# Testing of Diamond-Like Carbon Coatings under Slip-Rolling Friction Monitored by Acoustic Emission

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Keywords: Wear life, DLC coatings, Slip-rolling friction, Acoustic Emission Testing

## Abstract

Wear tests were carried out on different DLC coatings under lubricated and dry slip-rolling friction in a Twin-Disc Testing rig. The analysis of Acoustic Emission (AE) provided an easy and comfortable tool for monitoring the wear life of DLC coatings. A DLC coating in-service was indicated by the appearance of no or just single AE events due to single damages (crack, small spallings, etc.) to the coating and the counterbody. A high AE activity consisting of events of low energy was due to large spallings in the coating which led to the undesirable contact between steel substrate and steel counterbody. However, a high AE activity caused by events of high energy indicated the end-of-service of the coating. In each and every test this AE activity was observed, the coating failed in the end. The AE responded much quicker and was much more sensitive than any changes in the Coefficient Of Friction (COF).

## 1. Introduction

Diamond-Like Carbon (DLC) coatings show good resistance to wear and low coefficients of friction (COF) under sliding friction, especially under dry conditions [1]. DLC coatings also lead to low wear on counterbodies made of steel [2]. DLC coatings are already used in ball bearing races or cages, as protection of hard disc drives, as well as in a number of moving parts inside automobile engines (e.g. in diesel engine injection systems) [3]. The field of applications can extremely be widened, if they are resistant to slip-rolling conditions at contact pressures $P_0$ > 2,2 GPa. As the wear life of such DLC coatings is important for the performance of the respective ball bearings, etc., DLC coatings were tested here. The performance of different DLC coatings under slip-rolling friction was examined.

The well known analysis of Acoustic Emission (AE) is already used successfully in tribology. Sliding tests of CrN [4] and DLC [5],[6] coatings were monitored using the AE. In contrast at slip-rolling conditions both samples are in motion and as a consequence the application of AE is more demanding. The AE was introduced to monitor in-situ the tests of different DLC coatings under slip-rolling friction.

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## 2. Setup-Tribology

In an Amsler type twin-disc wear tester steel samples with a thin DLC coating were tested under lubricated and dry slip-rolling friction. Paraffin oil without any additives was used as lubricant (boundary lubricated conditions). In figure 1 the testing condition is illustrated. The tribological test conditions are listed in table 1. Two specimens (altogether a test sample) in motion rolled on each other with a slip of about 10%. Maximum initial Hertzian Pressures $P_0$= 1,5 GPa; 1,9 GPa and 2,3 GPa could occur in the centre of the contact area. The tests under dry slip-rolling conditions were carried out with a Hertzian Pressure $P_0$= 1,5 GPa.
A bearing steel (100Cr6) served as the substrate of the DLC coating and also as the material of the counterbody. Only the cylindrical specimen was coated with an interlayer and a DLC coating with a thickness of about 1-10 µm. The coatings of six different suppliers were monitored using AE. The properties of these coatings are listed in table 2. Their tribological behavior in the tests is discussed in detail elsewhere [7]. For further information about each test use the code number presented here. In the code numbers the different letters denote different suppliers and the numbers indicate the test number of a DLC coating tested of one supplier.

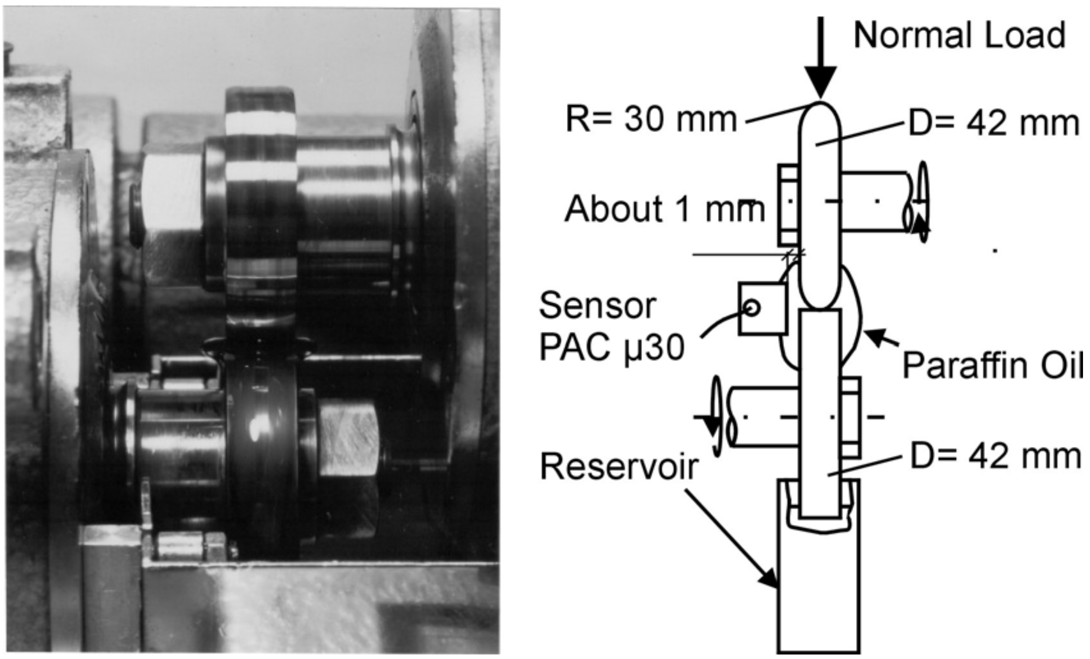


Figure 1: Picture of a test sample in the Amsler type twin-disc wear tester and sketch of the acoustic coupling of the AE sensor under lubricated slip-rolling friction.

## 3. Setup-Acoustic Emission

The AE signals were detected by means of an AE sensor (PAC-µ30) with a resonant frequency of about 270 kHz. The sensor was arranged in a distance of about 1 mm to the center of the contact area. The sketch in figure 1 shows that the oil served also as couplant. Under dry slip-rolling conditions the ambient air served as couplant. The output of the sensor was amplified 40 dB (lubricated) or 60 dB (dry) by a PAC 2/4/6 preamplifier. Every AE signal which exceeded the threshold of 61 dB or 45 dB originated from the contact area and was stored as a hit by an AE measurement system (PAC-DISP). The energy in aJ (atto=$10^{-18}$) of each hit was used. The energy E was calculated by

$$E = \frac{1}{R} \cdot \int_{t=0}^{t*} \left[ A(t)^2 \right] \cdot dt$$

with R = 10 kΩ the resistance in the preamplifier, t the time in seconds, A the time-dependent Amplitude in Volt during exceeding the threshold of 61 dB or 45 dB and t* the duration in seconds. The calculation of the AE energy excluded the influence of different preamplifications. The configuration of the AE measurement is listed in table 3.

## 4. Wear time history and Sources of Acoustic Emission

DLC coatings were tested here as a wear protection coating in steel contacts. The coating had to avoid a contact between the steel surface of the substrate and the counterbody. In doing so, the DLC coating was in-service. The service life ended when a coating exhibited spallings which were large enough to render the direct contact of steel against steel. Figure 2 shows the different stages of the use of a DLC coating and the corresponding AE under lubricated slip-rolling friction:

Fig. 2a) DLC coating perfect in-service. At the beginning of the test the counterbody was polished from rough ($R_Z$~ 3 µm) to fine ($R_Z$< 3 µm). The coating showed no damage. As long as no damage occurred neither to the coating nor to the steel no AE was recorded due to the proper threshold setting.

Fig. 2b) DLC coating in-service. The coating showed only minor defects after a total number of 1 million revolutions. The corresponding counterbody stayed polished. In accordance to this desirable tribological behavior only a few AE hits were recorded due to single damage events to the coating.

Fig. 2c) DLC coating end-of-service. After a total number of 60.000 revolutions the coating showed a spalling about 1 $mm^2$ in size. A high AE activity occurred after a total number of about 40.000 revolutions and stayed till the end of the test. The high AE activity which formed the basic noise was the result of the direct contact between the steel of the substrate and the steel of the counterbody. In addition, a few hits with AE energies of 10.000 aJ and more were registered. These hits were associated with single damage events to the coating, because the counterbody here stayed polished without damage. In general, spallings larger than 1 $mm^2$ led to heavy damages like plastic deformations to the counterbody (see section 5.2).

Fig. 2d) DLC coating out-of-service. As reference, a test sample without any coating in the steel contact was examined. The corresponding counterbody and the uncoated substrate showed small spallings, plastic deformations and cracks after a total number of 1.000.000 revolutions. These events were associated with single hits mainly with AE energies of 10.000 aJ and larger. A high AE activity as a basic noise was observed due to the direct contact of steel against steel.

Figure 3 shows the different AE sources, which can occur in the lubricated tribological contact. The AE hits showed an AE energy in a range of 100 to 100.000.000 aJ. The different AE sources were located in the DLC coating and steel. AE sources "subsurface" to the coating (under the coating surface) were difficult to observe. However, AE activity without damage to the coating and the counterbody would be assumed as hits out of delaminations, cracks and other AE sources "subsurface" to the coating. Considering the wear time history a separation of different AE sources and locations were possible. As long as a DLC coating was in service all AE hits had one origin (location): the coating itself. Only cracks which occurred in the wear track of the counterbody led to hits mainly with an AE energy above 10.000 aJ. These cracks were relief cracks and weren't critical. However, they only appeared rarely. Spallings with a critical size led to a high AE activity. The base noise indicated the undesirable direct contact between steel against steel (end-of-service) of a DLC coating. Furthermore, the AE activity got more intensive due to the number of single damage events from the coating and the counterbody.

The tests under dry slip-rolling friction conditions were too small for an analysis of the AE sources. Only five tests were taken out. More details are discussed elsewhere [8]. Fretting was a source of intense AE with large AE energies (see figure 7).

## 5. Wear tests monitored by Acoustic Emission

The classical cause for the failure of rolling elements in bearings are localized defects, in which large pieces of the contact surface are dislodged during operation. The appearance of such a defect with the size of 1 mm² was chosen as the criterion to mark the end of the wear life of the DLC coating tested (1 mm² -criterion) [7],[8]. If a DLC coating did not exhibit a defect of such a size after about one million revolutions, it was called resistant to slip-rolling fatigue. In order to determine the end of the wear life the tests were divided into logarithmic intervals to examine the surfaces with an optical microscope. This procedure led only to a rough estimation of the wear life, because after the test the sizes of spallings larger than 1 mm² varied in a large scale. In order to get a finer estimation of the wear life and to save time, it was important to have a technique, which was able to detect defects in-situ, while the tests were in progress. The wear tests were monitored by AE and the Coefficient Of Friction (COF) was recorded. In general, if a system is nearing the end of its wear life, the friction torque is dramatically increasing. However, under lubricated slip-rolling friction conditions the COF did not change significantly, even if the DLC coating was removed completely from inside the wear track [7]. Under dry conditions the COF wasn't able to detect undesirable damages to the DLC coatings clearly. Complicated tribochemical reactions were probably the reason for the different behavior of the COF [7]. In contrast, the tests were monitored successfully with AE.

## 5.1 Results

In figure 4 all results are presented of the tests under lubricated slip-rolling friction. Test samples with a bold and underlined number were monitored by AE. In all cases in which test samples reached the 1 mm²-criterion, a high AE activity could be registered, too. In many cases the AE criterion indicated the end-of-service very early.

## 5.2 Early Detection by Acoustic Emission

Spallings smaller than 1 $mm^2$ and damage events “subsurface” to the DLC coatings could lead to a high AE activity (AE criterion). In figure 5 the test sample with the code number A10 is shown. This test was terminated after a total number of 690.000 revolutions and a high AE activity was registered. This high AE activity occurred already after a total number of 23.000 revolutions, too. The base noise was caused mainly by the direct contact steel against steel. The small damage events in the wear track of the counterbody were mainly caused by the hard edges of the spalling in the coating. The damage events of the coating and of the counterbody were indicated by single AE hits. The AE energy of these hits was in most cases higher than the base noise.

Damage events “subsurface” to the DLC coating were another reason of an early detection. These damage events indicated the oncoming termination of the test (1 $mm^2$-criterion). Some samples, which per definition were called resistant to slip-rolling fatigue, were tested further to the 10.000.000 revolutions. The test samples B7, E3, F1 and G3 reached the 1 mm²-criterion after 1.000.000 revolutions. A high AE activity was recorded much earlier. Figure 6 shows the results of the test sample B7 and G3. The AE graphs show the AE in the test interval, in which the high AE activity began. The sources of AE had to be “subsurface” to the DLC coating like cracks and delaminations, because the amount of “visible” damage events were too small for the high AE activity observed.

## 5.3 Large Spallings

Spallings larger than 1 $mm^2$ could heavily damage the wear track of the counterbody. Under lubricated slip-rolling friction the counterbody showed plastic deformations and spallings. Under dry conditions a DLC coating, which was partly rubbed out off the wear track, could lead to fretting conditions between the steel surfaces. Such damage events led to a high AE activity in addition to the base noise. The base noise got more intense with increasing size of the spalling. Figure 7 shows three DLC coatings with a spalling larger than 1 $mm^2$. In all cases a very powerful and intensive AE activity was recorded:

Fig. 7a) The edges of the large spalling in the coating had dramatically damaged the respective counterbody. The high AE activity, which was recorded immediately, was due to the direct contact of steel against steel and to the observed plastic deformations and spallings of the counterbody.

Fig. 7b) After a total number of 483.000 revolutions this sample reached the 1 mm²- criterion with a very large spalling. Most of the coating was removed suddenly inside the wear track. The counterbody showed plastic deformations. The AE graph showed the AE activity from a number of 100.000 to 483.000 revolutions. In this test interval the AE activity changed from a high AE activity to a very high AE activity. The very high AE activity was due to the contact of steel against steel and to the heavy damaging of the counterbody and the coating, both appeared suddenly. Probably the high AE activity occurred earlier due to crack events in the coating.

Fig. 7c) The DLC coating was partly rubbed out off the wear track. Parts of the coating covered the transfer layer in the wear track of the respective counterbody. The AE graphs shows the evolution of the damages which could be observed after the tests. The removal of the coating happened suddenly. Nearly no AE activity was registered before a very high activity level of AE was reached. The high AE activity was mainly caused by fretting events through the contact steel against steel and appeared suddenly at a total number of 90.000 revolutions. The AE hits showed an AE energy in a different range as under lubricated conditions due to the different threshold settings.

## 6. Conclusion & Discussion

Wear life tests of DLC coatings on a steel substrate under slip-rolling friction conditions were carried out. The coated substrate ran against a counterbody made of steel. The test parameter changed as follows:

- DLC coatings of different suppliers
- Hertzian Pressures $P_0$ in a range of 1,5 – 2,3 GPa
- Lubricated with paraffin oil without additives and dry
- Coating thickness/ Surface finishing of the counterbody

The single tests were terminated by the 1 mm²-criterion. The AE was an excellent tool for an in-situ estimation for the end of the wear life of the DLC coatings tested here. Under lubricated conditions a high AE activity (AE criterion) indicated in-situ all samples which actually failed later on in the tests. A high AE activity was able to detect the failure much earlier than the 1 mm²-criterion. The sources of a high AE activity were as follows:

- Mainly originating from the contact of steel against steel. Sizes of spallings in the coating less than 1 mm² could lead to a contact of the substrate and the counterbody, both made of steel. The high AE activity appeared as a base noise.
- Continuous damaging to the surface of the counterbody. The edges of larger spallings in the coating were able to damage the counterbody plastically.
- Many events, like cracking and delamination, under the coating surface. These events could indicate the catastrophic failure which appeared suddenly.

The spallings showed different sizes above 1 mm ² after the termination of the tests. The total amount of coating material removed changed with the type and especially with the individual

quality of each coating. The AE reacted very sensitive to the end-of-service of a coating. A high AE activity as a base noise indicated the undesirable contact between steel against steel due to spallings in the coating. The AE turned out to be a quick and easy monitoring tool of wear tests to save test time and to get more comparable results. Furthermore the AE provided a useful tool for the observation of the wear time history. Tests could be stopped, if a damage event occurred. Regarding the wear time history of the DLC coatings in a steel contact a separation of different locations of the AE sources was possible.
Further tests under dry conditions have to be carried out. However a high AE activity occurred due to fretting conditions which were induced by a large and deep spalling in a DLC coating.
Throughout using the AE the advantages were as follows:

- Detecting the onset of undesirable wear
- Early Detection of the end-of-service
- In-situ view of the tribological history
- Remote monitoring
- Save time and money, especially in long time tests.

## Acknowledgement

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| Table 1: Test conditions in the Amsler type twin-disc wear tester. | |
|---|---|
| Disc dimensions | Diameter D: 42 mm, Thickness t: 10 mm |
| Sample | Substrate: cylindrical lateral area<br>Counter body: spherical lateral area (R: 30 mm) |
| Tested materials | Substrate: bearing steel (100Cr6), fine polished with a thin DLC coating<br>Counter body: bearing steel (100Cr6), fine or rough polished |
| Initial Normal loads<br>(Hertzian Pressure $P_0$) | 1,5 GPa<br>1,9 GPa<br>2,3 GPa |
| Speed | Substrate: 390 RPM; Counter body: 354 RPM |
| Tribological use | Slip-rolling friction with a sliding velocity of 0,08 m/s (10% slip) |
| Test atmosphere | Ambient air (temperature 20 °C, relative humidity 50 %) |
| Lubricant | Paraffin oil (without additives, $\eta_{20°C}$: 110-230 mPa·s), dry |

Table 2: Properties of the different DLC coatings, which were monitored using Acoustic Emission.

| Code numbers | Type of DLC coating | interlayer | Doping | Thickness of the DLC coating | modulus of elasticity E |
|---|---|---|---|---|---|
| A7 – A11 | a-C:H:Si | Si | Si | 10 µm | 161 GPa |
| B5 – B7 | a-C:H:Me | Ti, W | Ti, W | 2-3 µm | No information |
| D1 - D4 | a-C:H | Ti | No | 2 µm | 256 GPa |
| D6 - D9 | | | | 1 µm | |
| E2 - E5 | a-C:H:Me | Cr | W | 1-2 µm | 150 – 170 GPa |
| F1 – F4 | a-C:H:Si | Si | Si | 3-5 µm | 251 GPa |
| F5 – F7 | No information | | | | |
| G1, G3, G4 | a-C | Ti | No | 3 µm | 500 GPa |

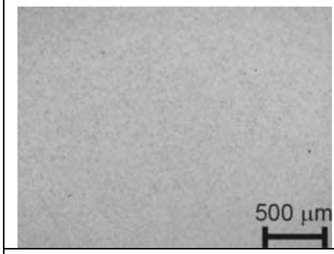
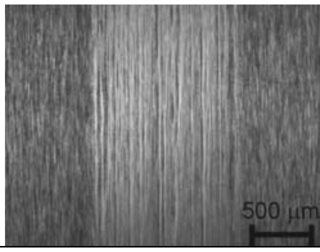
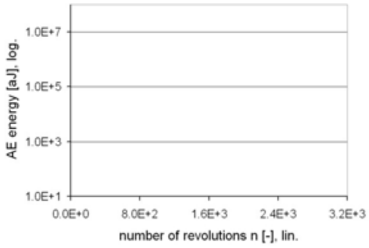
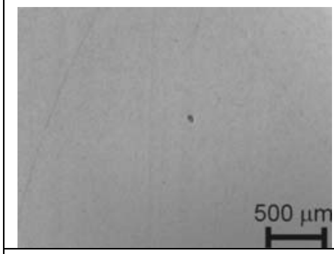
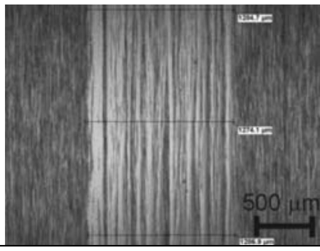
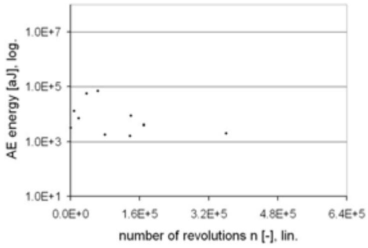
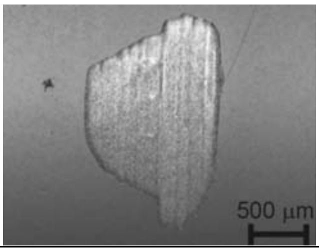
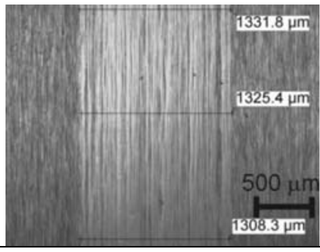
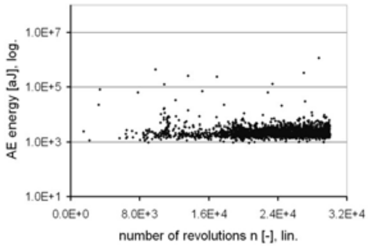
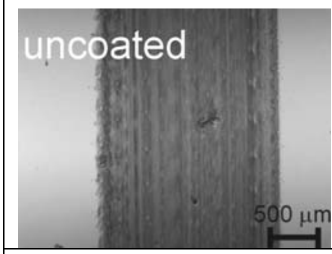
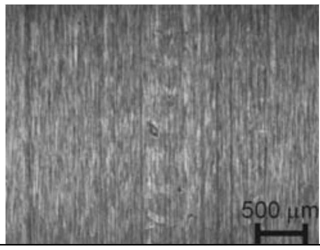
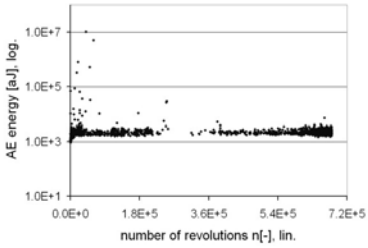
| Substrates coated | Counterbodies | AE graphs |
|---|---|---|
| a) perfect in-service<br> |  |  |
| b) in -service<br> |  |  |
| c) end-of-service<br> |  |  |
| d) out-of-service<br> |  |  |

Figure 2: AE indicated the different stages of the wear life of DLC coatings in steel contact under slip-rolling friction. The undesirable contact between steel against steel was indicated immediately by a high AE activity (base noise).

a) D2, lubricated, $P_0$= 1,9 GPa, n= 0-3.000 revolutions
b) D2, lubricated, $P_0$= 1,9 GPa, n= 615.000-1.250.000 revolutions
c) D9, lubricated, $P_0$= 1,9 GPa, n= 30.000-60.000 revolutions
d) uncoated, lubricated, $P_0$= 2,3 GPa, n= 320.000-1.000.000 revolutions

| Table 3: Configuration of the Acoustic Emission (1 dB corresponds to 1 µV). | | |
|---|---|---|
| Sensor | PAC µ30 (resonant frequency $f_R$ of about 270 kHz) | |
| Couplant | Dry:<br>ambient air | Lubricated:<br>paraffin oil |
| Preamplification PAC 2/4/6 (integrated filter: 100-1200 kHz) | 60 dB | 40 dB |
| Threshold | 45 dB | 61 dB |
| Front-end filter | 2 Counts | 3 Counts |
| AE parameter | AE energy of each hit in aJ (atto=$10^{-18}$) | |

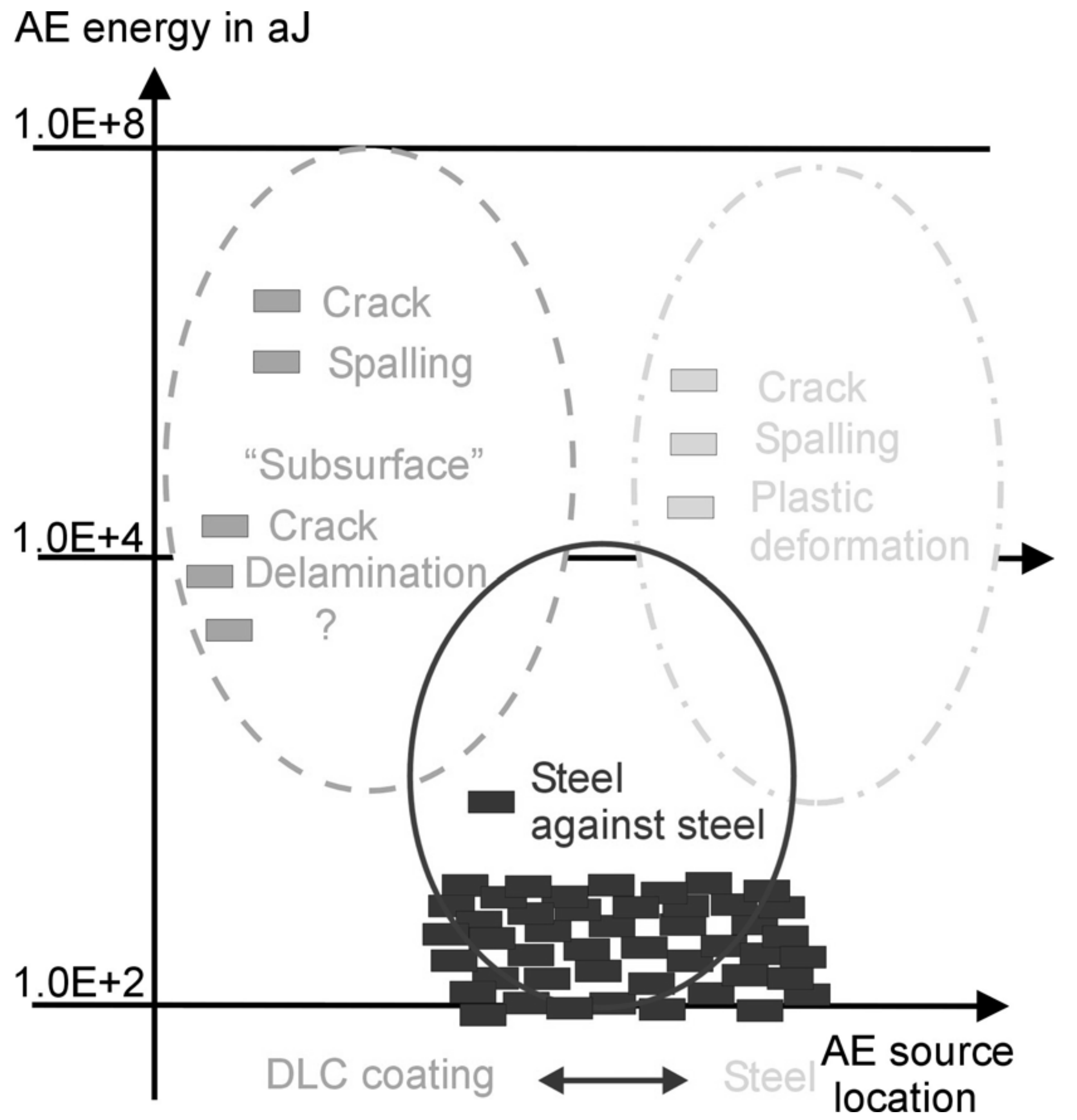



Figure 3: AE energy of hits from different sources and locations under lubricated slip-rolling friction

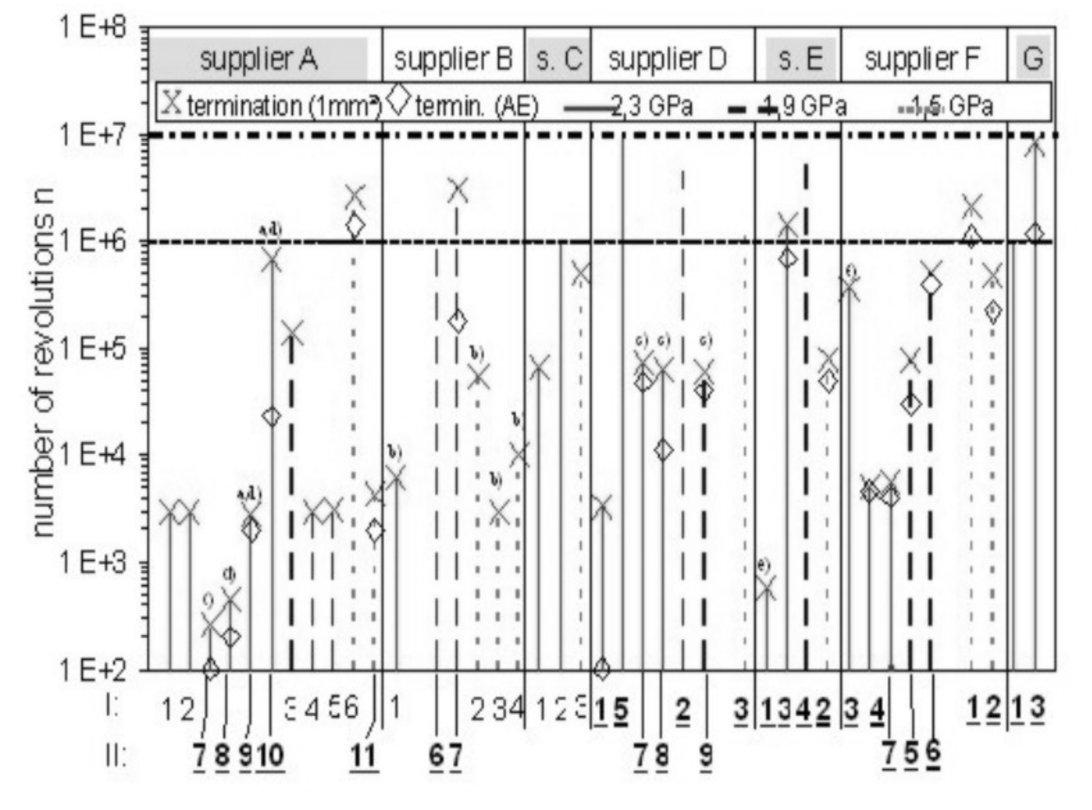



Figure 4: Results of the wear tests of the DLC coating under lubricated slip-rolling friction. Test samples monitored by AE are marked bold and are underlined. In all tests which were terminated (1 mm$^2$-criterion), high AE activities (AE criterion) were observed, too.

<table>
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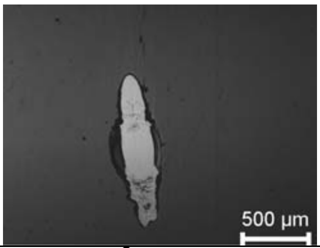
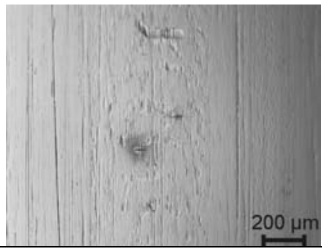
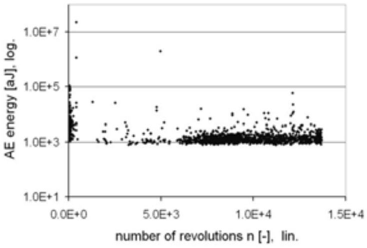
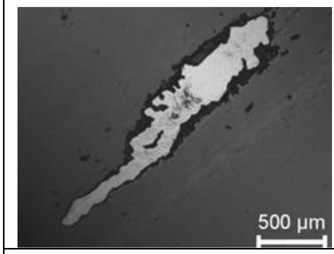
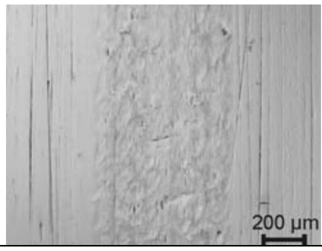
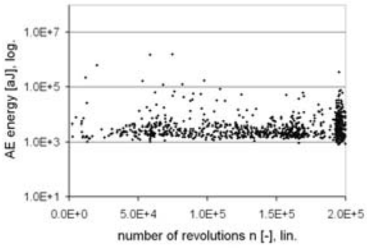
<tr><td>a) AE criterion<br><br></td><td><br></td><td><br></td></tr>
<tr><td>b) 1 mm$^2$- criterion<br><br></td><td><br></td><td><br></td></tr>
<tr><td colspan="3">Figure 5: Early detection by AE. Spallings with a size less than 1 mm$^2$ could lead to a contact between steel and steel. As result a high AE activity as a base noise indicated this undesirable tribological situation. Furthermore the edges of the spallings damaged the wear track of the counterbody plastically.<br>e) A10, lubricated, $P_0$= 2,3 GPa, n= 16.000-30.000 revolutions<br>f) A10, lubricated, $P_0$= 2,3 GPa, n= 490.000-690.000 revolutions</td></tr>
</table>

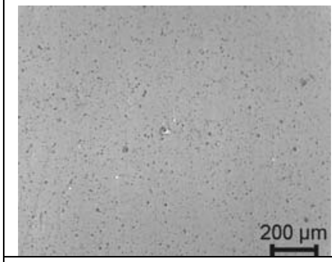
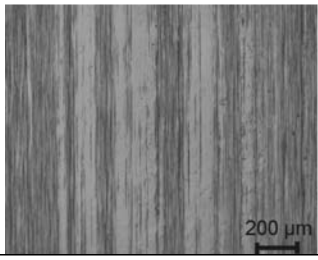
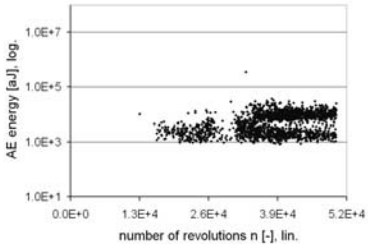
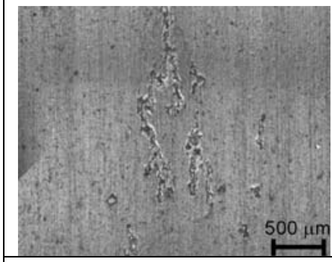
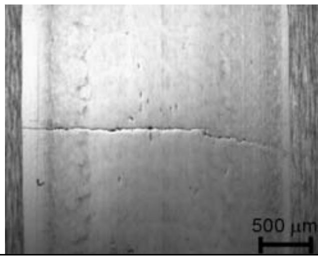
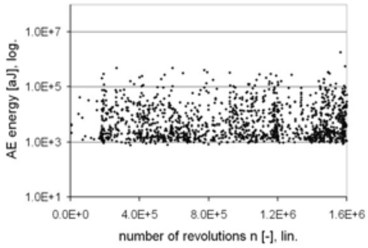
| Substrates coated | Counterbodies | AE graphs |
|---|---|---|
| a) failed: 3.000.000   |   |   |
| b) failed: 9.600.000   |   |   |

Figure 6: Early detection by AE. A high AE activity due to events "subsurface" of the DLC coatings indicated very early the reaching of the 1 $mm^2$-criterion.

g) B7, lubricated, $P_0$= 1,9 GPa, n= 150.000-200.000 revolutions
h) G3, lubricated, $P_0$= 2,3 GPa, n= 1.000.000-2.600.000 revolutions

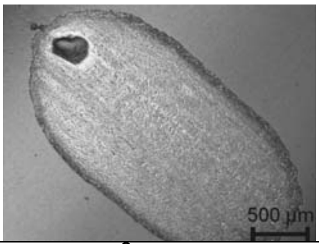
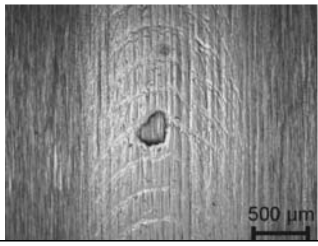
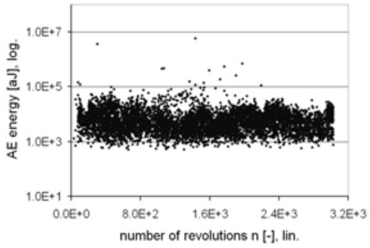
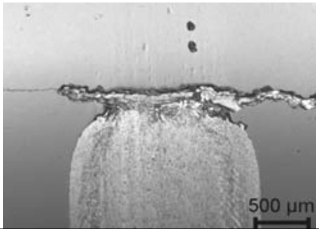
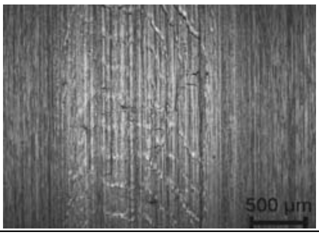
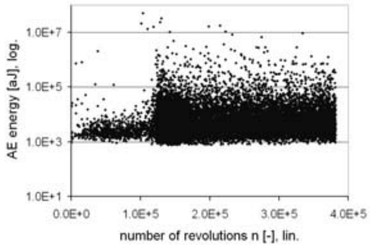
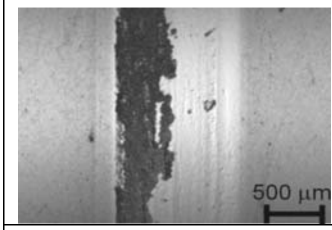
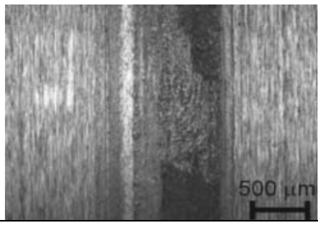
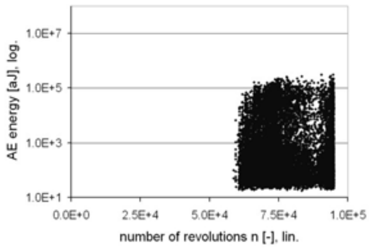
| Substrates coated | Counterbodies | AE graphs |
|---|---|---|
| a) 1 $mm^2$-criterion<br><br> | <br> | <br> |
| b) 1 $mm^2$-criterion<br><br> | <br> | <br> |
| c) 1 $mm^2$-criterion<br><br> | <br> | <br> |

Figure 7: Spallings larger than 1 $mm^2$ damaged the counterbody heavy. In addition to the base noise a powerful and intense AE activity was observed.

a) D1, lubricated, $P_0$= 2,3 GPa, n= 0-3.000 revolutions
b) F2, lubricated, $P_0$= 1,5 GPa, n= 100.000-483.000 revolutions
c) D4, dry, $P_0$= 1,5 GPa, n= 30.000-130.000 revolutions