Influence of Nb addition on microstructure evolution and superplastic behavior of Ti-5Al-5Mo-5Cr-2Zr-xNb titanium alloy at 923 K

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Abstract: Ti-5Al-5Mo-5Cr-2Zr-*x*Nb with different Nb (abbreviated as Ti-5552-*x*Nb, *x*=3, 6, 9, 12, wt.%) contents were stretched at 923 K to study their superplastic behavior and mechanical properties below recrystallization temperature. The microstructure of as-cast Ti-5552-*x*Nb alloy is consisted of a single β phase, and the β grain size increases slightly with the increase of Nb content. The thermal effect in the process of high temperature drawing leads to the precipitation of α phase. The addition of Nb in Ti-5552 titanium alloys reduces the α/β phase transformation temperature, which causes a decrease in the volume fraction of α phase. Reducing the α phase content reduces incompatibility, but too low a proportion of α phase will lead to premature fracture, so tensile strength and plasticity firstly increase and then decrease. The results show that Ti-5552-9Nb titanium alloy shows the best tensile strength (307.2 MPa) and superplasticity (106%). The superplastic mechanism of Ti-5552-9Nb alloy is mainly caused by relative sliding of β grain boundaries and dislocation movement.

Keywords: titanium alloy; niobium element; superplastic; mechanical properties

CLC numbers: TG146.23 **Document code:** A **Article ID:** 1672-6421(2026)01-055-07

1 Introduction

Titanium alloy is widely used in various industries because of its excellent mechanical properties and high corrosion resistance[1-3]. With the development of aerospace, the application of titanium alloy has been paid more and more attention[4-6]. Cast titanium alloy has poor mechanical properties because of its coarse β grains, thus proper thermal deformation is considered necessary[7, 8]. Because of its softening mechanism, titanium alloy shows excellent superplasticity when temperature is higher than 1,073 K[9, 10]. In contrast, the plasticity is lower at lower temperatures. If superplasticity at 923 K can be achieved, it plays an important role in reducing deformation energy consumption and shortening the production process[11, 12]. For this reason, achieving superplasticity of titanium alloys at lower temperatures has attracted widespread attention from researchers. Among the current methods, alloying is one of the most promising ways.

Nb is an important alloying element in titanium alloy, which significantly improves the mechanical properties of titanium alloy[13]. In the previous research of our group, the addition of Nb element into the titanium alloy resulted in a significant improvement in room temperature strength[14]. In addition, Nb is also a β-stable element that plays a role in regulating the ratio of α/β phase[15]. Researchers have found that the ratio of α/β phase directly affects the superplasticity at 1,023 K[9]. At present, the research on superplasticity is still conducted at higher temperatures, and the mechanism by which Nb influences superplasticity at lower temperatures still needs to be revealed.

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Received: 2024-11-18; Revised: 2025-02-10; Accepted: 2025-02-17

A temperature of 0.5T_m (T_m is the melting point of the material, K) is usually defined as the critical temperature of superplasticity. Research on the superplasticity of titanium alloys has predominantly focused on temperature ranges above 0.5T_m, and the superplastic deformation behavior and mechanisms at temperatures below this threshold remain unclear. Therefore, this work delved into the influence mechanisms of the superplasticity of the newly developed ultra-high-strength and ductile Ti-5Al-5Mo-5Cr-2Zr-*x*Nb alloys by our team[14]. Through the addition of varying Nb contents, the effect of Nb on the mechanical properties and plasticity of the alloy at 923K (below 0.5T_m) were elucidated.

2 Experimental materials and methods

The raw materials with composition Ti-5Al-5Mo-5Cr-2Zr-*x*Nb (wt.%, *x*=3, 6, 9, 12, abbreviated as Ti-5552-*x*Nb) used in the experiment were melted by vacuum induction melting, and the specific method was reported by our previous work[14]. The α/β phase transition temperature of Ti-5552-*x*Nb alloys measured by the metallographic observation method was 1,098 K, 1,073 K, 1,048 K, and 1,023 K. The tensile sample with the gauge length of 5 mm×2 mm×20 mm was cut from the ingot. After holding for 5 min at 923 K, the tensile test was completed at a speed of 0.5 mm·min^{-1} on the universal testing machine (Instron 5500R), and was heated continuously during the stretching process. After the stretching was completed, quickly removed the sample and water cooled to room temperature to maintain the microstructure at 923 K. The pretreatment process of samples was also shown in previous work[14]. The metallographic microstructure was observed using a laser confocal microscope (VL2000DX-SVF18SP). Further microstructure characterization was performed with a Quanta 200F scanning electron microscope and a Talos F200×transmission electron microscope.

3 Results and discussion

3.1 Microstructure and phase constitution of as-cast Ti-5552-*x*Nb alloy

Figure 1 shows the melting point, phase transformation point, and critical temperature of superplastic deformation of Ti-5552-*x*Nb alloy. As shown in Fig. 1, the melting point of titanium alloy increases gradually from 1,945 K to 1,972 K with the increase of Nb content, because Nb is a high melting point element. Correspondingly, the superplastic temperature also increases. On the contrary, the β stable element Nb reduces the α/β phase transformation temperature from 1,098 K to 1,023 K, which indicates that the stability of β phase is improved with the increase of Nb content.

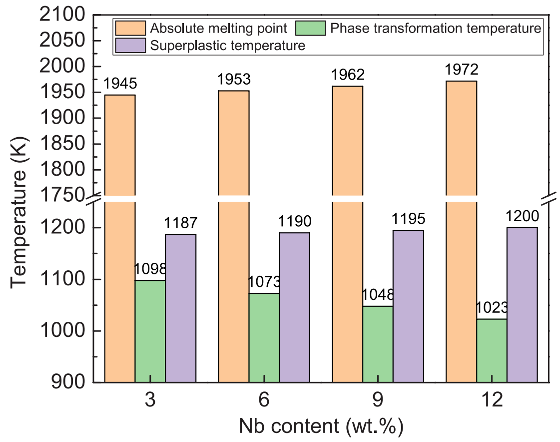

Fig. 1: Melting point, phase transformation points, and superplastic temperature of Ti-5552-*x*Nb alloys

The microstructure of as-cast titanium alloy is shown in Fig. 2. The observation in the microstructure shows that the structure is composed of clear grain boundary and single β phase, and the precipitation of α phase is not observed. The β grain of Ti-5552-3Nb alloy is approximately equiaxed, and its aspect ratio increases with the increase of Nb content. As shown in Fig. 2(a), the average β grain size of the Ti-5552-3Nb alloy is approximately 190 μm. As the Nb content increases to 6wt.%, the size of the β grains in the lengthwise direction increases to about 240 μm. As shown in Fig. 2(c), when the Nb content is 9wt.%, the average β grain size is about 370 μm. The largest β grain size is observed in Fig. 2(d), with an average value of about 470 μm, due to the continuous increase in Nb content to 12wt.%. In general, the β grain size increases with increasing Nb content[16]. Nb is a β stabilizing element that supresses the β to α phase transition by weakening component supercooling[17, 18]. Therefore, grains continue to grow in a single β phase. Eventually, the size of the β grain in the microstructure shown in Fig. 2 increases with the increasing Nb element content.

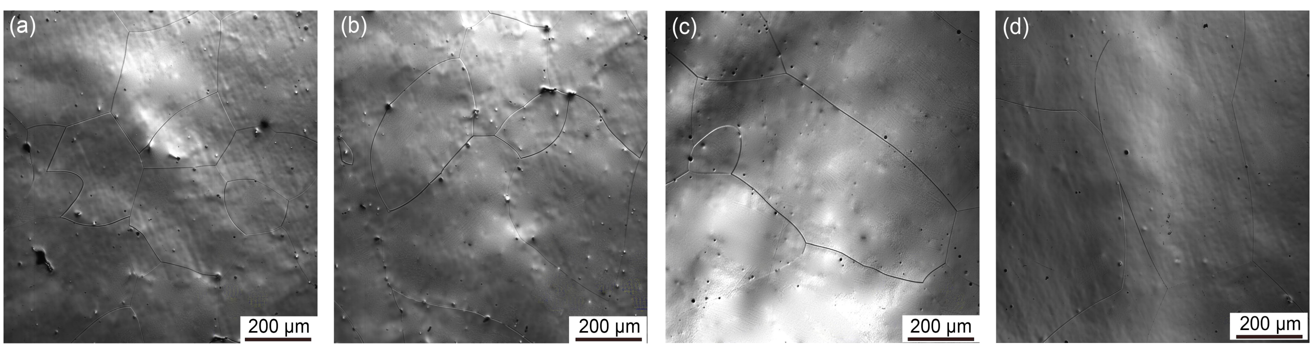

Fig. 2: Microstructures of as-cast titanium alloy with different Nb contents: (a) 3wt.% Nb; (b) 6wt.% Nb; (c) 9wt.% Nb; (d) 12wt.% Nb

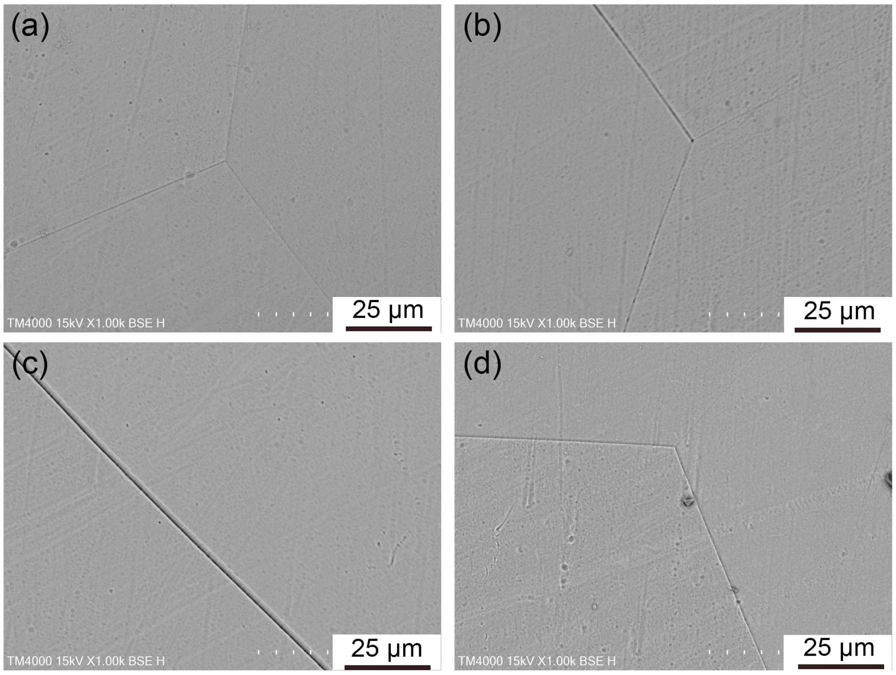

Fig. 3: SEM microstructure of as-cast titanium alloy with different Nb contents: (a) 3wt.% Nb; (b) 6wt.% Nb; (c) 9wt.% Nb; (d) 12wt.% Nb

To characterize the as-cast microstructure in detail, the SEM images of as-cast titanium alloys with different Nb contents are shown in Fig. 3. The microstructure is composed of a single β phase, and no grain boundary α phase is observed at the straight β grain boundary. The above results show that the increase of Nb content does not change the phase constitution of Ti-5552-*x*Nb alloy, because the β stable element Nb does not promote the precipitation of α phase.

3.2 Stress-strain curves and microstructures of 923 K high temperature tensile sample

The Ti-5552-*x*Nb alloy was stretched at 923 K. The stress-strain curves and morphologies before and after testing are shown in Fig. 4. The macroscopic morphology shows that the Ti-5552-*x*Nb alloy is oxidized to different degrees; all the tensile samples show necking phenomenon, and the fracture surface morphology presents a dimpled rupture pattern. The stress-strain curves show that with the increase of Nb content in the range of 3wt.%–9wt.%, the stress of Ti-5552-*x*Nb alloys increases from 158.6 MPa to 307.2 MPa, and the elongation increases from 75% to 106%. It can be seen that Ti-5552-9Nb alloy not only shows higher strengths, but also shows superplasticity as high as 106%. When the Nb content further increases to 12wt.%, the stress is 212.1 MPa and the strain is 100.1%, which is slightly lower than that of Ti-5552-9Nb alloy.

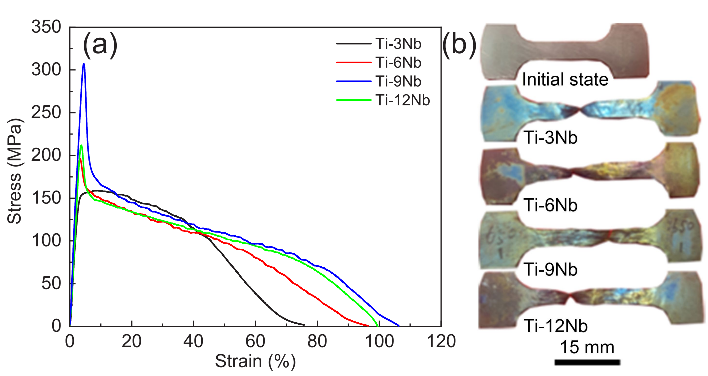

Fig. 4: Stress-strain curves at 923 K (a) and sample morphologies before and after testing (b)

In the process of high temperature tension, the heat treatment furnace of the tensile testing machine continues to heat the sample, which is equivalent to heat treatment. Therefore, the microstructure of the unstressed area of the sample is observed, as shown in Fig. 5. As shown in Fig. 5(a), the microstructure of Ti-5552-3Nb alloy after high temperature tension is composed of β phase, intragranular lamellar α phase and grain boundary α phase. Some of the nearly equiaxed α phases in the crystal may be caused by the orientation relationship, and the spatial orientation is perpendicular to the visual field. In Fig. 5(b), the type of α phase in the microstructure does not change, but its content and aspect ratio decrease. This is because the increase in Nb content lowers the α/β phase transformation temperature, resulting in a decrease in the driving force for α phase precipitation. In Ti-5552-9Nb alloy [Fig. 5(c)], the morphology of intragranular α phase is mainly equiaxed, the grain boundary α phase is still continuous, and the content of α phase is about 15%. As shown in Fig. 5(d), as the Nb content increases to 12wt.%, the α phase content further decreases. The Nb content has little effect on the morphology of the α phase, and it still maintains a short lamellar state.

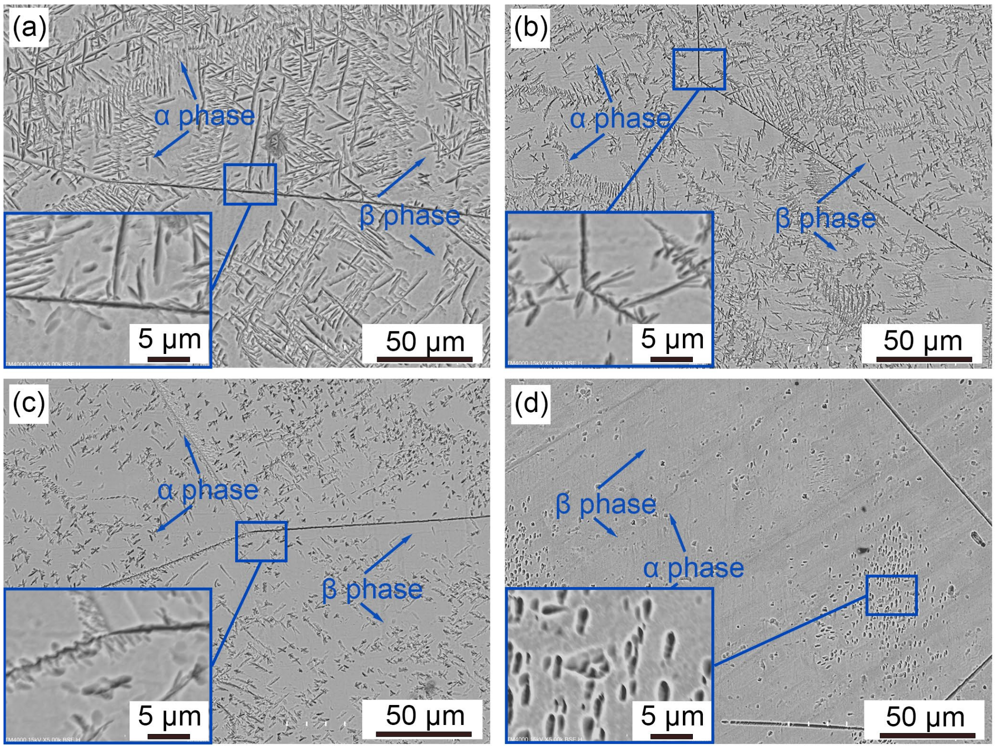

Fig. 5: Microstructure of Ti-5552-*x*Nb alloy in the non-stressed zone after high temperature stretching: (a) 3wt.% Nb; (b) 6wt.% Nb; (c) 9wt.% Nb; (d) 12wt.% Nb

The materials before and after high temperature tension are characterized by XRD, and the results are shown in Fig. 6. There is only a single peak of β phase in the as-cast microstructure. Due to the lattice distortion effect caused by Nb element, the peak of β phase shifts to the left compared to the Ti-5552-3Nb alloy. Nb exists in the β phase as a substitutional solid solution, and its atomic radius is slightly lower than that of Ti atom, which increases the crystal plane spacing of the β phase, resulting in a decrease in the diffraction angle θ. After high temperature stretching, the peak of α phase is observed in the microstructure. Since the content of α phase decreases with the increase of Nb content, the number and strength of the peaks decreases.

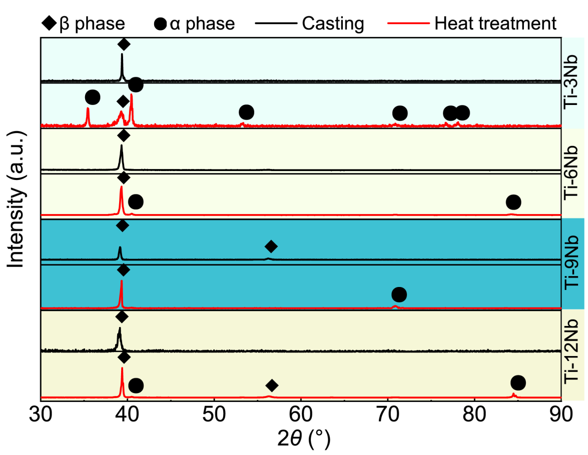

Fig. 6: XRD analysis results of titanium alloys with different Nb contents before and after tensile testing

3.3 Influencing mechanism of Nb on superplasticity

The microstructure near the fracture surface after high temperature tension is shown in Fig. 7, in which Figs. 7(a) and (b) show the microstructure of Ti-5552-3Nb alloy and Figs. 7(c) and (d) show the microstructure of Ti-5552-9Nb alloy. As shown in Fig. 7(a), the higher content of α phase precipitated during the heat preservation process before stretching captures most of the dislocations, and the dislocation density in the β matrix is low. As shown in Fig. 7(b), dislocation accumulation can be seen near the α phase in the TEM at a higher resolution, forming a dislocation wall, and the dislocation density inside the α phase is low. As shown in Fig. 7(c), the α phase content in the Ti-5552-9Nb alloy is low, and dispersed dislocations are observed in the β phase. It can be seen that the addition of Nb element pins the movement of dislocations in the β phase, which is an important reason for its significant improvement in strength. As shown in Fig. 7(d), a large number of dislocations are deposited near the α phase, and the dislocation density is significantly higher than that of the Ti-5552-3Nb alloy. The lower α phase content of the Ti-5552- 9Nb alloy makes it easier to adapt to deformation because the incompatibility between the α/β phases is suppressed. Therefore, the elongation of Ti-5552-9Nb alloy can reach 106%, which means that superplasticity is achieved.

The dislocation resolution near the α phase of Ti-5552-3Nb and Ti-5552-9Nb alloys is shown in Fig. 8. As shown in Figs. 8(a–c), the dislocation density of the α phase of the Ti-5552-3Nb alloy is low. The phase type is calibrated in combination with the SAED shown in the inset of Fig. 8(c).

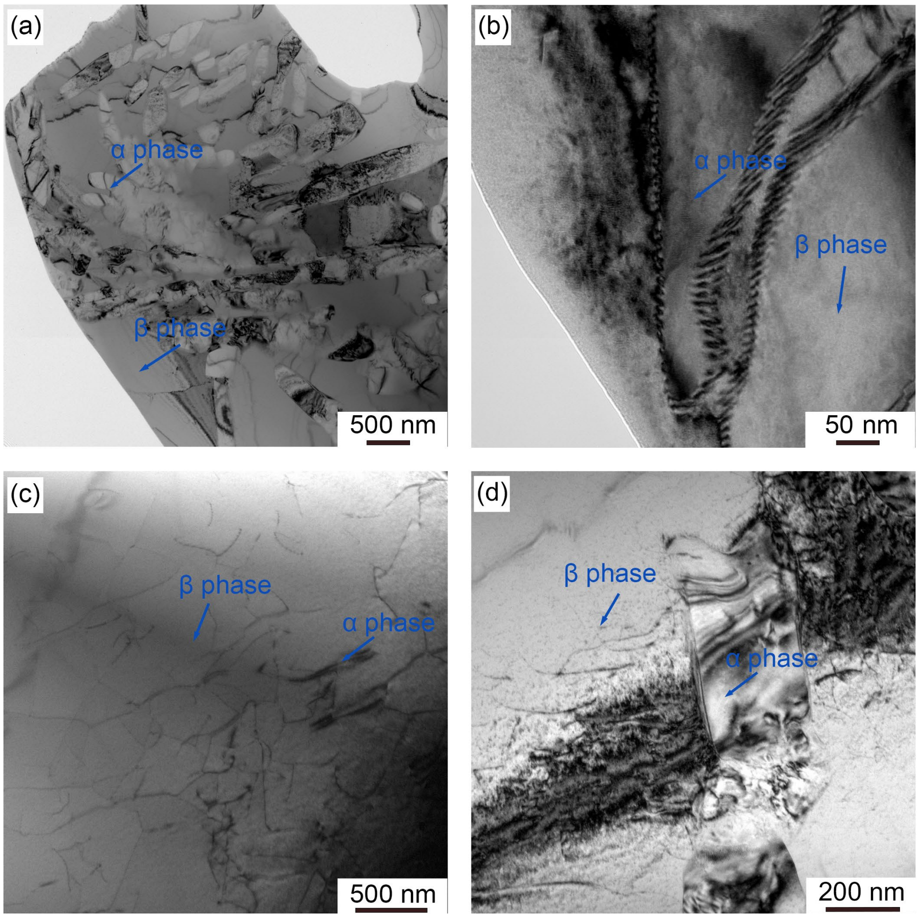

Fig. 7: Microstructure near the fracture surface after high temperature tension: (a, b) Ti-5552-3Nb alloy; (c, d) Ti-5552-9Nb alloy

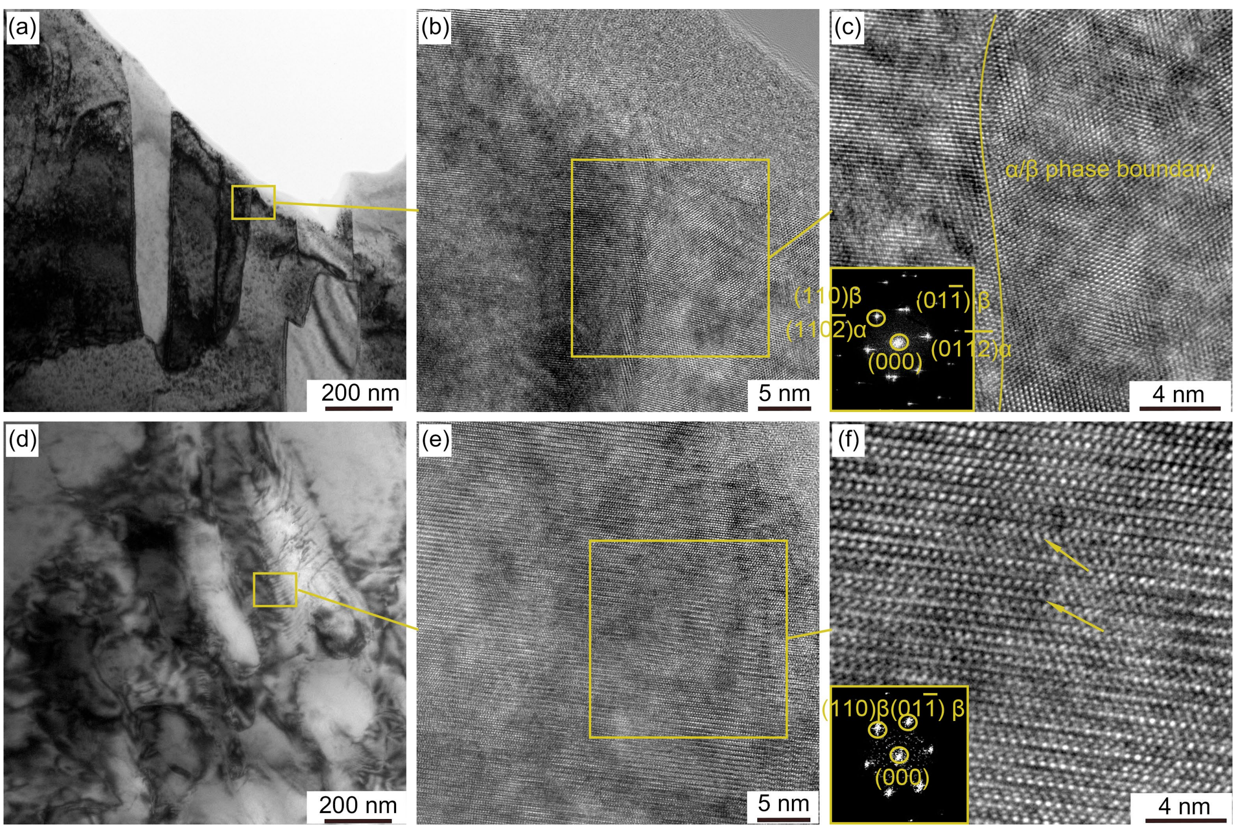

Fig. 8: Dislocations in α phase in high resolution: (a, b, c) Ti-5552-3Nb alloy; (d, e, f) Ti-5552-9Nb alloy

HRTEM shows that the orientation mismatch between the α and β phases is about 15°, and the β phase produces obvious lattice distortion at the position indicated by the arrow. Figures 8(d–f) show HRTEM at the interface of α and β phases in Ti-5552-9Nb alloy. The results show that since α phase hinders the slip of dislocation in the β phase, a large amount of dislocation plugging exists in the β phase near the α phase interface, as shown by the arrow, which leads to lattice distortion. This also confirms that the addition of Nb makes it easier for Ti-5552 titanium alloys to pin dislocations and thus obtain higher strength at 923 K.

Figure 9 shows the element distribution diagram near the α phase of Ti-5552-3Nb alloy. The Nb concentration difference between the α phase and β phase is lower than that of Cr and Mo elements due to the limitation of Nb addition and its solid solubility in the α phase (about 2.5wt.%). With the increase of Nb addition, the concentration difference between the two phases is bound to increase. It also can be seen the unlimited solid solution of Nb in the β phase exerts a significant strengthening effect. This leads to the formation of higher density of dislocations in high temperature stretching, which is the main reason for the significant increase in both high temperature strength and plasticity of titanium alloys with an increase in the content of Nb.

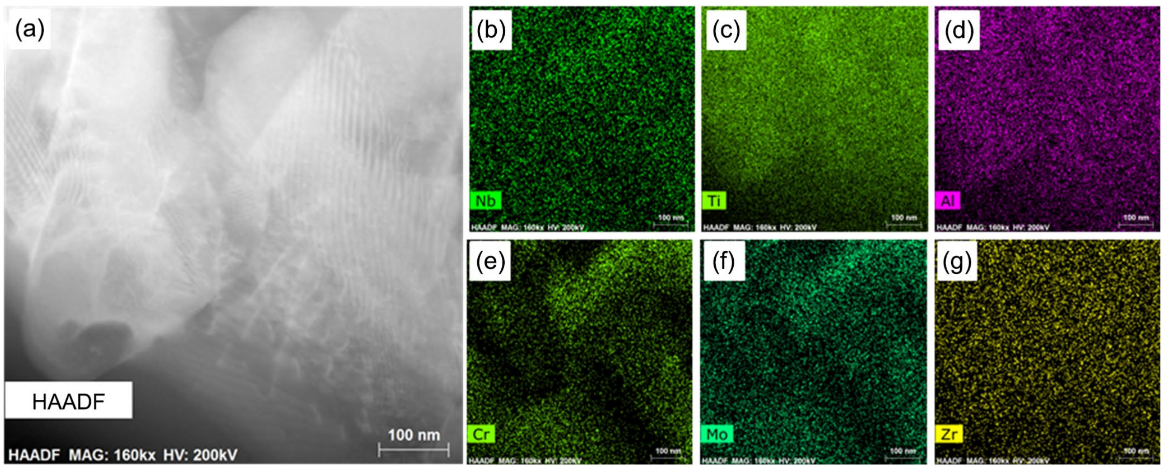

Fig. 9: Element distribution diagram near the α phase of Ti-5552-3Nb alloy (a–g)

4 Conclusions

In this study, the effect of Nb addition on superplasticity at 923 K was revealed. The specific conclusions are as follows:

(1) The as-cast microstructure of Ti-5552-*x*Nb alloy is consisted of a single β phase. With the increase of Nb content, the size of β grain in the microstructure increases gradually, and the peak of β phase shifts to the left.

(2) The addition of Nb element reduces the precipitation trend of α phase during high temperature tensile at 923 K. When the Nb content increases from 3wt.% to 9wt.%, the tensile strength increases from 158.6 MPa to 307.2 MPa, and the elongation increases from 75% to 106%.

(3) The dispersed dislocations in β phase and the stacking effect of α relative dislocations in Ti-5552-9Nb alloy are the main reasons for high superplasticity.

Acknowledgments

The authors are grateful to the financial support by the Major Science and Technology Achievement Transformation Project in Heilongjiang Province (ZC2023SH0075), the National Natural Science Foundation of China (52425401, U2441255, 52474377, and 52371015), and the Young Elite Scientists Sponsorship Program by·CAST (2021QNRC001), and the Henan Provincial Key Research and Development & Promotion Special Program (251111231400).

Conflict of interest

Prof. Rui-run Chen is an EBM of *CHINA FOUNDRY*. He was not involved in the peer-review or handling of the manuscript. The authors have no other competing interests to disclose.

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