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The interfacial β_0 phase strengthening the creep properties of powder hot isostatic pressing γ -TiAl alloy

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ABSTRACT

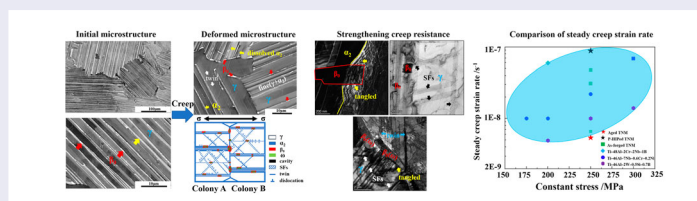
Interfacial β_0 phase strengthening on the creep properties of TNM alloy has been investigated. The initial fully lamellar microstructure with fine-dispersed interfacial β_0 phase was fabricated by powder hot isostatic pressing (P-HIP) and then carried out ageing treatment. The results indicated that the aged condition presented better microstructural stability and excellent creep resistance than the P-HIPed condition. Moreover, the ω phase precipitated within the β_0 phase by stress-inducing. The $\beta_0/\beta_0(\omega)$ phase was not only effectively restricted the dislocation motion, twins and stacking faults propagation, but also delayed the α_2 lamellae decomposition to maintain the integrity of the fully lamellar structure.

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TiAl alloy; β_0 phase; hot isostatic pressing; creep; phase transformation



IMPACT STATEMENT

The fine-dispersed β_0 precipitated at the α_2/γ lamellar interface by ageing treatment was effectively hindered the dislocation motion and significantly strengthened the creep properties. The strength mechanism was revealed.

1. Introduction

TNM alloys with a nominal composition of Ti-43.5Al-4Nb-1Mo-0.1B (at. %)[1], as the third generation TiAl alloy, are exhibiting a lower density, high specific modulus, good high-temperature mechanical strength, which is considered to be attractive for high-temperature structure materials [2–4]. Creep resistance and structure stability are both significant prerequisites for high-temperature structure TiAl components and determine the service temperature range [5]. Therefore, it is essential to improve the creep property of TiAl alloy.

The mechanical properties of TiAl alloy are shown a stronger dependence on the microstructure and chemical composition [5]. Due to the refractory elements (such as Mo, Nb.) that are generally strong β stabilizer elements in TNM alloy, the ordered β_0 phase with hard and brittle characteristics remains at room temperature and is difficult to eliminate [6,7]. Previous research has

been studied on chemical composition on the TNM alloy, the fully lamellar microstructure (FL) of TNM alloy was obtained by adding the 0.3% C and 0.2% Si elements (in at. %), which presented the best creep resistance than the near fully lamellar microstructure (NFL) of TNM alloy, i.e. (NFL + γ_g + β_0) and (NFL + β_0) [8]. Since the blocky β_0 phase with a body-centered crystal structure is located at the colony boundaries, which facilitates the diffusion processes at high temperatures due to the lower dense-packed [9,10]. As for the cast TNM alloy with 1.5 Mo element addition, the β_0 phase was irregular particle and the volume fraction of the β_0 phase could be controlled at a range of 3–18%. However, its effect on the creep properties was uncertain [11]. Especially, the β_0 phase distributed around the colony boundary and presented a continuous net shape acting like a ‘low creep strength film’ to reduce creep resistance or result in local stress concentration [12,13]. In addition, the ω phase was

easier precipitated within the β_0 phase and the $\beta_0/\beta_0(\omega)$ phase with a brittle characteristic at grain boundary caused deformation incompatibility and resulted in local stress concentration, which was treated as nucleation site of cavity or cracks [14,15]. The crack propagation always took place on the $\beta_0/\beta_0(\omega)$ phase area and, up to the creep failure. Therefore, a higher volume fraction of blocky $\beta_0/\beta_0(\omega)$ phase located at the colony boundaries might be a negative effect on the creep resistance.

The conventional peritectic solidification γ -TiAl alloy was easier to obtain fully lamellar microstructure [16]. With W, Mo, Nb β -stabilization element addition, the fine dispersed interfacial β_0 phase precipitated within α_2 lamellar after ageing treatment, and the dispersed β_0 phases provide precipitation strengthen and hindered the dislocation motion [17,18]. Moreover, the dispersed interfacial β_0 restricted the migration of ledges along with the α_2/γ interface, thereby delaying the α_2 decomposition. Hence, the creep resistance is improved. However, the β_0 was precipitated in the single-phased γ areas and in the lamellar colonies, which in turn induces the formation of W-depleted bands in the borders and the reduction of the width of α_2 lamellae, as well as some tungsten segregation in these precipitates. Therefore, the strength and creep resistance was deteriorated [19]. The impact of β_0 on creep performance is controversial and complex, which is stronger dependent on the grain size, distribution, volume fraction, etc.

In this work, the fully lamellar TNM alloy was fabricated by powder hot isostatic pressing, and the fine dispersed particle β_0 phase precipitation was obtained by ageing treatment. The influence of the interfacial β_0 phase on the microstructure stability and creep properties were discussed in detail.

2. Materials and methods

The fully lamellar TNM powder ingots (Ti-43.98Al-4.01Nb-1.04Mo-0.17B, actual composition in at.%) with a diameter of 30 mm and a height of 300 mm were consolidated by powder hot isostatic pressing (P-HIP) at 1260°C under 170 MPa for 4 h. Cylindrical samples with a diameter of 12 mm and a height of 80 mm were cut along the central axis and then ageing treated at 1050°C for 2 h with furnace cooling. For evaluating the creep behavior, cylindrical samples were machined by computerized numerical control (CNC) machining to a gauge length of 25 ± 0.1 mm and a diameter of 5 mm. Uniaxial creep experiments were tested on the NCS GNCJ-100E creep tests machine at 750°C under the constant stress of 250 MPa for 140 h. Samples for scanning electron microscope (SEM) and transmission electron microscope (TEM) were cut from the gauge area along the load

direction and then prepared by grinding and then followed by electron polishing procedure. The FEI Helios G4 CX SEM was used to observe the microstructure characteristic utilizing the backscattered electron mode (BSE). The phase volume fraction was used Image-Pro plus software to statistic 5–10 SEM pictures. Thin foils for TEM observation were prepared by twin-jet electron polishing from 60 μ m thick, 3 mm diameter disks using a solution of 500 ml electrolyte (5% perchloric, 35% butanol, 65% methanol) at -35°C , 30 V. The chemical composition were analyzed by using energy dispersive spectroscopy (EDS) system. The high angle annular dark-field (HAADF) and TEM images were tested on the FEI Talos F200X microscopy at 200 kV.

3. Results and discussion

Figure 1(a) shows a fully lamellar structure of the P-HIPed condition. The insert image depicted γ lamellar in dark gray and α_2 lamellar in light gray and a very small amount of β_0 phase in white located at the colony boundaries. The volume fraction of β_0 and γ phases are about 0.3% and 72.1%, respectively. Moreover, the bright-field (BF) TEM image in Figure 1(b) depicted that there was no precipitation observed within the α_2 lamellae. And the corresponding selected area electron diffraction (SAED) patterns only presented γ and α_2 phase diffraction spots, which obeyed orientation relationship as followed by: $\langle 10\bar{1} \rangle_\gamma // \langle 11\bar{2}0 \rangle_{\alpha_2}$ and $1\bar{1}1\gamma // 0001\alpha_2$. The microstructure characteristic of the aged condition of TNM alloy is shown in Figure 1(c,d). With the α_2 lamellae decomposition during ageing treatment, i.e. $\alpha_2 \rightarrow \gamma$ and $\alpha_2 \rightarrow \beta_0$ phase transformation. The proportion of β_0 phase and γ phase increased to 1.4% and 79.1%, respectively. Many interfacial β_0 phase precipitated within the α_2 lamellae marked with red arrows in Figure 1(c), and fewer β phases were located within γ lamellae like ‘isolated island’ because of the α_2 lamellae dissolved. Since the excess and metastable α_2 phase tends to decompose to achieve phase equilibrium after elevated temperature treatment [20,21]. The BF image in Figure 1(d) shows that the β phase marked with red arrows was nucleated at the α_2/γ interface, the corresponding SAED pattern of β phase taken along $[001]\beta_0$ direction show that there was no precipitation within the β_0 phase after aged treatment.

Figure 2(a) displays the creep curves of different conditions at 750°C under 250 MPa constant loading, i.e. the gray curves represent of aged condition and the red curves represent of P-HIPed condition. The ageing treatment was not only stabilize the lamellar structure but also form finely dispersed β_0 precipitates at the α_2/γ lamellar interface. The interfacial β_0 phase of the aged condition

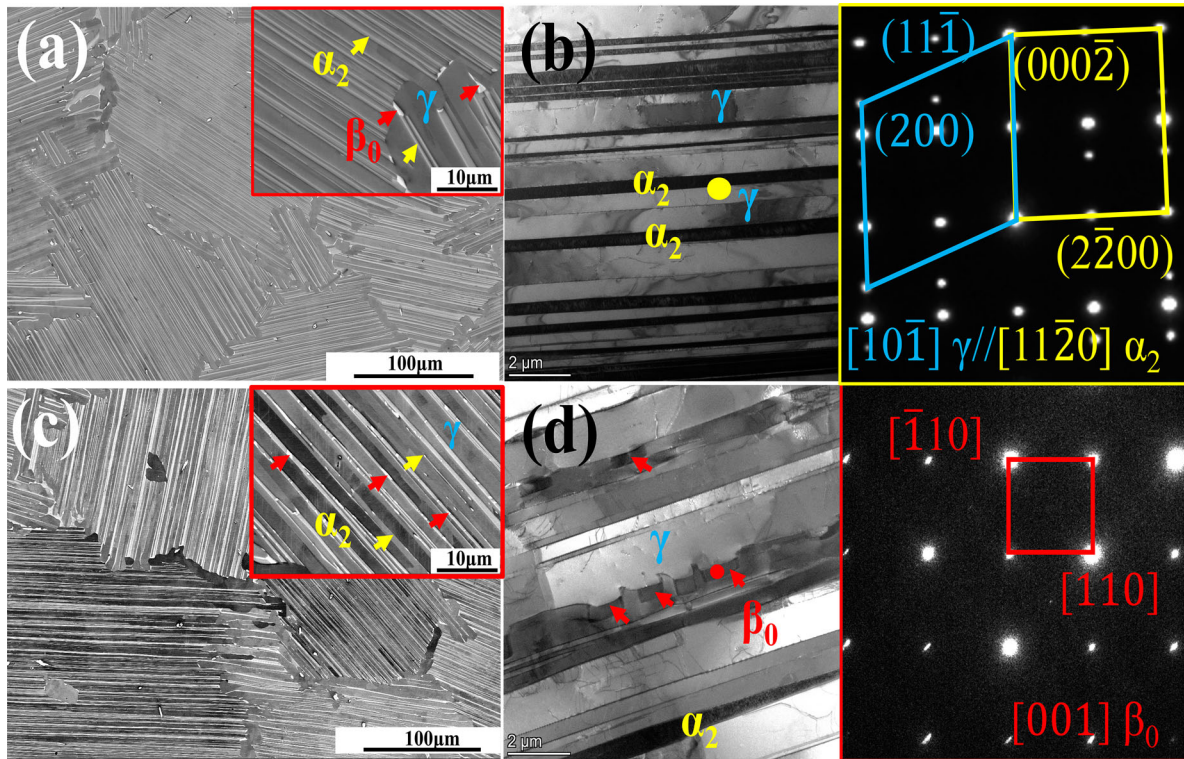


Figure 1. (a) BSE image of the P-HIPed condition, the insert image shows high magnification, (b) BF image of α_2/γ lamellae, the corresponding SAED patterns of α_2/γ lamellae, (c) BSE image of Aged condition, the insert image shows high magnification, (d) BF image of the interfacial β_0 , the corresponding SAED patterns of β_0 phase along $[001]\beta_0$ direction.

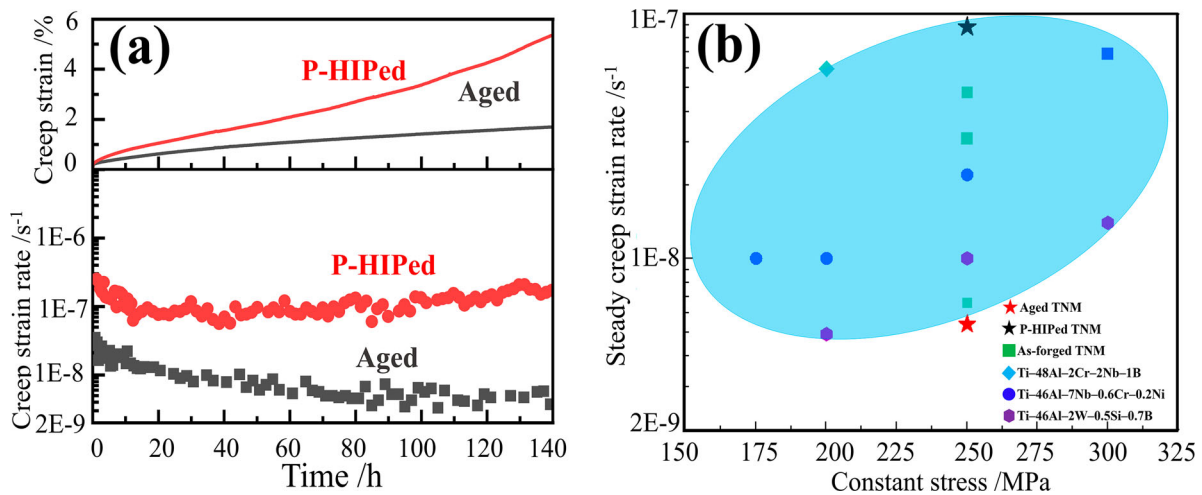


Figure 2. (a) Creep curves at 750°C under 250 MPa for 140 h, (b) comparison of steady creep strain rate.

showed a significant improvement in the creep resistance. The aged condition with 1.7% creep strain was over three times lower than the P-HIPed sample with 5.4%. Furthermore, the steady-state creep strain rate of the aged condition with $5.64 \times 10^{-9} \text{ s}^{-1}$ was seven times slower than the P-HIPed condition of $8.89 \times 10^{-8} \text{ s}^{-1}$. Compared to the other TiAl alloy at 750°C in Figure 2(b) [8,22], the steady-state creep strain rate of the aged condition presented the lowest creep rate than the fully lamellar TNM and Ti-46Al-2W-0.5Si-0.7B.

Figure 3 shows the deformed microstructure characterization after the creep test. The microstructure of the P-HIPed condition displays severe microstructure degradation in Figure 3(a). The excess α_2 lamellar decomposition caused massive phase transformation during creep, in which the volume fraction of γ phase increased from 72.1% to 84.9%. It destroyed the accumulated defects and caused microstructure instability. With the creep strain increased, the strain incompatibility at the colony boundaries caused local stress concentration

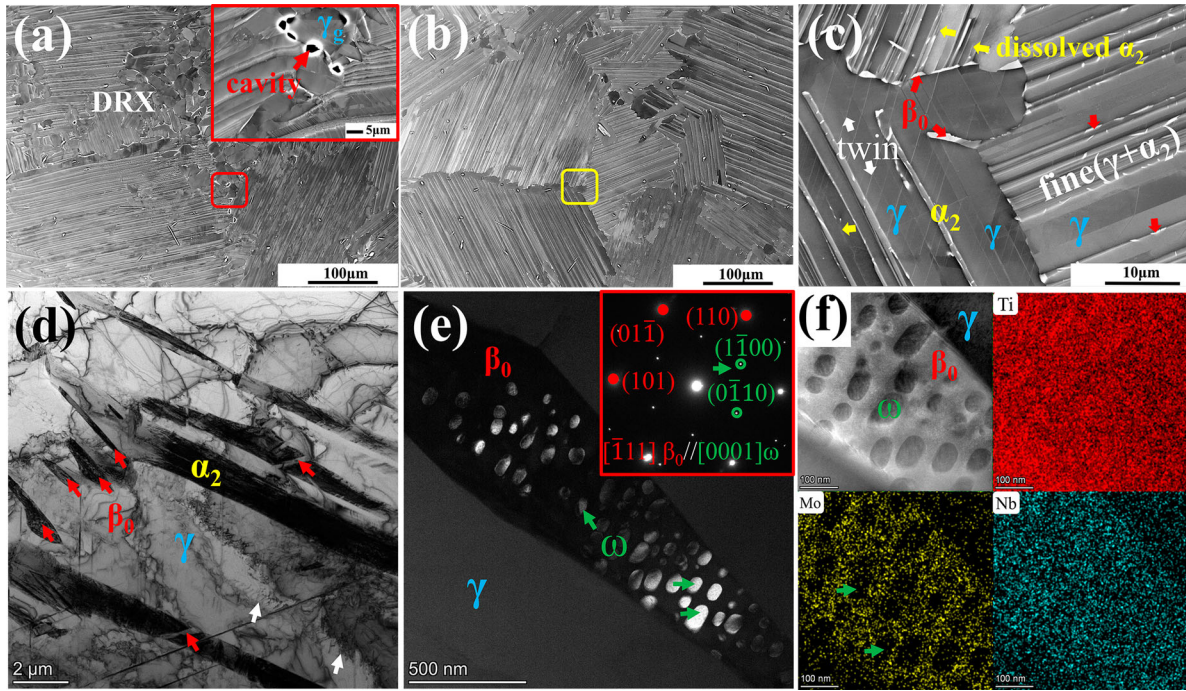


Figure 3. (a) BSE image of the P-HIPed condition, the insert image shows high magnification; (b-c) BSE image of the aged condition; (d) BF of the aged condition; (e) DF image of ω phase, the insert image shows the SAED patterns of β_0 and ω phase; (f) the corresponding HAADF and EDS image of $\beta_0(\omega)$.

[23,24]. The high magnification of the insert image in Figure 3(a) shows that the P-HIPed condition was released local stress concentration by dynamic recrystallization (DRX) of γ phase and the nucleation of the cavity. It deteriorated the microstructure stability as well as creep resistance. Figure 3(b,c) presents the deformed microstructure of the aged condition, which depicted better microstructure stability. The fully lamellar structure was not damaged, and no DRX occurred. There were some twins observed within the γ lamellae. Compared to the P-HIPed condition, the volume fraction of β_0 and γ phase increased to 2.6% and 84.1%, respectively. Due to the α_2 dissolution, some β_0 phases were located within γ lamellar, which look like 'isolated islands' in Figure 3(c,d). The dark-field (DF) TEM image in Figure 3(e) shows that there was some ellipsoid ω phase precipitated within the β_0 phase. Based on the insert image of the SAED pattern which concludes two sets of diffraction spots. The ω phase and β_0 phase obey orientation relationship as $\langle -111 \rangle \beta_0 // \langle 11-20 \rangle \omega$ and $110\beta_0 // 0001\omega$. A previous study found that the ω phase was also precipitated at β_0 phase at 750–825°C for long-term heat treatments under unloaded conditions, its growth mechanism is dominated by element diffusion [25]. Due to the introduction of the applied stress, it was attributed to accelerating the element diffusion, and the formation of the ω phase was also promoted. According to the HAADF and the corresponding EDS

Table 1. TEM-EDS spot measurements within pure β_0 and ω phase chemical composition (at.%).

Phase	Al	Ti	Nb	Mo
ω	30.8 ± 1.5	60.5 ± 1.6	8.2 ± 0.6	0.5 ± 0.2
β_0	30.7 ± 1.2	55.7 ± 1.8	6.7 ± 0.5	6.9 ± 0.5

images (Figure 4(f)) and TEM-EDS spot measurements in Table 1, ellipsoid ω phase was enriched Nb element and depleted Al element. As the growth of ω phase consumes Nb elements and rejects Mo elements, it led to a stable β_0 phase by dissolving more Mo elements. However, the γ phase has lower solubility of β -stabilizer elements, i.e. Mo and Nb elements [26]. Consequently, the $\beta_0(\omega)$ phase could not be transformed into γ phase. It was treated as the second particle to reinforce the creep resistance, and the strengthening mechanism will be discussed as follow.

Figure 4 shows the HAADF, two-beam bright-field (TBBF), and weak-beam dark-field (WBDF) TEM images of the defects interaction of the aged sample by using the diffraction vector at $g = [1-11]$. As can be seen from Figure 4(a,d), the HAADF images depicted that the particle precipitations in white were enriched by the high β -stabilizer elements (Ti-30.5Al-6.8Nb-6.9Mo, at. %), and the insert image shows that the precipitates identified by the convergent beam electron diffraction (CBED) of the β_0 phase along the $[-111]\beta_0$ direction. Consequently, these precipitates were proved to

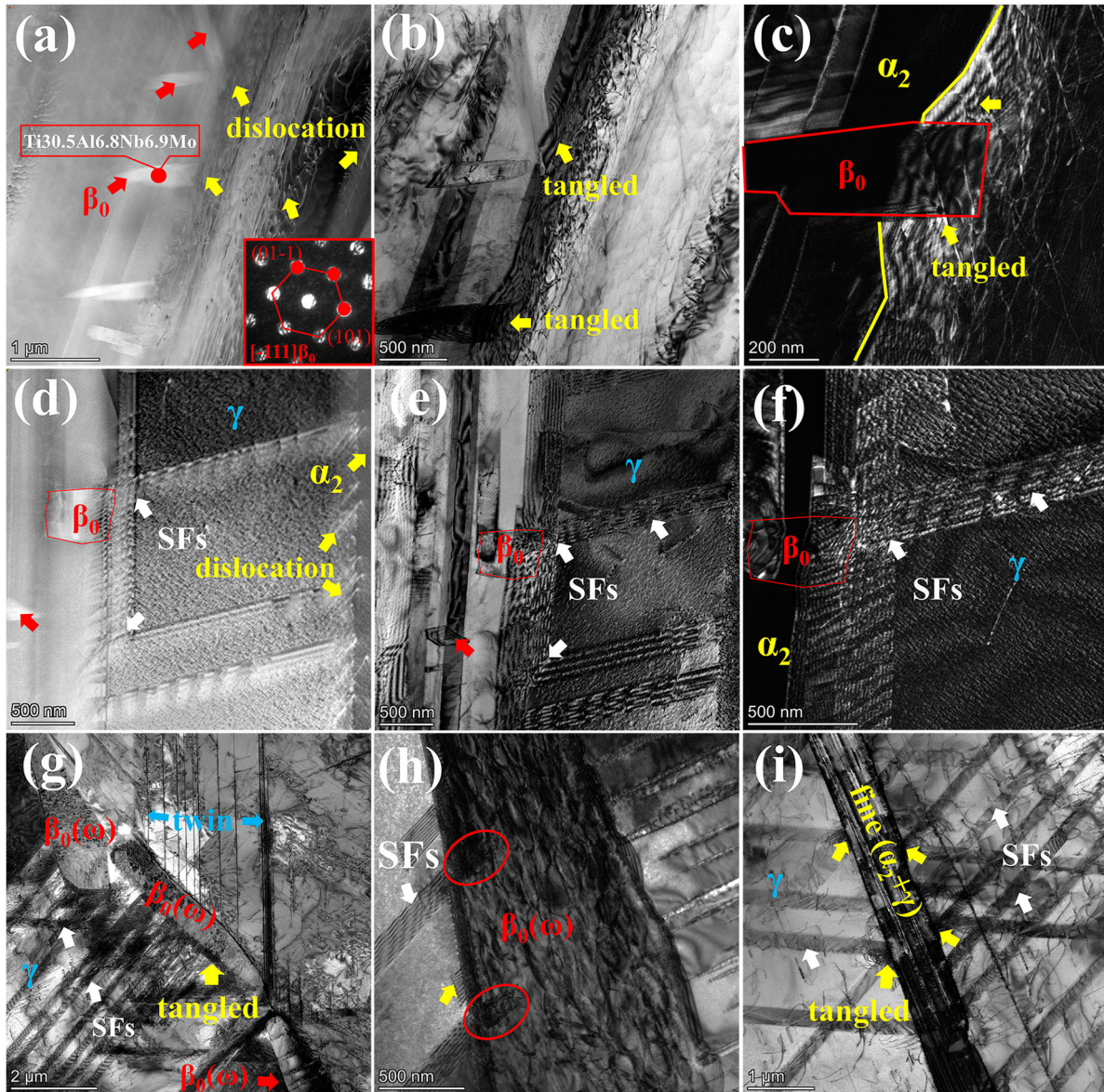


Figure 4. The defects interaction of aged sample after creep test using the diffraction vector at $g = [1-11]$. (a, d) HAADF images of β_0 phase, the insert images is the CBED of the β_0 phase; (b, e) TBBF images and (c, f) WBDF images of the interaction between interfacial β_0 phase and defects, respectively; (g, h) TBBF image of the interaction between grain boundary $\beta_0(\omega)$ phase and defects; (i) TBBF image of the interaction between lamellae and defects.

be β_0 phase. Moreover, a high-density dislocation was observed at the α_2/γ lamellae interface in Figure 4(a,b). Since the interfacial dislocation emission is related to the α_2 lamellae decomposition during creep [27], the interfacial β_0 phases could effectively pin the dislocation motion and result in dislocation tangled at the phase boundaries in Figure 4(b,c). With the α_2 lamellae decomposition, the interfacial β_0 phase with bulged morphology was attributed to hindering the dislocation and delayed the rate of α_2 lamellae dissolved. Moreover, the stacking faults (SFs) presented in two directions parallel and inclined to the γ lamellae, it was also found that the interfacial β_0 phase hindered the SFs propagation in

Figure 4(d-f). The fine dispersed interfacial β_0 phase provided precipitation strengthen for creep resistance. Furthermore, the grain boundary $\beta_0(\omega)$ phase hindered the dislocation movement, twins, and stacking faults propagation in Figure 4(g,h). Since the ω phase precipitation stabilizes the β_0 phase and improved effectively the hardness up to high temperature [28]. The grain boundary $\beta_0(\omega)$ phase was acting as a secondary phase particle strengthening. Moreover, the partial dislocations were transferred into the $\beta_0(\omega)$ as a slip dislocation marked with red circles in Figure 4(h). Compared to the P-HIPed condition, the occurrence of DRX was related to the lamellar cylinderization and edge spheroidization [29].

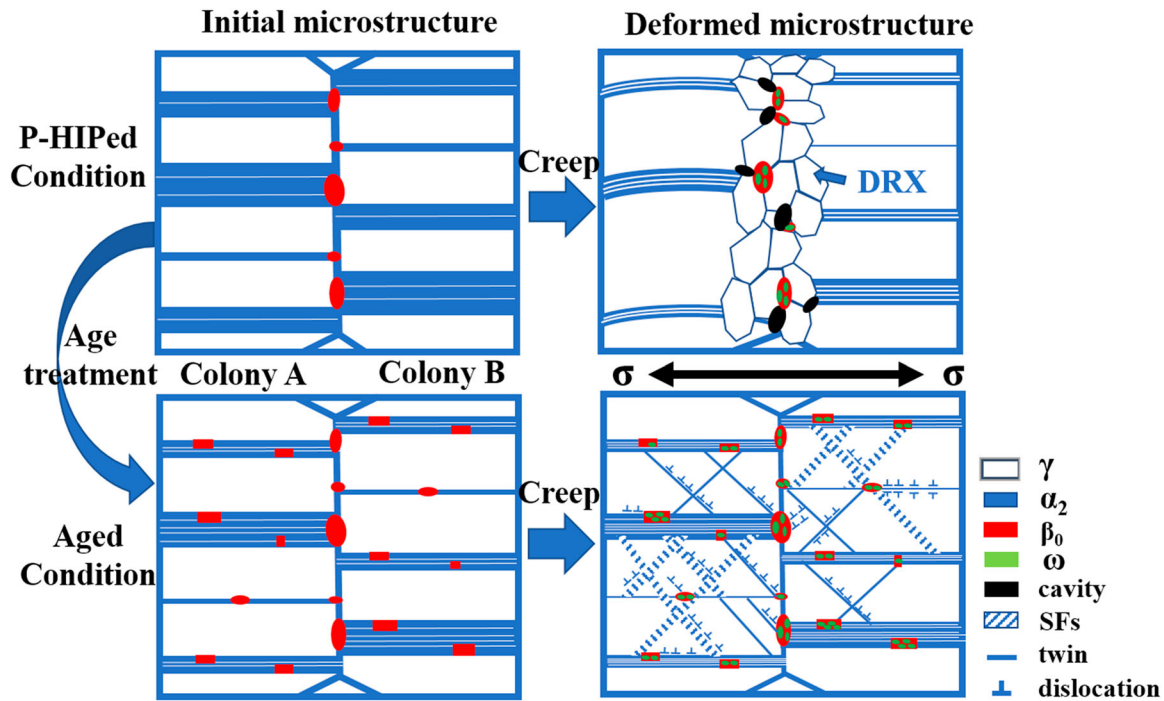


Figure 5. The schematic diagrams showing deformed microstructure.

After the ageing treatment, the volume fraction of phases were reached a relative equilibrium condition, and the precipitation of interfacial β_0 phase was formed and stabilized the α_2 lamellae. Figure 4(i) shows that the intact lamellar could retard the motion of dislocation during creep and provided obstacles for SFs and twin propagation. Conventionally, the interfaces presented obstacles for dislocation and served as dislocation sources. SFs that are highly stable conditions can block the dislocation motion [30,31]. A higher density of defects was observed within the lamellar colony, and the dislocation was pile-up at the lamellar interface and SFs conjunction area marked with yellow arrows. Moreover, the SFs were transferred into the fine ($\alpha_2 + \gamma$) lamellae as a slip dislocation marked with red circles in Figure 4(i). These were attributed to strengthening the creep resistance.

The schematic diagrams of the deformed microstructure after the creep test is shown in Figure 5. The deformed microstructure of the P-HIPed condition showed severe microstructure degradation, such as massive phase transformation, deformed lamellae, DRX of γ phase, and the nucleation of cavities. On the contrary, The aged condition was not only stabilize the lamellar structure but also formed a fine dispersed β_0 phase at the γ/α_2 lamellar interface. The interfacial β_0 phase has been shown to significantly improve the creep resistance by hindering dislocation motion, twin, and SFs propagation. It also delayed the α_2 lamellae decomposition and maintain the integrity of the lamellar structure.

4. Conclusion

A fine and uniformly dispersed interfacial β_0 phase were precipitated within α_2 lamellae after age treatment. The β_0 phase effectively blocks the dislocation motion, twin and stacking faults propagation. Moreover, the interfacial β_0 phase also delays the α_2 lamellae decomposition and stable the lamellae integrity. Finally, the aged condition exhibited better microstructure stability as well as creep properties.

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