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MECHANICAL PROPERTIES OF SILICON CARBON COATING

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ABSTRACT: *The aim and objective of this investigation was to investigate the mechanical properties of amorphous hydrogenated silicon carbide (a-SiC:H) layers. The layers were prepared using Plasma enhanced chemical vapor (PECVD) technology on polished tool steel. For formation layers, we used vapors of liquid precursor Hexamethyldisiloxane (HMDSO) diluted in argon and mixed with various flow of methane (18, 23 and 29 sccm) for comparison. We investigated influence of methane (CH_4) flow on mechanical properties of a-SiC:H coatings such as adhesion, coefficient of friction, nanohardness, thickness and roughness. The adhesive strength of layers was evaluated by scratch test (through Acoustic Emission) and by static indentation. Nanohardness was studied and compared using nanoindentation tester with Vickers indenter.*
KEYWORDS: *PECVD, a-SiC: H, Nanohardness, Adhesion, Coefficient of friction*

INTRODUCTION

The plasma enhanced chemical vapor deposition technique (PECVD) has been in use for over two decades. Today, this technology is widely established together with another commercial technique (most frequently PVD) for the deposition of a variety of hard thin films. The main advantage of the PECVD technique is the ability to deposit films at relatively low substrate temperatures (typically less than 500°C) and the aptitude to deposit a curvature surface, such as gear wheels, bio-implant applications, cavities of mechanical parts and other. The PECVD films are used not only for mechanical applications, but also in the microelectronics.

Amorphous silicon carbon layers (a-SiC) combine series of interesting properties, such as a low friction coefficient, high hardness and wear resistance. Due to these attributes they provide a substantial potential for new applications, such as machine parts. Except the possibility of mechanical application the a-SiC layers are used successfully in semiconductor industry. Therefore, in recent years a large number of investigation has been carried out on opto-electronic properties of semiconductor films as amorphous silicon (a-Si, a-Si:H) and amorphous hydrogenated silicon carbide (a-SiC, a-SiC:H) films. In practice, a-SiC:H films have been widely used as an emitter layer in solar cells [1-6].

Nanoindentation technique is modern and useful tool and it develops rapidly for over twenty years. This technique has an important role in the characterization of mechanical properties of materials on submicron scale. The ultra-low load indentation systems provide continuous records of load and of displacement as an indentor which is pushed into a surface. Load-displacement data (indentation curve) can be analyzed to give a number of useful parameters and information for complete assessment of elastic, plastic and fracture properties of surface at the scale of real asperity contacts [7].

However, the mechanical properties of a-SiC:H layers, such as adhesion strength, nanohardness and other properties have been studied relatively little in spite of the fact that essential mechanical properties are key factors for success application.

The purpose of the present work was to investigate the mechanical properties (adhesion strength, coefficient of friction, nanohardness, thickness and roughness) of carbon a-SiC:H layers and compare their results in connection with the various flow of methane (CH_4 flow: 18 sccm, 23 sccm and 29 sccm). The article contains the description of the deposition process of silicon carbide in connection with the change of methane flow too.

EXPERIMENTAL

The deposition of silicon carbon layers were performed in an experimental PECVD reactor. The deposition chamber is 500 mm in diameter and 700 mm in height. The scheme of the whole technique is reported early [8]. It is designed for the production protective coatings as well as for plasma nitriding. The vacuum chamber contains a heated table and the temperature thermocouples for observing and controlling of temperature.

Amorphous hydrogenated silicon carbide (a-SiC:H) layers were deposited on polished tool steel substrates. The substrates of size of 20 mm in diameter and 10 mm in thickness were polished prior to the deposition, to roughness of surface in the value of R_a = 0,02. Then the samples were cleaned in isopropylalkohol in ultrasonic bath for 15 minutes. After cleaning, the samples were loaded immediately into the plasma chemical reactor and put on working electrode. Before depositing layers, the surfaces of samples were precleaned in argon immersion (Ar flow: 45 sccm) at the bias voltage of 700 V for 15 minutes. To improve the adhesion of a-SiC:H layers, Si layers were formed about 40 nm in thickness , from liquid Hexamethyldisiloxane (HMDSO) whose vapors were diluted at argon flow of 42 sccm (HMDSO was vaporized in a tank at 103 °C) .

The a-SiC:H layers were prepared through the decomposition from HMDSO (1g/h) diluted argon (flow : 42 sccm) and mixed with methane under various flow (flow of CH_4 : 18 sccm, 23 sccm and 29 sccm). The working pressure was 20 Pa and during the deposition process the total pressure was held constant. The power input was kept at 200 W and bias voltage 800 V. The deposition rate was approximately 1 μm.h^{-1}. In the experimental series, the deposition parameters, except the flow of methane, were kept constant.

We have investigated roughness, thickness, both dynamic and static adhesion strength, coefficient of friction and nanohardness. The roughness was carried out by Hommel Tester T1000 and the measurement of coating thickness was performed by using kalotest method, which uses steel ball for grinding of spherical cap. The thickness of thin film is counted from grinding annulus [8].

The adhesion strength was investigated by the scratch test and by static indentation. Both methods have been using Rockwell diamond ball indenter.

The adhesion of a-SiC:H films was carried out using scratch tester CSEM Revetest in the mode of changing normal force, which was linearly increased to maximum value 80 N. During the scratch test, the acoustic emission signal in dependence on normal force was registered and also tangential force for calculation of coefficient of friction. The critical load (L_c), which was measured by an acoustic sensor during the scratch test, was defined as the load at which spalling or flaking of the films occurred [9].

For specification, the adhesion strength was evaluated by static indentation with Rockwell indenter (radius 0,5 mm and 0,2 mm) too.

Morphologies of adhesion tracks were recorded after running of the scratch test and also morphologies were documented of indents after static indentation test by light microscopy.

The nanohardness of the layers has been evaluated by nanoindentation test (nanoindenter Shimadzu DUH 202). The equipment uses a Vickers shaped diamond tip with load resolution of 0,1 mN. Microhardness was calculated from the load-displacement curves using an analysis programmer. The specimen was put on the horizontal holder with a microscope directly located above the selected area. The conditions of the measurement are following: at a room temperature (25°C) and at a relative humidity around 55 %. The displacement during loading gives the universal hardness H_U, which is expressed by the following equation:

$$H_U = \frac{F_{max}}{26,43 . h^2} , \qquad (1)$$

where F_{max} is the peak indentation load and h is the indenter penetration depth at F_{max}. The load – displacement experiments were repeated on 2 independent locations of the surface of each specimen.

Measurements were performed gradually under the maximum load of 200g to obtain the microhardness values of the substrate, which is influenced by thin film a-SiC:H. Further, measurement of nanohardness was performed under 25g load to obtain the microhardness occurring near the borderline of the thin film and under the load of 5g to obtain the microhardness values of thin film itself.

RESULTS

After 2 hours of depositing in plasma chemical reactor, the layers a-SiC:H achieved thickness approximately 5 μm for all the flow of methane (CH_4). The result roughness of coatings was the same as the substrate R_a = 0,02.

In Figure 1 the running of acoustic emission (AE) of adhesion test is recorded. We can see that the film with flow of methane 23 sccm has the critical load (L_c) approximately 10 N. The coating which was prepared under 29 sccm has the critical load (L_c) about 12 N. On the other hand, coating with lowest methane flow of 18 sccm has achieved the critical load L_c about 21 N.

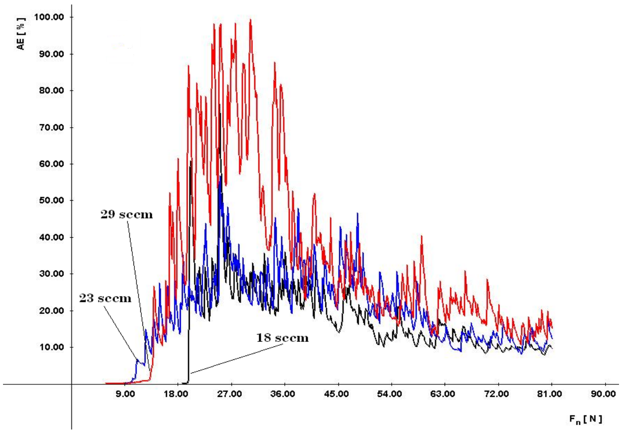

Figure 1: Record of running of acoustic emission

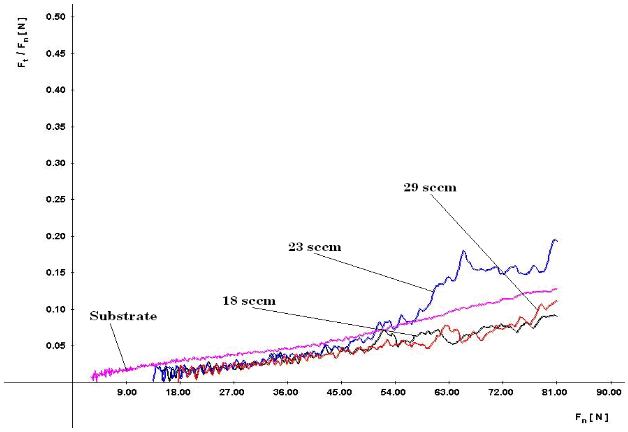

Figure 2 : Record of coefficient of friction

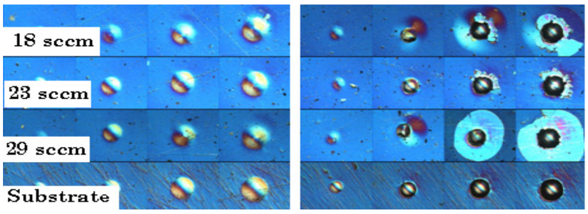

Figure 3 : Morphologies of static indentation (indenter: left - 0,5 mm right - 0,2 mm)

Another adhesion test is the test of coefficient of friction, which is depicted in Figure 2. On this record there are four trails of friction coefficient. One of them is the trail of the substrate and the others are trails of coatings.

The friction coefficient of the substrate linearly increases from value which is very near to zero to up 0,13 at normal force 80 N. All friction coefficient of a-SiC:H coatings linearly increased too, however the value of friction are below the record of the substrate, except running of coating, which was prepared under methane at flow 23 sccm. At this coating the friction coefficient at the normal force 50 N suddenly enhanced upper running of substrate.

The final test from adhesion series is the test of static indentation, which is shown in Figure 3. This test was applied in order to get further information of adhesion behavior of deposited coatings. For contradistinction two measurements were performed. First measurement was performed using Rockwell diamante ball indenter with diameter 0,5 mm and the second one using Rockwell ball indenter with diameter 0,2 mm. The applied load increased gradually at 20, 40, 60 and 80 N (columns).

From morphologies (Figure 3) of indenter 0,5 mm we can see, that all records are without failure. However, the morphologies of indenter 0,2 mm have brittle failure around the indent. The coating that was prepared under 23 sccm flow of methane shows minimal failure.

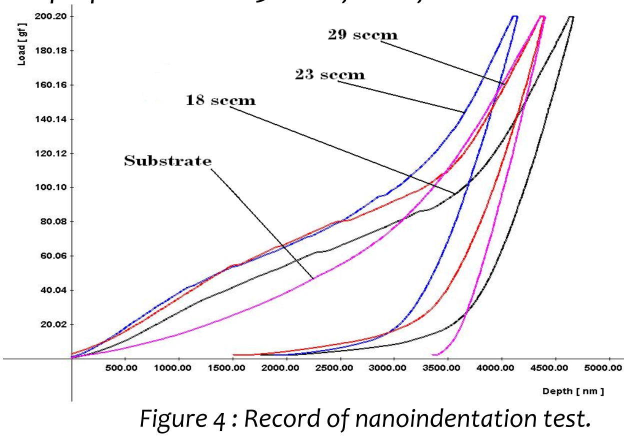

Figure 4 : Record of nanoindentation test. Maximal load 200 g

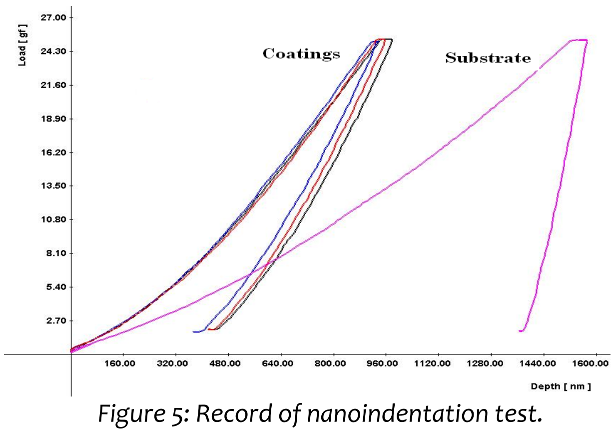

Figure 5: Record of nanoindentation test. Maximal load 25 g

Next mechanical analysis which was applied to coatings was nanoindentation test. Test was performed at three loads; 200 g, 25 g and 5 g. All hardness were counted according formula (1). The record of load-unload displacement curves for maximal load 200 is depicted in Figure 4. We can see that loading curve of substrate is among other curves of layers. The load 200 g is too high for assessment of thin films a therefore running of curve is the same as curves of substrates with films. For flow of CH_4 /18 sccm was counted nanohardness 3,4 GPa, for flow of CH_4 /23 sccm was counted nanohardness 4,5 GPa and flow of CH_4 /29 sccm was counted nanohardness 4,5 GPa as the same as substrate. Slopes of unloaded curves are under the same angle at all samples.

In Figure 5 there is running of curves of nanohardness test for maximal load 25 g. We can see that curve of substrate is outside the group of coating curves. The nanohardness of the substrate is 3,9 GPa and it is the same as previous measurement for maximal load 200 g. Nanohardness for coatings is approximately 10,5 GPa for all coatings. Slope of unloading part of coating curves is higher than substrate. That is why the coatings show higher rate of elastic deformation compared with substrate.

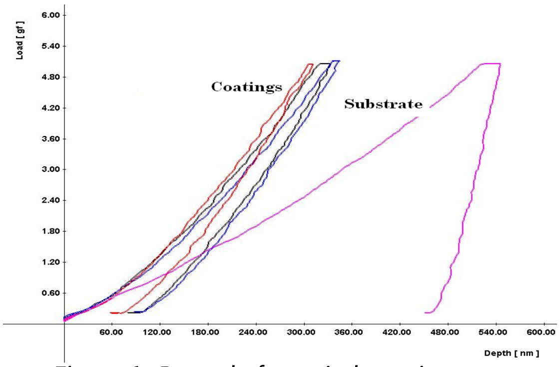

Figure 6 : Record of nanoindentation test. Maximal load 5 g

In Figure 6 is shown load-unload curves of nanoindentation test for maximal load 5 g. This measurement is very important due to assessment of hardness of single a-SiC:H thin film. It is important to mention that the hardness of coating itself must fulfill the condition, that depth of indenter has to be less than one tenth of value of total thickness of coating. The condition for measurement at load 5 g was fulfilled; because the overall thickness of all coatings was about 5000 nm and the depth of indent does not exceed value of 350 nm. The hardness of all coatings is approximately 21 GPa.

The substrate achieved the hardness approximately 6 GPa. The hardness of this substrate is higher compared with previous measurements (3,9 GPa at load 200 g and 25 g). Increasing of hardness was caused by polishing and thus hardening of surface of substrates before depositing. By polishing, a hardening layer is called Beilby layer. This layer has thickness approximately to 100 nm.

CONCLUSIONS

The hydrogenated amorphous silicon carbide (a-SiC:H) coatings were prepared using PECVD technique. After 2 hours, the thickness of coatings of all samples achieved approximately 5 μm, independently on flow of methane (CH_4). The roughness of coatings was the same as the substrate R_a = 0,02. Acoustic emission record from adhesion test gave these results: the critical load (L_c) is about 10 N for the sample prepared under the flow of methane 23 sccm. The critical load (L_c) is 12 N for the coating prepared under the methane flow 29 sccm. Finally, the critical load (L_c) is approximately 21 N for sample prepared under the flow of 18 sccm.

The layers which were prepared under flow 18 sccm and 29 sccm show lower friction coefficient than the substrate. The layer which was prepared under the flow 23 sccm shows running of friction coefficient below running of substrate, too. However, this running suddenly increases above the running of substrates at the normal force 50 N.

Tests of static indentation shows, that all records of Rockwell indent tip 0,5 mm were without failure. The morphologies of indenter 0,2 mm, however, show brittle failure around the indent. The coating, which was prepared under 23 sccm flow of methane, shows the least failure.

The results from nanoindentation test show, that steel substrate gives bulk hardness 3,9 GPa, while surface hardness of substrate is 6 GPa (due to thin Beilby layer) for measurement of load 5g. The single a-SiC:H film attained a level of hardness 21 GPa (at loading 5g).

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