Synthesizing a nanocomposite tungsten carbide-cobalt metal oxide

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ABSTRACT

In this work a nanocomposite WC-Co metal oxide was synthesized by using a solid state mixing techniques. Certain metals oxide were used in this process (Al, Mg, Si, and Zr) to enhance the property of the powder. The nano-powder was consolidated and sintered with a fast sintered techniques to produce a very hard metal with an outstanding properties,

In fabrication techniques, it is important to maintain the powder properties during consolidation process. In addition, Chemical and mechanical characterization were performed for the consolidated metal. The mechanical characterization shows a very hard material with an outstanding properties.

Keywords: nanocomposite, metal oxide, solid state, consolidation

1 INTRODUCTION

WC has a high melting point of about 2900 °C, which results in the difficult preparation of dense bulk pure WC by conventional sintering techniques [Imasato et al., 1995]. However, the addition of metal Co binder to WC result in improved sintering and increases its strength and toughness; but on the other hand, the hardness and wear resistance of the cemented carbides are significantly decreased. Moreover, the additions of such metallic binders are inferior to WC in corrosion and elevated temperature applications [Sherif El-Eskandarany, 2000]. All of these factors have led to limited applications of such WC-metal binder composites. El-Eskandarany(2000) replaced the traditional metal Co binder with a nonmetal-binder nanocrystalline MgO material [Kim et al., 2008] to fabricate full dense bulk nanocomposites that enjoyed excellent mechanical properties. This was due to the use of nanocrystalline TiC [Sherif El-Eskandarany, 2005] and Al_2O_3 [Imasato et al., 1995; Jiang et al., 2007] as nonmetal-binders to extend the service life of WC materials and improve their mechanical and physical properties. Cemented carbides [Raihanuzzaman et al., 2014] such as tungsten carbide-cobalt composites are characterized by high hardness and wear resistance resulting from their high carbide content [Su et al., 2014]. These classes of materials have been widely used as cutting tools and wear-resistant components due to the singular combination of their properties which include high hardness, moderate toughness, and excellent wear resistance.

The present work addresses, in part, to study the effect of adding ZrO_2-2mol% Y_2O_3 nanocrystalline fine grains to small concentrations (0 to 7 wt%) in improving the fracture toughness and wear resistance of mechanically mixed 93WC/7Co nanocomposite powders. $ZrO_2$was selected because of its high thermal stability and excellent mechanical properties such as high bending strength and excellent fracture toughness [Anstis et al., 1981]. This study also proposes a powerful tool for obtaining fully dense bulk nanocomposites using spark plasma sintering (SPS) technique on mechanically mixed WC-ZrO2ceramics powders.

The top-down approach [Sherif El-Eskandarany, 2009] which involves using high energy ball milling method [Sherif El-Eskandarany, 2001] is considered as the most powerful technique for synthesizing and fabricating wide varieties of metastable [Sherif El-Eskandarany and Inoue, 2007] and nano-scaled materials with grain sizes of less than 10 nm in diameter [Sherif El-Eskandarany, 2014]. The fabrication of nanocomposite materials via high energy ball milling method dates back to 1998 when El-Eskandarany [Sherif El-Eskandarany, 1998] used it for the preparation of homogeneous SiCp/Al nanocomposite with high volume fractions of SiC nanograins reaching up to 25%. Since then, many attractive nanocomposite systems have been fabricated using this technique for a variety of machining, cutting, drilling, and other applications [Suryanarayana and Al-Aqeeli, 2013].

2 EXPERIMENTAL

2.1 Powder consolidation

Commercial WC powder was used to prepare WC-based nanocomposite material. The product obtained after 20 h of ball milling consisted of ultrafine WC grains of about 5 nm in diameter (Figure 1a). The selected area diffraction pattern (SADF) (Figure 1b) shows the formation of single hexagonal closed packed (hcp) WC nanograins.

The end product was mixed with different wt% of metallic ZrO_2, Y_2O_3, and Co powders to obtain the desired nominal composition. The mixed powders of each ZrO_2-2mol% Y_2O_3 concentration were sealed in a glove box under argon gas atmosphere. The ball-milling experiments were carried out at room temperature for 75 h, using Fritsch P5 high-energy ball mill (Germany) at a rotation speed of 4.2 s^{-1}.

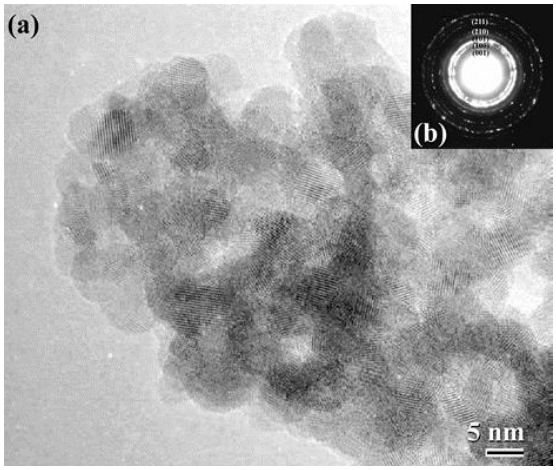

Figure 1: (a) High resolution transmission electron microscope (HRTEM) and the corresponding SADF; (b) High-energy ball milled WC powders obtained after 20 h of milling.

In order to consolidate the nanocomposite powders into perfect cubic high-dense shapes with a dimension of 1cm ×1cm ×1 cm, first, the powders were dried in a vacuum oven at 150ºC for 24 h. Then the activated powders were gently ground inside a glove box filled with argon, using agate mortar and pestle. The powders were then placed in sealed screw bottles under argon gas atmosphere and stored under vacuum in dissectors. Nanocompositepowders with different ZrO_2-2mol% Y_2O_3 concentrations were individually consolidated into bulk samples using graphite molds SPS system. The consolidation procedure took place in a vacuum at 1600ºC and 30MPauniaxial pressure. In order to avoid any undesired grain growth, the sintering process was applied for only 0.18 ks without the addition of any binding materials. More experimental details of SPS are shown elsewhere [Kim et al., 2008; Jiang et al., 2007].

2.2 Sample characterization

The total structural investigations of the nanocomposite powders with different ZrO_2-2mol% Y_2O_3 concentrations were examined by powder X-ray diffraction (XRD) technique, high resolution transmission electron microscope (HRTEM), and energy-dispersive x-ray spectroscopy (EDS). Analyses were carried out to investigate the local structure and composition of synthesized nanocomposite powders. The topological and surface properties of the fabricated bulk nanocomposites samples were investigated by contact-mode AFM technique.

The synthesized nanocomposite powders and their corresponding bulk materials (after consolidations) were examined by the electron probe microanalyzer (EPMA). However, the morphology of the selected samples was investigated using the field emission scanning electron microscope (FE-SEM) equipped with energy dispersive X-ray (EDS).

The microhardness of all consolidated samples was determined with Vickers hardness test, using a load of 20 kg. In these experiments, microhardness values were the average of at least 60 readings performed for each sample. The fracture toughness of the consolidated samples was determined using the VHT indenter crack-length measurements approach.

Nanoindentation technique was employed to determine the nanomechanical properties of consolidated nanocomposite samples. This technique allows the investigation of nano-hardness and Young's modulus (modulus of elasticity) values of the bulk samples produced. To achieve this, more than 300 indents were developed for each sample using single indent continuous multicycle ramp approach with an applied force of 400 mN.

Wear test, using a pin-on-disk machine (Microtest, Spain) was employed to determine wear behavior of the consolidated nanocomposite samples with ZrO_2-2mol% Y_2O_3 concentrations. The samples were tested in pairs under non-abrasive conditions of 150 N applied force and 100 m sliding distance. In the experiments, the specimen was placed on a flat circular disk rotated at 400 rpm speed. The sample was positioned perpendicular to a WC-12Co ball. This WC ball (pin) was pressed against the sample on a disk at a specified load by means of a lever and attached weights. Wear results were reported as volume loss in cubic millimeters for the pin and the disk separately. The amount of wear was determined by weighing the sample and WC ball before and after the test.

3 RESULTS AND DISCUSSION

When the WC powders were subjected to high energy ball milled for 20 h, their grain sizes were reduced to 10 nm in diameter. Moreover, the milled WC grains tended to have a spherical-like morphology, as shown in Figure 2b.

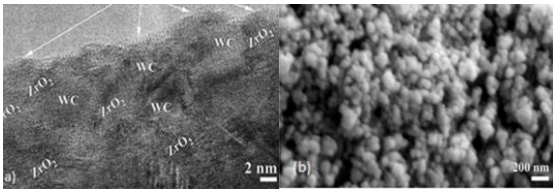

Figure 2: (a) HRTEM image of nanocomposite 93WC/7ZrO_2-2 mol% Y_2O_3 powders obtained after 75 h of ball milling; (b) WC powders obtained after 20 h of the ball milling time.

The end-product of milling 93WC/7ZrO_2-2mol% Y_2O_3 powders consisted of ultrafine grains (about 4-5 nm in diameter) of WC coexisting with ZrO_2, as shown in the HRTEM image in Figure 2.

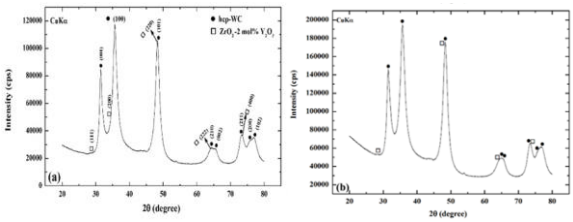

Figure 3: (a) XRD pattern of nanocomposite 93WC/7ZrO_2-2mol% Y_2O_3 powders obtained after ball milling for 75 h; (b) XRD pattern of the powders obtained after consolidation at 1600ºC, using SPS technique.

The metallographic examinations of the consolidated bulk nanocomposites with different concentrations (x) of ZrO_2-2mol% Y_2O_3 are shown in Figure 4. The prismatic shape with sharp edges of WC large grains (87 μm to 633 μm) embedded into the Co matrix can be clearly seen in Figure 4a. Unlike the 93WC/7Co specimen, the microstructure of the cemented carbides obtained upon replacing Co with 4 wt% of ZrO_2-2mol% Y_2O_3 (Figure 4b) showed significant improvement on the WC grain sizes, which became finer with an average size of 74 μm. We believe that the hard phase of fine ZrO_2-2mol% Y_2O_3 grain played the role of a micro-milling media and led to the refinement ofWC powders to the micro level. In addition, the decreased percentage of WC angular grains and increased portion of cubic/rounded grains can be seen.

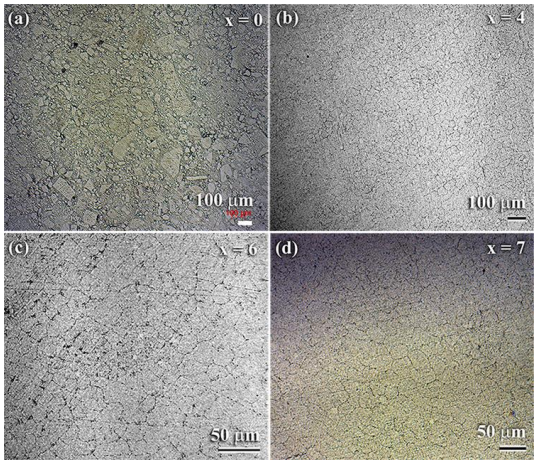

Figure 4: Optical micrographs of the cross-sectional view for bulk 93WC/7(Co)/xZrO_2-2mol% Y_2O_3nanocomposite materials with ZrO_2 concentrations of (a) 0, (b) 4, (c) 6 and (d) 7 wt.%.

As the concentrations of ZrO_2-2mol%, Y_2O_3added to WC increased to 6 (Figure 4c) and 7 wt% (Figure 4d), the microstructure of WC became fine with almost around shape morphology. Moreover, the average WC grain sizes were dramatically reduced to 23 - 49μm.

4 CONCLUDING REMARKS

Nanocomposite 93WC/7Co/xZrO_2-2mol% Y_2O_3 powders with different ZrO_2-2mol% Y_2O_3 concentrations (xwt%) were successfully prepared by high energy ball milling technique. The end-product obtained after ball milling for 75 h were consolidated into full, dense bulk nanocomposite with relative densities. This consolidation did not lead to significant grain growth in the WC grains maintained in nanocrystalline structure. Based on the results of this study, ZrO_2-2mol% Y_2O_3 additives led to improved fracture toughness (K_{c1}) for the nanocomposite93WC/7ZrO_2-2mol% Y_2O_3 system. The wear properties indexed by the coefficient of friction (COF) for nanocomposite 93WC/7Co/xZrO_2-2mol% Y_2O_3 systems were improved upon increasing ZrO_2-2mol% Y_2O_3 content.

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