Acoustic emission analysis of hard coatings cracking during indentation test

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Abstract. Hard coatings are widely used in industries where the good behaviours of surface properties play the main role in a life cycle of machine components. The main material characteristic of coatings which determines behaviour of surface is Hardness. In the past, Hardness of materials was considered to be the most important material property, which influences wear properties of coatings as well as value of maximal elastic tension needed for initiation of cracks. However, Adhesion of hard coatings depends on mechanical properties such as Young modulus E, hardness H and fracture toughness R. Initiation/Formation of fractures and their propagation in hard coatings is commonly investigated using indentation tests. Creation of Hard coating crack during indentation is usually detected as a wide frequency AE signal. In this article, the initiation of cracks is investigated using data measured during the indentation. The cracks formation/propagation and spalling in hard coatings during indentation were observed and quantified by using AE. AE frequency analysis was calculated from the signals measured during the indentation and it was evaluated from recorded AE signals.

1. Introduction

Basically, three main types of hard coatings cracks deposited on ductile substrate have been identified:

- circumferential cracks which appear at the periphery of plastic zone,
- channel cracks, which are initiated under big stress caused by contact between indenter and coating,
- radial cracks, which come from the middle of indentation imprint and propagate outside in the form of beams.

If the indentation loading force is high enough and the substrate is plastically deformed, the coatings start losing adhesion with surface and then delamination or spalling can occur. Based on this type of damage, it is inevitable to find an appropriate method for diagnostics of hard coatings cracking. Hard coatings cracking and spalling generate the energy which propagates through the material in the form of elastic waves, the formation of which could be detected by an acoustic emission measuring device during instrumented indentation.

When the type of hard high-performance film is designed, it is of great importance to learn the material properties of coating-substrate system. Due to size limitation of coatings the best method for evaluation of material properties of coating-substrate systems are indentation methods. Indentation is
widely used for measuring material parameters of coating-substrate systems and many studies have been
done and a lot of approaches have been developed in order to evaluate measured data from indentation
and to calculate all the main material characteristic parameters. If indentation loading force is high
enough and the substrate is plastically deformed, the coatings start losing adhesion with surface and then
subsequently delamination or spalling can occur.

Hard coatings usually have a brittle character, and a fracture failure often occurs in a hard thin film
when it is subjected to high stress and it is called cracking. This failure of coating adhesion to the
substrate usually results in interfacial debonding. One of the most important parameters of coating which
indicates its ability to remain attached to the substrate is Adhesion. At present there is no standard
test procedure for calculating adhesion of coating, but some authors published methods based on stress or
energy approach [3,8]. Authors used the data from indentation and define fracture toughness of the
coating by using calculated parameters from indentation data and they estimate critical force \( F_c \) from
indentation curve where the coating fracture is represented by pop-in. However, the estimation of critical
force from loading curves can be difficult for some coatings.

During indentation the stress activated on the border coat – substrate causes creation of cracks which
propagates through the surface. When coating breaks, the cracks initiation and propagation release the
energy, that propagates through material and can be measured and quantified with Acoustic Emission
device. The analysis of acoustic emission signals provides information of releasing this energy into
material. Creation and propagation of crack release high energy than acoustic signal exceeds threshold
and this event is recorded by AE device. The critical force \( F_c \) when the first crack of coating appears
could be determined by analysis of AE data. Then the value can be used for calculations of coating’s
adhesion parameters.

Under contact pressure by indentation, the coating undergoes elastic deformation until brittle fracture.
The substrate is deformed plastically at relatively high loads. The first crack of hard coating which
occurs under loading by indentation is circumferential crack with the radius \( c \) see figure1
When the first crack occurs, indentation force equals to the critical force \( F_c \). Using folowing equation
it’s possible to calculate the radius \( c \) of the plastic zone in the ductile substrate [7]

\[
c = \frac{3F_c}{2\pi\sigma_y}
\]  

\( F_c \) is the load carried by the substrate at the critical indentation depth at which pop-in occurs. The sign
\( \sigma_y \) is the yield stress of the ductile substrate [4,7].

2. Experimental procedure
In this study we investigate acoustic signals generated during indentation test. As an experimental device
we used materials testing device Micro-Epsilon UMZ-3k. Indenter Rockwell C was carried out by this
device. Acoustic Emission signals released during macroindentation were measured by DAKEL-ZEDO

![Figure 1. Schematic cross-section of deformation profile of a hard brittle film on an elastic-plastic substrate under indentation [4].](image-url)
Device (DAKEL). Surface cracking and spalling of the coatings after indentation test were examined by the JEOL JSM 7600 F high resolution scanning electron microscope (HRSEM).

The studied experimental sample was fabricated using PLATIT π80+DLC PVD technology. Nanocomposite hard coating nACRo based on Cr and silicon were deposited with BIAS 85V on the high-speed steel (HSS 6-5-2-5, STN19 852).

To be able to reach every possible generated acoustic signal the AE device DAKEL was more sensitive during this test what was set up by the increase of the amplification.

3. Macroindentation and AE measuring

The well-known Rockwell C indentation test is prescribed by the VDI 3198 norm, as a destructive quality test for coated compounds [1, 2]. In this study the surface has been loaded with Rockwell C indenter and the test has been executed according Daimler-Benz method (standard VDI 3198) with constantly increasing force at maximum load 50N (instrumented indentation). The applied force through indenter generates firstly elastic and then plastic deformation of sample surface. When coating breaks, the cracks initiation and propagation and spalling of coatings release high energy, which propagates through material and can be measured and quantified by Acoustic Emission device.

Indentation method - „Mercedes test“ is unpretending for execution and is frequently used often when the wear behaviour between coat and substrate is investigated. The stress on the interface coat – substrate is caused by indenter, by static indentation of indenter. The stress activated on the interface coat – substrate causes the creation of cracks which propagates through the surface. The advantage of indentation method is the fast execution of tests, minimal demand on measuring device and possibility of coating behaviour evaluation with acoustic emission monitoring directly on the researched specimens without any further destruction of specimens.

Indenters which are Rockwell cones with the top angle of 120° has been carried by a special device for mechanical behaviour measurement of materials UMZ-3K (Micro-Epsilon). This device can measure the load force in [N] and there is also possibility to adjust the speed of this movement. The Indenter movement stopped automatically when the adjusted force is reached.

Acoustic emission signals generated during the test in the specimens were measured by DAKEL-ZEDO device. DAKEL-ZEDO is modern powerful modular system for detection and evaluation of acoustic emission signals. In this case it’s used for detection of coating cracks and delamination during Mercedes test.

Many authors pointed out that it is possible to identify cracking of coating from the load-displacement curve. During the crack formation, a significant amount of energy is dissipated, which causes the discontinuity in the loading curve [3,4]. This discontinuity is called pop-in event and could be caused by different physical mechanisms. Occurrence of cracks in coating is one of more sources of this discontinuities in the loading curves [5]. In the case that we want to distinguish from the loading curve when a critical Force of first crack occurs could be a real issue. The critical force could be used for calculating of radius of the plastic zone in the ductile substrate or a fracture toughness of a hard brittle coating on the basis of the fracture pattern caused by a Rockwell indentation of the film/substrate system [6]. Also, it is possible to express if coating is susceptible for cracking by low indentation force.

The maximum reached indentation load was 50 N and has been reached without any delay in loading phase. The 50N load was selected based on the previous experiments confirming that the first acoustic signals caused by the coating breakdown occurred in the range of 10 to 20 N. For better diagnostic of acoustic signals, the amplification was increased to 96 dB. Indentation test parameter are shown in table 1.

| Max. force (N) | Holding force (N) | Holding delay (s) | Movement speed (mm/min) | Amplification (dB) |
|----------------|-------------------|-------------------|--------------------------|-------------------|
| 50             | -                 | -                 | 0.2                      | 96                |
AE parameters used for evaluation were

- **Hit** - parameter which was registered when AE signal amplitude exceeds adjusted threshold. It indicates number of all cracks and other structural instabilities which were arisen and growth in specimen.
- **Hits Energy** gives information about energy which is released into material caused by crack initiation and propagation. Bigger crack initiation causes more energy released into material. Bigger amount of energy is usually released when hard coating spalling or delaminating occur.

4. Results and discussion

The problem to find the pop-in in the loading curves is also a point in our experimental macroindentation loading curve. In the figure 2. is load-displacement curve measured by test device UMZ 3K with maximum indentation load 50N. From the load displacement curve isn’t possible to define exactly the localisation of “pop-in” neither the critical load force. It is possible to see a lot of instabilities on the curve but it’s difficult to find out when the first “pop-in” occurred.

![Figure 2. Load-displacement curve, $F_{\text{max}} = 50$ N, sample nACRo with BIAS 85V](image)

For the reason with determination of pop-ins in load-displacement curve, the acoustic signal Hits curve which represents the number of AE events (possible instabilities in the material) recorded during loading phase were added in Figure 3.

![Figure 3. Total Number of AE Hits, Indentation test at $F_{\text{max}} = 50$ N on sample nACRo with BIAS 85V](image)

In Figure 4 can be seen recorded curve of Hits0 signals which was generated from the beginning of indentation up to maximum force 50N. To every acoustic event has been assigned loading force by which it was recorded. The first accorded Hit correspond to indenter bump into surface. From this point
it is possible observe very small increasing of loading force. Another two Hits can be seen by Force $F_0 = 0.5 \, N$. Next acoustic events the system of AE detected at Forces $F_2 = 1.5 \, N$, $F_3 = 7 \, N$ and $F_4 = 11.5 \, N$. Than start a series of Hits which continuously increasing until maximum load 50 N. Because of very high hardness and very good tribological behaviour of tested coating there are just 23 recorded Hits in total. This is an excellent result and it has been confirmed by AE that this coating is not susceptible for cracking at low loads.

However, even if we use Hit parameter curve it’s not possible exactly determine the critical force of the first crack from Hit curve. There are more hits by different loading forces seen in figure 4. Because the acoustic events could be caused by different physical phenomena which happens into the materials under loading it’s not possible to identify exactly by which force was the first crack generated. From the experience we know that creation and propagation of crack cause relatively big dissipation of energy into material. When dissipated energy from crack creation is recorded then also the amplitude of recorded signal is high and of course the recorded acoustic signal energy must be high as well. In this case another parameter which is evaluated from recorded AE signals can help us to identify the pop-in that corresponds to the first crack. This parameter which can be used for quantification of events registered by system of AE is the Hit Energy and the curve recorded during indentation is shown in figure 5. Using this parameter, it is possible to determine how much energy has been dissipated into the material by crack initiation and growing.

\textbf{Figure 4.} Indentation test at $F_{\text{max}} = 50 \, N$ on sample nACRo with BIAS 85V - Total number of Hits - zoom.

\textbf{Figure 5.} Indentation test at $F_{\text{max}} = 50 \, N$ on sample nACRo with BIAS 85V - Hits Total Energy - zoom

\textbf{Figure 6.} Indentation test at $F_{\text{max}} = 50 \, N$ on sample nACRo with BIAS 85V - Picture of imprint scanned by SEM

\textbf{Figure 7.} Indentation test at $F_{\text{max}} = 50 \, N$ on sample nACRo with BIAS 85V - Visualisation of calculated substrate plastic zones c
Table 2. Calculated plastic zones $c$ with different $F_C$

| Max. force $(N)$ | Value of critical Force $(N)$ | Yield stress of ductile substrate $\sigma_y$ (GPa) | Plastic zone diameter $(\mu m)$ |
|------------------|-------------------------------|---------------------------------|-------------------------------|
| $F_1$            | 0.5                           | 35                              | 26                            |
| $F_2$            | 2.5                           | 35                              | 58                            |
| $F_3$            | 7.0                           | 35                              | 98                            |
| $F_4$            | 11.5                          | 35                              | 125                           |

In the table 2 the plastic zones are calculated according to Equation 1. From the load-displacement curve and related acoustic signal parameters is possible assume that first recorded Hit is not related to creation of crack. The first crack occurs according to Hit Energy curve in the figure 5 by the loading force $F_3 = 7 \, N$ shown in the figures 4 and 5.

After the indentation the indenter imprint was scanned by SEM. The picture from SEM of the imprint is in the figure 6. In figure 7, the calculated dimensions of substrate plastic zones $c$ are illustrated. From the figures 6 and 7, it is possible to confirm that around calculated diameter of plastic zone $c_3$ are located visible coating circumferential cracks.

5. Conclusion

In this study, the sample with nACRo coating deposited on high-speed steel (STN19 852, HSS 6-5-2-5) substrate was investigated during indentation by measuring of AE signals. According to the acoustic emission signals represented by number of hits and the energy of acoustic signals (cracks) recorded during the modified Daimler-Benz adhesion test, we can track creation of cracks in the coating. From the AE data analysis is also possible define when exactly the first crack occurs. Subsequently it is possible to assign what is the value of Force when the first crack occurs. This value is the critical force which is important for calculation of tribological and adhesion parameters of coatings.

The acoustic emission signal detection and evaluation makes it possible to evaluate:

- susceptibility of coating to untimely crack at low loading forces,
- determining of critical force when the first crack occurs.

The aim of this paper is to find a more effective and accurate methodology to determine tribological properties of hard coatings deposited on hard substrates. One of the most outstanding advantages of this methodology is very fast and precise detection of the coating’s cracking.

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References

[1] Verein Deutscher Ingenieure Normen, VDI 3198 1991VDI-Verlag, Dusseldorf
[2] The VDI 3198 indentation test evaluation of a reliable qualitative control for layered compounds, https://doi.org/10.1016/S0924-0136(03)00300-5
[3] Fu K, Tang Y, Chang L 2016 Toughness Assessment And Fracture Mechanism Of Brittle Thin Films Under Nano-indentation Fracture Mechanics - Properties, Patterns and Behaviours, Ed. Lucas Alves, IntechOpen doi: 10.5772/64117
[4] Mercier D 2018 Pop-In Documentation
[5] Navamathavan R et al. 2006 Mater. Chem. Phys. 99 410–413
[6] Krabbe M 2014 Fracture Toughness Of Thin Films Estimated By Rockwell C Indentation Phd Dissertation Thesis, Aarhus University, Department of Engineering, Denmark, ISBN 978-87-93237-27-8
[7] Bahr D F, Woodcock C L, Pang M, Weaver K D, Moody N R 2003 Indentation induced film fracture in hard film – soft substrate systems *Int. J. Fract.* **119/120** 339–349

[8] Malzbender J, Den Toonder J M J, Balkenende A R, De With G 2002 Measuring mechanical properties of coatings *Mater. Sci. Eng.* **R36** 47-103