Research Article

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Microstructure and mechanical properties of NbC–Ni cermets prepared by microwave sintering

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Abstract: This study shows the application of microwave sintering (MS) on preparing NbC–10Ni and NbC–12Ni cermets, as well as the effect of its microstructure, phase formation, and mechanical properties. Results indicated that NbC–Ni cermets with a fine and uniform structure can be obtained by MS in a relatively short time. And the sintering temperature greatly influenced the microstructure and mechanical properties of NbC–Ni cermets, whereas the effect of dwell time is relatively small. With the increase of the sintering temperature, the microstructure of NbC–Ni cermets experienced sintering densification and grain growth. The strength and toughness increased first and then decreased, and the hardness increased with the increase of sintering temperature. According to the comprehensive mechanical properties, the optimized sintering process is 1,390°C and the dwell time is 15 min. At this time, no new phase formed, but the diffraction peak of the Ni phase shifted. Through analysis, it is found that the improvement mechanisms for comprehensive mechanical properties of NbC–Ni cermets mainly include grain refinement, crack deflection and bridging, and energy absorption of the ductile phase of Ni.

Keywords: NbC–Ni cermets, microwave sintering, microstructure, mechanical property

1 Introduction

Since the 1920s, WC-Co cemented carbides have been extensively used as cutting tools, molds, and wear-resistant tools in the machinery, petrochemical, and automobile industries due to their higher hardness, strength, and toughness. However, WC-Co cemented carbides is easily oxidized to generate $CoWO_4$ and WO_3 [1] during its use, and the maximum service temperature is 800–900°C [2]. When processing high-strength and high-hardness materials, these cutting tools soften and react, then lose efficacy. Meanwhile, with people's attention to environmental protection, the toxicity of bonding phase of Co and its oxides have been paid more attention [3]. In recent years, NbC-based cermets have become an alternative candidate for WC-Co cemented carbide tool materials [3], owing to its higher melting point, lower density [4], higher temperature performance [5], as well as lower solubility [6,7] in steel than WC.

Co, Ni, and Fe are used as bonding phases of NbC-based cermets [8,9]. While Ni with its better wettability gradually replaces Co as the first choice. NbC–Ni cermets have improved fracture toughness than that of NbC-Co materials but have lower hardness [5]. Adding the second-phase carbides Mo_2C and VC, the wettability of hard phase and bonding phase in the cermet has increased, the microstructure has refined, the grain growth has inhibited, and the hardness has increased [10–13]. Adding TiC and WC facilitate the formation of the "core-ring" structure of NbC–Ni cermets and increase its hardness [11,14]. Different sintering methods also have a significant impact on the microstructure and properties of NbC–Ni cermets. Results [5] show that the hardness of materials sintered by rapid pulse electric current sintering (PECS) is higher than that of vacuum liquid-phase sintering (LPS), nevertheless, the fracture toughness is lower.

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The heating rate and sintering time of microwave sintering (MS) are between LPS and PECS, which can compensate for the problem of grain growth caused by the excessively long sintering time of LPS and the uneven distribution of bonding phase caused by the short sintering time of PECS. Studies show that MS can obtain TiC-based cermets [15,16] with a complete core-ring structure. Therefore, in this article, MS is used to prepare NbC–Ni cermets and systematically study the grain growth, microstructure, and mechanical properties evolution.

2 Experimental process and method

In the experiment, NbC powder (Brofos, FSSS = 1.5 μm, purity ≥99.9, China, as shown in Figure 1a) and Ni powder (Yingtai, FSSS = 1–3 μm, purity ≥99.98, China, as shown in Figure 1b, the agglomeration occurs due to the pressure of the vacuum compression package) were used as initial raw materials to prepare NbC–Ni cermets with NbC–10 vol% Ni and NbC–12 vol%Ni, respectively, marked as 10Ni and 12Ni. At first, the raw materials are placed in a stainless-steel ball mill tank, then anhydrous ethanol is added as the ball milling medium, then 4 wt% paraffin wax is added as a forming agent, and then YG6 cemented carbide milling balls are selected to mill the mixture. The ball-to-material ratio is 5:1. The ball milling speed is 300 rpm, and the time of ball milling is 24 h. Later, ball-milled slurry is placed in the electric heating incubator to be heated and dried at a temperature of 80°C. The morphology of NBC–12Ni powder after ball milling and drying is shown in Figure 1c, indicating that the agglomeration of Ni powder after ball milling is basically eliminated. Then the powder is formed by uniaxial compression at 250 MPa. After that, it is heated to 400°C in the vacuum furnace to degrease for 1 h, then placed in a vacuum MS furnace (MW1306V) and heated to 1,270–1,450°C. The heating rate is 10–15°C·min^{-1}, and the dwell time is 5–30 min. Eventually, the sintered samples with a size of (21 ± 0.5) × (7 ± 0.25) × (6 ± 0.25) mm are formed.

After being ground and polished, the sintered samples were carefully measured and observed. The density was measured by Archimedes principle; the hardness of

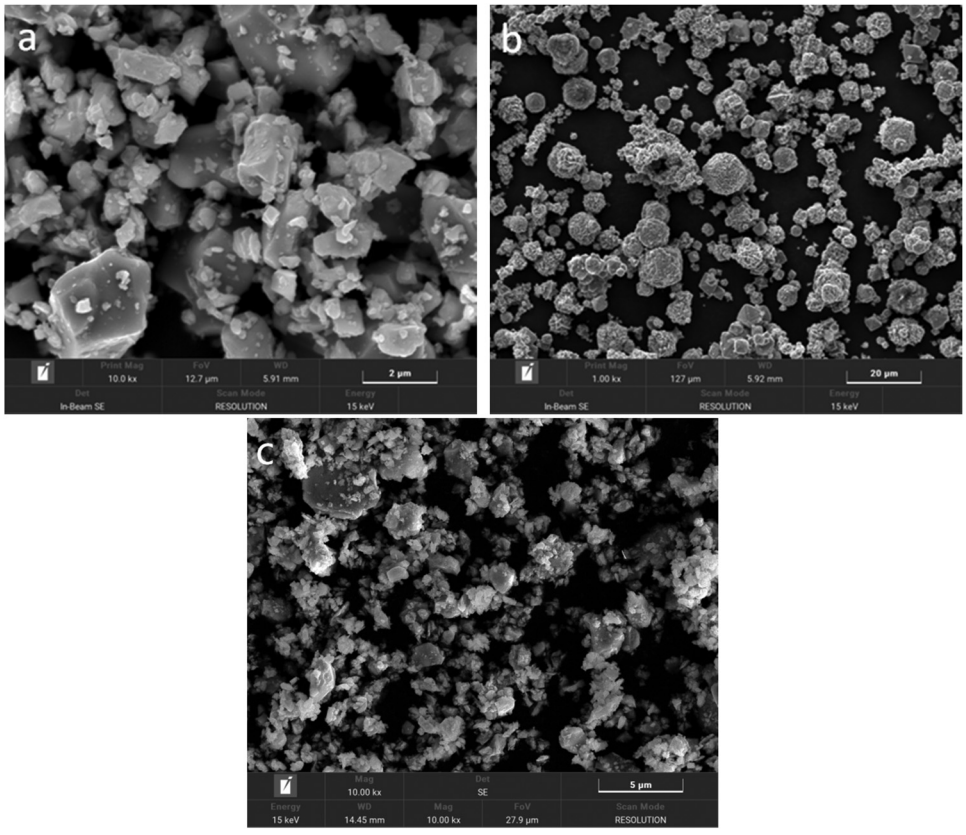

Figure 1: Morphology of the raw material powders: (a) NbC, (b) Ni, and (c) NbC–12Ni after ball mill.

the sample was measured by Vickers hardness tester, at the time, the indentation load was 294.2 N, the dwell time was 10 s; and its fracture toughness K_{IC} was used to calculate by the Palmqvist formula (equation (1)) [17], taking the average of five times.

$$K_{IC} = 0.0889\left(\frac{HV \cdot P}{\sum l}\right)^{\frac{1}{2}}, \quad (1)$$

where P represents the experimental payload (N), HV is the Vickers hardness (GPa), and $\sum l$ is the sum of crack length (μm). While the sintered samples have been machined to the shape of bars of about 20 $mm^3 \times 6.5\ mm^3 \times 5.25\ mm^3$, the bending strength of the samples was measured by the three-point bending method on a universal testing machine. During the test, the span was 12.5 mm and the loading speed was 0.1 $mm \cdot s^{-1}$. X-ray diffractometer was used to analyze the phase of the sintered body, and the microstructure and cross-section of samples were observed with a scanning electron microscope (SEM, Zeiss Sigma 300) with energy disperse spectroscopy (EDS).

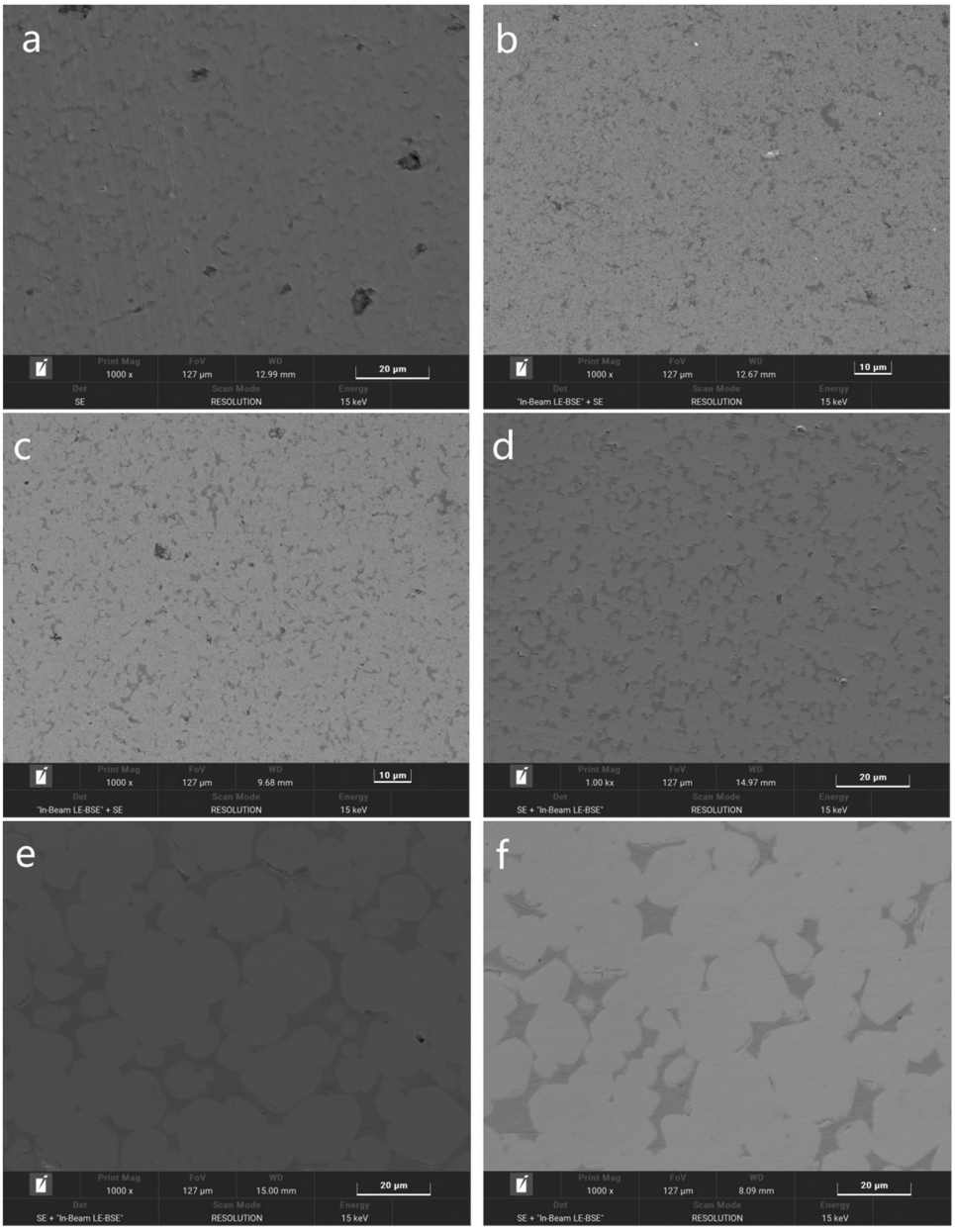

Figure 2: SEM micrograph(s) of NbC–12Ni cermets with different sintering temperatures (SE + BSE mode): (a) 1,270°C, (b) 1,300°C, (c) 1,330°C, (d) 1,390°C, (e) 1,420°C, and (f) 1,450°C, and dwell time 15 min each.

3 Results and discussion

3.1 Microstructure

Figure 2 shows the microstructure (SE + BSE) of 12Ni cermets when it is sintered at different sintering temperatures for 15 min. The grey-white part is NbC, and the grey-black part is the bonding phase of Ni. It can be seen that they have large pores in the 10Ni cermets at 1,270°C, and the sintered body is not dense enough; when the temperature rises to 1,300–1,330°C, the 12Ni cermets have no obvious pores. The bonding phase Ni and NbC particles are tiny and uniform; as the sintering temperature rises to 1,390°C, some larger particles appear in the structure of the 12Ni cermets. When the sintering temperature is further increased to 1,420°C, the NbC particles grow rapidly. At this time, the interface between the NbC particles and the bonding phase is clear; when the temperature rises to 1,450°C, the NbC particles further grow, but at this time the microstructure of 12Ni cermets is uniform and no obvious defects are found. In particle size analysis parts, it shows that the maximum NbC particles are about 20 μm at 1,420°C, and about 30 μm at 1,450°C. While the microstructure of 10Ni cermets (Figure 3) changes with temperature and is similar to that of 12Ni, dense and tiny NbC–Ni cermets can be obtained by sintering at 1,390°C, and the particles grow obviously at 1,420°C, some defects can be seen during sintering at 1,420 and 1,450°C.

According to the phase diagram of NbC–Ni [14], the liquid-phase formation temperatures of 10Ni and 12Ni cermets are about 1,265 and 1,280°C, respectively. NbC–% Ni cermets with 9.75–10 wt% carbon content can obtain an NbC + Fcc two-phase structure at room temperature. While carbon content less than 9.75 wt% will form the $NbNi_3$ phase, while over 10 wt% will form the graphite phase and a three-phase structure. The carbon content of 10Ni is 10.11 wt%, and the carbon content of 12Ni is 9.85 wt%. Therefore, the graphite phase of 10Ni may appear. In this article, 1,270°C is the solid-phase sintering stage according to the phase diagram, and larger pores appear in the NbC–Ni cermets' structure; sintering above 1,300°C is LPS, and a dense sintered microstructure is obtained. The LPS mechanism of NbC–Ni cermets is the Ostwald ripening

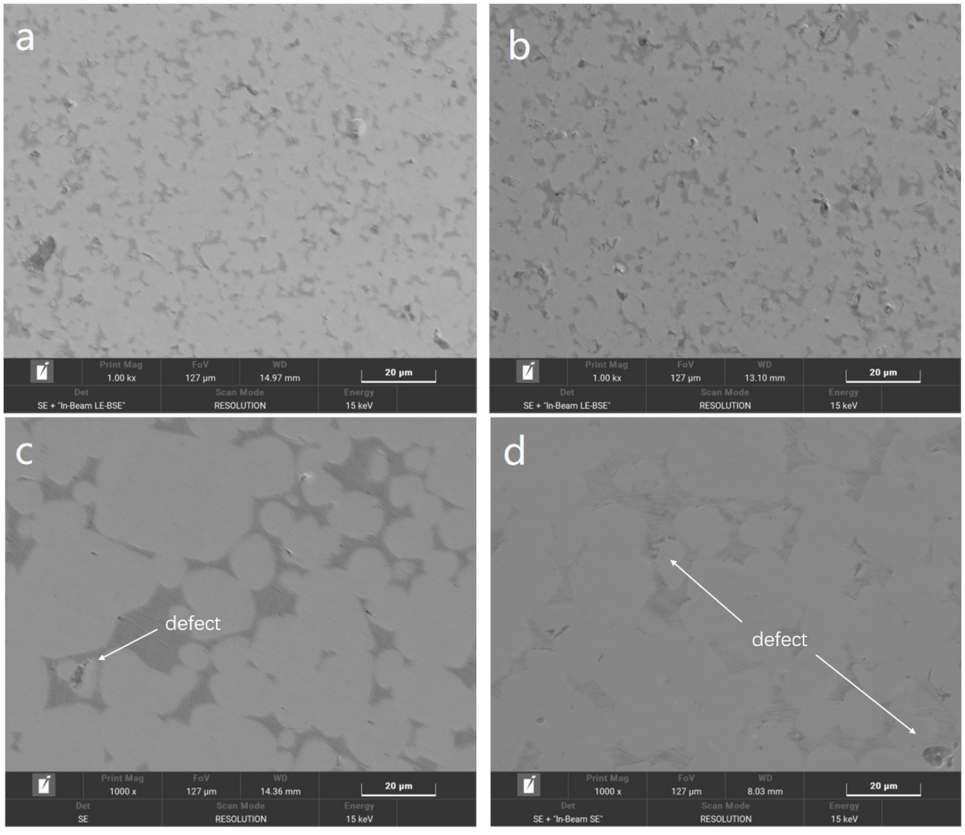

Figure 3: SEM micrograph(s) of NbC–10Ni cermets with different sintering temperatures (SE + BSE mode): (a) 1,360°C, (b) 1,390°C, (c) 1,420°C, and (d) 1,450°C, and dwell time 15 min each.

mechanism. During high temperature sintering process, NbC dissolves in the bonding phase Ni and precipitates. In the cooling process, it is conducive to the sintering density of materials. Therefore, a fine and uniform cermet is obtained when sintered above 1,300°C. At the same time, the solubility of NbC in Ni increases with the increase of temperature, and the solubility of NbC in Ni is about 0.5–1.0% [11] at 1,250°C, the solubility of NbC in Ni is 7 wt% [18] at 1,400°C. As the temperature increases, the solubility of NbC in Ni increases, and during the heating process, the tiny NbC particles dissolve in the liquid phase. While during the cooling process, NbC preferentially precipitates in the undissolved large NbC particles, which promotes the growth of NbC. The main driving force for the dissolution of small particles and the growth of large particles is the decrease of the surface energy of solid particles [11]. At the same time, NbC particles may also grow due to the atomic diffusion across the solid–solid boundary [11]. The microstructure in the temperature range of 1,330–1,390°C is slightly larger than that at 1,300°C, and the structure grows significantly at 1,420–1,450°C.

The EDS line scanning of the interfacial boundary of NbC–12Ni at 1,390°C in Figure 4 shows the diffusion and dissolution of elements at the interface. There is solid solution of NbC phase in the Ni phase in the middle region 3, while Ni is not dissolved in the NbC phase in the two sides' region 1, and the element distribution in the transition region 2 is uniform and continuous. It shows that the NbC/Ni system has good wettability, and the interface between NbC phase and Ni phase is well combined.

The EDS map scanning analysis of Figure 5 shows that the binding phase Ni is evenly distributed around the NbC framework, indicating that Ni has good wettability for NbC. At the same time, the defect in the 10Ni cermets is graphite, which may be related to its higher initial carbon content.

The microstructure of cermets (SE + BSE) with different dwell times is shown in Figure 6. When holding for 5 min, tiny pores can be seen in both 10Ni and 12Ni cermets, indicating that the dwell time is insufficient; when the dwell time is extended to 30 min, the microstructures of 10Ni and 12Ni cermets are uniform, and the microstructure is not much different from that at 15 min of dwell time (Figures 2d and 3b), but there is a growing trend, especially for 12Ni cermets. However, compared with grain growth caused by 30–1,420°C increase in sintering temperature (Figure 3), the particle growth caused by prolonged incubation time was not obvious.

3.2 Phase composition

The XRD diffraction spectrums of the NbC–Ni cermets sintered at different temperatures are shown in Figure 7. XRD diffraction spectrums showed NbC and Ni did not react and did not form a new phase during the MS process. Compared with the raw material, the main diffraction peak of NbC has no obvious change after MS, which is also no different from ordinary sintering [2]. Compared with the pure phase of Ni, the diffraction peaks of Ni in the sintered body are significantly shifted to a lower diffraction angle since NbC has a certain solubility in Ni [10,17], and the diffraction peak of Ni nearly disappears at 1,420°C.

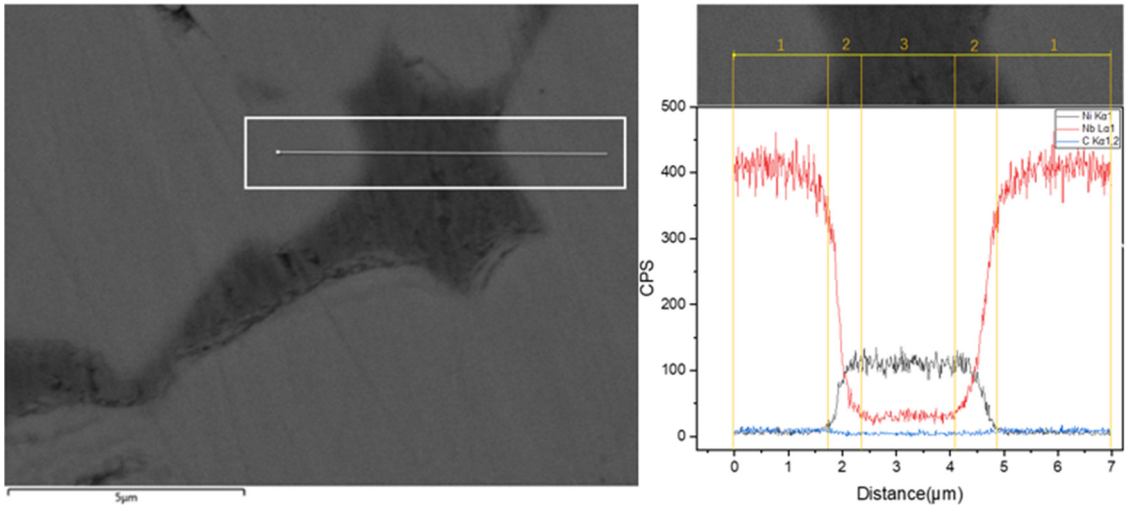

Figure 4: EDS line scanning of NbC–12Ni interfacial boundary at 1,390°C.

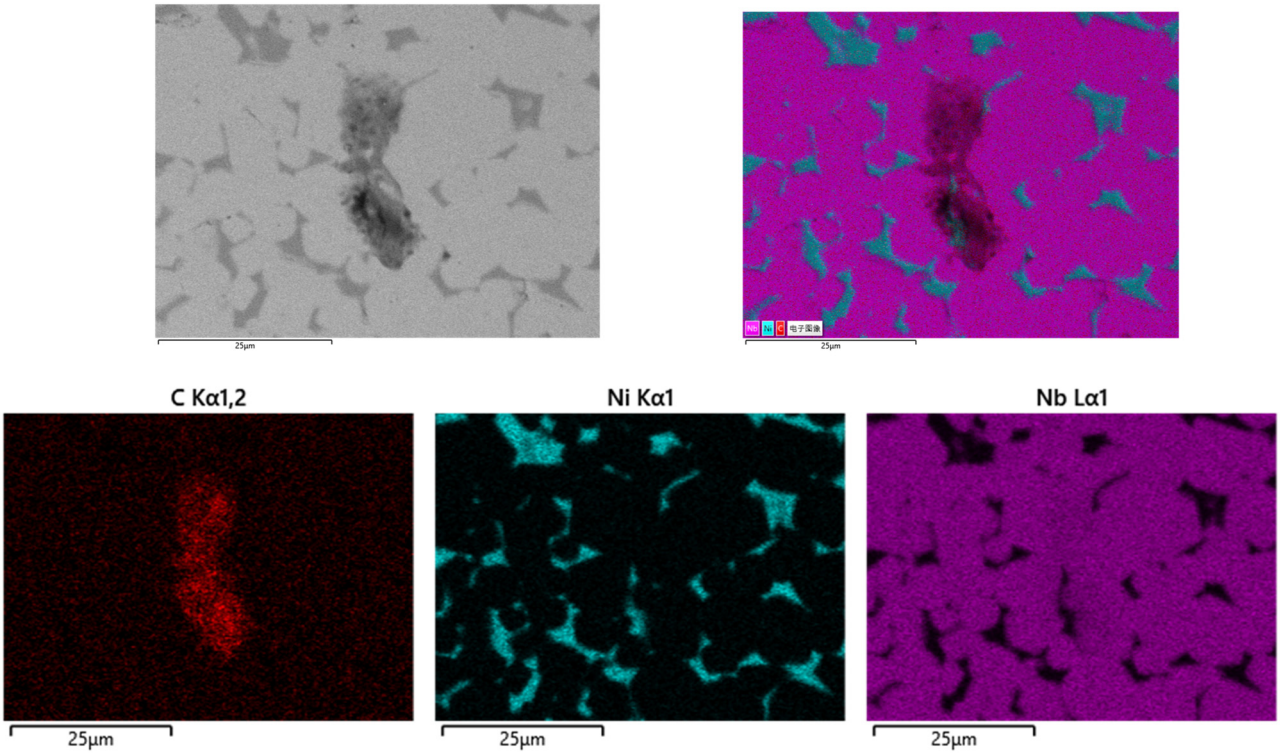

Figure 5: EDS map scanning of NbC–10Ni cermets sintered at 1,450°C.

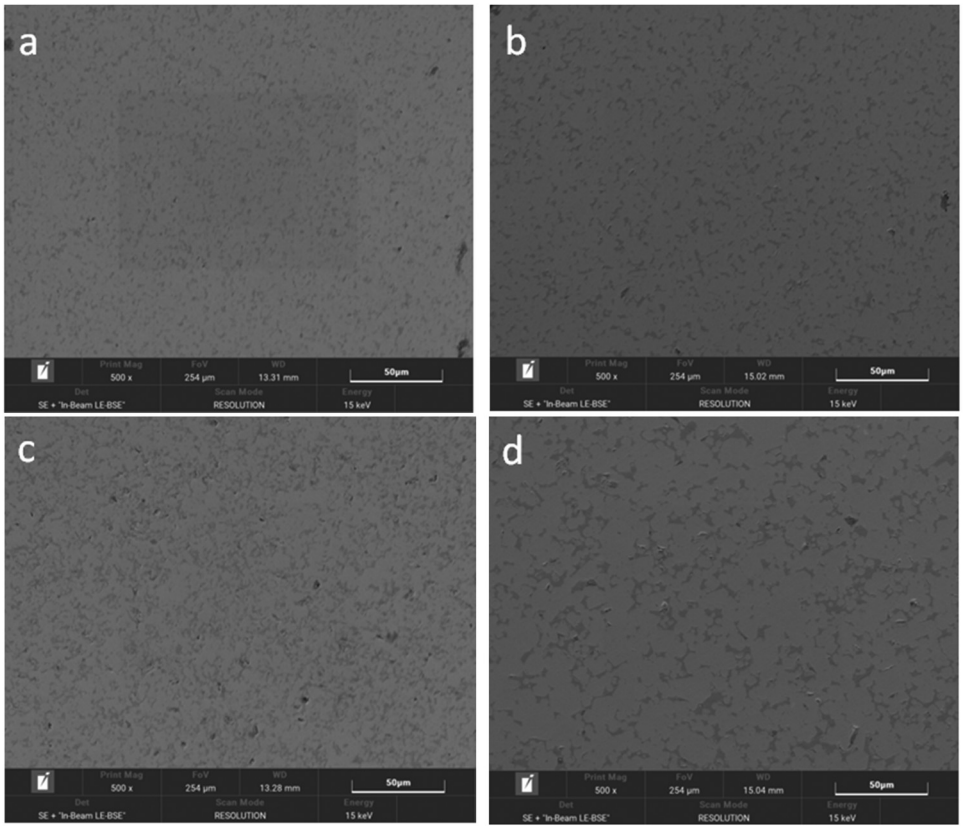

Figure 6: SEM micrograph(s) of NbC–Ni cermets with different dwell times (SE + BSE mode): (a) NbC–10Ni 5 min, (b) NbC–10Ni 30 min, (c) NbC–12Ni 5 min, and (d) NbC–12Ni 30 min, and sintering temperature 1,390°C each.

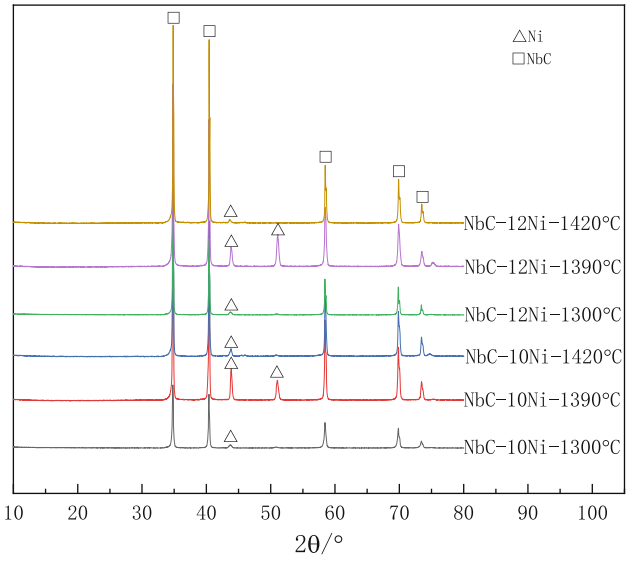

Figure 7: XRD diffraction spectra of raw materials and NbC–Ni cermets at different sintering temperatures.

3.3 Mechanical properties

The mechanical properties of 10Ni and 12Ni cermets sintered at different temperatures are shown in Figure 8. With the increase of temperature, the Vickers hardness of NbC–Ni cermets shows an upward trend, while the fracture toughness and bending strength show an increase first and then a decrease trend with the increase of sintering temperature, reaching the extreme value at 1,390°C.

Compared with 10Ni, 12Ni has higher fracture toughness and bending strength. The Vickers hardness, fracture toughness, and flexural strength of 10Ni at 1,390°C are 1,119 kg·mm^{-2}, 12.5 MPa·m$^{1/2}$, and 1,139 MPa, respectively. While, those of 12Ni are 1,158 kg·mm^{-2}, 13.12 MPa·m$^{1/2}$, and 1,231 MPa, respectively.

The relationship between the hardness and its porosity of powder metallurgy materials can be expressed as [19,20] $H = H_0 \exp(-b\rho)$, where ρ is the porosity of sintered body and H_0 and b are constants. At the same time, according to the Hall-Petch relationship, the relationship between the hardness of the sintered body and the grain size of hard phase can be expressed as [19,20] $H = H_0 + kd^{(-1/2)}$, where d is the average grain size of hard phase and H_0 and k are constants. Sample density at each sintering temperature is shown in Table 1. As the sintering temperature increases, the density of the material increases, and the porosity decreases, while its hardness increases. However, the analysis of the structure in Figures 2 and 3 shows that the cermets particle size grows significantly when sintered at 1,420 and 1,450°C, but the hardness of the NbC–Ni cermets still increases with the

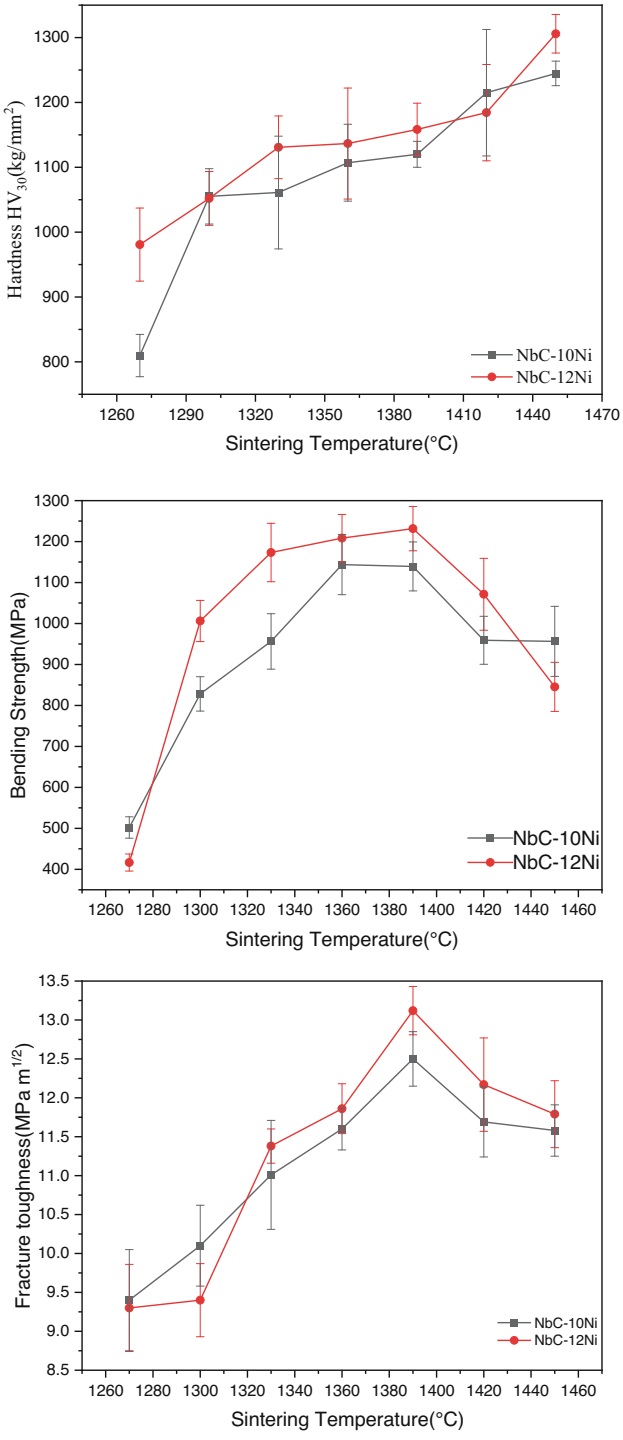

Figure 8: Mechanical properties of NbC–Ni cermets at different sintering temperatures.

increase in temperature. This may be related to the increase of NbC content caused by a small amount of volatilization of Ni in NbC–Ni.

The fracture characteristics of 12Ni at different sintering temperatures are shown in Figure 9. As it can be

Table 1: Relative density of samples at different sintering temperatures

Sintering temperatures (°C)	Relative theoretical density (%)
1,330	99
1,360	99.5
1,390	100
1,420	101
1,450	102

seen in the solid-phase sintering stage (1,270°C), the NbC–Ni cermet particles are tiny, but there still have numerous small pores. The fracture mode of 12Ni is a mixture of transcrystalline and intergranular fracture. While the sintering temperature rises to 1,300°C as the liquid phase is just formed, the small pores are still not completely eliminated, the particle interface is clear, mainly showing as transcrystalline fracture. The existence of small pores makes the mechanical properties of 12Ni cermets sintered at 1,270–1,300°C lower. The sintering temperature continued to rise to 1,360–1,390°C, the small pores almost disappeared completely, and the river-like patterns appeared in the fractures. The appearance of the river-like patterns caused the cracks to fracture and deflect along the habitual cleavage planes in the grains, which increased the crack propagation path [12], thereby improving the strength and toughness. The sintering temperature continued to rise to 1,420–1,450°C, through the observation and analysis of the fracture microstructure of cermet, it is found that the grain grows obviously, and obvious cracks were seen, which led to the decrease of its mechanical properties. The 10Ni cermet also has a similar evolution rule at different sintering temperatures, as shown in Figure 10, but the growth of the structure occurs at the stage of 1,360–1,390°C, which causes its mechanical properties to be lower than that of 12Ni cermets. The possible reason for this phenomenon is that the high carbon content in the 10Ni cermets promotes the growth of its structure.

The indentation cracks of 12Ni cermets sintered at 1,390°C for 15 min are shown in Figure 11. The cracks mainly propagate along the NbC grain-to-grain gaps, split the NbC grains, and pass through the Ni binder phase. The main toughening mechanisms are crack deflection and bridging, and the absorption of crack propagation energy by Ni as the crack propagates through the ductile phase.

The Vickers hardness of the microwave sintered NbC–Ni cermets is comparable to that of the traditional sintering method (LPS) [2] and PECS [5] of the same composition, but the fracture toughness and bending strength

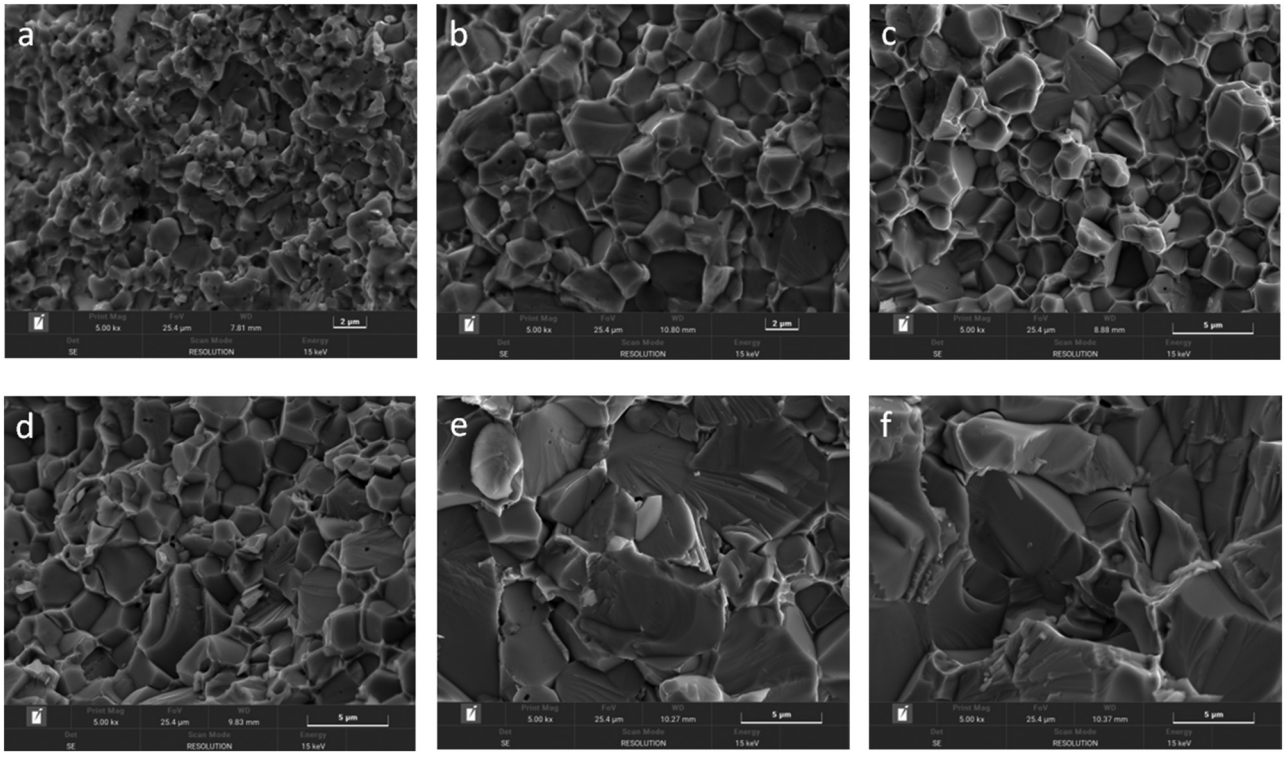

Figure 9: Fracture morphology of NbC–12Ni cermets with different sintering temperatures: (a) 1,270°C, (b) 1,300°C, (c) 1,360°C, (d) 1,390°C, (e) 1,420°C, and (f) 1,450°C, and dwell time 15 min each.

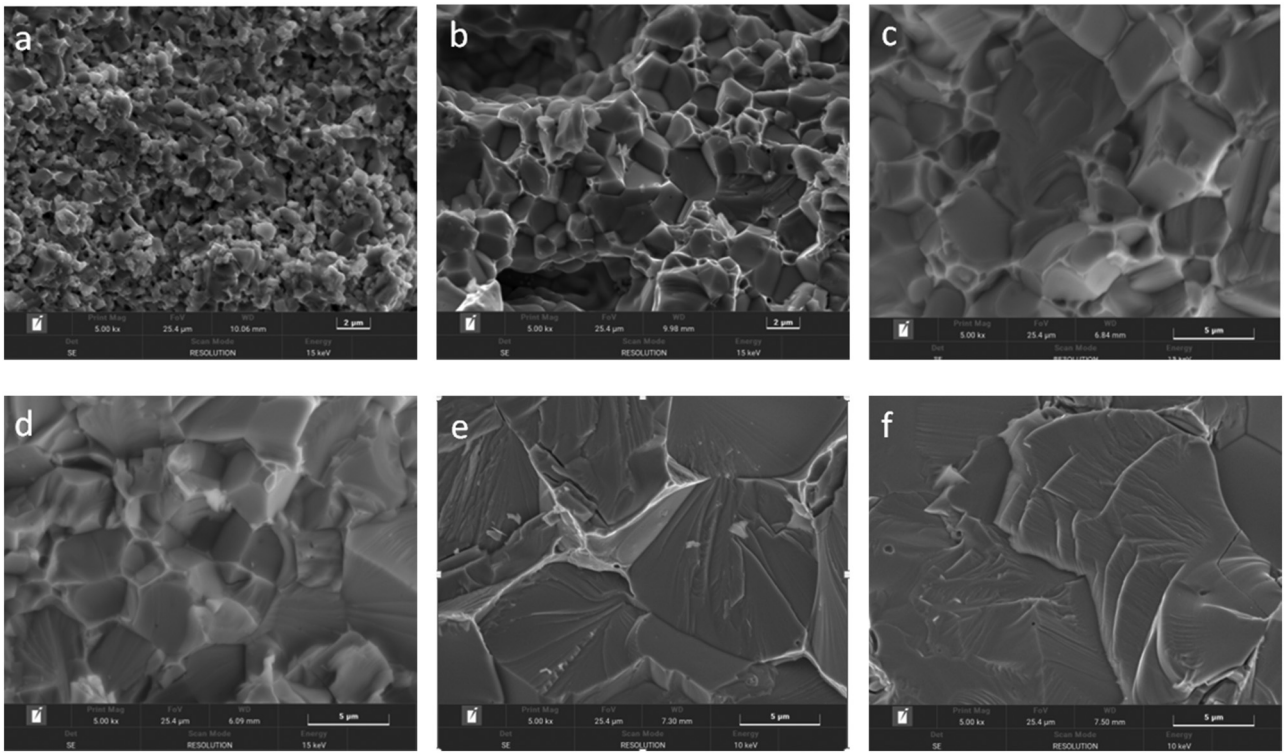

Figure 10: Fracture morphology of NbC–10Ni cermets with different sintering temperatures: (a) 1,270°C, (b) 1,300°C, (c) 1,360°C, (d) 1,390°C, (e) 1,420°C, and (f) 1,450°C, and dwell time 15 min each.

have a significant improvement. The sintering process system of LPS sintering is 1,420°C for 1 h so that the NbC–Ni cermet is fully dense, but it will obtain a coarse structure [2,10,11]. The sintering process system of PECS is 1,280°C for 5 min, sintering fast and at low temperature, which hinders the Ostwald ripening mechanism of the sintering process and the growth of particles and the uniform distribution of the bonding phase [5], reducing the mechanical properties. The heating rate and dwell time of MS are between the traditional sintering and spark plasma sintering. It has the ability to shorten the heating time and form a uniformly distributed bonding phase.

At the same time, the body heating characteristics of microwave heating are conducive to the removal of pores in the material matrix [21,22]. A compact structure and uniform bonding phase distribution can be obtained at 1,390°C for 15 min, thereby obtaining higher comprehensive mechanical properties.

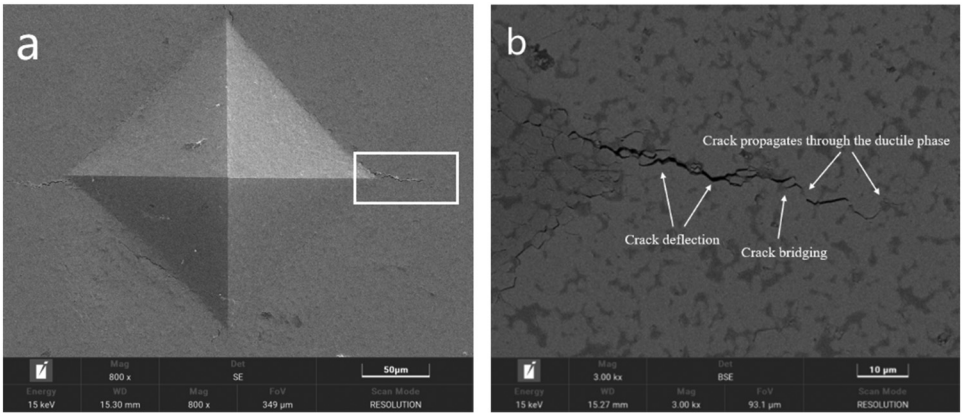

Figure 11: Indentation crack of 1,390°C sintered NbC–12Ni cermets: (a) exemplary indentation and (b) crack propagation.

4 Conclusion

1) 10Ni and 12Ni cermets with compact microstructure and excellent mechanical properties are prepared by rapid microwave heating. The sintering temperature has a greater influence on the microstructure and mechanical properties of 10Ni and 12Ni cermets, and the effect of dwell time is relatively small. The microstructure experienced the process of sintering densification and grain growth with the increase of sintering temperature. With the increase of sintering temperature, the strength and toughness increased first and then decreased, and the hardness increased with the increase of sintering temperature.
2) According to the comprehensive mechanical properties, the optimized sintering process is sintering at 1,390°C for 15 min. At this time, the mechanical properties of 10Ni cermets are 1,119 $kg{\cdot}mm^{-2}$, 12.5 $MPa{\cdot}m^{1/2}$, and 1,139 MPa; the mechanical properties of 12Ni cermets are 1,158 $kg{\cdot}mm^{-2}$, 13.12 $MPa{\cdot}m^{1/2}$, and 1,231 MPa.
3) The microwave-sintered NbC–Ni cermet did not generate a new phase, but the NbC dissolved in the Ni phase shifted the diffraction peak of Ni.
4) The improvement mechanisms of the comprehensive mechanical properties of NbC–Ni cermets sintered by MS are mainly crack deflection and bridging, as well as the absorption of energy by toughness relative to Ni.

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Author contributions: Siwen Tang drafted the manuscript and conducted the experiments, Hao Zhang and Zhifu Yang carry out the experiments, Qian Liu and Zheng Lv guiding experiments, Cong Ouyang and Xinyi Qiu help to analyzed the experimental data and modified and polished the draft.

Conflict of interest: The authors declare no conflicts of interest.

Data availability statement: The data used to support the findings of this study are included within the article.

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