The synthesis and characterization of W- 1wt. % TiC alloy using a chemical method

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1. Introduction

The tungsten and its alloys have been used in many application due to their excellent mechanical and thermal properties such as high melting point, high thermal conductivity, high strength at elevated temperatures, low sputtering yield in radiation environment and low tritium inventory.[1-3] Moreover, they are considered as the most promising candidate for plasma facing components for future nuclear fusion reactors.[4] Nevertheless, the serious problems are issued with tungsten such as low temperature embrittlement, high ductile-to-brittle transition temperature and low recrystallization temperature. It is known that the body centred cubic crystal structure of tungsten has almost no ductility.[5,6] Therefore, a novel tungsten-based materials should be developed to overcome the problems stated above.

In the past few decades, many studies have demonstrated the dispersed second phase nano-particles in the tungsten matrix inhibit the grain growth and recrystallization, besides they improve the ductility and the irradiation resistance by hindering grain boundary sliding and stabilizing the microstructure.[7] La_2O_3, Y_2O_3, TiC or ZrC particles are usually added to tungsten. However, some crucial issues should be solved such as the uniform distribution of these second phase particles and the industrial mass production. By using typical mechanical alloying and powder metallurgy, nano-particles tend to be agglomerated and concentrated at the grain boundaries due to the high surface energies introduced. Moreover, the milling process often produces detrimental phase by the wear of the milling equipment and media.

Xia et al. have firstly reported core-shell structured W/TiC using ammonium metatungstate ($(NH_4)_6W_7O_{24}\cdot XH_2O$, AMT) and hydrochloric acid. Nano-sized TiC particles are coated uniformly by AMT precipitation formed by the addition of hydrochloric acid to the AMT solution. The core-shell structure particles were examined by TEM. In addition to this work, they have reported that use of Polyvinylpyrrolidone as dispersion agent to achieve better distribution. Also, L. Lou et al. have reported a use of ammonium paratungstate ($(NH_4)_{10}H_2W_{12}O_{42}\cdot XH_2O$, APT) with oxalic acid to form core-shell structure rather than the use of AMT. They have used spark plasma sintering as a consolidation method. The relative density achieved was 99.0%.

To achieve uniform distribution of TiC nano-particles, a wet chemical method is essential rather than typical mechanical alloying. Also, the use of the chemical method of alloying can be easily applied to the industry with cost-effective and environmental benefits.

The purpose of this study is to achieve uniform distribution of TiC nano-particles within the tungsten matrix, as the literature has studied. Moreover, the chemical methods could be applied to other refectory metals such as molybdenum.

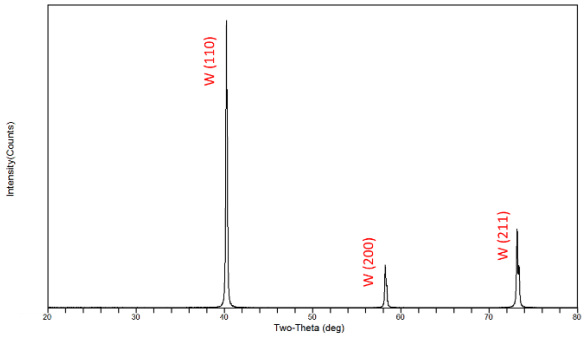

Fig. 1. XRD patterns of the reduced W/TiC powder.

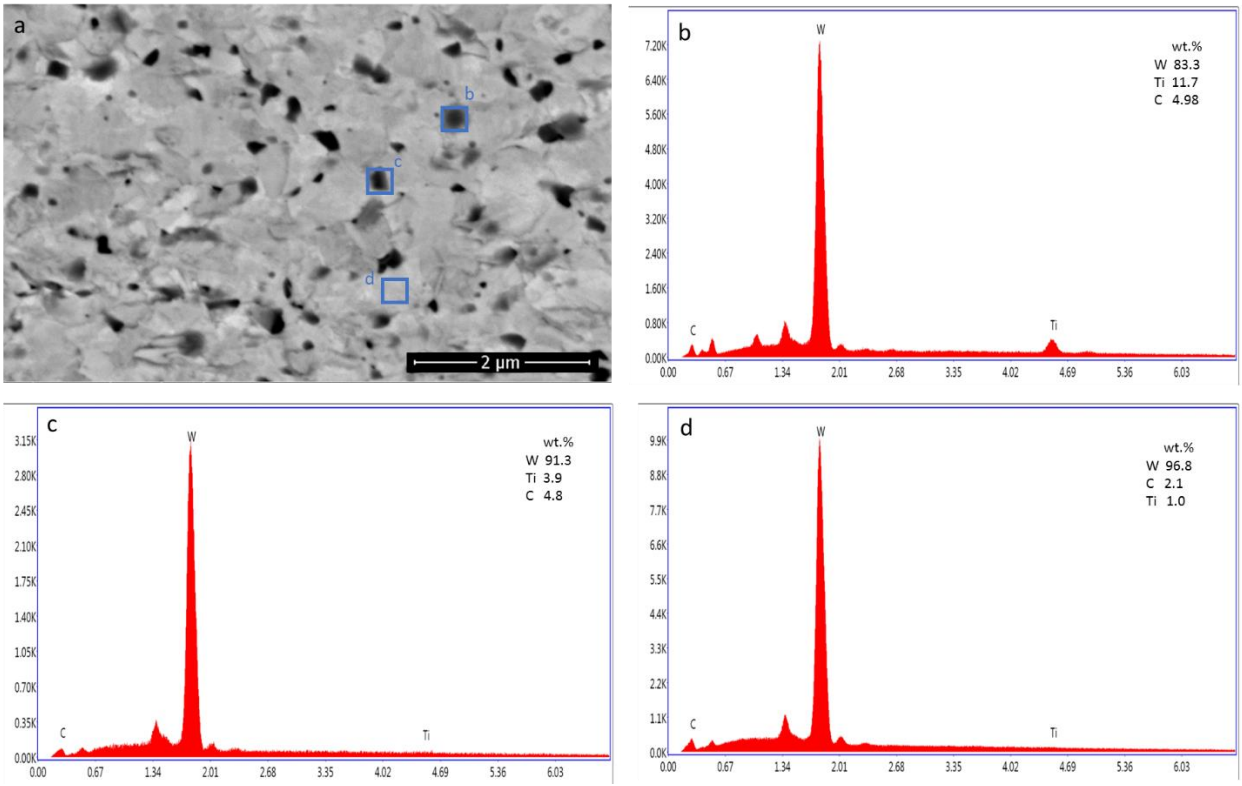

Fig. 2. (a) FESEM image of the sintered W/TiC; (b), (c) and (d) EDS spectrum of the selected square regions.

2. Experimental methods

0.500g of oxalic acid ($C_2H_2O_4 \cdot 2H_2O$) was dissolved in 250 ml of deionised water to form a clear solution, 0.0500g of TiC nano-sized particles (commercial powder, particle size about 50 nm, purity 99.9%, Alfa Aesar Korea) was added to the solution. The amount of TiC nano-powder was calculated by stoichiometry. The mixture was stirred for 30 minutes to ensure that TiC particles are well distributed in the solution. 7.500g of ammonium paratungstate (purity 99.9%, Alfa Aesar) was added to the above solution. After stirring the solution for 5 hours, the W/TiC precursor was obtained by stirring and evaporating the mixture solution in phenyl methyl silicone oil bath at 165°C. The obtained powder was ground and then reduced by high purity hydrogen flow in a tube furnace at 800°C for 60 minutes. The reduced powder was consolidated using Spark Plasma Sintering (SPS) technique. The powder is loaded in an electrically and thermally conductive graphite die with a diameter of approximately 13 mm.

3. Characterization

The crystal phases of W/TiC reduced powders were identified using X-ray diffraction (XRD). In addition, the microstructure of the sintered sample was characterized using Field-emission scanning electron microscopy (FE-SEM) and energy dispersive X-ray spectrometry (EDS). The density of the sintered sample was measured by Archimedes principle.

4. Results

Fig. 1 displays the XRD pattern of reduced W/TiC powders, which consisted of the body-centered cubic crystalline structure of pure tungsten. No peaks for TiC were observed due to the low content of TiC. It is the indication that the powders were completely reduced using the tube furnace at 800°C for 1 hour.

The micro-structure of sintered W/TiC alloy is illustrated in Fig. 2. The number of black particles appears in the grey tungsten matrix. The black particle diameter ranging from 60 nm to 1200 nm,

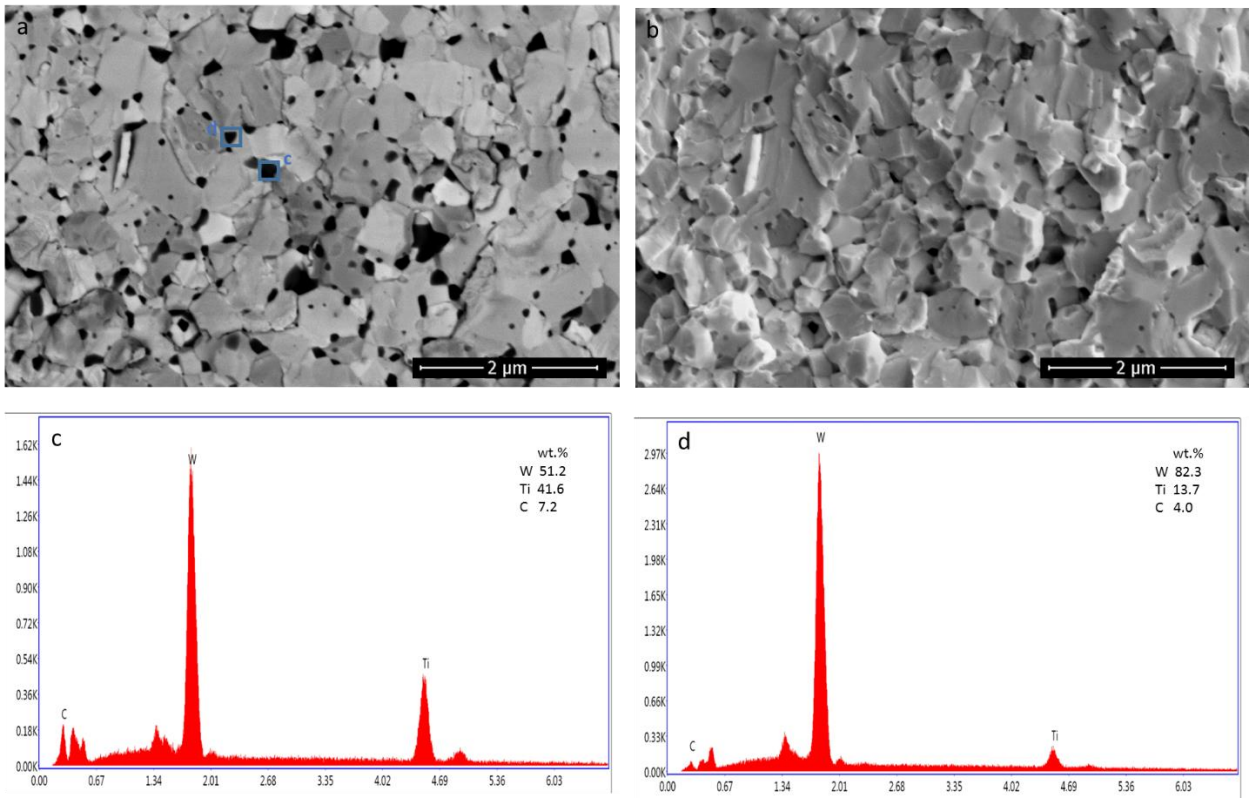

Fig. 3. (a) Back scattered electron image (b) secondary electron of the fracture surface sintered W/TiC; (c) and (d) EDS spectra of the selected square regions

however some aggregations of black particles was observed with the diameter of several microns. The most of the large particles are distributed in the grain boundaries. Furthermore, the relative density of the sintered sample is 97.4%.

The EDS spectrum of selected rectangular region "b" indicates the black particle is TiC phase which is shown in Fig. 2b. Also, the EDS spectrum of Fig. 2c and d consist of Ti, C and W which indicates the tungsten matrix contains small amount of TiC particles.

The facture surface of the sintered sample is displayed in Fig. 3. A number of micro-pores were found within the fracture surface. Moreover, both trans-granular and inter-granular fractures can be observed on the surface. Fig. 3c and d show the EDS spectra which verify the presence of TiC particles.

5. Conclusion

Core-shell structured W/TiC precursor was successfully synthesized using a chemical method. The obtained powder was reduced and sintered using SPS technique. The relative density of sintered sample achieved was 97%. Furthermore, the microstructure of the sintered sample was analyzed using FE-SEM and the presence of TiC particles at the grain boundary and grain interior was confirmed by EDS. both trans-granular and inter-granular fractures can be observed on the surface.

Acknowledgment

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