Int. Journal of Refractory Metals and Hard Materials 33 (2012) 53–57

Contents lists available at SciVerse ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

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

Synthesis and characterization of hexagonal nano tungsten carbide powder using multi walled carbon nanotubes

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ARTICLE INFO

Article history:
Received 19 September 2011
Accepted 17 February 2012

Keywords:
Nano-particle
Hexagonal tungsten carbide
Wire explosion
MWCNT
WAXD
HRTEM

ABSTRACT

Tungsten carbide nano particles were formed by carburizing tungsten/tungsten oxide/non-stoichiometric tungsten oxide particles obtained from a wire explosion process with multi walled carbon nano tubes (MWCNT). The produced powder is confirmed to be hexagonal tungsten carbide from wide angle X-ray diffraction analysis. Carburization of tungsten nano particles with MWCNT shows the presence of tungsten sub-carbides in the produced tungsten carbide particles. Transmission electron micrographs show that the tungsten carbide particles are of spherical shape and the geometric mean size of the particles obtained is about 19 nm.

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1. Introduction

Transition metal carbides have many superior properties such as good thermal, structural and excellent electronic characteristics. Specifically, hexagonal tungsten carbide (WC) is an important tool and die material because of its high melting point and high hardness, with good thermo-mechanical and thermo-chemical properties. Tungsten carbide is also a potential alternative for materials such as Pt and Ru in fuel cell applications because of its good electro-catalytic properties [1–3]. Further, it is well known that nano structured materials have unique chemical, electrical, optical, mechanical, magnetic properties, etc., and can be selectively controlled by engineering the morphology and composition of the particles [4,5]. Thus, obtaining nano tungsten carbide is essential to achieve the best characteristics of the material, and to meet the demands of industrial applications.

Recently, a number of methods such as gas–solid reactions [6,7], gas-phase reactions [8], and electrochemical, and sonochemical methods [9,10] have been reported to obtain nano particles of transition metal carbides. Tungsten carbide is conventionally synthesized by powder metallurgical techniques involving the heat treatment of a mixture of metallic tungsten powder and carbon black in a furnace under an atmosphere of flowing hydrogen [11]. The temperature of carburization reaction should be high, in the range of 1400–1800 °C or above 2500 °C. The particles thus obtained are in the large micrometer size range [12]. Shi et al. produced WC through reduction and carbonization processes using MWCNT and tungsten oxide precursors by molecular level mixing and calcinations, resulting in particles in the size range of 20–100 nm [13]. Kim et al. generated WC by chemical vapor condensation process using CO and hexa-carbonyl as precursors; the resulting material WC_{1-x} co-existed with pure tungsten [14]. Debalina et al. have generated nano tungsten carbide by wire explosion process, where nano tungsten sub-carbides were present [15]. Koc et al. produced WC by carburization of tungsten oxide with particles of sub-micron size [16].

This paper deals with the production of hexagonal tungsten carbide by a two-stage process. The first stage involves generation of nano tungsten/tungsten oxide (WO_3)/non-stoichiometric tungsten oxide (WO_{3-x}) particles by wire explosion process (WEP). In the second stage, these particles are carburized with MWCNT to generate nano tungsten carbide particles by carburizing the nano tungsten oxide/non-stoichiometric tungsten oxide with MWCNTs. The WC particles thus produced are characterized through wide angle X-ray diffraction (WAXD). The size and shape of the produced powders are analyzed by transmission electron microscope (TEM). The particle size distribution follows a log-normal probability distribution. It is found that the tungsten carbide produced by carburizing nano tungsten contains sub-carbides. The amounts of MWCNT to be added to produce nano tungsten carbide from nano tungsten oxide particles are detailed.

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doi:10.1016/j.ijrmhm.2012.02.010

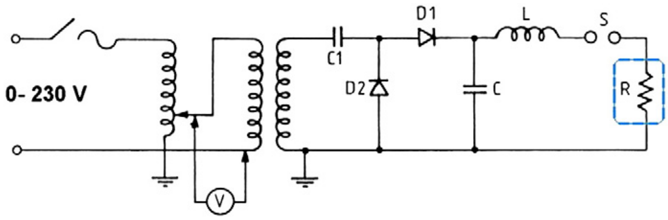

Fig. 1. Experimental setup of the wire explosion process.

2. Experimental details

The circuit used for exploding a wire to synthesize nano-powder is shown in Fig. 1. It includes a charging unit, a discharging unit, and a wire explosion unit. The charging unit is a high-energy capacitor charged to a required direct current (DC) voltage. The discharging unit consists of a trigatron switch S, and the explosion unit involves the conductor R to be exploded. The conductor is placed in a closed chamber filled with the desired gaseous ambience at an appropriate pressure. In the present work, tungsten wire has been exploded in argon/oxygen medium to form nano tungsten/tungsten oxide particles. To form non-stoichiometric tungsten oxide, the tungsten conductor is exploded in an argon–oxygen mixture of 1:3 or 3:1 ratio.

To start with, the capacitor is charged from the DC source and discharged through the wire. In the present work, the capacitor is of 3 μF capacitance. The dimensions of the exploding conductor are chosen in such a way that the current density is constant in the entire wire. The diameter and length of the conductor are 0.25 mm and 50 mm respectively.

In the present work, the circuit parameters match the condition for an under-damped RLC circuit, i.e.,

$$\frac{R^2}{4L^2} < \frac{1}{LC}. \tag{1}$$

Then, the magnitude of current flow in the circuit can be written as

$$i(t) = \frac{Ee^{\frac{-R}{2L}t}}{\sqrt{\frac{L}{C} - \frac{R^2}{4}}} \sin\left(\sqrt{\frac{1}{LC} - \frac{R^2}{4L^2}}t\right) \tag{2}$$

where C is the capacitance of the capacitor, E is the charging voltage of the capacitor, R is the exploding wire resistance, and L is the contribution from the internal inductance of the capacitor and the lead inductance. This suggests that the choice of a capacitor with low L/C ratio is suitable for the present work.

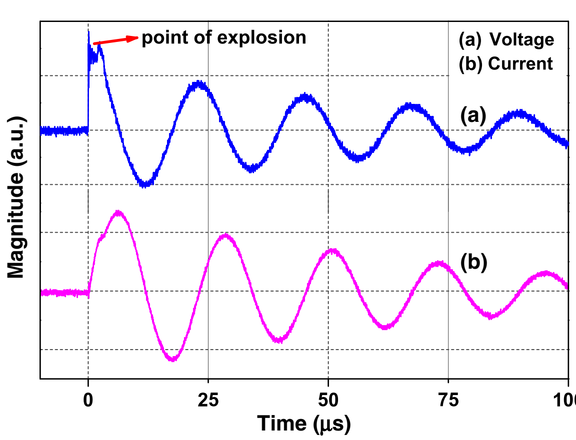

Fig. 2. Typical (a) voltage and (b) current waveforms of tungsten wire exploded in oxygen gas.

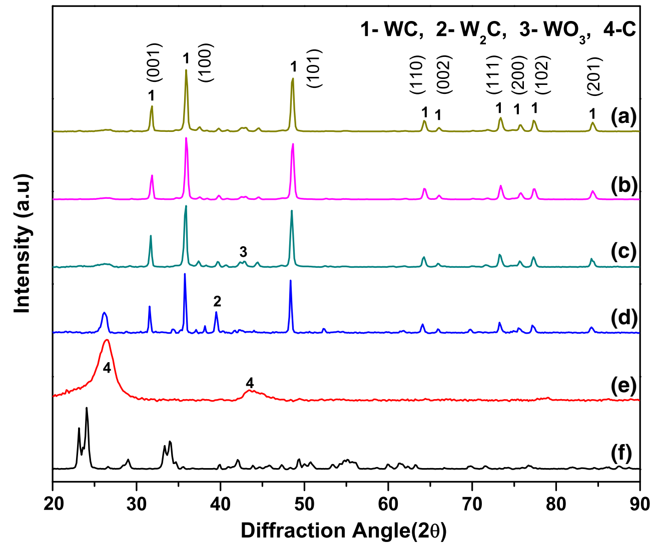

Fig. 3. Results of WAXD investigations of tungsten carbide particles obtained by carburizing the tungsten oxide (type-a) particle with different proportions of MWCNT (a) 1:0.5, (b) 1:1, (c) 1:1.5 and (d) 1:1 with tungsten nano particle, (e) MWCNTs and (f) reference WO_3 (Type-a).

The voltage across R and the current flow through it during the wire explosion process were measured using a voltage probe (PPE-20 kV, LeCroy®) and a current probe (Pearson Electronics, USA, Model No-101) respectively. The typical applied voltage and the current measured during the wire explosion process are shown in Fig. 2.

The nano tungsten/tungsten oxide particles produced by WEP were mixed with different proportions of MWCNT by weight and carburized to form nano tungsten carbide. MWCNT (obtained from Alfa Aesar, USA) has an internal diameter in the range of 1–3 nm and an outer diameter of 3–20 nm, with a length of 0.1–10 μm. The carburization process was carried by placing the nano powder obtained from the WEP in a sealed quartz tube and heating it in a furnace stabilized at a temperature of 1250 °C for 7 h. The heating rate was 7 °C/min and the cooling rate was 30 °C/min.

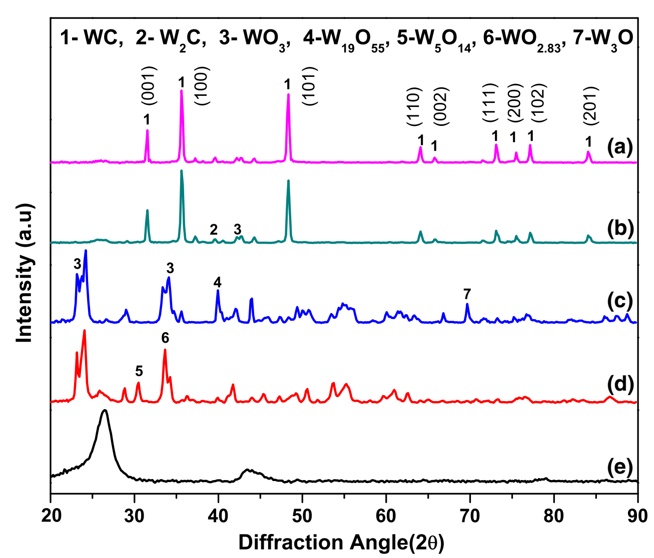

Fig. 4. WAXD pattern of tungsten carbide particle obtained by carburizing non-stoichiometric tungsten oxide with MWCNT in the ratio of 1:1 (a) carburized Type-b particles, (b) carburized Type-c particles, (c) Type-b particles, (d) Type-c particles, and (e) MWCNT.

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