

PAPER • OPEN ACCESS

Precipitation-hardened refractory Ti-Nb-Hf-Al-Ta high-entropy alloys

To cite this article: E Panina *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1014** 012041

View the [article online](#) for updates and enhancements.



240th ECS Meeting ORLANDO, FL

Orange County Convention Center **Oct 10-14, 2021**

Abstract submission deadline extended: April 23rd

SUBMIT NOW

Precipitation-hardened refractory Ti-Nb-Hf-Al-Ta high-entropy alloys

E Panina*, N Yurchenko, S Zherebtsov, G Salishchev and N Stepanov

Laboratory of Bulk Nanostructured Materials, Belgorod State University, Belgorod 308015, Russia

*Corresponding author: panina_e@bsu.edu.ru

Abstract. This study reports the structure and mechanical properties of new refractory $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$, $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$, and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ (at.%) high entropy alloys. After annealing at 1200 °C for 24 h, the program alloys had a single-phase B2 structure. Further annealing at 600 °C for 24 h resulted in the formation of Widmanstätten orthorhombic particles (O-phase) in the bcc matrix. The $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloys annealed at $T = 1200$ °C showed moderate strength and good ductility (>50%) at 22 and 600 °C; while the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$ alloy was stronger, but less ductile at both temperatures. Subsequent annealing at $T = 600$ °C significantly increased the strength of the $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ alloy at 22 and 600 °C, maintaining compressive sufficient ductility at room-temperature .

1. Introduction

Recently introduced refractory high entropy alloys (RHEAs), which demonstrated an outstanding capability to maintain high strength at temperatures up to $T = 1600$ °C [1], seem promising candidates for next-generation turbines. Despite the attractive high-temperature strength, one of the major drawbacks of most RHEAs is the modest mechanical performance at ambient temperatures.

It is believed that balanced properties can be obtained by developing precipitation-strengthened RHEAs. One of the promising examples of such RHEAs are alloys with the bcc/B2 structure [2, 3]. The superalloy-like microstructure with a bcc solid solution, strengthened by coherent cuboidal B2 nanoparticles, provides a good combination of strength and ductility at temperatures $T \leq 600$ °C. However, the development of bcc/B2 RHEAs is a non-trivial task due to weakly established composition-structure relationships and poor fidelity of thermodynamic modeling in the case of the B2 phase [4, 5].

In this work, we presented a series of RHEAs strengthened by nanosized Widmanstätten orthorhombic (O-phase) particles. The O-phase precipitation was found to be an attractive option to achieve a notable strength enhancement both at room and elevated temperatures with only a slight loss in ductility.



2. Materials and methods

Ingots of the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$, $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$, $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloys were produced by vacuum arc melting of pure (≥ 99.9 wt%) elements. The alloys were annealed in quartz tubes at 1200 °C for 24 h. Some samples were further annealed at 600 °C for 24 h. The phase composition and microstructure of the alloys were studied using transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The densities of the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$, $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$, $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloys, determined by the hydrostatic weighing method, were 7.23 ± 0.03 , 7.07 ± 0.03 , 7.87 ± 0.03 g/cm³, respectively. Isothermal compression was carried out in air at 22 °C or 600 °C using an Instron 300LX test machine.

3. Results

3.1. Microstructure

After annealing at 1200 °C, the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$, $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$, and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloys had a coarse-grained single-phase microstructure with the average grain size ~ 300 μm , (Figure 1a-c); no secondary phases were observed.

After further annealing at 600 °C, precipitation of profuse Widmanstatten second phase particles was revealed. The particles located adjacent to grain boundaries were coarser than the particles within the grain interior. The volume fraction of the second phase was nearly equal in all program alloys and estimated to be $\sim 35\%$.

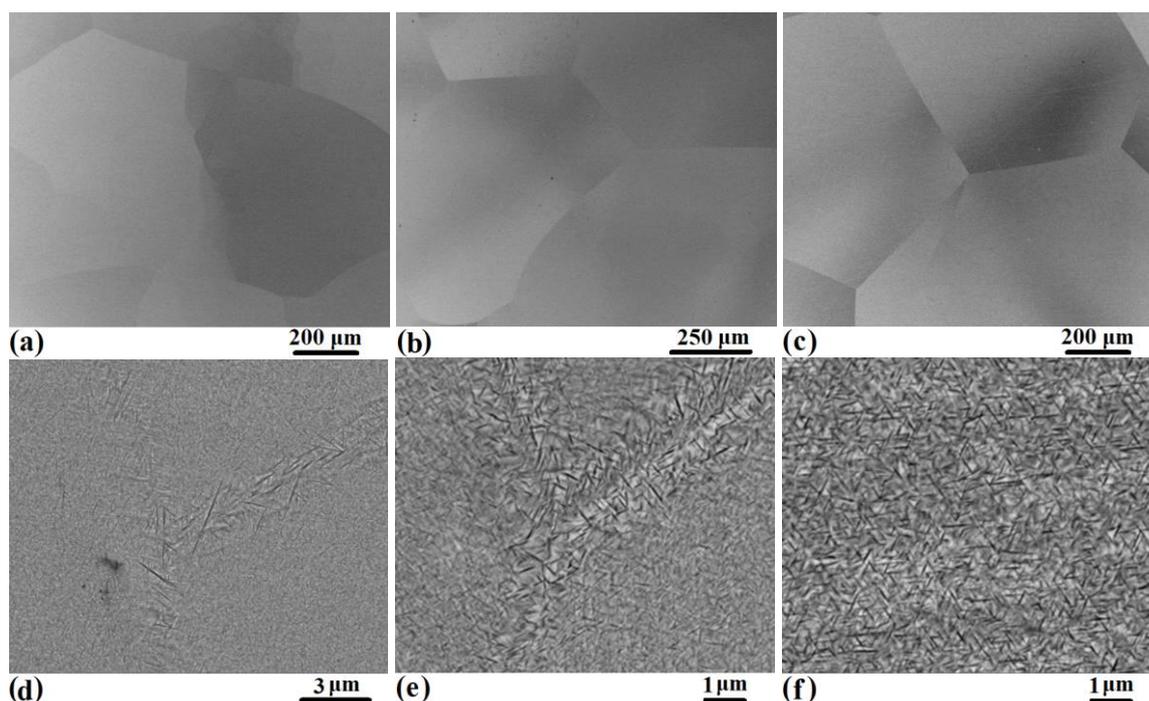


Figure 1. SEM images of the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$ (a, d), $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ (b, e), $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ (c, f) alloys after annealing at 1200°C, 24 h (a, b, c) and further annealing at 600°C, 24h (d, e, f).

After annealing at 1200 °C, all the program alloys had a single-phase B2 structure as revealed by the selected area electron diffraction (SAED) patterns. TEM analysis revealed numerous anti-phase boundaries in the matrix grains (denoted in figure 2a-c as APBs); no secondary phases were found in all alloys.

The phases found after further annealing at 600 °C, were identified using TEM analysis: the matrix had the B2 ($\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$) or bcc ($\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$) structure, whilst the particles were defined as the O-phase.

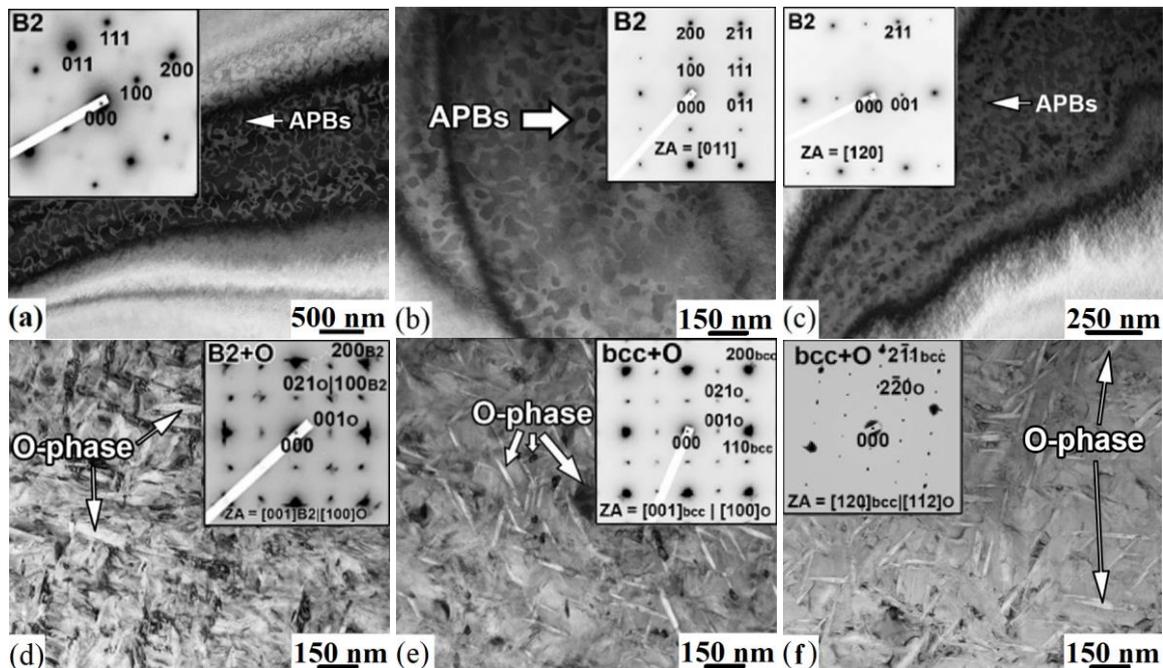


Figure 2. TEM bright-field images of the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$ (a, d), $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ (b, e), $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ (c, f) alloys after annealing at 1200 °C, 24 h (a, b, c) and further annealing at 600 °C, 24h (d, e, f). Selected area electron diffraction patterns used for phase identification are shown in the inserts.

3.2. Mechanical properties

Stress-strain curves of the program alloys annealed at 1200 °C, i.e. with the single B2 phase structure, are presented in figures 3a, b. At 22 °C, the $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloys demonstrated moderate strength (830 and 1075 MPa, respectively), high strain hardening capacity and compressive ductility. Meanwhile, the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$ alloy was the strongest (1645 MPa), albeit fractured after 1% of plastic deformation. At 600 °C, the strength of the $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloys reduced to 635 and 700 MPa, respectively. The $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$ alloy exhibited a notable decrease in yield strength to 810 MPa with a simultaneous increase in compressive ductility to 12%.

Subsequent annealing at 600 °C (which caused precipitation of the O-phase particles in the bcc/B2 matrix) changed the mechanical properties of the program alloys markedly (Figures 3c, d). The $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ alloy demonstrated a ~50% strength increment both at 22 and 600 °C while maintaining reasonable compressive ductility at 22 °C. A similar annealing effect on room-temperature mechanical properties was also found for the $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloy; however, at 600 °C, the alloy performance degraded inevitably; i.e. both strength and ductility became worse in comparison with annealing at 1200 °C. In the case of the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$ alloy, annealing at 600 °C increased its high-temperature strength, but drastically diminished compressive ductility. The obtained findings suggest complex relationships between the chemical composition, structure, and mechanical properties of the O-phase strengthened RHEAs since similar microstructural changes (precipitation of the O-phase particles) had a drastically different effect on the mechanical performance of the program alloys. Further

studies are required to reliably establish the relationship between the composition and the structural properties of these alloys.

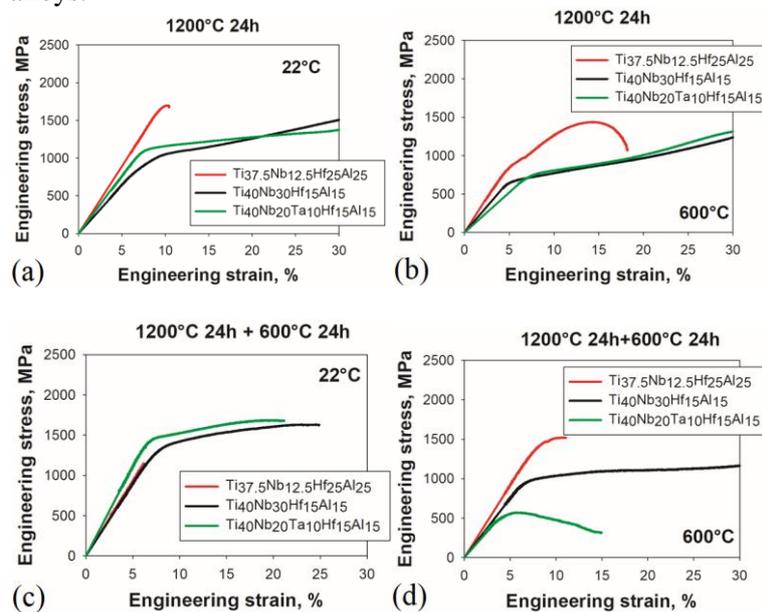


Figure 3. Engineering stress-strain curves of the program alloys after annealing 1200 °C (a, b) and further annealing at 600 °C (c, d) obtained during compression at 22 (a,c) and 600 °C (b,d).

4. Conclusions

Microstructure and mechanical properties of the refractory $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$, $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$, and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ high entropy alloys were studied. The following conclusions were drawn:

- After annealing at 1200 °C, all the program alloys have a single-phase B2 structure. Further annealing at 600 °C led to the formation of a mixture of the B2 ($\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$) or bcc ($\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$, $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$) matrix and nanosized Widmanstätten O-phase particles.
- In the single-phase state, the $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloys demonstrated moderate strength and high compressive ductility at 22 and 600 °C. The $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$ alloy showed the highest strength, but very limited ductility. The precipitation of the O-phase enhanced the strength of the $\text{Ti}_{40}\text{Nb}_{30}\text{Hf}_{15}\text{Al}_{15}$ alloy at 22 and 600 °C without notable sacrificing in compressive ductility at 22 °C. However, the mechanical properties of the $\text{Ti}_{37.5}\text{Nb}_{12.5}\text{Hf}_{25}\text{Al}_{25}$ and $\text{Ti}_{40}\text{Nb}_{20}\text{Ta}_{10}\text{Hf}_{15}\text{Al}_{15}$ alloys deteriorated after annealing at 600 °C.

Acknowledgments

This study was supported by the Russian Science Foundation, grant № 19-79-30066.

References

- [1] Senkov O N, Wilks G B, Scott J M and Miracle D B 2011 *Intermetallics* **19** 698
- [2] Soni V, Senkov O N, Gwalani B, Miracle D B and Banerjee R 2018 *Sci. Rep.* **8** 1
- [3] Senkov O N, Couzinie J-P, Rao S I, Soni V and Banerjee R 2020 *Materialia* **9** 100627
- [4] Soni V, Senkov O N, Couzinie J-P, Zheng Y, Gwalani B and Banerjee R 2020 *Materialia* **9** 100569
- [5] Whitfield T E, Pickering E J, Owen L R, Jones C N, Stone H J and Jones N G 2020 *Materialia* **13** 100858