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EVALUATION OF COMPOSITE HARDNESS AND WEAR MECHANISM OF COPPER THIN FILM COATING

Sumaiya Islam, Raafat N. Ibrahim
Department of Mechanical & Aerospace Engineering, Monash University, Australia

ABSTRACT
To measure the mechanical characteristics and the wear mechanism of the coating material, nano indentation and the nano scratching method were employed respectively with diamond Berkovich tip having tip radius of 100 nm. The results showed that the hardness decreased with the increase of the indentation depth. Therefore experimental data was interpreted with the Korsunsky model. Moreover the plie up along the indentation mark reveals that the ploughing mechanism is responsible for the material deformation in this range of indentation depth. After single scratching operation, the percentage of elastic recovery for this film/substrate combination decreased and the pile up volume increased with the increase of the depth of cut. The propagation of the pile up volume along the scratch length was clearly observed and there was no sign of crack propagation. Therefore for this film/substrate system ploughing wear mechanism is the dominant mechanism for the nano level deformation.

Key Words: Nano indentation, Nano scratch, Composite hardness

1. INTRODUCTION

Thin film coating is a promising technology and it has lot of applications in the field of semiconductor and biomedical industries. In order to increase the lifetime and reliability of thin film coatings, it is necessary to measure their mechanical properties and to characterize their wear mechanisms. In this paper nano indentation and nano scratching were chosen to investigate the composite hardness and the wear mechanism of the thin film coating.

Composite hardness describes the phenomenon where the measured hardness changes as depth of indentation increases, due to the combined responses of both the substrate and the film. The most commonly used model for composite hardness, the Bückle rule, which states that if the indentation depth is less than 10% of the film thickness, the hardness measured will be that of the film alone[1]. However, it is believed that the rule cannot accurately represent both hard film/soft substrate systems and soft film/hard substrate systems. Therefore there are different models available [2-7] to accurately represent the hardness data. However, for a specific thin coated system no specific model can be recommended. Generally a simple law of mixtures was used to describe the proportional contributions of both the film and substrate hardness, in relation to the ratio of film thickness to indentation depth. Beegan et. al [8] measured the hardness of copper films on oxidized silicon substrates (soft film / hard substrate), for different film thickness and indentation depths. Three distinct regions was identified with the increment of the indentation depth and shown in Fig 1. For a shallow penetration depths (region I), the measurement was that of the film only (H_f). As the depth increases (region II) the composite hardness was measured (H_c). Then, at a large depth in region III the response was purely that of the substrate surface (H_s).

To enhance the tribological properties of the surface, such as reducing the wear hard coating on a soft substrate

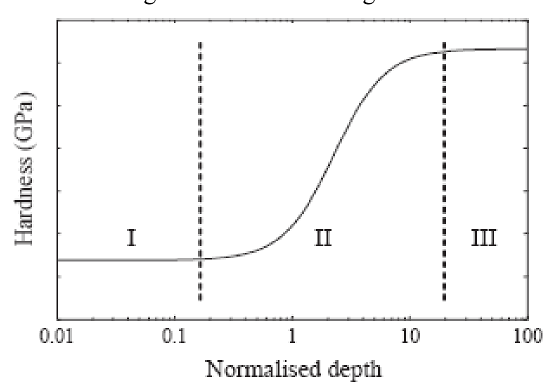

Fig 1. Composite Hardness for a soft film/hard substrate system [8]

was used [9]. The wear due to the contacting of the two metal surfaces was classified as abrasive wear. Recently it is speculated that ploughing mechanism is one of the dominant mode of abrasive wear [10]. However, the physics of such wear mechanism in the nanometer range still remains unclear because of the lack of experimental data. Therefore to establish the wear mechanism elastoplastic deformation behaviour can be considered as an indicator. Moreover elasto-plastic behaviour of the material have been reported by the variation of the normal force using Atomic Force Microscope (AFM) [11-14]. AFM is a well recognised tool to measure the mechanical characteristics by the micro/nano scratching method[15, 16] The conventions of the AFM based lithographic technique is that the machined surface scale of the structure is solely determined by the geometry of the AFM probe[17-19].Therefore the nano indenter produced by Hysitron utilises the same mechanism to create a scratch; however, a diamond probe is placed with the tool holder instead of the cantilever attachment used in AFM. The advantage of using nano indenter is to avoid the effect of the cantilever's geometry, stiffness and their respective calibration reliability on the measured nano machining parameters[20-22]. Moreover, AFM cannot directly measure the lateral force during scratching.

Therefore the objective of this study was to investigate the composite hardness and the wear mechanism studying elastoplastic deformation behaviour in hard film/soft substrate system utilizing indentation and scratching method for a nano meter range of dimensions. The effects of both the normal and the lateral cutting forces due to nano range variations in the depth of cut are reported in this study. The post scratch profiles were used to calculate the elastic deformation and pile up volume to identify the wear mechanism of the coated system.

2. EXPERIMENTAL CONDITION

Nickel was used as the substrate material in this study. The substrate materials were cut by the electro discharge machine (EDM) for making a completely flat surface. Before the deposition of the coating material, the substrate surfaces were polished with 1 μm polishing pad to obtain a scratch free parallel surface[23].The substrate specimens then underwent ultrasonic cleaning progressively in acetone and ethanol, and were subsequently dried in a vacuum dryer. After cleaning the substrate, Ni was electroplated at a current density of 3.5 Amps/dm² and at a deposition rate of 1.8 μm/minute to form the Cu coatings. In the electroplating process, an electronic grade Cu metal sheet was used as the anode, while the substrate (Ni) acted as the cathode. After coating, the samples were not subjected to any further treatment to avoid additional effects resulting from surface preparation techniques (such as mechanical hardening due to polishing). For the measurement of composite hardness and elastoplastic behaviour to identify the wear mechanism nano indenter was used and the experimental conditions are presented in Table 1.

The topographic scanning method was employed to analyse the elastoplastic deformation behaviour. The area of the topographic measurement was 10x10 μm^2.In scanning mode, 20 μm/s tip velocity and 1 Hz scan rate were used. Before the scratching operation, the roughness of the surface was measured with the scanning mode. The average surface roughness for the investigated samples was found to be approximately 2 nm.

3. RESULT & DISCUSSION

3.1 Composite Hardness Evolution

In fig 2 the results for copper samples clearly showed that the hardness value decreased with the increase of indentation depth. The hardness was determined from the load displacement curve, using Oliver and Pharr method[24]. The variation of the hardness indicated that the composite hardness was being measured and therefore the response of the material to indentation changed with the increasing depth. As the indentation depth was equal to the film thickness, therefore the measured hardness could be the hardness of the substrate. The horizontal line at the bottom end represented the hardness of the substrate. It was observed in sample1 (thickness 300nm), the substrate hardness at 300 nm displacement of the indenter whereas sample2 (thickness 400nm), the substrate hardness at 400 nm. The individual substrate and the film hardness were 2.85 GPa and 15.95 GPa respectively

Table 1: Experimental Condition

Machine used	Triboindenter (Hysitron)
Cutting tip	Berkovich tip (100nm)
Room	20-24° C
Relative Humidity	50%
Nano indentation	
Sample 1	Coating Thickness 300 nm
Sample 2	Coating Thickness 400 nm
Displacement	50,75,100,150,200,300,400 nm
Nano scratching	
Material	Copper Coating
Feed rate	0.33 μm /sec
Depth of cut	50,100,200,300 nm
Scratch length	10 μm

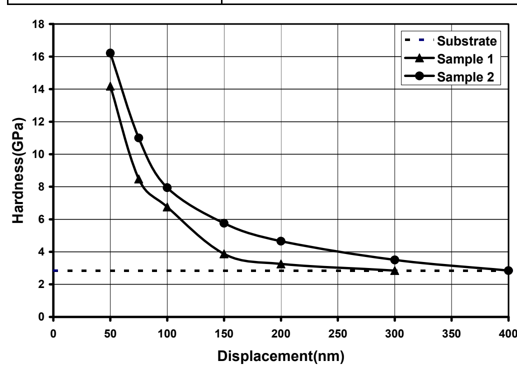

Fig 2. Variation of the Hardness with the Displacement

Different hardness models were compared to the collected data. The model which correlated best with the experimental data were found to be a Korsunsky model [6]. The equation used for the Korsunsky model is given below.

$$H_c = H_s + \frac{H_f - H_s}{1+\kappa\beta^2}$$

(1)

$$\beta = \frac{\delta}{t}$$

where β, δ and k represent the normalised depth, indentation depth and constant related to the film thickness.

Fig 3 showed that the Korsunky model [6] fits the experimental data well. The constant k was found to be 17.93 using equation 1. The trend of the data correlates with the trends of the models shown in [8] and [25]. There is variation in the fitted model at the smaller depths. This could be attributed to the uncertainty in hardness measurements at small depths, due to the very thin film used and large surface roughness. However, it can be seen that the Korsunsky model provides a sensible model for composite hardness of this thin film coatings.

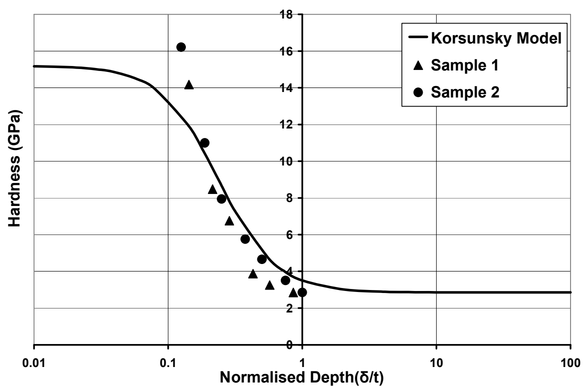

Fig 3. Fit of Korsunsky model to data

Fig 4 shows the surface topography and the indentation marks produced on a sample at different depths. The residual impression resembles the shape of the three sided pyramidal diamond tip. The slightly raised material at the indentation edges, in conjunction with the absence of cracks, was characteristics of the plastic flow behaviour of the ductile material. The smooth side walls of the residual impression and the absence of any surface or subsurface crack indicate that plastic flow was the principle deformation process in the nano indentation process.

3.2 Wear Mechanism Evaluation

To investigate the wear mechanism of the thin coated system nano scratching method was employed. The nano level elastoplastic deformation characteristics in the scratching operation was utilized for this purpose. The elastoplastic deformation of the material was determined by the percentage of elastic recovery and the pile up volume. The elastic recovery (%) was calculated using the equation (2).

$$\text{Elastic Recovery (\%)} = \frac{h_1 - h_2}{h_1} \times 100 \qquad (2)$$

where h_1 is the depth of cut and h_2 is the plastic depth that was measured from the post scratching profile.

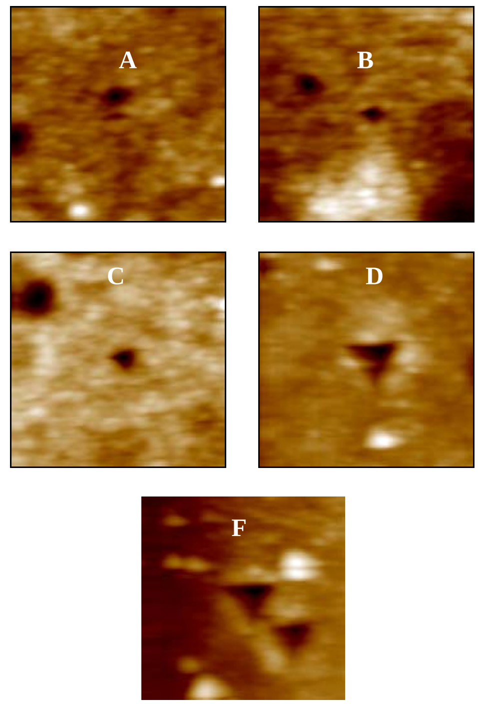

Fig 4. Indentation at different depth in sample 1 (A) 50 nm (B) 100 nm (C) 150nm (D) 200nm and (E) 300nm.

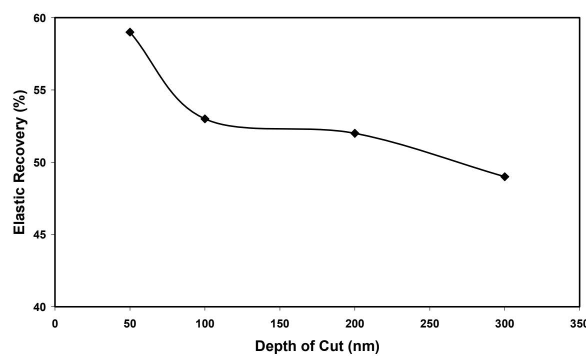

Fig 5. Variation of the elastic recovery (%) with the depth of cut.

Fig 5 presents the effect of the elastic recovery (%) with the variation of depth of cut. It was shown that percentage of elastic recovery decreased with the increase in the depth of cut. When the depth of cut increased the generated normal and lateral forces also increased as presented in fig 6. As a result, due to the increment of both forces elastic recovery was decreased and the plastic deformation of the material was increased. The upward dislocation of the material due to plastic deformation is termed as pile up [26-28]. This pile up is the indicator to identify the ploughing abrasive wear mechanism.

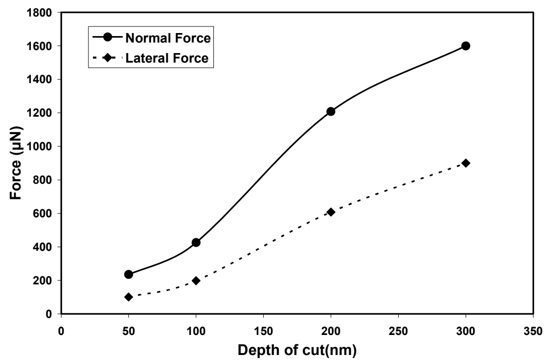

Fig 6. Variation of the normal and Lateral force with the depth of cut.

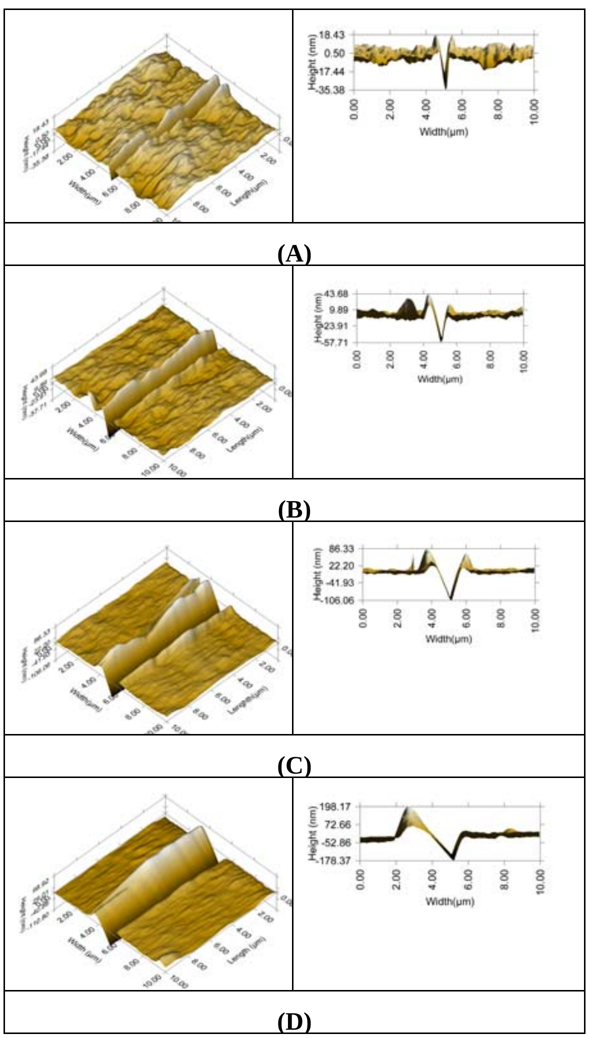

Fig 7. Scratches were done by using different depths of cut. (A) 50nm (B) 100 nm (C) 200nm and (D) 300 nm.

Fig 7 presented post scratch surface topography at different depths of cut. The pile up volume was generated at one side of the scratch length during this nano scratching operation .Generally the pile up volume was generated at the edge of the cutting tool. Cutting tool (Berkovich tip) used in this experiment has an asymmetric shape [29]. The cutting edge faces at one side of the scratch length producing hydrostatic pressure that enables the plastic deformation.

Fig 8 shows the changes in the pile up volume with the depth of cut. The pile up volume increased with the increase of the depth of cut. This establishes that a higher plastic deformation leads to an increase in the pile up volume, which also implies a greater amount of plastic energy dissipation from the machining operation. Therefore, pile up volume was indicated that ploughing wear mechanism was the dominant factor for making this groove. Moreover, no crack was observed in this range of machining condition. Therefore it reveals that the wear has occurred due to the ploughing mechanism rather than the crack in this soft film hard substrate system.

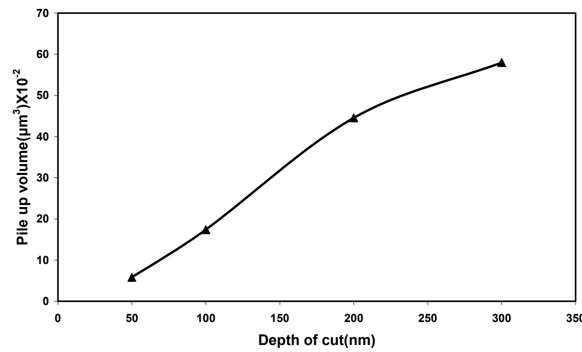

Fig 8. Variation of the pile up volume with the depth of cut.

4. CONCLUSION

The hardness and wear characteristics of copper film/nickel substrate systems were experimentally investigated utilizing nano indentation and nano scratching technique. In view of the presented results and discussions, the following conclusion can be drawn.

- A composite hardness trend was observed for each of the samples. The hardness decreased with the increasing of the indentation depth. These hardness values provided the composite value for the hard film soft substrate system. The Korsunsky model was recommended to be a good fit for the data. Pile up volume was observed along the indentation mark rather than the crack.

- However, the hardness value changed with the increment of the indentation depth, therefore the wear mechanism remained unchanged. Hence the ploughing is the dominant mode for the wear mechanism during this range of indentation depth.

- The percentage of the elastic recovery of the coated system decreased with the increase of the depth of cut due to the increase of the generated normal and cutting forces during scratching. Therefore the pile up volume increased with the increase of the depth of cut. Pile up along the scratch length reveals the ploughing mechanism. Therefore wear occurred due to the ploughing mechanism of the coated system in this range of depth of cut. Further more no crack or subsurface crack was observed along the scratch length.

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6. NOMENCLATURE

Symbol	*Meaning*	*Unit*
δ	Indentation depth	nm
T	Film thickness	nm
H_f	Film hardness	GPa
H_s	Substrate hardness	GPa
H_c	Composite hardness	GPa
h_1	Depth of cut	nm
h_2	Plastic Depth	nm
K	Constant	
β	Normalised depth	

7. MAILING ADDRESS

Raafat N. Ibrahim
Department of Mechanical & Aerospace Engineering,
Monash University
Clayton, Victoria 3800, Australia.
Corresponding author. Tel.: +61-3-99051982;
Email: raafat.ibrahim@eng.monash.edu.au