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 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, scratches hardness, copper

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 [1-3]. An extensive study of nanoindentation on copper films has been done by Beegan et al. [4, 5]. Hardness i.e. scratch hardness can also be measured from the scratch track made by the scratch tester. The scratch test is a simple and widely used method for investigating adhesion in thin films [6]. 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 of the scratch obtained after the test is completed. For indenter tips of circular cross-sectional area scratch hardness [7] is defined as

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

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. [7]

2 EXPERIMENTAL DETAILS

Copper films were deposited in a Leybold Lab 500 rf magnetron sputtering system (rf 13.56 MHz) with a pure Cu target. Nanoindentation testing on copper samples was carried out using a CSM™ nano hardness tester (NHT). mechanical properties were calculated using the Oliver and Pharr method. Scratch testing was carried out on all samples using the CSM™ microscratch tester (MST). The MST was used as a progressive loading device with a Rockwell C diamond indenter of radius of 200 µm. The load can be increased from 0 to 30 N at a specified loading rate. Load can be measured at any place as we know the loading rate and the length of the track. Some scratches were then imaged with the Zygo white light interferometer to obtain more information on the width and depth of the scratch tracks.

3 RESUTS AND DISCUSSION

In order to fully investigate the indentation behavior of copper films an investigation of the films was carried out by nanoindentation. First the load-displacement curves were analyzed and hardness was obtained via the Oliver and Pharr method. It is seen that the hardness at P_{max} decreases as the film thickness decreases. This is due to the decreasing influence of the substrate on the measured hardness. For the films with thickness in the range 100 to 500 nm we see a continuous decrease in hardness as the load decreases, reaching a minimum value of ~2.2 GPa for the 500 nm film. The hardness of copper films was also measured by combining the nanoindentation and AFM residual area analysis mentioned by Chowdhury et al. [2] and Beegan et al. [4] and thus it was possible to determine the total projected contact area and more accurate value for the film hardness. It was found that at low load, hardness measured by conventional unloading curve analysis by Oliver and Pharr is similar to the hardness found from residual indent area analysis by AFM as there is no pile up and sink-in effect at lower load indents [4].

At the beginning of scratch test with ramped load the load and depth of penetration are very low along the scratch track. Scratch hardness is measured at that low load and depth measuring the load and the width of the track by profilometer. Load can be measured at any place as we know the loading rate and length of the track. Fig. 1 (a-d) shows the interferometry images of the scratch tracks and depth profiles. From the interferometry images it is possible to obtain valuable information such as the scratch track width and depth as well as the height of the edge ridges. The measurements of the scratch track widths from the interferometry are used to calculate the scratch hardness of a number of films. Images of a 100 nm copper film at low normal loads is shown in Fig. 1 (a-d). Scratch track width is measured at a load of 0.25 N and the track depth is found to be 16.2 nm.

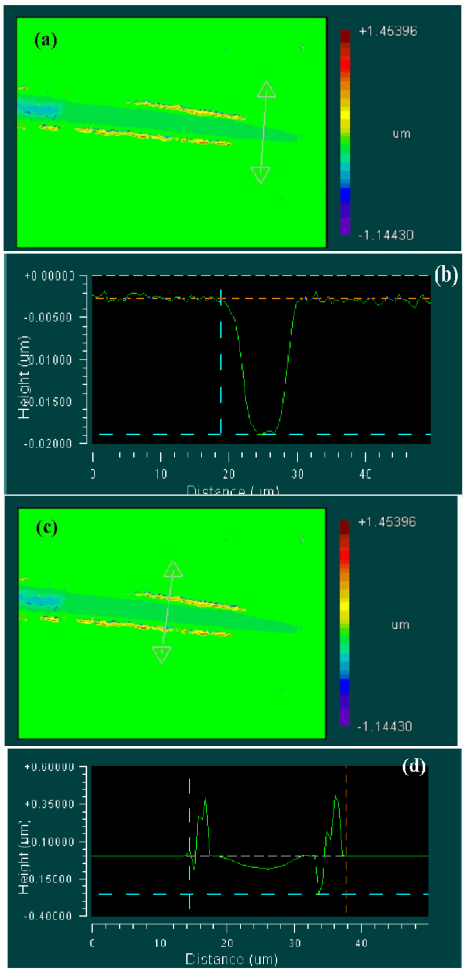

Figure 1: (a-d) Interferometry image of a scratch in a 100 nm copper film with two measurements of scratch track width

The calculated scratch hardness is compared with the nanoindentation hardness values for the same films. The results are shown in the plotted in Fig. 2. Hardness values (H_{NHT}) are given for these films at the lowest load of 0.5 mN to minimise the effect of the harder silicon substrate. The scratch hardness (H_S) values are calculated at low loads where the substrate would have the least effect on the measured values. 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. As the loads increase the scratch hardness increases.

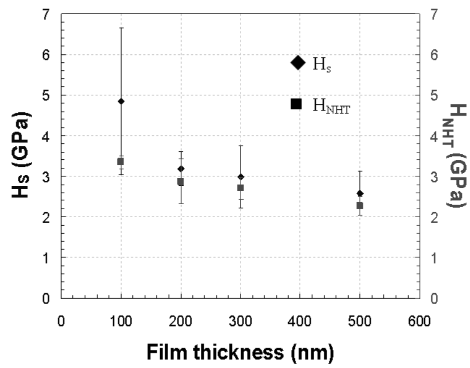

Figure 2: Comparison between the scratch hardness (H_S) and the indentation hardness (H_{NHT}) for as-deposited copper films.

4 CONCLUSION

We have measured hardness i.e. scratch hardness from the scratch tester usually used for investigating adhesion in thin films and compare this with hardness measured by the nanoindentation technique on copper thin films. We defined scratch hardness as the load per unit load-bearing area during scratching, calculated from applied load and the width of the scratch obtained after the test is completed. Copper films with different thickness range (100 nm to 500 nm) have been deposited by rf magnetron sputtering on silicon substrate. Scratch hardness was determined by CSM TM scratch tester using scratch widths at low loads. The measured hardness values were compared with conventional nanoindentation hardness measurements using a 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.

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