average grain size statistics of pure Ti after different ECAP passes

ECAP pass	923 K			1023 K		
	Distribution range/ $\mu\text{m}$	Average grain size/ $\mu\text{m}$	Refinement ratio/%	Distribution range/ $\mu\text{m}$	Average grain size/ $\mu\text{m}$	Refinement ratio/%
0	0~200	63.79	-	0~300	97.11	-
1	0~20	3.71	94	0~15	3.22	97
2	0~15	2.84	23	0~15	4.61	43
4	0~15	2.81	1	0~15	3.71	20
Total	-	-	95.6	-	-	96.2

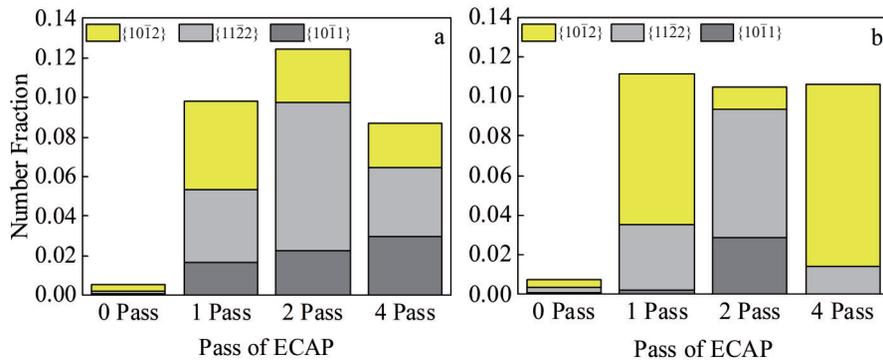


Fig.5 Deformation twinning statistics of pure Ti annealed at 923 K (a) and 1023 K (b) followed by different passes of ECAP

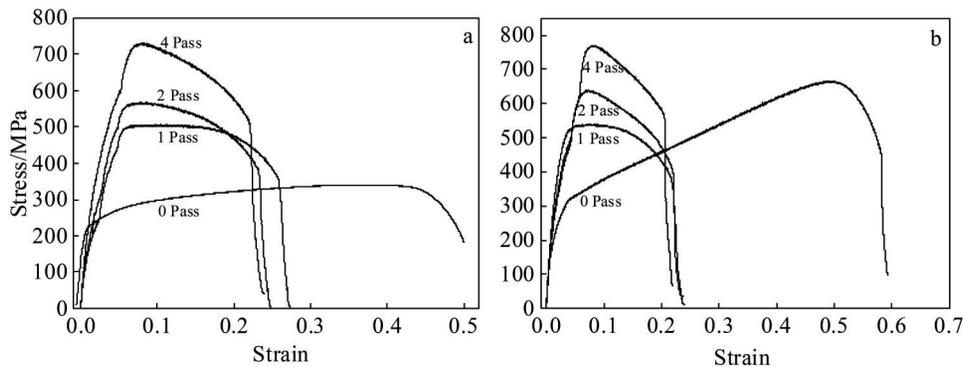


Fig.6 Engineering stress-strain curves of pure Ti annealed at 923 K (a) and 1023 K (b) followed by ECAP process

**Table 2** Mechanical properties of pure Ti after ECAP process

ECAP pass	923 K					1023 K				
	$\sigma_s/\text{MPa}$	$\Delta\sigma_s/\%$	$\delta/\%$	HV/MPa	$\Delta\text{HV}/\%$	$\sigma_s/\text{MPa}$	$\Delta\sigma_s/\%$	$\delta/\%$	HV/MPa	$\Delta\text{HV}/\%$
0	215	-	58	1290	-	190	-	68	1360	-
1	435	102	30	1550	20	440	132	25	1690	24
2	500	15	25	1730	12	470	7	21	1930	14
4	600	20	22	2000	16	535	14	19	2060	7
Total	-	179	-	-	55	-	182	-	-	51

displayed in Fig.6. The mechanical properties are tabulated in Table 2. It can be seen that there is excellent strengthening after 4 passes of ECAP: the yield stress increases from 215 MPa to 600 MPa in pure Ti annealed at 923 K and 190 MPa to 535 MPa in pure Ti annealed at 1023 K correspondingly. After 1 pass of ECAP, the largest increase ratio appears, which agrees with the largest grain refinement ratio. Obvious work

hardening is observed in annealed samples, while little work hardening occurs in severely deformed samples. The general paradox of strength and ductility appears<sup>[31]</sup>, but there is also reasonable ductility with an elongation to failure of 22% and 19% after 4 passes of ECAP, respectively. Although the yield stress of the sample annealed at 1023 K is lower than that annealed at 923 K, it is higher than the samples annealed at

923 K after 1 pass of ECAP. The phenomenon is related to the grain refinement ratio, dislocation density and number fraction of deformation twins.

The microhardness of pure Ti annealed at 923 and 1023 K is increased by about 55% and 51% after 4 passes of ECAP, respectively, which can be seen in Table 2. The microhardness increases as the grain size decreases during the ECAP<sup>[32]</sup>. It can be noted that the largest increase appears after 1 pass of ECAP, which is owing to the sharp decreasing of grain size, the obvious increasing of dislocation density and number fraction of deformation twins and unbalanced substructures.

It is well known that the grain size plays a predominant role in the mechanical properties of metal materials, which is fitted to the Hall-Petch equation (Eq.(1)). The values of yield stress shown in Table 2 are plotted against the inverse square root of grain size ( $d^{-1/2}$ ), as shown in Fig.7, and a straight line is fitted. For the samples both annealed at 923 and 1023 K and subsequently processed by ECAP, the yield stress and average grain size have a typical Hall-Petch relationship with the  $\sigma_0$  values of about 121.2 and 130.8 MPa, and  $k_y$  values of about 21.8 and 21.4 MPa·mm<sup>1/2</sup>, corresponding to the slope of the fitted line and its intercept on the y axis, respectively. This result suggests that the deformation twinning plays a very important role in the deformation process. Calvillo also reported values of  $\sigma_0=327.03$  MPa and  $k_y=5.11$  MPa·mm<sup>1/2</sup><sup>[33]</sup>. These results are far from the current result because the deformation is controlled by dislocation slip in their study. But when the grain size is smaller than a certain critical value, the data will show a negative deviation from Hall-Petch line or even an opposite result. A recent experimental report observed an inverse Hall-Petch relationship in high pressure torsion (HPT) pure Mg when the grain size was less than 1.57  $\mu\text{m}$ <sup>[21]</sup>.

### 2.3 Microstructure evolution: dislocation slips and deformation twinning

From the high dislocation density in TEM micrographs (Fig. 2b~2d) and lots of deformation twins in twinning statistics (Fig. 5a and 5b), it is evident that the deformation mechanisms of pure Ti during the ECAP include dislocation slips and deformation twinning. Generally, twinning has a little direct effect on deformation. Its role is to change the orientation of the crystal and to release the stress concentration, which will stimulate further dislocation slip. As a result, twinning mainly affects the dislocation slips to assist the plastic deformation of the materials<sup>[34,35]</sup>. The appearance of deformation twins can refine the grain, which is Hall-Petch strengthening<sup>[36]</sup>. Meanwhile, it can increase the internal interface of the microstructures, resulting in an increase in the resistance to dislocation slips and a stress concentration, which has a significant strengthening effect on the mechanical properties of the materials. Due to the severe shearing force, a large number of inhomogeneously distributed dislocations are generated in pure Ti during the ECAP. These dislocations gather together to form the dislocation cells. With further deformation, the dislocation cells deform and the dislocations gather at the edge of the cells to form substructures.

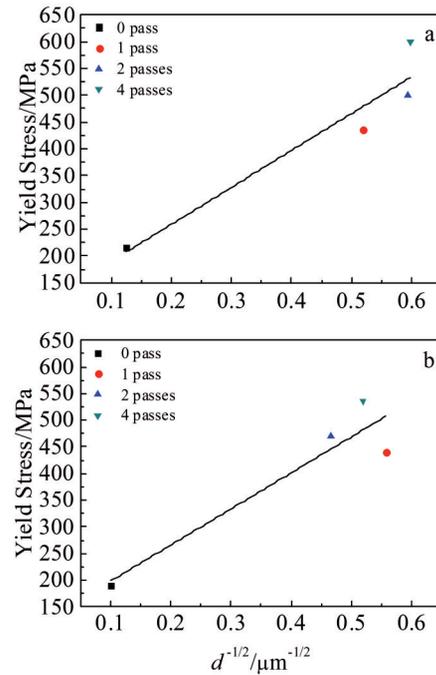


Fig.7 Relationship of average grain size and yield stress of pure Ti annealed at 923 K (a) and 1023 K (b) followed by ECAP

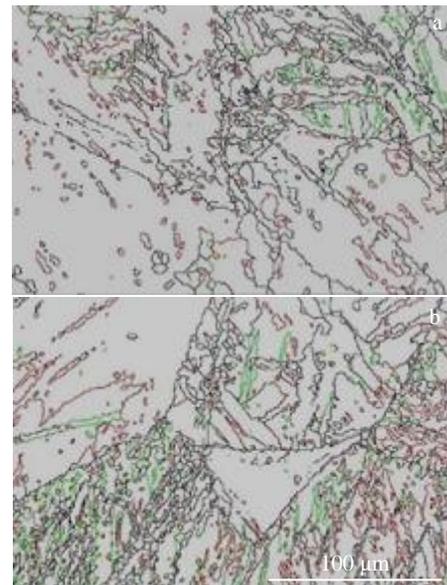


Fig.8 EBSD orientation data of special grain boundary distribution characterization for pure Ti annealed at 923 K (a) and 1023 K (b) after 1 pass of ECAP

Fig.8 shows special grain boundaries distribution characterization for pure Ti annealed at 923 and 1023 K after 1 pass of ECAP, which identifies different types of deformation twinning based on EBSD grain orientation data. The main deformation twins of pure Ti during the ECAP at room temperature include  $\{10\bar{1}2\}/86^\circ$  (red) tensile twins and  $\{11\bar{2}2\}/64^\circ$  (green),  $\{10\bar{1}1\}/56^\circ$  (yellow) compression twins. The shear

strain energy of  $\{10\bar{1}2\}$ ,  $\{11\bar{2}2\}$  and  $\{10\bar{1}1\}$  twins is 0.175, 0.225 and 0.105, respectively. As we know that the lower the shear strain energy of deformation twins, the easier it will be activated during the ECAP. However, the activation of  $\{10\bar{1}1\}$  twins requires a higher temperature and pressure<sup>[37,38]</sup>. Minonishi reported that the  $\{10\bar{1}1\}$  twins appear when the deformation temperature is higher than 673 K<sup>[39]</sup>. However, the ECAP process has a large shear strain and high strain rate, so the  $\{10\bar{1}1\}$  twins will be activated at a lower temperature. It is found that the  $\{10\bar{1}1\}$  twins may be the key factor in refining grain size effectively<sup>[40]</sup>. The main types of deformation twins are  $\{10\bar{1}2\}$  tensile twins and  $\{11\bar{2}2\}$  compression twins, and the number fraction of  $\{10\bar{1}1\}$  compression twins is small, especially in pure Ti annealed at 1023 K after 1 pass and 4 passes of ECAP.

### 3 Conclusions

1) The grains of pure Ti are continuously refined during the ECAP. After 1 pass of ECAP, the pure Ti annealed at 1023 K with a larger initial grain size has a more significant grain refinement effect. After 4 passes of ECAP, the microstructure of pure Ti annealed at 923 K with a smaller initial grain size is finer and more uniform. The samples annealed at 1023 K have average grain size of 3.71  $\mu\text{m}$  and 85.83% grains smaller than 5  $\mu\text{m}$ , while the values are 2.81  $\mu\text{m}$  and 93.37% for samples annealed at 923 K, respectively.

2) During the ECAP, the yield stress and microhardness increase with the increase of ECAP pass, and the increment after 1 pass of ECAP is the largest, which is in agreement with the grain size refinement ratio and the number fraction of deformation twins.

3) The deformation mechanisms of pure Ti are dislocation slips and deformation twinning during the ECAP, the twinning types include  $\{10\bar{1}2\}$ ,  $\{11\bar{2}2\}$  and  $\{10\bar{1}1\}$  twins, and the number fraction of deformation twins is affected by the initial grain size. The larger the initial grain size, the easier the deformation twins to activate, and the more deformation twin assisted deformation.

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## 原始晶粒尺寸对ECAP变形纯钛组织性能的影响

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**摘 要:** 室温下, 对923及1023 K退火1 h所得的不同原始晶粒尺寸的工业纯钛进行ECAP变形。通过TEM、EBSD、室温拉伸和显微硬度测试研究原始晶粒尺寸对ECAP变形纯钛组织性能的影响。探讨纯钛ECAP变形孪生行为和变形机制。结果表明, 退火温度越高, 原始晶粒尺寸越大。1道次变形后, 1023 K退火纯钛的晶粒细化效果更显著。4道次变形后, 923 K退火纯钛的组织更细小均匀。随着变形道次的增加, 屈服强度不断增大, 1道次变形后增幅最大, 约为100%, 且原始晶粒尺寸越大, 强度增幅越大。纯钛ECAP变形机制包括位错滑移和孪生, 原始晶粒尺寸越大, 孪晶数量越多。

**关键词:** 纯钛; ECAP; 晶粒尺寸; 孪晶; 显微组织; 力学性能

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