

ANALYSIS OF AE-SIGNAL GENERATED DURING FRICTION TEST OF DLC-LAYERS

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Summary

In the present work the results of tribological and simultaneous AE measurements of the DLC layers deposited on Si substrate are presented. The layers studied were 18nm, 200nm and 650nm thick. The best properties were obtained for the films with thickness of 18nm and 650nm. The value of average friction coefficient was stable and equal to 0,1 and no wear was observed. The 200nm layer had a very low adhesion to the substrate. Intense acoustic emission signals accompanied the friction.

Keywords: DLC-film, Acoustic Emission, sliding friction, adhesion, damage.

ANALIZA EMISJI AKUSTYCZNEJ GENEROWANEJ PODCZAS PRÓBY TARCIA WARSTW DLC

Streszczenie

W pracy przedstawione zostały wyniki badań tribologicznych i równoczesnych pomiarów emisji akustycznej na warstwach DLC na podłożu Si. Badano warstwy o grubościach: 18nm, 200nm i 650nm. Najlepszymi właściwościami charakteryzowały się warstwy 18nm oraz 650nm. Współczynnik tarcia był stabilny i wynosił 0,1 a warstwy nie uległy uszkodzeniom. Bardzo słabą adhezją do podłoża odznaczała się warstwa o grubości 200nm. Tarcia towarzyszyła intensywnej emisji sygnałów akustycznych

Słowa kluczowe: warstwa DLC, emisja akustyczna, tarcie ślizgowe, adhezja, uszkodzenia.

1. INTRODUCTION

Excellent mechanical, tribological, optical and electronic properties of diamond like carbon (DLC) coatings are exploited in different domains of industry. The major application of DLC is a chemical hard coating for mechanical or chemical protection [1]. Among numerous practical examples the DLC-fine film is used for resistant-coverage and anti-wear coating of optical-infrared elements, where good resistance of abrasion is very important.

In this paper the adherence and tribological properties of DLC-coating are described.

Simultaneous acoustic emission (AE) measurements are used to obtain additional information on the process and the phenomena occurred during the sliding friction in DLC layers.

2. EXPERIMENTAL DETAILS

2.1. Specimens

The DLC-fine films were deposited on Si-substrate by RF reactive sputtering, using a graphite target under various pressures of Ar and H₂. Finally, three thicknesses of DLC films were chosen for analysis: 18nm, 200nm and 650nm.

As the substrate three separate Si-wafers of 2" in diameter with thickness of 0.5mm were used. The surface of substrate was polished (average roughness Ra=5nm on the measurement section 400µm).

2.2. Experimental equipment

Tribological properties and adhesion of the studied films were determined using a "ball-on-disc" tribometer. During dry friction sliding the momentary friction forces were recorded. Then they were converted to the momentary friction coefficient. Three sensors of acoustic emission (AE) measure simultaneously the acoustic signals generated during friction. Schematic diagram of the set-up is presented in Fig. 1.

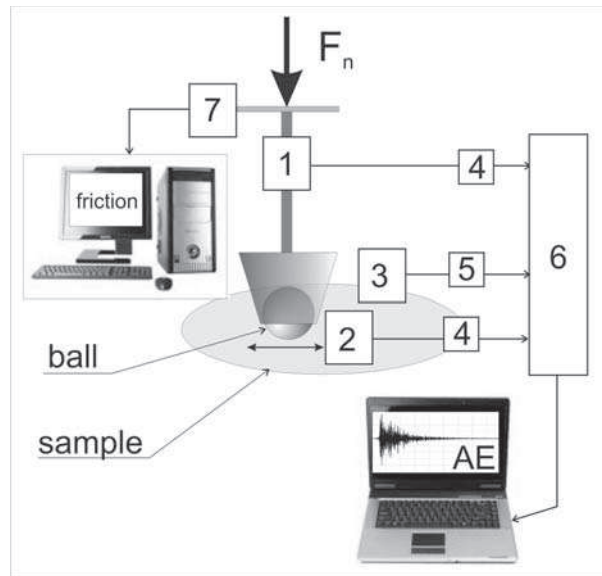


Fig. 1. Schematic diagram of the set-up for the measurement of friction and acoustic signals.

- 1 – AE sensor freq. Range: 400-750 kHz
- 2 – AE sensor freq. Range: 60-1950 kHz
- 3 – Accelerometer freq. Range: 5-60 kHz
- 4 – AE Preamplifier
- 5 – Signal Conditioner
- 6 – Data Acquisition Card
- 7- Friction force Sensor

The proposed set-up for the measurements of vibroacoustic and acoustic emission is composed with three sensors: two high-frequency sensors (1) and (2), and accelerometer (3) shown in Fig. 1. Sensors (2) and (3) were fixed on the surface of samples with DLC-film in a distance of about 15mm to the wear track. The sensor (1) is placed in the holder of ball with intention to measure acoustic emission signals generated from damages of the ball. Registration of the vibroacoustic and acoustic emission signals was in the 60 s periods in the same way for all tests.

Tribological conditions are described in [2]. The performed tests were a kind of multi-pass with sliding velocity 0.21 mm/s and wear trace length.

2.3. Methodology

The aim of this work is an analysis of the relation between the tribological properties and the generated and recorded AE signals. The values and changes of the momentary friction coefficient and of the AE signals amplitudes recorded as a function of test-time in several time periods (A, B, C...) are compared. We verify if the fluctuations of momentary friction coefficient are accompanied by simultaneous impulses of AE signals. AE signal is analyzed in relation to impulses number, their duration and reproducibility determined from AE-

time plots. The frequency spectra were calculated. Due to the non stationary character of AE signals the Short-Time Fourier Transform (STFT) was applied. The STFT transform divides up the signal into small time segments and performs Fourier transforms on each segment of time to derive the spectra. The Kaiser-Bessel window was used due to its higher selectivity than other types of gaps [3-4].

During the friction test several AE signal measurements were performed in dependence of the appearance of fluctuation of the momentary friction coefficient. We named the tests as stage A, stage B.

3. RESULTS AND DISCUSSION

Generally, DLC-films are characterized by very low friction coefficient approx. 0,1 [1, 5] and their key feature is an excellent adhesion to the substrate [5, 6].

In this work we present the results of tribological tests of DLC-films on Si-substrate characterized by different adhesion to the support.

The DLC layers of 18nm thick were characterized by high wear resistance and excellent adhesion to the Si substrate. Multi-pass friction tests performed on the 18nm DLC layer did not show substantial surface damages. The abrasion of the counterbody was observed only together with a transfer of wear products. The corresponding AE signals described the equipment noise and the ball abrasion. Some AE impulses appeared in data registered by sensor1 located in the support of the counterbody. The results obtained due to friction on the 650nm layer are presented in Fig. 2. The undamaged DLC-film resulted in non- AE activity [4, 5].

During the friction test the value of average friction coefficient decreases from 0,15 to 0,08, however in the 60s periods: A, B, C its variations are related to the changes of the movement direction of sliding. The arrows mark one pass of the ball shift. Few AE impulses were recorded and their amplitudes were very low. They were registered only by sensor1. From this fact and from the analysis of the friction trace one can conclude that the AE signals originated from small damages of the ball. Spectra obtained for the subsequent friction stages showed the higher amplitude of the registered AE signals in the frequency range of several to 100kHz.

Average value of friction coefficient for the 18nm DLC layer was about 0,10 during the entire test. AE impulses were not generated and the background level did not change in the next stages of friction. The increase of the load also did not cause any damage on the layer.

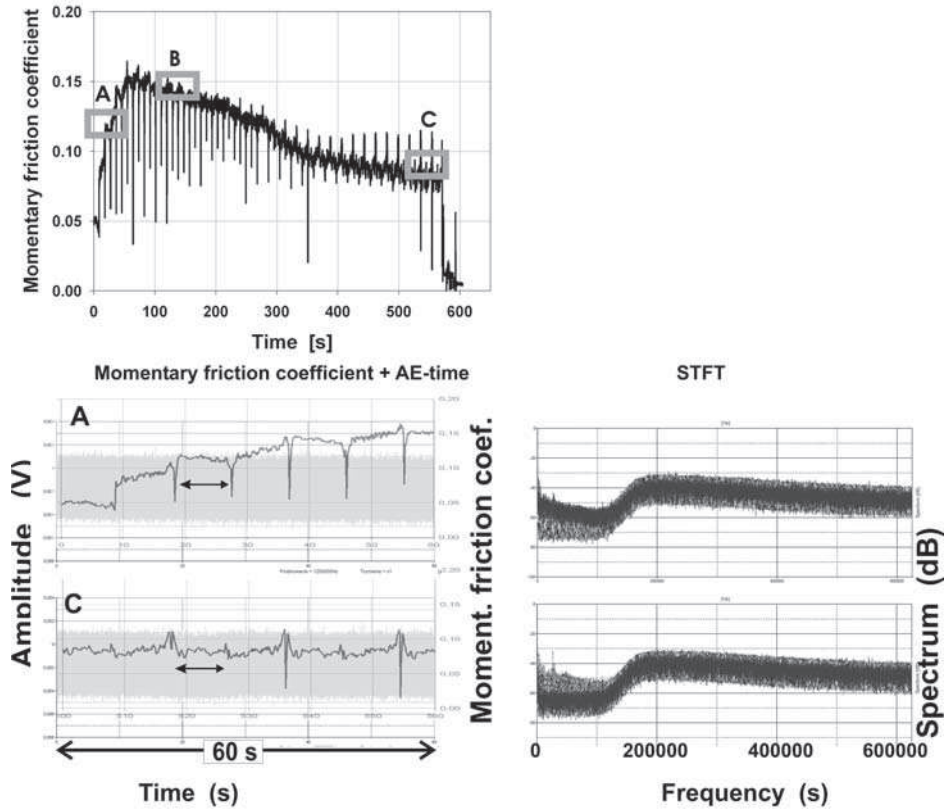


Fig. 2. Results of tribological and AE signal measurement on 650nm thick DLC layer, registered by sensor1

Completely different properties revealed the 200nm thick DLC layer. During the friction test three phases of the average friction coefficient values were observed (Fig. 3).

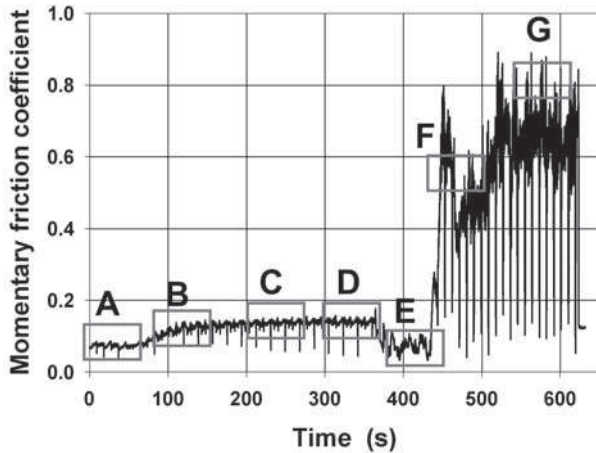


Fig. 3. Plot of changes of the friction coefficient values during test on 200nm thick DLC layer

In the first phase (stages A to D), the value of average friction coefficient was constant ($\mu=0,08$). It lasted up to 200-300s and corresponded to 20-30 passes. In the next phase a decrease of average friction coefficient was registered, however, significant fluctuations were observed. This phase lasted about 100s (10 passes of ball). After that the average friction coefficient value increased catastrophically up to 0,7 with momentary

fluctuations in the range of 0,55 to 0,9. The boxes in Fig. 3 mark the registered AE signals. Our intention was the starting of the recording of AE signals when the first changes of the momentary friction coefficient values occur.

First phase of friction indicated by low momentary friction coefficient values shows similar aspects of the generated AE signals as those obtained in the case of 18nm thick layer. The diagrams of AE amplitudes variation and the plots of momentary friction coefficient superimposed with the same time axis are presented on the left in Fig. 4. On the right in Fig. 8 the STFT spectra for A and D stages are shown.

The amplitude of AE signal is comparable for all measurements of this non-wear phase. The STFT spectra for the whole range of frequencies from several to 600kHz do not manifest any rise of amplitude. The single peaks for frequencies: 156kHz, 280kHz and 320kHz are the result of counterbody movements and the activity of tribometer.

The amplitudes amplification is stable for the whole range frequencies and does not exceed -50dB.

The comparison of these results and the amplification level of noise -90dB provide information about increase of amplitudes of AE continuous signals. The next two phases of friction presented in Fig. 5 have numerous AE impulses recorded.

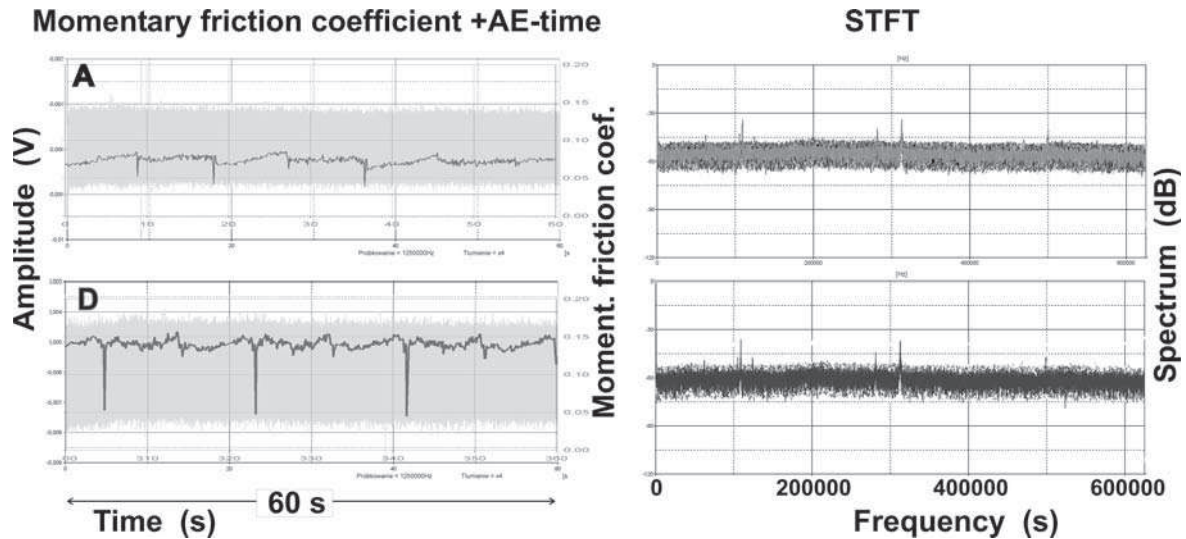


Fig. 4. Diagrams of momentary friction coefficient (dark line) and AE-time (gray lines) registered on 200nm DLC layer and STFT spectra for A and D stages of friction

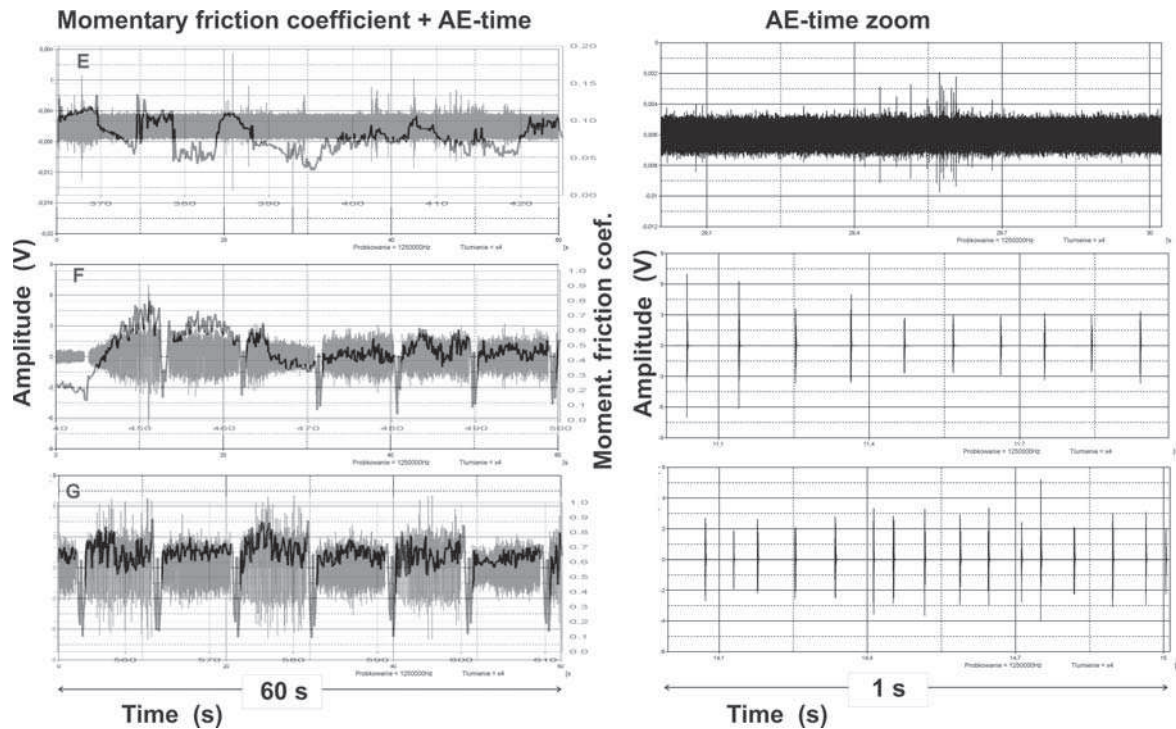


Fig. 5. Diagrams of momentary friction coefficient (dark line) and AE-time (gray lines) registered on DLC layer of 200nm thick

During E stage of friction non-stationary, incoherent groups of transient impulses were generated. Analysis of the AE signal and the momentary friction coefficient shows weak correlation between these parameters. Spectra STFT for this stage show the amplitude rise for frequencies range from 480kHz to 560kHz. During friction at the E stage transients amplitudes increase 200 times. Perturbations of momentary friction coefficient correspond to AE-time. The rise of momentary friction coefficient is accompanied by augmentation of AE amplitude. Regular pauses decline of AE signal and low value of friction

coefficient coincide with change of movement direction of counterbody. It permits they exact superimpose and comparison of diagrams. At G stage the momentary friction coefficient value increases as well as number of transients. At both F and G stages simple impulses generated are repetitive. Phenomenon of reappearance is presented by zoom of one second diagram AE time (Fig. 5 on the right). Intervals between successive transients indicate the tendency to diminution of break during friction-time. Thus for F stage intervals among impulses is 100ms in average, however for G stage is 65ms. At both cases

a certain signal modulation occurred. Higher frequency is obtained for G stage than for F stage. Another difference between tribological behavior at F and G stages was shown by AE signals. Only during G stage the distinction of AE signals takes place. Values of amplitudes depend on the sliding direction. They have much higher values in a return direction. Parallely momentary friction coefficient is varied. STFT spectra calculated for AE signal of G stage are presented in Fig.6. STFT spectra calculated from whole 60s AE show amplitudes magnification for all range of frequencies. Diagrams in Fig.6 b and c expose 8s section of AE registered respectively by sensor1 and sensor2 (see Fig. 1). This selected AE time part includes strong signals generated during one pass of friction.

In contrary to the results for the whole range of AE at G stage, STFT spectra of part AE time show some preferences of frequencies. For the signal registered by sensor2 fixed to the surface of the sample a higher amplification of amplitudes exists at a frequency about 262kHz. Different appearance is observed for the spectrum STFT calculated from AE-time registered by sensor1 attached to holder of counterbody. Fig.6c presents the rank of frequencies: 110kHz, 140-170kHz, 183kHz, 198kHz, 240kHz, 267kHz, 345kHz, 445kHz, 476kHz characterizing transients with maximal amplitudes.

Between impulses recorded by sensor1 and 2 we do not observe phase difference. Impulses have similar profiles and they happen at identical time.

Duration of these two multiple impulses is comparable. But it seems that sensor 2 is more sensible and it recorded the biggest number of tribological events.

Vibroacoustic sensor 3 recorded small number of impulses and only for terminal friction stage. STFT spectra exhibit impulses at level -40dB and in the range of frequencies from several to 15kHz.

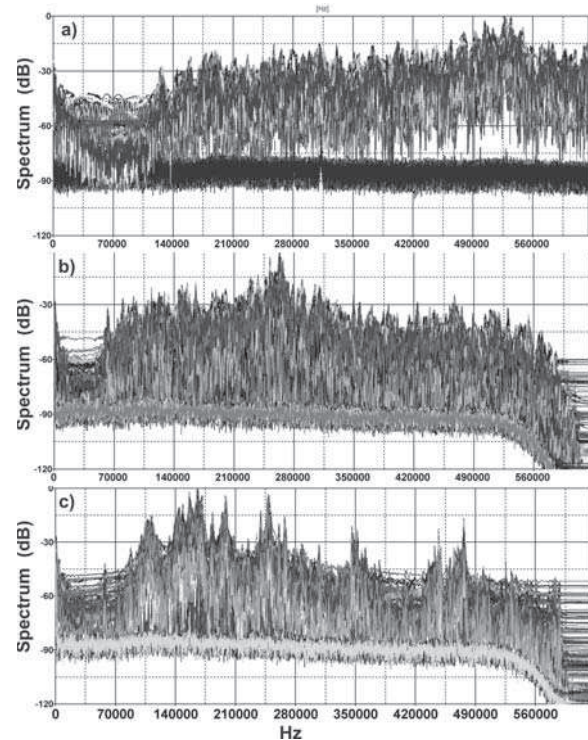


Fig. 6. STFT spectra calculated for G stage a) from 60s AE recorded by sensor2, b) from 8s part of AE recorded by sensor2, c) from 8s part AE, recorded by sensor1

The presented results of tribological and AE measurements were correlated with the topography of wear traces. DLC layer 18nm thick and 650nm thick were not damaged. The only effects of friction are the abrasion and product of counterbody transferred on the surface of sample. This kind of wear generates AE response with higher amplitude of continuous signal. Similar results obtained for plastically deformation were described at [7].

The friction of the sample with 200nm thick layer significantly damaged its surface, but damages appeared after relatively long time of friction. When tribological test was stopped during first phase of friction where average friction coefficient was low and AE impulses were not recorded, we did not observe any wear (by optical microscope with Nomarski contrast). In E stage first microcracks and spalling of DLC layer became visible. Next cycles of friction catastrophically demolished surface. Fig. 8 presents SEM images after 630s of friction, it is after 70 passes of ball.

The topography of wear trace was observed by scanning electron microscope – Fig. 7. For analysis of all wear tracks succeeding scans were realized with 200 time magnification.

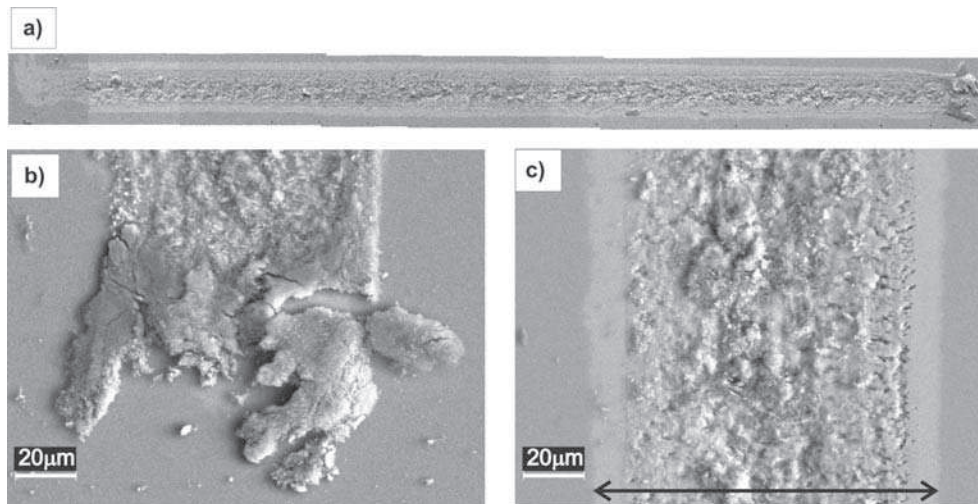


Fig. 7. SEM images of wear trace after friction on DLC layer 200nm thick
a) topography whole track b) zoom of track extremity

In whole trace area shown in Fig. 7a appear almost uniformly spread damages: spallings, microcracks, agglomerations of wear particles. On one extremity of trace presented in Fig. 7b occurred large accumulation of wear particles. It is crushed, pressed conglomerate of wear particles. Fig. 7c shows magnification of central trace fragment. Width of trace marked by arrow, is about 100µm. Clear gray strip zone along wear track is showed on the Si-substrate. Sideways wear trace were occurred delaminating of DLC layer. Probably the break took place during E stage of friction. Products of wear: DLC, Si (counterbody), Si-substrate particles were mixed agglomerates of various dimensions. On the right of trace (Fig. 8c) numerous microcracks of Si-substrate are manifested. The surface of trace is very rough and wear particles are strongly agglomerated. During F stage number of fine cracks are slightly increased, but they are covered by wear particles.

3. CONCLUSION

Tribological properties of DLC layers depend on their thickness, on the method and parameters of processes. The DLC layers 18nm and 650nm thick produced by RF sputtering were coated by one sputtering process and they characterized very good abrasion resistance and excellent adhesion to Si-substrate. The average friction coefficient is stable and have low value of 0,1.

In this case the identification of wear using a registration of AE signal wasn't useful and non provided new information for discussion of results. The amplification of amplitudes signal level is sign of slight 200nm abrasion of counterbody. The DLC layer has a limited abrasion and delamination resistance. In the beginning of friction damages do not appear. This non-wear phase is at 200-300s, according to associated events. The first changes are small spallings, short, single cracks and plastic

deformation of DLC-coating. These damages generated measurable activity of AE signal. During this stage when adhesion is lost the average friction coefficient is diminished. In this moment non-regular and non-stationary AE signals appear. Their frequencies is the range: 480-560kHz. Terminal phase of friction consists of the damages formation and the transfer of wear products. The average friction coefficient rises catastrophically until value 0,9. In this stage the friction is accompanied by numerous AE transients. Size of destruction intensity can be described by quantity of AE impulses generated per second. This number rises from 10 to 15 impulses/second in terminal 70th pass of friction. It means 50% rise of the origin number of AE impulses or number of damages (tribological events). Considering continuous regularity of impulses it is probably appearance of cracks in substrate.

The measurement AE signal adopted at tribological tests made possible the damage detection and identification of the beginning of cracking of DLC layer, microcutting of Si-counterbody and breakage of Si-substrate.

Friction monitoring by registering AE signals provided observation of wear intensification, that was not visible by measuring the changes of momentary friction coefficient.

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