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ARTICLE

Study on Microstructure and Properties of Pure Ti flat Wire

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ABSTRACT

The titanium round wires could be prepared to the high precision flat wires with the sizes of 0.63mm×5.03mm by nine-pass cold continuous rolling process.Mechanical property and metallograph analysis were used to analyze the possibilities of preparing flat wire. Results show that the grain kept equiaxed grain in the rolling processes,and a large number of dislocation and a small amount of twin existed in the microstructure of final rolling flat wire.During the whole rolling processes, when the accumulated rolling deformation degree was at low degree, the deformation of titanium was controlled under the mutual control of dislocation slip and twinning. When the accumulated rolling deformation degree was at high degree, the deformation of titanium was controlled by the dislocation slip.

1. Introduction

Pure titanium has good properties such as high specific strength, good biocompatibility, corrosion resistance and so on. It has a wide application prospect, and is very popular in aerospace, petrochemical and other fields. However, because the α-Ti, with hcp lattice structure at room temperature is difficult to achieve the plastic deformation of pure Ti by slip alone, it is necessary to rely on twins to assist in the deformation [1]. In unidirectional cold rolling of high purity titanium by Ruoyu Zhang [2], it is found that both slip and twin participate in deformation when the deformation is less than 50%, and the twins contribute more in the deformation process, so a large number of twins appear in the microstructure. If the amount of deformation is not less than 50%, the slip is less than 50%. The contribution of shape gradually increases and exceeds the effect of twins, and the twins in the microstructure will gradually decrease and the deformed bands will be formed at the same time. Shankun Chen [3] of Xi'an Jiaotong University found that the original equiaxed microstructure of ultra-fine grained TA1 pure titanium plate prepared by repeated rolling process was gradually elongated and evolved into fine grain. When the equivalent effect was more than 2.4, it was found that the original equiaxed structure was gradually elongated and evolved into fine grain. A large number of substructures formed by dislocation entanglement were observed in TEM images, but no twins were observed, and the dislocation motion was the main deformation mechanism.

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The cold continuous rolling process was used to flatten industrial pure titanium. The microstructure evolution and deformation mechanism of industrial pure titanium during flat rolling were studied by mechanical properties test and microstructure observation. In order to explore the stable forming process of pure titanium flat wire with high precision.

2. Trial

2.1 Test Material

The industrial pure Ti filament treated with Φ 2.5 mm was used in the experiment. The main chemical composition of the filament was shown in Table 1.

Table 1. Chemical composition of commercial pure titanium wire（wt%）

Ingredient	Ti	Fe	C	N	H	O
content	allowance	0.20	0.08	0.03	0.015	0.18

2.2 Rolling Mill Practice

Industrial pure Ti flat wire was prepared by two high and three tandem rolling mill. The rolling is divided into nine passes, and the roll diameter is 150. The rolling process is cooled and lubricated with 10 # oil, the rolling speed is 18 m / min, the pass reduction is distributed as shown in Table 2. The rolling process is divided into 9 passes, and the roll diameter is 150. The rolling speed is 18 m / min, and the rolling speed is 18 m / min.

Table 2. Reduction distribution of commercial pure titanium wire（unit：mm）

Pass	Outgoing gauge	Pass compression ratio	Cumulative compression ratio
1	1.99	20.40%	20.40%
2	1.77	11.06%	29.20%
3	1.56	11.86%	37.60%
4	1.37	12.18%	45.20%
5	1.20	12.41%	52.00%
6	1.05	12.50%	58.00%
7	0.86	18.10%	65.60%
8	0.65	24.42%	74.00%
9	0.63	3.080%	74.80%

2.3 Mechanical Property Test and Microstructure Observation

The tensile tests at room temperature for Φ 2.50mm pure titanium round wire and each pass flat wire were carried out on the WDW- 100 10 ton electronic drawing machine. The total length of single specimen was 160 mm, 50mm and the tensile speed was 1 mm/min. The hardness test was carried out on the Waubert 401 MVD digital display microhardness tester. The loading force was 300 gf, holding time of 10s. The hardness test was divided into two types: hardness test in the direction of flat wire rolling (longitudinal) and hardness test on its cross section. Olympus - G51 metallographic microscope for metallographic observation, corrosion agent HF3ml, HN O315ml, H2O30ml. Tem test was carried out at Xi’an Jiaotong University.

3. Test Results and Analysis

3.1 Mechanical Properties of Flat Wire under Different Rolling Conditions

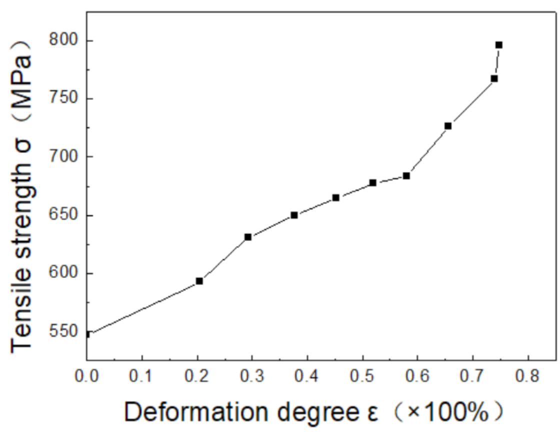

Figure 3. The relation between tensile strength and deformation of commercial pure titanium wire

As can be seen from Figure 3, the original tensile strength of industrial pure Ti wire with Φ 2.50mm size after warm drawing is 547 MPA, which is cold rolled according to technological regulations. It is found that the trend of tensile strength changes with the increase of deformation degree, and the tensile strength of titanium wire increases continuously. When the total compression ratio of the first rolling stage is 37.60%, the tensile strength of the titanium wire reaches 649.7 MPA, the tensile strength of the titanium wire reaches 796 MPA when the total compression ratio of the second rolling stage is 58.00%, and the tensile strength of the titanium wire reaches 796 MPA when the total compression ratio of the third rolling process reaches 74.8%. After three rolling periods, compared with the round wire before unrolled The increment of tensile strength of titanium wire is 102.7 MPA, 37.3MPa and

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112MPa respectively. Therefore, the change rule of tensile strength of industrial pure Ti flat wire during rolling is as follows: with the increase of deformation degree, the tensile strength of Ti wire increases greatly, but the work hardening degree of the second rolling process is smaller.

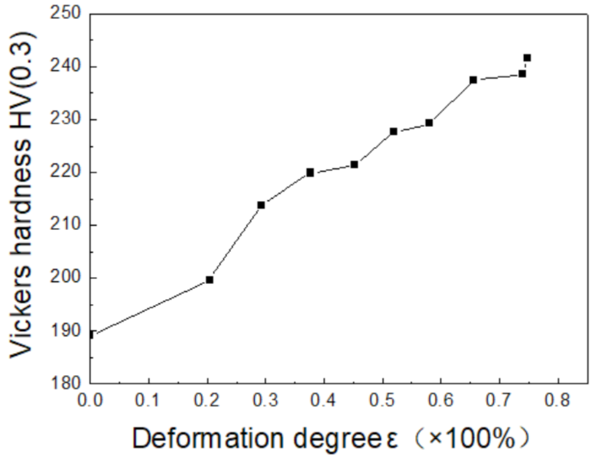


Figure 4. The relation between microhardness and deformation of commercial pure titanium wire

In Figure 4, the microhardness of Φ 2.50mm's industrial pure Ti filaments is 189.1HV (0.3). It is found that the change trend of microhardness is the same as that of tensile strength: with the increase of deformation degree, the change trend of microhardness is the same as that of tensile strength. The Vickers hardness of Ti filament increases continuously. When the compression ratio is 37.60%, the microhardness is 219.9HV (0.3). In the sixth pass, when the compression rate is 58.00%, the microhardness is 229.2Hv (0.3), and when rolling to the ninth pass, the total compression rate reaches 74.8%, the microhardness is 241.6HV (0.3). The increment of microhardness after rolling is 30.8HV (0.3), 40.1HV (0.3) and 52.5HV (0.3) respectively) compared with the round wire before rolling. Therefore, the microhardness of industrial pure Ti flat wire during rolling is as follows: with the increase of deformation degree, the microhardness increases. The hardness of industrial pure Ti flat wire is also tested. The industrial pure Ti wire is rolled to the finished product according to the rolling schedule shown in Table 2, and the total reduction rate reaches 74.8%. The hardness distribution of the hardness point on the cross section is shown in Figure 5, and the hardness distribution of the cross section is obtained. As shown in Figure 6.

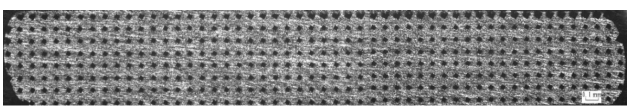


Figure 5. Distribution of microhardness points on cross-section of commercial pure titanium flat wire

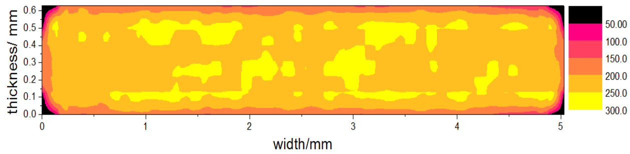
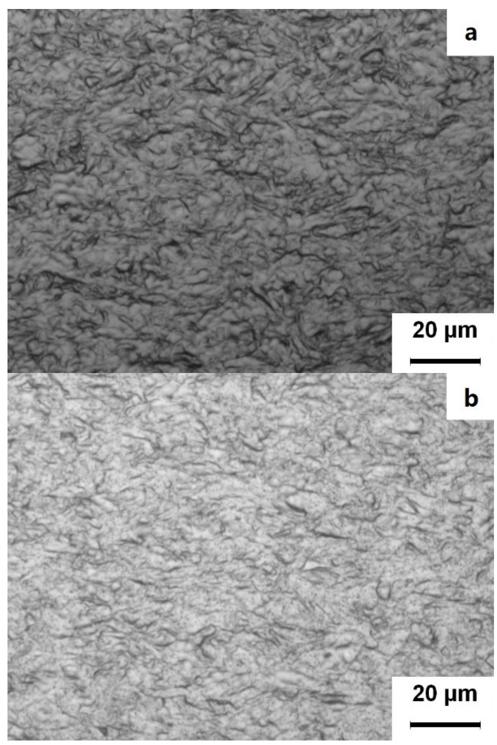


Figure 6. Distribution of microhardness on cross-section of commercial pure titanium flat wire

Figure 6 when the total reduction rate of industrial pure Ti flat wire reaches 74.8%, the hardness distribution of cross-section increases gradually from the edge to the cross-section center in the wide direction, and the hardness of the cross-section increases from the rolling surface to the cross-section center in the thickness direction. The maximum hardness of the core and the minimum hardness of the edge are 281.8HV (0.3) and 103.4HV (0.3), respectively. The central high hardness region is X-shaped, and X extends to the whole cross-section along the direction of extension, and the hardness of the rest tends to be uniform. This is because with the increase of deformation, the deformation zone will gradually expand to the contact area and the wide area, so that the deformation of the entire section will be more uniform.

3.2 Microstructure of Flat Wire under Different Rolling Conditions



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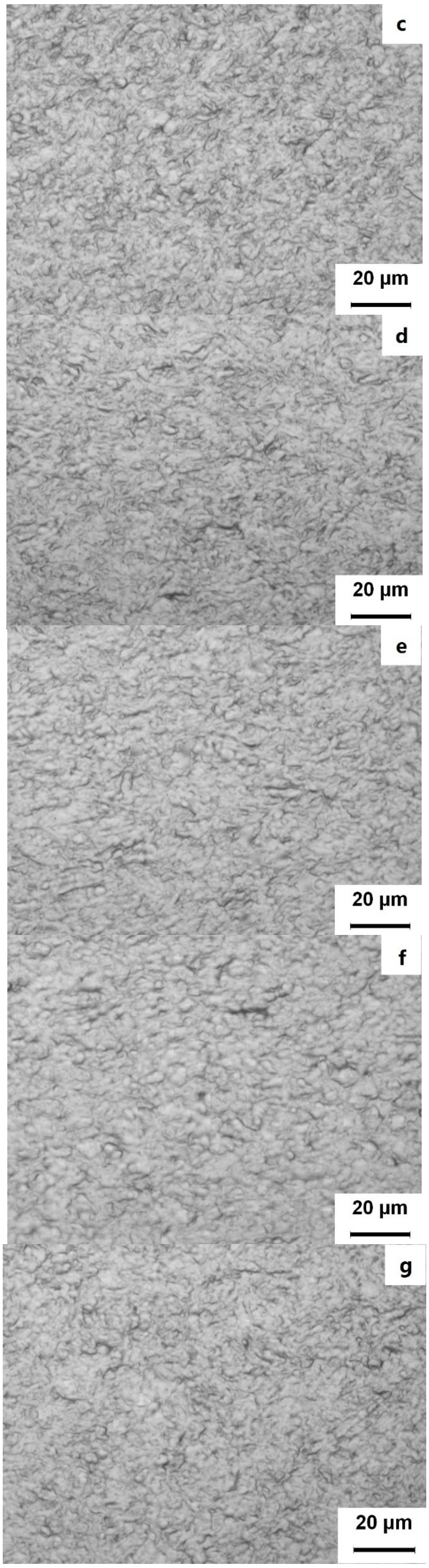

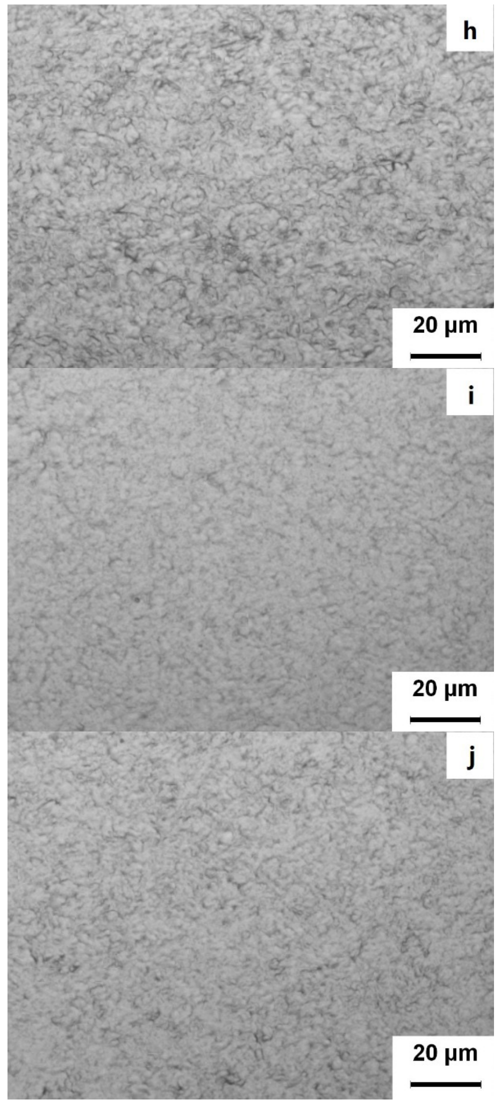

Figure 7. The microstructures of commercial pure titanium round wire and after rolling in accordance with the process planning

Notes: a. Commercial pure titanium round wire; b. Flat wire after one-pass rolling process; c. Flat wire after two-pass rolling process; d. Flat wire after three-pass rolling process; e. Flat wire after four-pass rolling process; f. Flat wire after five-pass rolling process; g. Flat wire after six-pass rolling process; h. Flat wire after seven-pass rolling process; i. Flat wire after eight-pass rolling process; j. Flat wire after nine-pass rolling process.

The microstructure of industrial pure Ti is a phase with dense hexagonal (hcp) crystal structure at room temperature. In this experiment, the microstructure of industrial pure titanium filaments drawn at medium temperature is α phase, the morphology of which is part of equiaxed structure and a small number of irregular grains with flat ellipse or long strip shape. As can be seen from Figure 7 (a) - 7 (j), during the rolling process from the first pass of 20.40% to the final cumulative reduction rate of 74.80%, the basic

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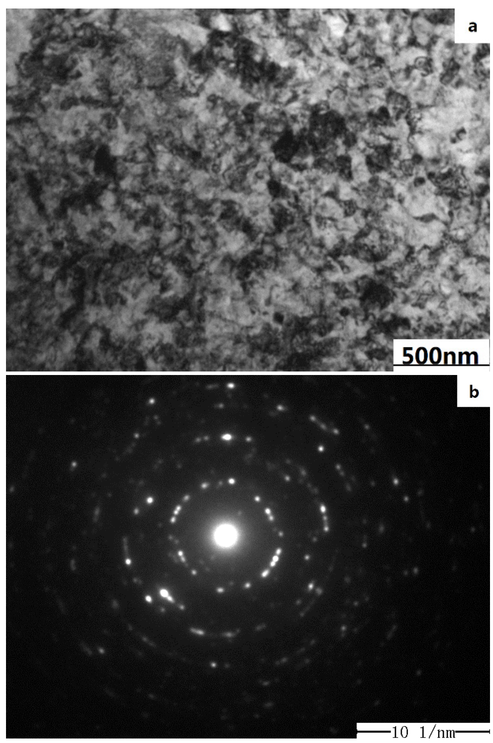
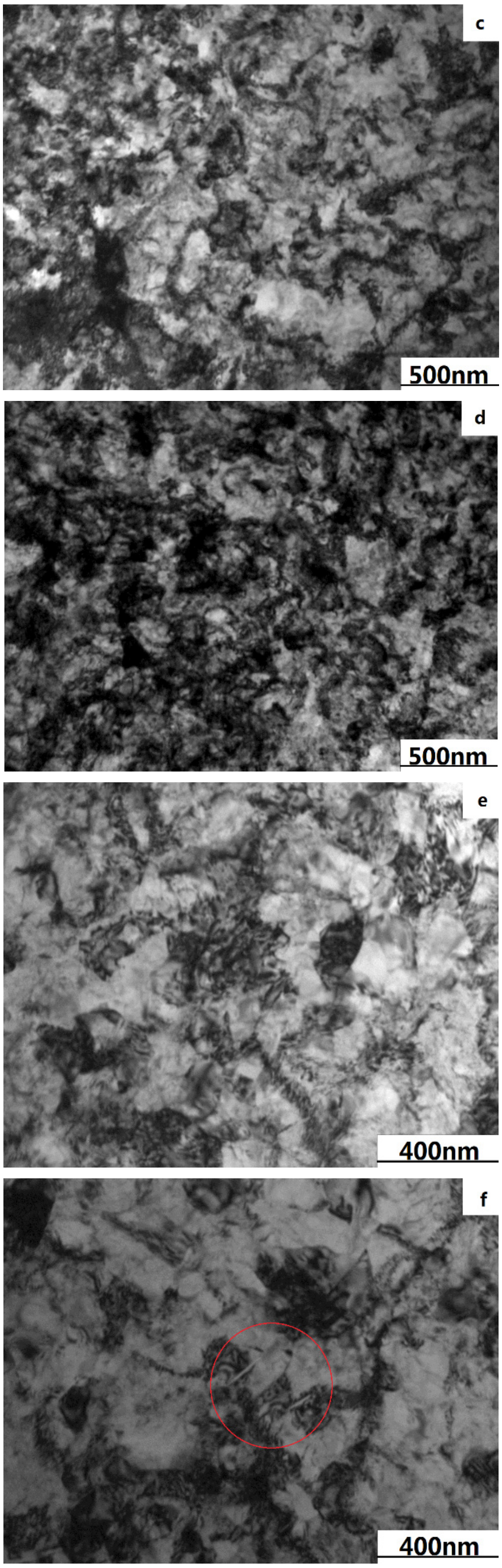
shape of grain does not change, and most of the grains are equiaxed. Only with the increasing of the reduction rate, the equiaxed structure is more refined and the original grain boundary is blurred. This indicates that in rolling plastic deformation, intra and intergranular deformation occurs. The deformation mechanism plays an important role. This is because slip is not easy to occur during plastic deformation of hcp crystal. In the initial rolling stage, only a small number of grains with favorable orientation will slip, and there will be interaction between adjacent grain boundaries and shear stress at the grain boundary. When the shear stress increases to overcome the resistance of grain sliding to each other, the grain boundary will slip. For the grains in the unfavourable orientation, the intergranular interaction will gradually turn to the orientation conducive to slip. Neither of the above two intergranular deformation modes can change the original morphology of the grain and promote the intra-crystal deformation. That is, when there are more crystals, When the grain is in a favorable slip direction, the slip and twin-dominated intra-crystal deformation will play a major role. In Figure 7, the grain refinement of the final rolling structure is related to the slip and the twinning of the grain refinement in the (j). This also explains the reason for the increasing tensile strength and hardness in Figure 1 and Figure 2, that is, fine grain strengthening.

Dislocations and twins in α-Ti microstructure have great influence on plastic deformation, but these structures are not visible under OM. In order to find out the change of microstructure in pure Ti deformation, the TEM analysis of the flat wire rolled to the ninth pass is carried out as follows.

Figure 8 (a) - 8 (g) are all TEM photographs of flat wire after the ninth pass cold rolling. As can be seen from Figure 8 (a), there are a large number of high density dislocations along the grain boundary in the α-Ti, microstructure with single phase after rolling, a large number of dislocation cells formed by dislocation entanglement and a small number of subcrystalline structures in some regions of the grain boundary. According to the (b) diffraction speckle in Figure 8, there are nanocrystalline grains formed after the grains are broken and the diffraction spots are circular, indicating that there is internal stress in the microstructure and the lattice is distorted and deformed violently. Figure 8 (c) is a local area of the same organization in which both dimensions are found The dislocations are concentrated in the equiaxed grains and entangled. In the large grain regions, there are already formed dislocation cells, and the dislocations are concentrated on the cell walls to form dislocation walls, and the dislocations are concentrated on the cell walls, and in the large grain regions, the dislocations are mainly distributed on the cell walls, and the dislocations are concentrated on the cell walls to form dislocation walls. However, there are very few intra-cellular dislocations. Figure 8 (d) shows dislocation cells formed by a large number of dislocation entanglement in the microstructure, which indicates that dislocation slip plays an important role in cold rolling plastic deformation of pure Ti, and as can be seen from figure (a), (c), (d). After cold rolling, the microstructure of industrial pure Ti is not uniform, and its inhomogeneity is reflected in grain size and sub-structure distribution. Figure 8 is more visible in the organization as shown in (e) The results show that the dislocation in the inner part of the cell wall is attracted to the dislocation wall of the cell wall and rearrangement and cancellation occur, the dislocation in the central region decreases, the orientation difference of the grain boundary increases, and a large angle grain boundary is formed, which indicates that the dislocation of the inner part is attracted to the dislocation wall of the cell wall in the process of deformation. The organization was refined. Although the plastic deformation modes of pure Ti are slip and twinning, there are very few twinning structures under the condition of large deformation with a total reduction rate of 78.40%. As shown in Figure 8 (f), the twins are fine needle-like with a large number of dislocations distributed on both sides. Figure 8 the twinning staggered motion in (g) causes the grain to be segmented so that the grain can be refined and a large number of fine equiaxed crystals can be obtained.





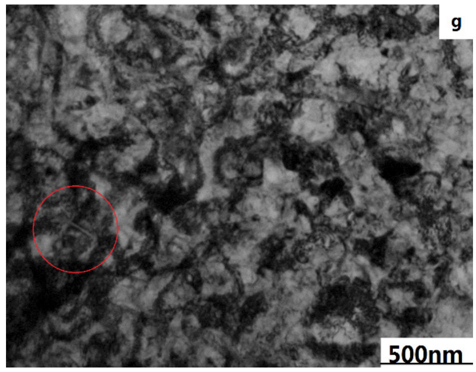


Figure 8. The TEM microstructures of commercial pure titanium flat wire after nine-pass rolling process

3.3 Cold Rolling Deformation Mechanism of Industrial Pure Ti Wire

The plastic deformation of pure Ti is complicated. It is difficult to satisfy the deformation conditions of the five independent slip systems in plastic deformation by simple type slip. It is necessary to open the slip-like system on the cone or to accompany the twin deformation at the same time. As two main plastic deformation modes of dense hexagonal metals, slip and twinning compete with each other and complement each other [5]. In the microstructure with large deformation, there are few twins, a large number of dislocation entanglement and substructure are distributed in the microstructure, and a large number of fine equiaxed crystals are formed. The evolution rule of microstructure is as follows: in the initial stage of deformation, some grains in the microstructure take the lead in the dislocation slip due to the dominant orientation, and the slip system which is easy to slip is first opened, and when the movement of dislocation slip is obstructed, the movement of dislocation slip is blocked. A large number of dislocations will be plugged into the barrier, resulting in stress concentration and interruption of slip. In this case, the height of stress concentration will cause the shear stress to be higher than the critical shear partial stress required for twin deformation, thus inducing the formation of twins. Twinning is a kind of shear change. Shape, the essence of which is to change the orientation of the crystal so that it rotates to a position conducive to slip, so that the previously stopped or unopened dislocation motion retakes place, allowing the plastic deformation to continue, and in addition, the twins can absorb some of the energy. The local stress concentration is alleviated, the crack is restrained, and the flexibility of the material is increased. With the increasing of accumulated deformation and density of dislocation, the stress concentration becomes more and more obvious, the high

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stress makes twins continue to form, and when the rotation of crystal is not ideal, the twin will continue to occur, and the stress concentration is more and more obvious, and the twinning will continue to occur when the orientation rotation of the crystal is not ideal. The size of the crystal will also continue to decrease until it is rotated to the appropriate orientation, when the crystal changes from hard orientation to Due to the soft orientation and more slip systems, the mutual motion of the slip system makes the crystals cut each other, and a large number of dislocations will be entangled at the grain boundary to form dislocation walls or dislocation cells, and the dislocations will decrease the density by rearrangement or cancellation. In the process of deformation, the original grain is divided into a large number of fine grains in order to coordinate the deformation. Under the action of external force, the large angle orientation between the grains will be poor through the rotation of the fine grains. Small equiaxed grains are formed in the local region. At this time, twins are rare in the tissue, and a large number of high-density dislocations are distributed in the tissue. Apart from the dislocation slip and twinning mechanism mentioned above, the plastic deformation lifting of pure Ti is carried out. In addition, grain rotation and grain boundary sliding also promote the plastic deformation of pure Ti, which not only maintains the original grain shape of α phase but also coordinates the slip deformation.

4. Conclusion

The main results are as follows:

(1) under the condition of this experiment, the diameter of Φ 2.50mm warm-drawn industrial pure Ti round wire was adopted, and the flat Ti wire with the cross-section size of 0.63mm × 5.03mm was obtained by cold nine continuous rolling.

(2) during the deformation process, the tensile strength and microhardness of the wire increase with the increase of the deformation amount;

(3) during the cold rolling deformation of industrial pure Ti wire, the grains in the microstructure keep equiaxed at the same time, and there are a lot of dislocations and a few twins in the deformed structure.

(4) the main deformation mechanism of pure Ti at room temperature is the slip and twinning mechanism in the small deformation stage, the slip mechanism in the gradual increase of cumulative deformation, and the slip mechanism in which the accumulated deformation increases gradually. And the grain rotation and grain boundary sliding mechanism of auxiliary deformation during rolling deformation.

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