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ARTICLE

Effect of Heat Treatment on Microstructure and Mechanical Properties of Ultra-fine Grained Ti-55511 Near β Titanium Alloy

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Abstract: The ultra-fine grained (UFG) Ti-55511 near β titanium alloy with grain size 0.1~0.5 μm was prepared by hot rolling. The effects of heat treatment on the microstructure and mechanical properties were investigated using SEM and TEM. The results indicate that both strength and hardness increase firstly and then reduce with increasing of heat treatment temperature from 350 ℃ to 650 ℃. The peak strength (1486 MPa) appears at 450 ℃. The strength dramatically reaches to 1536 MPa and then is stablized with increasing of the holding time when heat treated at 450 ℃. While the elongation increases firstly and then decreases. The microstructure analysis shows that the dynamic recovery occurs and the grain sizes remain at smaller than 1 μm during annealing. The recovery stimulates the grain refinement effect by eliminating the hardening process and stimulating the grain boundary/phase boundary to be stable. The phase transformation of $\alpha\rightarrow\alpha_2$ and $\beta\rightarrow\omega\rightarrow\alpha$ enhances the second phase particle dispersion effect during annealing. However, the ductility of the alloy could significant decrease when the second phase particles grow up to a certain size. The mechanical properties evolution during annealing are mainly related to the effect of strengthening mechanisms.

Key words: Ti-55511 alloy; ultra-fine grain; heat treatment; microstructure; mechanical property

High strength and toughness near β titanium alloys such as Ti-55511(Ti-5Al-5Mo-5V-1Cr-1Fe) and Ti-1023(Ti-10V-2Fe-3Al) have a number of advantages such as good processability, easy to achieve near-net shape forming, and their excellent comprehensive mechanical properties can be obtained by adjusting strength and toughness[1-5]. Thus such a kind of high performances of lightweight and high strength structural materials are not only required in the area of aviation[6,7], but also widely used in the fields of automobile, vessel and sport appliances.

It is a very important method to improve the performance of titanium alloy by refining grain[8,9]. The severe plastic deformation (SPD) such as equal-channel angular pressing[10], high-pressure torsion[11], accumulative roll bonding[12] and multi-axial forging[13] is a significant technique to prepare the UFG titanium alloy. However, the conditions of SPD technique to realize grain refinement of titanium alloy are very rigorous[14-16]. However, based on the characteristic of microstructure evolution during deformation process of near β titanium alloy, it is possible to realize the grain refinement by adjusting the microstructure before deformation to reduce the requirements of SPD. Li Chao etc.[17] acquired UFG with grain size 0.2~0.8 μm by thermocompression. But the research about the correlation of microstructure evolution and mechanical properties of this UFG near β titanium alloy is still blank.

At home and abroad, the research of near β titanium alloy heat treatment was focused on the microstructure with grain size 3~20 μm which was prepared by near β forging or $\alpha+\beta$ forging. Taking an example of Ti-55511 alloy[18], the typical process of heat treatment was solid solution under T_β–20~50 ℃×1~1.5 h→ furnace cooling to T_β–80~120 ℃×1~3 h→ air cooling to room temperature→300~650 ℃×2~6 h. The microstructure after the heat treatment contained 30%~40%

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globular α, 10% thick lamellar α and 10% thin lamellar α in volume fraction. After the heat treatment, the strength and the toughness of the near β titanium alloy will be matched between tensile strength 1080~1280 MPa, fracture toughness 55~75 MPa·m$^{1/2}$ [19].

However, there exist high residual stress and distortion energy in the UFG microstructure prepared by SPD. The conventional heat treatment might significantly increase grain sizes, and reduce grain refinement effects. Therefore it is necessary to design a new kind of strengthening-toughening heat treatment on the premise to guarantee the UFG characteristic. A lot of research showed that the heat treatment of UFG titanium alloy usually adopted the single stage annealing at low temperature of 200~600 ℃. D. Kent etc.[20] studied the thermostability of UFG metastable β titanium alloy Ti-25Nb-3Zr-3Mo-2Sn, and discovered that single stage annealing at 400~600 ℃ could reduce the density of dislocation/defect by taking advantage of recovery and dispersed precipitating of second phase, and enhanced the stability of the subgrain/grain on the premise to guarantee the UFG characteristic. In addition, there might be a series of special phase transformation in the UFG titanium alloy during low temperature annealing under the influence of high residual stress and distortion energy. W. Xu etc. [10] studied the Ti67.4Nb24.6Zr5Sn3 (TNZS, at%) β titanium alloy, and discovered the $\beta\rightarrow\alpha''$ stress-induced martensitic transformation occurred during ECAP, further reverse transformation of $\alpha''\rightarrow\beta$ would happen during aging process. However, in the domestic and overseas it was rarely reported about the research of the microstructure evolution and the phase transformation of high strength and toughness near β titanium alloy such as Ti-55511 during heat treatment process. So, it is of important theoretical value to research the heat treatment of UFG near β titanium alloy.

1 Experiment

The as received Ti-55511 alloy was provided by Xiangtou Goldsky Titanium Industry with the composition (in wt%) of 5.75Al, 5.42Mo, 4.48V, 0.75Cr, 1.2Fe, balanced Ti, impurity less than 0.3, and the β-transus temperature is 875 ℃±5 ℃. The microstructure of the as received billet is shown in Fig.1.

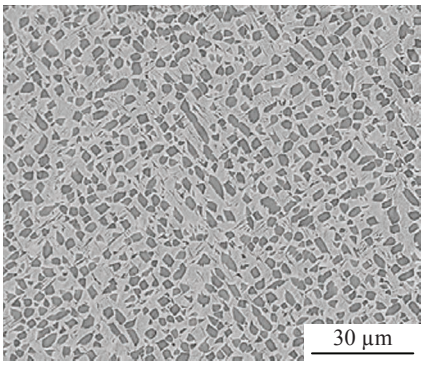

Fig.1 Microstructure characteristic of as received Ti-55511 billet

According ref.[17], UFG microstructure could be obtained by hot compression at a 0.7 true strain for which the initial microstructure had 50~60 vol% acicular α (0.1~0.5 μm wide, 1~5 μm long). In the present work, samples were first heat-treated at 920 ℃ for 1 h, followed by water quenching and then aged at 700 ℃ for 1 h. After the heat treatment, the samples with the microstructure similar to the described above were used for UFG hot rolling. They were hot rolled to 3 mm thickness with 2 passes on the LO-500 rolling machine, the deformation degree was 50% for the first pass and 40% for the second pass. In order to retain the deformed microstructure, the samples were water quenched immediately after hot rolling. Tensile samples were gained along the rolling direction (RD) and then soaked at 350~650 ℃ for 0.5~32 h in tube resistance furnace with Ar protecting.

Tensile experiment was conducted on Instron8802 mechanic machine. HRC hardness was tested on Huayin 200HRS-150 Rockwell apparatus with a loading of 1470 N and a hold time of 5 s. The specimens for microscopy were etched in the solution of HF-HNO_3 (the volume ratio of HF-HNO_3-H_2O was 1.5:3:100) and observed on NOVATM Nano SEM 230 scanning electron microscope (SEM). TEM analyze was carried on using JEM-2100F transmission electron microscope with acceleration voltage as 200 kV. TEM specimens were prepared as follows: specimens were first wire-electrode cut into 0.3 mm slices, polished on abrasive paper to the thickness of 80 μm and punched into Φ3 mm circles. Then the circles were thinned till piercing by TENUPOL-5 twin-jet electropolishing machine using a solution of 600 mL CH_3OH, 350 mL $CH_3(CH_2)_3OH$, 50 mL $HClO_4$ at –30 ℃~–35 ℃.

2 Results and Discussion

2.1 Microstructure before and after rolling

The microstructures of Ti-55511 alloy before and after rolling are shown in Fig.2. From the microstructure before rolling (Fig.2a), it can be found that through 920 ℃×1 h→water quenching→700 ℃×1 h, about 50 vol% acicular α precipitates on β grain boundary or inside of β grain. The width of grain boundary α is 0.2~0.4 μm. The acicular α inside of β grain has the boundary of 0.1~0.3 μm in width and 1~5 μm in length. The β matrix is divided by the acicular α into small sections of 0.2~0.8 μm. From the microstructure after rolling (Fig.2b), the initial continuous grain boundary and the acicular α through break/spheroidize form with size of 0.1~0.4 μm equiaxial α. Further observation of TEM microstructure (Fig.2c) indicates that the β grains are broken and their sizes are 0.3~0.5 μm. The high magnification microstructure (Fig.2d) reveals the mass of dislocations tangle inside the broken α grain and the β grain, and the α/β phase boundary is twisted.

2.2 Annealing microstructure of UFG

Fig.3 shows the microstructures of UFG Ti-55531 alloy after annealing at different temperatures for 4 h. In the range of 350~650 ℃, no obvious grain growth is observed. Compared

with the microstructure before annealing (Fig.2c), the microstructure after annealing at 350 ºC reveals that there exist lots of remnant dislocations and the interfaces of α/β are indistinct (Fig.3a). When the annealing temperature reaches 450℃, the density of dislocation drops, but the interfaces of α/β are still indistinct (Fig.3b). After annealing at 550 ºC, the interfaces of α/β and β/β gradually become distinct, the polygonization is obvious and the grain sizes are 0.2~0.8 μm (Fig.3c). When annealing at 650 ℃ (Fig.3d), the grain sizes of α and β slightly grow and the subgrain boundaries of α/α and β/β disappear. The main factor for the phenomenon described above can be explained as follows: upon annealing at the range of 350~650 ℃, a recovery occurs in the UFG alloy and the degree of the recovery rises with the temperature increment. Through slipping and climbing, dislocations move to phase boundaries and grain boundaries during the recovery. Thus the α phase and β phase are divided into subgrains, and the subgrain boundaries gradually become distinct with the temperature increasing in the range of 350~550 ℃. When the temperature reaches 650 ℃, the α and β subgrains begin to merge and grow up, resulting in leading to the subgrain boundary disappearance. In addition, comparing the diffraction pattern of Fig.2c with that of Fig.3, it is obvious that after rolling deformation, the diffraction patterns are continuous and the rings are clear, which means that the misorientation of the grains are large. The diffraction patterns become less continuous with the increase of the annealing temperature, which means the misorientation of the grain decreases.

Upon holding at 250~500 ℃, a series of complex phase transformations such as $\beta\rightarrow\omega\rightarrow\alpha$ and $\beta\rightarrow\alpha$ may take place in Ti-55511 near β titanium alloy. In this study, the selected area electron diffraction of TEM was used to analyze the phase transformation of UFG during annealing. Fig.4a shows that some acicular precipitates with length less than 100 nm and

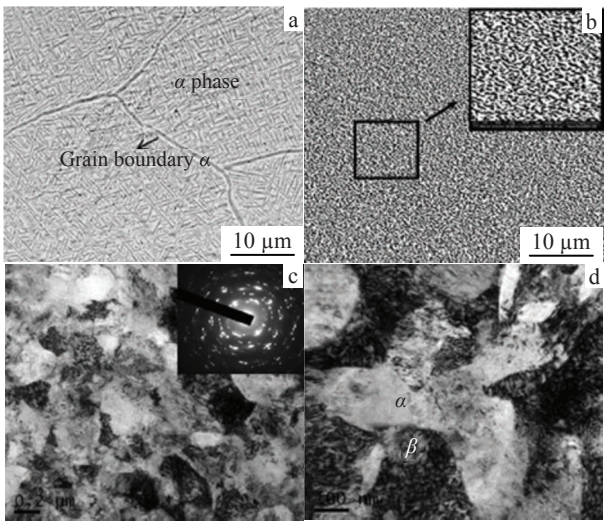

Fig.2 Microstructure characteristics of Ti-55511 alloy before (a) and after rolling (b~d)

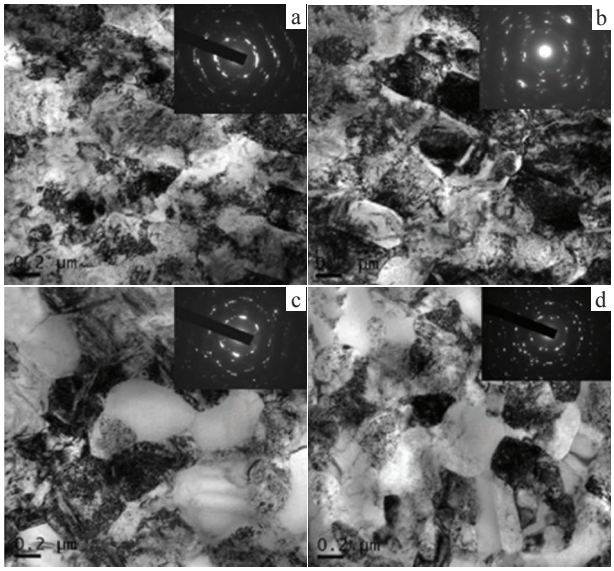

Fig.3 Microstructure characteristics of Ti-55511 alloy after annealing for 4 h at different temperatures: (a) 350 ℃, (b) 450 ℃, (c) 550 ℃, and (d) 650 ℃

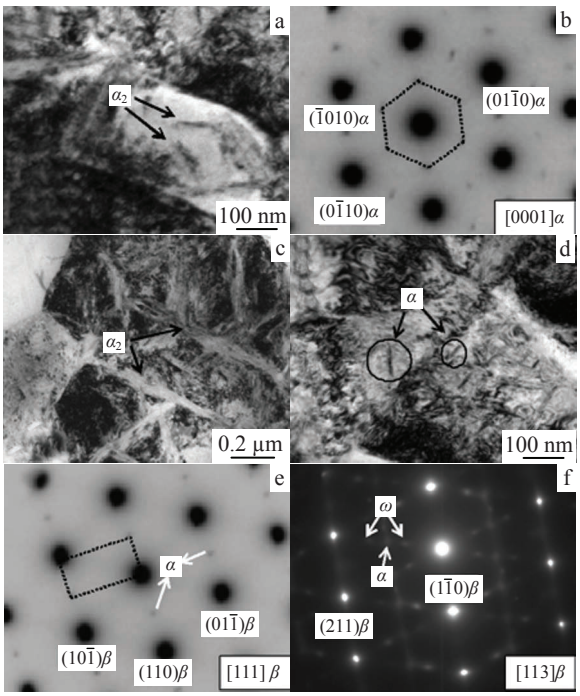

Fig.4 Phase transformations during annealing: (a, b) 450 ℃×4 h, $\alpha\rightarrow\alpha_2$, (c) 450 ℃×8 h, $\alpha\rightarrow\alpha_2$; (d~f) $\beta\rightarrow\omega\rightarrow\alpha$

width 5~20 nm precipitate within the α phase. In the diffraction pattern image at [0001]α zone, superlattice spots emerge at the location of [h/2, k/2, l]. In titanium alloys, the superlattice spots are caused by the long-range order transformation from α to α_2. The α_2 is a kind of long-range order phase based on Ti_3Al with a D019 structure and P63/mmc space group. α_2 mainly precipitates from α phase through the coherent relationship. The Al

atoms orderly distributed on the (0001) of α form lattice constant which is twice a axis of α phase, resulting in leading to the appearance of superlattice spots. The α_2 precipitates meet the orientation relationship of $(0001)\alpha_2//(0001)\alpha$ and $<11\bar{2}0>\alpha_2//<11\bar{2}0>\alpha$. During the annealing of Ti-55511 UFG alloy, the acicular α_2 gradually grows with the increase of annealing temperature and holding time from 450 ℃ to 550 ℃ (Fig.3c) and for 4 h to 8 h (Fig.4c), respectively. When the temperature reaches 650 ℃, no α_2 precipitates inside α phase. Normally, the α_2 phase is found in α, near α and $\alpha+\beta$ titanium alloys with high content of α-stable elements [21, 22]. However, in the present research the α_2 precipitates inside α phase is firstly found in near-β titanium alloy. The main reason for this phenomenon is probably related to the impact of high distortion energy in the UFG microstructure.

Fig.4d shows the microstructure after annealing at 450 ℃ for 4 h. Some acicular precipitates with length less than 100 nm and width of about 5~15 nm precipitate inside the β phase. Through the analysis of the diffraction pattern of the $[111]\beta$ zone and $[113]\beta$ zone (Fig.4e and 4f), it is found that ω and α phases both exist in the β phase matrix. N. G. Jones [23] et al research suggested that upon soaking in the range of 350~500 ℃, the $\beta\rightarrow\omega$ phase transformation took place in the near-β titanium alloys. With the increase of the soaking time, the ω phase accelerated α phase nucleation and growth on or from a certain distance of the ω/β interface, which makes the acicular α thin and dispersive. The ω and α phases can be observed simultaneously when the UFG Ti-55511 is aged at 450℃ which indicates both taking place of the phase transition of $\beta\rightarrow\omega$ and $\beta\rightarrow\omega\rightarrow\alpha$. However, when the annealing temperature increases from 450 ℃ to 550 ℃ and 650 ℃ (Fig.3c and 3d), respectively, no precipitation of acicular α in the β matrix is observed. Because 550 ℃ is higher than the phase transformation temperatures of $\beta\rightarrow\omega$ and $\beta\rightarrow\omega\rightarrow\alpha$ and the direct phase transformation of $\beta\rightarrow\alpha$ dominates. During the direct phase transformation of $\beta\rightarrow\alpha$, α phase nucleates at the interface of α/β containing high dislocation density and grows attaching to α phase. Because of α phase nucleation near the interface of α/β and its growth and attaching to the phase boundary, the indistinct and twisted phase boundary (Fig.2d) of the UFG before annealing gradually become distinct and smooth (Fig.3c and 3d).

2.3 Mechanical properties

In order to evaluate the mechanical properties of Ti-55511 alloy with UFG microstructure, the tensile strength and hardness were tested. The tensile strength of UFG Ti-55511 alloy is 1304 MPa, which is much higher than that of the forged alloy with 1080 MPa. Through the analysis of the microstructure, grain refinement and deformation strengthening are considered to be the two main strengthening mechanisms. On one hand, after deformation the grain sizes are about 0.1~0.5 μm and the grain boundary/phase boundary increases, which leads to the huge blocking effect on dislocation slippage. On the other hand, alloys with severe plastic deformation contain high density of dislocation, which makes the elastic stress field increasing and the interaction between dislocations strengthened. Thus the motion of dislocation becomes more difficult and causes work hardening.

Fig.5 shows the relationship between heat annealing temperature and mechanical properties of UFG Ti-55511 alloy annealed for 4 h. It is clear that with the increase of annealing temperature from 350 ℃ to 650 ℃, tensile strength and hardness both present the tendency of first increasing and then decreasing. The peak hardness (45.4) and the peak tensile strength (1486 MPa) both appear at 450 ℃. Fig.6 shows the relationship between holding time and mechanical properties of UFG Ti-55511 alloy upon annealing at 450 ℃. With the extension of holding time, tensile strength gradually increases and reaches the peak of 1536 MPa at 16 h, then tends to stablizing. The elongation presents the tendency of first increasing and then decreasing. The elongation reaches 8.54% at 4 h and decreases rapidly to 4.47%. This phenomenon is closely related to microstructure evolution.

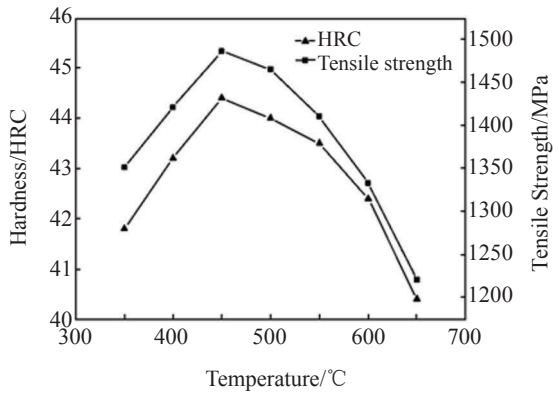

Fig.5 Relationship between heat annealing temperature and mechanical properties of ultra-fine grained Ti-55511 alloy upon holding for 4 h

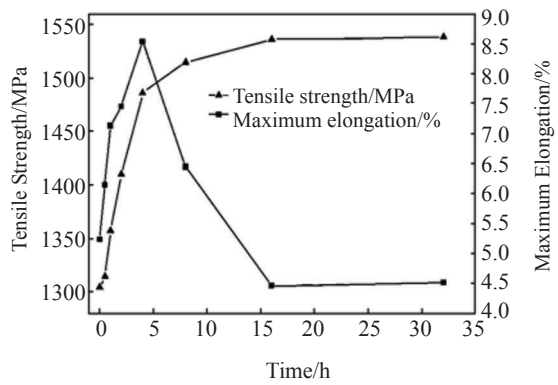

Fig.6 Relationship between holding time and mechanical properties of ultra-fine grained Ti-55511 alloy upon annealing at 450 ℃

2.4 Relationship between mechanical properties and microstructure

After annealing at 350~650 ℃ for 4 h, tensile strength significantly increases in the range of 350~450 ℃ and decreases with further rise of temperature. Under all sorts of heat treatment condition, the density of dislocation greatly drops. The residual stress, defect density and distortion energy decrease which lead to the reduction of work hardening effect. However, grain/phase boundaries of α/α, α/β and β/β tend to be stable and dislocations tangle transforms into subgrain. Thereby the hindrance effect of grain/phase boundary on the movement of dislocation enhances and the effect of fine grain strengthening is obvious. Meanwhile, phase transformations of $\alpha\rightarrow\alpha_2$ and $\beta\rightarrow\omega\rightarrow\alpha$ occur during annealing in the range of 350~450 ℃. The second phase particles dispersion strengthening effect increases due to the precipitation of acicular α_2 and α in α and β phase, respectively. Under the double effects of fine grain strengthening and second phase particles dispersion strengthening increases, tensile strength and hardness of the alloy rise. When the temperature is above 500 ℃, the direct phase transformation of $\beta\rightarrow\alpha$ occurs due to the temperature being higher than that of the precipitation interval of ω phase. In this case, α phase nucleates at the interface of α/β which contains high density of dislocation. Then α phase growing attached on the broken α phase leads to no precipitation of dispersed acicular α and the decrease of second phase strengthening effect, thus the tensile strength decreases. When the temperature is above 550 ℃, a further decrease of second phase particles dispersion strengthening and the tensile strength can be observed due to the temperature being above that of the both precipitation interval of ω and α_2 phase. The combination and growth of β subgrain occur due to the recovery of β phase as the temperature rises above 650 ℃ and the strengthening effect of grain/phase boundary weakens, so the tensile strength reaches the bottom. The tensile strength and hardness of the alloy reach the peak due to the highest effect of fine grain strengthening and second phase particles dispersion strengthening when annealed at 450 ℃ for 4 h.

Upon holding at 450 ℃, the tensile strength and the elongation of the alloy increase rapidly with the extension of the holding time from 0~4 h. During 4~32 h, the tensile strength slowly increases and tends to be constant, but the hardness decreases significantly. When annealing time is between 0 and 4 h, the phase transition of $\alpha\rightarrow\alpha_2$ and $\beta\rightarrow\omega\rightarrow\alpha$ develop rapidly and the sizes of α_2 and α increase remarkably, leading to the sharp increase of the tensile strength. Affected by the decrease of dislocations, defects and distortion energy during the early stage of annealing, work hardening is eliminated and the elongation increases notably. As the extension of annealing time, the phase transformations of $\alpha\rightarrow\alpha_2$ and $\beta\rightarrow\omega\rightarrow\alpha$ are completed. Thereby the tensile strength increases slowly and trends to be steady. However, the acicular α_2 and α phase would grow up with the extension of annealing time. When the acicular α_2 and α reach a certain size, the hindrance on the slippage/climbing of dislocations increases, which lowers the elongation of the alloy.

Through the analysis above, the main strengthening mechanism of UFG Ti-55511 titanium alloy during annealing can be realized: fine grain strengthening and second phase particles dispersion strengthening. The process of recovery eliminates the work hardening and accelerates the stabilization of grain/phase boundary, which promote the effect of fine grain strengthening. Through the phase transition of $\alpha\rightarrow\alpha_2$ and $\beta\rightarrow\omega\rightarrow\alpha$ during annealing, second phase of acicular α_2 and α precipitate inside α and β phase, and the effect of second phase particles dispersion strengthening increases. When the size of the second phase particles increases to a certain level, however, the elongation decreases significantly.

3 Conclusions

1) The α/β grains in Ti-55511 UFG near-β titanium alloy prepared by rolling can be refined into the sizes of 0.1~0.5 μm and contain a lot of dislocations. The tensile strength of the alloy is 1304 MPa and the elongation is 5.24%. In the annealing range of 350~650 ℃, a recovery mainly takes place in the alloy. The sizes of the grains are less than 1 μm and no obvious growth is observed. Phase transformations of $\alpha\rightarrow\alpha_2$ and $\beta\rightarrow\omega\rightarrow\alpha$ will occur during annealing.

2) As the annealing temperature increases, the tensile strength and the hardness increase first and then decrease. The highest hardness (45.4) and tensile strength (1486 MPa) appear at 450 ℃. Upon annealing at 450 ℃, the tensile strength increases with the extension of holding time and reaches 1536 MPa at 16 h and then tends to stabilization. The elongation increases and reaches 8.54% at 4 h, then decreases rapidly to 4.47% at 16 h.

3) The main strengthening mechanisms of UFG Ti-55511 titanium alloy during annealing include: fine grain strengthening and second phase particles dispersion strengthening. The process of recovery will eliminate the work hardening and accelerates the stabilization of grain/phase boundary, thus promotes the effect of fine grain strengthening. Through the phase transition of $\alpha\rightarrow\alpha_2$ and $\beta\rightarrow\omega\rightarrow\alpha$ during annealing, the effect of second phase particles dispersion strengthening increases. When the size of second phase particles increases to a certain level, however, the elongation decreases significantly.

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