CLASSIFICATION OF INTERNAL DAMAGES OF THE SAMPLE WITH A LAYER BASED ON ACOUSTIC EMISSION AND MICROSCOPIC OBSERVATIONS

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Summary

An acoustic emission (AE) method was used to detect internal damages of diamond-like-carbon (DLC) thin layer deposited on silicon substrate.

The AE signals were recorded during the entire indentation test using a broadband piezoelectric microsensor with measuring range 80Hz-1950kHz. Depending on the indenter's load, observations of surface by scanning electron microscopy (SEM) did not reveal any damage or showed single small cracks in the Vickers impression area. Therefore, it was assumed that the majority of the AE pulses originate from internal damages. Using the method of Focused Ion Beam (FIB) ion milling, indenter impression cross sections were performed. In this way, internal damages caused by indentation, such as decohesion of DLC-layer, DLC-layer cracking and the Si-substrate cracking, were revealed. As a result of microscopic and AE signal data comparison, it was possible to identify the type of failure and to describe the progress of damage during indentation.

The results will be helpful in the selection of parameters during hardness tests of layered materials and in the analysis of the strength properties of the samples with layers.

Keywords: acoustic emission, indentation, cracks, decohesion, FIB.

KLASYFIKACJA USZKODZEŃ WEWNĘTRZNYCH PRÓBKI Z WARSTWĄ ZA POMOCĄ METODY EMISJI AKUSTYCZNEJ I OBSERWACJI MIKROSKOPOWYCH

Streszczenie

W niniejszej pracy metoda pomiarów emisji akustycznej (EA) została zastosowana do detektowania wewnętrznych uszkodzeń cienkiej warstwy DLC nałożonej na krzemowe podłoże.

Uszkodzenia realizowane były w teście indentacji, a rejestracji sygnałów EA dokonywano podczas całego procesu obciążania i odciążania wgłębnika Vickersa. W zależności od wielkości maksymalnego obciążenia na powierzchni odcisków obserwowano za pomocą mikroskopu skaningowego brak bądź występowanie małych pęknięć warstwy DLC. Zarejestrowane sygnały EA pochodziły głównie z uszkodzeń wewnętrznych, które były ujawnianie za pomocą trawienia wiązką jonów. Zaobserwowano uszkodzenia różnego rodzaju, takie jak decohezja i pęknięcia lateralne warstwy i/lub podłoża. Za pomocą parametrów sygnału EA możliwe było określenie rodzaju oraz zasięgu wewnętrznych uszkodzeń.

Słowa kluczowe: emisja akustyczna, indentacja, pękanie, dekohezja, FIB.

INTRODUCTION

Deposition of layers, thin films or multilayered structures contributes significantly to technology in the field of electronics as well as in biomedicine or devices construction.

The determination if a method of deposition is suitable for technological application depends on the tests results including microhardness tests for scratching resistance and wear strength. For this group of materials in the first stages of damages the most common are decohesion of the layer and the substrate or internal cracks in the layer or substrate.

Observations of the surface morphology (scratches [1], impressions of indentation [2], wear traces [3]) are not able to reveal damages like internal cracks or delamination but the presence of these damages will affect the results of strength test.

Diagnosis of the actual state of the studied layer-substrate system has a major significance in the evaluation of properties and suitability of the technological – structural solution.

Simultaneous measurement of AE signals and performance of diagnostic tests is an example of complementary characterization methods. Registration of AE signals has been successfully applied in the detection of damages under heavy load conditions.

The aim of this study is determination of effectiveness of application of the AE method for a diagnosis of the first damages of a substrate with a layer.

Damages were introduced in the form of indenter imprints during microindentation tests. The authors undertook the study of micro- and nanodamages and investigated the relationship between AE data and the microscopic images, including the SEM micro cross-sections of impression. This study is supplementary to the previous work of other authors [6].

1. EXPERIMENTAL DETAILS

The indentation tests were executed with the Vickers indenter. The maximal loads of indentation test were 100mN, 200mN, 300mN 400mN and 500mN. We made a few tests with the same indentation load, and names of tests consist of the value of load and of a letter (a, b, c..) for subsequent tests.

During test indentation the AE signals were registered simultaneously. The AE measurement equipment was described in the publication [3].

The study concerns DLC layer deposited on monocrystalline Si substrate by RF PACVD (Radio Frequency Plasma Assisted Chemical Vapour Deposition). The thickness of amorphous DLC layer was 1μm.

The impressions were observed by the scanning electron microscope Auriga-type (Zeiss). Next, sequences of cross-sections of the impression were performed by milling system FIB (Focused Ion Beam) as shown in Fig.1.

The AE-signals were analyzed using PlexPro program and were elaborated in time and frequencies domains. From the total registered signal AE signals peak-type signals were extracted for which the maximum amplitude, duration, envelopes, and the signal energy (equivalent to signal strength) were determined. In the frequency domain was obtained STFT (Short Time Fourier Transformation) map.

2. EXPERIMENTAL RESULTS

The microscopic observations of imprints surfaces indicated that above the 300mN of load, the surfaces are damaged in each test. Due to the large number and variety of failures the imprints without any or with small surface damages, were selected for further analysis.

The increase of the maximal load causes the increase of the number of generated peaks of AE signals.

A presence of AE-peaks for indentation test at 100mN is presented in Fig.2.

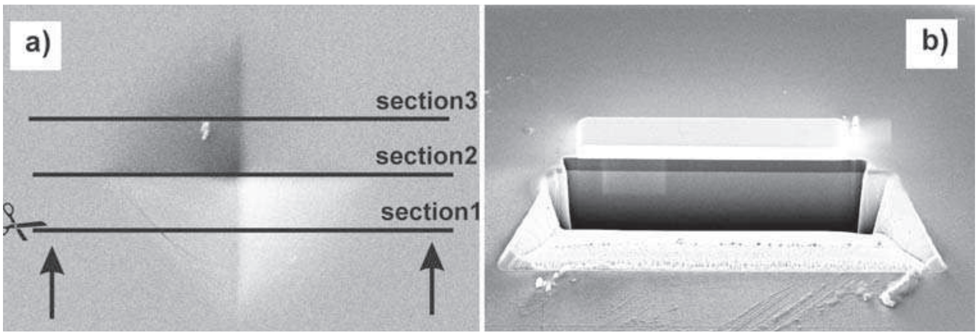

Fig. 1. The method of cross-section a) position on the impression,
b) example of cross-section by the FIB system

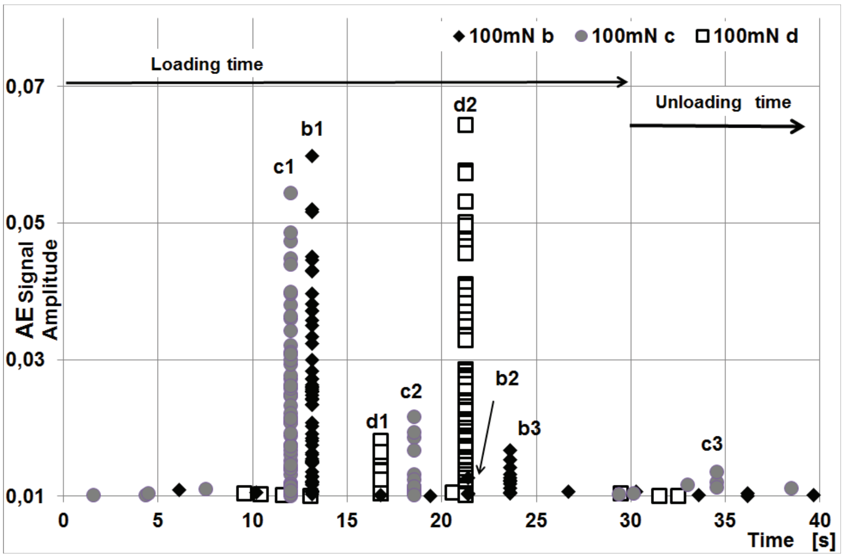

Fig. 2. The AE peaks registered during indentation tests under 100mN load

Under the load of 100mN two or three AE-peaks were generated. At load of 200mN 5 to 9 AE-peaks were observed, and at 300mN the number of AE-peaks exceeded 20. The maximal amplitude of the registered peaks increased up to 0.35V at 300mN. It was five times greater than the AE-peak amplitude at 100mN of load.

Even providing the same parameters of the indentation test it results in significant differences in damages. The 100b and 100e tests are carried out by generating just 3 and 2 AE-peaks, but caused damage of a completely different type, as shown in Fig.3.

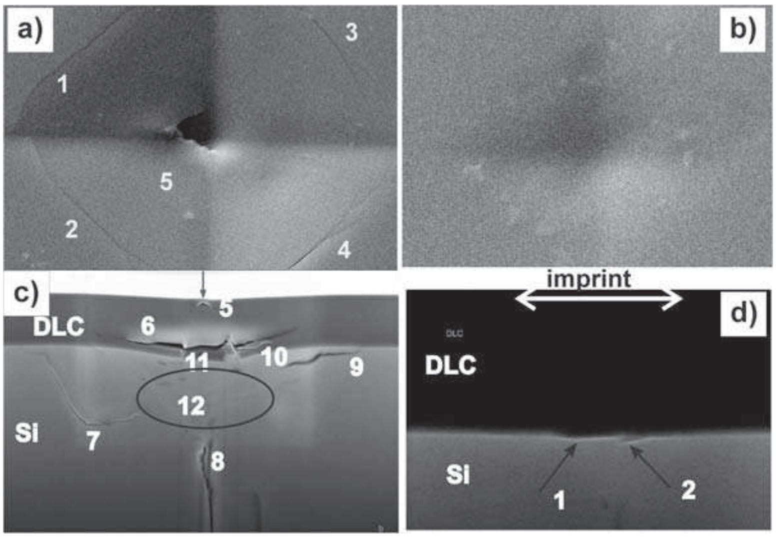

Fig. 3. SEM images of two impressions : a) top view 100b, b) top view 100d, c), d) correspondig cross-section made by FIB system, numbers indicated surface and cross-section failures

The surface of impression 100b, presented in Fig.3a is damaged with four circular surface cracks and one small hole in the center. Below the surface three large opened cracks of DLC and three extended cracks of Si-substrate were detected. Fig.3c. A total of twelve large defects detected at the surface consist of fife fissures and nine cracks of inside. In addition, the image reveals a very slight deformation of the Si material in the form of darker spots (near the side of the ellipse). All this destruction may be the source of the AE-signals. As shown in Fig.3b the look of 100d impression is quite different. The surface of the imprint is not failed. Visible is only the plastic deformation of DLC layer. The inside of impression is slightly damaged. The arrow1 indicated delamination at the DLC layer/ Si-substrate and the arrow2 highlighted the presence of a short diagonal crack at the Si-substarte. The size of this delamination is very small – about 200 nm long Determination of the kind of a failure, i.e. distinction between delamination or cracks, is possible basing on the values of the total signal energy of the registered peaks. This parameter is described by the formula:

$$\Sigma\int(AE)^2dt$$

This parameter describes the total amount of the AE signals power during whole indentation-test.

The value of total energy for AE peaks in test 100d is by 1/4 larger than the value calculated for AE-peaks from 100b test, for which damages were significantly larger. Fig.4. presents the bars of total energy values calculated for three indentation tests at 100mN load. The SEM images of 100b and 100d tests were presented in Fig.3. For the 100e test the types of damages were similar to that of 100d test, but the size of decohesion was slighthly larger. The smallest value of the Total Energy corresponds to the open damage and as shown in Figure 4. remains minimal even at numerous internal failures. The damages in the tests 100d and 100e are similar. The difference in Total Signal Energy parameter is not significant, but it rises with increasing size of individual defects.

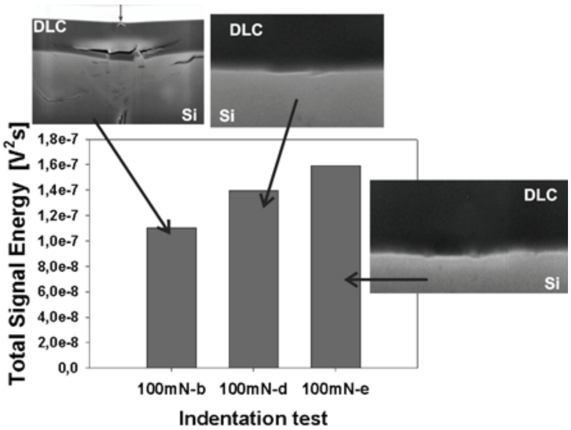

Fig.4. Comparison of the total energy for different damages under the 100mN load

Similar results were obtainded for the tests under 200mN load.

Fig.5. shows the results for two indentation tests, which caused defects of different types and size. The bigger Total Signal Energy value was calculated from the AE signals recorded during the 200b test. The damage resulting from indentation occurred only inside the sample as several lateral cracks of DLC layer and small cracks in Si substrate. The damages of smaller size and lower number in the test 200d were the source of a larger number of individual pulses. But these pulses correspond to smaller amplitude and shorter period of time as a result of which the calculated Total Signal Energy parameter is much smaller than that obtained for the test 200b. It should be noted that for the 200d test, one of the failures occurred on the surface of the DLC layer.

When failures become more extensive and their number increases, as it is at load of 300mN, the Total Signal Energies of AE-peaks at the imprints with damages on the surface are of a similar values.

The delamination can be related to the AE-peak with significantly smaller energy than the one originating from the crack. This relationship concerns similar damages and failures that are in a similar distance from the AE sensor.

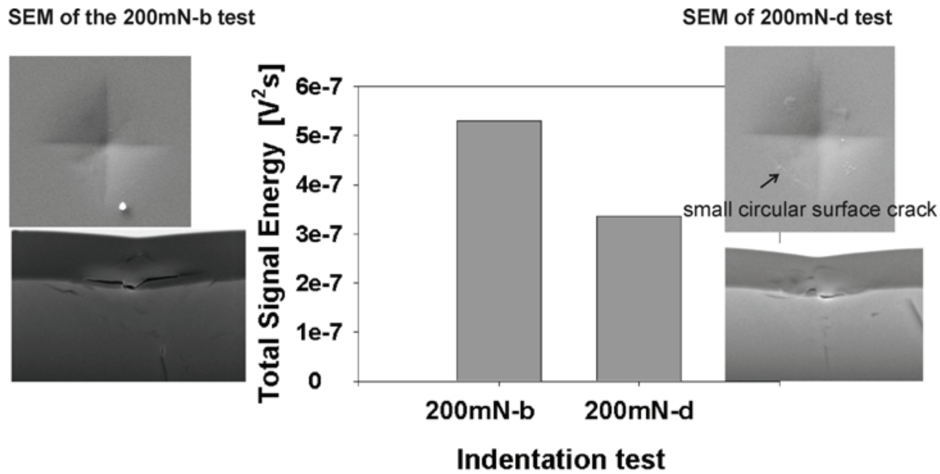

Fig. 5. Comparison of the total energy for different damages under the 200mN load

3. SUMMARY

Damages caused by the indentaion can be:

- Open, that are manifested on the sample surface.
- Internal, only inside under the surface of the sample.

The type od damage can by indicated by the parameter "Total Signal energy". It was found that for open damages this parameter is lower than for internal damages. But a similar number of defects is required in order to make the comparison reliable. The Total Signal Energy parameter calculated for a single pulse is also helpful to identify the type of a damage. Only for internal damades, when the value of Total Signal Energy increases, the AE signal was due to the decohesion of the layer from the substrate. When the value of Total Signal decreases, the source of AE signal was the internal crack.

The result of this research may be possibly applied to the investigations:

- Selection of the parameters of hardness measurements for thin films
- Determination of the origin of scattering of hardness results
- Application for quick comparative test of the adhesion of the layer to the substrate.

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