Nanomechanical and Morphological Characterizations of Spark Plasma Sintered WC-5Co-1Cr-3MgO-0.7VC-0.3Cr_3C_2 Nanocomposite Powders Prepared by Solid-State Mechanical Mixing Technique

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ABSTRACT

Nanocomposite WC-5Co-1Cr-3MgO-0.7VC-0.3Cr_3C_2 powders were prepared at room temperature, using a high-energy ball mill technique. The powders obtained after 75 h of ball milling had spherical-like morphology with an average particle size diameter of 0.75 μm. The powders were composed of nanoscaled grains of less than 10 nm in diameter. These obtained powders were consolidated at 1500 ^{o}C with a pressure of 30 MPa into full-dense bulk samples, using spark plasma sintering (SPS) approach. This consolidation step did not lead to a serious grain growth and the material maintained its nanocrystalline characteristics after compaction.

The nanomechanical properties, indexed by nanohardness, Young's modulus were investigated by nanoindentation technique at room temperature and they were found to be 18.67 GPa and 406.33 GPa, respectively. Atomic force microscope (AFM) was employed to characterize the nanoindents. Vickers microhardness test was used to determine the fracture toughness (K_c) of the consolidate bul samples. The average K_c value, which was estimated from 60 Vickers indents, was found to be 17.36 MPa.m$^{1/2}$.

Keywords: hard materials, metal-matrix-composites, nanoindentation, atomic force microscope.

1 INTRODUCTION

WC-Co-based composites possess unique and useful properties that make them desirable materials for wide range of industrial applications such as tips for cutting and drilling tools, wear resistant parts in wire drawing, extrusion and pressing dies and wear-resistant surfaces in many types of machines [1]. The present study has been undergone in order to fabricate new nanocomposite of WC-5Co-1Cr-3MgO-0.7VC/0.3Cr_3C_2 (wt.%) powders by mechanically-induced solid state mixing, using high energy ball milling technique. The present study aims to improve the homogeneity of the synthesized materials and the corresponding mechanical properties at the micro-and nano-levels.

2 EXPERIMENTAL PROCEDURE

The starting materials of WC, Co, Cr, VC, Cr_3C_2 and MgO were first balanced in a glove box under argon gas atmosphere to give the average nominal composition shown in title of the paper. Then, the mixed powders were charged into WC vials and sealed together with 25 balls made of WC (15 mm in diameter) to give an average ball to powder weight ratio of 20 to 1. The ball milling process was carried out at room temperature using high energy ball milling with a rotation speed of 250 rpm for 75 h of milling time.

The structure of the synthesized powders and the consolidated bulk samples were investigated by X-ray (XRD) and high resolution transmission electron microscope/energy dispersive X-Ray spectrometer (HRTEM/EDS). Field emission scanning electron microscope (FESEM) and electron probe micro analyzer electron microscope/EDS (EPMA/EDS) were utilized to analyze the local elemental compositions of the nanocomposite powders. the as-fabricated powders were consequently consolidated into dense bulk materials (Fig. 1) at a temperature of 1500 ^{o}C with a pressure of 30 MPa, using SPS technique.

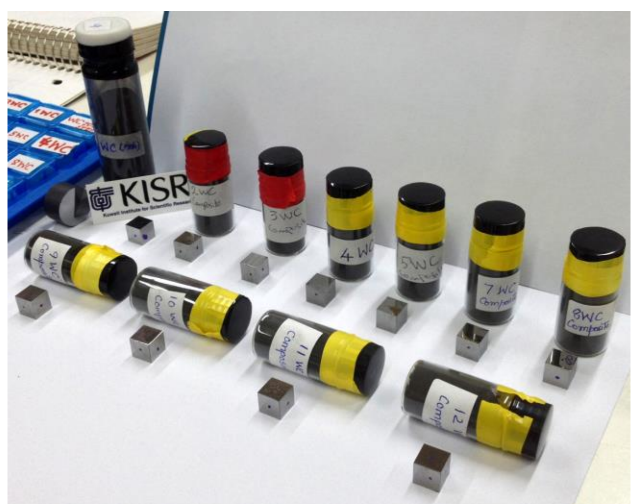

Figure 1: The Outer shape of different samples of WC-5Co-1Cr-3MgO-0.7VC-0.3Cr_3C_2 nanocomposite after consolidation by SPS at 1500 ^{o}C with a pressure of 30 MPa.

Whereas the microhardness of the consolidated samples was investigated by Vickers hardness tester, their nanohardness and Young's modulus properties were determined via nanoindentation approach. AFM technique was employed to characterize the topographic properties of the consolidated samples.

3 RESULTS AND DISCUSSION

Figure 2 shows the SEM micrograph of the powders obtained after high energy ball milling for 75 h. The powders possess excellent morphological characterizations indexed by their spherical-like morphology and narrow particle size distributions (0.5 – 1 μm in diameter).

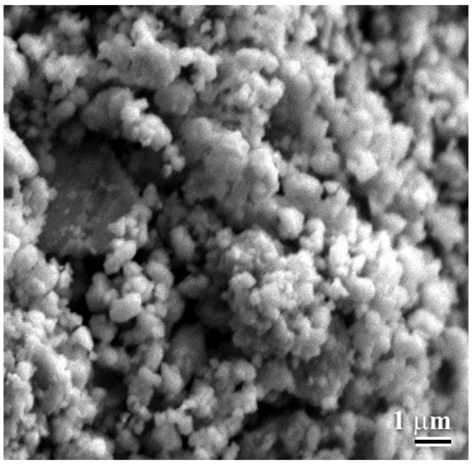

Figure 2: SEM micrograph of the powders obtained after 75 h of ball milling.

The bright field image (BFI) of the powder after this stage of ball milling shows the formation of homogeneous nanocomposite powders, indicated by the excellent distribution of the reinforcement materials in the metal matrix (Fig. 3).

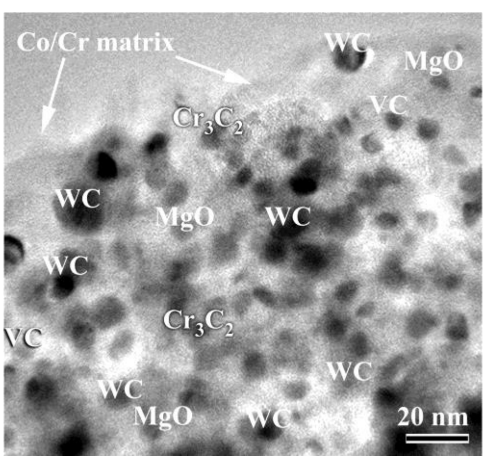

Figure 3: BFI of the nanocomposite powders obtained after ball milling for 75 h.

The BFI of the consolidated sample (Fig. 4) shows fine grains with nanostructured characterizations of less than 60 nm in diameter. This indicates that the consolidation step did not lead to a serious grain growth and the bulk material still maintains its nanocrystalline characterizations.

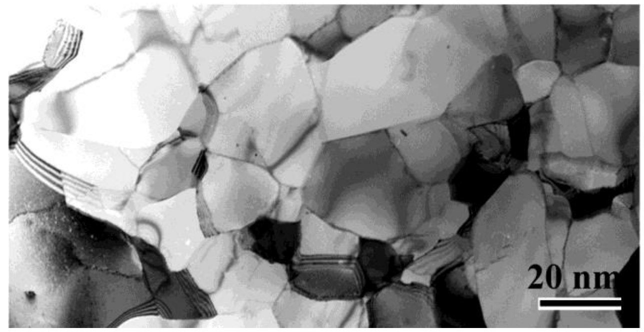

Figure 4: BFI of as-consolidated bulk sample.

The SEM micrograph of the Vickers microhardness indentation of the cubic consolidated sample is presented in Fig. 5. The average value of the hardness calculated from 60 measurements (10 measurements of each face of the cube) was found to be 19.32 GPa. The cracks displayed in the micrograph that developed in the product during indentation and extended to 192 μm away from it, were used for estimating the fracture toughness, K_c. This value was found to be 17.36 MPa.m$^{1/2}$, well above that of commercial WC/Co composites (12-14 MPa.m$^{1/2}$).

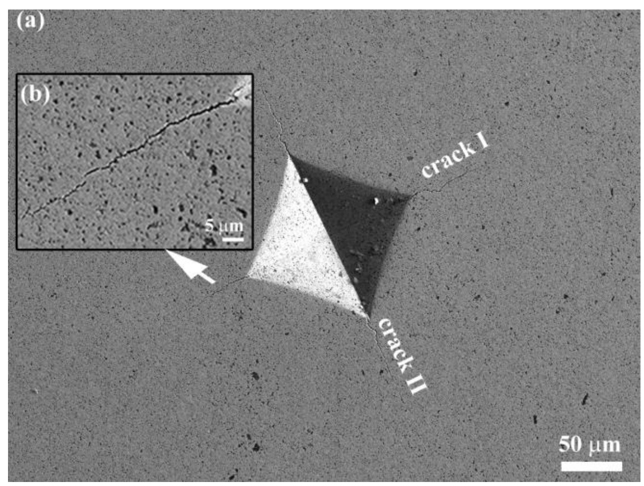

Figure 5: (a) SEM of the Vickers hardness indentation developed by applying a load of 20 kg on the consolidated sample. A one-side radial crack emanating from the Vickers indentation is shown in (b) in a different scale.

The metallographic examinations of the powders after consolidation (Fig. 6(a)) shows fine grain microstructure of WC grains that are embedded into the Co/Cr metal matrix. The average grain size of WC grains were measured and found to be 50 μm in diameter. The triangle indents located in different grains and at the grain boundaries (Fig. 6(a)) refer to the Berkovich indents developed upon examining the sample by nanoindentation. This advanced approach was employed to study the degree of uniformity in the mechanical properties (hardness and Young's modules) at the sub-microscale and nano-scaled. In order to tackle this task over than 300 indents were developed, using single indent continuous multicycle ramp approach. The results have shown that both of the nanohardness and Young's modules are not dramatically varied from grain to grain (Fig. 6(b)) implying the absence of a serious compositional gradient at the micro-scaled. The average values of nanohardness and Young's modules calculated from at least 300 tests are found to be 18.67 GPa and 406.33 GPa, respectively. These values together with the measured value of the fracture toughness (17.36 $MPa.m^{1/2}$) indicate the possibility of fabrication new families of WC-Co based nanocomposites with superior properties at the micro- and nano-scaled.

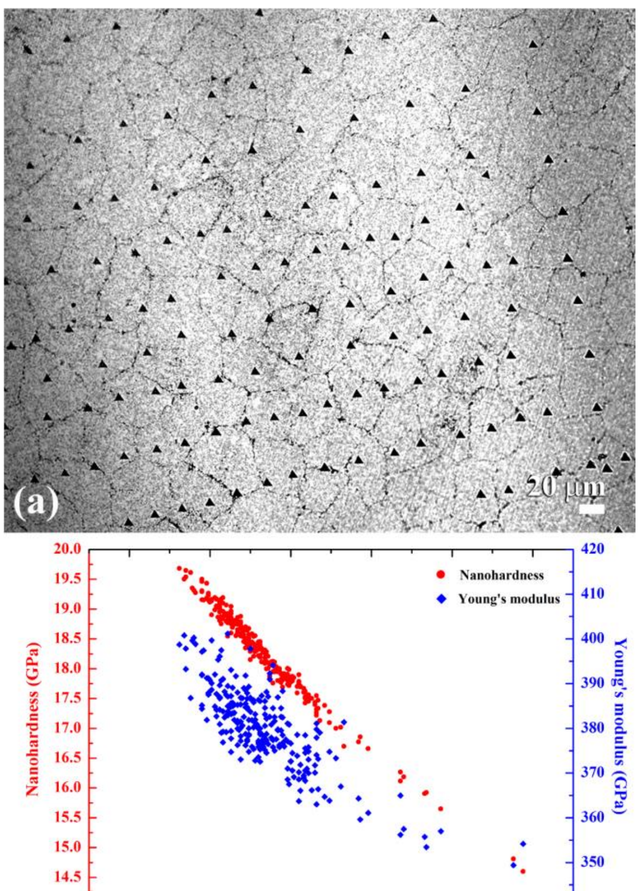

Figure 6: (a) SEM micrograph of the cross-sectional view for the consolidated sample, which shows part of Berkovich indents in different examined zones. The nanohardness-Young's modulus-contact depth relationships of the examined sample are presented in (b).

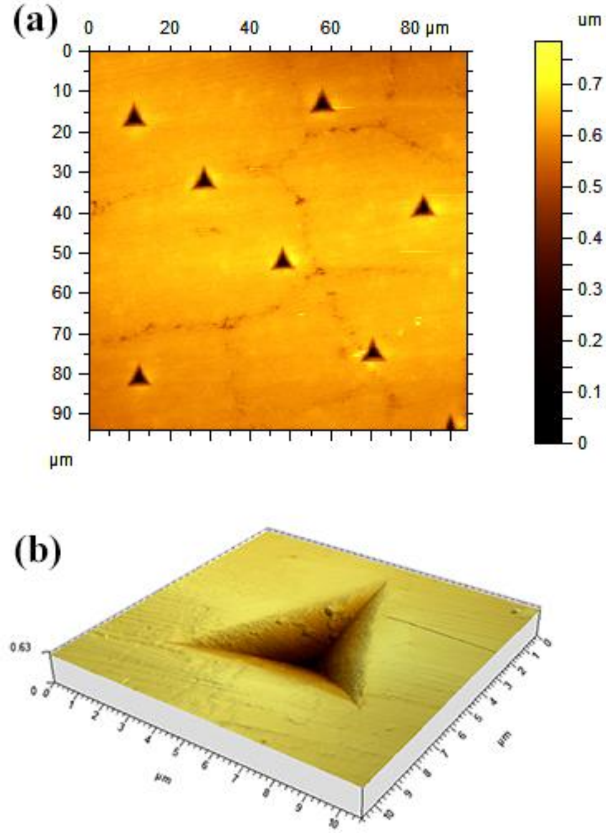

Figure 7: AFM of (a) selected nanoindents image and (b) 3-D image of a single nanoindent for consolidated WC-Co based nanocomposite powder.

AFM micrographs of the nanocomposite bulk material for selected nanoindents developed during Berkovich probe nanoindentation tests, using a load of 400 mN are presented in in Fig. 7. The multi-indents presented in Fig. 7(a) have almost the same dimensions, indicating the homogenity in composition and nanomechanical properties of the nanocomposite materials.

The micrograph of a single nanoindent taken at higher magnification (Fig. 7(b)) does not show sink-in behavior. Sinking-in is expected in materials which exhibit a low value of E/Y (where E is the Young's modulus and Y is the yield strength) [2].

In order to investigate the plastic deformation behavior of as-consolidated WC/Co based nanocomposite material prepared in the present study, high magnification AFM micrographs for the developed nanoindents were performed. The AFM micrographs for selected nanoindents taken at higher magnifications are shown in Fig. 8. The nanoindents exhibit pile-up behavior as shown in both Figs. 8(a) and 8(b). We should emphasis that such pile-up behavior was performed a result of the deflection of WC grains which are pushed upwards by the indenter, a suggested by Ndlovu et al [2].

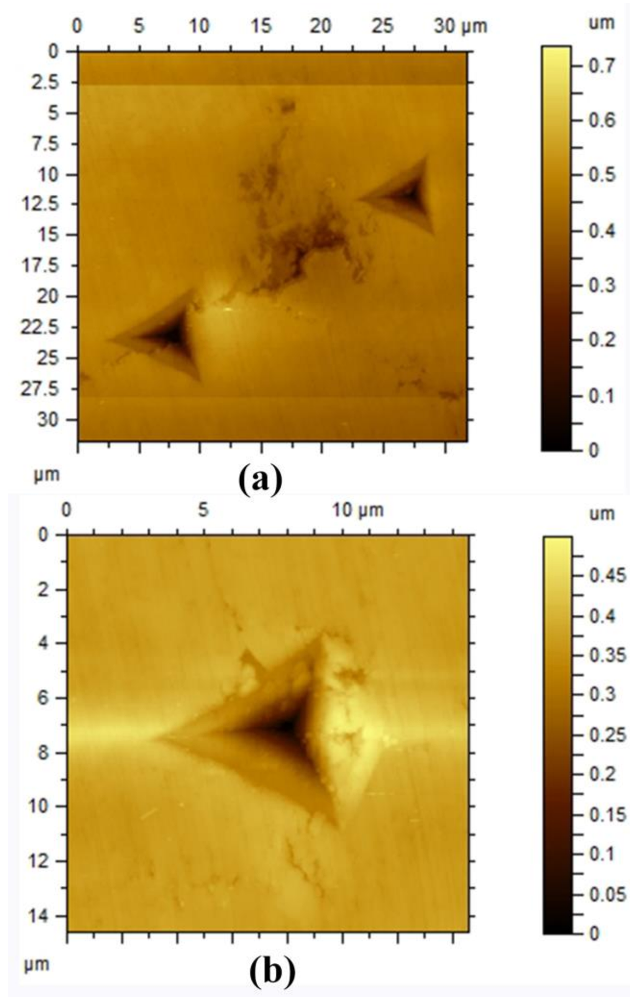

Figure 8: AFM micrographs of (a) two selected nanoindents and (b) single nanoindent for consolidated WC-Co based nanocomposite powder. Both micrographs indicate that the nanoindents exhibit pile-up behavior.

SEM investigations showed that the hard grains of WC located around the nanoindent contained slip lines (see Fig. 9), suggesting that the slip was performed according to the plastic deformation. The formations of such slips were also found in WC grains upon Vickers testing [3].

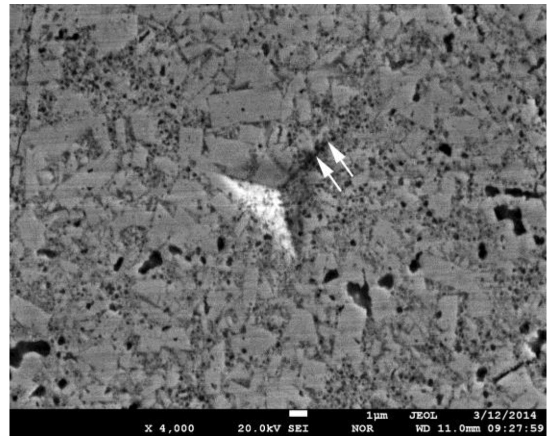

Figure 9: SEM micrograph of as-consolidated WC/Co based nanocomposite. The slip line shown in the WC grains, which are indexed by the arrows, refer to the plastic deformation taken place upon nanoindentation testing.

4 CONCLUSIONS

New nanocomposite system of WC-5Co-1Cr-3MgO-0.7VC-0.3Cr_3C_2 powders were produced by high energy ball milling the elemental powders at room temperature. The end-product powders obtained after 75 h of ball milling were consolidated into full dense bulk materials, using SPS technique. The as-consolidated material showed excellent nanomechanical properties with average nanohardness and fracture toughness of 18.67 GPa 17.36 MPa.m$^{1/2}$, respectively. The AFM technique showed that the nanoindents exhibit pile-up behavior, which suggests the deflection of WC upon introducing a load of 400 mN.

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