

# Improvement of microstructure and mechanical properties of Ti42Al6Nb2.6C alloy through Er doping and in-situ formation of Er<sub>2</sub>O<sub>3</sub> particles

Hong-ze Fang<sup>1,2</sup>, Ke-xuan Li<sup>1</sup>, Xiao-kang Yang<sup>1</sup>, Hong-yuan Zhai<sup>1</sup>, Xian-fei Ding<sup>3,4</sup>, and \*Rui-run Chen<sup>1,2</sup>

1. National Key Laboratory for Precision Hot Processing of Metals, Harbin Institute of Technology, Harbin 150001, China

2. Zhengzhou Research Institute, Harbin Institute of Technology, Zhengzhou 450000, China

3. Cast Titanium Alloy R&D Center, Beijing Institute of Aeronautical Materials, Beijing 100095, China

4. Baimtec Material Co., Ltd., Beijing 100094, China

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**Abstract:** The improvement of the morphology and distribution of ceramic particles is a crucial aspect in enhancing the high-temperature strength and service temperature of TiAl-based composites. In this study, the Er element was successfully used to modify the morphology of the Ti<sub>2</sub>AlC reinforcing phase and improve room-temperature and high-temperature mechanical properties of the alloy. Results show that as the Er content increases from 0.04at.% to 0.08at.%, in-situ Er<sub>2</sub>O<sub>3</sub> particles are distributed at the surface of Ti<sub>2</sub>AlC reinforcing phase and within the lamellar colony. Both the lamellar colony and Ti<sub>2</sub>AlC reinforcing phase are significantly refined. The orientation relationship between Er<sub>2</sub>O<sub>3</sub> and Ti<sub>2</sub>AlC is {111}<sub>Er<sub>2</sub>O<sub>3</sub></sub>//{0001}<sub>Ti<sub>2</sub>AlC</sub> and {110}<sub>Er<sub>2</sub>O<sub>3</sub></sub>//{11 $\bar{2}$ 1}<sub>Ti<sub>2</sub>AlC</sub>. The higher surface energy of Ti<sub>2</sub>AlC and the favorable orientation relationship allow the Er<sub>2</sub>O<sub>3</sub> particles to nucleate on the Ti<sub>2</sub>AlC reinforcing surface. As a result, the growth of Ti<sub>2</sub>AlC is restricted by the fine Er<sub>2</sub>O<sub>3</sub> particles, preventing its uncontrolled expansion and ultimately leading to the formation of fine, short rod-like structures. The fine Er<sub>2</sub>O<sub>3</sub> and Ti<sub>2</sub>AlC act as nucleation sites for  $\beta$  grains, which is fundamental for obtaining fine lamellar colony. The Ti42Al6Nb2.6C0.08Er alloy exhibits superior room-temperature compressive properties with a compressive strength of 2,106 MPa and a compressive strain of 26.8%. The high-temperature compressive properties also demonstrate a great balance between strength and plasticity. The improvement in compressive properties is attributed to significant changes in microstructures, including the notable refinement of the lamellar colony, precipitation of Er<sub>2</sub>O<sub>3</sub> particles, load transfer effect of the refined Ti<sub>2</sub>AlC reinforcing phase, and solid solution strengthening of Er solutes.

**Keywords:** TiAl alloy; Ti<sub>2</sub>AlC reinforcing phase; refinement; Er<sub>2</sub>O<sub>3</sub> particle; mechanical properties

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## 1 Introduction

With the rapid development of aerospace and automotive industries, high-temperature structural

materials are evolving towards higher strength and lightweight design<sup>[1,2]</sup>. To meet the increasing demands of service conditions and mechanical properties, the preparation of TiAl-based composites is an effective approach to improve the high-temperature strength and service temperature of TiAl alloys<sup>[3,4]</sup>. Ti<sub>2</sub>AlC is considered one of the most promising reinforcement materials due to its similar coefficient of thermal expansion to the TiAl matrix and excellent interface bonding strength<sup>[5-8]</sup>. Compared to externally added reinforcement particles, the in-situ self-generation technique ensures a clean and strong bonding interface between the Ti<sub>2</sub>AlC reinforcing phase and the TiAl matrix<sup>[9]</sup>. However, for as-cast TiAl alloys, the size, morphology, and distribution characteristics of the

### \*Rui-run Chen

Ph.D., Professor, Doctoral Supervisor. He is the winner of the National Science Fund for Distinguished Young Scholars, and one of the leading talents of the scientific and technological innovation of the "Ten-thousand Talents Program". His research interests primarily focus on the solidification theory and casting technology of titanium aluminum alloys, niobium silicon alloys, high entropy alloys, metal based hydrogen storage materials, and solar grade silicon. To date, he has published more than 240 SCI indexed papers, and has been authorized 24 patents for inventions of China.

E-mail: ruirunchen@hit.edu.cn

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reinforcement particles are difficult to predict and control during the solidification process<sup>[10-13]</sup>. Coarse and clustered  $\text{Ti}_2\text{AlC}$  particles can lead to an imbalance between strength and plasticity<sup>[14]</sup>. Therefore, achieving uniformly fine carbide particles is crucial for improving the microstructure and mechanical properties.

Rare earth elements are important elements for improving the microstructure, and are commonly used as inoculants in steel and magnesium alloys<sup>[15, 16]</sup>. These elements are widely recognized for their ability to increase the undercooling of the alloy melt, hinder dislocation slipping, form second-phase particles, and purify the melt<sup>[17]</sup>. For TiAl alloys, the lamellar colony structure can be significantly refined by adding a small amount of Y<sup>[18, 19]</sup>. Doping Y also leads to the formation of multi-scale Y-rich phases, with these nanoscale precipitates distribute at the  $\text{B}_2/\gamma$  phase interfaces, resulting in significant dispersion strengthening<sup>[20]</sup>. Huang et al.<sup>[21]</sup> prepared high-density 3D-printed TiAl alloys by adding La to avoid cracking. Hadi et al.<sup>[22]</sup> studied the effects of adding La or Er on the oxidation behavior of TNM alloys, and found that the addition of 0.1at.% Er or La effectively reduced the oxidation rate by lowering cation diffusion. Therefore, the addition of even small amounts of rare earth elements has a significant impact on the microstructure and mechanical properties of TiAl alloys. Although the action mechanism of rare earth elements on TiAl matrices has been widely studied, the mechanism by which rare earth elements improve the morphology and the distribution of the reinforcing phase still requires further investigation. This is crucial for designing  $\text{Ti}_2\text{AlC}/\text{TiAl}$  composites with excellent overall performance.

Based on the above analysis, in this study, as-cast  $\text{Ti}_{42}\text{Al}_{16}\text{Nb}_{2.6}\text{CxEr}$  alloys were prepared. The morphology of the lamellar colonies,  $\text{Ti}_2\text{AlC}$  reinforcing phases, and  $\text{Er}_2\text{O}_3$  particles was observed using SEM and TEM. The phase constitution and their relative content were identified by XRD and EDS. The aspect ratio of  $\text{Ti}_2\text{AlC}$  particles was statistically analyzed, and the compressive properties at room temperature and high temperature of the alloy were tested. The refinement mechanism of the  $\text{Ti}_2\text{AlC}$  reinforcing phase by  $\text{Er}_2\text{O}_3$  particles was elucidated, along with the microstructural evolution mechanism induced by doping Er, and the underlying mechanisms responsible for the improved mechanical properties.

## 2 Experimental materials and methods

Sponge titanium (99.99%), aluminum particles (99.99%), aluminum niobium master alloy (75wt.% Nb), carbon powder, and high-purity erbium particles (>99.99%) were utilized as the raw materials of the  $\text{Ti}_{42}\text{Al}_{16}\text{Nb}_{2.6}\text{CxEr}$  alloys ( $x=0.04, 0.06, 0.08, 0.1, 0.4, 0.8$ , in at.%, hereinafter referred to as 0.04Er, 0.06Er, 0.08Er, 0.1Er, 0.4Er, 0.8Er alloys). The vacuum non-consumable electric arc furnace was used to melt button ingots. Each button ingot weighed 80 g and solidified in a copper crucible chilled with water. The vacuum degree was maintained below  $10^{-3}$  Pa until the completion of melting to

prevent oxidation.

Each button ingot was remelted five times in an argon environment to guarantee composition consistency. To evaluate the microstructure, the button ingot was ground, polished, and chemically etched with Kroll reagent [ $5 \text{ HF} + 10 \text{ HNO}_3 + 85 \text{ H}_2\text{O}$  (vol.%)] for 5–7 s. An Olympus GX71 optical microscope (OM) was used to analyze the microstructural characteristics, and the Quanta 200F scanning electron microscopy (SEM) with an energy dispersive spectrometer (EDS) was utilized to examine microstructure evolution and element distribution. An X-ray diffraction examination was carried out to qualitatively examine the phase constitution, and the  $K\alpha$  spectrum of copper target was chosen, with a diffraction angle of  $20^\circ$ – $90^\circ$ . The interface characteristics of in-situ  $\text{Er}_2\text{O}_3$  and  $\text{Ti}_2\text{AlC}$  particles were further observed through Talos F200X transmission electron microscopy (TEM) analysis, including bright field (BF) image, high angle annular dark field (HAADF) image, select area electron diffraction (SAED) pattern, and scanning transmission electron microscopy (STEM).

Mechanical characteristics tested in this study included room-temperature and high-temperature compressive properties. On the Instron 5569, a cylinder with a diameter of 6 mm and a height of 9 mm was deformed at a loading rate of  $0.5 \text{ mm} \cdot \text{min}^{-1}$  under a pressure head of 250 kN/50 kN. The temperatures for high-temperature compression were selected as  $700^\circ\text{C}$ ,  $800^\circ\text{C}$ ,  $900^\circ\text{C}$ , and  $1,000^\circ\text{C}$ , and the loading rate was the same as at room temperature compression test. At least three samples were measured for all the TiAl samples with different Er contents to reduce inaccuracy.

## 3 Results

### 3.1 Microstructure characteristics of $\text{Ti}_{42}\text{Al}_{16}\text{Nb}_{2.6}\text{CxEr}$ alloys

The microstructures of the  $\text{Ti}_{42}\text{Al}_{16}\text{Nb}_{2.6}\text{CxEr}$  alloys are shown in Fig. 1, with enlarged inset figures. The microstructures mainly consist of gray lamellar colony, with bright white  $\text{B}_2$  phase at the lamellar colony boundaries and small-sized white phase (the area marked by red dashed circle) inside the lamellar colony. As Er content is 0.04at.%, microcracks are observed near the  $\text{B}_2$  phase and  $\text{Ti}_2\text{AlC}$  particles. These microcracks are formed due to interfacial stress caused by the mismatch in thermal expansion coefficients among the  $\text{B}_2$  phase,  $\text{Ti}_2\text{AlC}$  particles, and the TiAl matrix [Fig. 1(a)]. The long needle-like morphology and clustered distribution of  $\text{Ti}_2\text{AlC}$  particles also contribute to stress concentration. As the Er content increases from 0.04at.% to 0.08at.%, the lamellar colony is significantly refined [Fig. 1(c)], with their average size decreasing from  $57.9 \mu\text{m}$  to  $25.6 \mu\text{m}$ . The morphology of the  $\text{Ti}_2\text{AlC}$  particles gradually transitions from long needle-like to short rod-like, and their distribution changes from a clustered aggregation pattern to a more uniform dispersion. Notably, the microcracks near the  $\text{B}_2$  phase are eliminated with the addition of Er exceeding 0.04at.%, contributed by the reduction in the segregation. Additionally, no microcracks are observed

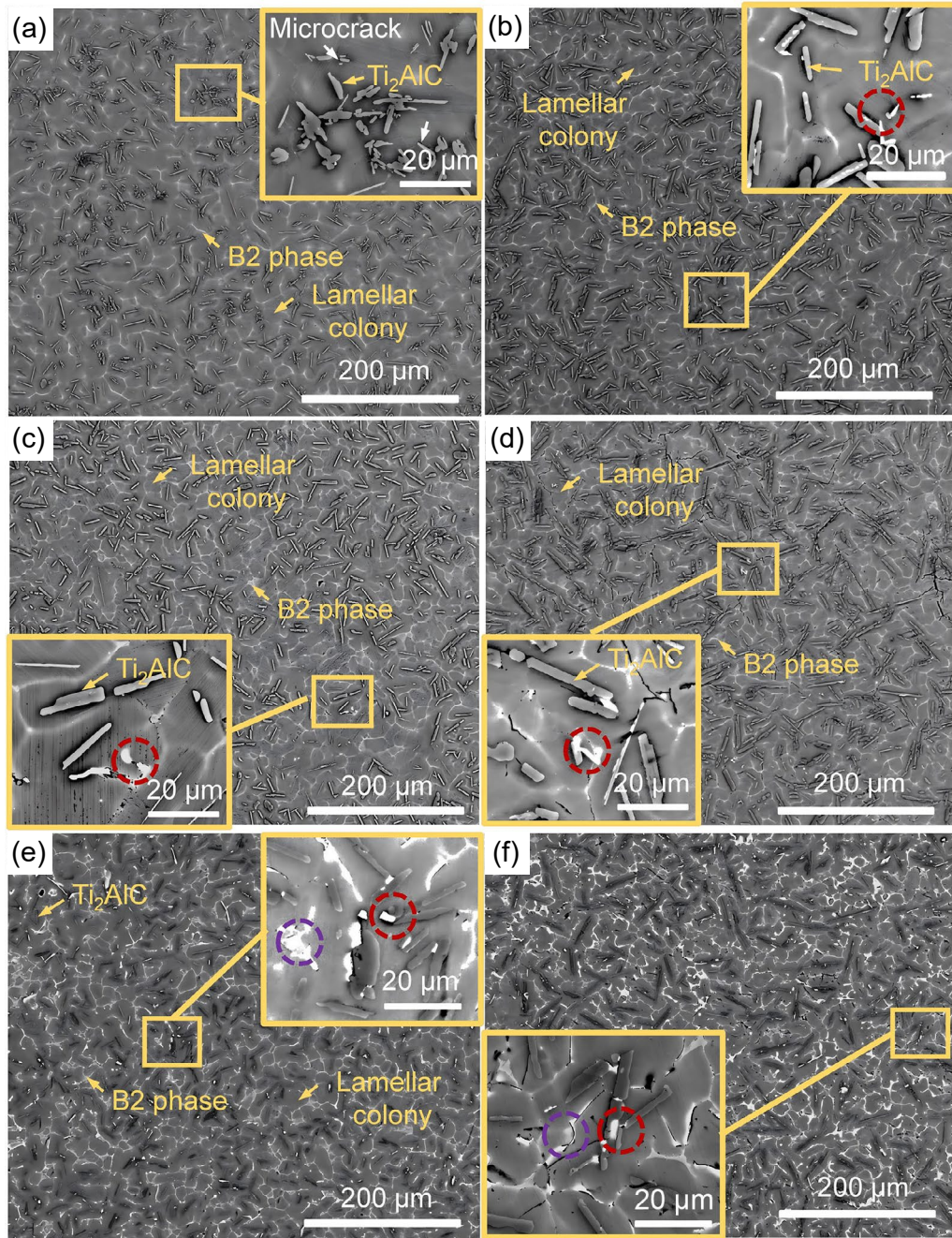


Fig. 1: SEM images of microstructures of Ti42Al6Nb2.6CxEr alloys: (a) 0.04Er; (b) 0.06Er; (c) 0.08Er; (d) 0.1Er; (e) 0.4Er; (f) 0.8Er

between the  $\text{Ti}_2\text{AlC}$  particles and the TiAl matrix, which is attributed to the transformation of  $\text{Ti}_2\text{AlC}$  particle morphology from long needle-like to short rod-like and distribution from aggregation to uniform. When the Er content further increases to 0.4at.% and 0.8at.%, another bright white phase (the area marked by purple dashed circle) appears in an irregular shape at the lamellar boundary, and its size is much larger than that of the white phase inside the lamellar colony [Figs. 1(e–f)]. The phase constitution mentioned above needs to be further confirmed by energy dispersive spectroscopy (EDS).

### 3.2 Elemental distribution and phase constitution of Ti42Al6Nb2.6CxEr alloys

Figure 2 focuses on the elemental distribution of the

0.08Er and 0.8Er alloys to confirm the element distribution characteristics of small-sized white particle and irregularly shaped white precipitates. Five positions in Figs. 2(a) and (c) were selected for analysis: inside the lamellar colony, inside the  $\text{Ti}_2\text{AlC}$  reinforcing phase, and at the lamellar boundary. At Point 1, the atomic ratio of Ti to Al is close to 1:1, and the Nb content is moderate, indicating that Point 1 corresponds to the lamellar colony. At Point 2, the atomic ratio of Ti, Al, and C is close to 2:1:1, with a small amount of Nb, suggesting that this region is the  $\text{Ti}_2\text{AlC}$  reinforcing phase. At Point 3, the composition is primarily composed of Er and O, with an atomic ratio close to 2:3, indicating the formation of  $\text{Er}_2\text{O}_3$  particles. Point 4 shows high levels of Nb and Ti, with low contents of Al, C, and Er, which is identified as the B2 phase.

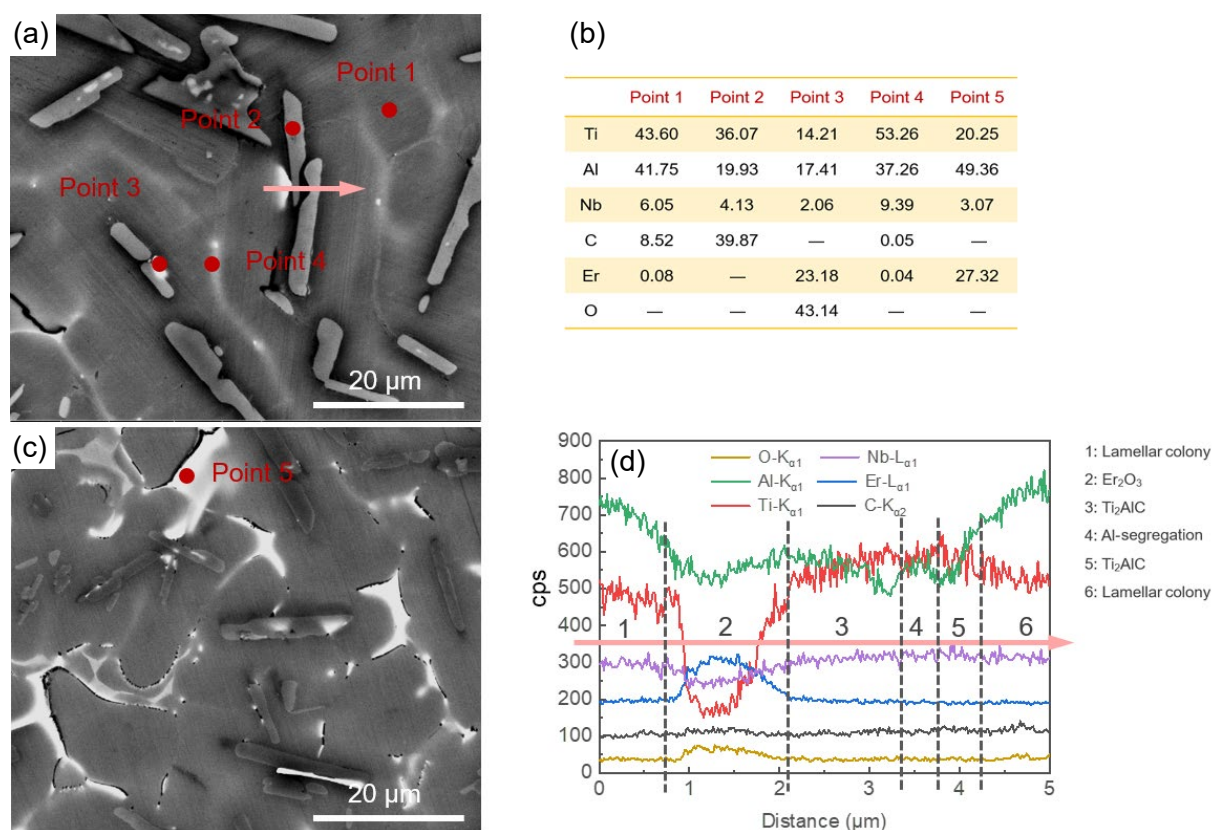


Fig. 2: Elemental distribution of 0.08Er and 0.8Er alloys: (a) locations of Points 1 to 4 in 0.08Er alloy; (b) results of point analysis; (c) location of Point 5 in 0.8Er alloy; (d) line scanning results of Fig. 2(a)

Point 5 exhibits higher levels of Er and Al, with an atomic ratio of approximately 1:3, confirming the presence of  $\text{ErAl}_3$  phase. Therefore, the 0.08Er alloy mainly consists of lamellar colony,  $\text{Ti}_2\text{AlC}$  reinforcing phase,  $\text{Er}_2\text{O}_3$  particle, and B2 phase, while, in the 0.8Er alloy the  $\text{ErAl}_3$  phase is also observed. As previously reported, the  $\text{ErAl}_3$  phase, being a hard and brittle phase, can accelerate the fracture of the alloys<sup>[23]</sup>. Figure 2(d) presents the line scanning results along the pink arrow in Fig. 2(a), which clearly shows that the scan passes sequentially through the lamellar colony,  $\text{Ti}_2\text{AlC}$  reinforcing phase, Al segregation region,  $\text{Ti}_2\text{AlC}$  reinforcing phase, and lamellar colony again.

The XRD patterns of TiAl alloys doped with different Er contents are shown in Fig. 3(a). The  $\gamma$  phase,  $\alpha_2$  phase,  $\text{Ti}_2\text{AlC}$  reinforcing phase, and a small amount of  $\text{Er}_2\text{O}_3$  phase are detected in the TiAl alloys, and their contents are determined according to the XRD pattern. As Er increases, there are fluctuations in the content of  $\alpha_2$  phase and  $\gamma$  phase, but overall, the relative content of the  $\alpha_2$  increases, while the relative content of the  $\gamma$  slightly decreases. The relative content of  $\text{Ti}_2\text{AlC}$  reinforcing phase remains almost unchanged [Fig. 3(b)]. The above result corresponds to the diffraction peak intensities of each phase. As the Er content increases, the diffraction peak intensity of the  $\alpha_2$  phase increases, while no significant changes are observed in the diffraction peaks of  $\text{Ti}_2\text{AlC}$  reinforcing phase. This indicates that an appropriate amount of Er can help increase the relative content of the  $\alpha_2$  phase, which contributes to the increase in the number of lamellar colonies<sup>[22, 23]</sup>. Higher number of lamellar colonies is beneficial

for enhancing strength. The lattice parameters of the (111) $\gamma$ , (0002) $\alpha_2$ , and (0001) $\text{Ti}_2\text{AlC}$  crystal planes were calculated, as shown in Fig. 4. The lattice distortion of the  $\gamma$  phase is more significant. The atomic radius of Er is significantly larger than that of Ti and Al. When Er atom is dissolved in  $\gamma$  phase, it will preferentially occupy specific positions in the lattice, leading to lattice distortion. As a rare earth element, Er enhances the covalency of Ti-Al bonds. Stronger covalent bonds tend to shorten the Ti-Al bond length along the  $a$ -axis direction, while along the  $c$ -axis direction, the Ti-Al bond length still expands due to the size effect of Er<sup>[24, 25]</sup>, resulting in an increase in the  $c/a$  value of the  $\gamma$  phase. It is well-known that the  $\gamma$  phase has a face-centered cubic (FCC) structure, while the  $\alpha_2$  phase and  $\text{Ti}_2\text{AlC}$  reinforcing phase have hexagonal close-packed (HCP) structure<sup>[26]</sup>. Due to higher lattice flexibility and greater number of slip systems and slip directions of the FCC structure<sup>[27]</sup>, the  $\gamma$  phase exhibits enhanced capacity to accommodate atomic size mismatches, facilitating the incorporation of Er atoms.

### 3.3 Orientation relationship and interfacial characteristics of $\text{Ti}_2\text{AlC}$ and $\text{Er}_2\text{O}_3$

Figure 5(a) presents the band contrast (BC) map after doping with 0.08at.% Er. The average size of the lamellar colony is approximately 30  $\mu\text{m}$ , with short rod-shaped  $\text{Ti}_2\text{AlC}$  reinforcing phases distributed within the lamellar colony, while long rod-shaped  $\text{Ti}_2\text{AlC}$  reinforcing phases serve as bridges connecting two adjacent lamellar colonies. Consistent with the description in Fig. 1(c), no crack initiation is observed

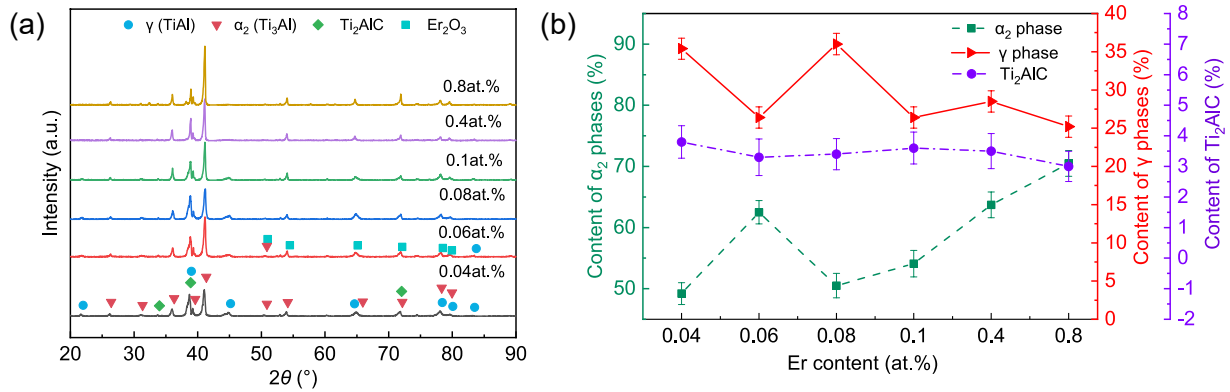


Fig. 3: XRD patterns of TiAl alloys doped with different Er contents (a) and relative content of different constituent phases within the alloys (b)

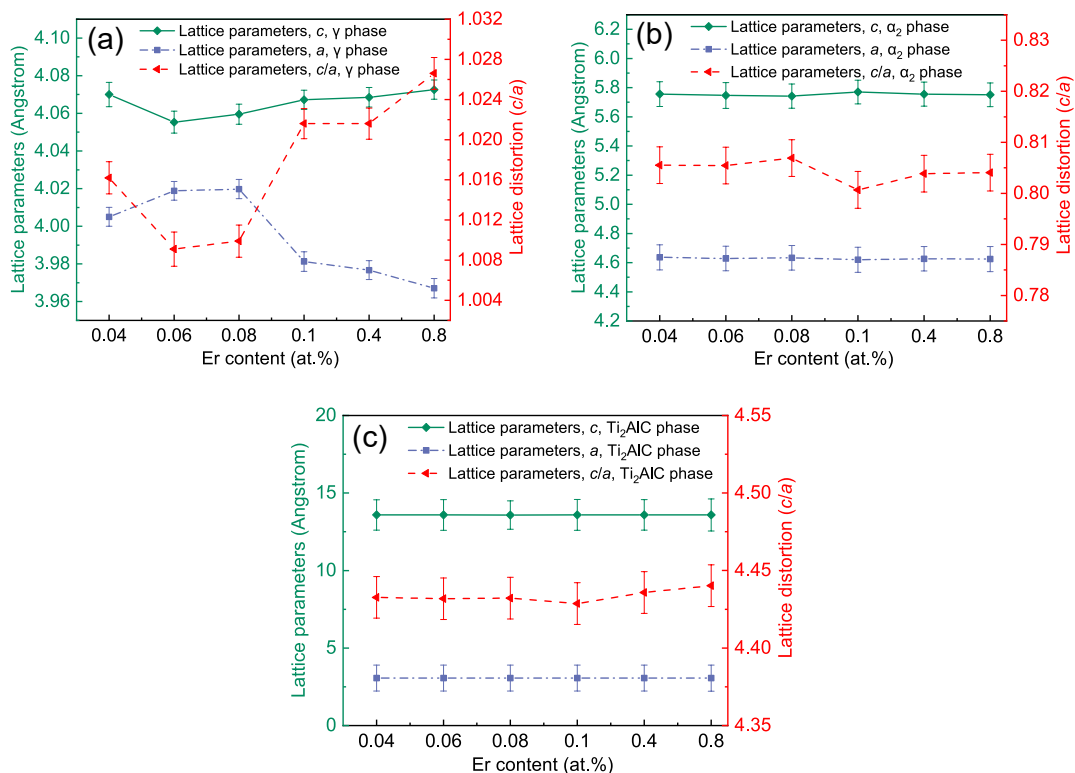


Fig. 4: Lattice parameters of different constituent phases: (a)  $\gamma$ ; (b)  $\alpha_2$ ; (c) Ti<sub>2</sub>AlC

at the interface between the lamellar colony and the Ti<sub>2</sub>AlC reinforcing phase. Figure 5(b) shows the inverse pole figure (IPF) of Fig. 5(a) overlaid with the phase diagram of Ti<sub>2</sub>AlC reinforcing phases (in green). These equiaxed lamellar colonies exhibit different orientations. Combined with the Kernel average misorientation (KAM) map, it can be observed that the interface between the lamellar colony and the Ti<sub>2</sub>AlC reinforcing phase exhibits a higher degree of plastic deformation. Compared to the  $\alpha_2/\gamma$  phase interfaces, the differences in physical properties such as the coefficient of thermal expansion and elastic modulus between the lamellar structure and Ti<sub>2</sub>AlC reinforcing phase lead to distinct plastic deformation, causing stress to concentrate at the interfaces<sup>[28, 29]</sup>. When the applied stress reaches the yield strength, these interfaces become the sources of dislocation emission and slip. The aspect ratio of Ti<sub>2</sub>AlC reinforcing phase is divided into three ranges: less

than 5, 5–10, and greater than 10, as shown in Fig. 5(d). As the Er content increases from 0.04 at.% to 0.08 at.%, from the overall trend of change, the content of Ti<sub>2</sub>AlC reinforcing phase with an aspect ratio less than 5 increases significantly from 23.8% to 83.1%. This indicates that an appropriate amount of Er can effectively refine the size of the Ti<sub>2</sub>AlC reinforcing phase. By overlaying the phase diagram of Er<sub>2</sub>O<sub>3</sub> particles on the KAM map, the white rectangular area in Fig. 5(c) was selected for interface orientation analysis. The pole figure (PF) of Er<sub>2</sub>O<sub>3</sub> particles and Ti<sub>2</sub>AlC reinforcing phase [Fig. 5(e)] shows that the two phases have the orientation relationship of  $\{111\}_{\text{Er}_2\text{O}_3} // \{0001\}_{\text{Ti}_2\text{AlC}}$ ,  $\{110\}_{\text{Er}_2\text{O}_3} // \{11\bar{2}1\}_{\text{Ti}_2\text{AlC}}$ , which helps reduce the interfacial energy between these two phases, thereby promoting the growth of Er<sub>2</sub>O<sub>3</sub> particles attached to the phase interface of Ti<sub>2</sub>AlC.

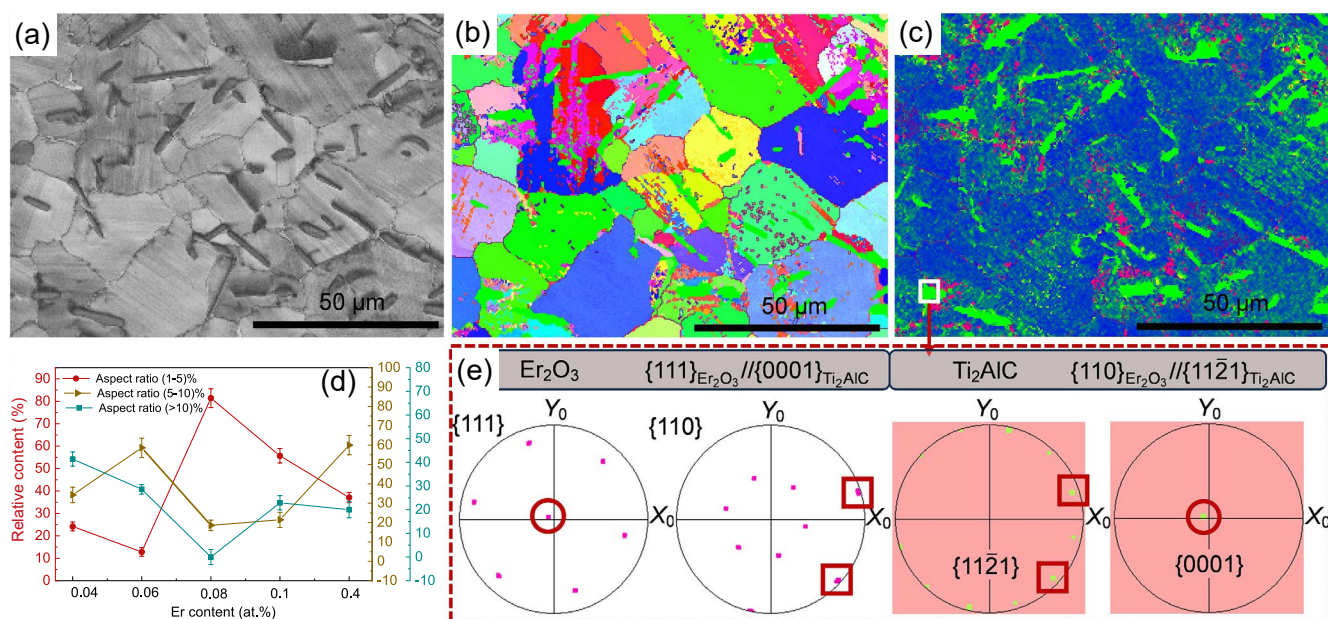


Fig. 5: EBSD analysis of 0.08Er alloy: (a) BC map; (b) IPF map; (c) KAM map with additional phase map of  $\text{Er}_2\text{O}_3$  phase; (d) aspect ratio of  $\text{Ti}_2\text{AlC}$  reinforcing phase; (e) PF map of  $\text{Er}_2\text{O}_3$  particle and  $\text{Ti}_2\text{AlC}$  reinforcing phase

To further investigate the effect of  $\text{Er}_2\text{O}_3$  particles on the  $\text{Ti}_2\text{AlC}$  reinforcing phase and lamellar colony, TEM images of the phase interfaces of 0.08Er alloy were analyzed, as shown in Fig. 6. From the HAADF images and corresponding elemental distributions in Figs. 6(a)–(g), it can be observed that  $\text{Er}_2\text{O}_3$  particles are distributed at the  $\alpha_2/\gamma$  interface, where high dislocation density and more distinct dislocation slip features are present. The  $\text{Er}_2\text{O}_3$  particles exhibit smooth contours and a size of approximately 200 nm. When distributed as nanoparticles at the lamellar interfaces, they do not induce severe stress concentrations; instead, they allow dislocations to bend and bypass them during slip. As a result, stress is uniformly distributed across the phase interfaces and within the phases as dislocations move, enabling uniform plastic deformation in the TiAl alloy prior to fracture. Additionally,  $\text{Er}_2\text{O}_3$  particles are also aligned along the phase boundaries of the  $\text{Ti}_2\text{AlC}$ , as shown in Figs. 6(h)–(j). These fine  $\text{Er}_2\text{O}_3$  particles adhere to both the long and short axes of the  $\text{Ti}_2\text{AlC}$  reinforcing phase, thereby closing and modifying the phase boundaries, which acts as barriers to inhibit the growth of the  $\text{Ti}_2\text{AlC}$  reinforcing phase. No dislocation entanglement or accumulation is observed at the interfaces between the two phases, indicating that the presence of the nanoscale  $\text{Er}_2\text{O}_3$  particles has no effect on the stress distribution at the phase interfaces and further helps to prevent the formation of severe interphase stresses. The SAED patterns in Figs. 6(k)–(l) correspond to the blue boxed regions in Fig. 6(j), identifying the  $\text{Er}_2\text{O}_3$  particles and  $\text{Ti}_2\text{AlC}$  reinforcing phase as body-centered cubic (BCC) and hexagonal close packed (HCP) structures. In summary,  $\text{Er}_2\text{O}_3$  particles not only help disperse the stress at the  $\alpha_2/\gamma$  lamellar interfaces but also adhere to the surface of the  $\text{Ti}_2\text{AlC}$  phase, thereby modifying the morphology of  $\text{Ti}_2\text{AlC}$ . Therefore, the formation of  $\text{Er}_2\text{O}_3$  particles contributes to improving the microstructures of the TiAl alloy.

### 3.4 Compressive properties at different temperatures of Ti42Al6Nb2.6CxEr alloys

The room-temperature compressive properties of the TiAl alloys with varying Er contents and the high-temperature compressive properties of the TiAl alloy with 0.08at.% Er are shown in Fig. 7. As the Er content increases from 0.04at.% to 0.08at.%, the room-temperature compressive strength increases from 1,528 to 2,106 MPa. However, when the Er content continues to increase to 0.8at.%, the compressive strength decreases to 1,580 MPa, while the compressive strain shows an insignificant decrease. The decrease in compressive strength is associated with the continuous precipitation of  $\text{ErAl}_3$  phases at the lamellar colony boundaries. Therefore, the optimal compressive properties are achieved when the Er content is 0.08at.%. Subsequently, the high-temperature compressive properties of the Ti42Al6Nb2.6C0.08Er alloy are evaluated, as shown in Fig. 7(b). As the temperature increases to 700 °C, the alloy exhibits a compressive strength of 1,684 MPa and a compressive strain of 49.7%. As the temperature rises to 800 °C, the compressive strength decreases to 1,587 MPa, while the compressive strain significantly increases to 66.9%. As the temperature further increases to 900 °C, the compressive strength continues to decrease and the compressive strain continues to increase. At 1,000 °C, the compressive strength drops to 714 MPa, while the compressive strain reaches 64.7%. Therefore, the alloy exhibits a great balance between strength and plasticity at 800 °C. Excessive compression temperature not only accelerates dislocation motion but also accelerates oxidation of the alloy. As a result, the compressive properties of the alloy cannot be further improved during the compression deformation process.

Based on Fig. 8, the fracture mode of the Ti42Al6Nb2.6CxEr alloys at room temperature can be further clarified. Figure 8(a)

shows the fracture morphology at 0.04at.% Er. It can be observed that with the addition of a small amount of Er, the fracture mode of the alloy is predominantly intergranular cracking, with cracks propagating along the boundaries of lamellar colonies in different orientations<sup>[30, 31]</sup>. When Er content increases to 0.08at.%, a mixed fracture mode of interlamellar and translamellar cracking is observed [Fig. 8(b)]. Additionally, from the magnified figure [Fig. 8(c)], the pull-out and crack of short rod-shaped  $\text{Ti}_2\text{AlC}$  reinforcing phases are evident, indicating that under compressive stress, the  $\text{Ti}_2\text{AlC}$  reinforcing phase, as a hard phase, effectively resists plastic deformation compared to the TiAl matrix. Moreover, the  $\text{Ti}_2\text{AlC}$  reinforcing phase modified by  $\text{Er}_2\text{O}_3$  increases

the deflection path of cracks, preventing crack propagation into the lamellar colonies [Fig. 8(d)]. When the Er content further increases to 0.8at.%, the alloy exhibits an intergranular fracture mode again [Figs. 8(e)–(f)], which is caused by the precipitation of the brittle  $\text{ErAl}_3$  phase along the boundaries of the lamellar colonies. This leads to the formation of a network-like microstructure. This distribution pattern increases the extent of cleavage of the TiAl matrix. When microcracks form at the boundaries of the lamellar colonies, the large difference in deformability between the  $\text{ErAl}_3$  phase and the lamellar colony leads to rapid crack propagation along the  $\text{ErAl}_3$  phase boundaries, ultimately accelerating the fracture of the TiAl alloy.

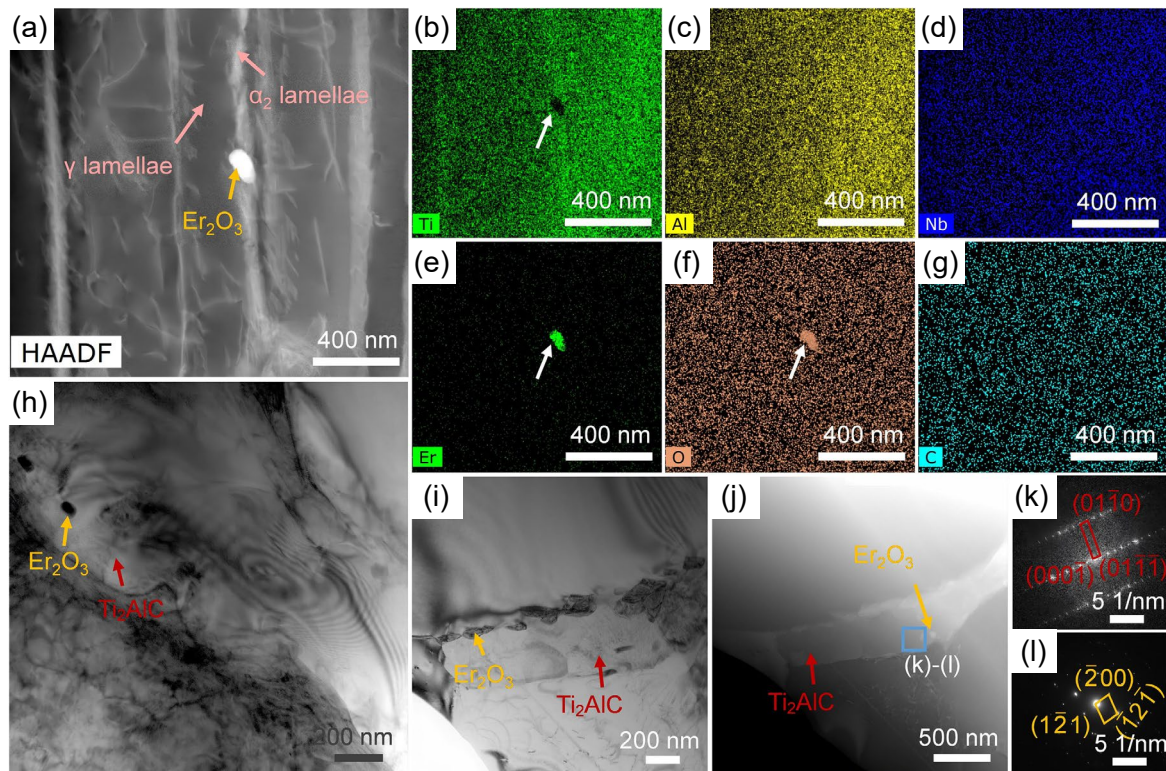


Fig. 6: TEM analysis of the interface of  $\text{Er}_2\text{O}_3$  particles and  $\text{Ti}_2\text{AlC}$  reinforcing phase in 0.08Er alloy: (a) HAADF of  $\text{Er}_2\text{O}_3$  particle,  $\gamma$  lamellae, and  $\alpha_2$  lamellae; (b)–(g) corresponding element mapping distribution of Fig. 6(a); (h)–(j) BF of  $\text{Ti}_2\text{AlC}$  reinforcing phase and  $\text{Er}_2\text{O}_3$  particle; (i) HAADF of  $\text{Ti}_2\text{AlC}$  reinforcing phase and  $\text{Er}_2\text{O}_3$  particle; (k) SAED pattern of  $\text{Ti}_2\text{AlC}$  reinforcing phase; (l) SAED pattern of  $\text{Er}_2\text{O}_3$  particle

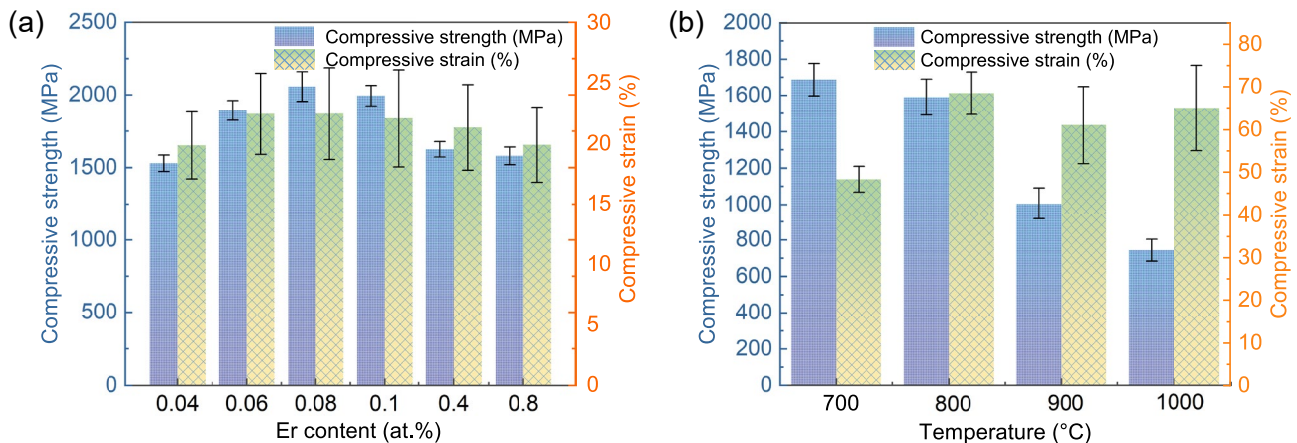


Fig. 7: Room-temperature compressive properties of alloys with different Er contents (a) and high-temperature compressive properties of 0.08Er alloy (b)

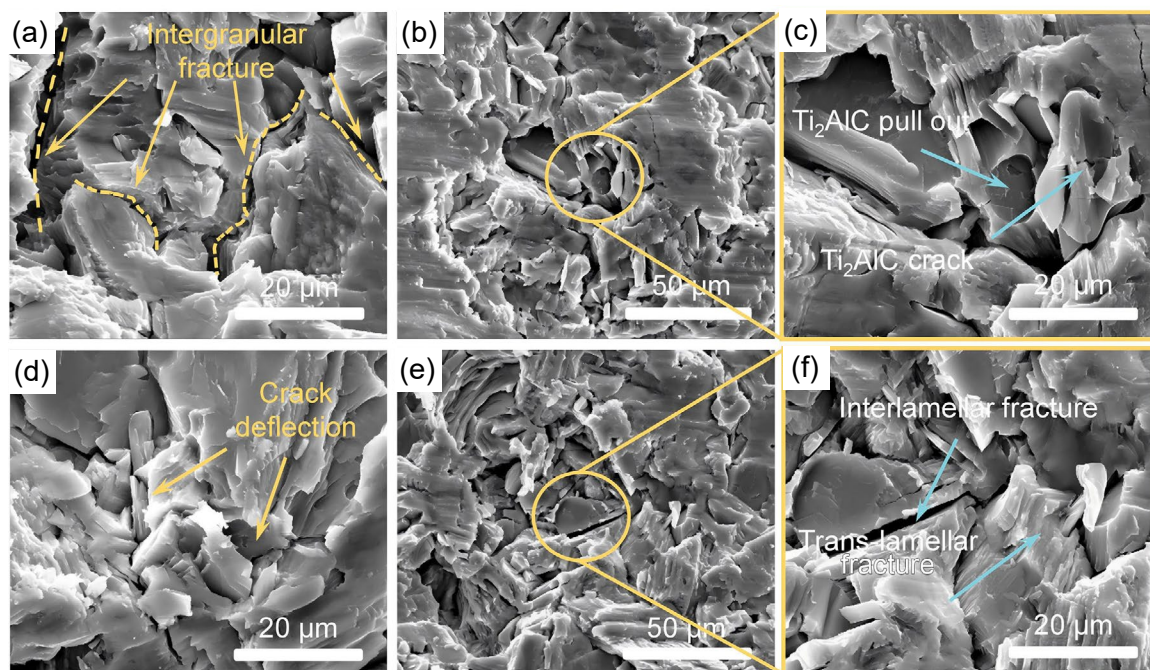


Fig. 8: Compressive fracture morphologies of Ti42Al6Nb2.6CxEr alloys at room temperature: (a) 0.04Er; (b) 0.08Er; (c) magnified figure of (b); (d) 0.1Er; (e) 0.8Er; (f) magnified figure of (e)

## 4 Discussion

### 4.1 Effect of Er content on microstructures of Ti42Al6Nb2.6CxEr alloys

As addition of Er element ranges from 0.04at.% to 0.8at.%, and the oxygen content is strictly controlled during the alloy preparation process, the content of  $\text{Er}_2\text{O}_3$  particles is low. According to previous study<sup>[32]</sup>, Er element has little effect on the nucleation of  $\text{Ti}_2\text{AlC}$  reinforcing phase. Based on the Al content in the alloy, the solidification path of the TiAl alloy follows the  $\beta$ -solidification mode. During the phase transition from liquid to solid,  $\text{Ti}_2\text{AlC}$  reinforcing phase preferentially nucleates from the liquid phase. As the process consumes Al elements, an Al-enriched dark gray region forms around the  $\text{Ti}_2\text{AlC}$  reinforcing phase. From the analysis of Figs. 5 and 6, it can be observed that nanoscale  $\text{Er}_2\text{O}_3$  particles are adsorbed onto the surface of  $\text{Ti}_2\text{AlC}$  reinforcing phase, where they subsequently grow. The  $\text{Ti}_2\text{AlC}$  reinforcing phases adsorbed by  $\text{Er}_2\text{O}_3$  particles are unable to grow further. Therefore, after the addition of Er element, the aspect ratio of  $\text{Ti}_2\text{AlC}$  reinforcing phases gradually decreases, and their morphology changes from elongated rod-like shapes to short rod-like shapes. Subsequently,  $\beta$  grains nucleate from the liquid phase at the sites of nanoscale  $\text{Er}_2\text{O}_3$  particles and refined  $\text{Ti}_2\text{AlC}$  reinforcing phases. The nucleation rate of  $\beta$  grains significantly increases, and the size of the  $\beta$  grains is greatly refined. When the temperature decreases to the  $\beta \rightarrow \alpha$  transformation region,  $\alpha$  grains are also refined, which lays the foundation for the formation of small-sized lamellar colonies<sup>[33]</sup>.

As the temperature further decreases, the  $\alpha \rightarrow \alpha_2 + \gamma$  phase transformation involves the diffusion of Al elements via a stepwise mechanism, leading to the enrichment of Al elements

at the boundaries of the lamellar colonies. As the Er content increases to 0.08at.% or above, the content of  $\text{Er}_2\text{O}_3$  particles no longer increases, and the remaining Er elements will continue to react with the Al elements at the lamellar colony boundaries to form the  $\text{ErAl}_3$  phase. As a result, the relative content of  $\gamma$  lamellae decreases, and the relative content of  $\alpha_2$  lamellae increases due to the consumption of Al. In addition, the doping of rare earth elements has been reported to delay the  $\alpha \rightarrow \alpha_2 + \gamma$  phase transformation, thus preserving a higher fraction of ordered  $\alpha_2$  phase<sup>[34]</sup>. This is because the atomic radius of Er is much larger than that of Ti and Al, and as a solute atom, it occupies lattice gaps or substitution sites, causing local lattice distortion. Lattice distortion forms an elastic strain field that hinders the long-range diffusion of Al atoms, thereby delaying the compositional segregation required for the  $\alpha \rightarrow \alpha_2 + \gamma$  phase transition. Er element reacts with oxygen to generate  $\text{Er}_2\text{O}_3$ , pinning the  $\alpha/\alpha_2$  interface and inhibiting the migration of the  $\gamma$  phase boundary. During the solidification process, the pinning effect and diffusion inhibition work together to preserve a more ordered structure of the  $\alpha_2$  phase under non-equilibrium condition. These factors promote the presence of more  $\alpha_2$  lamellae rather than  $\gamma$  lamellae within the lamellar colonies. The sharp-edged, brittle  $\text{ErAl}_3$  phase, under the interface stress, weakens the bonding strength at the lamellar colony boundaries and ultimately becomes a stress concentration region, aggravating the accumulation and propagation of microcracks.

### 4.2 Refining mechanism for $\text{Ti}_2\text{AlC}$ reinforcing phase by Er dropping

As the Er content increases to 0.08at.%, the aspect ratio of  $\text{Ti}_2\text{AlC}$  reinforcing phase gradually decreases. The content

of  $\text{Ti}_2\text{AlC}$  reinforcing phase with an aspect ratio less than 5 increases from 23.8% to 83.1%. According to the Er-O binary phase diagram<sup>[35]</sup> and the strictly controlled oxygen content during the melting process, the temperature at which  $\text{Er}_2\text{O}_3$  particles precipitate from the liquid phase is lower than 1,600 °C, which is below the temperature at which  $\text{Ti}_2\text{AlC}$  reinforcing phase precipitates. Therefore,  $\text{Er}_2\text{O}_3$  particles have no effect on the nucleation process of  $\text{Ti}_2\text{AlC}$  reinforcing phase. Compared to the liquid phase, the surface of the stable precipitated  $\text{Ti}_2\text{AlC}$  reinforcing phase has a higher surface energy, making it an ideal site for the nucleation of  $\text{Er}_2\text{O}_3$  particles. Thus,  $\text{Er}_2\text{O}_3$  particles tend to move to the surface of  $\text{Ti}_2\text{AlC}$ . These  $\text{Er}_2\text{O}_3$  particles preferentially adhere to the growth active sites of  $\text{Ti}_2\text{AlC}$  through chemical adsorption, forming a physical barrier. This covering effect directly hinders the diffusion and adhesion of atoms Ti, Al, and C to the crystal surface of  $\text{Ti}_2\text{AlC}$ . Due to limited diffusion pathways,  $\text{Ti}_2\text{AlC}$  grains cannot grow sufficiently, thus forming fine and more uniform structure. Figure 5(e) shows that there is an interface orientation relationship between  $\text{Er}_2\text{O}_3$  particles and  $\text{Ti}_2\text{AlC}$  reinforcing phase, with  $\{111\}_{\text{Er}_2\text{O}_3} // \{0001\}_{\text{Ti}_2\text{AlC}}$  and  $\{110\}_{\text{Er}_2\text{O}_3} // \{11\bar{2}1\}_{\text{Ti}_2\text{AlC}}$ , which also facilitates the development of  $\text{Er}_2\text{O}_3$  particles on the surface of  $\text{Ti}_2\text{AlC}$  reinforcing phase. In addition,  $\text{Er}_2\text{O}_3$  particles, as highly thermal-stable phase, alter the local heat conduction, leading to uneven temperature fields at the growth front of  $\text{Ti}_2\text{AlC}$  reinforcing phase and inhibiting its coarsening. These three factors lead to the constraint of  $\text{Ti}_2\text{AlC}$  reinforcing phase by fine  $\text{Er}_2\text{O}_3$  particles, preventing its free growth, ultimately resulting in the formation of finer short rods in the liquid region. As the temperature decreases to the solid-state phase transition stage, the refined  $\text{Ti}_2\text{AlC}$  reinforcing phase and fine  $\text{Er}_2\text{O}_3$  particles act as heterogeneous nucleation sites for the primary  $\beta$  phase. This increases the number of nucleation sites for  $\beta$  grains, thereby increasing the number of  $\beta$  grains and reducing the size of  $\beta$  grains. Therefore, the in-situ  $\text{Er}_2\text{O}_3$  particles have a different refinement mechanism for  $\text{Ti}_2\text{AlC}$  particles compared to lamellar colonies. Essentially, in-situ  $\text{Er}_2\text{O}_3$  particles inhibit the growth of  $\text{Ti}_2\text{AlC}$  reinforcing phase while enhancing the nucleation rate of  $\beta$  grains.

### 4.3 Strengthening mechanisms of TiAl alloys doped with Er

The improvement in compressive performance is primarily attributed to the significant changes in the microstructure: (1) significant refinement of lamellar colonies; (2) precipitation of nano-sized  $\text{Er}_2\text{O}_3$  particles; (3) load transfer effect of the refined  $\text{Ti}_2\text{AlC}$  reinforcing phase; and (4) solid-solution strengthening by Er atoms.

As heterogeneous nucleating particles, Er,  $\text{Er}_2\text{O}_3$  particles, and the  $\text{Ti}_2\text{AlC}$  reinforcing phase increase the undercooling of the TiAl melt, which promotes the nucleation rate of  $\beta$  grains. When atomic diffusion is suppressed with decreasing temperature, grain growth becomes constrained, and further grain coarsening ceases. The movement of grain boundaries requires atomic diffusion. When diffusion slows down, grain

boundary migration becomes difficult, and grains are unable to grow by consuming smaller neighboring grains. Further considering the driving force and resistance to grain growth, it can be noted that the driving force for grain growth is the reduction of grain boundary energy. In other words, the system tends to lower its total energy by reducing the total grain boundary area. The migration of grain boundaries relies on atomic diffusion. When the diffusion rate decreases, the speed of grain boundary migration decreases, leading to a slowdown or even termination of grain growth process. In this way, during the solidification process of the TiAl alloy, the grains do not have enough time or driving force to continue growing, thus maintaining a smaller size. This results in a significant refinement of lamellar colonies at room temperature, increasing the boundary density of these lamellar colonies. Under the elastic interactions of dislocations and the short-range stress fields at the grain boundaries, dislocations are prevented from entering or passing through the boundaries and instead accumulate at the boundaries<sup>[35]</sup>. The increased grain boundary density allows for more dislocations to be accommodated, which helps to shorten the dislocation pile-up length without reducing compressive strain<sup>[37, 38]</sup>. Prior to fracture, the stress required for dislocation motion must exceed the stress at the grain boundaries to sustain plastic deformation, thus significantly increasing the compressive strength of the TiAl alloys. Additionally, a greater number of lamellar colony boundaries reduce the stress concentration within individual lamellae, facilitating more uniform deformation of the lamellar colonies, thereby promoting stress redistribution. The refinement of lamellar colonies contributes to the improvement in both compressive strength and compressive strain.

It is known that Er dissolves as solute atoms in the TiAl matrix. Due to the difference in atomic radii among Er, Ti, and Al, lattice distortion occurs, which increases both the chaos degree and the internal energy of the system<sup>[39, 40]</sup>. The interaction between Er atoms and the stress field generated by the moving dislocations increases the resistance to dislocation motion, thereby raising the stress required for dislocation sliding. The excellent compressive strength of the TiAl alloy is closely related to the precipitation of  $\text{Er}_2\text{O}_3$  particles.  $\text{Er}_2\text{O}_3$  particles, which possess high elastic modulus and hardness, effectively hinder dislocation motion. Although some dislocations may escape from the pinning effect of  $\text{Er}_2\text{O}_3$  particles, the stress generated by bending deformation weakens dislocation propagation<sup>[41]</sup>. Therefore, continuous dislocation slip requires higher stress, and the macroscopic plastic deformation of alloy demands higher external force, which in turn enhances the compressive strength.

The increase in compressive strength is also associated with the refined  $\text{Ti}_2\text{AlC}$  reinforcing phase. As one of the MAX-type ceramics,  $\text{Ti}_2\text{AlC}$  combines the excellent properties of both metals and ceramics. When  $\text{Ti}_2\text{AlC}$  is incorporated into the TiAl alloy, its aspect ratio directly influences the size of the  $\beta$  phase.  $\text{Ti}_2\text{AlC}$  with a small aspect ratio promotes the formation of fine  $\beta$  phase. Since  $\text{Ti}_2\text{AlC}$  has higher hardness than the

lamellar colony, it bears most of the applied stress<sup>[42-45]</sup>. After adding Er, the distribution of  $\text{Ti}_2\text{AlC}$  reinforcing phases becomes more uniform, reducing the degree of stress concentration within the microstructure. As shown in Fig. 8,  $\text{Ti}_2\text{AlC}$  reinforcing phase exhibits bridging and pull-out effects. When cracks encounter  $\text{Ti}_2\text{AlC}$ , these reinforcements act like bridges, connecting the two sides of the crack, thereby preventing further crack propagation. When the interface bonding strength between  $\text{Ti}_2\text{AlC}$  reinforcing phase and lamellar colony is high enough, the refined  $\text{Ti}_2\text{AlC}$  reinforcing phase can effectively bridge cracks and improve the crack resistance of the alloy<sup>[46]</sup>. Furthermore, when cracks propagate and bypass  $\text{Ti}_2\text{AlC}$  reinforcing phases, these reinforcements are pulled out, consuming energy and slowing down the crack propagation rate. In addition, when cracks encounter  $\text{Ti}_2\text{AlC}$  reinforcements, multiple deflections occur, which further enhances the compressive performance.

Figure 9 shows the microstructure morphology of  $\text{Ti42Al6Nb2.6C0.08Er}$  alloy after compression deformation at 800 °C. As shown in Fig. 9(a), high-density nanotwins are generated in the  $\gamma$  lamellae, and there are abundant dislocations distributed between these nano-twins. Chen et al.<sup>[47]</sup> reported that after tensile deformation, three types of dislocations are formed in polysynthetic twinned  $\text{TiAl}$  single crystals, among which the super dislocation can be decomposed into two dislocations, accompanied by the formation of twins. Twinning is initiated through dislocation decomposition, at the same time, act to hinder dislocation movement. This mechanism mitigates severe work hardening and helps preserve high-temperature plasticity. In addition, it can be observed from Fig. 9(b) that the  $\text{Er}_2\text{O}_3$  particles distributed in the  $\text{Ti}_2\text{AlC}$  reinforcing phase and  $\text{TiAl}$  matrix act as obstacles to dislocation slip, helping to maintain the high-temperature strength. At the same time, the  $\text{Ti}_2\text{AlC}$  reinforcing phase undergoes morphological changes, as evidenced by the following features: (1) its short axis is about 200 nm, representing a significant reduction in size compared to the

as-cast state; (2) its long axis exhibits a consistent orientation; (3) its morphology of  $\text{Ti}_2\text{AlC}$  is no longer regular rod-like, but instead displays a tortuous boundary. These characteristics indicate that during the high-temperature compression process, the  $\text{Ti}_2\text{AlC}$  reinforcing phase adapts to plastic deformation through self-fragmentation, refinement, and consistent orientation. Dislocations are emitted at the interface between  $\text{Ti}_2\text{AlC}$  reinforcing phase and  $\gamma$  phase. Most dislocations reduce interfacial stress concentration by sliding towards the  $\gamma$  phase, while a few dislocations can also slip into the interior of the  $\text{Ti}_2\text{AlC}$  reinforcing phase. This indicates that at this temperature, the slip system in  $\text{Ti}_2\text{AlC}$  phase is also activated, which is beneficial for enhancing the coordinated deformation ability of different phases. Therefore, the interaction between the second phases of  $\text{Er}_2\text{O}_3$  and  $\text{Ti}_2\text{AlC}$  and dislocations, as well as the nano twinning of  $\gamma$  lamellae, are the main reasons for the excellent strength-plasticity balance exhibited by  $\text{Ti42Al6Nb2.6C0.08Er}$  alloy at 800 °C.

It is worth noting that when the Er content exceeds 0.08at.%, the compressive properties at room temperature no longer increase, but instead decrease, which can be attributed to three aspects. First, the brittle  $\text{ErAl}_3$  phase induces obvious stress concentration. The elastic modulus and fracture toughness of  $\text{ErAl}_3$  phase are significantly different from those of the  $\text{TiAl}$  matrix. Under compressive load, stress concentration is prone to occur at the interface between  $\text{ErAl}_3$  phase and matrix, which becomes a source of crack initiation. Second, the introduced  $\text{ErAl}_3$  phase weakens the bonding of lamellar interfaces and hinders dislocation motion. During compression process, the interface between  $\text{ErAl}_3$  and the lamellar colony is prone to delamination.  $\text{ErAl}_3$  phase impedes dislocation slip and climb, leading to dislocation accumulation at the phase boundary, causing local strain concentration and accelerating the nucleation of micropores. Third, the presence of  $\text{ErAl}_3$  at lamellar colony boundaries destroys the integrity and continuity of the original  $\alpha_2/\gamma$  lamellar structure. The strengthening effect of the lamellar structure is weakened, resulting in a decrease in yield strength.

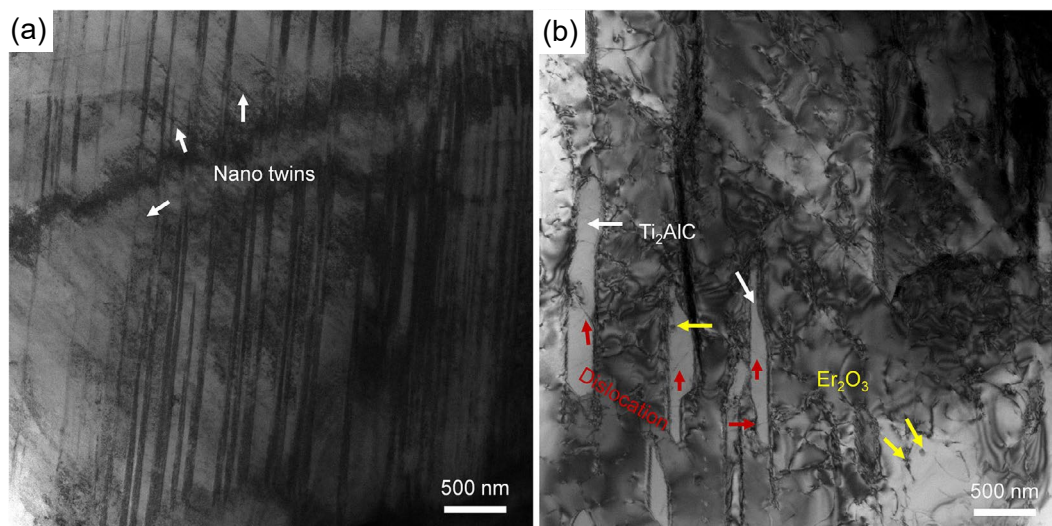


Fig. 9: Microstructure morphologies of  $\text{Ti42Al6Nb2.6C0.08Er}$  alloy after compression deformation at 800 °C: (a) BF of nano-twins inside  $\gamma$  lamellae; (b) BF of  $\text{Ti}_2\text{AlC}$ ,  $\text{Er}_2\text{O}_3$ , and dislocation

## 5 Conclusions

The addition of Er significantly alters the microstructure of the Ti42Al6Nb2.6C alloy. The in-situ formed nanoscale Er<sub>2</sub>O<sub>3</sub> particles notably refine the lamellar colonies and Ti<sub>2</sub>AlC reinforcing phase. The phase constitution and solidification path were analyzed using SEM, XRD, and TEM. The room-temperature and high-temperature compressive properties were tested, and the refinement mechanisms of the microstructure by Er were clarified. The improvement mechanisms of the refined Ti<sub>2</sub>AlC and in-situ Er<sub>2</sub>O<sub>3</sub> particles on the microstructure, as well as the strengthening mechanisms of the Ti42Al6Nb2.6C alloy, were ultimately revealed. The main conclusions are summarized as follows:

(1) As Er content increases from 0.04at.% to 0.08at.%, the average size of lamellar colonies decreases from 57.9 to 25.6 μm. The decrease in aspect ratio of Ti<sub>2</sub>AlC reinforcing phase leads to its refinement and more uniform distribution, thereby promoting overall microstructural refinement.

(2) The high surface energy of Ti<sub>2</sub>AlC reinforcing phase, along with the favorable orientation relationship between Er<sub>2</sub>O<sub>3</sub> and Ti<sub>2</sub>AlC, facilitate the nucleation of Er<sub>2</sub>O<sub>3</sub> particles on the surface of Ti<sub>2</sub>AlC. Therefore, the growth of Ti<sub>2</sub>AlC is constrained by Er<sub>2</sub>O<sub>3</sub>, leading to the formation of finer, shorter rod-like structures. Both Er<sub>2</sub>O<sub>3</sub> particles and refined Ti<sub>2</sub>AlC act as nucleation sites for β phase, which is the fundamental for obtaining finer lamellar colonies.

(3) Ti42Al6Nb2.6C0.08Er alloy exhibits superior compressive properties, with a compressive strength of 2,106 MPa and a strain of 26.8%. Its high-temperature compressive properties show great strength-plasticity balance, especially at 800 °C, the compressive strength reaches 1,587 MPa, and the compressive strain increases to 66.9%.

(4) The improvement in compressive properties is attributed to the significant changes in the microstructures, including significant refinement of the lamellar colonies, the precipitation of nanoscale Er<sub>2</sub>O<sub>3</sub> particles, the load-transfer role of the refined Ti<sub>2</sub>AlC reinforcing phase, and the solid-solution strengthening of Er atoms.

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## 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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