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Effect of heat-treatment of in-situ synthesized composite powder on properties of sintered cemented carbides

C.B. Wei, X.Y. Song*, J. Fu, X.M. Liu, Y. Gao, H.B. Wang, S.X. Zhao

College of Materials Science and Engineering, Key Lab of Advanced Functional Materials, Education Ministry of China, Beijing University of Technology, Beijing 100124, China

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ABSTRACT

The present study was focused on the subsequent heat-treatment of the in-situ synthesized WC–Co composite powder and its effect on the properties of the cemented carbides prepared by sinter-HIP. The element composition, phase constitution, microstructure characteristics and properties of the prepared cemented carbides were analyzed quantitatively. The heat-treatment of the composite powder before sintering plays a significant role in the characteristics of the microstructure and properties of the WC–Co bulk material. The optimized heat-treatment parameters for the composite powder were obtained, from which the excellent combination mechanical properties were achieved for the sintered WC–Co bulk material.

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1. Introduction

Cemented tungsten carbide has many industrial applications due to the much desired properties of high hardness and good wear resistance. It is widely used in metalworking drilling and mining industries under high pressure, high temperature, and corrosive environments. It's hardness, compressive strength, and transverse rupture strength (TRS) depend on the composition and the microstructure [1–3]. Substantial investigations have shown that the refinement of WC grain size improves significantly the mechanical properties for the cemented tungsten carbide. Thus the productions of ultrafine/nanoscale WC or WC–Co composite powder are crucial [4–8]. With regard to the synthesis of ultrafine/nanoscale WC or WC–Co composite powder, there has also been a large amount of research and significant technology developments to produce the ideal powders [9–12]. Recently, our group proposed a novel rapid method for preparing ultrafine WC–Co composite powder by the in-situ solid-state reactions in vacuum [13]. In this approach, metal oxides together with suitable amount of carbon black were used as the raw powders, and the ultrafine WC–Co composite powder can be obtained in a single-step procedure through the in-situ reduction and carbonization reactions.

While numerous techniques for producing the ultrafine/nanoscale WC or WC–Co composite powder have been developed, only a few have been proven at the industrial level for economic considerations. There are two drawbacks of the ultrafine or nanoscale powders. One is the aggregation of the fine particles. The other is the high chemical activity due to the very large specific surface. The surface of the ultrafine/nanoscale WC or WC–Co composite powder may contain oxygen or other impurities in the surface oxide film and in adsorbed water [14]. The ultrafine WC–Co composite powder prepared by in-situ solid-state reactions may contain excess free carbon co-existing with the carbon-lacking intermediate phases (e.g. η phase) [13]. The above factors may affect obviously the phase constitution and microstructure so as to the properties of the finally sintered WC–Co bulks. In order to obtain the superior properties in the bulk material, it is very important to study and control the excess free carbon, η phases and oxygen in the composite powder before the sintering process.

In the present work, efforts were focused on the heat-treatment of the in-situ synthesized composite powder which contains free carbon, η phase and oxygen, followed by the preparation of WC–Co bulk specimens by sinter-HIP. The effects of heat-treatment for the in-situ synthesized composite powder on the chemical elemental compositions content, phase constitutions, the microstructure characteristics and various properties of the sintered WC–Co bulk material were analyzed quantitatively. The optimized heat-treatment temperature of in-situ synthesized composite powder was proposed according to the excellent combination mechanical properties of the sintered WC–Co bulk, especially the high TRS.

2. Experimental

2.1. Preparation of WC–Co composite powder

Take WC–10 wt% Co bulk as the target material in the present study. The WC–Co composite powder was synthesized by in-situ

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* Corresponding author. Tel./fax: +86 10 67392311.
E-mail address: xysong@bjut.edu.cn (X.Y. Song).

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reduction and carbonization reactions of metallic oxides and carbon black [13]. The detailed procedures are described as follows: the blue tungsten oxide ($WO_{2.9}$) powder with 99.5% purity, cobalt (II, III) oxide (Co_3O_4) powder with 98.5% purity and carbon black powder with 99.5% purity were used as the raw materials. The 0.20 wt% vanadium carbide and 0.80 wt% chromium carbide were added into the raw powders as the grain growth inhibitors. The raw powders were milled for 20 h in a planetary mill (Planetary Milling 20 L) using pure ethanol as the liquid medium. The balls with a diameter of 8.0 mm were made of cemented carbides to avoid the introduction of impurities during ball milling. The ball-to-powder weight ratio was 3:1 and the rotation speed was 180 rpm. Subsequently, the as-milled powder mixture was put into the vacuum furnace and heated up to 1000 °C with a heating rate of 15 °C/min and held for 3 h at this temperature to perform the in-situ reduction and carbonization reactions. The synthesized composite powder was then refined by ball milling for 10 h.

2.2. Processing of WC–Co composite powder and its densification conditions

The in-situ synthesized composite powder was loaded in a vacuum tube furnace at a desirable temperature range for 60 min in the Ar atmosphere with the heating and cooling rates of 6 °C/min. The powders were mixed with 1.50 wt% polyethylene glycol (PEG 1500), which was used as the forming binder, and compressed to form a number of compacts. The green compacts were dewaxed in vacuum at 400 °C for 60 min then densified by the sinter-HIP technique with a heating rate of 8 °C/min and sintering at 1450 °C for 60 min, during this period there was an isothermal holding for 30 min which was performed in the Ar atmosphere with a gas pressure of 5 MPa. Prior to the isothermal holding with Ar pressure of 5 MPa, the specimens were presintered in the vacuum environment.

2.3. Characterizations of composite powder and bulk materials

The contents of the free carbon and total carbon contained in the powder were measured by the gravimetric method according to the international standards ISO 3908-1985 and ISO 3907-1985, respectively. The composition of the powder was examined by the chemical analyses. The phases in the composite powder and the sintered bulk specimens were detected by X-ray diffraction (XRD) with Cu Kα radiation. The thermal gravimetric-differential scanning calorimeter (TG/DSC) measurements of the composite powder were performed with a NETZSCH STA 449C thermogravimetric analyser at a heating rate of 10 °C/min. The morphology of the composite powder and the grain structure of the sintered bulk were observed by the field-emission scanning electron microscope (FESEM, JSM 6500, JEOL). The WC grain sizes were measured with the linear intercept method performed on a series of SEM micrographs. The microstructure details were studied by the transmission electron microscopy (TEM), which was carried out with the JEOL JEM-3010 operated at 300 kV.

The density of the sintered bulk specimens was measured by the Archimedes method. The hardness was measured by the Vickers hardness tester with a load of 30 kg according to the ISO-3878 standard. The fracture toughness was determined based on the measurements on the length of cracks generated by the Vickers indentation and calculations with the equation $K_{IC}=0.0028(HvP/L)^{1/2}$ [15,16], where Hv is the indentation hardness, P is the indentation load, and L is the total crack length. The TRS was measured on the prismy and polished specimens with dimensions of 20 mm × 6.5 mm × 5.25 mm according to the standard of ISO3327:2009. At least five specimens were prepared for the measurements of each property.

3. Results and discussions

3.1. The in-situ synthesized WC–Co composite powder

The XRD analysis of phase constitutions and the morphology of the in-situ synthesized WC–Co composite powder are shown in Fig. 1. It is seen from Fig. 1(a) that the composite powder consists of mainly the WC and Co phases, and a small amount of the complex carbonized compound, η (Co_6W_6C) phase, exists in the composite powder. Combine the results shown in Fig. 1(a) and the composition analysis in Table 1, it is known that the dominant phases in the in-situ synthesized composite powder are WC and Co. It indicates that the tungsten oxide and cobalt oxide were completely reduced during the in-situ reactions. Moreover, the particle sizes in the composite powder distribute mainly in a narrow range of 150–250 nm (see inset Fig. 1(b)) and the mean particle size is obtained

Table 1
Composition analysis of the in-situ synthesized and heat-treated WC–10 wt% Co composite powder at different temperatures (wt%).

Temperature	Composition				
	W	Co	Total carbon	Free carbon	Oxygen
In-situ synthesized	83.55	10.03	5.65	0.280	0.48
850 °C	83.63	10.32	5.59	0.160	0.29
900 °C	83.80	10.11	5.56	0.140	0.28
950 °C	83.44	10.38	5.45	0.048	0.29

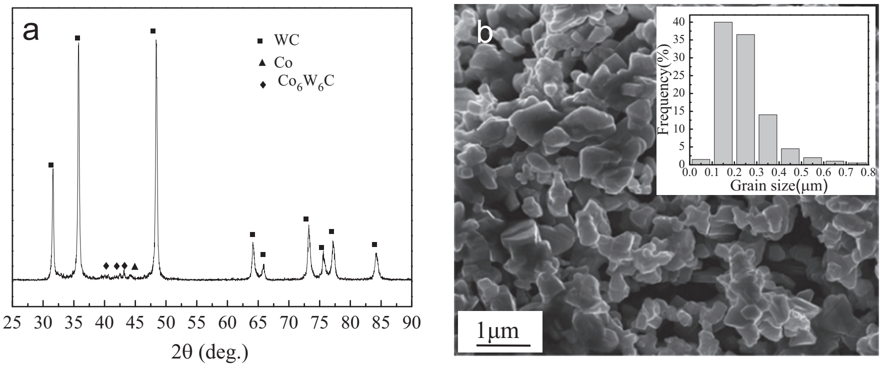

Fig. 1. (a) XRD analysis of phase constitutions; (b) SEM micrograph of the morphology of the in-situ synthesized composite powder.

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