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On the determination of the film hardness in hard film/substrate composites using depth-sensing indentation

N.A. Sakharova[a,*], M.C. Oliveira[a], J.M. Antunes[b], J.V. Fernandes[a]

[a]CEMUC-Department of Mechanical Engineering, University of Coimbra, Rua Luís Reis Santos, Pinhal de Marrocos, 3030–788 Coimbra, Portugal

[b]Escola Superior de Tecnologia de Abrantes, Instituto Politécnico de Tomar, Rua 17 de Agosto de 1808, 2200 Abrantes, Portugal

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Abstract

The main difficulty in the mechanical characterisation of thin films using depth-sensing indentation is the determination of the relative substrate and film contributions to the measured properties of the film/substrate composite. In this study, a three-dimensional numerical simulation of the Vickers hardness test is used to study the influence of the substrate and film mechanical properties on the composite's behaviour under depth-sensing indentation. The particular case of hard films on soft substrates is analysed. In order to understand the behaviour of the composite, a study of the plastic strain distribution under indentation of several composites is performed. A methodology to determine the relative film hardness, i.e. H_F/H_S ratio, is proposed. The methodology is successfully verified using fictitious and real composite materials.

Keywords: B. Composites; C. Hardness; Vickers indentation; Numerical simulation

1. Introduction

Hardness is one of the most important mechanical properties in the mechanical characterisation of materials. The depth-sensing indentation technique is commonly used for evaluation of the hardness of bulk and coating materials. In the case of thin coatings, elastic and plastic deformations can occur in the film and the substrate, during the hardness test. Therefore, the main difficulty in evaluating film hardness is to distinguish the contribution of the substrate from that of the film and the measured composite hardness, H_C, depends on the film and substrate hardness, H_F and H_S, respectively. This is especially so for small thicknesses and/or when the ratio between the film and substrate hardness (H_F/H_S) is very different from unity [1]. Consequently, for the maximum indentation depths commonly used in experimental tests, the composite hardness, H_C, is a function of the film and substrate mechanical properties. In this context, the composite hardness depends on the maximum applied load or, as usually expressed, on the relative contact indentation depth, h_c/t, *i.e.* the ratio between the contact indentation depth, h_c, and the film thickness, t.

In some applications, the film thickness can be lower than ten nanometres. In these cases, low indentation depths are required for determining film hardness, since the plastic response of the composite can significantly deviate from that of the film, for indentation depths of only around 10 to 15% of the film thickness (see, for example, [2,3]). As previously stated, the critical indentation depth depends on the film and substrate mechanical properties, namely hardness (or yield stress) and Young's modulus ratios [1,4]. Moreover, in an investigation [5] on the critical indentation depth rule, according to which the substrate has a negligible effect on the composite hardness, divergences were found. These authors established that the critical indentation depth is sensitive to film structure (crystal, polycrystalline or amorphous), which determines the plastic deformation mechanisms and, consequently, the size of the region under the indenter that suffers from plastic strain. This shows the important role that this region plays in the composite behaviour for relatively low indentation depths, as stated in [6].

*Corresponding author. Tel.: +351 239790700.
E-mail address: nataliya.sakharova@dem.uc.pt (N.A. Sakharova).

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The experimental procedure associated with the use of depth-sensing indentation tests requires a careful preparation of the sample surface. This is particularly important, when using very low indentation depths, which reduce the accuracy of the evaluated mechanical properties. In fact, uncertainties in the depth-sensing indentation data due to the roughness of the sample surface and the indenter's geometrical imperfections are inevitable, to a certain extent (see, for example, [7–9]).

Therefore, alternative methods have been proposed for determining the film's hardness considering the effect of the substrate on the film mechanical properties. The typical approach for solving this problem consists of performing hardness tests at relatively high indentation depths, then separating the contributions of the film and the substrate in the composite response. In a number of experimental and theoretical studies, different phenomenological and empirical weight functions for extracting the film hardness from the composite indentation data have been proposed, e.g. [10,11]. These functions are expressed by the general formulation:

$$\frac{H_C - H_S}{H_F - H_S} = \alpha(h_c/t), \tag{1}$$

where $\alpha(h_c/t)$ is a function of the ratio h_c/t (or its inverse t/h_c).

In some models, the $\alpha(h_c/t)$ function contains parameters with fixed values, in such a way that a unique equation can be used, whatever the film and substrate mechanical properties. For example, in case of the well-known Jönsson and Hogmark model [10], this function is:

$$\alpha = 2D\left(\frac{t}{d}\right) - D^2\left(\frac{t}{d}\right)^2, \tag{2}$$

where d is the diagonal of the Vickers indentation ($d = 7h_c$) and D is a predetermined parameter ($D \approx 1$ if, during indentation, only plastic strain occurs; and $D \approx 0.5$ if cracking also occurs in the coating). For applying this type of model, it is only necessary to perform a single hardness measurement on the composite, for a given h_c/t ratio, which is an advantage.

However, experimental results clearly show that the behaviour described by the $\alpha(h_c/t)$ function is not unique, but depends on the film and substrate mechanical properties and, possibly, on the indentation size effect of both materials (see for example [6,12,13]). In fact, experimental plots of $(H_C - H_S)/(H_F - H_S)$ *versus* h_c/t (or t/h_c) pointed out that enormous differences in this behaviour can be observed, see for example [1,11].

A few $\alpha(h_c/t)$ functions have been proposed to be fitted to the experimental evolution of the composite hardness results *versus* the relative contact indentation depth, h_c/t; the parameters of these functions depend on the properties of the composite material. For example, in the case of the Korsunsky model [14]:

$$\frac{H_C - H_S}{H_F - H_S} = \frac{1}{1 + k(h_c/t)^2}, \tag{3}$$

where k is a dimensionless parameter, given by the ratio: $k = t/\gamma$; in which γ, that has the dimension of length, is linked to the fracture toughness. For the fracture-dominated case (when facture occurs) γ is proportional to the film thickness, t; while for the plastically-dominated case (when fracture does not occur), γ mainly depends on the ratio H_F/H_S and is weakly dependent on t. The quality of the fit used in this model depends on the experimental hardness data obtained over a wide range of h_c/t values, which implies that nanoindentation data are taken in account. Otherwise, there are insufficient data points for values close to zero to provide a good quality of the fit. Therefore, the main difficulty associated with this type of models is obtaining the correct extrapolation for h_c/t equal to zero.

In the literature, experimental results show three different regions in the plot of $(H_C - H_S)/(H_F - H_S)$ (or H_C) as a function of t/h_c (or h_c^{-1}, for a given film thickness of the composite) (see for example [1,15]). In one of these regions, which corresponds to relatively low indentation depths (i.e. high values of t/h_c), the evolution tends to gradually saturate ($(H_C - H_S)/(H_F - H_S) \rightarrow 1$). In the opposite region, for relatively high indentation depths (low values of t/h_c), the value of $(H_C - H_S)/(H_F - H_S)$ remains close to zero, from the origin of the axes up to a t/h_c value, depending on the composite, after which it shows a positive concavity. Finally, a relatively large region between the two previous regions with a linear evolution of $(H_C - H_S)/(H_F - H_S)$ *versus* t/h_c is observed, (see for example [13,16,17]).

In this context, we report a three-dimensional numerical simulation of the Vickers hardness test of several composites in order to study the linear region of the function $(H_C - H_S)/(H_F - H_S)$ *versus* t/h_c. Based on the numerical simulation results, a methodology for evaluation of the film hardness is developed. This approach consists of a simple method, which considers the hardness behaviour of the composite described by a linear $\alpha(h_c/t)$ function, with parameters that depend on the film/substrate system, and which avoids the use of indentation data obtained for very low indentation depths. The proposed methodology is validated using numerical and experimental indentation results.

2. Theoretical aspects

Depth-sensing indentation is commonly used for determining materials' hardness and Young's modulus. The hardness, H_{IT}, is evaluated by:

$$H_{IT} = \frac{P}{A}, \tag{4}$$

where P is the maximum load and A is the indentation contact area at maximum load.

The mechanical property results strongly depend on the accuracy of the value of the indentation contact area. In the case of Vickers or Berkovich ideal indenters, the contact area,