Study on microstructure and mechanical properties of W-Ni₃Al alloys prepared by ball milling and two-step sintering

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Abstract

In this paper, W-Ni₃Al alloys with the different Ni₃Al contents were prepared via ball milling and two-step sintering processes. The relationship of Ni₃Al contents, microstructural evolution, and mechanical properties of W-Ni₃Al alloys were also clarified. The results show that the milled powder has a lamellar structure and cold welding phenomenon. This structure encloses a small amount of ethanol, and this causes an Al₂O₃ phase to form when it undergoes an oxidizing reaction of Ni₃Al with oxygen in ethanol during ball milling and drying. The W-rich phase is the network structure, and this structure is finer and denser with an increase in Ni₃Al contents as a result of the grain refining effect of Al₂O₃ particles and the densification effect of liquid sintering. Under a load, the Al₂O₃ particles at the interface of W/binder phases increase the resistance of crack growth and compressive strain, which improves the mechanical properties and deformability of W-Ni₃Al alloys. The Ni₃Al binder provides a new strategy for improving the mechanical properties of tungsten alloys.

1. Introduction

Tungsten alloys are used as rocket nozzles, die-casting molds, bullet cores, contacts, and heating elements because of their high density, high melting points, high-temperature strength, and good conductivity of heat and electricity [1–4]. Cu, Fe, Co, Ni, and Mn are binders commonly used in tungsten alloys to reduce sintering temperature, shorten densification time, and enhance comprehensive mechanical properties [5–8]. However, the addition of these binders deteriorates the hardness, corrosion resistance, and high-temperature strength of the alloys. To overcome these disadvantages, doping minute amounts of Y₂O₃, ZrO₂, and TiC or a substitute binder phase is an approach that has been attempted [9–12]. Ni₃Al can be used as a binder candidate because of its good corrosion resistance, high hardness, and high-temperature strength [12]. Thus, application of Ni₃Al as a binder in WC cemented carbides has been successful. Zhang et al [13] studied the microstructure and mechanical properties of WC-10Ni₃Al cerments after compression in the temperature range of 650 °C–900 °C; the results show that the Ni₃Al binder phase has anomalous mechanical properties at high temperature, improving the ability of the cerments to resist stress at high temperature. This effect is coordinated with plastic deformation of WC, enhancing the high temperature strength of the WC-10Ni₃Al alloy. Li et al [14] investigated the corrosion behavior of the WC-10Ni₃Al alloy via a comparison with the WC-8Co alloy in strong acid at room temperature. They found that Ni dissolution is the main corrosion mechanism, indicating that the WC-10Ni₃Al alloy has better corrosion resistance. In our earlier works [15], the effects that substituting Ni with Ni₃Al has on phase, microstructure, and properties of W-6Ni-4Co alloys were evaluated. The results show that Ni₃Al mainly reacts with Co, restraining some brittle phases as NiW and NiW₂ from forming. Ni₃Al also increases the densification parameter compared with W-6Ni-4Co alloys at lower sintering temperature. Therefore, Ni₃Al is also a promising binder for tungsten alloys.

To further study the effects that Ni₃Al full substitution has on microstructure and properties of W-Ni₃Al alloys, W-Ni₃Al alloys with different Ni₃Al contents were prepared in this paper using ball milling and two-step
sintering processes. The relationships of Ni$_3$Al contents, microstructural evolution, and mechanical properties of W-Ni$_3$Al alloys were also clarified.

2. Materials and methods

W and Ni$_3$Al powders with the same characteristics are reported in [8] and [15] were used as raw materials in this paper. Alloy powders with Ni$_3$Al contents of 2 wt. %, 4 wt. %, 6 wt. %, and 8 wt. % were ball mixed for 25 h under argon atmosphere using a planetary ball mill (XQM-0.4 A) with YG-cemented carbide balls and jars with a rotation speed of 510 rpm and a ball-to-powder ratio of 6:1. To disperse and refine the powder more adequately, balls with diameters of 10 mm, 8 mm, and 5 mm were used with a ratio of 1:2:6. Moreover, ethanol (CH$_3$CH$_2$OH) was used as a milling suspension with a powder-to-suspension ratio of 1:0.45. The milled powders were ground after vacuum drying to avoid agglomeration, and then a uniaxial press was used to press the powders under 476 MPa into cuboid samples with dimensions of 30 mm $\times$ 7 mm (width) $\times$ 3.5 mm (height). Finally, bulk W-Ni$_3$Al alloys were obtained via a two-step sintering process in a vacuum tubular furnace (GSL-1700X-II) in an atmosphere of flowing Ar at 20 ml min$^{-1}$. The two-step sintering process included solid phase sintering and liquid phase sintering, and the temperature and soaking time were 1250 $^\circ$C for 30 min and 1500 $^\circ$C for 60 min, respectively. The samples were heated and cooled inside the furnace with average heating and cooling rates of 5 $^\circ$C min$^{-1}$. The sintered samples were wet polished and then cloth polished using metallographic sandpapers and Al$_2$O$_3$ suspension to a size of 25 mm $\times$ 6 mm $\times$ 3 mm for testing the microstructure and mechanical properties.

To further analyze the microstructural evolution and mechanical properties, a scanning electron microscope (Scios, FEI and JSM-6390LV) was used to observe micrographs and to analyze the element distributions of the two-step sintered samples. The volume fraction of the second phase ($V_f$) was estimated using the ratio of the area of the second phase to the area of the entire microstructure in SEM micrographs. The relative area ($A_f$) of the second phase can be calculated using the weight method. Using the equations $A_f = G_f/G_G$ and $V_f = A_f$, where, $G_f$ and $G_G$ are the weights of the areas of the second phase and the entire microstructure, respectively. In SEM images, the weight of microstructure was measured by electronic balance. The the volume fraction of the second phase can be estimated [16].

A transmission electron microscope (TEM, Tian ETEM G2) was used to determine the ultimate microstructure of the W-6Ni$_3$Al alloy. The phase structure was characterized using an X-ray diffractometer (D/max-2500/PC). The average hardness of the W-Ni$_3$Al alloys was estimated using a microhardness tester (HVS-1000) under loads of 100 g for 20 s, with five testing points taken at the different locations for each sample. A mechanical tester (MP-200) was used to measure the compressive strength and flexural strength of the sintered samples under loading rates of 0.08 mm min$^{-1}$ and 0.5 mm min$^{-1}$, respectively.

The density of cold pressed compacts and sintered samples were obtained using the Archimedes principle. Before testing, samples were wax-sealed to prevent liquid from getting into the pores of the samples. The densification parameter was calculated as follows [8]:

$$D = \frac{\rho_S - \rho_G}{\rho_T - \rho_G}$$  

where $D$ is the densification parameter of W–Ni$_3$Al alloys with different Ni$_3$Al contents prepared via the two-step sintering process. $\rho_S$, $\rho_G$, and $\rho_T$ are the sintered density, green compact density, and theoretical density, respectively.

3. Results and discussion

3.1. Characterization of W–6Ni$_3$Al milled powders

We present the W–6Ni$_3$Al milled powders as an example because of the similar characteristics that the milled powders with different Ni$_3$A contents have. Figure 1 shows SEM images and EDS line scans of W–6Ni$_3$Al milled powders. A lamellar structure is observed, and each tablet was layered. Therefore, it can be deduced that plastic deformation of tungsten powder with a polyhedron shape and Ni$_3$Al with an irregular morphology are present in the lamellar structure. Moreover, the cold welding phenomenon is also observed on the basis of the close bonding of the interface, and this can be verified by the EDS lines, which indicate obvious element diffusion (see the yellow double-headed arrow in figure 1). This can be attributed to the cyclic effect of plastic deformation $\rightarrow$ strain hardening $\rightarrow$ fracture $\rightarrow$ cold welding $\rightarrow$ secondary fracture $\rightarrow$ cold welding $\rightarrow$ third fracture, and so on during high energy ball milling process [17]. Next, we will analyze and discuss the microstructural evolution process of the above prealloy powder under one-step sintering and two-step sintering.
3.2. Phase evolution of W-Ni3Al alloys

To study the phase transition from milled powders to the sintered bulk, XRD patterns of milled powders and the bulk prepared via one-step sintering (solid phase sintering) and the bulk with different Ni3Al contents prepared via two-step sintering are shown in figure 2. The peaks were identified using Jade 6 software. The main phases in the milled powders and sintered bulk are W (BCC, Im3m), Al3Ni (Orthorhombic, Pnma), Ni3Al (Tetragonal, P4/mmm), and α-Al2O3 (Trigonal, R-3c). In addition, the α-Al2O3 phase was not found in the W–Ni–Co and W–Ni–Cu alloys in previous studies [8, 15], and this indicates the oxidation of mixing powders during ball milling and two-step sintering processes. A vacuum and subsequent argon atmosphere can be used to isolate oxygen from the atmosphere under a vacuum degree of 10⁻¹ Pa and 99.5% pure argon. Thus, the oxygen source of the α-Al2O3 phase can only come from ball-suspension (CH₃CH₂OH). This is because oxygen in the organic solvent can be absorbed into the mixed powders via ball-milling when ethanol is used as a ball-milling suspension, and this is similar to that of WC-Ni3Al alloys in [18]. Hence, we estimated the phase composition to help in studying the phase transition.
process from milled powders to the sintered bulk using equation (2) for simplicity, and the results are listed in table 1.

\[ V_i = \frac{A_i}{\sum_{i=1}^{n} A_i} \% \]  

(2)

where \( A_i \) is the area of the diffraction peaks of the W, Al\(_3\)Ni, and \( \alpha\)-Al\(_2\)O\(_3\) phases; \( V_i \) is the volume fractions of the W, Al\(_3\)Ni, and \( \alpha\)-Al\(_2\)O\(_3\) phases. The volume fractions of the Al\(_3\)Ni and \( \alpha\)-Al\(_2\)O\(_3\) phases are 0.09\% and 0.2\% in the W-6Ni\(_3\)Al milled powders, and this indicates the oxidizing reaction of Ni\(_3\)Al with CH\(_3\)CH\(_2\)OH during ball milling and the drying processes. By comparison, the volume fractions of the \( \alpha\)-Al\(_2\)O\(_3\) phase in the W-6Ni\(_3\)Al alloy that was sintered via one-step sintering (solid phase sintering) increased to 0.5\% because of the continuous oxidizing reaction of Ni\(_3\)Al with the remaining CH\(_3\)CH\(_2\)OH in the milled powders after drying. Moreover, there was a further increase in the \( \alpha\)-Al\(_2\)O\(_3\) phase to 0.66\% in the W-6Ni\(_3\)Al alloy that was sintered via two-step sintering, and this confirms that there was residual oxygen in the W-6Ni\(_3\)Al alloy that was sintered via one-step sintering. Also, the volume fractions of Al\(_3\)Ni and \( \alpha\)-Al\(_2\)O\(_3\) phases increased with Ni\(_3\)Al content. This also demonstrates the oxidizing reaction of Ni\(_3\)Al with CH\(_3\)CH\(_2\)OH during ball milling, drying, and two-step sintering processes. Next, we will discuss the effects that the Al\(_3\)Ni and \( \alpha\)-Al\(_2\)O\(_3\) phases have on the microstructural evolution of the W-Ni\(_3\)Al alloy that was sintered via two-step sintering.

### 3.3. Microstructural evolution of W-Ni\(_3\)Al alloys

Figure 3 shows the microstructure and EDS map of the W-6Ni\(_3\)Al alloy that was sintered via one-step sintering. Light gray indicates the W-rich phase, dark gray indicates the binder phase, and there is a black strip phase. A network structure is composed of the W-rich phase and binder phase. The black strip phase is mainly distributed inhomogeneously, as seen in figure 3(a), and these areas are identified as regions that are rich in Al and O, according to the EDS map shown in figure 3(b). The distribution of the W-rich phase is strip-type in these inhomogeneous areas, and this is similar to the lamellar structure of milled powder, as seen in figure 1. Thus, it can be verified that the oxygen came from the ethanol that was wrapped by the lamellar structure of milled powders, which was assumed above. Figures 4 and 5 show microstructures and EDS maps of W-Ni\(_3\)Al alloys with the different Ni\(_3\)Al contents that were produced via two-step sintering. Compared with the W-6Ni\(_3\)Al alloy that was sintered via one-step sintering, the distribution of the \( \alpha\)-Al\(_2\)O\(_3\) phase in the alloy that was sintered via two-step sintering is more uniform, and the W-rich and binder phases retain a network structure. Also, their grain sizes increase from 1.33 \( \mu \)m to 5.15 \( \mu \)m, as measured using the intercept method, and this resulted in liquid flow from the Al\(_3\)Ni phase during the two-step sintering process (figures 4(c) and 5(c)). The oxygen content in the W-6Ni\(_3\)Al alloy that was produced via two-step sintering also increased from 4.57 wt.% to 5.09 wt.%
Figure 4. SEM images of W-Ni$_3$Al alloys with the different Ni$_3$Al contents sintered via two-step sintering: (a) W-2Ni$_3$Al alloy, (b) W-4Ni$_3$Al alloy, (c) W-6Ni$_3$Al alloy, (d) W-8Ni$_3$Al alloy.

Figure 5. EDS maps of W-Ni$_3$Al alloys with the different Ni$_3$Al contents sintered via two-step sintering: (a) W-2Ni$_3$Al alloy, (b) W-4Ni$_3$Al alloy, (c) W-6Ni$_3$Al alloy, (d) W-8Ni$_3$Al alloy.
(figures 3(b) and 5(c)), indicating that there was a continuous oxidizing reaction of Ni$_3$Al and remaining oxygen in the alloy that was produced via one-step sintering. The Ni$_3$Al content also has a significant impact on the microstructural evolution of W-Ni$_3$Al alloys that were produced via two-step sintering. Figure 4(a) shows the microstructure of the W-Ni$_3$Al alloy with 2 wt.% Ni$_3$Al content. Two types of Ni-rich phases were observed, as shown in figures 4(a) and 5(a), and their Ni contents were 22.17 wt.% for the darker one and 28.24 wt.% for the lighter one, as indicated by EDS point analysis. However, this effect on Ni-rich phases became weaker with an increase in the Ni$_3$Al content from 2 wt.% to 6 wt.%(figures 4(a)–(c) and figures 5(a)–(c)). This is because liquid fluidity from Al$_3$Ni and residual Ni$_3$Al phases (not detected by XRD) are enhanced with an increase in binder phase content, improving the Ni-rich phase uniformity. Also, liquid flow cracks are clearly observed in the W-rich phase, as seen in enlarged view of figure 4(c). In this case, the α-Al$_2$O$_3$ phase was also uniformly distributed in the interface of Ni-rich and W-rich phases. This can refine grain size of the W-rich phase from 14.23 μm in the W-2Ni$_3$Al alloy to 7.11 μm in the W-4Ni$_3$Al alloy and to 5.15 μm in the W-6Ni$_3$Al alloy; this has an inhibiting effect on solution precipitation of W, according to Ostwald ripening theory [19]. The average grain size of the W-8Ni$_3$Al alloy was the smallest (4.46 μm) because of it had the highest Ni$_3$Al content; however, the Ni-rich phase was again nonuniform, and this was caused by flow inhibiting effect of the binder phase that resulted from an excess α-Al$_2$O$_3$ phase, as seen in figure 5(d). Thus, the α-Al$_2$O$_3$ phase has an important impact on microstructural evolution of W-Ni$_3$Al alloys. In addition, the values of $V_{c_1}$ for the α-Al$_2$O$_3$ phase of the alloys with Ni$_3$Al contents from 2% to 8% are 0.26%, 0.55%, 0.62% and 0.29%, respectively. A sum of $V_{c_2}$ for Al$_3$Ni and Ni$_3$Al phases can be obtained, not alone because it is difficult to identify both of them. The corresponding values of $V_{c_2}$ for Al$_3$Ni and Ni$_3$Al phases are 0.05%, 1.17%, 1.64% and 3.32%, respectively, and these are consistent with XRD results.

To further study the arrangement of the binder and W-rich phases in detail, TEM analysis was also conducted, as shown in figure 6. Figures 6(c)–(e) is the selected area diffraction pattern that corresponds to the white phase, gray phase, and that phase with black stripe in figures 6(a), (b). The white phase, which has a polygonal shape with cell parameters of $a = b = 4.73$ Å and $c = 12.711$ Å, is the α-Al$_2$O$_3$ particle phase which
is consistent with that of XRD, and it is mainly distributed in the interface of two other phases; it restrains grain growth of the matrix phase, and this restraint is more efficient with an increase in the Al2O3 phase (figure 4). The gray phase and the phase with the black stripe are Al3Ni and W-rich phases, respectively. The black stripe in the W-rich phase is crisscrossed and dense, which indicates that there is a lot of residual internal stress in the W-rich phase. This is because microstructural defects, such as dislocation and faults caused by ball milling, cannot be completely eliminated after two-step sintering, and this further confirms the blocking effect on solution precipitation of W in the binder phase. This causes incomplete recovery and recrystallization of a deformed W phase during two-step sintering.

To compare the densification evolution of W-Ni3Al alloys, figure 7 shows a histogram of the densification parameter of W-Ni3Al alloys with different Ni3Al contents. Obviously, the densification parameter increased with an increase in Ni3Al content. This can be proven from the microstructural evolution that occurred with different Ni3Al contents, as described above. The amount of liquid phase increased with an increase in Ni3Al content because of its low melting point (1293 °C), which added the flow region of the liquid phase (figures 4(c), (d) and matrix color evolution of EDS maps in figure 5). Therefore, body shrinkage was accelerated, improving the densification parameter of the W-Ni3Al alloys.

3.4. Mechanical properties of W-Ni3Al alloys

Table 2 shows the microhardness (HV), bending strength (σbb), and yield strength (σ0.2) of W-Ni3Al alloys with different Ni3Al contents. The yield strength (σ0.2) and strain (ε) were calculated using the true stress-strain curves from the compressive test in figure 8. The hardness of the alloy with 2 wt.% Ni3Al content was the lowest (434 HV), and this increased to 512 HV with an increase in the Ni3Al contents from 2 wt.% to 6 wt.%. This is because the densification parameter and grain size are the main factors that influence resistance to the deformation of alloys, namely, it has higher hardness. However, the hardness decreases with an increase in Ni3Al content to 8 wt.% because there is an excess of Al2O3 phase (figure 2 and figure 5(d)), and this weakens the interface of W/W and W/binder phases. The trends of σbb and σ0.2 are similar to that of microhardness. We also organized the data of the densification parameter, grain size, Al2O3 phase content, and σ0.2 to verify the deductions above; these data are shown in figure 9. The results show that microstructural evolution (such as phase transition and grain growth) are closely related to Ni3Al contents, and the acquired microstructural characteristic has an important impact on the mechanical properties of W-Ni3Al alloys (see curve trend in figure 9). Strain is used to characterize the deformability of alloys. With an increase in Ni3Al contents, the strains of the alloys first increased and then decreased, and the alloy with 4 wt.% Ni3Al content has the largest strain of 30.16%.

The fracture morphology of W-Ni3Al alloys was observed using SEM to analyze the influence of compressive strain, and the results are shown in figure 10. There were main W-W grain boundary separation (I) and W-particle grain boundary separation fracture modes (II). According to the microstructure characteristics of W-Ni3Al alloys shown in figures 4–6, pits and humps on the W-rich phase result from the Al2O3 particles being distributed in the interface of the W-rich phase. Al2O3 particles can rotate during compressive testing, and such rotations deflect and disperse stress, relieving local stress concentration. This increases the resistance of crack growth and compressive strain [20]. However, this strengthening effect is weakened or even reversed because of
aggregation of $\text{Al}_2\text{O}_3$ particles. In this case, $\text{Al}_2\text{O}_3$ particles become the source of cracks, decreasing compressive strain. This phenomenon is observed in the fracture morphology of the W-8Ni$_3$Al alloy, as shown in figure 10(d), and this causes the lowest compressive strain (9.97%). Thus, the microstructural evolution of W-Ni$_3$Al alloys has an important impact on their mechanical properties.

4. Conclusions

W-Ni$_3$Al alloys with different Ni$_3$Al contents were prepared via ball milling and two-step sintering. The phase transition, microstructural evolution, and corresponding mechanical properties were investigated. The conclusions are as follows:
A lamellar structure and cold welding are observed in milled powder that is produced via ball milling, and a small amount of ethanol is wrapped in the milled powder. This causes an Al₂O₃ phase to form via oxidizing reaction of Ni₃Al with oxygen in ethanol during ball milling and drying. The content of this phase increases with an increase in Ni₃Al contents during the subsequent two-step sintering process, and this promotes grain refinement.

The alloy with a network structure of the W-rich matrix phase fine grains (4.46 μm) and that is densely compact (98.66%) achieves high hardness (512 HV), bending strength (572 MPa), yield strength (1238 MPa), and strain (30.16%). It is proven that grain refinement and interface strengthening by the Al₂O₃ phase and the dense compactness make significant contributions to improving the mechanical properties of W-Ni₃Al alloys. However, there is an adverse effect when there is an excess of Al₂O₃ content. Moreover, the relationships of Ni₃Al contents, microstructural evolution, and mechanical properties are clarified.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
Author contributions

Conceptualization and data curation, Bobo Liu; funding acquisition, Hongfeng Dong; methodology, Hongfeng Dong; writing original draft, Bobo Liu; writing, review, and editing, Hongfeng Dong, Peiyou Li, Taotao Ai and Wenhu Li.

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