Iranian Journal of Materials Forming 12 (1) (2025) 10-17

Online ISSN: 2383-0042

Iranian Journal of Materials Forming

Journal Homepage: http://ijmf.shirazu.ac.ir

Research Article

The Effect of Tungsten on Microstructure and Mechanical Properties of Sintered Tantalum by Hot Pressing in a Vacuum

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ARTICLE INFO

Article history:

Received 28 November 2024
Reviewed 24 December 2024
Revised 4 February 2025
Accepted 15 February 2025

Keywords:

Tantalum alloy
Tungsten
Hot pressing
Mechanical properties
Resilience modulus

Please cite this article as:

Najafzadegan, F., Khodabandeh, A., Youzbashizadeh, H., & Tamizifar, M. (2025). The effect of tungsten on microstructure and mechanical properties of sintered tantalum by hot pressing in a vacuum. *Iranian Journal of Materials Forming*, *12*(1), 10-17. https://doi.org/10.22099/IJMF.2025.51766.1309

ABSTRACT

In this study, the effect of tungsten addition on the microstructure and mechanical properties of tantalum using hot pressing-sintering is investigated. Pure tantalum and tungsten powders were used to produce Ta-W alloys with varying tungsten content, including pure Ta, Ta-2.5 wt.% W, Ta-5 wt.% W, and Ta-7.5 wt.% W. The morphology of the powders was examined by scanning electron microscopy. Consolidation was performed via hot pressing-sintering at 1600 °C under vacuum conditions. Density was measured using the Archimedes method, and the microstructure was analyzed by optical microscopy. Hardness measurements were conducted on the sintered samples, and their deformation behavior was evaluated using uniaxial compression test at room temperature. The results indicated that the relative density of pure Ta is 97.5%, which decreases to 92.5% with the addition of 7.5 wt.% tungsten. With increasing tungsten content, the average grain size decreased while the porosity percentage increased. The properties of Ta-W alloys are influenced by three main characteristics: tungsten content, porosity percentage, and the grain size. The addition of tungsten to tantalum increased most mechanical properties, including hardness, compressive strength, yield strength, toughness, and resilience modulus, although ductility decreased by 10%.

1. Introduction

Refractory metals exhibit unique characteristics, including high melting points, exceptional strength at elevated temperatures, mechanical stability, and outstanding resistance to corrosion compared to other metals [1]. Among them, tantalum and its alloys are particularly significant in various industrial applications due to their high density and excellent formability, even at low temperatures. Because of these properties, tantalum is widely used in high-strain-rate applications [2-6]. Its combination of a high melting point, suitable

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https://doi.org/10.22099/IJMF.2025.51766.1309

elastic modulus, and excellent ductility at room temperature makes it valuable in power industries and aerospace engineering [7]. Notably, pure tantalum possesses mechanical properties similar to low carbon steel, with a relatively low elastic modulus of 186 GPa [8].

Tantalum and its alloys are primarily produced through casting, which results in a coarse-grained structure. However, despite this structure, tantalum exhibits remarkable ductility [7, 9]. Powder metallurgy has emerged as an alternative production method to achieve a dense tantalum microstructure optimized for broader applications [5, 10].

Despite its many advantages, the high cost of tantalum has limited its widespread use [2, 3]. As a result, several studies have focused on reducing production costs while simultaneously eliminating heterogeneous structures caused by casting or insufficient sintering time and temperature in powder metallurgy samples to enhance its applicability [4, 11].

Tantalum powder, due to its high melting point and low thermal conductivity, requires prolonged sintering at high temperatures. Additionally, tantalum has a strong affinity for interstitial elements such as oxygen and nitrogen, necessitating vacuum conditions and elevated temperatures during the metallurgical process [6, 12]. To maintain or enhance its high-temperature strength, tantalum is often alloyed with other elements. These alloying elements are categorized based on their atomic radius relative to tantalum into interstitial and substitutional elements. Tungsten, a substitutional element, increases the hardness of the solid solution at room temperature and enhances the mechanical properties of Ta-W alloys [13, 14]. Efe et al. [15] studied the dissolution and deposition of oxygen and carbon in pure tantalum powder under different oxygen concentrations after hot pressing. Other studies have explored the role of oxygen in structural development in tantalum powder metallurgy [16, 17]. Angerer et al. [18, 19] analyzed the microstructure and residual stress in hot-pressed pure tantalum samples. Additionally, previous research has employed a combination of cold isostatic pressing, electron beam melting (EBM), forging, and rolling to produce Ta-W alloys with improved mechanical properties under high-strain-rate deformation [20]. Favorable mechanical properties have also been reported in parts produced via powder metallurgy, where plasma pressure compaction was used for material consolidation [21].

In this study, the hot vacuum pressing method was employed to produce high-density tantalum parts with a controlled microstructure. The primary objective was to enhance the mechanical properties of tantalum, contributing to advancements and innovation in the application of refractory metals. To achieve this, the effect of tungsten addition to tantalum was investigated under hot pressing conditions.

2. Materials and Methods

This study investigates the microstructure and variations in mechanical properties of tantalum alloys influenced by tungsten content. Ta-W alloys with different tungsten concentrations were synthesized using pure tantalum and tungsten powders. The morphology of tantalum and tungsten powders was analyzed using scanning electron microscopy (Fig. 1). The average particle sizes of tantalum and tungsten powders were 1.44 and 1.582 µm, respectively. The powders were blended in a turbo mixer at 100 rpm for 40 minutes to produce Ta-W compounds with a mass ratio of 2.5 wt.% W, 5 wt.% W, and 7.5 wt.% W. Pure tantalum powder was also analyzed as a reference. Fig. 2 presents the powders after mixing.

A graphite mold was utilized to compact the samples, as schematically illustrated in Fig. 3, along with its dimensions. Graphite foil was used on the inner surfaces of the mold to prevent mold wear and chemical reaction with tantalum powder.

The mold and powder mixture were placed inside a hot press machine with a 15-ton hydraulic capacity, where hot compression was conducted under vacuum conditions. Table 1 provides the hot pressing parameters, while Table 2 outlines the test methods.

The hot-pressed specimens were cylindrical, with a diameter of 12.5 and a height of 10 mm. Their density was measured using the Archimedes method, following the ASTM B 527 standard.

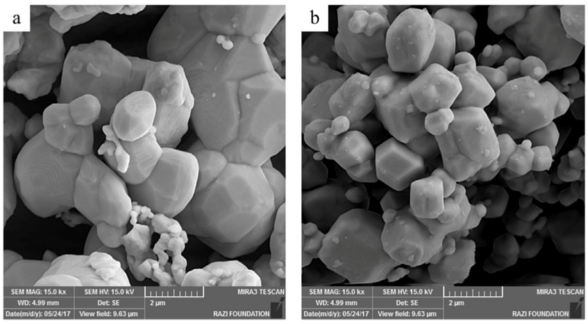

Fig. 1. Distribution of pure powders: (a) tantalum and (b) tungsten.

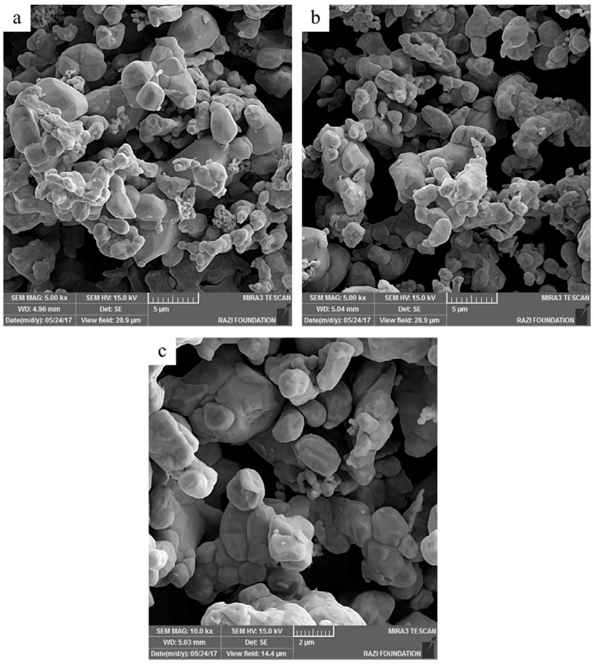

Fig. 2. Powder distribution for Ta-W alloys with varying tungsten content: (a) Ta-2.5 wt.% W, (b) Ta-5 wt.% W, and (c) Ta-7.5 wt.% W.

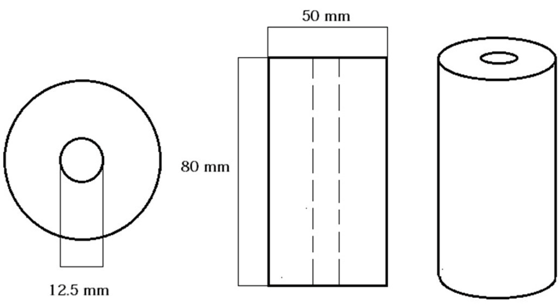

Fig. 3. Schematic representation of the graphite mold.

To study the microstructure, the samples were initially prepared and ground, followed by final polishing with diamond paste. Etching was performed for 30 seconds using a solution of H_2O: HNO_3 and HF: H_2SO_4 in a 1: 1: 1: 3 ratio. The microstructure was then examined with an optical microscope. Grain size was determined using microscopic image analysis software following the planimetric method as described in ASTM E112, by measuring more than 90 grains per sample to calculate the average grain size.

Vickers hardness was measured using 9.807 N loading in accordance with ASTM E384. To investigate the effect of tungsten on the mechanical properties of tantalum-tungsten alloys, compression tests were conducted at ambient temperature per ASTM E9 requirements. For these tests, samples were prepared with an initial height-to-diameter ratio (h_0/d_0) of 1.1 using a wire-cut machine, and the parallelism of the sample ends was ensured by grinding. The compression tests were performed using an Instron machine at a load speed of 1 mm/min.

Table 1. Hot pressing setup conditions

Mold diameter	12.5 mm
Powder in each sample	12 g
Pressure	40 MPa
Heating rate	100 °C/min
Vacuum	5×10^{-2} mbar
Thermocouple type	Type-R

Table 2. Specimen conditions

Sample	Temperature (°C)	Time (min)	Tungsten wt.%
1	1600	25	0
2	1600	25	2.5
3	1600	25	5
4	1600	25	7.5

3. Results and Discussion

One of the most critical physical properties that influence other characteristics is density. In this study, density is expressed as relative density, defined as the ratio of the measured porous density (obtained using the Archimedes method) to the theoretical density. The theoretical density of each alloy was calculated using Eq. (1), which is derived from the rule of mixtures [22]:

$$\rho_{th} = (\rho_{Ta} \times V_{Ta}) + (\rho_W \times V_W) \tag{1}$$

In the above equation, ρ_{Ta} and ρ_w represent the densities of tantalum and tungsten, respectively, while V_{Ta} and V_W are their corresponding volume fractions. Fig. 4 shows the variation of relative density with the weight percent of tungsten.

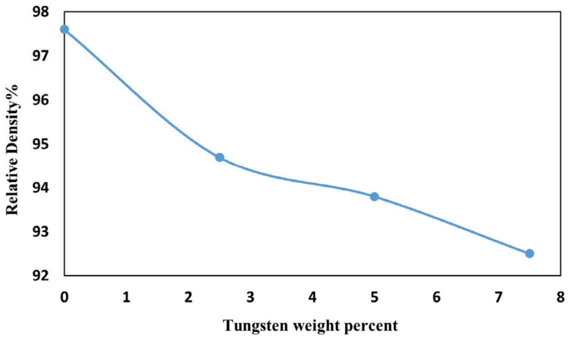

Fig. 4. Variation of relative density with tungsten weight percent.

The results showed that pure tantalum achieved a relative density of 97.6%. However, with the addition of tungsten up to 7.5 wt.%, the relative density decreased by approximately 5 wt.%, reaching 92.5%. Although tantalum and tungsten have densities of 16.6 and 19.25 g.cm^{-3}, respectively, one might expect that adding tungsten would increase the alloy's density relative to pure tantalum. Instead, the observed decrease in relative density with increasing tungsten content can be attributed to tungsten's low ductility as an additive. The incorporation of tungsten reduces the plasticity of the particles during compression, which prevents pores from being completely eliminated and ultimately lowers the overall density. Additionally, tungsten's higher melting temperatures of 3422 °C compared to 2996 °C for tantalum may slow atomic diffusion during simultaneous compression and sintering, causing pores to persist or change very little in size. Consequently, Ta-W samples with higher tungsten content exhibit lower densities than pure tantalum.

Fig. 5 shows the microstructures of specimens after hot pressing. Grains, grain boundaries and porosities are clearly obvious. As shown, at higher content, the grains become smaller (for example, compare Fig. 5(b) and 5(d)); however, increased porosity is also observed. Porosities are primarily located along grain boundaries, although some porosities are evident within the grains.

The microstructure examinations revealed that the grain size in Ta-W alloys decreased with increasing tungsten content. Fig. 6 shows the average grain size of samples with varying tungsten weight percentages. The addition of tungsten to tantalum causes tungsten atoms to aggregate at the tantalum grain boundaries, thereby inhibiting grain growth [20]. Consequently, as indicated in Fig. 6, the average grain size decreased from 17.3 µm to 7.2 µm as the tungsten content increased from 0 wt.% to 7.5 wt.%.

Another contributing factor is the formation of a Ta-W solid solution. Since tantalum and tungsten are completely soluble in solid-state, tungsten atoms can substitute into the tantalum lattice, creating a strain field that reduces grain boundaries or dissolves in a solid solution. The addition of tungsten effectively restricts grain boundary movement, resulting in smaller grains.

To calculate the percentage of porosity in the samples, Eq. (2) was used based on the density [23]:

$$P = 1 - \frac{\rho}{\rho_0} \tag{2}$$

In this equation, P is the percentage of porosity, ρ is the density of the porous material, and ρ_0 is the theoretical density.

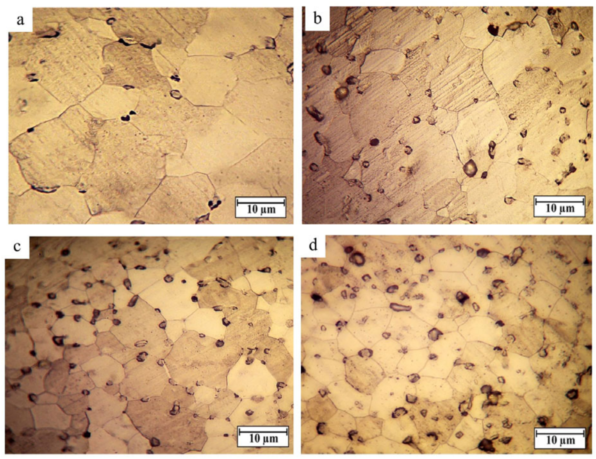

Fig. 5. Microstructures of hot-pressed samples: (a) pure tantalum, (b) Ta-2.5 wt.% W, (c) Ta-5 wt.% W, and (d) Ta-7.5 wt.% W.

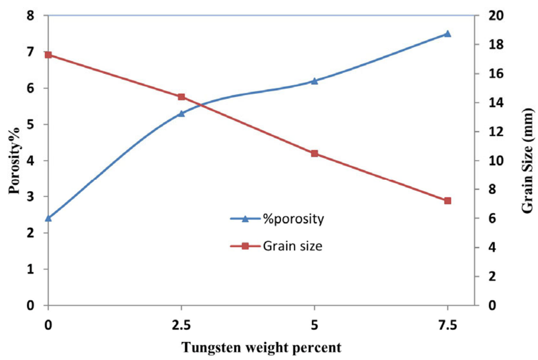

Fig. 6. Average grain size and porosity % influenced by tungsten weight percent.

Fig. 6 also shows the variations of porosity with tungsten; as the tungsten content increases, the porosity in the microstructure rises from 2.4% to 7.5%.

As previously discussed, adding tungsten powder to tantalum increases the concentration of solute atoms in solid solution while simultaneously increasing porosity fraction and decreasing the grain size (Fig. 6). These three parameters—tungsten content, grain size, and porosity—affect the overall properties of hot-compressed Ta-W samples, but often in opposing ways. In other words, while one factor may enhance a given property, another may detract from it. Consequently, these factors compete with each other in determining the final properties of the material and their influence may vary depending on the specific property under consideration.

Fig. 7 shows that the hardness of the samples increases with tungsten content. For example, pure tantalum exhibits a hardness of 345 HV, whereas the Ta-7.5 wt.% W alloy reaches 511 HV—an increase of approximately 166 HV. This hardness enhancement can be attributed to several factors. First, tungsten content raises the number of solute atoms in solid solution which creates a strain field that impedes dislocations motion. Second, as shown in Fig. 6, the grain size decreases with increasing tungsten; resulting in a higher grain boundary area per unit volume. Grain boundaries act as barriers to dislocations movement due to the difference in crystallography orientation between adjacent grains, leading to further hardening. On the other hand, increased porosity generally decreases hardness by reducing the effective load-bearing cross-section during indentation. Despite this, the combined effects of grain refinement and solute strengthening appear to dominate, as evidenced by the overall increase in hardness observed in Fig. 7. Thus, the beneficial effects of a reduced grain size and increased solute atom concentration outweigh the detrimental impact of increased porosity on the hardness of hot-compressed Ta-W samples in different manner. It will decrease hardness by decreasing the resisting material against deformation due to indentation during hardness test. According to above results that have been observed in Fig. 7, it can be said that the effect of grain size and solute atom on hardness are more than porosity fraction.

Fig. 8 shows the true stress-true strain diagrams obtained during the compression tests of samples with different tungsten contents, while the corresponding mechanical properties extracted from these diagrams are presented in Fig. 9.

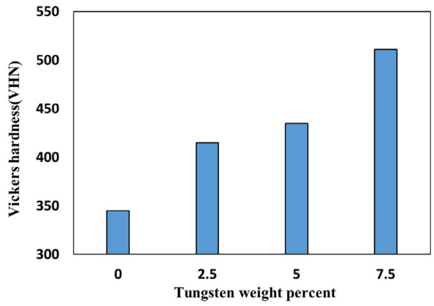

Fig. 7. Hardness variation with tungsten weight percent.

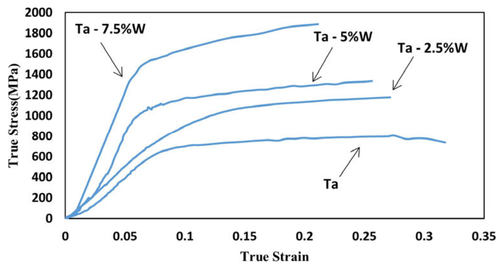

Fig. 8. True stress-true strain diagram.

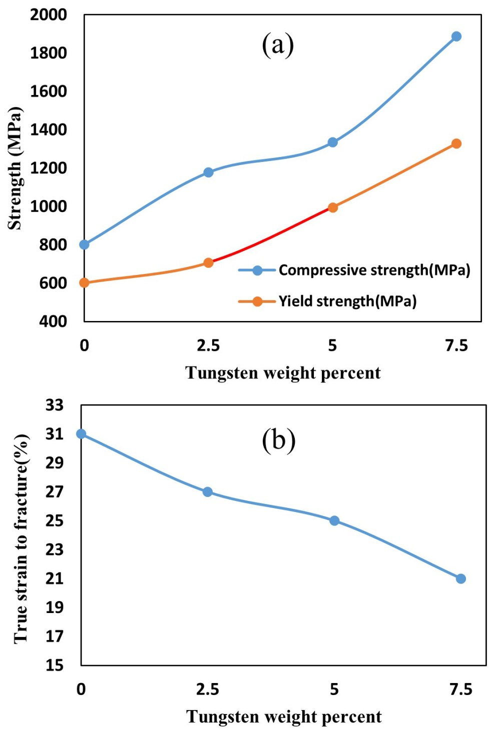

Fig. 9. Variation of mechanical properties with tungsten weight percent: (a) compressive and yield strength and (b) true strain to fracture (%).

As indicated in Fig. 9, the addition of 7.5 wt.% tungsten to tantalum reduced the true strain to fracture from 31% to 21% corresponding to a 10% decrease in ductility. This reduction in ductility is primarily attributed to an increase in the porosity percentage (see Fig. 6). Porosity acts as a site for stress concentration; therefore, an increase in porosity intensifies stress concentration and consequently diminishes ductility. Additionally, as shown in Fig. 6, grain size decreases from 17.3 μm to 7.2 μm (~ 58% reduction), further limiting dislocation slip and reducing ductility. The presence of solute tungsten atoms also contributes to this effect by impeding dislocation motion. Therefore, it can be concluded that all three factors—tungsten content (solute atoms), porosity percentage, and reduced grain size—collectively contribute to the observed decrease in ductility.

As can be seen, adding 7.5 wt.% tungsten to pure tantalum increases the compressive strength from 802 MPa to 1887 MPa, and the yield strength (calculated using the 0.2% offset criterion) rises from 602 MPa to 1328 MPa. This corresponds to an increase of 120% in yield stress and 135% in compressive strength indicating that tungsten has a slightly greater effect on compressive strength than on yield strength. The enhanced strength can be attributed to both an increased concentration of solute tungsten atoms and a reduction in grain size (Fig. 6). Adding tungsten to tantalum lowers the stacking fault energy (SFE), making cross-slip more difficult and thereby increasing strength. Moreover, the significant difference in shear modulus between tantalum (160 GPa) and tungsten (69 GPa) [13-14] means that tungsten atoms locally alter the shear modulus of tantalum, increasing the dislocation strain field of energy, and reducing dislocations mobility; as a result, a higher stress is necessary for dislocation glide. Additionally, the reduced grain size contributes to strengthening, as summarized by the Hall-Petch equation:

$$\sigma_{ys} = \sigma_i + kd^{-1/2} \quad (3)$$

In this equation, σ_{ys} is the yield strength, σ_i is the frictional stress (influenced by the presence of solute atoms), k is the Hall–Petch constant, and d is the average grain size [24]. Thus, with an increase in frictional stress (solute atoms), and a decrease in grain size, the yield stress increases.

Conversely, as shown in Fig. 6, increasing tungsten content also raises the porosity fraction. Porosity reduces the effective cross-sectional area under load and create sites for stress concentration, which can promote crack growth and decrease strength. Overall, the strengthening effects of a reduced grain size and increased solute atom concentration outweigh the weakening impact of increased porosity, as evidenced in Fig. 9.

Two additional mechanical properties of importance in certain applications are toughness and modulus of resilience, both of which were determined in this study.

Toughness was calculated according to Eq. (4), which represents the area under the stress-strain curve:

$$\frac{W}{V} = \int_0^{\varepsilon_f} \sigma d\varepsilon \quad (4)$$

W/V represents the energy per unit volume, where σ is stress, and ε_f is strain at failure [24].

The ability of a material to absorb energy during elastic deformation is measured usually by modulus of resilience. The resilience modulus, U_R, was calculated from the compression test results using Eq. (5):

$$U_R = 0.5\, \sigma_y \varepsilon_y \quad (5)$$

U_R is modulus of resilience, while σ_y and ε_y are yield strength, and strain at yield stress, respectively [25].

Fig. 10 presents the results for toughness and the modulus of resilience as a function of tungsten content. With the addition of 7.5% tungsten to tantalum, the toughness increases from 215 $J.m^{-3}$ to 300 $J.m^{-3}$, and the resilience modulus rises from 21 $J.m^{-3}$ to 35 $J.m^{-3}$. This corresponds to an approximate increase of 39% in toughness and 67% in the resilience modulus. In other words, tungsten has a more significant effect on elastic behavior (modulus of resilience) than plastic behavior (toughness).

Elastic behavior is primarily determined by atomic bonding, whereas plastic behavior is influenced by dislocation mobility and grain boundaries. Therefore, tungsten's effect on strengthening appears to stem more from its influence on atomic bonding than from its role in impeding dislocation glide. Since tungsten is completely soluble in tantalum, it acts as a solute atom that decreases the stacking fault energy, thereby hindering cross-slip and reducing deformability. Consequently, higher loads and stresses are required for deformation, leading to more pronounced work hardening with increasing tungsten content. Moreover, the combined effects of solute strengthening and grain refinement on toughness and the modulus of resilience seem to outweigh the adverse influence of increased porosity, which generally tends to reduce these properties.

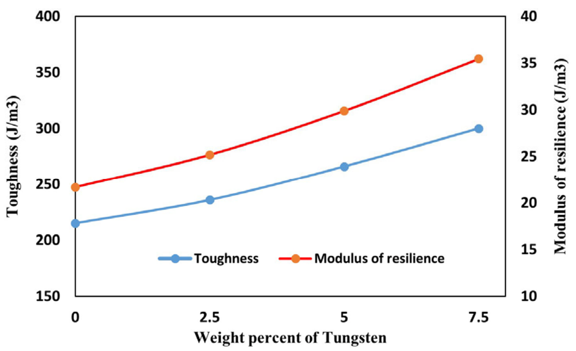

Fig. 10. Toughness and modulus of resilience with tungsten weight percent.

4. Conclusions

This study aimed to enhance the mechanical properties of tantalum using tungsten addition, thereby fostering development and innovation in the use of refractory metals. The effects of tungsten addition on the microstructure and mechanical properties of tantalum by hot pressing were thoroughly investigated. The key findings of this research are as follows:

1. With tungsten addition up to 7.5 wt.%, the relative density of tantalum decreased from 97.5% to 92.5%.
2. Tungsten addition significantly reduced the grain size and increased the porosity, which in turn affected other material properties.
3. Hardness, compressive strength, yield strength, toughness, and modulus of resilience all increased with tungsten addition, while ductility decreased by 10%.
4. The results indicate that tungsten has a greater effect on the elastic behavior of tantalum than on its plastic behavior.

Conflict of interest

The authors declare no conflict of interest.

Funding

The authors did not receive funding to conduct the research.

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