



Nanoindentation study of WC–12Co hardmetals obtained from nanocrystalline powders: Evaluation of hardness and modulus on individual phases

V. Bonache*, E. Rayón, M.D. Salvador, D. Busquets

Instituto de Tecnología de Materiales, Universidad Politécnica de Valencia, Camí de Vera s/n. E-46022, Valencia, Spain

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ABSTRACT

With the development of finer hardmetals, the study of mechanical properties of the different constituents down the micrometric level is a question of concern for materials optimization. Nanoindentation has been developed in last years in order to cope with mechanical characterisation at the nanolevel, but difficulties on phase detection are an issue. In the present work, individual hardness and Young's modulus of the constituents of WC–12Co composite were obtained by the use of very shallow nanoindentations (30 nm depth), with the aid of in situ 3D imaging to identify these. By this method three different phases at the sub-micrometric level have been identified and characterised: namely cobalt matrix, tungsten carbide and η phase. The presence of the latter phase and its characterisation is of paramount importance in understanding the behaviour of hardmetals. Values of hardness from 8 (cobalt matrix) to 25 GPa (η phase) have been obtained. Also, for these phases Young's modulus varied from 250 to 400 GPa respectively. Furthermore, it is firstly reported these values for the WC prismatic planes $\{1010\}$ being in the range of 40–55 and 700–900 GPa respectively. These values decrease to a hardness in the range of 25–30 GPa and modulus in the range from 450 to 550 GPa for the WC basal plane $\{0001\}$.

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1. Introduction

Hard metals based in cemented carbides, cermets, are widely used in cutting, forming and machining tools in many different industrial sectors (for instance from metal to wood or mining industry), because of their high hardness and strength, good fracture strength, high temperature capabilities and excellent wear resistance [1,2]. The WC–Co cermets are basically constituted by a homogeneous distribution of hard WC faceted grains embedded in a ductile matrix of cobalt.

The properties of these materials are mainly dependent on its composition, microstructure and chemical purity of carbides [3]. In general, reducing WC particle size produces a marked improvement of the mechanical properties, increasing hardness, wear resistance and transversal rupture strength, without significant loss of fracture toughness [2]. This superiority of the submicron and ultrafine grades, with the ability to have micrometer dimension and rounded shaped tools, has led to the introduction and rapid expansion in different market for these grades. These encompass some special applications as wood cutting tools and microdrilling

for printed circuit boards (PCBs) to a great variety of applications as cutting tools for non ferrous materials and plastics, mining, iron and steel casting and wear parts [4].

Although the advantages of reducing WC grain size are generally recognized, the production of these materials presents a number of unresolved processing difficulties. Nanocrystalline powders present a faster sintering kinetics, which renders more difficult to control the phenomena leading to grain growth [5–7]. In addition, its higher specific surface area causes a higher moisture and oxygen absorption increasing oxidation rate. This can lead to a severe decarburization during sintering, which involves significant formation of secondary phases (η and η' phase), with detriment of the mechanical properties [8].

The mechanical properties of bulk nanostructured WC–Co composites, such as hardness, fracture toughness, and wear resistance, were reported in several studies [9]. However the mechanical response of a single crystal or phase in the fine and ultrafine cemented carbides has not been studied before. Some authors [10–12] showed the mechanical response of the cermets by coupling nanoindentation and SEM techniques although the mechanical properties of single phases were ruled out in the study. Deepening the knowledge of nanomechanical properties of the composite would be useful to develop new models to simulate the macroscopic mechanical properties and vice-versa. For this reason and in order to perform these analyses in the nanometric range a specific technique as the nanoindentation is required. In fact, by

* Corresponding author at: Instituto de Tecnología de Materiales, Edif. 5E.

1ª Planta, Universidad Politécnica de Valencia, Camí de Vera s/n. E-46022, Valencia, Spain. Tel.: +34 692512435; fax: +34 963877629.

E-mail address: vicbobe@doctor.upv.es (V. Bonache).

carrying out indentations at very low loads, indentation marks fall below the size of single phases in the fine and ultrafine composite materials.

The nanoindentation technique has shown an important development to measure the mechanical properties of thin films and multiphase materials [13] by precise control and measuring of very small displacements and loads applied by the indenter. The main mechanical characteristics that can be obtained from a nanoindentation technique are hardness, Young's modulus, adhesion, friction coefficient and crack growth [14–17].

Due to the small marks produced by the low loads in a nanoindentation experiment, the traditional optical measurements cannot be performed. Instead of this, the basic method to obtain the hardness and modulus values without direct observation of mark in nanoindentation is based in the Oliver and Pharr procedure [18,19]. They showed that the contact stiffness, S of a material can be experimentally measured from the slope of the unloading data in a typical load–displacement curve. The unloading curve is described by a power law and the unload contact stiffness is then established differentiating the power law at the maximum load, $F_{\max}$ and maximum depth, h_t reached in an indentation. Based on the theory of rigid contact relationships developed by Sneddon [20,21] the reduced modulus can be calculated by the follow relationship of the contact stiffness and the contact area, A :

$$E_r = \frac{\sqrt{\pi}S}{2\beta\sqrt{A}} \quad (1)$$

where β is a geometry indenter constant ($\beta=1.05$ and for a Berkovich tip). The contact area function describes the projected area of the indenter assuming that sink-in and not pile-up occurred in the elastic contact, being $A = 24.56h_c^2$ for a Berkovich tip. The contact depth h_c is usually $h_c < h_t$ for elastic materials. The elastic modulus, E of sample is determined using the expression:

$$E_r = \left[\frac{(1-\nu_i)^2}{E_i} + \frac{(1-\nu)^2}{E} \right]^{-1} \quad (2)$$

For this calculation, the Poisson's ratio and elastic modulus of the indenter, ν_i^2 and E_i respectively, is required (for diamond $\nu_i^2 = 8.72e^{-13}$ and $E_i = 1141$ GPa). Hardness is calculated by the quotient of $F_{\max}$ and contact area

$$H = \frac{F_{\max}}{A} \quad (3)$$

In this way, this method works well for the majority of metallic and ceramic materials, although presenting some deficiencies as errors in the measurements when pile-up occurs and the impossibility of measuring time–response materials. Furthermore, the fact that the stiffness contact is measured only at the maximum penetration implies to get a limited number of measures in depth.

An improved technique for determining hardness and elastic modulus is based in a dynamic test, also called continuous stiffness measurement (CSM) [22]. The CSM method enables to obtain continuous hardness and modulus profiles during loading with high accuracy and resolution, improving also the surface detection [23,24]. Moreover, the viscous behaviour can be directly calculated and time-dependent response materials analyzed [25–27].

The CSM technique superimposes a dynamic load of a set frequency, ω on the single load–unload test. The displacement oscillates at the same ω that the force, but lags by a phase angle, ϕ . This mode permits us to measure the phase and amplitude deviation observed between an excitation force, F_0 and the resulted force F :

$$F(t) = F_0 e^{i\omega t} \quad (4)$$

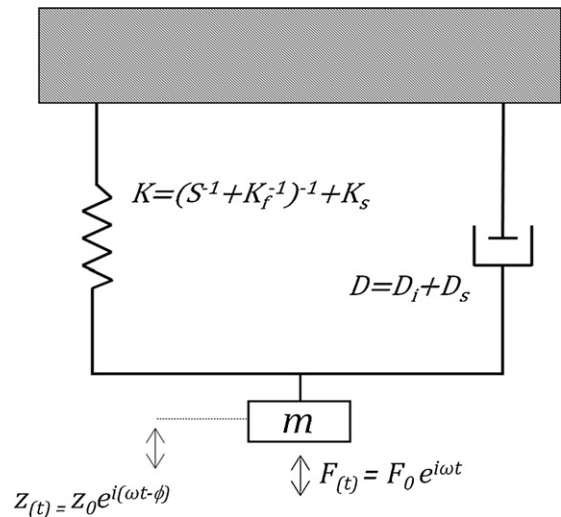


Fig. 1. Simple harmonic oscillator scheme used to model dynamic response of the CSM head.

and between the displacement amplitude z_0 in air (no contact), and response wave $z(t)$, in the form:

$$Z(t) = z_0 e^{i(\omega t + \phi)} \quad (5)$$

The phase and amplitude of the material's response to the force oscillations are characteristic of the contact stiffness as well as the damping coefficient of the material, D . Fig. 1 shows an equivalent harmonic oscillator model that agree well with an indentation head system which motion is restricted to one dimension. Due to the effect of each element of the oscillator module, an equivalent stiffness K is required in order to include the stiffness of the contact, the load frame stiffness K_f , and the stiffness of the support springs, K_s that is:

$$K = (S^{-1} + K_f^{-1}) + K_s \quad (6)$$

The equivalent damping coefficient D , is the result of the indentation head's damping coefficient D_i and sample's coefficient D_s

$$D = D_i + D_s \quad (7)$$

The analysis of this simple harmonic oscillator subject to a force summation on the mass, m , is analyzed by an ordinary differential equation:

$$F(t) = mz'' + Dz' + Kz \quad (8)$$

Substituting the particular solution of Eq. (5) in Eq. (8) and equating magnitudes yields:

$$S = \left[\frac{1}{F_0/h_0 \cos \phi - (K_s - m\omega^2)} - K_f^{-1} \right]^{-1} \quad (9)$$

Once S is obtained, the continuous elastic modulus and hardness profiles are calculated by means of Eqs. (2) and (3) respectively.

Either the way, this technique permits to take measures of hardness and modulus at very low depths, but the problem of where the indentations are performed is still present. In fact, the marks produced for a Berkovich tip is 6 times the depth reached. This means that for a 30 nm depth indentation, an 180 nm mark size would be obtained on the surface of sample. When indentations are done in a composite or multiphase material knowing where the indentation was performed is the critical issue. To solve this question, some strategies have been followed. One consists of printing a nanoindentation and after study the marks in an AFM, TEM or SEM microscopes [28–30]. However, this sequential procedure

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