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Berkovich nanoindentation and deformation mechanisms in a hardmetal binder-like cobalt alloy

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ABSTRACT

A cobalt-base solid solution is the most used binder for hardmetals (WC–Co cemented carbides) in a wide range of industrial applications. In the composite material such cobalt alloy is surrounded by hard carbides grains; thus, a direct evaluation of its intrinsic mechanical properties is not an easy task. In order to overcome this inconvenience, a model cobalt alloy with a composition similar to that exhibited by typical hardmetal binder (containing W and C in solid solution) was processed following a powder metallurgy route. Nanoindentation testing at different length scales has been performed to study the mechanical properties as well as to introduce plastic deformation in individual grains of the dilute face centered cubic (fcc) stabilized model Co–W–C alloy. It is found that it exhibits an isotropic mechanical response, without any evidence of indentation size effects. The main deformation mechanism activated during the indentation tests resulted to be deformation twinning, although combined with limited planar slip but with no evidence of stress-induced phase transformation. Such finding is related to the high W and C amounts dissolved in the model Co alloy studied, and its effect on stabilizing the fcc configuration and increasing the stacking fault energy. The effective use of small-length scale mechanical characterization protocols is finally discussed for understanding deformation response of binder-like alloys and optimizing microstructural design of hardmetals.

1. Introduction

WC–Co cemented carbides, also known as hardmetals, are well-established materials for cutting and forming tools (e.g. indexable inserts, drills, end-mills and pressing dies), mining bits, and wear resistant and structural components in a variety of industrial applications [1]. Main reasons behind it are the exceptional combination of hardness, toughness and wear resistance that they exhibit, optimized for each specific application by means of microstructural design. From this viewpoint, it must be underlined that cemented carbides may be micromechanically described as composites of ceramic nature reinforced by ductile metallic ligaments, strengthened through the constraint imposed by the very rigid carbide skeleton. Hence, mechanical properties and overall performance of hardmetals are directly dependent upon the intrinsic mechanical response and corresponding deformation mechanisms exhibited by the metallic binder [2]. An example of this statement is the fact that improvements through microstructure control on fracture strength and/or toughness do not directly translate into similar beneficial effects on the fatigue behavior of WC–Co cemented carbides [3–6]. Such behavior has been rationalized on the basis of the double role played by the ductile binder as both toughening and fatigue susceptible agent. Accordingly, fatigue sensitivity becomes then dependent upon the deformation mechanisms within the binder (intimately related to chemical nature of the hardmetal under consideration [3,7,8]); and thus, a deeper understanding of those emerges as a key factor for optimal microstructural tailoring of cemented carbides.

Since the invention of hardmetals in the 1920s, cobalt has been the most widely used binder metal because of the very low interfacial energy, nearly perfect wettability and very good adhesion in the solid state exhibited by the WC and cobalt couple [9]. However, the binder phase within hardmetals is not pure cobalt but rather a cobalt-base solid solution containing small amounts of dissolved carbon and tungsten, i.e. a Co–W–C alloy. Furthermore, as a direct consequence of this (W,C) solid-solution nature, Co-base binder alloys within hardmetals generally exhibits the Co high-temperature face centered cubic (fcc) configuration, instead of the

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room-temperature hexagonal close packed (hcp) one [9,10]. In this regard, existing literature on the mechanical response and deformation mechanisms of (fcc) hardmetal binder-like alloys is quite limited, as the few existing studies have focused on chemical composition – tensile response / corrosion behavior relationships [10–16]. Within this framework, it is the purpose of this study to document and analyze the nanoindentation response and associated deformation mechanisms in a hardmetal binder-like Co–W–C alloy.

2. Experimental procedure

2.1. Material processing and specimen preparation

The Co–W–C alloy specimens were processed through hot isostatic pressing (HIP). The powder used was a pre-alloyed one produced by gas atomization. It was filled into capsules made of pure nickel in order to avoid any diffusion of atoms from the capsule into the powder. Powder consolidation was performed by HIP at pressure and temperature of 100 MPa and 1200 °C respectively, during 120 min. Finally, the samples were machined to remove the encapsulating container and sliced by band sawing and milling.

The as-obtained samples were sequentially polished using silicon carbide paper, diamond suspensions until 0.25 μm, and a neutral suspension of 20 nm alumina particles as final step. X-Ray diffraction data were analyzed by the Rietveld method and refined using the MAUD software [17], considering the presence of a mixture of Co, W and C. For the probed volume, the analysis yielded a composition in weight percent of 87.03% Co, 12.25% W, 0.27% C, and 0.45% of other minor elements. According to the results published by Roebuck et al. [11], such W and C contents are in the relatively high range (i.e. above 8% W and 0.16% C) and must result in a completely stabilized fcc crystal configuration for the alloy under consideration.

2.2. Berkovich nanoindentation

The mechanical characterization of the hardmetal binder-like alloy included the evaluation of their effective hardness (H) and elastic modulus (E) through instrumented indentation. Nanoindentation tests were performed on a nanoindenter XP (MTS) equipped with a continuous stiffness measurement module, the latter allowing a dynamic determination of the mechanical properties (such as hardness and elastic modulus) during the indentation. The mechanical characterization was done with a Berkovich tip, and experimental data were analyzed using the Oliver and Pharr method [18,19]. The indenter shape was calibrated with a fused silica standard [18]. Two different sets of experiments, at micro- and nanometric length scale, were done. The mechanical response of the polycrystalline alloy was assessed as the average behavior of 16 indentations, organized in a regularly spaced 4 by 4 array, at 2000 nm penetration depth (or until reaching maximum applied load, i.e. 650 mN). A constant distance between each imprint of 50 μm was kept in order to avoid any overlapping effect. On the other hand, attempting to evaluate crystal orientation effects on hardness and elastic modulus, as well as to induce plasticity within single grains, three different spaced arrays of 400 imprints (20 by 20, and separated about 5 μm from each other) were done at 200 nm of maximum penetration depth.

2.3. Crystallography and deformation mechanisms characterization

Crystal orientation was assessed by means of electron backscattered diffraction (EBSD). It was conducted in a field emission scanning electron microscopy (FESEM) using a JEOL 7001F unit equipped with an orientation imaging microscopy system. The diffraction response of grains oriented with a surface normal near the basal direction was sufficient for indexing with beam current in the order of 1 nA. EBSD measurements were performed with a constant scanning step (50 nm) at an acceleration voltage of 20 kV. Phase orientation analysis confirmed, as it was referred above, the fcc crystal configuration for the as-sintered Co–W–C alloy (Fig. 1)

Transmission electron microscopy (TEM) was used for detailed study of mechanisms associated with deformation induced during nanoindentation. Hence, TEM lamellae were extracted by focused ion beam (FIB) using a dual beam Workstation (Zeiss Neon 40). In doing so, prior to milling a thin platinum layer was deposited on residual imprints to be studied. A Ga^{+} ion source was used, and current and acceleration voltage were continuously decreased up to a final polishing stage at 5 kV and 10 pA. Deformation features within the FIB-milled lamellae were examined first in a scanning transmission electron microscopy (STEM) coupled with the Zeiss Neon 40 unit, and later in a TEM equipment (JEOL JEM 2100) operating at 120 kV.

3. Results and discussion

3.1. Hardness and elastic modulus by instrumented indentation at micro- and nanometric length scale

Hardness and elastic modulus as a function of the maximum penetration depth is shown in Fig. 2 for the fcc cobalt-base binder model alloy at micrometric length scale. Surface roughness and indenter tip effects on the mechanical response are discerned at very low penetration depths. However, hardness and elastic modulus values tend to stabilize, within the experimental error range, as penetration depth gets higher than 125–250 nm. After it, mechanical properties remained stable for the range of loads and penetration depths of study, i.e. size effects were not evidenced. Hardness and elastic modulus for the alloy studied are found to be 4.8 ± 0.2 GPa and 230 ± 7 GPa respectively, at the micrometer length scale. These hardness values are somehow higher than those previously reported by Roebuck et al. [11] on similar dilute Co–W–C alloys. Main reason for such a difference is the higher carbon content dissolved in the alloy studied in this investigation. On the other hand, elastic modulus values are in satisfactory

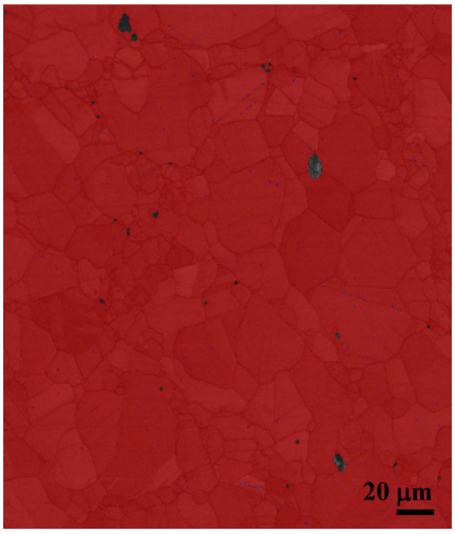

Fig. 1. EBSD map (phase image superimposed to the quality image) corresponding to the microstructure for the hardmetal binder-like cobalt alloy. Pixel size: 50 nm. The fcc phase is presented in red and the non-indexed pixels in black.