

Letter

## Critical relative indentation depth in carbon based thin films

Ruben Bartali\*, Alessandro Vaccari, Victor Micheli, Gloria Gottardi, Rajesh Pandiyan,  
Amos Collini, Paolo Lori, Gianni Coser, Nadhira Laidani

Fondazione Bruno Kessler – Ricerca Scientifica e Tecnologica, Center for Materials and Microsystems, via Sommarive 18, 38050 Povo (Trento), Italy

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### Abstract

The thin film hardness estimation by nanoindentation is influenced by substrate beyond a critical relative indentation depth (CRID). In this study we developed a methodology to identify the CRID in amorphous carbon film. Three types of amorphous carbon film deposited on silicon have been studied. The nanoindentation tests were carried out applying a 0.1–10 mN load range on a Berkovich diamond tip, leading to penetration depth-to-film thickness ratios of 8–100%. The work regained during unloading ( $W_e$ ) and the work performed during loading ( $W_l$ ) was estimated for each indentation. The trend of unload-to-load ratio ( $W_e/W_l$ ) data as a function of depth has been studied.  $W_e/W_l$  depth profiles showed a sigmoid trend and the data were fitted by means of a Hill sigmoid equation. Using Hill sigmoid fit and a simple analytical method it is possible to estimate CRID of carbon based films.

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**Keywords:** Nanoindentation; Carbon film; Critical relative indentation depth;  $W_e/W_l$

### 1. Introduction

Carbon-based thin films show wide range of mechanical properties as a function of hydrogen content, sp<sup>2</sup>–sp<sup>3</sup> ratio, stress, density and structure [1,2]. The mechanical properties of thin film estimated by means of nanoindentation are influenced by substrate [3]. Different approaches can be used in order to remove any substrate effect [4–7], but the main approach is that the critical relative indentation depth (CRID) should not exceed 10% of the total film thickness. These approach is quick but not accurate, for instance in the case of aluminum on silicon [8]. In this work we developed a methodology that allows CRID evaluation directly from the energy response of the material during the indentation process. Special interest is given to the unload to-load work of nanoindentation,

which represents the ratio between the total work ( $W_l$ ) done and the recovered work (elastic) during indentation as shown in Fig. 1. It allows us to separate the indentation work into elastic ( $W_e$ ) and plastic components ( $W_p$ ) since  $W_l = W_p + W_e$ . The  $W_e/W_l$  ratio determination is straightforward from the load–unload curves. It does not need any model application and is not affected by shape of stylus, state of material (bulk or thin film) or of the applied load which generally are critical for hardness and elastic modulus estimation [9]. Moreover, for carbon based coatings,  $W_e/W_l$  as function of depth have a sigmoidal trend and this trend can be fitted by sigmoid Hill equation [10]. For this reason we first study the Hill sigmoid equation to estimate CRID point under mathematical point of view. And then we apply the method developed for the  $W_e/W_l$  trend of the carbon-based films. In the end we compare the CRID points obtained with results using equation suggested by Bull to estimate CRID value in the thin film [11].

### 2. Experimental details

In this experiment three types of a-C:H coatings were used [12]. The first kind of coating was deposited by sputtering

\*Corresponding author. Tel.: +39 0461 314471; fax: +39 0461 810 851.

E-mail address: [bartali@fbk.eu](mailto:bartali@fbk.eu) (R. Bartali).

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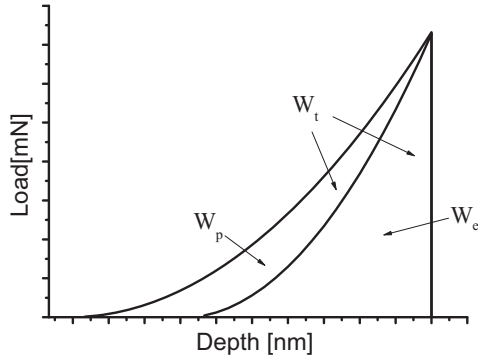


Fig. 1. Total ( $W_t$ ), plastic ( $W_p$ ) and elastic ( $W_c$ ) works of indentation.

from a graphite target using a RF (13.56 MHz in continuous wave mode) with a thickness of  $\sim 400$  nm. This was indicated as

C-1. The second kind (indicated as C-2) was deposited at the same condition but it was thicker than the C-1 ( $\sim 530$  nm). The third film (C-3) was deposited by pulsed plasma and the thickness of the coating was 300 nm.

The substrates for all coating was the 400  $\mu\text{m}$  thick n-type Si (100). All the coatings grew at room temperature. The pressure was 0.05 Torr and an Ar/H<sub>2</sub> mixture gas was used.

2.1. Mechanical measurements

Film thickness was measured using a KLA Tencor P15T profilometer. The mechanical properties were measured using a CSM Nano Hardness Tester. The indentations were obtained by a three side pyramid diamond Berkovich indenter, and the Oliver and Pharr model [13] has been applied for the analysis of the data. All data reported here are average values over four measurements for each load, and the error by root mean square (rms) has also been calculated. The mechanical properties are reported as a function of indentation depth normalized to the film thickness ( $D$ ). The roughness of the film surface was profiled by Atomic Force Microscopy (AFM), in contact mode with a SIS Ultra Object Instrument. For the present films, the values of roughness ( $Ra$ ) are measured on an area of  $5 \mu\text{m} \times 5 \mu\text{m}$ .

3. Treatment of data

Hill equation generally describes the cooperative or non-cooperative transition from two states of a bi-component system [14,15,16]. In a heterogeneous structure composed by a coating on a substrate there are two states as well: a zone where the mechanical properties are related to the coating (C) and a zone where the mechanical properties are related to the substrate (S), and both of them are filled by  $n$  binding sites. The  $W_e/W_t$  ratio as a function of depth might then be described by a Hill sigmoid

$$y = A_1 + (A_2 - A_1) \frac{x^n}{k^n + x^n} \tag{1}$$

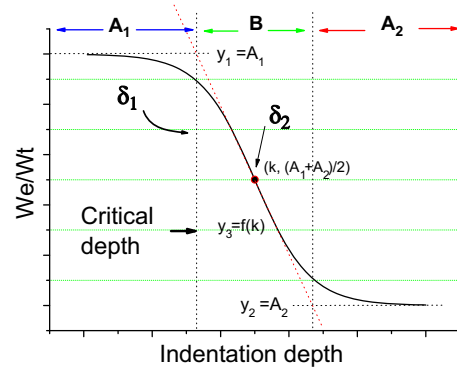


Fig. 2. Trend of Hill sigmoid equation,  $\delta_1$  is the critical relative indentation depth,  $\delta_2$  is the center of the sigmoid.  $A_1$  is the region where the mechanical properties may be correlated only to the film,  $B$  is the transition zone,  $A_2$  is the region where the properties are mainly due to the substrate.

where  $y$  denotes the  $W_e/W_t$  ratio, the independent variable  $x$  is indentation depth to the film thickness ( $D$ ),  $A_1$  and  $A_2 < A_1$  are the upper and lower limits for  $y$  ( $A_1 \leq y < A_2$ ), and  $k$  is the abscissa of the sigmoid center. Fig. 2 graphically illustrates all of these parameters. We point out two important values named critical depths  $\delta_1$  and  $\delta_2$ . The latter point  $\delta_2$  corresponds to the sigmoid center abscissa  $k$ , where  $y$  gets the value of  $(A_1 + A_2)/2$ . On the other hand,  $\delta_1$  is the singular point near the surface indicating the boundary of the region  $A_1$ . The point  $\delta_1$  is obtained from the intersection of the straight line  $y = A_1$  and the tangent to the sigmoid passing through its center, that is the point of the  $xy$  plane with coordinates  $x_0 = k$  and  $y_0 = (A_1 + A_2)/2$ . The slope of the tangent line at the sigmoid center is

$$\left(\frac{dy}{dx}\right)_{x=x_0} = \frac{(A_2 - A_1)n}{4k} \tag{2}$$

And  $\delta_1$  is obtained as the  $x$  solution of the following system of equations:

$$\begin{cases} y - y_0 = \left(\frac{dy}{dx}\right)_{x=x_0} * (x - x_0) \\ y = A_1 \end{cases} \tag{3}$$

After simplification one finally obtains

$$\delta_1 = k * (1 - 2/n) \tag{4}$$

Therefore, as suggested by Eq. (4), knowing only two parameters of the fit, the critical point  $\delta_1$  can be analytically determined. The  $A_1$  represents the superficial region where the mechanical properties of the films are dominant and  $\delta_1$  is the critical relative indentation depth, CRDI. To understand if the evaluation of CRID is correctly estimated we have compared the results and the critical relative depth derived by the equation suggested by Bull [11]

$$\delta_B = 0.4 \left(\frac{H}{E}\right)^{1/2} \tag{5}$$

where  $\delta_B$  is the critical relative indentation depth (CRID B),  $H$  is the hardness and  $E$  is the elastic modulus.

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