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Comparison between nanoindentation and scratch test hardness (scratch hardness) values of copper thin films on oxidised silicon substrates

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Abstract

In this paper we measured for the first time the scratch hardness of copper thin films and compared this with hardness measured from nanoindentation. Copper films with thicknesses in the range 100 nm to 500 nm were deposited by rf magnetron sputtering on silicon substrates. Scratch hardness was determined by CSM™ scratch tester using scratch widths at low loads. The measured hardness values were compared with conventional nanoindentation hardness measurement by CSM™ nanohardness tester. At these low indentation depths there is a good correlation between the scratch hardness and the nanoindentation hardness, with an increase in hardness as the film thickness decreases.

Keywords: Scratch test; Nanoindentation; Copper; Hardness

1. Introduction

Nanoindentation has been established as an important tool for measuring mechanical properties of both bulk solids and thin films on the submicron scale [1–9]. An extensive study of nanoindentation on copper films has been done by Beegan et al. [10–12]. In nanoindentation, the depth of penetration together with the known geometry of the indenter provide an indirect measure of the area of contact at full load and thus hardness is obtained by dividing the maximum applied load by that contact area. On the other hand microhardness testing i.e. Vickers microhardness, is dependent on visual resolution of the residual indent for accurate measurement. In the nanoindentation technique hardness and Young's modulus can be determined by the Oliver and Pharr method [4], where hardness (H) can be defined as, $H = \frac{P_{\max}}{A_c}$, $P_{\max}$ is maximum applied load, and A_c is contact area at maximum load. Fig. 1 shows the typical load–displacement curve, showing the values used in the Oliver and Pharr method and cross-section of an indent during indentation. In nanoindentation, the reduced modulus, E_r, is given by:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}} = \frac{\sqrt{\pi}}{2}\frac{1}{C}\frac{1}{\sqrt{A_c}} \qquad (1-1)$$

Thus, the Young's Modulus, E, can be obtained from:

$$\frac{1}{E_r} = \frac{1-v^2}{E} + \frac{1-v_i^2}{E_i} \qquad (1-2)$$

Where v_i = aggregate Poisson ratio of the diamond indenter is 0.07 [7] and E_i = Young's modulus of the diamond indenter.

The scratch test is a simple and widely used method for investigating adhesion in thin films [13–15]. In the scratch test a Rockwell C diamond indenter is lowered on to the sample at increasing loads as the indenter tip is moved across the surface of the sample. Hardness i.e. scratch hardness can also be measured from the scratch track made by the scratch tester. We defined scratch hardness as the load per unit load-bearing area during scratching, taking into account the formation of ridges and a prow. As it is generally difficult to measure the load-bearing area during the scratching experiment, it is calculated from the width

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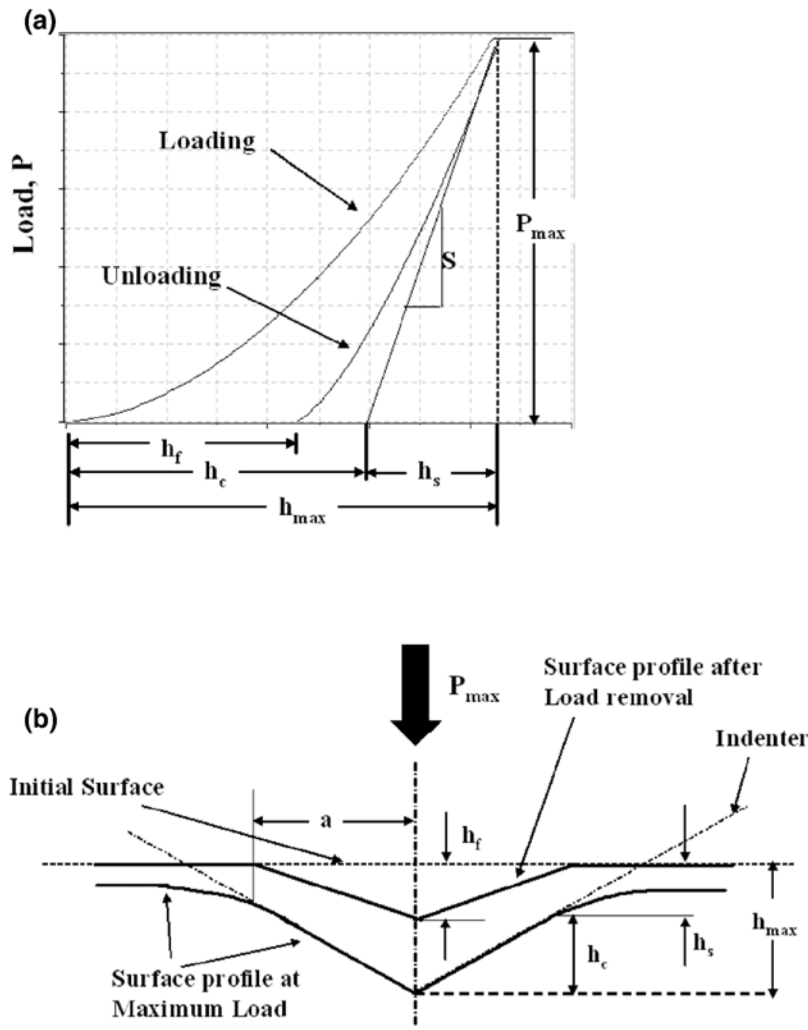

Fig. 1. (a) Load–displacement curve, showing the values used in the Oliver and Pharr method and (b) cross-section of an indentation.

of the scratch obtained after the test is completed. For indenter tips of circular cross-sectional area (cones, spheres, and parabolas) the equation for scratch hardness is defined as [16]

$$H_S = \frac{8F_N}{\pi b^2} \qquad (1-3)$$

where F_N is the applied normal load and b is the scratch track width. An advantage of scratch hardness relative to indentation hardness measurements is the possibility of studying hardness variations along the scratch. The hardness of different phases can be determined by making one single scratch. The absolute values at specific positions can be calculated by measuring the groove width and by applying the appropriate hardness formula.

2. Experimental details

Copper films were deposited in a Leybold Lab 500 rf magnetron sputtering system (rf 13.56 MHz). The chamber base pressure was below 1.0×10^{-3} Pa and the process pressure was 0.4 Pa. A 99.9999% pure Cu target was used for film deposition with an applied rf power of 40 W. The Cu deposition rate was 9.6 nm/min and film thicknesses were varied by varying deposition time. In this paper we look at Cu films of thicknesses 100 nm to 500 nm. An in-situ quartz crystal oscillator was used to monitor the film thickness. Film thickness was measured using an interferometry technique using the Zygo white light interferometer.

Nanoindentation testing on copper samples was carried out using a CSM™ nanohardness tester (NHT). The nanohardness

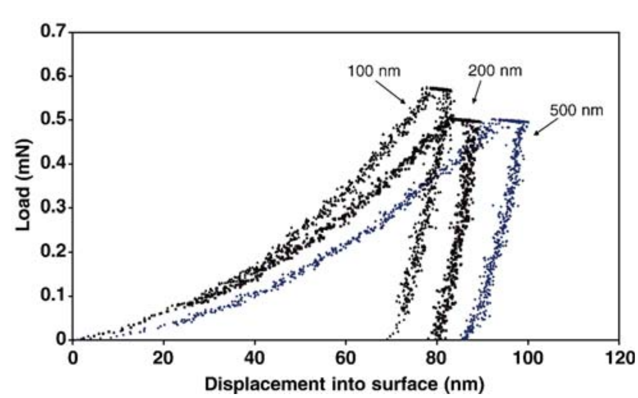

Fig. 2. Load–displacement curves for copper films on SiO_2/Si substrates at 0.5 mN load.