Scripta Materialia 120 (2016) 103–106

Contents lists available at ScienceDirect

Scripta Materialia

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

Regular Article

Microwave-assisted synthesis of vanadium and chromium carbides nanocomposite and its effect on properties of WC-8Co cemented carbides

Zhiwei Zhao

College of Materials Science and Engineering, Henan University of Technology, Zhengzhou 450001, China

ARTICLE INFO

Article history:
Received 13 March 2016
Accepted 16 April 2016
Available online 27 April 2016

Keywords:
Cemented carbides
Grain growth inhibitor
Microwave sintering
Microstructure
Properties

ABSTRACT

A new method of in-situ microwave heating is used to prepare vanadium and chromium carbides (V_8C_7-Cr_3C_2) nanocomposite and their alloys. The as-prepared nanocomposite was used as the grain growth inhibitor of cemented tungsten carbides. Well dispersed V_8C_7-Cr_3C_2 nanocomposite with a mean diameter of about 30 nm can be synthesized at lower temperature. The prepared nanocomposite has an important effect on the microstructure and properties of WC-8Co alloys. The microstructure of WC-8Co-0.8(V_8C_7-Cr_3C_2) alloys is more uniform than that of WC-8Co alloys. Vickers hardness and fracture toughness of the former can be increased by about 7% compared with those of the latter.

The transition metal carbides have some superior properties such as high melting points and hardness, extreme corrosion and wear resistances, excellent electronic characteristics and good catalytic properties [1–3]. Therefore, they have wide uses in metallurgy, electronics, catalysts, etc [4]. Particularly, vanadium carbide (V_8C_7) shows very high melting point (2810 °C), good wear resistance and high mechanical hardness. Chromium carbide (Cr_3C_2) has high melting point (1890 °C), good strength and high corrosion properties [5–7]. Research shows that V_8C_7 and Cr_3C_2 are the most effective grain growth inhibitors of cemented tungsten carbides, owing to their appreciable solubility and mobility in liquid cobalt at lower sintering temperatures [8]. However, single V_8C_7 (Cr_3C_2) or the mixture of V_8C_7 and Cr_3C_2 cannot effectively inhibit the growth of WC grains. V_8C_7-Cr_3C_2 nanocomposite combines the advantages of V_8C_7 and Cr_3C_2, and can effectively inhibit the growth of WC grains and improve the mechanical properties of cemented carbides [9,10]. In addition, the grain growth can be inhibited to some extent by using some rapid sintering technologies, such as rapid hot pressing sintering, microwave sintering and spark plasma sintering (SPS). These sintering technologies can obviously accelerate the heating rate, increase the densification rate, decrease the sintering temperature and shorten the holding time [11].

Commonly, carbide powders are synthesized by carbon thermal reduction of micron-sized oxides and carbon. This method has some disadvantages such as a high reaction temperature (>1400 °C), a long reaction time (>4 h) and a coarse-grained level (μm-range) [12]. Presently, carbide powders have been successfully synthesized by other methods, e.g. direct element reaction [13], mechanical alloying [14,15], temperature programmed reaction [16] and gas reduction-carburization [17]. However, industrial applications of these methods are still limited due to the low yields, agglomeration problems, wide size distributions, complex monitoring and high costs.

In this study, V_8C_7-Cr_3C_2 nanocomposite was prepared by an in-situ microwave heating method at and nanometer carbon black were used as raw materials, which exhibit a higher specific surface area and activity. Thus the reaction temperature and time can be reduced and shortened, respectively. Furthermore, microwave heating is a novel heating technology with higher effectiveness than the traditional heating method. It generates first heat within the material and then heats the entire volume. This heating mechanism is advantageous due to uniform, rapid, high reaction rates and selectivity, dramatically reduced reaction times, and high product-yields [18–20]. As far as we know, V_8C_7-Cr_3C_2 nanocomposite was firstly synthesized by the method. Besides, effect of the prepared nanocomposite on the microstructure and properties of WC-8Co cemented carbides prepared by the microwave heating method was also firstly researched.

Nanometer V_2O_5 (average particle size < 50 nm), nanometer Cr_2O_3 (average particle size < 60 nm) and nanometer carbon black (average particle size < 50 nm) were used as raw materials. 68 wt.% oxides (V_2O_5 and Cr_2O_3) and 32 wt.% C were put into a ball milling pot according to the reaction equation between oxides and C ($4V_2O_5 + 20C = V_8C_7 + 20CO\uparrow$, $3Cr_2O_3 + 13C = 2Cr_3C_2 + 9CO\uparrow$), and the milling medium was absolute ethyl alcohol. After being milled for 24 h, the mixture was dried at 90 °C for 12 h. Finally, the mixture was heated at different temperatures (900 °C, 1000 °C, 1100 °C and 1200 °C) in a multimode 2.45 GHz RWS microwave furnace (Zhongsheng Thermal Technology Co., Ltd., China) in argon gas atmosphere to prepare V_8C_7-Cr_3C_2 nanocomposite. The prepared V_8C_7-Cr_3C_2 nanocomposite (0.8 wt.%) was

E-mail address: zhiwei_zhao@haut.edu.cn.

http://dx.doi.org/10.1016/j.scriptamat.2016.04.024
1359-6462/

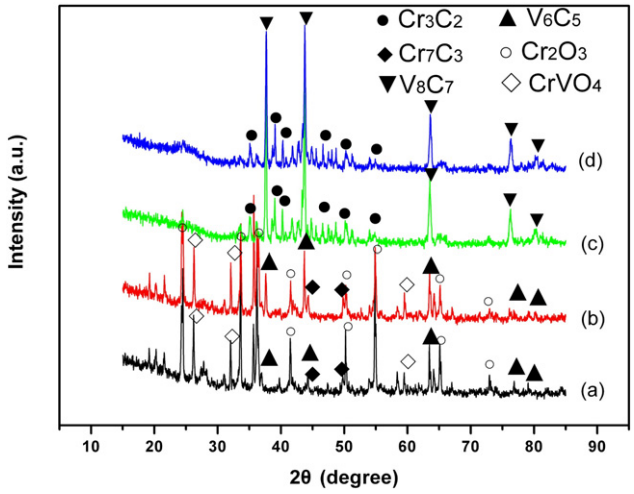

Fig. 1. X-ray diffraction patterns of the nanocomposite synthesized by microwave heating at different temperatures for 1 h: (a) 900 °C; (b) 1000 °C; (c) 1100 °C; (d) 1200 °C.

mixed with WC (purity > 99.9%, average particle size < 200 nm) and 8 wt.% Co (purity > 99.9%, average particle size < 50 nm) powders by wet mixing technology. The mixture was dried at 95 °C for 10 h after it being milled for 24 h. The compacts were shaped into cylinder and sintered in the mentioned above microwave furnace in argon gas atmosphere.

The structure of the powder and the cemented carbides samples was examined at room temperature via X-ray diffraction (X'Pert Powder, Philips, Netherlands). Particle morphology and size of the synthesized powders were observed by JEM-1000CX transmission electron microscopy (TEM). The microstructure of the cemented carbides samples was observed by JSM-6700F scanning electron microscopy (SEM). The bulk density was measured according to the Archimedes principle. Hardness (HV_{30}) measurements were carried out using a HVS-50 Vickers hardness tester (Shanghai material testing machine factory) at a load of 300 N.

The fracture toughness (K_{IC}) of the alloys was determined according to the following equation [21]:

$$K_{IC} = 0.15\frac{HV_{30}}{\sum l} \quad (1)$$

where K_{IC} is the fracture toughness (MPa $m^{1/2}$), HV_{30} is the Vickers hardness (GPa) at a load of 300 N, $\sum l$ is the sum of the lengths of the crack tip from the hardness indent (mm).

Fig. 1 shows the XRD patterns of V_8C_7-Cr_3C_2 nanocomposite synthesized by microwave heating method at different temperatures for 1 h. As shown in Fig. 1(a) and (b), the peaks are identified as Cr_2O_3 (JCPDS 38-1479), $CrVO_4$ (JCPDS 38-1376), V_6C_5 (JCPDS 65-9718) and Cr_7C_3 (JCPDS 11-0550), indicating that the oxidation-reduction reactions between oxides and carbon have not completed under these conditions. When the temperature reaches 1100 °C, the product is mainly composed of V_8C_7 (JCPDS 35-0786) and Cr_3C_2 (JCPDS 35-0804), indicating that the carbothermal-reduction reactions among V_2O_5, Cr_2O_3 and C have completed at 1100 °C for 1 h, and the reactants are V_8C_7 and Cr_3C_2, as shown in Fig. 1(c). The synthesis temperature and time required by this approach are at least 300 °C and 3 h lower and shorter than those of the conventional method [12], respectively. Furthermore, the synthesis temperature is at least 100 °C lower than that of the precursor method [22]. Compared with the methods mentioned above, the current method has the advantages of lower reaction temperature and shorter reaction time, which can be attributed to the combination of nanoscale raw materials and microwave heating technique [20,23]. V_6C_5 (JCPDS 65-9718), V_8C_7 (JCPDS 35-0786), Cr_7C_3 (JCPDS 11-0550) and Cr_3C_2 (JCPDS 35-0804) may be regarded as substoichiometric carbides [24], yielding a range of site occupancy defects that control the phase stability via specific ordering of vacancies which can influence

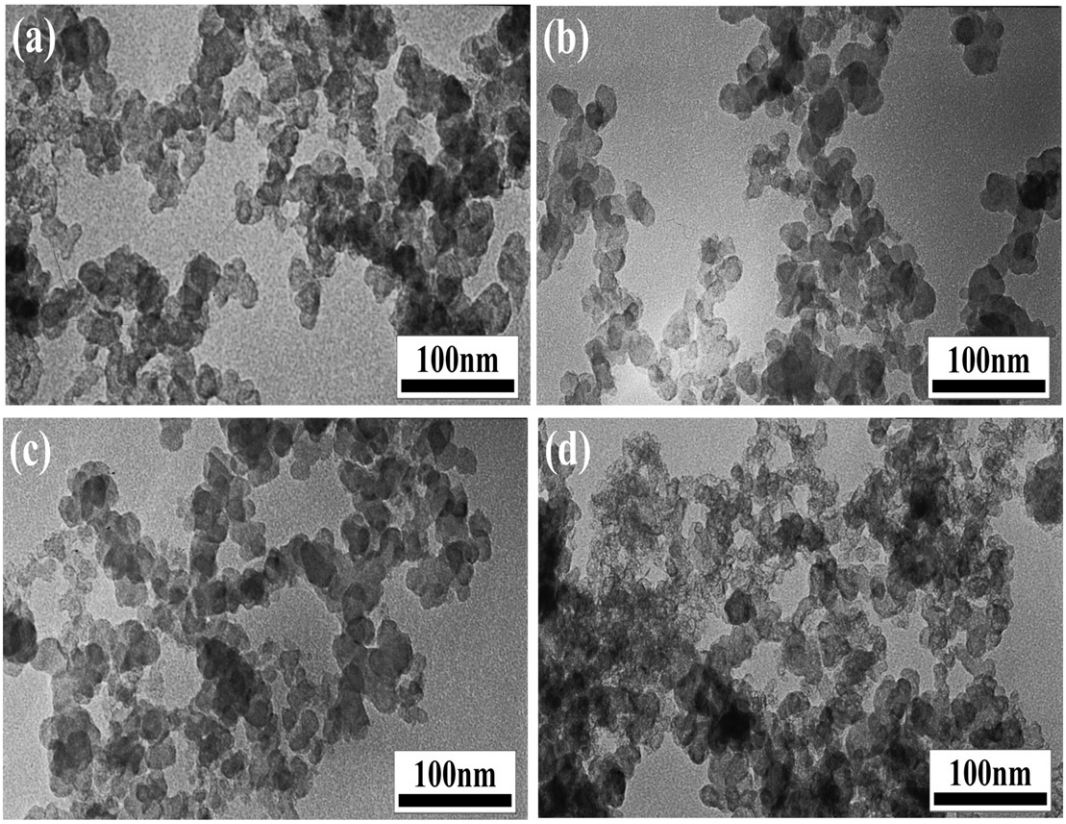

Fig. 2. TEM micrographs of the nanocomposite obtained by microwave heating at different temperatures for 1 h: (a) 900 °C; (b) 1000 °C; (c) 1100 °C; (d) 1200 °C.