

Effect of Post-Heat Treatment on the Microstructure and Mechanical Properties of SPS-Processed Cobalt-Free WC-Fe Cemented Carbides

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Abstract

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This study investigates the influence of spark plasma sintering (SPS) and subsequent heat treatment on the microstructure, and mechanical properties of cobalt-free WC-Fe cemented carbides. The aim of the work was to evaluate Fe-based binders as a potential alternative to cobalt in tungsten carbide hard alloys while maintaining high structural integrity and mechanical performance. WC-Fe powder mixtures were consolidated by SPS at temperatures of 1250°C and 1300°C, followed by post-sintering heat treatment at 700°C for different holding times. The results showed that increasing the annealing time improved hardness. After SPS at 1250°C, the hardness increased from 1260±0.1 to 1273±0.45 HV, whereas for samples sintered at 1300°C, the hardness increased from 1510±0.4 to 1525±0.7 HV after 3h of heat treatment. The results show that heat treatment after fusion significantly influences mechanical properties, confirming the potential of Fe binders as a promising substitute for cobalt in WC-based cemented carbides.

Keywords: WC-Fe; microhardness; heat treatment; SPS; microstructure

1. Introduction

Improving the performance of material and products, as well as extending their service life in one of most relevant tasks in modern material science. In this connection, research is being actively carried out on alloys and protective coatings capable of providing high wear resistance, strength and thermal stability under heavy-duty conditions. Among such materials, tungsten carbide-based cemented alloys are particularly prominent and are widely used in various industries to protect tools and structural components [1-3]. Their performance is demonstrated by high hardness and durability, which is especially important when manufacturing cutting tools and parts to operate under increased mechanical loads [4]. In cemented carbide, tungsten carbide acts as the main solid phase, providing high hardness and wear resistance of the alloy. The material has a significant crack, reaching approximately 15-20 MPa·m^{3/2} [5-7]. By combining these properties, such materials can be used to protect components operated in extreme conditions, including elements of nuclear power plants where high temperatures and significant thermal loads are possible. Traditionally, cobalt has been used as a metal binder in cemented carbide, which plays an important role in moistening tungsten carbide particles and forming a strong metallic matrix. This helps to increase the material's strength at a bend of up to 3-4 GPa. However, cobalt has a number of significant disadvantages, including relatively low corrosion and oxidation resistance as well as

toxicity, which limits the service life and application of such materials. Therefore, alternative linking systems based on elements such as Ni, Fe [8,9], Cr and Ti have been actively explored in recent years. The use of nickel binders produces alloys with a high hardness (up to ~1400 HV) and an increased destructiveness viscosity (about 7.5 MPa·m^{3/2}) [10-12]. Various technological methods are used to obtain such alloys, including laser smelting, vacuum smelting, traditional sintering and other powder metallurgy techniques. However, the best combination of strength and hardness is usually achieved using modern sintering techniques, among which IPS attracts special attention. The SPS method is based on the application of impulse electric current, which activates processes of compaction and reaction sintering of powder materials.

It is known that the mechanical properties of WC-based solid alloys with Fe-Ni metal bonding can be improved not only by optimizing the parameters of the spark plasma sintering, but also by subsequent heat treatment. According to the literature, additional heat treatment contributes to redistribution and structural stabilization of the connecting phase, reduction of residual stress levels and modification of intergrain boundaries, which has a positive effect on the material's performance. In the work of González and co-authors [13], it is shown that isothermal quenching leads to a change in the microstructure of the binding phase and contributes to an increase in cracking (rupture viscosity) without significant decrease in hardness. In this connection, carrying out heat treatment after SPS-consolidation is of practical interest to improve the complex of physico-mechanical properties of WC-Fe-Ni alloys.

The aim of this work was to obtain non-cobalt solid alloys from the WC-Fe and WC-Fe-Ni systems by a spark-plasma process and to study the subsequent thermal treatment of their microstructure. The scientific novelty of the work consists in a comparative study of SPS-consolidated systems WC-Fe with evaluation of the influence of draft methods on processes of structure compaction and formation of mechanical properties. The results allow for an assessment of the possibility of using iron-based binder as an alternative to cobalt in the development of solid alloys for cutting tools.

2. Materials and Methods

In this study, WC powder (99.5% purity, grain size 30-50 µm, density: 15.63 g/cm³) and Fe powder (99.5% purity, grain size 30-50 µm, density: 7.87 g/cm³) were used as source materials provided by Hebei Suoyi New Material Technology (Hebei Province, China). In order to obtain an ultra-hard carbide layer and manage the batches of metal binder, increasing the viscosity of the entire composite by about 50% without loss of hardness, heat treatment was carried out. For the heat treatment process, the MF-1200TX mill furnace was used at 700 °C with a 1- and 3-hour storage followed by slow cooling in the furnace allowing us to study in pure form the influence of temperature on activation of diffusion processes in iron-tungsten system (Hefei, China). This included preliminary grinding of samples with abrasive materials (silicon carbide) with gradually diminishing granulometry (P400, 1200, 2500) to the Trojan GP-2A double disc grinding and polishing machine (Suzhou, China). To identify the features of the microstructure, chemical etching by reactants was performed. To identify the features of the microstructure, chemical grafting was performed by Murakami reagents prepared by dissolving potassium ferricyanide (K₃Fe(CN)₆) and potassium hydroxide (KOH) in distilled water at a volumetric ratio of 1:1:10 respectively. This composition ensures effective selective grafting of tungsten carbide (WC) without damaging the binding phase [14,15].

The Tescan Vega 4 (Brno, Czech) scanning electron microscope (SAM) with tungsten thermionium cathode was used to study the specimen and obtain detailed surface images. The mechanical properties were assessed by measuring microwarp with a HLV-1DT Vickers (Shanghai Hualong Test Instruments Corporation, Shanghai, China) solider at 0.2 N load and 10 s soaking time, giving an idea of local changes in hardness throughout the composite. This approach allowed the mechanical properties of different sections of the composite to be assessed, taking into account differences in particle distribution and matrix interactions with particles. The combination of structural and mechanical analysis made it possible to compare the features of the microstructure with the performance characteristics and obtain a complete picture of the properties of the composite material.

3. Results and Discussion

The WC-Fe system was chosen for the study because iron is an affordable and cost-effective binder compared to Ni and Co. In addition, the Fe binder provides satisfactory wetting properties of WC particles and

promotes a dense structure during sintering. However, WC-Fe systems are characterized by an intense grain growth and the probability of forming fragile η phases, which requires further investigation of the processes of sintering and heat treatment. In order to evaluate the influence of heat treatment on the element composition and structural evolution of the material, WC-Fe samples, pre-pressed by the SPS method at 1250 °C and 1300 °C, underwent an isothermal quenching at a temperature of 700 °C with storage 1 hour and 3 hours in the atmosphere of the argon (fig.1).

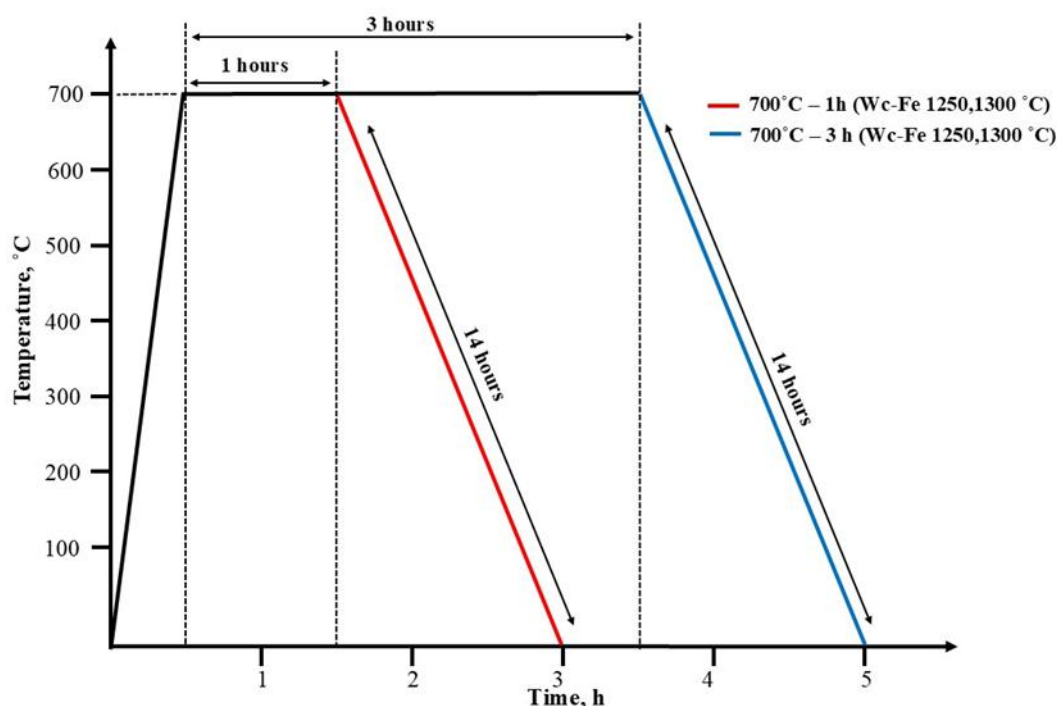


Fig 1. Schematic illustration of the heat treatment (annealing) procedure applied to WC-Fe composites after SPS at 1250°C and 1300°C. The samples were heated to 700°C, held for 1h or 3h, and subsequently cooled inside the furnace.

To evaluate the effect of annealing duration at 700°C on the elemental composition of WC-Fe samples fabricated by SPS at 1250°C, energy-dispersive X-ray spectroscopy (EDS) analysis was performed. The elemental distribution maps presented in Figures 2c, 2q, 2k, and 2o indicate significant microstructural changes after heat treatment 1h or 3h.

Previous study [16] demonstrated that during annealing of the W/Fe (cast iron) system within the temperature range of 1085-1125°C, the thickness of the WC-Fe layer increases according to a parabolic growth law with an activation energy of approximately 184kJ/mol, indicating a diffusion-controlled growth mechanism. In the present study, despite the considerably lower annealing temperature of 700°C applied to SPS-consolidated WC-Fe samples sintered at 1250°C, a similar diffusion-assisted microstructural rearrangement was observed. In particular, increasing the annealing time from 1h or 3h resulted in a decrease in W content accompanied by an increase in Fe and C contents according to the EDS results. Despite the reduction in tungsten concentration with prolonged annealing, the overall compositional trend remained relatively stable, as illustrated in Figures 2d and 2h.

However, samples 3 and 4, sintered by SPS at 1300°C and subsequently annealed at 700°C for 1h and 3h, exhibited more pronounced growth of WC grains compared with the samples sintered at 1250°C, as shown in Figures 2j and 2n, indicating the dominant role of the carbide phase in the microstructure.

According to the literature [16], the growth of the ceramic WC phase is governed by the diffusion of both carbon and tungsten atoms. Carbon atoms can diffuse perpendicular to the tungsten phase, while tungsten atoms diffuse in the opposite direction. Furthermore, the interfacial reaction kinetics are considerably faster than the diffusion kinetics of carbon within the WC phase under annealing conditions. From a kinetic perspective, WC formation during isothermal annealing is therefore controlled predominantly by diffusion processes.

A higher fraction of carbide precipitates was observed in the WC-Fe sample sintered at 1250°C and annealed for 3h, where the carbide content reached 13.37%, while the fraction of WC particles decreased to

77.29%. This behavior suggests redistribution of the constituent elements and ongoing diffusion-driven phase transformations during annealing.

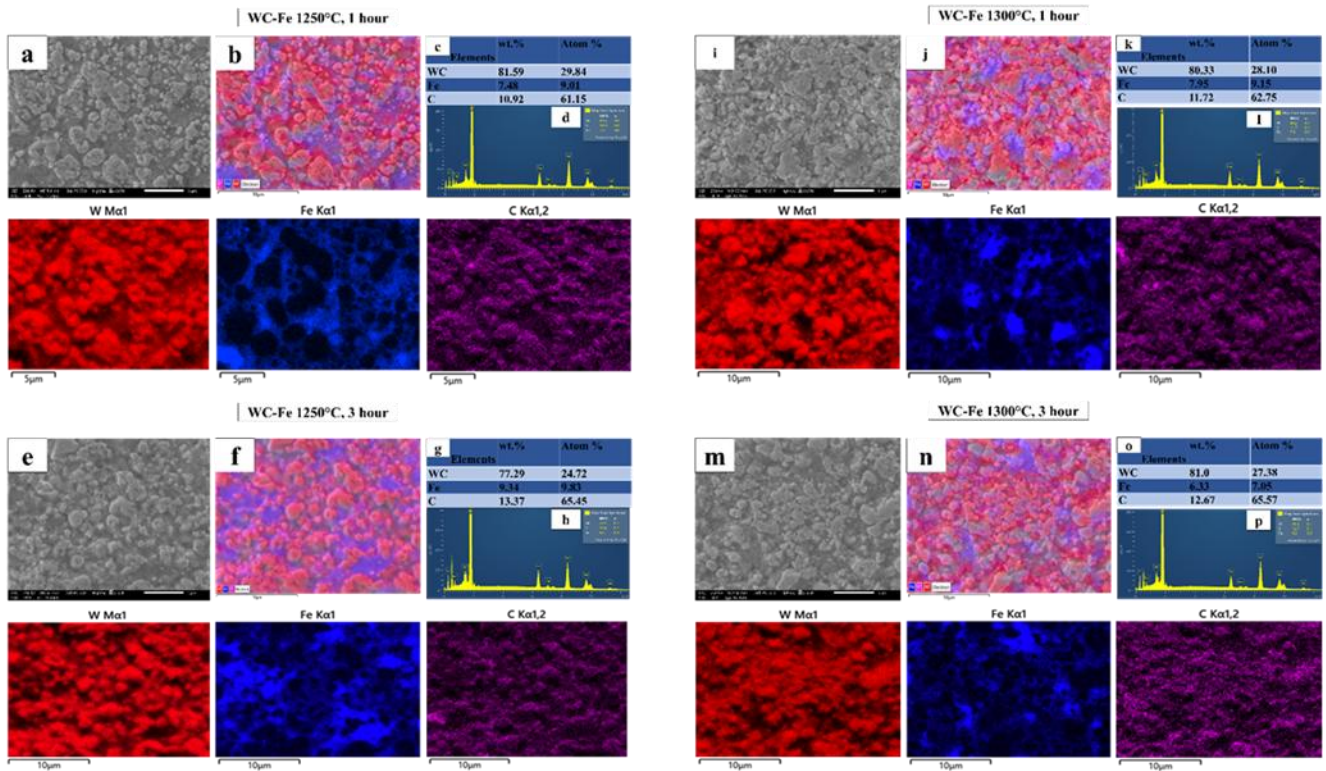


Fig 2. SEM micrographs, EDS elemental mapping, energy-dispersive X-ray spectra, and quantitative elemental composition of WC-Fe alloys after SPS and subsequent heat treatment at 700°C.

(a-d) sample sintered at 1250°C and annealed for 1h;

(e-h) sample sintered at 1250°C and annealed for 3h;

(i-l) sample sintered at 1300°C and annealed for 1h;

(m-p) sample sintered at 1300°C and annealed for 3h.

The SEM images illustrate the microstructural morphology and degree of densification after heat treatment. The EDS elemental maps show the distribution of W, Fe, and C across the sample surfaces. The accompanying tables present the weight and atomic concentrations of the constituent elements, while the EDS spectra confirm the presence of the principal components of the WC-Fe system.

The microhardness values of the WC-Fe alloys after heat treatment at 700°C for 1h and 3h are summarized in Table 1. The results demonstrate that increasing the annealing time leads to a gradual increase in microhardness for both sintering temperatures. For the samples sintered at 1250 °C, the microhardness increased from 1260 ± 0.1 HV_{0.2} after 1 h of annealing to 1273 ± 0.45 HV_{0.2} after 3 h. A similar tendency was observed for the samples sintered at 1300 °C, where the microhardness increased from 1510 ± 0.4 HV_{0.2} to 1525 ± 0.7 HV_{0.2} with increasing annealing duration. The higher microhardness values observed for the samples sintered at 1300°C indicate the beneficial effect of improved densification and a more developed carbide framework formed during SPS.

Table 1. Microhardness of WC-Fe alloys subjected to annealing at 700°C (heating rate: 10°C/min; holding time: 1h and 3h)

Sample	WC	Binder type	Temperature °C	Holding time, (h)	Microhardness, HV
Alloy sintered at 1250°C	88 wt.%	12 wt.% Fe	700	1	1260±0.1
Alloy sintered at 1250°C	88 wt.%	12 wt.% Fe	700	3	1273±0.45

Alloy sintered at 1300°C	88 wt.%	12 wt.% Fe	700	1	1510±0.4
Alloy sintered at 1300°C	88 wt.%	12 wt.% Fe	700	3	1525±0.7

The subsequent heat treatment further contributes to microstructural stabilization and diffusion-assisted redistribution of the constituent elements within the binder phase. In addition, the undissolved WC particles act as effective barriers to dislocation motion and grain boundary migration, resulting in dispersion strengthening of the material. The highest microhardness value, approximately 1525±0.7 HV_{0.2}, was obtained for the WC-Fe alloy sintered at 1300°C and annealed at 700°C for 3h, indicating the beneficial combined effect of elevated SPS temperature and post-sintering heat-treatment on the mechanical performance of the cobalt-free hard alloy.

4. Conclusions

In the present study, the morphology and mechanical properties of cobalt-free WC–Fe cemented carbides produced by spark plasma sintering (SPS) followed by subsequent heat treatment were investigated, and the influence of processing parameters on the microstructural evolution and material properties was evaluated. Based on the obtained results, the following conclusions can be drawn:

It was established that both the SPS temperature and subsequent heat treatment have a significant influence on the microstructure and microhardness of WC–Fe alloys. Annealing at 700°C resulted in a further increase in microhardness, which can be attributed to diffusion processes and microstructural rearrangement occurring during heat treatment. For samples sintered at 1250°C, increasing the annealing time from 1h to 3h increased the microhardness increased from 1510±0.4 to 1525±0.7 HV_{0.2} under the same heat treatment conditions, followed by furnace cooling for 14h.

The obtained results confirm the decisive role of SPS processing conditions and subsequent heat treatment in controlling the microstructural evolution and mechanical properties of cobalt-free WC-based cemented carbides. The WC-Fe system exhibited high structural stability and improved mechanical performance, demonstrating the potential of Fe binders as an alternative to conventional cobalt-containing cemented carbides for cutting and wear-resistant tool applications.

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