



Preparation of alumina–tungsten carbide nanocomposite by mechano-chemical reduction of WO_3 with aluminum and graphite

M. Zakeri*, M.R. Rahimipour, S. Kh. Sadrnezhad, R. Yazdanni-rad

Ceramic Department, Materials and Energy Research Center, P.O. Box 31787/316, Karaj, Iran

ARTICLE INFO

Article history:

Received 28 August 2009
Received in revised form 29 October 2009
Accepted 2 November 2009
Available online 13 November 2009

Keyword:

Mechanochemical processing

ABSTRACT

Alumina–tungsten carbide composite powders were successfully obtained through reactive milling of WO_3 , Al and graphite as precursor at room temperature. The structural and morphological evolutions of the powders were studied by X-ray diffraction (XRD) and scanning electron microscopy. Physical properties such as density, surface area and micro-hardness of the ball milled powders were also measured. Results showed that the mechano-chemical reactions began in the first 5 h of milling and completed by the end of the milling (80 h). Very fine particles with a homogeneous microstructure with the hardness of 1450 kg/mm^2 were obtained at the end of milling. An annealing step did not lead to extensive grain growth, and the as-annealed sample still maintained its nanocrystalline characteristics with a minimum lattice strain.

© 2009 Elsevier B.V. All rights reserved.

1. Introduction

Cutting tool materials with improved mechanical properties and chemical inertness capable of operating at high cutting speeds is an ever-growing need. Ceramic materials are the prime candidates to fulfill these requirements because of their excellent physical properties such as thermal stability, high hardness, and good corrosion resistance.

One of the most widely used materials as ceramic cutting tools is alumina. The addition of a hard secondary phases such as WC, TiC, TiB_2 , $Ti(C,N)$, ZrO_2 particles, and SiC whiskers to alumina matrix provides great improvement in mechanical properties [1–8].

Another possible mechanism to improve mechanical properties is to prepare these materials in nano structure. Al_2O_3 –WC nanocomposite can be obtained easily by direct mixing of nano alumina and tungsten carbide [2,9]. But the resulting heterogeneous microstructure and high cost of the starting materials are two important set backs of this method. Alternatively, nano-metric Al_2O_3 –WC powders can be obtained through high-energy reactive milling of mixtures of WO_3 , Al and C powders. The occurrence of reactions during milling depends on several factors. Of course, thermodynamic is the determining factor, followed by parameters that controlling the reaction kinetics, such as atomic and thermal diffusivity and the mechanical properties of the reactant phases [10]. Mechanical alloying (MA) [11] has been used for preparing thermally stable metallic glasses and amorphous alloys

[12], nanocrystalline and nanocomposite materials [13,14], and refractory hard materials [15], carbides [16], hydrides [17]. Sherif Eskandarany [18] obtained WC–32 at.% Al_2O_3 nano composite from WO_3 , Al and graphite mixture after 100 h of milling. Also Pallone et al. [19] performed a similar study, but they obtained very different results. For more clarification we also performed the above study, but with more emphasis on the various conditions of the starting materials, milling time and annealing temperatures. Ball milled powders were characterized in different ways. In general the aim of this work is to obtain alumina–tungsten carbide composite powder by ball reactive milling of WO_3 , Al and graphite at the best conditions of milling and annealing.

2. Experimental

Mechanical alloying was performed in a planetary ball mill at nominal room temperature with a vial rotation speed (cup speed) of 500 rpm. Pure Al (Fluka Co, 99.9%, $<200 \mu\text{m}$), graphite (MERCK, 99.9%, $<50 \mu\text{m}$) and WO_3 (BDH, 99.9%, $<100 \mu\text{m}$) powders were mixed to give the desired $Al_2O_3(34)$ –WC(66) weight percent composition (77.85% WO_3 , 18.12% Al and 4.03% graphite wt.). The ball to powder weight ratio (BPR) was 10:1. Five balls with 20 mm, two ball with 15 mm and two balls with 10 mm of diameter were used in the mechanical alloying experiments. Energy of the ball mill can be increased by using a distribution of balls with different sizes. Ball size distribution increases the frequency of the ball–ball and ball–wall impacts and leads to the higher energy of the ball mill. The mixture of the powders and the steel balls was charged into a steel vial (150 ml) in Ar atmosphere. Samples for analysis were removed by interrupting the milling process at various intervals.

X-ray diffraction profiles were recorded on a Philips diffractometer (30 kV and 25 mA) with $Cu K\alpha_1$ radiation ($\lambda = 1.5404 \text{ \AA}$). All XRD experiments were performed with a step size of 0.02° and a time per step of 1 s. The recorded XRD patterns were used for the calculation of crystallite size and strain. Prior to calculations from the XRD peaks, the background was automatically removed and the $K\alpha_2$ radiation (Eq. (1)) was stripped from the scans using the computer software X-pert High Score

* Corresponding author. Tel.: +98 261 6204131; fax: +98 261 1888.
E-mail address: M.zakeri@merc.ac.ir (M. Zakeri).