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Analysis in real time of isolated cracks within a coating

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Physical vapour deposited coatings have a complex structure, and there is a need to characterize the failure of such coatings and this has been carried out by a non-destructive method, namely acoustic emission. In this paper simple scratch tests are described in order to study some characteristics of nitrogen-doped stainless steel coatings on 40CrMo4 constructions steel. Scratch tests were performed in order to induce a mechanical failure in the coatings. It is shown that the nucleation of isolated cracks mechanism that lead to failure can be monitored by certain characteristics of the acoustic signal.

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1. Introduction

The development of coatings deposition techniques during the last years has offered new possibilities to improve the tribological properties of surfaces in sliding contact [1, 2]. Metallic, as well as ceramic coatings [3, 4], are used in order to provide better tribological properties to bulk materials. The choice of a coating is often made on the basis of high hardness in order to give better wear properties. But the higher the hardness of a coating is, the more brittle it becomes. The spalling tendency of coatings is caused by the initiation and propagation of cracks [5, 6, 7]. Therefore, it becomes important to understand the mechanisms of crack initiation and propagation in metallic and ceramic coatings. It is well known that the scratch test is a useful method for this purpose. Cracks within a coating may have various shapes, distributions and/or morphology. They can be parallel or perpendicular to the substrate surface (Fig. 1) [8]. In the first case, the cracks can lead to loss of the coating by spallation when fragments of material are ejected due to impact or stress process. The failure process is most severe at the bond coating-substrate interface and the crack most commonly occurs within the coating propagating near to the highly stressed regions of the bond coat asperities [5, 8-10]. In the second case, the perpendicular cracks may not necessarily contribute to removal of the coating and also may have the beneficial effect of arresting the cracks before they cause substantial damage to the overall coating integrity [11].

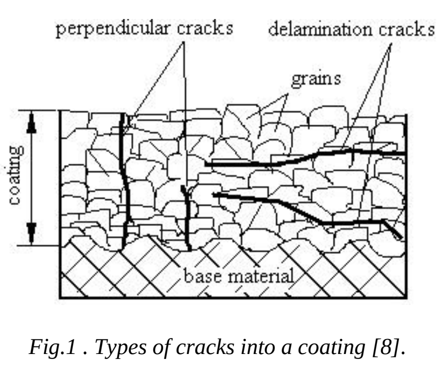

Fig.1 . Types of cracks into a coating [8].

A major difficulty is to assess the severity of any cracking within the coating. Acoustic emission (AE) methodology lends itself to qualitative measurements of crack nucleation activity. It also has the advantage of being a passive (non-interfering) technique, i.e. measurements can be carried out "on line" during the test period and, finally, they may become quantitative [12-14].

Adhesion is an extremely important property of coatings and the scratch test is widely used as an indicator of adhesion strength. Spalling and buckling failures of coatings occur as a result of compressive stress fields ahead of the moving stylus. This leads to crack nucleation at the edge of the scratch channel. In conformal cracking, semi-circular cracks occur within the scratch tracks only ahead of the indenter. Tensile cracking is similar to conformal cracking. However, the semi-circular cracks appear behind the indenter. Spalling results from complete delaminating while buckling is due to partial coating delaminating at a certain distance from stylus. A practical approach for the coatings study is to consider certain structural characteristics of sandwich substrate-coating as it is shown in Fig. 2 [15].

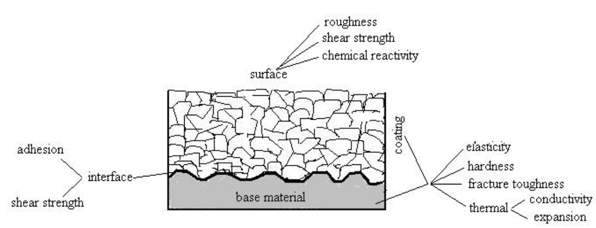

Fig. 2. Some properties of different zones of the surface, coating, interface and substrate.

The basic principle of acoustic emission (AE) methodology is that crack nucleation release elastic strain energy which then propagates throughout the material and influences the response of a transducer [12]. A piezoelectric transducer having resonance frequency of 150 kHz was used in the case of this particular experiment.

The characterisation of a noise source is important in both AE detection and AE analysis. The detection process of the AE signal can be perturbed due to some vibrations which appear in experimental set-up during scratch test. These vibrations can interfere and the resulted pulse will have a certain magnitude. This pulse has peaks that, at low thresholds, produce false triggers and the interference contributes to the steady state noise that largely affects the baseline signal in a transient wave. A typical AE device is equipped with so called 150 kHz sensors having a band pass of 100-300 kHz. The 150 kHz piezoelectric serves to isolate the signals with low frequency associated to machinery noise (threshold interference) and those with high frequency due to electrical noise (steady state). The waves measured by the 150 kHz sensor have a low enough frequency. That is why signals can still be detected in both low and medium damped materials.

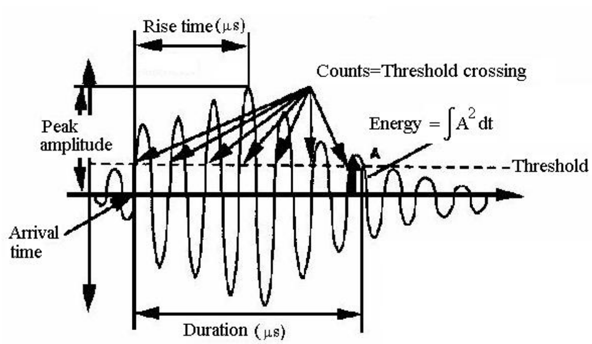

Fig. 3. AE Signal Characteristic Features [12].

For a rapid and continuous detection of signals in the AE frequency regime, the typical parameters of signal are measured in real time. In Fig. 3 are presented the main parameters of the acoustic emission signal. They are: signal duration and time to peak (rise time), peak amplitude, number of threshold crossing and integrated energy. The set of AE parameters has practical significance for experiments [12].

2. Experimental procedure

Experiments were performed at "Laboratoire de Science et Génie des Surfaces", Ecole des Mines, Nancy, France. The scratch tests were carried out using a semi-automatic LSRH-Revêtest scratch tester provided by CSEM Swiss Center for Electronics and Microtechnology, Inc., Neuchâtel, Switzerland equipped with a standard off-line Zeiss metallographic microscope (magnification: ×200). Fig. 4 shows the experimental set-up.

Fig. 4. The experimental set-up.

A complete digitalised, state-of-the-art, Vallen AMSY4 AE system, provided by Vallen-Systeme GmbH Company, Icking, Munich, Germany that includes a personal computer was used in conjunction with a piezo-electric sensor DECI model SE 150-M (delivered of Dunegan Engineering Corporation Company Inc.) that operates in the resonance mode. The sensor was mounted on the indenter shaft of the scratch tester (Fig. 5). The indenter shaft of the scratch tester was equipped with a standard Rockwell-C diamond stylus of 0.2 mm radius, supplied by Moessner GmbH Company, Pforzheim, Germany.

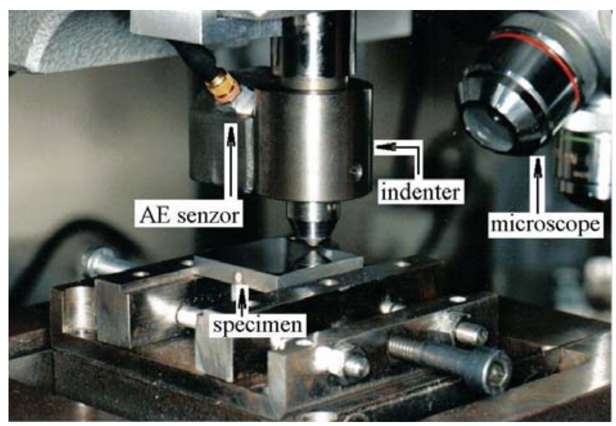

Fig. 5. LSRH-Revêtest scratch tester with the acoustic sensor on the indentation shaft The micrograph of a scratching test.

The technical characteristics of LSRH-Revêtest scratch tester are:

- Normal load: 0÷50 N.
- The loading rate: 100 N/min.
- The sliding speed of the horizontal specimen mounting table: 10 mm/min.
- The maximum sliding distance: 10 mm.

Specimens with nitrogen-doped stainless steel coatings obtained by PVD method of coatings having a thickness of 12 μm on 34CrMo4 steel substrate were investigated. In order to get isolated cracks in the coating, simple mechanical tests were performed.

First of all, static indentation was performed until a load of 50 N. A ring crack (Fig. 6) opened at a certain value of normal load. It was very important to know precisely the value of the load when the crack appeared. This is why the acoustic emission is an extremely powerful method of investigation. The electronic impulse generated by the crack opening was transformed into an acoustic signal by the piezo-electric sensor. This signal, in its turn, was picked up by the Vallen Systeme and shown up on the computer monitor as an individual train of waves with particular characteristics.

Fig. 6. Circular indentation crack.

Secondly, a scratch test was performed with uniform increasing normal load, up to 50 N, in order to determine the critical normal load for crack opening during sliding contact. The critical load corresponding to crack opening was possible to be determined, again, with acoustic emission signal monitoring.

Finally, a scratch test was performed with constant normal load equals with the critical load previously determined. This test was the one giving important information about the coating's practical adherence [16-18].

3. Results and discussion

Five different groups of nitrogen-doped stainless steel coatings deposed on 40CrMo4 constructions steel were tested. Table 1 shows concentrations of nitrogen in the coatings, the brittleness of the specimens, and the coating failure after the scratch test in each case.

Table 1. Characteristics of the tested specimens and failure of the coating after the scratch test.

Sample of RO 752 steel coated with nitrogen steel	Hardness of the surface	Concentration % of N_2	Coating failures
S1	800 HV	5	No cracks, perfect adherence with the substrate, the coating was "folded" at the end of the scratch.
S2	900 HV	7	Single cracks easy to identify with AE and optical microscopy. Critical normal load F_c = 38 N
S3	1000 HV	9	Perfect adherence with the substrate, no "folding" at the end of the scratch.
S4	1100 HV	10	Several failure modes: spallation, chipping, cracks, impossible to be separated by AE.
S5	1200 HV	11	Several single cracks and delaminations very closed one with another difficult to separate them with AE.

The experimental procedure for the scratch tests was as follows:

- First step: application on coatings of a uniform increasing normal load from 0 to 50 N. The normal load corresponding to appearance of the first crack was considered as critical load, F_c.
- Second step: application on coatings of a constant normal load having different values as (100 %F_c, 80 %F_c, 60 %F_c).

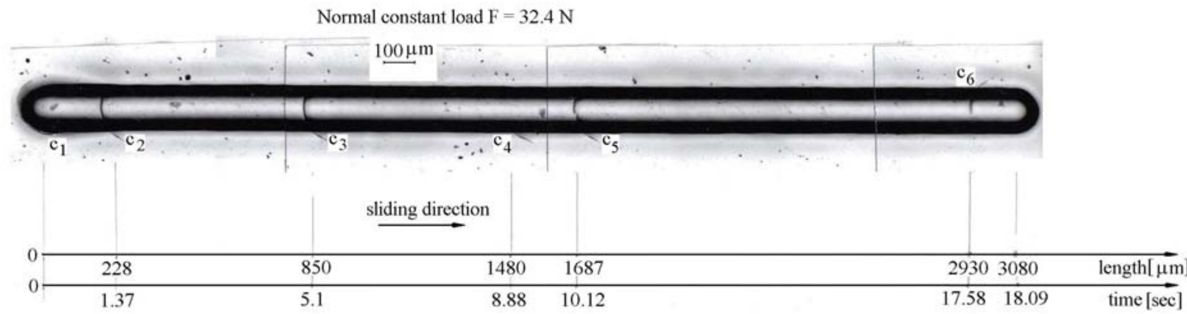

Fig. 7. Micrograph of a scratching trace.

After each and every test a complete observation under the microscope was made. Perfect post synchronization with the off-line micrograph of the cracks was achieved. The acoustic signal generated by each cracking event was characterised by the complete acoustic parameter set as well as the wave trains.

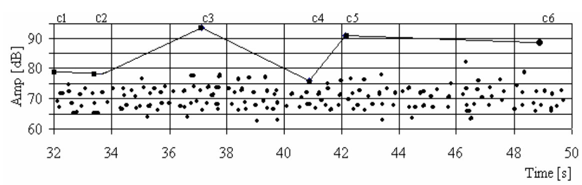

Fig. 8. AE signal corresponding to the scratch test from fig. 7.

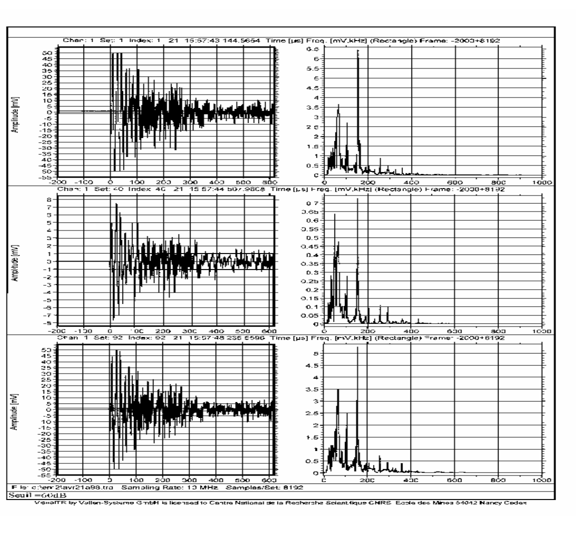

Fig. 9. Wave trains corresponding to the cracks c_1, c_2, c_3 shown in fig.7.

Fig. 7 presents the microscopic image of a 3 millimetres long trace on the nitrogen-doped stainless steel coating, for specimen S3. Isolated cracks were denoted c_1, c_2, c_3, c_4, c_5, and c_6. The microscopic images were taken on an Olympus Vanox AHMT 3 system with Normarsky contrast enhancement or by a scanning electron microscope (Philips SEM 505). Below the micrograph is the plot in real time of amplitude peaks (Fig. 8). The amplitude peaks of the acoustic signal correspond to each crack mentioned above. In Fig. 9 the plots of acoustic signal emitted during appearance of three of the above mentioned cracks are displayed. In the left column represent the variation of the electronic impulse amplitude versus time, while in the right column is presented the dependence of frequency of the emitted acoustic signal versus time.

Based on the analysis of the acoustic emitted signal due to appearance of cracks, five parameters were noticed in order to identify an isolated crack event in the coating:

1. The threshold value is 60 dB, a lower limit can be considered white noise.
2. The rise time is smaller than 60 µs, and corresponds to instantaneous release of energy of the crack trigger.
3. The length of the acoustic impulse is under 600 µs.
4. There is a time window before the first trigger, called „pre-trigger” time, of 200 µs where another threshold is noticed at 0.17 dB.
5. The pre-trigger rise time is maximum 5 µs.

5. Conclusions

A crack that appears in a coating of a substrate is dangerous. A lot of human lives can be saved with a device that can pick up, in real time, the very moment of a crack nucleation and let the supervisor know about it.

This paper presents a set of five parameters that were found out by experimental method, that make the difference between a single crack nucleated in a coating and the “white noise”. Every isolated crack, regardless the size, has the same acoustic “signature”. The parameters set can be used for building a “crack alarm” device.

The isolated cracks were induced in nitrogen-stainless steel coating deposited by PVD method on a construction steel substrate. The method of crack inducing was the scratch test, accompanied by measurement of acoustic emission signal during appearance of each crack. After each and every test, a complete analyze by microscope was done. The goal was to determine, in real time, the precise moment of the crack trigger, the corresponding normal load, and the AE main parameters. The stainless steel coatings with various concentrations of nitrogen had different behaviour during scratch test. Regarding the coating behaviour, the coating with 9 % nitrogen and surface hardness 1000 HV had the best adhesion to at the substrate, no cracks or other type of failure appeared while 10 % and 11 % nitrogen had multiple failure types. Single cracks were obtained on the specimen with 7 %nitrogen.

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