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Recrystallization Kinetics of Ti-Ta Alloy

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In the paper**,** recrystallized behaviour of binary Ti-Ta alloy wire under various deformation amounts is studied. The recrystallization annealing test is carried on the alloy under different deformation amount, and recrystallization activation energy Q is calculated by using the formula of $G=G_0 \bullet exp^{(-Q-RT)}$. The recystallization rate of titanium tantalum alloy wire increase with the increase of the deformation amount, which is attributed to the decrease in the recrystallization activation energy, Q.

Keywords: *Ti-Ta (Titanium-Tantalum) alloy, deformation amount, recrystallization activation energy*

1. Introduction

Titanium tantalum alloy getter wires are used in some vacuum equipments in order to keep and increase the vacuum degree of the equipment [1]. But the report about the process technique of this titanium tantalum alloy wire has not appeared in Chinese journal to this day.

The processes of titanium tantalum from ingot to wire are separated into forging wore,hot rolling, grinding and drawing deformation. The drawing deformation so easily leads to work-hardening that the annealing-in-process is extremely important. When the annealing schedule is not reasonable, the direct results are broken wire during drawing process and the lower ratio of finished products. In industry, it is more difficult to work the fine wire than to work the coarse wire, so the annealing-in-process under drawing deformation is the key to make fine wire which diameter is less than 1.0 mm [2]. So the study to find the connection of deformation ratio, work-hardening and annealing-in-process during drawing the titanium tantalum alloy wire has a extraordinary important meaning. In this paper, the recrystallizing behaviour of titanium tantalum wire which diameter is less than 1.0mm was studied too, and the recrystallizing kinetics curve of titanium tantalum wire was obtained. These conclusions are very useful to improve drawing technique and the quality of the titanium tantalum alloy fine wire.

2. Experimental Procedure

In the paper, the authors apply the formula (1) to calculate the deformation amount in drawing γ1 process.

$$\Delta = (1-\frac{\gamma_2}{\gamma_1}) \times 100\% \quad (1)$$

In the formula, Δ represents the deformation amounts; represents the cross section radius of wire after drawing deformation; γ_2 represents the primal cross section radius of wire before drawing deformation. The test samples were Obtained from different deformation amount wire. The recrystallization annealing of fine wire was executed in the electrical resistance furnace, and the wire surface was protected by the high temperature oxidation resistant coating, in which the principal constituent is silicides. When the furnace temperature reaches the annealing temperature, the sample was taken into the furnace for thermal treatment. Later, the sample was taken out and water quenched. In the whole test, the time of heat preservation is not easy to control in annealing treatment. In order to increase the test precision, the preparation test should be done, and the approximate time and temperature of recrystallization nucleus under different deformation amounts were controlled through the preparation test. The sample passing annealing treatment was inlayed in the ethoxyline resion for easing to make the metallographic specimen because the wire, diameter was smaller. The recrystallization nucleus forming and growing-up were observed by metallographical measurements of NEOPHT21 metallographic microscope. The recrystallization activation energy was calculated according to the time of the recrystallization grain number forming less than 5 percents. The recrystallization kinetic curve was obtained through researching the relaction of recrystallization volume fraction and recrystallization time.

3. Results and Discussions

3.1 Recrystallization Activation Energy

Figure 1a shows the microstructure of the drawing stage. The elongated grain is the fiber structure, and it is a sort of flowing line becoming on drawing process. The dynamic recrystallization grain doesn't appear in microstructure. So we can say that the obvious dynamic recrystallization behavior do not happen during the drawing process, the reason is that the temperature is too low to reach the point of recrystallization nucleus temperature. **Figure 1b** shows the recrystallization nucleus microstructure. The deformation is non-uniform, the deformation storage power of different deformation zone is different, the time that the storage power giveing off is different too [3]**.** Because the distortion energy is lower in the center grain than in the edge grain, recrystallization nucleus starts to appear in original grain boundary of wire edge, and it will grow up by swallowing the non-recrystallization grain which hold in center, the result is that the recrystallization grains come into being in whole sample, and the equiaxed grain occupies the whole cross section which is distortionless. This research conclusion is the uniform on the other alloy [4].

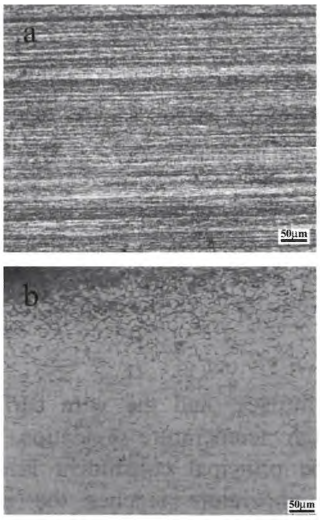
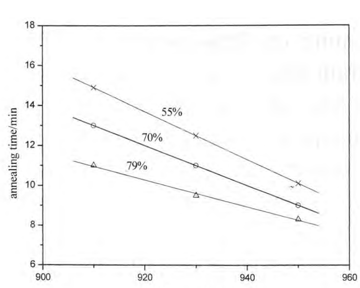


Figure 1. The microstructure
(a, Processing state; b, Recrystallization nucleus)

The time of recrystalliation nucleus is that the recrystallization nucleus is defied such numbers is not to exceed 5 percents on the whole microstructure. **Figure 2.** shows the relationship between incubation period of recrystallization nucleus and annealing temperature under the different deformation amounts. From the curve, we can discover that the time of incubation period of recrystallization nucleus is different in different zone of the alloy ample. The obvious rule is that the time of incubation period is shorter with the deformation amount being larger and annealing temperature being higher. The recrystallization process is a forming nucleus and growing up, its realization depends on the atom diffusion, but the condition which recrystallization nucleation could stably grow up is that the recrystallization nucleus size must be bigger than the critical nucleus. When the deformation amount is larger, the critical nucleus dimension is smaller and the atom diffusion is more easy, so the recrystallization nucleus dimension become smaller. As a result, the incubation period of recrystallization nucleus become shorter.



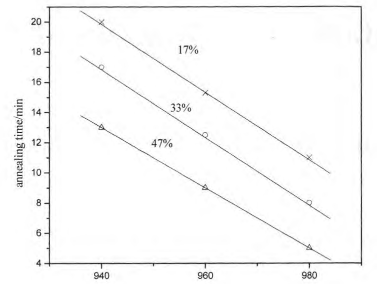


Figure 2. The relationship between incubation period of recrystallization nucleus and annealing temperature under different deformation amounts.

Since the metal recrystallization is a heat activation process, a formula to calculate the recrystallization activation energy can be applied. Arrhenius equation formula is expressed by the following equation[6]:

$$G = G_0 \cdot \exp^{(-Q/RT)}$$

in the formula, Q represents the recrystallization activate energy (J/mol) ;R represents the gas constant (8.3 14J/mol•K) ;T represents the Kelvin temperature (K) .
in the use of the equation, some scholars often suppose that the following formula can exist:
$G = X_v / t = G_0 \bullet \exp^{(-Q/RT)}$,here t represents the time(min) , X_v represents the recrystallization volume fraction(%). When X_v is a constant, the above formula will turn to thr formula or

$$1 / t = A \bullet \exp^{(-Q/RT)}$$

Figure 2 is used to calculate the value Int and $\frac{1}{T}$. **Figure3**. shows the relation curve of $Int \sim \frac{1}{T}$.

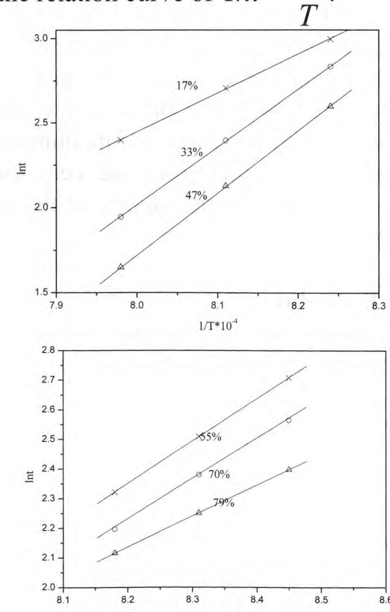


Figure 3. The relational curve of Int on 1/T

It is not difficult to find that the relationship of Int and 1/T is linear. The K represents the linear slope, and it is the ratio of recrystallization activation energy, Q, and gas constant R. The recrystallization activation energy can be obtained through calculating the linear slop K. The different deformation amounts correspond to the different recrystallization activation energy. For example, when the deformation amounts are separate 17%, 33 %, 47%, 55 %, 7 0%, 79%, the recrystallization activation energy, Q, is 190. 9kJ/mol, 169.5 kJ/mol, 119.1 kJ/mol, 118.8 kJ/mol, 113.1 kJ/mol, 86.8 kJ/mol, respectively The recrystallization uncleus first appear in structure defect such as dislocation and vacancy on the grain boundary. The recrystallization driving force is larger with the deformation amount being larger, the recrystallizaton tendency becomes bigger too. At the same time, the density of dislocation and that of vacancy on the grain boundary become bigger. The dislocation and vacancy will provide the more position to form the recrystallization nucleus. In other words, it is the advantaged channels for atom diffusion. On the other hand, the binding force of atoms is weaker in the dislocation defect and vacancy defect than in other positions. Therefore the thermal barrier decreases as well as the recrystallization activation energy decreases. In conclusion, with the deformation amounts increase, the distortion energy increases and the activation energy of recrystallization nucleus decreases, and the recrystallization driving force increases.

3.2 Recrystallization Kinetics Curcve and Microstructure

Recrystallization kinetics of the titanium tantalum alloy were studied. Figure 4. shows the recrystallizaiton kinetics curve. When the deformation amount is 55%, the recrystallizaiton kinetics curve at different temperature shows that the recrystallization incubation time is shorter with the temperature increase at the same deformation amount, and the time which finishing the recrystallization is shorter. For example, when the annealing temperature is 940°C, the finishing recrystallization time is approximate3 times 1 Ongre than the 980 °C , and the recrystallization incubation time is approximately 5 times longer. The other conclusion is that the recrystallization incubation and finishing time is shorter with the deformation amount increasing under the same annealing temperature. Under the annealing temperature of 960°C ,when the deformation amounts are at 47%, 55%, 70%, the difference of recrystallization incubation time is about 5-10 minutes, and the finishing recrystallization time is 20-30 minutes. All curves have the same phenomena, The speed of recrystallizaion beginning is low and it increases with the recrystallizaion process. When the recrystallizaiton volume fraction exceeds to 20%~70%, the speed is the largest, but it decreases subsequently. The speed is lower at the surplus process till the recrystallization finishing. Give an example applying the deformation amount of 55% and annealing temperature of 940E, the recrystallizs number increases from 1% to 17% when the annealing time is from 15 minute to 25minute and it be increase continuously from 20% o 70% in the later 25 minutes, the recrystallization finishes at 65minute.

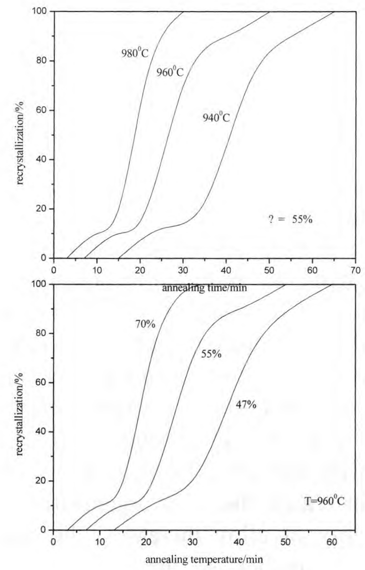


Figure 4. Recrystallization kinetics

Figure 5. shows the recrystallization microstructures under the different deformation amounts. The sizes of recrystallization grain are different under the different deformation amounts. The average size of recrystallization grain is 15μm at the 17% deformation amount and the average grain is 6μm under the deformation amount of 47%. When the deformation amount is 70%, the average grain is only 1μm. It can be seen that the size of recrystallization grain reduces with the in crease of deformation amount. It is not difficult to explain the above phenomenon applying the metallic theory. The recrystallization driving force is mostly the storage energy which is preserved by dislocation form when the deformation applies to the alloy. The nucleus ratio of recrystallization process increases with the deformation amount increase [7]), The storage energy is more with the deformation amount increasing, it will improve consumedly the nucleus ratio of recryatallization, as a result, the grain size is smaller.

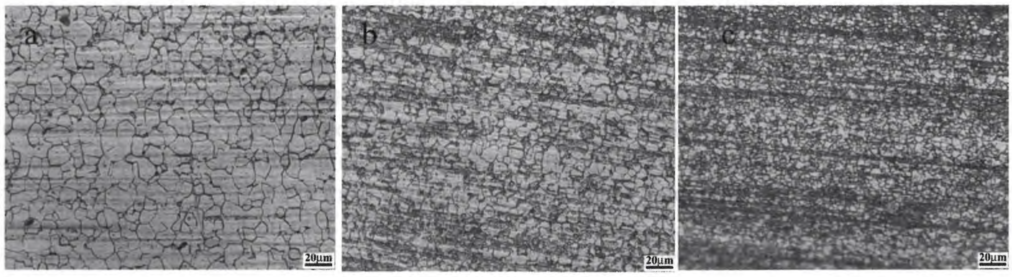


Figure 5. Recrystallization microstructures.
（a,△＝17％; b,△＝55％; c,△＝70％）

4. Conclusion

When titanium tantalum alloy is drawed into fine wire, the obvious dynamic recrystallization behavior do not happen because of drawing-work temperature being low. The recrystallization nucleus start to appear in the grain boundary of wire edge after annealing treatment.

The recrystallization activate engery decreases with the deformation amount increasing. The reason was relationship with the crystal lattice distortion. When the deformation increases, the alloy organization crystal lattice distortion energy of alloy increases, the recrystallization driving force is smaller, so the recrystallization activate energy was smaller. For example, when the deformation amount is 17%, the recrystallization activate engery is 190.9KJ/mol, when the deformation amount was 33%, the result is 169.5KJ/mol. In the paper, the author calculate the recrystallization activate energy on different deformation amount through the formale of $G = G_0 \bullet \exp^{(-Q/RT)}$, the result shows that when the deformation amount individual is 17%, 33%, 47%, 55%, 70%, 79%, the recrystallization activate energy individual is 190.9kJ/mol, 169.5 kJ/mol, 119.1 kJ/mol, 118.8 kJ/mol, 113.1 kJ/mol, 86.8 kJ/mol.

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