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ADHESION BEHAVIOUR OF Si LAYERS

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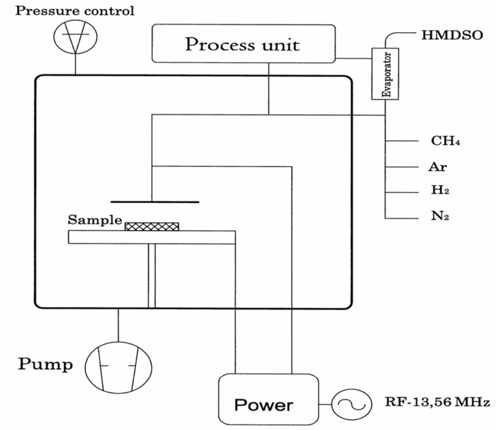
ABSTRACT: The aim of this investigation was to determine the adhesion strength and failure of Si layers. The layers were prepared using technology RF PACVD onto steel substrates. Si layers were prepared from HMDSO, its vapors were diluted under various carrying gases. The adhesion of layer systems was evaluated by scratch test, including acoustic emission signals and by static indentation (both Rockwell and Vickers indenter). The results of adhesion are documented by records of morphologies of failures. Results demonstrated that the adhesion behavior of Si layer is improved by introducing carbon.
KEYWORDS: RF PACVD, Adhesion, AE signal, scratch test

❖ INTRODUCTION

For engineering applications, diamonds like carbon films are suitable coatings, when high wear resistance and low friction is needed. One problem is their poor adhesion on steel substrates without intermediate layers [1]. This problem can be solved by using intermediate layers, specifically by Si layers. Si layers are very appropriate intermediate layers for DLC coating. Even, without Si intermediate layer DLC coating spontaneously spalling from substrate. Plasma-assisted chemical vapour deposition (PA-CVD) is a modern technique for depositing various hard coatings on different materials and geometrically complicated substrates. An electric glow discharge is used in this process to increase the activation of species which form the layer [2]. In scratch testing, a diamond stylus of defined geometry is drawn across the surface of a coated sample at a constant speed with a defined normal force over a defined distance. The normal force can be constant, progressively increase, or incrementally increase. The diamond stylus typically has Rockwell C geometry with an angle of 120 degrees and a spherical tip radius of 200 µm. Different tip radii can serve to change the contact pressure. The tangential force, the penetration depth, and the acoustic emission signals are recorded as secondary test data, along with the normal force. After completion of the test, the scratch track is microscopically analyzed for specific, well defined damage such as cracking, deformation, buckling, spallation, or delamination of the coating [3]. The damage to the coating may have different forms: cohesive, adhesive and conformal breaks, delamination of a coating from the substrate, chipping, flaking etc. A big number of possible coating damages in a scratch test make it difficult to interpret the results with certainty. Critical force is often associated with adhesion whenever the coating is damaged. Its chipped or flaked elements are removed by the moving indenter outside the scratch, revealing the substrate. However, they are also sometimes pressed into the coating. When the fragments of the coating removed on top of the track do not reveal the substrate, the critical force is not related to adhesion, but to the mechanical properties of coating. The factors affecting the value of critical load in the scratch test include: adhesion and cohesion of coating-substrate system, hardness and roughness of the substrate, hardness and roughness of the coating, thickness of the coating, friction coefficient between the substrate and the indenter as well as the coating stress and that on the edge between the coating and the substrate [4]. The aim of this study was to specify the adhesion behavior of thin Si films deposited by RF PACVD technique. It contains information concerning the deposition technology of Si films and results of adhesion and tribological behavior of Si layers. In this report are documented various methods of evaluation of adhesion behavior.

❖ EXPERIMENTAL

Si layers were prepared from Hexamethyldisiloxane – HMDSO, which was diluted and supported by various gases: Ar, H_2, N_2 in a RF PACVD (13, 56 MHz) chamber, which in figure 1 is shown. Before being loaded in the chamber the polished steels substrates AISI L2 (R_a = 0,002) were chemically cleaned with isopropyl alcohol in ultrasonic bath for 15 min.



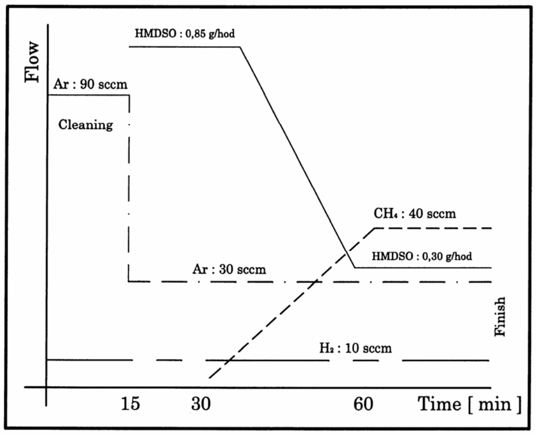


Figure 1: RF PACVD (13, 56 MHz) technique

Figure 2: Technology process of Gradient layer

Then the samples were dried very carefully under nitrogen's gas. Then they were loaded into the reaction chamber and put on working electrode where 13,56 MHz power was delivered through deposition. Because Ar sputtering of the substrate surface is effective in removing impurities and it improves its adhesion too, all the substrates were plasma cleaned in argon by RF PACVD plasma immersion at the bias voltage of 60 V for 15 minutes. The silicon layers were obtained through the decomposition from Hexamethyldisiloxane – HMDSO (HMDSO was vaporized in a tank at 102 °C) diluted with different gas, Ar, N_2 H_2. A flow of gases and others parameters is shown in Table 1. As well a thin film Gradient was prepared, whose technology process is exactly shown in figure 2. The gradient film is achieved by continuous feed of discharge gases.

Table 1: Parameters of preparing Si layers

HMDSO mixed by:	HMDSO [g/hod]	Ar [sccm]	N_2 [sccm]	H_2 [sccm]	Time [min]	Power [W]
Ar	0,85	100	-	-	90	250 W
N_2	0,85	75	30	-	90	250 W
H_2	0,85	75	-	45	90	250 W

The process gases Ar, N_2 H_2 as well as HMDSO were controlled by mass flow controls and the pressure was controlled by a butterfly valve independently of the total gas flow. The working pressure was 20 Pa and during the deposition processes the total pressure was held constant. The applied RF power input was kept at 250 W and bias voltage was 60 V.

We have investigated roughness, thickness, both static and dynamic adhesion and coefficient of friction prepared Si layers. The thickness of coatings for all the samples was evaluated by Calotest method. The adhesion was investigated by scratch test and statics indention. The scratch test and one of static indentation measurements were carried out using a Rockwell diamond indenter with radius of 0,5 mm. Another static indention was carried out by Vickers diamond indenter. The adhesion was carried out by scratch tester CSEM REVETEST in the mode of changing normal force, which was linearly increased at maximum value 80 N. During the test the holder with a specimen travelled at a constant speed beneath the indenter. The indenter was Rockwell stylus having spherical tip radius of 500 μm, which it was drawn along the sample. While, the normal force is linearly increased to maximum value 80 N, during scratch test the acoustic emission signal in dependence on normal force was registered and also tangential force for calculation of coefficient of friction. They were documented morphologies of failure after running of scratch test by light microscopy. Analogously they were documented morphologies of indents after static indentation tests [5]. The morphology of all measurements was accentuated by means of polarized light and Normansky differential interference contrast.

❖ RESULTS

The value of thickness of the coatings was calculated to be approximately 1,2 μm. The roughness of the coatings, about 20 nm, was approximately the same as the roughness of the substrate. Morphology of the scratches for the investigated Si layers is illustrated in Figure 3. In the figure line by line are demonstrated scratch tracks for :Si - H, Si - N_2, Si - Ar and gradient layer. Together with the scratch test, the acoustic emission signal in dependence on normal force from adhesion investigation is presented (Figure 4). According to the registration of acoustic emission measurement it is possible to determined that the failure occurred at critical load (*Lc*) ~ 42 N for sample Si-Ar, at critical load (*Lc*) ~ 53 N for layer Si-H and at critical load (*Lc*) ~ 70 N for gradient layer.

Figure 5 shows morphology of indents in dependence on normal force from adhesion investigation by the static indentation. This static adhesion was evaluated by Rockwell indenter with radius of tip 500 μm. The columns correspond by line to layer: Si-Ar, Si-H, Si-N_2 and gradient layer. The lines correspond to the values of normal force 50 N, 100 N, 150 N and 200 N.

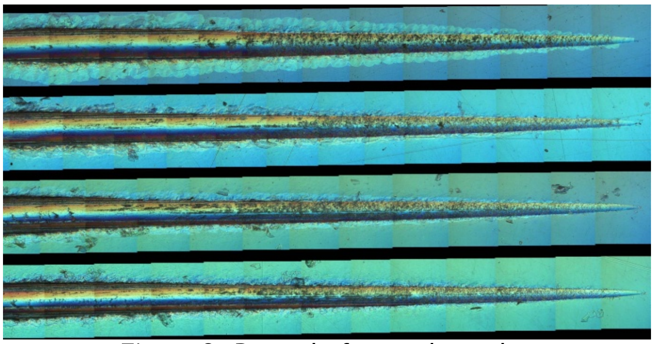


Figure 3: Record of scratch tracks.

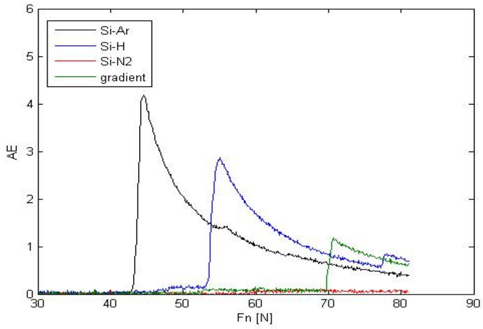


Figure 4: Record of AE signal.

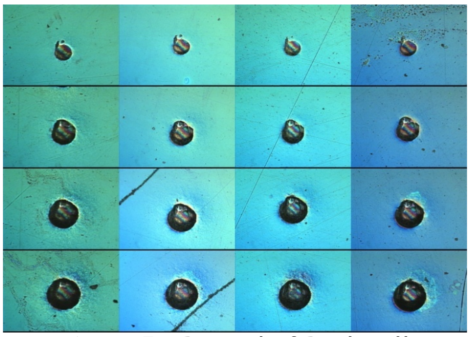


Figure 5: Record of Rockwell indentation

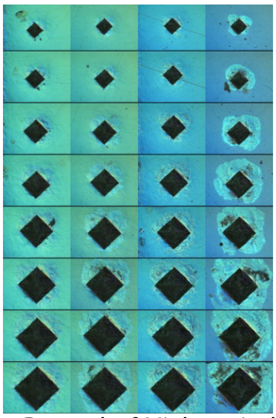


Figure 6: Record of Vickers indentation

In figure 6 the morphology of indents in dependence on normal force from adhesion investigation by the static indentation by means of Vickers indenter is presented. The columns correspond to layer gradient, Si-Ar, Si-H and Si-N_2. The lines correspond to the values of normal force 30N, 40N, 60 N, 80 N, 110 N, 140 N, 170 N a 200 N.

A Record of relationship between coefficient of friction and normal force is shown in Figure 7. The curves were registered during scratch test and that is way this dependence is again for diamond indenters with radius 500 μm. The character of curves is similar for all coatings. These coefficients of friction are very low to load 50 N. However, there is a change from load 50 N. The lowest value has Si layer diluted by N_2.

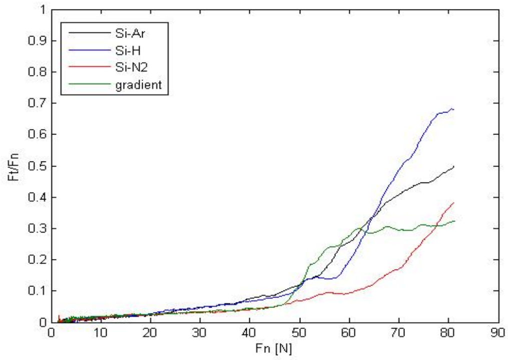


Figure 7: Record of the coefficient of friction

❖ CONCLUSIONS

In the paper, the relationship between adhesion heavier parameters of depositing process is demonstrated. Si layers were prepared from liquid HMDSO, which vapour was diluted by various carrying gas (Ar,H,N_2). Si gradient layer was made, too. All prepared coatings with thickness 1,2 μm had approximately the same morphology as the substrate. Various methods were tested to detect morphologies of failure of Si layers. The progressive load scratch test with a maximum load 80 N was applied to the samples. Records of scratch test were done after testing and the morphology of scratch test was monitored by signal of acoustic emission, too. Although both scratch track of scratch test looks similar, another detailed analyzes – AE shows more accuracy result. The highest resistance to adhesion failure was detected for Si gradient layer with critical force of failure *Lc* ~ 70 N. whereas others layers had a lower critical force: (*Lc*) ~ 42 N for Si layer diluted by argon and critical load (*Lc*) ~ 53 N for Si layer diluted by hydrogen. This is to certify that enriched Si layer by carbon makes better properties than Si layers without carbon. Another analyze was static indentation both Rockwell and by Vickers indenter. It is possible to see difference in failure and deformation from morphology for Si layers. Although from static indentation by Rockwell indenters are results for all samples similar, from indentation by Vickers indenter it is possible to see marked failure for layer Si-N_2. The record of the relationship between the coefficient of friction and the normal force shows character of curves similar for all coatings. The lowest value has Si layer which was diluted by N_2. So, it is important for future works to use more methods of evolution of the adhesion behavior of thin layers.

❖ ACKNOWLEDGMENT

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