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Alternative Ni-based cemented carbide binder – Hardness characterization by nano-indentation and focused ion beam

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ABSTRACT

The nano-hardness in the alternative ^{85}Ni-^{15}Fe binder phase of WC cemented carbide has been investigated. High-resolution scanning electron microscopy (SEM) imaging was used to measure the projected indentation area and a general pile-up correction, confirmed on selected indents, has been employed using atomic force microscopy (AFM). Focused ion-beam (FIB) cross-sections have been used to investigate the binder morphology below the indentations and the local binder hardness has been associated to the distance to the surrounding WC grains. Generally, decreasing distance to the WC grains leads to increased binder hardness. Furthermore, the nano-hardness for the cemented carbide binder has been corrected for the indentation size effect (ISE) to obtain the corresponding macroscopic hardness. A solid solution strengthening model for multicomponent bulk alloys was used to calculate the expected binder Vickers hardness considering the binder solubilities of W and C. Both the strengthening model and the ISE corrected hardness values, for larger binder regions, are in good agreement indicating that the intrinsic binder phase hardness is similar to that of a bulk metal alloy with similar composition.

1. Introduction

The binder of cemented carbide systems became the focus of recent research interest due to the potential need for alternative binder systems [1]. A prospective framework to systematically investigate new binder systems is the Materials Design approach embedded in the ICME (Integrated Computational Materials Engineering) methodology. The development of next generation material property models requires detailed experimental or theoretical data which is only available in limited form. The hardness of different binder systems is one of the missing links between the composition and composite properties which appears to be important for the development of a new model to predict the cemented carbide hardness [2]. Only limited data is available for the Cobalt (Co) binder hardness [3–7] but none for alternative binder systems. The available literature data for Co binders often gives average values around 8 GPa but it is suggested that the Co binder hardness takes values in the range from 4 to up to 17 GPa which is explained by a Hall-Petch like increase of the binder hardness with decreasing binder mean free paths [3,6–8]. A detailed investigation of the binder shape combined with nano-indentation has not been available yet making it difficult to provide a clear value for the intrinsic binder hardness, which is required for modeling purposes [2].

The present study analyzes nano-indentations, performed in the cemented carbide binder, and relates the indentations to the actual local 3D microstructure using a subsequent focused ion-beam (FIB) cross-sectioning.

2. Experimental information

A WC-^{85}Ni^{15}Fe cemented carbide [9] with about 20 vol% binder has been synthesized by liquid state sintering with a resulting WC grain size of 1.168 μm, a composite macro hardness of 1221 HV30 and a K_{IC} value of 15.6 MPa·m$^{1/2}$. The sample was mounted in resin and mechanically polished (9 μm & 1 μm diamond suspension and 0.02 μm colloidal silica) in preparation for the nano-indentation testing.

This work focuses on the investigation of the binder hardness using nano-indentations performed on a NanoIndenter II (Nano Instruments Inc., Oak Ridge, TN, USA) with a diamond Berkovich tip. The positioning control of the used instrument was too limited to perform individual indents in the micrometer-sized binder features. Instead, several matrices with varying number of indents and a fixed interval of 2 μm were made in proximity of the larger binder regions. With a generally limited size of the individual binder feature the indentation depth was set to 100 nm to keep the indentation volume small compared to the investigated features. This was necessary in order to investigate the binder hardness in regions with a binder-mean-free path

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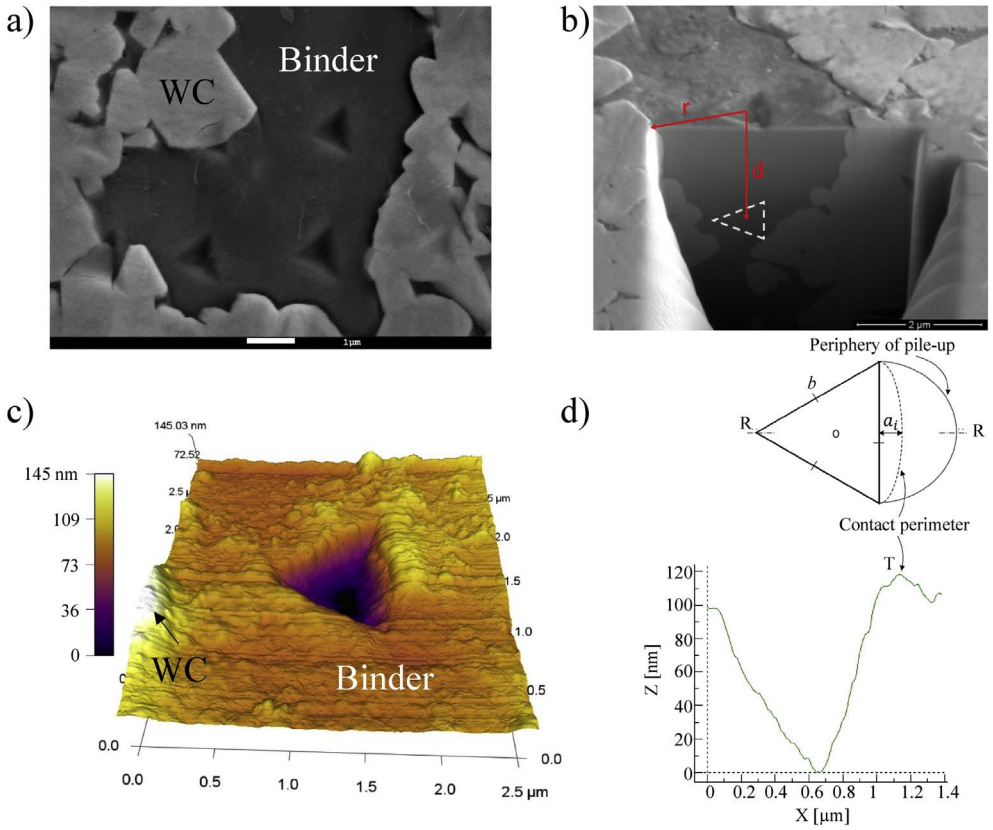

Fig. 1. a) example of indents positioned in the binder phase b) FIB cross-section aside a nano-indent c) AFM image to investigate the material pile-up d) schematic sketch of pile-up area measurement [11–13].

down to ~1 µm without significant interaction with the surrounding carbides. The tests have been load rate controlled (200 µN/s) with 20 s holding time at the peak load.

The projected indentation area, produced by the diamond Berkovich tip, was primarily investigated using high-resolution SEM (JEOL JSM-7800F) imaging. Selected indents were analyzed using an AFM instrument (D3000 Nanoscope from Digital Instrument with a silicon nitride tip) to obtain more detailed information about the projected area and the material pile-up emerging from the indentation tests. Usually the hardness evaluation is based on 2D images of the sample surface. To obtain a better understanding of the relation between the individual indention results and the morphology of the binder phase all indents have been analyzed with regards to the 3D morphology by FIB cross-sectioning. A FEI Nova 600 NanoLab dual-beam system, using an acceleration voltage of 30 kV and a current of 0.5 nA for the Ga ion-source, was used to perform the FIB cross-sectioning.

3. Indentation results and binder shape

High-resolution SEM has been used to confirm the correct positioning of the indents in the cemented carbide binder (Fig. 1a). In addition, FIB cross-sectioning gives an indication of the binder shape appearance below the nano-indent (Fig. 1b). By measuring the radius *r* and the depth *d* to the nearest WC grains, around or below the indent, it is possible to relate the indentation result to the real three-dimensional binder.

In Table 1 we present three different hardness values associated to the indents in the binder. The first value was calculated using the projected area as measured by high-resolution SEM and the maximum load. It is known [10] that material pile-up may be found in connection to nano-indents in pure Ni. From the visual inspection it is evident that all indents are subject to pile-up formation and this is confirmed by AFM on selected indents (Fig. 1c). The method proposed by Kese et al. [11,12] is used to correct the projected area for the pile-up contribution (Fig. 1d) and the second hardness value is obtained using this method. AFM measurements were performed for indents #11, 12 and 14 and for these the effect of the pile-up on the hardness is taken into account. These values are, on average, 17% lower and the hardness for the other indents that have not been measured by AFM, but where pile-up is evident from the high-resolution images, are thus reduced by 17%. The third hardness value is obtained after correcting for the indentation-size effect (ISE) which will be further discussed in Section 4.4.

4. Discussion

Considering the results previously shown, the discussion is divided into four parts focusing on the binder shape and its relation to the binder hardness (4.1 to 4.3) and a discussion on the ISE (4.4) to investigate the macroscopic binder hardness compared to a solid solution strengthening model for multicomponent bulk metals.

4.1. Bulk binder hardness

With increasing interest in alternative binder systems the assessment of the related mechanical binder properties is important for the development of new cemented carbide systems. From a modeling perspective, the lowest binder hardness is interesting since it gives an idea of the minimal achievable composite hardness and offers one opportunity to make different binder systems easily comparable. Several investigations [3,5–7] on WC-Co systems have proposed that the binder hardness is significantly larger compared to bulk Co alloys [14]. The binder phase in cemented carbides systems is surrounded by the WC grains which has been suggested to constrain the binder and thus to increase the measured hardness compared to a comparable bulk alloy.