Int. Journal of Refractory Metals and Hard Materials 58 (2016) 137–142

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

Microstructure and mechanical properties of functionally gradient cemented carbides fabricated by microwave heating nitriding sintering

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ARTICLE INFO

Article history:
Received 1 December 2015
Received in revised form 21 March 2016
Accepted 26 April 2016
Available online 27 April 2016

Keywords:
Functionally graded material
Cemented carbides
Microstructure
Mechanical properties
Microwave sintering
Nitriding

ABSTRACT

A new method is presented for the fast preparation of functionally graded cemented carbide materials by microwave heating nitriding sintering. The influence of composition and sintering temperature on the mechanical properties, microstructure, and phase composition of the materials was studied. Results showed that functionally graded cemented carbides with the desired mechanical properties can be obtained rapidly by microwave heating nitriding sintering. A gradient layer with a Ti(C, N)-enriched surface layer, and underneath a Co-enriched layer formed on the top of the hard alloy substrate. The nitriding process had little effect on the microstructure of the matrix. A lower surface roughness, and the similar layer thickness as seen in conventional heating nitriding was obtained by microwave heating nitriding sintering in a short period of time. The thickness of the gradient layer increased with increasing temperature. The high Ti content in the raw material was beneficial to the formation of the gradient layer; however, the Co content had little effect on the gradient layer thickness when it increased from 6% to 10%.

1. Introduction

Cemented carbide is an advanced composite material composed of: WC, Co, TiC, TaC, NbC, Mo_2C, etc., prepared, typically, by powder metallurgy, including mixing, drying, pressing, and sintering. It has good overall mechanical properties, is widely used in tools, moulds, drilling equipment, and cutting tools, but under some onerous working conditions, such as high-speed machining, the low wear resistance limits its application. A functionally graded, wear-resisting layer formed on the surface of cemented carbide to form functionally graded cemented carbide (FGCC) can effectively improve the cutting performance of a cutting tool [1–6]. At present, the functionally graded layer is mainly prepared through an in situ diffusion nitriding sintering process. The sintering process is primarily conducted in a traditional resistance furnace, in which the heat flow goes from the surface to the center. The binder melts at the surface during the nitriding process blocked the channel for outgassing, leading to a cessation of the densification process of the materials [7], thereby significantly reducing the performance thereof. To improve its mechanical properties, functionally graded cemented carbides need to be processed by vacuum sintering [3,8–10] or hot isostatic pressing process [3] to increase the density of material before or after nitriding sintering: the process is time-, and energy-consuming.

Microwave heating imparts rapid, selective, volumetric heating [11]. Compared with traditional resistance heating, microwave heating can reduce time and energy consumption, improve the material performance, is more environmental friendly, etc. [12]. More importantly, the heat flow direction of microwave heating is inside-out [13], this provides a potential means of preparing functionally graded cemented carbide material by combined sintering and nitriding from billet by microwave heating. However, the ability to absorb microwave energy of most materials at low temperature is low [12], so most of the microwave heating processes use SiC as an auxiliary heating medium to form a hybrid heating process [14–16], but this can cause a loss of energy and damage of the heat insulation material. Willert-Porada and Rödiger, et al. [16] reported the fabrication of hardmetal by microwave sintering. The result shows that microwave sintering can obtain a finer microstructure than traditional sintering. They also [17,18] report a method of fabricating functionally graded cemented carbides by microwave heating reactive sintering, which deals with sintering and nitriding simultaneously; but they used microwave hybrid heating sintering by using SiC for auxiliary heating. According to Luo, et al. [19], the pore length (L_p) of commercial pure Ti fabricated from TiH_2 by pure microwave sintering is half that fabricated from hydride–dehydride (HDH) Ti by microwave hybrid heating sintering, and the former has a higher density and tensile strength which show the acceleration of pure microwave sintering on material shrinkage behaviour.

Here, a new method for the preparing of functionally gradient cemented carbides tool materials by microwave heating nitriding sintering is presented. It includes two basic processes as follows: firstly,

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http://dx.doi.org/10.1016/j.ijrmhm.2016.04.013
0263-4368/

the rarefied nitrogen was used to form a plasma under microwave irradiation at low temperature. The radiant heat from the plasma was used to replace the heat produced by the SiC, which is currently commonly used in microwave heating as an auxiliary heating body which can accelerate the heating process. Secondly, pure microwave heating was achieved by adjusting the nitrogen pressure to suppress the plasma at higher temperatures; the densification and nitriding processes were conducted simultaneously on this material, therefore a high-performance functionally graded cemented carbide was formed. The combination of these two steps can avoid the material surface overheating due to the skin effect produced by microwave plasma that Willert-Porada and Rödiger reported [20], and obtain excellent mechanical properties of the final sintered body. The heating mechanism governing microwave heating nitriding sintering methods at different temperatures is shown in Fig. 1. At low temperature, the temperature inside the material is constant, the material undergoes no obvious shrinkage, and its porosity distribution is uniform. At high temperatures, pure microwave heating is achieved, the opposite temperature gradient is established, i.e. the internal temperature of the material exceeds that of the exterior, internal material shrinks before the external material, and porosity migrates along the yellow arrow (Fig. 1). When the surface temperature reached the sintering temperature, the pores moved further along the yellow arrow and out of the material: the material was thus densified.

Based on the method, this research prepared functional gradient cemented carbide cutting tool materials, investigated the effect of sintering temperature and alloy composition on the mechanical properties, formation and phase composition of the gradient layers.

2. Experimental methods

Cemented carbides samples from an industrial-grade mixture of WC, Co, and TiC were prepared by standard powder metallurgy methods. The nominal composition of the cemented carbides used here is given in Table 1. The green compacts were sintered in a vacuum sintering furnace heated to 400 °C for 1 h to remove the wax. The experimental apparatus is that used elsewhere [21]. The experiment was conducted in a microwave oven with an operating frequency of 2.45 GHz. Fig. 2 shows the typical temperature curve for preparation of FGCC, changed by controlling the input power, so giving different heating rates. To realise simultaneous sintering and nitriding, after a brief cryogenic vacuum degassing process, nitrogen, at a pressure of 0.02 MPa and a purity of 99.9% was saturated at about 300 °C. And the pressure was increased to 0.08 MPa upon reaching the required sintering temperature. The required sintering temperature ranged from 1380 °C to 1460 °C, and the soaking time was 15 min. The strength and surface hardness were evaluated by three-point bending test and a Vickers hardness tester, respectively. The surface roughness was measured by a Surf-gauge (Mahr, German). After sintering, the samples were cut, embedded in resin, and polished. The microstructures of the polished specimens were observed in cross-section by electro-probe micro-analyser (EPMA, JEOL Corp., Japan) in back-scattered-electron (BSE) mode. The phase compositions were evaluated by an X-ray diffractometer (XRD, Bruker Corp., Germany).

Table 1
Nominal composition of samples (wt.%).

Samples	WC	TiC	Co
FGCC-T5	Balance	5	10
FGCC-T15	Balance	15	6

3. Results

3.1. Strength and hardness

Fig. 3 shows the mechanical properties of the sintered FGCC samples. With increasing sintering temperature, the strength and hardness of FGCC-T5 and FGCC-T15 tended to decrease after an initial increase. The strength and hardness of FGCC-T5 were maximised, when sintered at 1400 °C for 15 min, at 2010.2 MPa and HV1580, respectively; however, the strength and hardness of FGCC-T15 were maximised, when sintered at 1430 °C for 15 min, at 1560 MPa and HV1835, respectively. Differences in composition result in differences in sintering temperature and mechanical properties. In addition, a matrix with more pores at low temperatures, upon being sintered, underwent grain coarsening and the volatilisation of its binding phase at high temperature, which caused the aforementioned mechanical properties changes.

3.2. Gradient structure and phase composition

Fig. 4 shows the typical cross-sectional view of the microstructure of FGCC-T5 sintered at 1400 °C for 15 min. A grey surface layer, about 10 μm thick was formed at the homogeneous bright white and grey matrix in EPMA/BSE mode (Fig. 4(a)). Combining elemental analyses by EPMA, it may be seen that a graded layer, rich in titanium and nitrogen, had formed on the surface of the matrix, in which the tungsten content was lower than that of the matrix (Fig. 4(b), (c), and (e)). A Co-enriched layer was located at the bottom of the graded layer adjacent to the matrix (Fig. 4(d)). From Fig. 4(f), the carbon content of the graded layer was greater than that of the substrate.

Fig. 5 shows the typical cross-sectional microstructure of FGCC-T15 sintered at 1430 °C for 15 min. The cross-sectional morphology of FGCC-T15 was similar to that of FGCC-T5, but the thickness of its surface

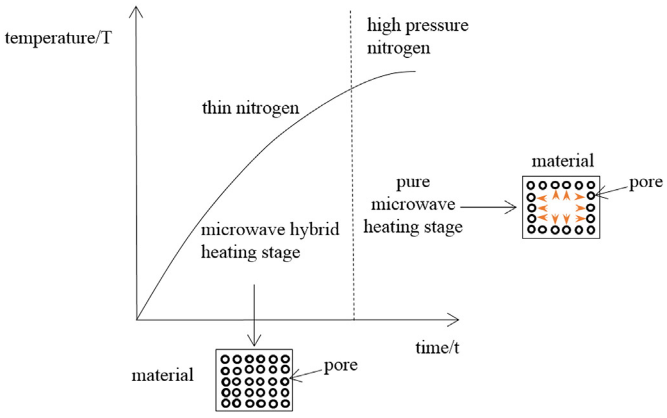

Fig. 1. Schematic diagram of heating mechanism underpinning the microwave heating sintering nitriding method at different temperatures.

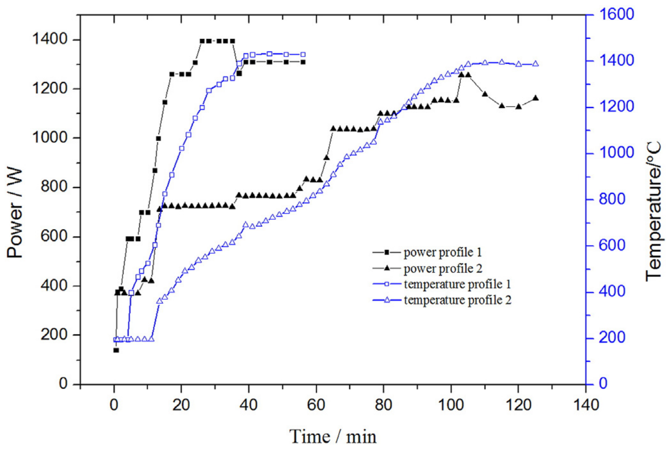

Fig. 2. Typical temperature curve.