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SCRATCH-PROFILES STUDY IN THIN FILMS USING SEM AND EDS†

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The adhesion of evaporated or sputtered thin films to substrates is one of the most important characterising parameters in their fabrication. It is a conventional method to scratch the films using a stylus and evaluate the shearing stress, which is proportional to the energy of adhesion. For the evaluation it is necessary to determine the so-called critical load and the profile of the scratch.

The aim during this experimental work was to find a method to evaluate the scratch profile from the X-ray-line profile and SEM pictures. From SEM pictures, the lateral dimensions and surface morphology of the scratches were studied. The thickness was also studied from X-ray-line profiles.

In this paper the thickness profile measuring method and the conclusion for the scratch method are discussed.

1. INTRODUCTION

The adhesion of thin films to their substrates and to one another has been one of the most important parameters in all thin film technology. Mittal[1] has recently reviewed comprehensively the adhesion measurement techniques for thin films. The well known scratch method has been shown to yield and hold some promise as a quantitative test under certain conditions. The scratch test consist of drawing a smoothly rounded tip of known radius, R, across the film surface and a vertical load, W, applied to the point, is gradually increased until the film is removed from the substrate forming clear track. The amount of load on the tip at film failure which is known as the critical load is related to the shearing force caused by film failure by a simple formula derived by Benjamin and Weaver.[2] So, that adhesion could be calculated as a shearing force, F:

$$\text{where} \quad F = \frac{AP}{\sqrt{R^2 - A^2}}, \quad A = \sqrt{\frac{W}{\pi P}} \tag{1}$$

where A is the radius of the circle of contact, P is the indentation hardness of the substrate material.

In the present work, the scratch test coupled with SEM and EDS has been used to study and discuss the scratch profiles were also studied and used to evaluate the adhesion strengths of thin films.

2. EXPERIMENTAL PROCEDURE

The scratch apparatus used in this work is provided with a microprocessor controlled

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(x–y) stage[3] on which the test specimen was attached. The substrate bearing the film under test was mounted under the stylus on the stage which could be moved horizontally in two directions using programmed tape. The motorized x-movement drives the stylus from right to left to make the scratch. Following the movement the stylus is unloaded by raising it off the substrate. The scratch is then inspected by transmitted light using an optical microscope to detect film failure. The stylus is brought down into contact with the specimen with a known force so that its vertical position is adjusted to scratch the specimen.

The load was then incremently increased, and the test was repeated on an adjacent area. A series of scratches has been made at successively higher loads until failure, an illuminated scratch path is observed in either the film or the substrate.

Vacuum deposition from directly heated and from electron-bombardment heated sources was used for Cu and Mn. A steady deposition rate was obtained before opening a shutter to expose the substrate.

Variation of film thickness could be obtained subsequent to initial substrate exposure by partial reinterposition of the shutter. The substrates used were of Corning glass 7059 cleaned by chemical solvents and also by glow discharge cleaning before film deposition.

The measurement was performed with a JSM-35 JEOL scanning electron microscope equipped with a PGT-1000 energy dispersive spectrometer. The electron beam diameter was about 15 nm with a beam current of 300 pA. The electron beam was scanned across the scratch perpendicularly. The scanning time was 15 s and it was repeated 100 times in each measurement. The lateral resolution was measured on a chemical etched calibration pattern, its value was 0,4 μm on 300 nm Mn sample. The real lateral resolution was better,[4] since the etched pattern was not exactly perpendicular step profile.

3. RESULTS AND DISCUSSION

Figure 1 shows a plot of the calibration curves of the CuK_α and MnK_α X-ray emission

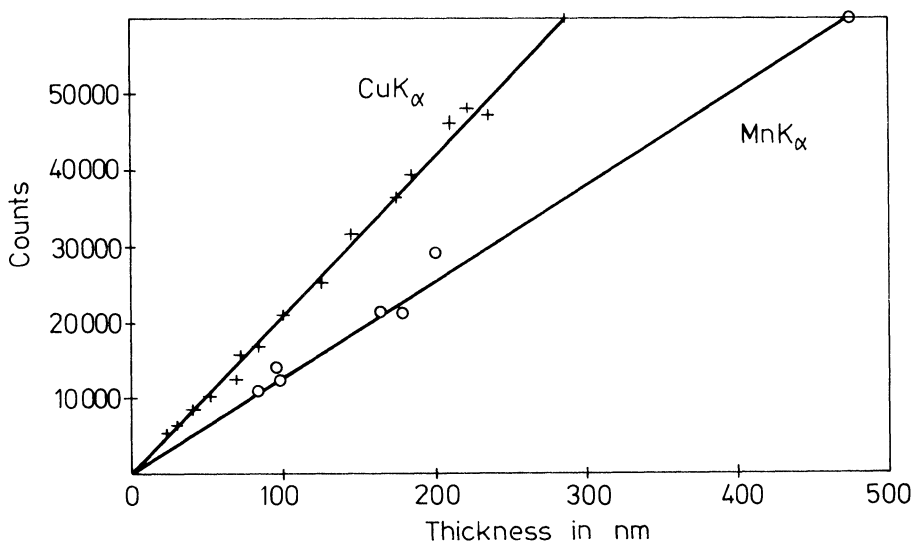

FIGURE 1 Calibration curves.

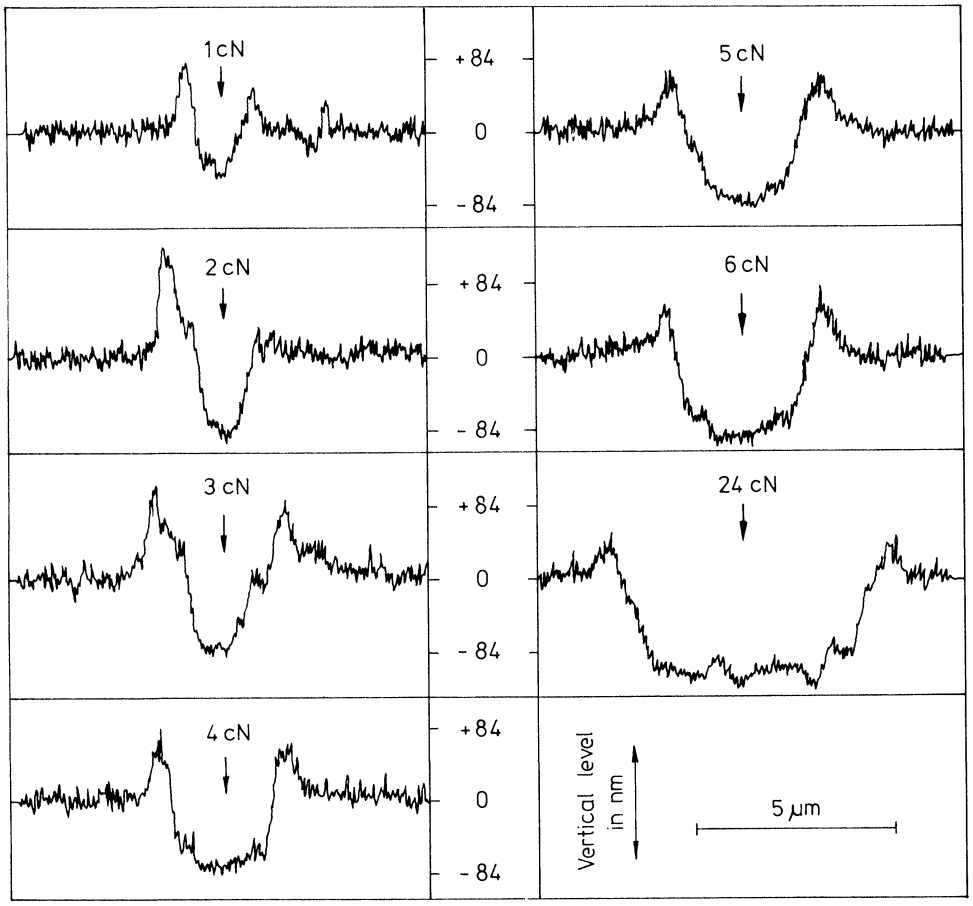

FIGURE 2 Scratch profiles in Cu (84 nm) layer.

intensities against the various thicknesses of both Cu and Mn layers. As can be seen, the intensities show a linear resolution to thickness at accelerating voltage of 20 kV.

Critical loads were first determined by optical and after that by scanning electron microscopy. The results were performed on selected sample of Cu layer of thickness 84 nm. A series of 24 scratch lines were made in the Cu film and each scratch was made with a different normal load within the range of 1–24 cN respectively. An electron beam of diameter 15 nm was used to excite the x-radiation, since the electron beam is scanned across the scratches perpendicularly. Using SEM and EDS, the profiles of the scratches could be estimated. Figure 2 shows the estimated profiles of the scratches number 1...6 and the last scratch number 24 at loads 1...6 and 24 cN respectively. From these profiles the depth can be recorded accurately. The measured depth are plotted against the applied loads shown in Figure 3. The results show that the depth was not increased above a definite value of the load say, 2 cN, since the layer completely removed from the substrate. The critical load as detected by EDS was found to be 2 cN while the critical load as detected by optical microscope was found to be 4 cN. So far, the EDS and SEM was found to be a more accurate technique for measuring the depth of the profiles.

Figure 3 shows also the variation between the width of the tracks against the applied load. It has been observed that the width of channels gradually increased with the

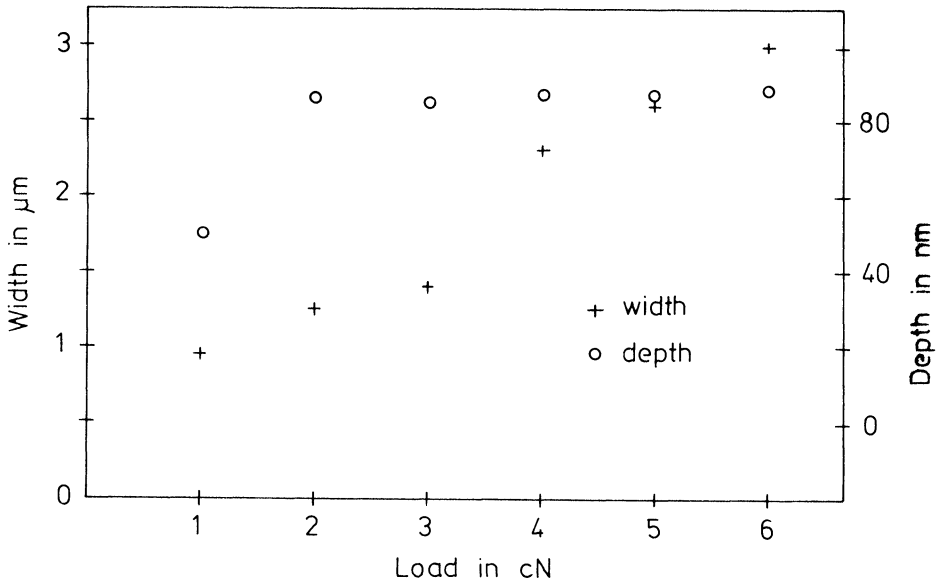

FIGURE 3 Changing of depth and width in scratched Cu (84 nm) layer.

applied load. At 4 cN there was a wide change in the width of the channels. The shear stress at the critical load observed both by SEM coupled with EDS and optical microscope was 78×10^7Pa and 56×10^7Pa respectively.

Figure 4 shows the detected profiles of Mn layer at 32 cN and 62 cN. As can be seen

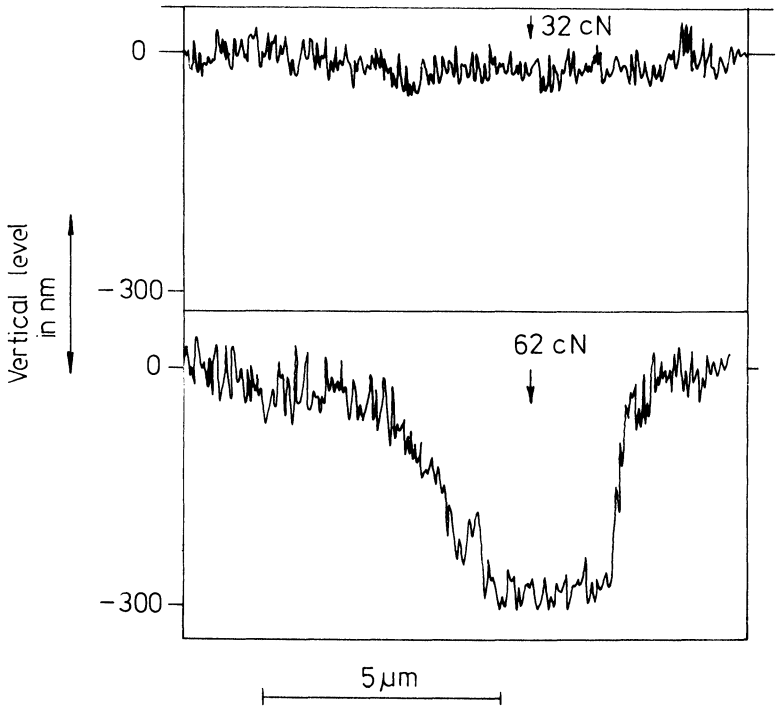

FIGURE 4 Scratch profiles in Mn (300 nm) layer.

that there was no dent at 32 cN, but at higher load say, 62 cN the layer was removed suddenly from the substrate. This was attributed to the brittleness of Mn layer.

CONCLUSIONS

1. The mechanism of film removal and the depth of the measured profiles depends on whether the film is soft or brittle.

2. For measuring the scratching profiles as detected by SEM and EDS, it seems to be that these techniques are more accurate and suitable for detecting the clear channels.

Knowing the real profile of the scratches, there will be a possibility of developing a model of the scratch process which incorporates the effect of plastic and elastic deformation of both the layer, and substrate as well as the function.

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