Study on increasing density of sintered W-1.5Ni alloy by Al-Cu-Zn alloy infiltration

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ASBTRACT

This paper investigates the densification process of W–1.5%Ni composite mixtures prepared by pressing and sintering methods, achieving an initial relative density of 77–80% to form a porous W skeleton. The isothermal sintering was conducted in a graphite tube furnace at 1700 °C for 3 hours under an Ar protective atmosphere. Nickel (1.5%) was employed as a sintering activator. Subsequently, the remaining pores were filled by infiltration with a 50%Al–30%Cu–20%Zn liquid alloy at 1000 °C for 1 hour. The Al–Cu–Zn alloy infiltration technique proved to be effective in reducing the porosity of the sintered structure and increasing the overall density by approximately 3–5%, thereby enhancing the mechanical properties and thermal stability of the sintered composite. creating a composite material for use as a heat sink material.

Keywords: Tungsten sintering; Alloy infiltration for porous W frame; Al-Cu-Zn alloy infiltration; Activated sintering.

1. INTRODUCTION

Tungsten composite materials consist of a group of immiscible elements, including W and the elements Cu, Ni, Al, and Zn, with a tungsten matrix and dispersed alloy particles. These materials have a wide range of technical applications in the mechanical and electrical fields, including high-voltage contacts, welding electrodes, high-temperature nozzles, radiators, as well as components for weapons and heat sinks in nuclear reactors. They combine excellent corrosion and erosion resistance, good thermal and electrical conductivity, low coefficients of thermal expansion, and good mechanical properties. However, the fabrication of these materials is limited by the complex shapes and the internal distribution of interphases [1–3].

To fabricate these materials, the powder metallurgy (PM) methods is chosen. Due to the large differences in melting temperature and the poor or negligible mutual solubility of Ni, Cu, Al, and Zn with W in both liquid and solid states, the fabrication of such composite materials by conventional processes such as casting or solid-state deformation is challenging. Therefore, pressing and sintering techniques used to create porous W skeletons are preferred to provide initial structural integrity for the material. These skeletons contain both interconnected and isolated pores in specific proportions. The material's density, mechanical properties, and electrical and thermal conductivities are subsequently enhanced by infiltrating a molten alloy into the pre-sintered W skeleton [1].

Activated sintering of tungsten (W) has been used to produce W skeletons. Small amounts of transition metals such as Ni act as sintering activators that reduce the activation energy of sintering, allowing lower sintering temperatures compared to conventional sintering (>2000 °C), while enhancing the diffusion rate of W atoms across grain boundaries in close contact. Owing to this effect, sintering temperatures of around 1700 °C can be employed to fabricate W skeletons. Activated sintering promotes the formation of open, interconnected pores that facilitate liquid metal infiltration, thereby increasing density and improving the overall properties of the composite [4–8].

Sintering activators (e.g. Pt, Pd, Ni, Co, Fe) are typically added in small amounts. These elements enhance atomic diffusion and interparticle bonding during low-temperature sintering by improving the wetting of W grain boundaries. In the case of Ni, several studies have demonstrated that sintering can occur below its melting point and still yield a W skeleton. However, due to the limited mutual solubility and poor wettability of liquid Ni with W, liquid-phase sintering is often accompanied by gravity-induced segregation. When the solid-state sintering temperature approaches or exceeds the melting point of Ni in systems with high W content, it becomes difficult to achieve full densification of the pressed powder, even when high-pressure-assisted sintering techniques are applied [1, 4, 5, 9, 10].

Due to the large differences in the melting temperatures of W, Ni, Cu, Al, and Zn (3425 °C for W, 1455 °C for Ni, 1085 °C for Cu, 660 °C for Al, and 419.5 °C for Zn), and because the melting point of W exceeds the boiling points of Ni (2913 °C), Cu (2595 °C), Al (2519 °C), and Zn (907 °C), it is almost impossible to form a liquid-phase bonding between W and these metals while the W phase remains solid. When infiltrating a Cu–Zn–Al alloy, pre-wetting the W skeleton with Ni significantly improves the ability of the molten Cu, Zn, and Al mixture to fill the pores between solid W particles, thereby producing composite materials with reduced porosity and improved structural uniformity.

The influence of activated sintering on the fabrication and properties of W-based composites has not been comprehensively investigated, and studies on the infiltration of various alloys into W skeletons for different applications remain limited. In this study, W composite materials were produced by Ni-activated sintering, followed by infiltration of a liquid Al–Cu–Zn alloy (50 wt% Al–30 wt% Cu–20 wt% Zn) to fill the residual pores. Microstructural observations and density measurements demonstrated that the Al–Cu–Zn infiltration technique is highly effective in reducing the porosity of the sintered structure, increasing the density of the compact by approximately 3–5%, and enhancing the mechanical integrity and thermal stability of the material. This method shows great potential for fabricating multi-metallic composites based on porous W skeletons.

2. EXPERIMENT

2.1. Sample preparation methods

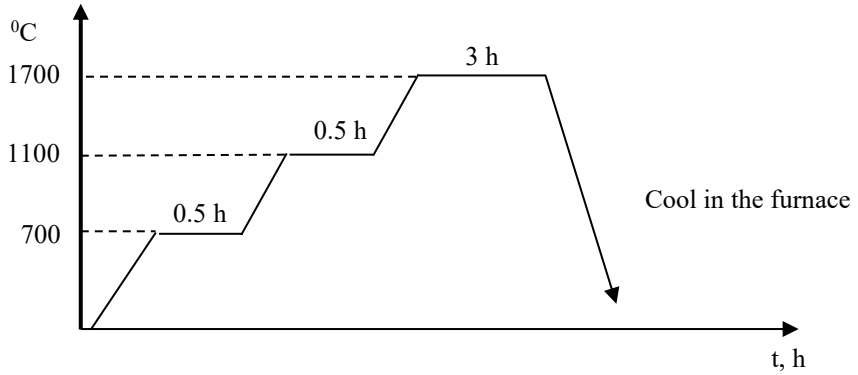


Figure 1. *Sintering diagram.*

In this study, tungsten powder with 99.95% purity and an average particle size of 6–7 μm was used. Nickel powder with 99.99% purity and a particle size of approximately 150 μm was added at 1.5 wt% as a sintering activator (The powders were made in China, AVIC Zhongmai Metal Materials Co., Ltd). The W–1.5% Ni mixture was blended with 0.5 wt% stearic acid as a process control agent and mixed for 4 hours in a sealed stainless-steel container with steel balls under an argon atmosphere. After mixing, the powder was washed with methanol and dried at 80 °C.The

mixed powder was then cold isostatically pressed, the pressures applied were: 963, 1123, 1204, 1284, 1445, and 1605 MPa, with a force holding time of 30 s. The sample size obtained after pressing is ϕ12.2 mm, and the workpiece height varies from 17 to 18 mm. The achieved density ranges from 65 to 70% of the solid density. The results after pressing showed that to achieve a density of 68-70% solid density, the pressing force only needed to be about 963 to 1084 MPa. Increasing the pressing force did not significantly increase the density.

The pressed samples were sintered in a graphite-tube furnace at 1700 °C, with a heating rate of 10 °C/min, and held for 3 hours in an Ag protective atmosphere, as shown in Figure 1. The sintering process was maintained at temperatures of 700 °C and 1100 °C for 0.5 hours to stabilize the samples, decompose the lubricant, and vaporize the lubricant.



Molds and presses.



Furnace for molten metal.



Tube furnace sintered graphite boat.



Sintered blanks.

Figure 2. *Experimental pressing, sintering of W block, and infiltration of Al-Cu-Zn alloy.*

A porous W skeleton was thereby formed, exhibiting a relative density of 76–80%. The infiltration of the liquid Al–Cu–Zn alloy (50 wt% Al–30 wt% Cu–20 wt% Zn) was then carried out at 1000 °C for 1 hour in ambient air. According to the Al–Cu–Zn ternary phase diagram (Figure 3), the 50Al–30Cu–20Zn composition has a melting point below 820 °C; thus, conducting infiltration at 1000 °C ensures complete melting and sufficient fluidity of the alloy to penetrate into the pores of the W skeleton [11–13]. The molds, pressing apparatus, sintering furnace, and infiltration setup are shown in Figure 2.

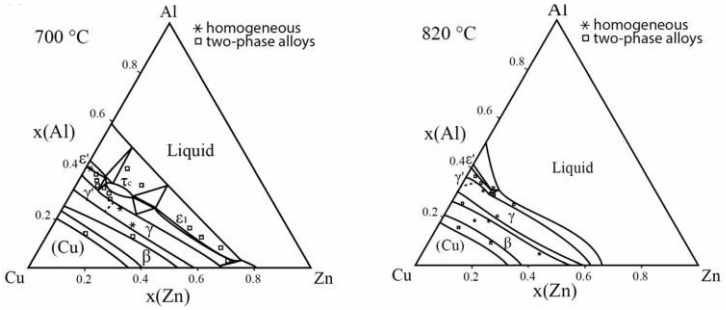


Figure 3. *Al-Cu–Zn alloy ternary phase diagram [12].*

2.2. Methods for investigating properties

The density of the pressed, sintered, and infiltrated samples was measured by the gravimetric method and the Archimedes technique. The relative density of the samples was calculated according to the rule of mixtures. For the W–1.5 wt% Ni alloy, the theoretical density was

determined to be 19.24 g/cm³. The relative density of the 50 wt% Al–30 wt% Cu–20 wt% Zn mixture, with a theoretical density of 4.05 g/cm³, was also calculated according to the rule of mixtures [2].

$$\frac{1}{\rho} = \frac{A}{\rho_A} + \frac{B}{\rho_B} + \frac{C}{\rho_C} \quad (1)$$

Where ρ is the theoretical density of the composite material, A, B and C are the mass percentages of the respective metals.

Microscopic analysis of the samples after infiltration was performed, the etched sample was performed using Murakami reagent, consisting of 10 g $K_3Fe(CN)_6$ (potassium ferricyanide), 10 g KOH or NaOH, 100 ml H_2O. Microstructure observations and elemental composition analyses were performed using a JEOL JSM-6510LV scanning electron microscope with energy-dispersive X-ray spectroscopy (SEM/EDS) and an Axiovert 40MAT optical microscope. Measure the density of the sample using a precision balance according to the Archimedes method.

3. RESULTS AND DISCUSSION

3.1. Optical microstructure

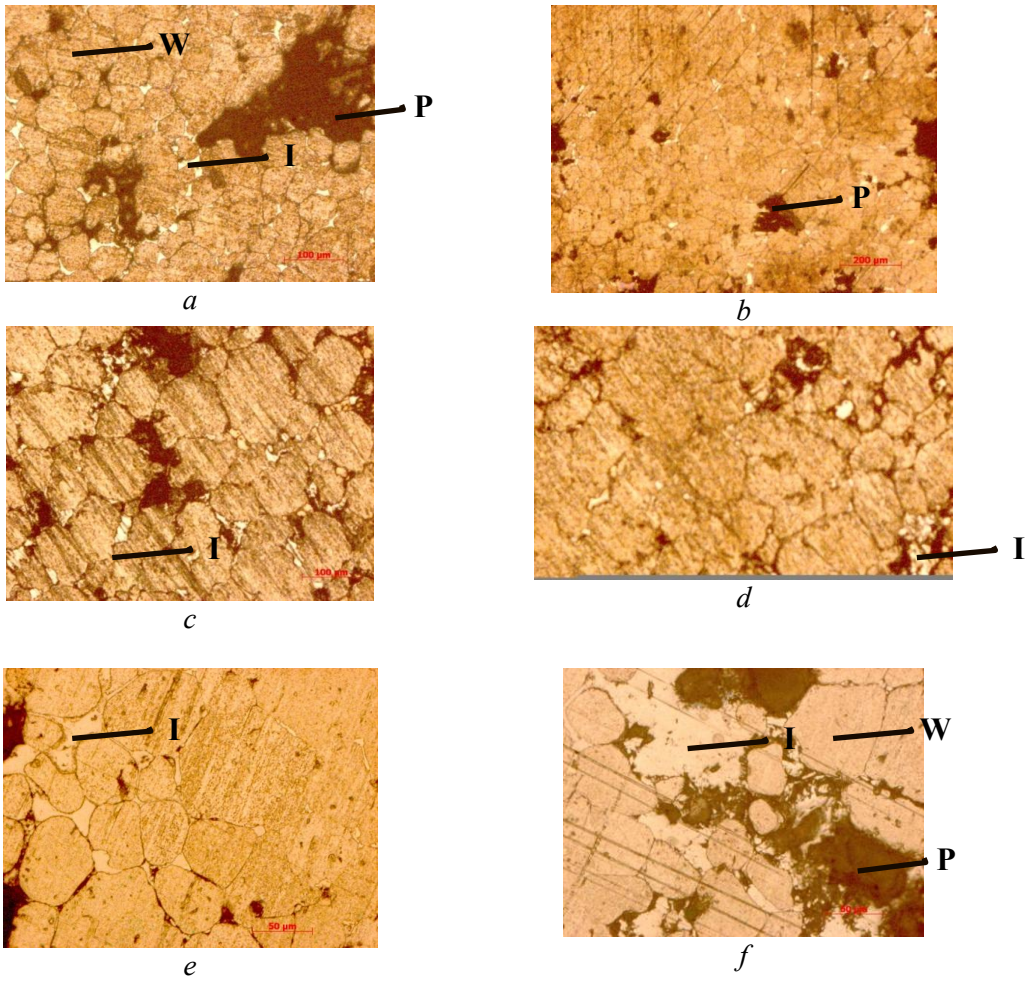


Figure 4. *Microscopic structure of the sample after infiltration*
W- Matrix W, P- Pore, I- Inifiltrator.

Microstructure characteristics of the fabricated post-infiltration W-1.5Ni composites are

shown in Figure 4. The microstructure of the W–Ni–Cu–Al–Zn composite shows that it consists of W particles ranging in size from 30 to 100 μm, which are bonded together through solid-state diffusion during sintering. Interconnected open pores are formed along the grain boundaries, mainly concentrated at triple junctions between grains. Both small and large pores, comparable in size to the W grains, are observed after sintering.

At the W–W grain interfaces, the etching contrast indicates that the Ni–Cu–Al–Zn alloy is concentrated along the grain boundaries (Figure 4). The pores at the W–W interfaces are filled with the infiltrated alloy, which enhances the structural continuity of the sintered body. Filling these pores increases the overall density and improves the mechanical properties, particularly by enhancing the plasticity of the composite under load. This improvement arises from the contribution of shear deformation along grain boundaries under external stress, which helps delay crack initiation and propagation.

To achieve complete filling of the pores with liquid metal, it is necessary to ensure appropriate conditions for the capillary-driven infiltration process. In addition to maintaining optimal porosity, the wettability of the powder particles must be improved. The pore size strongly affects the infiltration behavior, since capillary pressure is inversely proportional to pore diameter; thus, the infiltration rate increases in smaller pores.

However, a pressure drop in larger pores can cause gas entrapment, which hinders infiltration. Therefore, in a liquid alloy system, the melt more readily penetrates smaller capillary channels due to better wettability and smaller contact angles. In contrast, larger pores or channels are more difficult to fill because of trapped gas and poor wetting conditions. Closed pores, which are isolated from the capillary network, remain unfilled. This behavior is clearly illustrated in Figure 5. Hence, “permeability” should be considered a complex property that depends on the combined characteristics of the liquid alloy, the pore structure, and the infiltration atmosphere.

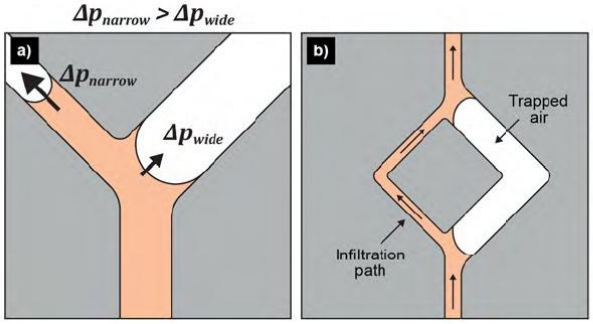


Figure 5. *Schematic diagram of capillary rise in Y-shaped channels of different radii [14].*

To ensure complete infiltration, the W skeleton must possess an interconnected network of open pores. Studies have shown that the optimal pore characteristics for infiltration include a pore size ranging from 1 to 4 μm and a length-to-width ratio between 1 and 8. The pore size is primarily determined by the particle size of the W powder used to form the skeleton and by the degree of sintering [1, 14-16].

3.2. Composition analysis by EDS spectrum of infiltration sample

Figure 6. Scanning electron microscopy (SEM) image of the sample after infiltration. The sample surface shows pores partially filled by the infiltration alloy, located between the W-W grain boundaries.

The results of EDS analysis of surface composition points from spectrum 22 to spectrum 36 are given in Figures 6, 7 and Table 1.

The results presented in Figure 6 and Table 2 show that the regions located within the grains consist of pure tungsten (Spec. 22). Several phases observed along the grain boundaries are mixtures of W and Ni (Spec. 23–27, 30), indicating that during sintering at 1700 °C, liquid Ni

interacted with solid W to form intergranular W–Ni compounds exhibiting different contrast levels.

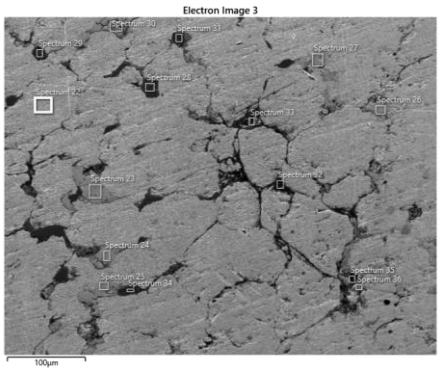


Figure 6. *SEM image of the infiltration sample and EDS spectrum analysis positions.*

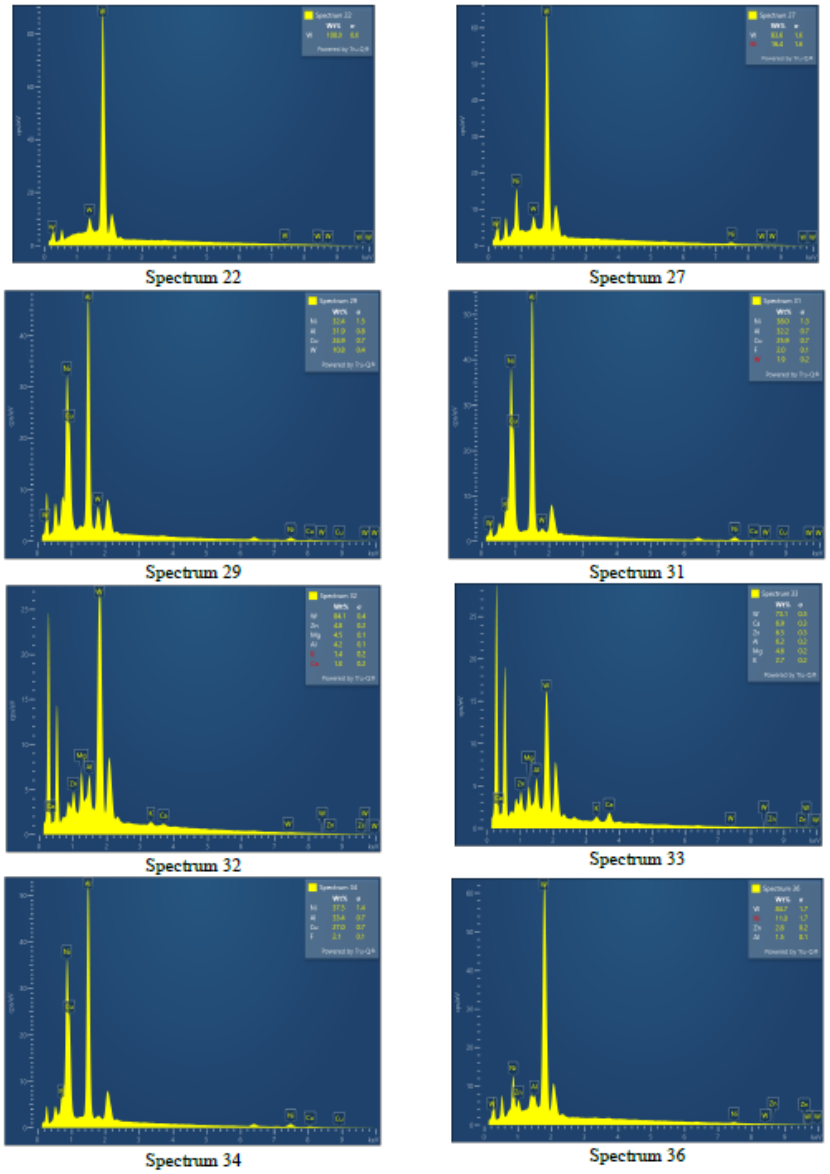


Figure 7. *EDS spectrum analysis results of the selected composition points.*

Table 1. *EDS analysis results of point composition, %.*

Location	% element,%w								
	W	Ni	Cu	Al	Zn	Ca	K	Mg	F
Spec 22	100	-	-	-	-				
Spec 23	82.4	17.0		0,1					-
Spec 24	81.5	18.5							-
Spec 25	84.3	15.7							-
Spec 26	81.3	18.7							-
Spec 27	83.0	16.4							-
Spec 28	2.7	35.6	27.0	30.6	2.2				1.8
Spec 29	10.8	32.4	24.9	31.9	2.2				-
Spec 30	81.6	12.5		2.0	1.8		1.5	0.7	-
Spec 31	1.9	38.0	25.9	32.2	2.2				-
Spec 32	84.1	-		4.2	4.8	1.0	1.4	4.5	-
Spec 33	72.1	-		6.2	6.5	6.9	2.7	4.6	-
Spec 34	1.4	37.0	26.6	32.9	2.2				-
Spec 35	82.1	-		3.4	4.7	5.3	0.7	3.8	-
Spec 36	84.7	11		1.5	2.8				-

At certain locations, the pores were filled with the infiltrated Al–Cu–Zn alloy (dark or black contrast) containing various proportions of Al, Cu, and Zn (Spec. 28, 29, 31, 34). These regions also contained a considerable amount of Ni, suggesting that nickel had infiltrated and coated the W grains, thereby promoting the wetting and infiltration of the liquid alloy. Since Ni is completely soluble in Cu, it facilitates the spreading of the liquid phase along the W interfaces.

The positions corresponding to Spec. 30, 32–36 represent fine grains located between larger W particles, where W accounts for approximately 70–80%, along with small amounts of Ni, Al, and Zn. Minor elements such as Ca, K, and F were also detected as impurities within the pores, possibly introduced during sample preparation or chemical etching.

It was also observed that larger pores remained only partially filled with the infiltration alloy compared with smaller ones, indicating that the infiltration duration of 1 hour was insufficient for the molten alloy to fully penetrate the pore network.

3.3. Density measurement

The results of density measurements for 3 samples by weighing, volume measurement, and Archimedes buoyancy measurement in water are shown in Table 2.

It can be observed that the increase in mass is directly related to the ability of the liquid alloy to penetrate into the pores of the sintered compact, which in turn depends on the pore characteristics, the infiltration time, and the atmospheric conditions during infiltration.

Table 2. *Density before and after infiltration.*

N^0	Density g/cm^3			% Volume increase
	After pressing (% theoretical)	Sintering (% theoretical)	Density after infiltrating	
1	13.0 (67.6)	15.3 (79.5)	16.0	4.6
2	12.7 (66.0)	14.9 (77.4)	15.47	3.8
3	13.4 (69.6)	15.3 (79.5)	15.75	2.9

For the sintered W–1.5Ni skeleton with a relative density of 76–80%, infiltration by immersion in the liquid 50%Al–30%Cu–20%Zn alloy for 1 hour in air resulted in an increase in density by approximately 3–5% by mass compared with the pre-infiltrated compact, in terms of volume, it fills an additional 6% of the sintered mass.

4. CONCLUSIONS

This paper investigates the increase in density of W–1.5Ni composite materials through the infiltration of a 50%Al–30%Cu–20%Zn molten alloy into a sintered W skeleton with a relative density of up to 80%. Based on the analysis of the microstructure and the composition of the infiltrated regions, the following conclusions can be drawn:

Activated sintering using Ni as an activator at 1700 °C produced a W skeleton with interparticle bonds measuring 30–100 μm, derived from the initial powder particles with an average size of 6 μm. The relative density of the W–1.5Ni composite reached 80% of the theoretical density. Ni interacted with W to form phases at the grain boundaries.

During infiltration, the 50%Al-30%Cu-20%Zn alloy fills the pores located at the grain boundaries, forming an alloy with Ni and hardly interacting with W. Non-connected pores are difficult to completely diffuse. Small, interconnected pores diffuse more easily, while larger pores are almost impermeable due to low capillary forces and the presence of trapped gas. Pore morphology plays a crucial role in determining the infiltration efficiency through capillary action, whereas it shows less dependence on the maximum sintered density achieved.

The W–1.5Ni composite sintered to 80% of the theoretical density and impregnated with the 50%Al–30%Cu–20%Zn alloy for 1 hour at 1000 °C exhibited a density increase of approximately 3–5%.

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TÓM TẮT

Nghiên cứu tăng mật độ hợp kim W-1,5Ni thiêu kết bằng phương pháp thấm hợp kim Al-Cu-Zn

Bài báo này nghiên cứu quá trình tăng mật độ của hỗn hợp W–1,5%Ni được chế tạo bằng phương pháp ép và thiêu kết, đạt mật độ ban đầu từ 77–80% để tạo thành khung W xốp. Quá trình thiêu kết đẳng nhiệt được thực hiện trong lò ống graphit ở nhiệt độ 1700 °C, giữ nhiệt 3 giờ trong môi trường khí bảo vệ Argon (Ar). Niken (1,5%) được sử dụng làm chất hoạt hóa thiêu kết. Sau đó, các lỗ rỗng còn lại được làm đầy bằng phương pháp thấm ướt với hợp kim lỏng 50%Al–30%Cu–20%Zn ở 1000 °C trong 1 giờ. Kỹ thuật thấm ướt bằng hợp kim Al–Cu–Zn đã chứng tỏ hiệu quả trong việc giảm độ xốp của cấu trúc thiêu kết và tăng mật độ thêm khoảng 3–5%, qua đó cải thiện các tính chất cơ học và độ ổn định nhiệt của vật liệu composite thiêu kết, tạo ra vật liệu composite dùng làm bộ phận tản nhiệt.

Từ khóa: Thiêu kết vonfram; Thấm hợp kim cho khung W xốp; Thấm hợp kim Al-Cu-Zn; Thiêu kết hoạt hóa.