

Developing (W,Ti)C-(Ni,Co) nanocomposite by SHS method

Vahid Balouchi^a, Fariborz Sharifian Jazi^a and Ali Saidi^b

^aYoung Researchers and Elite Club, Najafabad Branch, Islamic Azad University, Najafabad, Isfahan, Iran

^bMaterials Engineering Department, Isfahan University of Technology, Isfahan 84154-83111, Iran

(W,Ti)C-(Ni,Co) nanocomposite with various W/Ti weight ratios were produced by self-propagating high temperature synthesis (SHS) method. The effect of the W/Ti weight ratio of the initial mixture on the microstructure and carbide morphology of the final product was investigated. Experimental results indicate that (W,Ti)C particles exhibit fine size and homogeneous distribution in (Ni,Co) matrix, and solution of W in TiC is proved by X-ray diffraction (XRD) and SEM-EDS analysis. Moreover, with the increasing of W/Ti weight ratio, rounding and growth of the hard phase particles are observed.

Key words: (W,Ti)C-(Ni,Co) nanocomposites, Self-propagating high temperature synthesis (SHS), Microstructure.

Introduction

Ceramics have intrinsic characteristics like high melting point, high hardness, excellent wear resistance and no chemical reactivity which have resulted from their wide applications as functional materials at elevated temperatures. Today, advanced ceramics are used in various fields including cutting tools, extrusion molds and many high-temperature engine parts and so on [1-7].

TiC- and Ti (CN)-based cermets have been used as cutting tool materials because of their attractive mechanical properties [8]. These cermets typically have a core-rim structure. The cores are partially dissolved raw TiC or Ti (CN) particles, on which the rim structure grows via a dissolution/precipitation process. The rim phase, a (Ti,W,M)C- or (Ti,W,M)(CN) type solid solution, is known to improve the toughness of the cermets and has drawn much attention of researchers in this field [9, 10]. In addition, homogeneous (Ti,W)C or (Ti,W)(CN) cermets have been demanded by hard material industries since the 1980s [8, 9].

Many attempts have been made to synthesize of (Ti,W)C solid solution powders via the thermal reaction of a TiC/WC powder mixture at high temperatures or by the self-heat propagation synthesis (SHS) of a Ti, W and C powder mixture [11-13]. TiC is thermodynamically more stable than WC. (Ti,W)C is synthesized commercially by reacting TiC with WC in the temperature range of 1700-2100 °C for periods ranging from 10 to 20 h [11]. This method is expensive and always results in inhomogeneous powders with unreacted TiC as cores. Similar results were reported when (Ti, W) C was synthesized through SHS

[13]. It has been reported that homogeneous (Ti, Mo) C powders were produced by milling mixtures of elements such as Ti, Mo and C [14]. This approach involves production of nanosized powders. Thereby increasing the quality of (Ti,W)C ceramics powders. In this investigation synthesis of (Ti,W)C-Ni-Co composite by the planetary milling of a powders mixture is reported. The microstructure and mechanical properties of the sintered carbides were evaluated.

Experimental

Materials

The raw materials, Ti (> 99.9%, < 80 μm), W (> 99.9%, < 15 μm), Co (> 99.9%, < 2 μm), Ni (> 99.9%, < 100 μm) and C (> 99%, < 10 μm) powders, were mixed. Therefore the nanocomposite with different weight ratios of tungsten to titanium were produced.

Instruments

The mixed powders were then subjected to ball-milling using a planetary mill (Fritsch Pulverisette 7, Germany). Tungsten carbide balls were used as milling medium and sealed in the vial together with the starting reactant materials at a ball-to-powder weight ratio of 20 : 1. A tungsten carbide-coated bowl was used and all milling was conducted at a speed of 500 rpm for 60 h. The specification of the samples is summarized in table 1.

The final product of the powders was then consolidated into compacts, using a cold-press at a pressure of 500 MPa. The consolidated object was then sintered at 1100 °C for 1 h in a tube furnace under Ar atmosphere.

The microstructure of the sintered samples was investigated by scanning electron microscope (SEM, Philips XL30) with an energy dispersive X-ray spectrometer

*Corresponding author:

Tel : +98-91-26451156, 98-21-64542955

Fax: +98-21-66405846

E-mail: Jazi_sharifian@aut.ac.ir

Table 1. Composition of samples.

Sample code	W/Ti weight ratio	Ni (wt.%)	Co (wt.%)
A ₀	0.15	35	35
A ₁	0.2	35	35
A ₂	0.25	35	35
A ₃	0.3	35	35

(EDX). The phases in the samples were analyzed by means of a philips diffractometer (40 kv) with Cu K α radiation ($\lambda = 0.1542$ nm).

Vickers micro hardness measurements were carried out on the mounted and polished samples under a load of 1 Kg and a dwell time of 10 s for several indentations and average values of hardness were reported.

For the calculation of the lattice parameter of the hard phase in samples, used the geometrical relationship according to the Bragg law for the cubic system:

$$a = \sqrt{h^2 + k^2 + l^2}$$

Results and Discussion

X-ray diffraction results for the as-reacted materials for samples of A₂ and A₃ are shown in Fig.1, 2. As can be seen, the final product contains both TiC and Ni peaks; in addition NiTi and WC compounds. Tic and Ni are thermodynamically stable phases. In fact Ni peaks are solid solution of Ni-Co. The formation of

(W,Ti)C solid solution in all compositions is evidenced and peak shift corresponds to the change in the W/Ti ratio.

Fig. 3. shows the scanning electron micrographs of the products combustion-synthesized samples with different W/Ti weight ratio .The resulting microstructures of products consist of spheroidal carbide phase embedded in a nearly continuous (Ni,Co) binder. The carbide particle size increases at the content of W/Ti weight ratio increases from 0.15 to 0.3 wt. %. A similar tendency was also reported in the case of Ni, Co, NiAl and Ni3Al additives [15, 16].

The solution of the W in TiC can be found by the calculation of its lattice parameter and comparing it with lattice parameter of pure TiC. the calculated

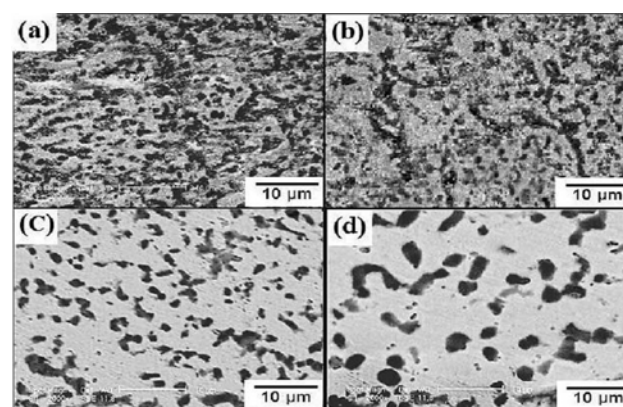


Fig. 3. SEM micrograph of (W,Ti)C-Ni-Co composites: (a) W/Ti = 0.15; (b) W/Ti = 0.2; (c) W/Ti = 0.25; (d) W/Ti = 0.3.

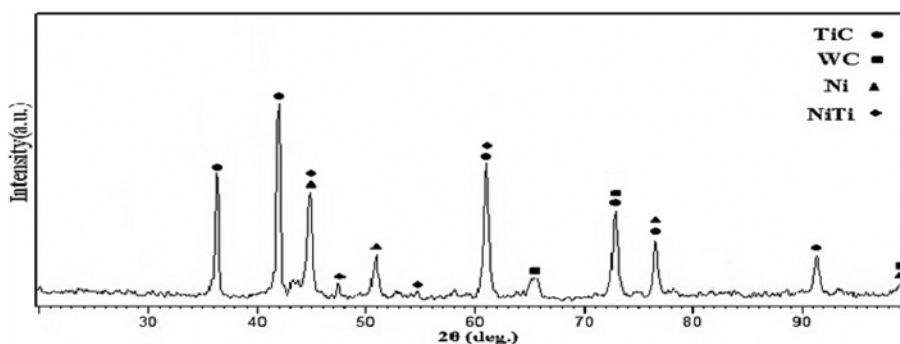


Fig. 1. XRD patterns of sample A₂.

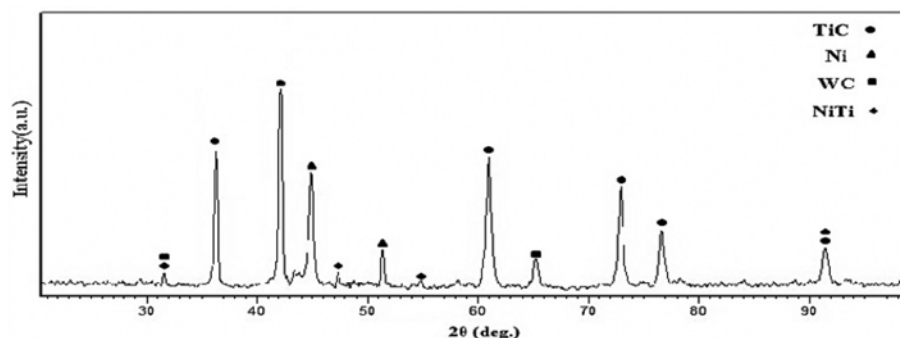


Fig. 2. XRD patterns of sample A₃

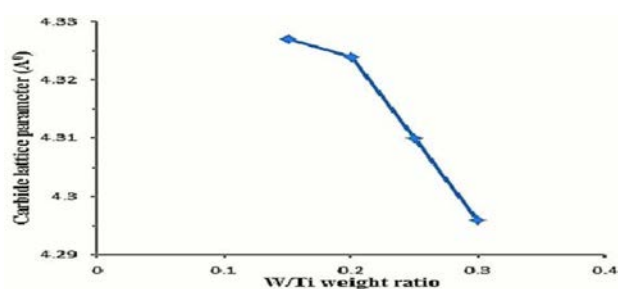


Fig. 4. Effect of the initial W/Ti weight ratio on the carbide lattice parameter.

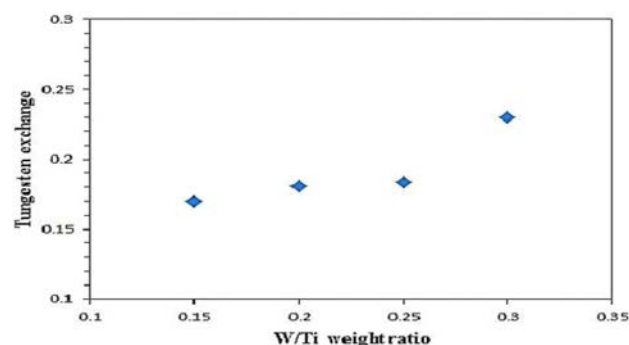


Fig. 5. Variation of tungsten exchange in carbide particles with W/Ti weight ratio.

parameter of TiC in the sample A₃ is 4.296 Å, which is less than that of pure TiC that is 4.329 Å. This means that the W atoms, which have smaller atomic radius than Ti, substitute some of the Ti atoms in TiC lattice and decreased its lattice parameter, as well in sample A₂ with TiC lattice parameter of 4.310 Å. As shown in Fig. 4, crystal lattice parameter of carbide phase decreased linear with increasing the W/Ti weight ratio.

Saidi and Barati also obtained the spherical (W,Ti)C grains by Combustion synthesis of Ti, W, C and Ni powder mixture with the molar ratios of W/Ti = 0.1-0.6 [16].

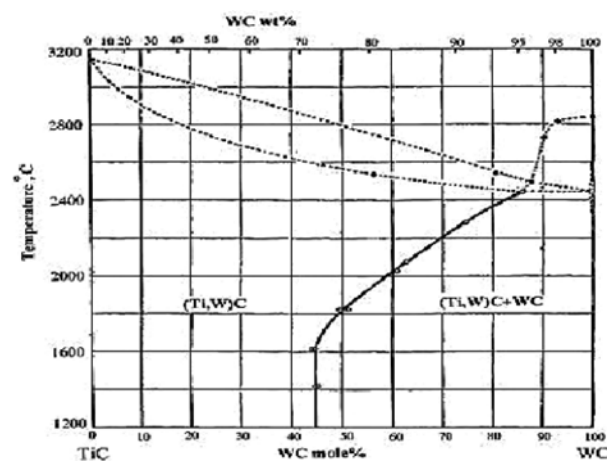


Fig. 7. TiC-WC phase diagram.

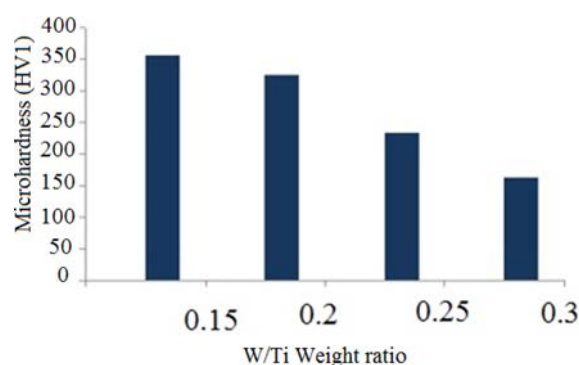


Fig. 8. Effect of W/Ti weight ratio on microhardness.

A number of Carbide particles in each sample were analyzed using SEM-Esther average tungsten exchange in the carbide particles then calculated for all of samples. The results are shown in Fig. 5, as plot of the variation of the tungsten exchange with the initial W/Ti weight ratio.

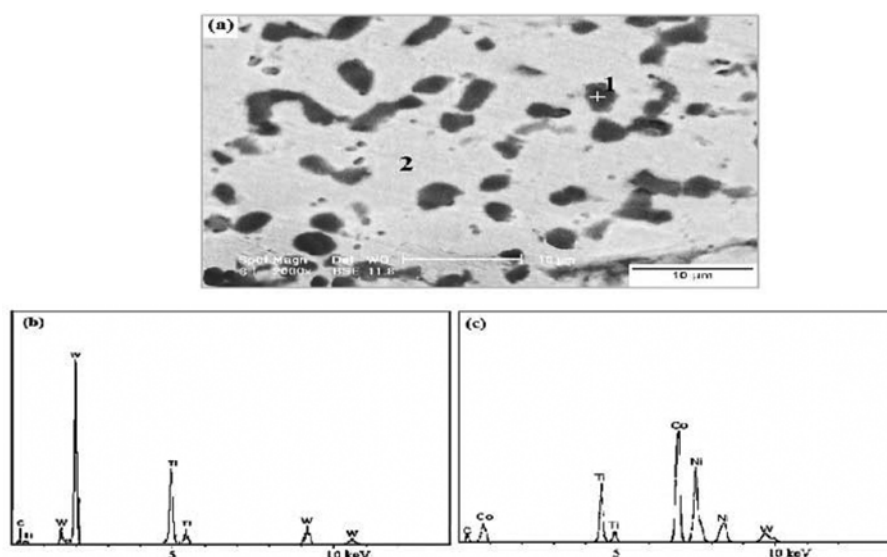


Fig. 6a. Microstructure of sample of A3 (b) EDX at point 1 and (c) EDX at point 2 as shown in (a).

The microstructure of the (W,Ti)C-Ni-Co composite (W/Ti = 0.3) is shown in Fig.6a, it can be found that there are many gray particles in the matrix. The result of microanalysis of these gray particle are rich in Ti, W and C. In fact the TiC-WC system has a complete solid solution according to its phase diagram in Fig. 7.

Fig. 6a shows the back scatter microstructure of the sample A₃. The composite was dense and no pores as observed. Microstructure contain gray and bright areas. EDX analysis (Fig. 6b, 6c) shows the gray areas are (W,Ti)C particles and lighter region is Ni/Co matrix. Carbide particles are uniformly dispersed in matrix with particle size in the range 0.5-3 µm. The composition of sample shows a typical without core-rim structure.

The micro hardness testing was conducted on the samples, and the results are given in Fig. 8. As can be seen, hardness decreased with increase of W/Ti weight ratio. Moreover, carbide particles size coarser with increased of W/Ti ratio, the effect of carbide particle size on the hardness as well as reported by [18-21].

Conclusions

1. The (W,Ti)C-Ni-Co nanocomposite produced by SHS method in smaller temperature range and has a different microstructure of carbide particles, because of they are without core- rim structure in compared with similar composites produced until now.

2. The (W,Ti)C particles generated in situ are uniformly dispersed in the matrix with particle size < 3 µm. Solution of W in TiC is proved by X-ray diffraction (XRD) and SEM-EDS techniques. Moreover, with the increasing of W/Ti weight ratio, rounding and growth of the hard phase particles are observed.

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