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Effect of Nano-Y₂O₃ Addition on Microstructure and Tensile Properties of High-Nb TiAl Alloy Prepared by Spark Plasma Sintering

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Abstract: Nano-Y₂O₃ reinforced Ti-47.7Al-7.1Nb-(V, Cr) alloy was fabricated by a powder metallurgy route using spark plasma sintering (SPS), and the influence of nano-Y₂O₃ contents on the microstructure and mechanical properties were investigated systematically. The results revealed that the ultimate tensile strength and elongation of the alloy were 570 ± 28 MPa and 1.7 ± 0.6% at 800 °C, 460 ± 23 MPa and 6.1 ± 0.4% at 900 °C with no nano-Y₂O₃, 662 ± 24 MPa and 5.5 ± 0.5% at 800 °C, and 466 ± 25 MPa and 16.5 ± 0.8% at 900 °C with 0.05 at% nano-Y₂O₃ addition, respectively. Due to the fine-grain strengthening and the second-phase strengthening, both tensile strength and elongation of the high-Nb TiAl alloy were enhanced with the addition of nano-Y₂O₃.

Keywords: high-Nb TiAl; nano-Y₂O₃; spark plasma sintering (SPS)



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1. Introduction

With the low density, good elevated-temperature strength, high resistance to oxidation, and excellent creep properties, TiAl alloys are one of the most important aerospace materials widely used in turbine blades, powerplant turbines, turbochargers rotors, and so on [1–4]. Nevertheless, compared with conventional TiAl alloy, high-Nb-containing TiAl alloys demonstrate high strength levels and good oxidation resistance between 800 °C and 900 °C, extending the application temperature of TiAl alloys [5,6].

However, metal matrix composites (MMCs) have been investigated worldwide in recent years because the MMCs can show greatly enhanced ambient and high-temperature strength and stiffness compared with the matrix alloys [7–9]. TiAl metal matrix composites are mainly composed by the addition of ceramic particles, such as Y₂O₃ [10–13], Ti₂AlC [14–19], TiB₂ [20–24], Al₂O₃ [25], WC [26], and B₄C [27,28], and etc. in the TiAl metal matrix. As one kind of rare earth oxide, the thermodynamic characteristics of Y₂O₃ are the most stable [29].

Ding et al. [30] investigated the effect of the Y₂O₃ particles on the fracture toughness of the directionally solidified Ti-45Al-2Cr-2Nb, and the results showed that the fracture toughness reduced with the increase of Y₂O₃ particles, and the cracking along the bonding interface and the ceramic particles. Tian et al. [12] fabricated the Y₂O₃ particles reinforced Ti-48Al-2Cr-2Nb alloy by induction skull melting (ISM), and it demonstrated that within a certain range, adding nano-Y₂O₃ could refine the microstructure and improve strength and elongation of TiAl alloy. Guo et al. [11] found that the content γ phase increased with the addition of nano-Y₂O₃, and the solidification pathway of TiAl alloy into high-Al side. The ultimate tensile strength (UTS) and elongation were 561 MPa and 1.49% for T4822-Y₂O₃ and 611 MPa and 0.79% for the T486-Y₂O₃ alloy. Xiao et al. [10] reported 0.15 at% nano-Y₂O₃ addition could increase ultimate tensile strength (UTS) and elongation of the TNV alloy.

The TNV-Y₂O₃ alloy displayed lower steady-state creep rates and longer creep life, and the stress exponent was 3.09 lower than the 4.03 of the TNV alloy. Gu et al. [31] prepared the β -solidifying TiAl alloy with YAl₂ and Y₂O₃ precipitates by spark plasma sintering, and the sintered specimen at 1150 °C had an ultimate tensile strength of 1113.1 MPa, yield strength of 881 MPa, and elongation of 1.41%. Due to the Y₂O₃ and YAl₂ precipitates, the grains and lamellar spacing were refined, dragged the grain boundary migration, and hindered the dislocation movement, leading to the improvement of the mechanical properties.

Therefore, adding Y₂O₃ powders is a feasible way to adjust the microstructure and properties, and there are rarely studies on the addition of nano-Y₂O₃ through the powder metallurgy method. This research concentrated on the high-temperature tensile properties and fractures behavior of high-Nb TiAl alloy with Y₂O₃ addition. The influence on microstructure and elevated-temperature tensile properties with different contents of Y₂O₃ addition was investigated.

2. Materials and Methods

The pre-alloyed powders of high-Nb TiAl alloy with chemical compositions of Ti-47.7Al-7.1Nb-1.1Cr-2.3V (at%) were prepared by plasma-rotating electrode process. As shown in Figure 1a, most of the pre-alloyed powder is spherical and a small part is satellite powder. The interior morphology exhibits a dendrite appearance and the particle sizes of pre-alloyed powders range from 11.68 μ m to 40.24 μ m, as shown in Figure 1b,c. The Y₂O₃ powders were supplied by Shanghai Naiou Nanotechnology Co. Ltd with an average particle size of 80 nm. The pre-alloyed powders and nano-Y₂O₃ powders were mixed in an argon atmosphere by ball milling with the weight ratio of balls to powders of 5:1, and a speed of 250 r/min for 6 h. After the mixed powders were sintered using the spark plasma sintering furnace (SPS-20 T-10 V, Shanghai Chen Hua Technology Co. Ltd., Shanghai, China), they were held for 3 min at 1350 °C under uniaxial stress of 45 MPa.

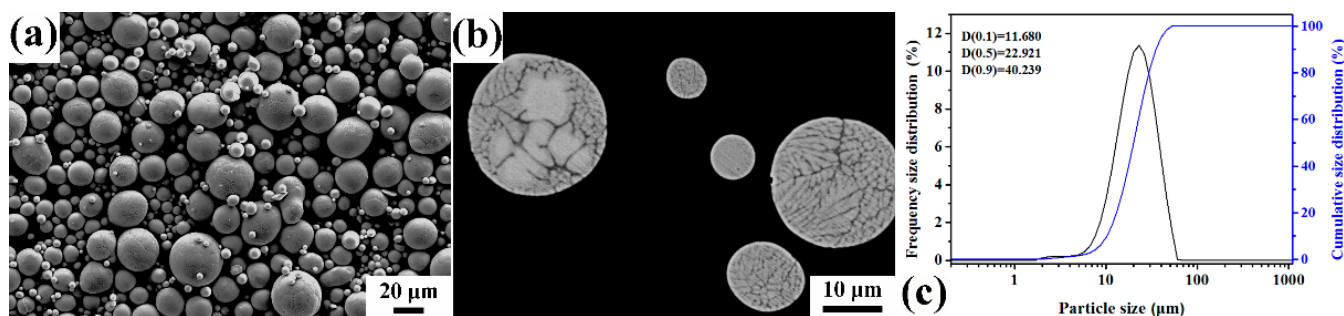


Figure 1. (a,b) SEM images of pre-alloyed powder; (c) particle size distribution.

The SEM samples were first ground with SiC papers to 1500 grit, then electron-polished in a solution of 60 vol% methanol, 35 vol% butanol, and 5 vol% perchloric acid. The fracture specimens were prepared by mechanical polishing with grinding up to 5000 mesh. The microstructure was characterized by scanning electron microscopy (SEM, Carl Zeiss AG, Jena, Germany), and the lamellar colony size and its volume fraction were measured by image-pro plus. A uniaxial tensile test was performed using the CMT4105 (Changchun Institute of Testing Machines, Changchun, China) machine at 800 °C and 900 °C with a constant displacement rate of $5 \times 10^{-4} \text{ s}^{-1}$. The tensile specimens had a gauge length of 10 mm, width of 2.5 mm, and thickness of 1.2 mm, and the surfaces of the tensile specimen were ground to 1500-grit SiC papers before testing.

3. Results and Discussion

3.1. Microstructure

The XRD pattern of Ti-47.7Al-7.1Nb-1.1Cr-2.3V (at%) pre-alloyed powders and four alloys with different content of nano-Y₂O₃ are shown in Figure 2. It can be seen that

the spectrum of pre-alloyed powders contained α_2 -Ti₃Al, γ -TiAl, and B2 peaks, but was dominated by the peaks of the α_2 phase. This was due to the high cooling rate of droplets about 10^4 – 10^6 K s^{−1} during the atomization, leading to the decrease of transformation $\alpha \rightarrow \gamma$ replaced by $\alpha \rightarrow \alpha_2$ ordering transformation [32]. After sintering, the high-Nb TiAl alloys with different content of nano-Y₂O₃ addition were mainly including γ -TiAl and α_2 -Ti₃Al phases but no obvious peaks of Y₂O₃ due to the small addition.

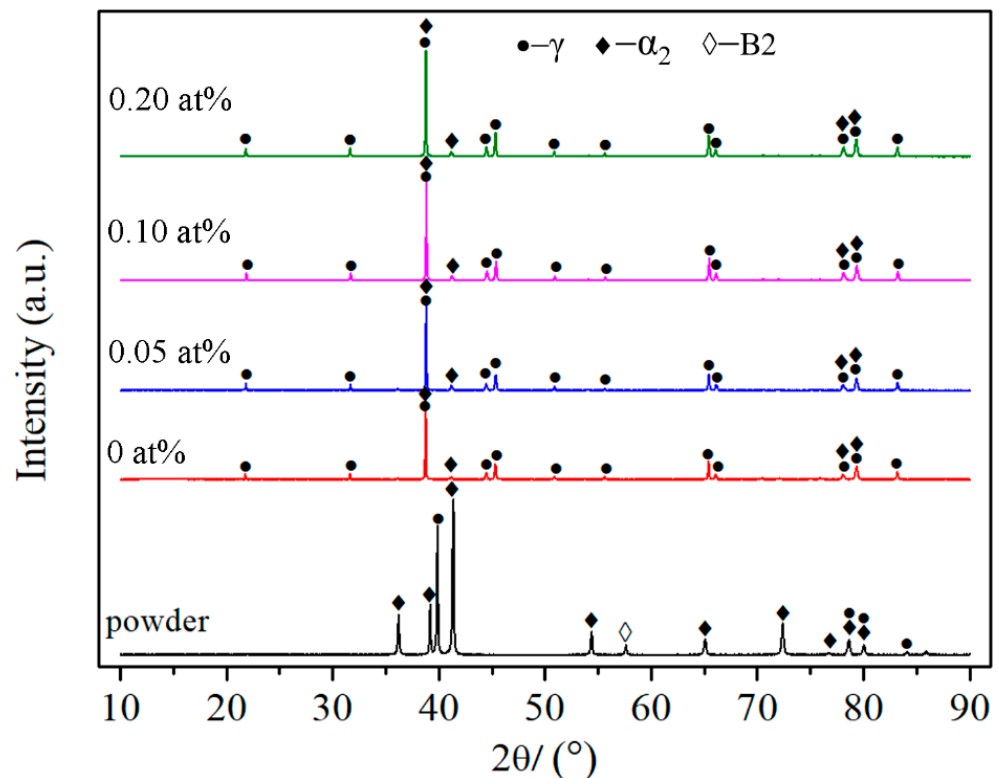


Figure 2. XRD pattern of pre-alloyed powders and four alloys with different content of nano-Y₂O₃.

Figure 3 shows the SEM-BSE microstructures of the experimental alloys with different content of nano-Y₂O₃ addition. It was found that the microstructure of Ti-47.7Al-7.1Nb-1.1Cr-2.3V alloy consisted of $\gamma + \alpha_2$ lamellar and a few γ , α_2 , and B2 phases around the boundaries of the lamellar colonies or in the $\gamma + \alpha_2$ lamellar without addition nano-Y₂O₃ as shown in Figure 3a,b. With 0.05 at% of Y₂O₃ addition, the sample showed a duplex microstructure including half approximately of the lamellar colony and more equiaxed γ phase exhibited in Figure 3c. Due to the nano-Y₂O₃ particles being distributed on the surface of pre-alloyed powders after ball milling, the Y₂O₃ particles displayed an obvious network structure in the TiAl matrix. It can be seen from Figure 3d that most of the still Y₂O₃ nanoparticles and a few of the micro-scale precipitates were in the TiAl matrix with the 0.05 at% nano-Y₂O₃ addition. The Y₂O₃ particles were dispersed in the γ phase, lamellar colony, and colony boundary. Obviously, with the content of Y₂O₃ increasing from 0.05 to 0.20 at%, the microstructure of the sintered sample changed from duplex to near lamellar as shown in Figure 3e,g. However, more micro-scale Y₂O₃ particles were observed, as shown in Figure. After 3 h, the content of nano-Y₂O₃ increased to 0.20 at%, due to the additional Y₂O₃ particles distributed on the surface of the pre-alloyed powders after ball milling during the sintering process and additional particle agglomeration and grow up.

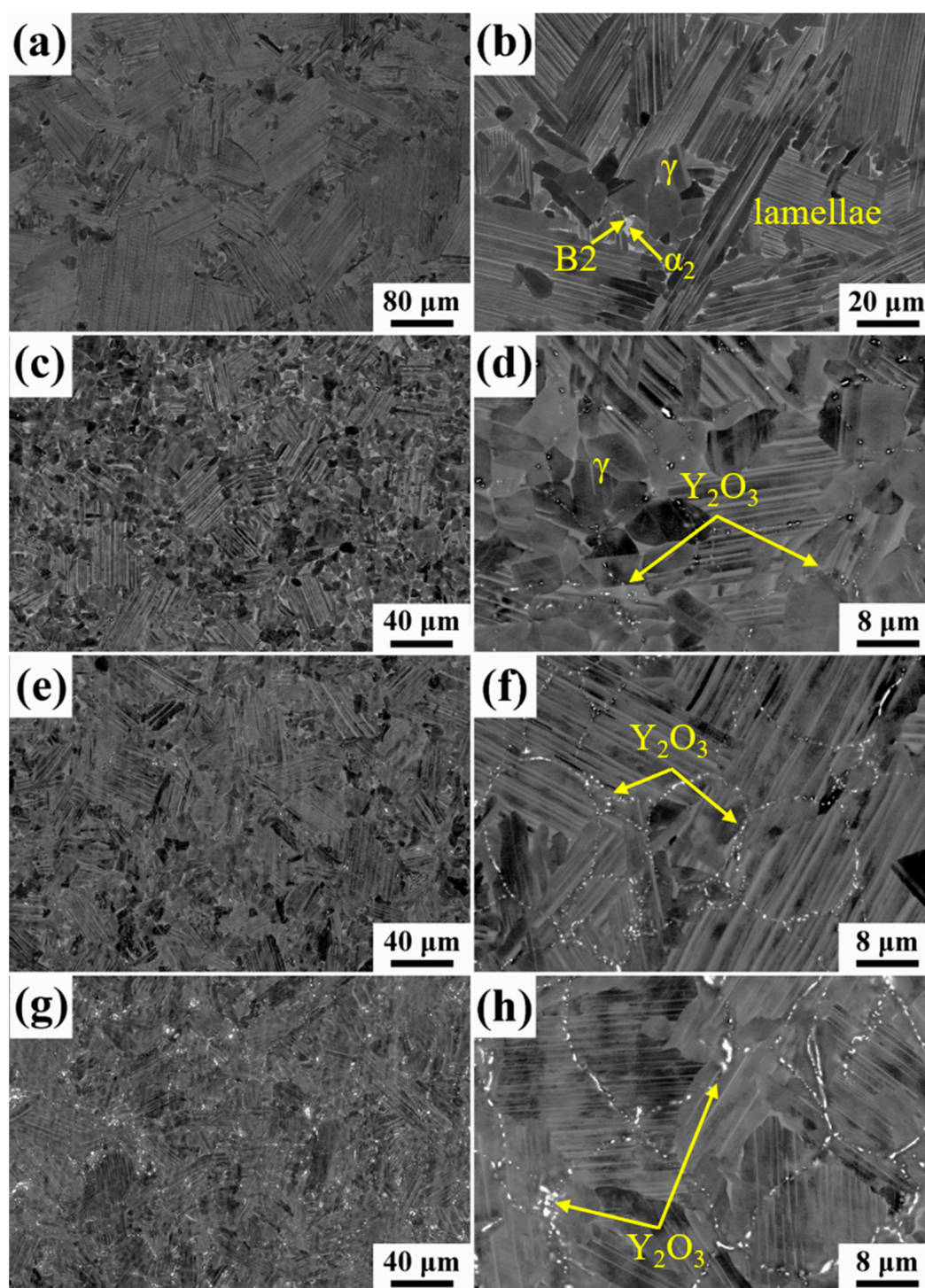


Figure 3. SEM-BSE images of samples adding difference content of nano- Y_2O_3 (a,b) 0 at%; (c,d) 0.05 at%; (e,f) 0.10 at%; (g,h) 0.20 at%.

Figure 4 displays the lamellar colony size and its volume fraction with different content of nano- Y_2O_3 addition using the Image-pro plus measurement. It can be seen that the mean lamellar colony size was $110 \pm 35 \mu\text{m}$ and its volume fraction was about $95 \pm 2\%$ without Y_2O_3 addition. With the content increase from 0.05% to 0.20%, the volume fractions of lamellar colony raised from $50 \pm 10\%$ to $93 \pm 5\%$. However, the average lamellar colony sizes with 0.05 at%, 0.1 at%, and 0.2 at% addition were $32 \pm 12 \mu\text{m}$, $35 \pm 10 \mu\text{m}$, $35 \pm 15 \mu\text{m}$, and not significantly different.

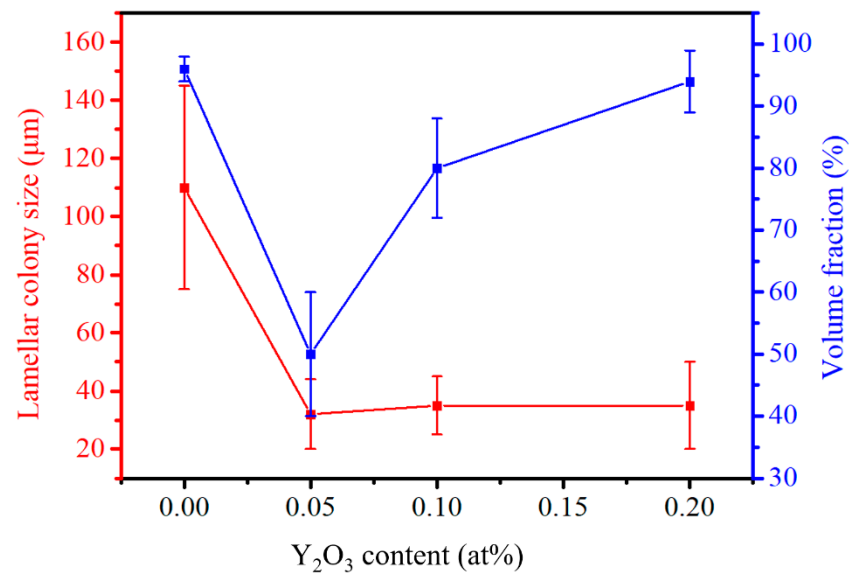


Figure 4. Lamellar colony size and its volume fraction with different content of Y₂O₃ addition.

The schematic diagrams of microstructure evolution before and after sintering is exhibited in Figure 5 without and with the addition of Y₂O₃. According to the Ti-Al quasi-binary phase diagram containing 8Nb (at%) in Ref. [33], the sintering temperature at 1350 °C was in the $\alpha + \gamma$ phase region. Furthermore, the pre-alloyed powder mainly consisted of α_2 phase and a few γ and B2 phases, and the lamellar microstructure was probably transformed by $\alpha \rightarrow \alpha + \gamma \rightarrow L(\alpha_2/\gamma)$ after the sintering holding 3 min at 1350 °C as shown in Figure 5a–c [33,34]. With the addition of nano-Y₂O₃, most particles were relatively uniformly distributed on the surface of the pre-alloyed powder after ball milling. Due to the ball milling speed not being too high, most of the pre-alloyed powders were still spherical before sintering. Figure 5d–f exhibits the microstructure evolution of the pre-alloyed powder mixing nano-Y₂O₃ particles before and after sintering. The Y₂O₃ particles seemed to limit the grain growth and played a prominent role in retaining a fine microstructure.

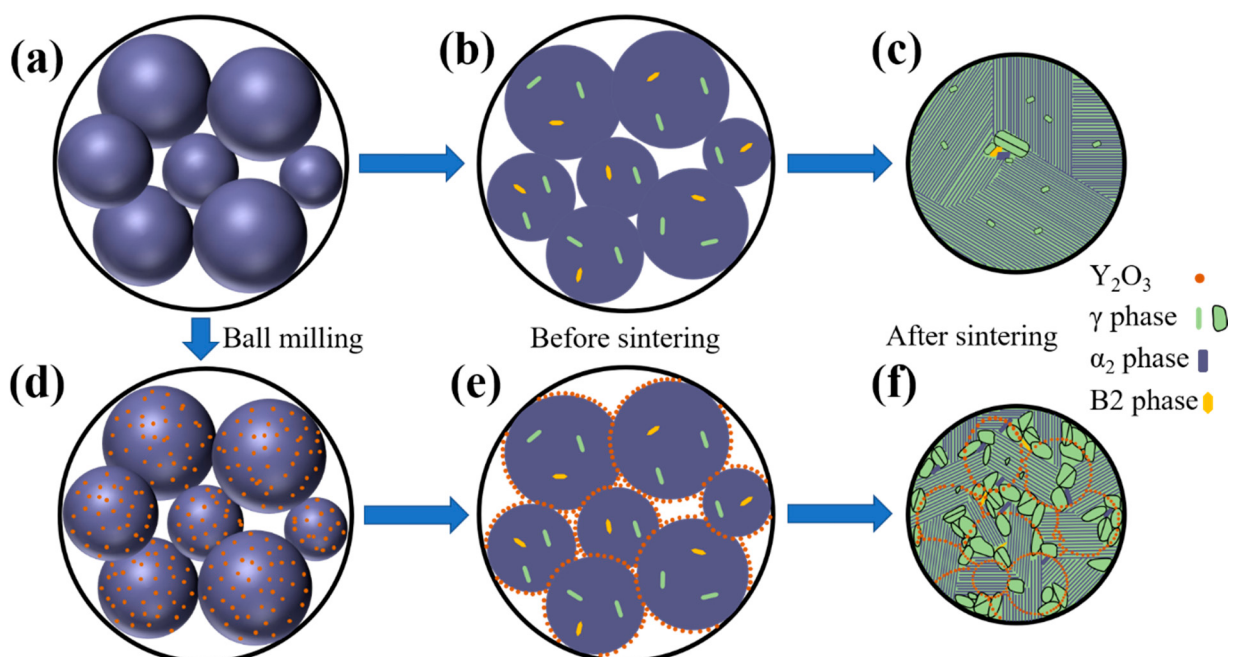


Figure 5. Schematic diagrams of the sintering process: (a–c) no addition nano-Y₂O₃; (d–f) addition nano-Y₂O₃.

3.2. Tensile Properties and Fractography

Tensile properties of the alloys at 800 °C and 900 °C with different content of nano- Y_2O_3 addition are illustrated in Figure 6. It can be seen from the results that the ultimate tensile strength (UST) increased with the Y_2O_3 addition and decreased with the improvement of the test temperature. At 800 °C, the ultimate tensile strength (UST) and elongation of Ti-47.7Al-7.1Nb-1.1Cr-2.3V (at%) alloy were 569 ± 25 MPa and 1.55 ± 0.3 % without adding Y_2O_3 . Compared with the high-Nb TiAl alloy matrix, adding 0.05 at% nano- Y_2O_3 , the UST increased about 82 MPa and the elongation improved about 4.05%. However, the UST had reduced with the increment of Y_2O_3 from 0.05 at% to 0.2 at%, and elongation was slightly different. Increasing the test temperature to 900 °C, the UST of the high-Nb TiAl alloy was 455 ± 15 MPa and did not change significantly with the addition of Y_2O_3 particles, but the elongation rose from 6.1 ± 0.5 % to 16.5 ± 0.6 % with the 0.05 at% addition. Moreover, the alloys with 0.1 at% and 0.2 at% addition did not have a significant influence on the high-temperature tensile properties as shown in Figure 6. Nano- Y_2O_3 addition improved both the ultimate tensile strength (UST) and elongation at elevated temperatures, and it mainly consists of two kinds of strengthening mechanisms. On the one hand, the lamellar colony size significantly reduced with the addition of Y_2O_3 particles. According to the Hall-Petch relationship, the refinement of the grain has a critical influence on the increase of mechanical property, and the interface of the fine lamellar can hinder the movement of the substructure [11,31,35]. On the other hand, most of the Y_2O_3 second phase is still nano size, and in a few parts of the particle agglomeration with 0.05 at% addition, the particles can block the movement of dislocation and promote the nucleation of twins both in the lamellar colonies and in the γ phase [31,35]. Due to the presence of Y_2O_3 particles, it forms dislocation loops and tangles when the dislocation moves through the particles by bending around, and the lattice dislocation energy increases with the bending of dislocation, leading to the improved strength [11].

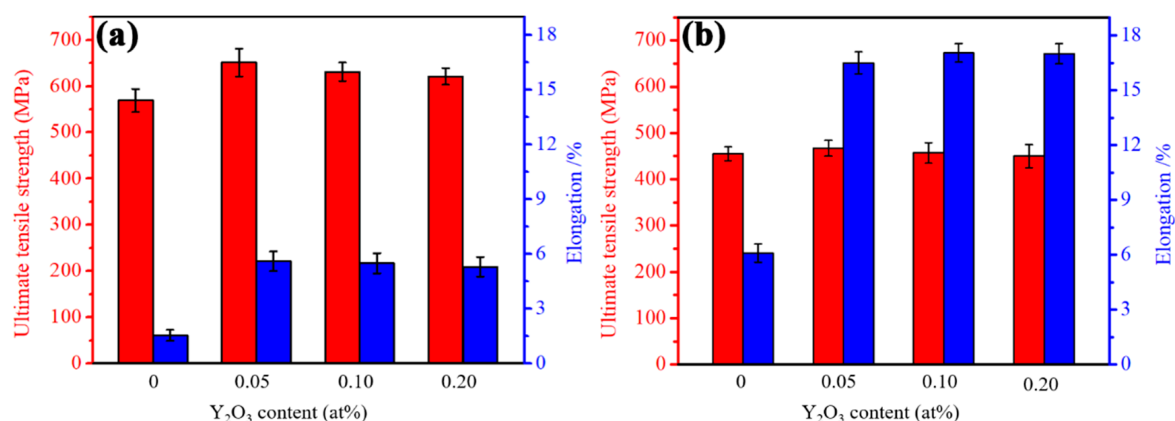


Figure 6. Ultimate tensile strength and elongation at elevated temperatures with different content of nano- Y_2O_3 addition: (a) 800 °C; (b) 900 °C.

Figure 7 exhibits the fracture side face after the tensile test at 800 °C with different content of Y_2O_3 addition. It was observed that the secondary cracks appeared in the inter-lamellar or lamellar boundaries, mainly concentrated near the fracture after tensile testing. During the tensile test, the micro-crack frequently nucleated at the interface of γ/α_2 lamellae due to the poor bonding strength between α_2 and γ lamellae [11]. Figure 7a,b exhibits the secondary crack propagates along the lamellar and changing of the direction in the boundaries of the lamellar without nano- Y_2O_3 addition. Owing to the addition of Y_2O_3 particles, the lamellar colony sizes were decreased and the fine lamellar microstructure restricted the growth of cracks as shown in Figure 7c–h. The combination of the nano- Y_2O_3 and the matrix worked very well, and there were no cracks or holes between the interfaces after tensile testing at 800 °C.

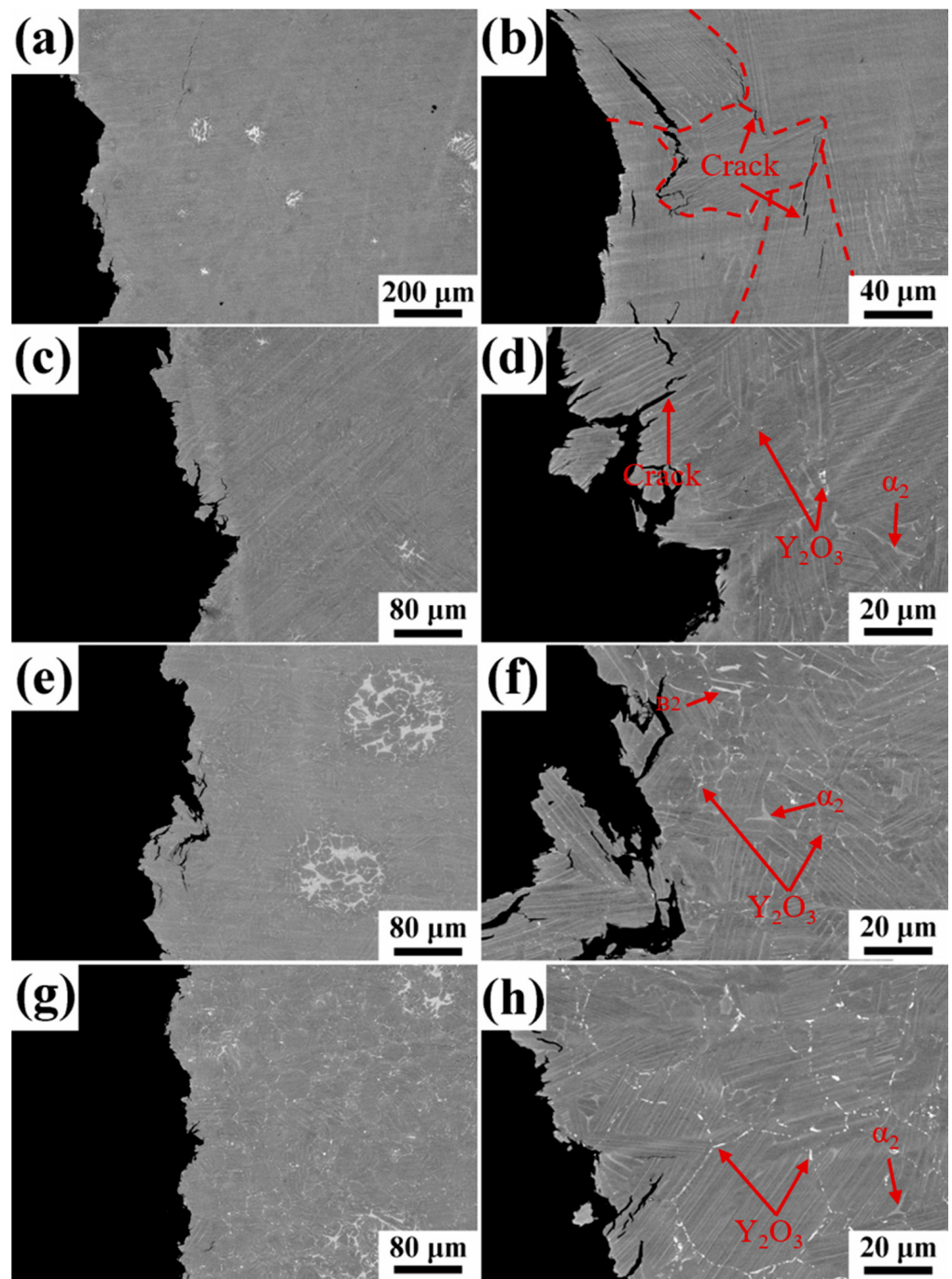


Figure 7. Fracture side face after tensile test at 800 °C: (a,b) 0 at%; (c,d) 0.05 at%; (e,f) 0.10 at%; (g,h) 0.20 at%.

The microstructure of the fracture side face after the tensile test at 900 °C is illustrated in Figure 8. The secondary cracks also extended along the direction of the lamellar or perpendicular to the lamellar without the addition of Y_2O_3 . Compared with the tensile test at 800 °C, there were significant second cracks and voids in the interface of the lamellar boundaries or the grain boundaries. The reason for this result was mainly due to the massive γ phase between the lamellar colonies in the duplex microstructure and near-lamellar microstructure. However, there were no obvious microcracks between the interfaces of Y_2O_3 particles and the matrix, which proves the good bonding strength of the Y_2O_3 and matrix. After the tensile test at 900 °C, it was observed that the lamellae curved in the alloys with different contents of nano- Y_2O_3 addition.

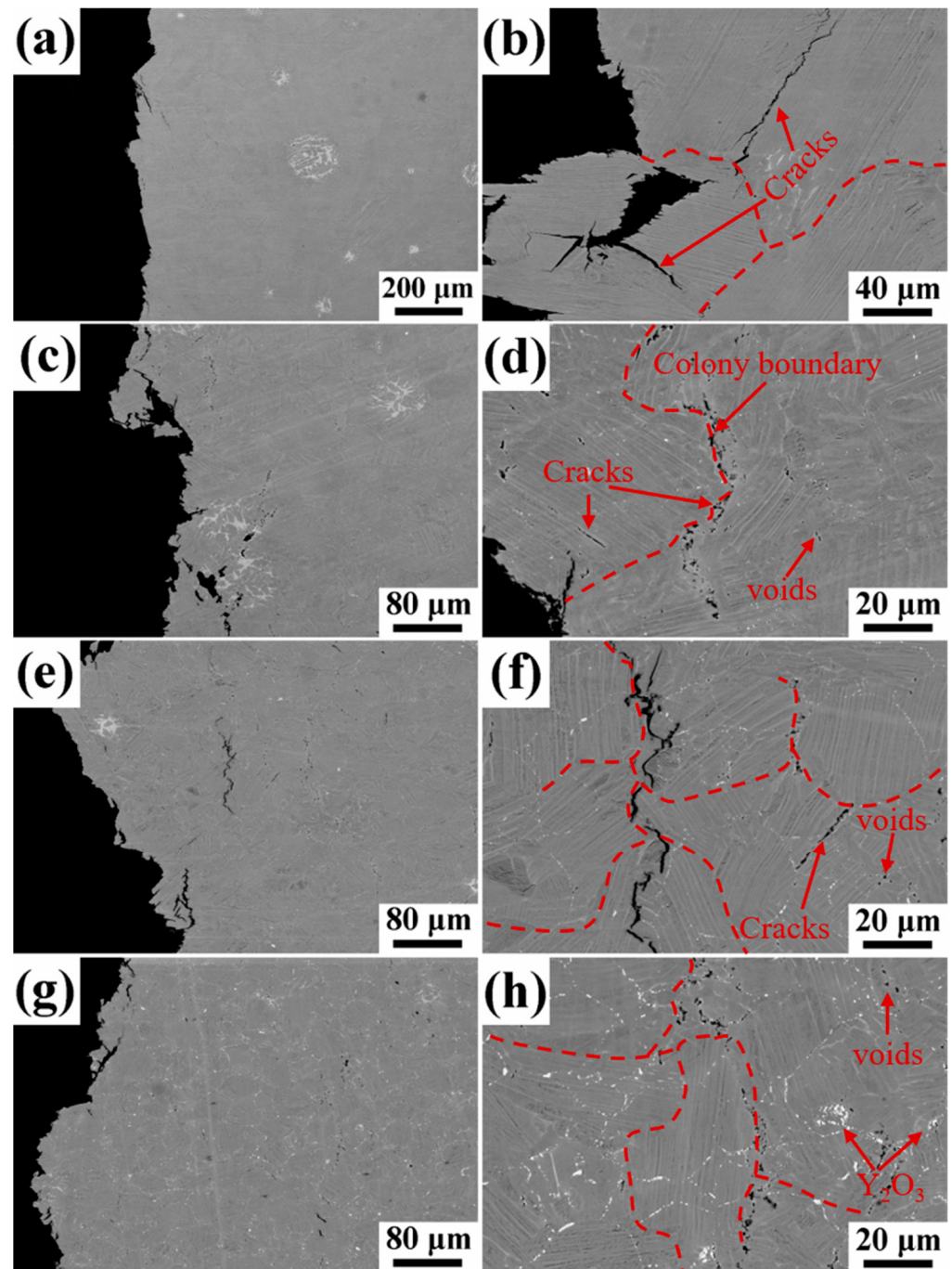


Figure 8. Fracture side face after tensile test at 900 °C: (a,b) 0 at%; (c,d) 0.05 at%; (e,f) 0.10 at%; (g,h) 0.20 at%.

Figures 9 and 10 exhibit the tensile fracture morphology at 800 °C and 900 °C with different content of Y_2O_3 addition. The fracture morphology of the alloy without adding Y_2O_3 was similar with no obvious dimples at both 800 °C and 900 °C, with the secondary crack, tearing ridges, and cleavage face as shown in Figure 9a,b and Figure 10a,b, and the fracture mechanisms still exhibited cleavage fractures. With the addition of nano- Y_2O_3 , the main reason for the failure of alloys at 800 °C and 900 °C was still cleavage cracks, and no obvious dimples were found in the fracture morphology. Therefore, the increase of the elongation at 900 °C was mainly caused by the second cracks and voids in the interface of the lamellar boundaries and the grain boundaries. Additionally, due to the fine grain strengthening and the second phase reinforcement, the mechanical property was improved.

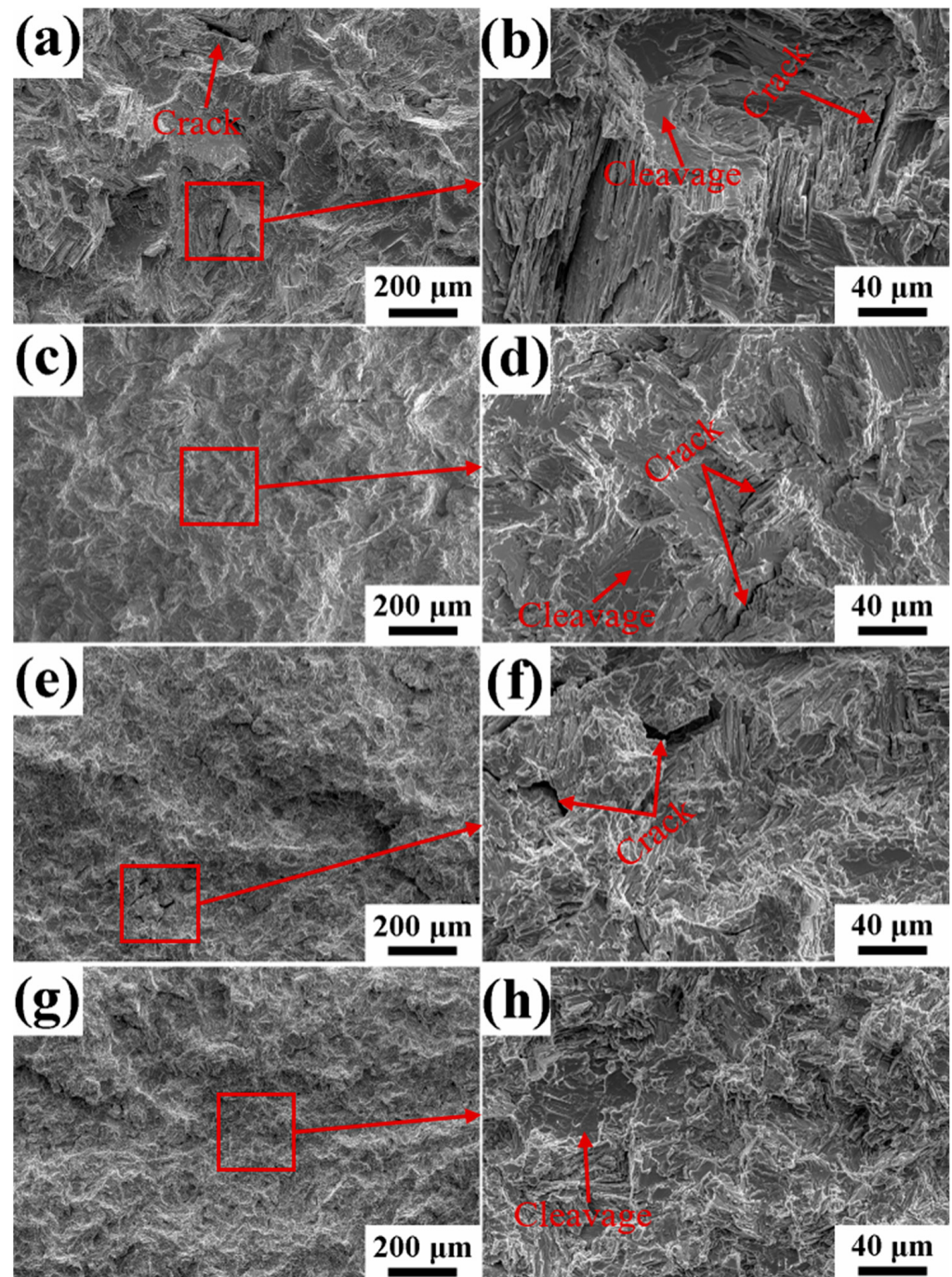


Figure 9. Tensile fracture morphology at 800 °C: (a,b) 0 at%; (c,d) 0.05 at%; (e,f) 0.10 at%; (g,h) 0.20 at%.

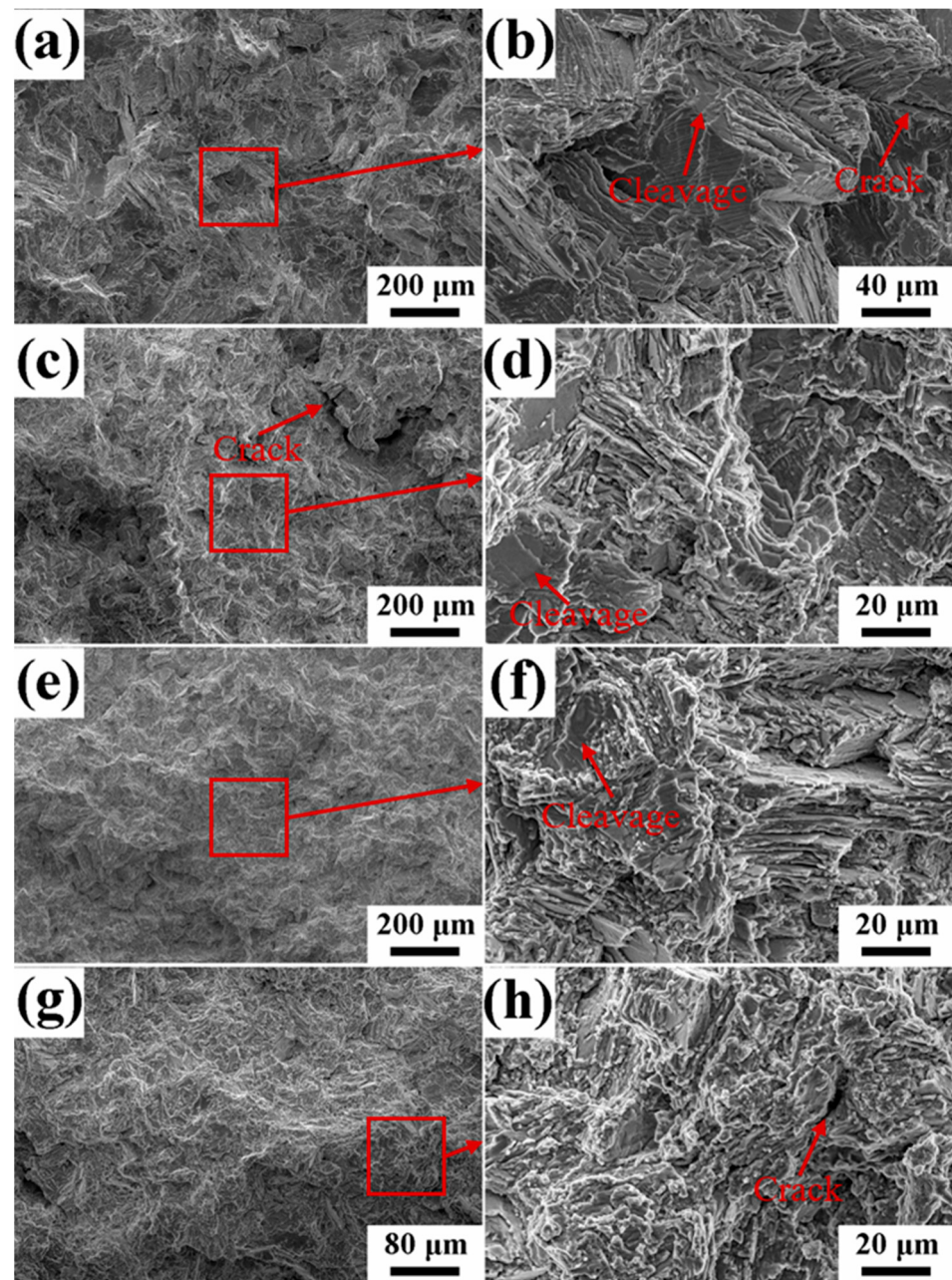


Figure 10. Tensile fracture morphology at 900 °C: (a,b) 0 at%; (c,d) 0.05 at%; (e,f) 0.10 at%; (g,h) 0.20 at%.

4. Conclusions

The present work mainly concentrated on the influence of adding different content of nano- Y_2O_3 on the high temperature tensile properties. The conclusions are summarized as follows.

(1) With the 0.05 at% addition of Y_2O_3 , the sample showed duplex microstructure including approximately half of the lamellar colony and a more equiaxed γ phase. Most of the particles were still nano-size, and the average lamellar colonies size decreased from $110 \pm 35 \mu\text{m}$ to $32 \pm 12 \mu\text{m}$.

(2) The ultimate tensile strength (UST) and elongation of the alloy were $569 \pm 25 \text{ MPa}$ and $1.55 \pm 0.3\%$ without adding Y_2O_3 . Compared with the high-Nb TiAl alloy matrix, the UST increased about 82 MPa and the elongation improved about 4.05% when adding 0.05 at% nano- Y_2O_3 .

(3) The enhancement of the high-temperature tensile property contributed to fine grain strengthening and the second-phase reinforcement. The interface of the fine lamellar can hinder the movement of the substructure and the second phase of Y_2O_3 can block the movement of dislocation.

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