ACOUSTIC EMISSION TECHNIQUE TO ASSESS MICROFRACTURES OF METALIC COATINGS WITH SCRATCH-TESTS

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ABSTRACT

The adherence of TiN and CrN films on steel substrates is analysed by means of the Acoustic Emission (AE) technique in scratch experiments. A first study shows a clear difference in the adherence behaviour of both kind of coatings: TiN is ductile and CrN is brittle. In the first case, four stages can be detected in the fracture process of the coating with the AE technique, corroborated by scanning electron microscope observations. Thus, the AE technique shows a high potential in the evaluation of adherence of coatings on metallic substrates.

INTRODUCTION

Acoustic Emission, (AE), is an important tool for the detection and characterisation of failures in the framework of non-destructive testing (NDE) [1-2].

In a previous paper [3] some of the authors analysed the detection of a TiN coating on a stainless steel sample. The analysed AE signal was obtained by a scratching test designed for adherence evaluation. The signal was treated in the frame of 1/f stochastic processes and harmonic analysis method, showing promising results. But more systematic experimental work is necessary. In this paper, seven samples with different coatings were analysed, and emphasis was put in the comparison between different stages in the scratching process, with the AE technique during the experiment and the after test scanning electron microscope observation. In the meantime, some other papers involving scratch tests appeared in the literature [4-7], but the subject is far from being concluded.

The organisation of the paper is as follows: First the AE technique is described. Then we describe procedure to obtain the TiN and CrN coatings on steel samples. In the next section the scratch test is described in detail. A thorough comparison between several AE parameters varying along the scratch test and the after test scanning electron microscope images follows. This permits to obtain encouraging results and conclusions.

THE ACOUSTIC EMISSION TECHNIQUE

Acoustic emissions (AE) are the stress waves generated by the sudden internal stress redistribution in materials or structures when changes in their internal structure are produced. Possible sources of AE can be: crack initiation and growth, crack opening and closure, deformation, dislocation movement, void formation, interfacial failure, corrosion, fibre-matrix de-bonding in composites, etc. These waves propagate through the material and eventually reach the surface, producing so small temporary surface displacements. Usually the stress waves are of low amplitude and of high frequency (normally, ultrasonic). This is the reason why very sensitive piezoelectric transducers (sensors) are required to capture them. Due to the low amplitude of AE waves, several steps must be sequentially incorporated after their capture and before the subsequent recording and analysis. A preamplifier is necessary to minimise the interference and prevent the signal loss, a filter to remove the noise and an finally an amplifier. Figure 1 shows a block diagram of this procedure.

Commonly, the sources of AE are related with the damage in the material. Thus, its detection and analysis can be used to evaluate the dynamical behaviour of the material, and so, to predict its failure. In fact, this procedure is a nondestructive evaluation (NDE) technique of materials and structures, the so called "Acoustic Emission Technique". The main difference with others NDE's methods (X-Ray, Ultrasounds, Radar, etc.) is its capability to detect in real time changes occurring inside the materials. Thus, by using an AE system, we can in-situ continuously monitor the start and progression of the damage of the specimen or structure, even when this is not possible by means of visual inspection. However, this technique is not free of some disadvantages which must be overcome in the future: limitations to carry out quantitative analysis, the unavoidable presence of noise, signal weakness, etc. Nevertheless, recent theoretical results and advances in electronics are allowing the high development of this technique for its use as a NDE method.

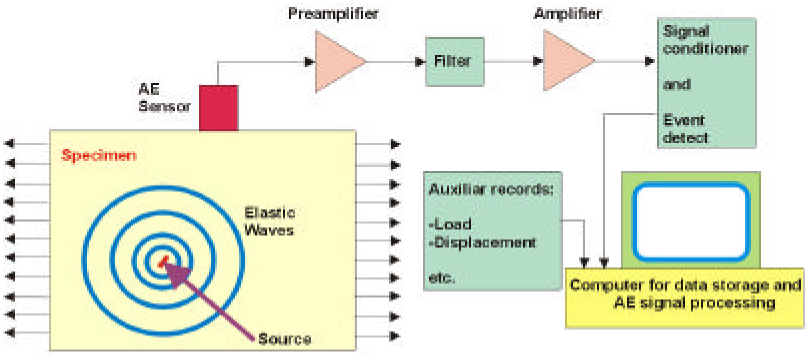

Figure 1: Acoustic Emission Technique

Keeping in mind the way to analyse the wave stress recorded with the sensors, the AE can be divided into two types. The historically first one, called "Non Quantitative Acoustic Emission" or "Traditional Acoustic-Emission" technique, is based on the obtaining of some parameters of the recorded signals and their statistical study along time. Contrarily, the "Quantitative Acoustic Emission" takes into account the source-function and the wave propagation inside the material and/or a signal processing of the recorded data by means of high-level techniques. Obviously, the second category of AE technique is more difficult to implement both in theory and practice, but allows a higher quality information. In this paper we use only traditional AE. More concretely, this type of analysis is based on the assumption that the elastic energy is emitted as a sequence of events as is shown in Figure 2, later on in the paper. Each event, detected when the AE signal overpasses a threshold, can be defined with some parameters: Amplitude (peak of the signal, normally measured in volts), duration (time between the first and the last cross of the threshold), number of counts (number of times the signal rises and crosses this threshold), energy (area under the envelope of the AE events) and rise-time (time between the first cross of the threshold and the peak). Moreover, the number of AE events and the total elastic energy recorded (accumulated along the load history) are parameters highly used to evaluate the AE activity into the material.

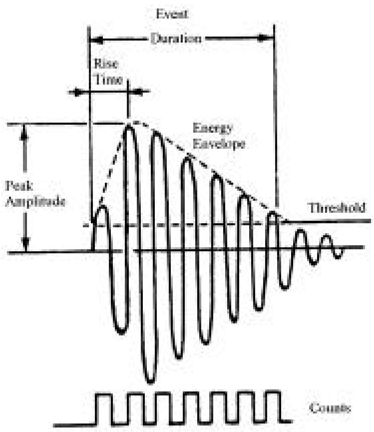

Figure 2: Traditional parameters of a AE event

MATERIAL

A number of seven samples were cut from a hot laminated slab and then polished. All samples were submitted to plasma nitriding in a process characterised by the following parameters: Temperature: 480 °C; Duration: 20 h; Gas composition: 25% N_2, 75% H_2; Total Pressure: 7 hPa; Pre-treatment: sputtering de-passivation during 2 h at a pressure of 2 hPa, in a gas composition of 50% Ar %50% H_2; The nitride layers depth was in all cases between 50 and 60 µm;

Sample	Coating	Re-polished prior coating
A	CrN	Yes
B	CrN	Yes
C	CrN	No
D	CrN	Yes
E	TiN	Yes
F	TiN	No
G	TiN	Yes

Table 1: Samples analysed

Five of the samples were re-polished. All the samples were then coated by PVD. The coating was either TiN or CrN. In this second deposition the layer depth was of some microns. The samples were nitrided by IONAR S.A. (Buenos Aires, Argentina) using industrial equipment. Details are given in Table 1.

EXPERIMENTAL METHOD

Scratch tests were performed under controlled conditions with a device that consisted of a loaded probe with a diamond indenter moving linearly along the sample with a constant speed and continuously increasing force. The steadily increasing contact load causes tensile stress behind the indenter tip (trailing edge) and compressive stress ahead of the cutting tip (leading edge). The detection system used was MISTRAS 2001 from Physical Acoustics Corporation (PAC). The piezoelectric sensor was located with a coupling wax on the topside of the sample holder. Then signals passed through preamplifiers (40 dB) and were measured by the AEDSP-32/16B card that has two channels for signal processing and wave shape detection. The parameters were: Pretrigger: 20 microseconds; Gain: 45 dB; Sampling Frequency: 4 Msamples/s; Duration: 2.2 s; Maximum load: 10 Kg. A scheme of the experiment is given in Figure 3. The breakdown of the coatings was determined both by AE signal analysis and optical and scanning electron microscopy. AE permits an earlier detection, because the shear stress is a maximum at certain depth beneath the surface, where a subsurface crack starts. Moreover with these coatings, the first cracks are so small that they are difficult to detect even under the microscope. They may even close-up within a few milliseconds and thus become optically "undetectable".

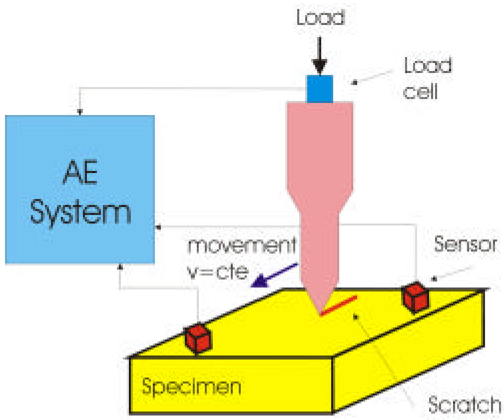

Figure 3: Scratch-test scheme

EXPERIMENTAL RESULTS

As mentioned previously, several samples covered with two kinds of coatings have been tested. Figure 4 shows an optical microscope image where we can see the four scratches carried out on sample E, with a TiN coating. In all cases, the duration of the experiment was 135 s and the real length of the scratch is 1.02 cm. For comparison, we show in Figure 5 a similar image corresponding to sample D, covered with CrN. A clear difference can be observed between both films behaviour: Whereas the TiN film breaks gradually, the CrN film suddenly breaks. Thus, we can conclude that the first one is a ductile coating and the second one is clearly a brittle one. This behaviour is also observed in the other samples, both with TiN and CrN. However, a more exhaustive analysis is necessary in order to better understand the fracture process and its acoustic emissions. For this purpose, Figure 6 shows a spatial sequential image obtained by electronic microscopy of the first scratch performed on sample E (TiN). In this picture, a scale graduated in ten per cent showing the position on the scratch has been incorporated to help in the interpretation of AE results.

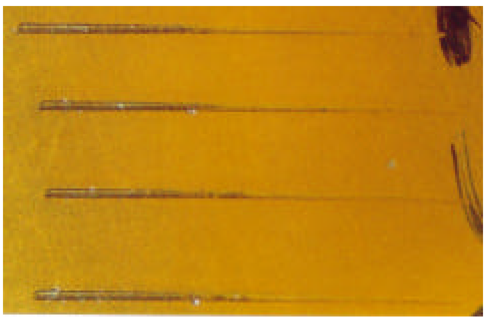

Figure 4: Scratches on sample E (TiN)
(Optical microscope)

Figure 5: Scratches on sample D (CrN)
(Optical microscope)

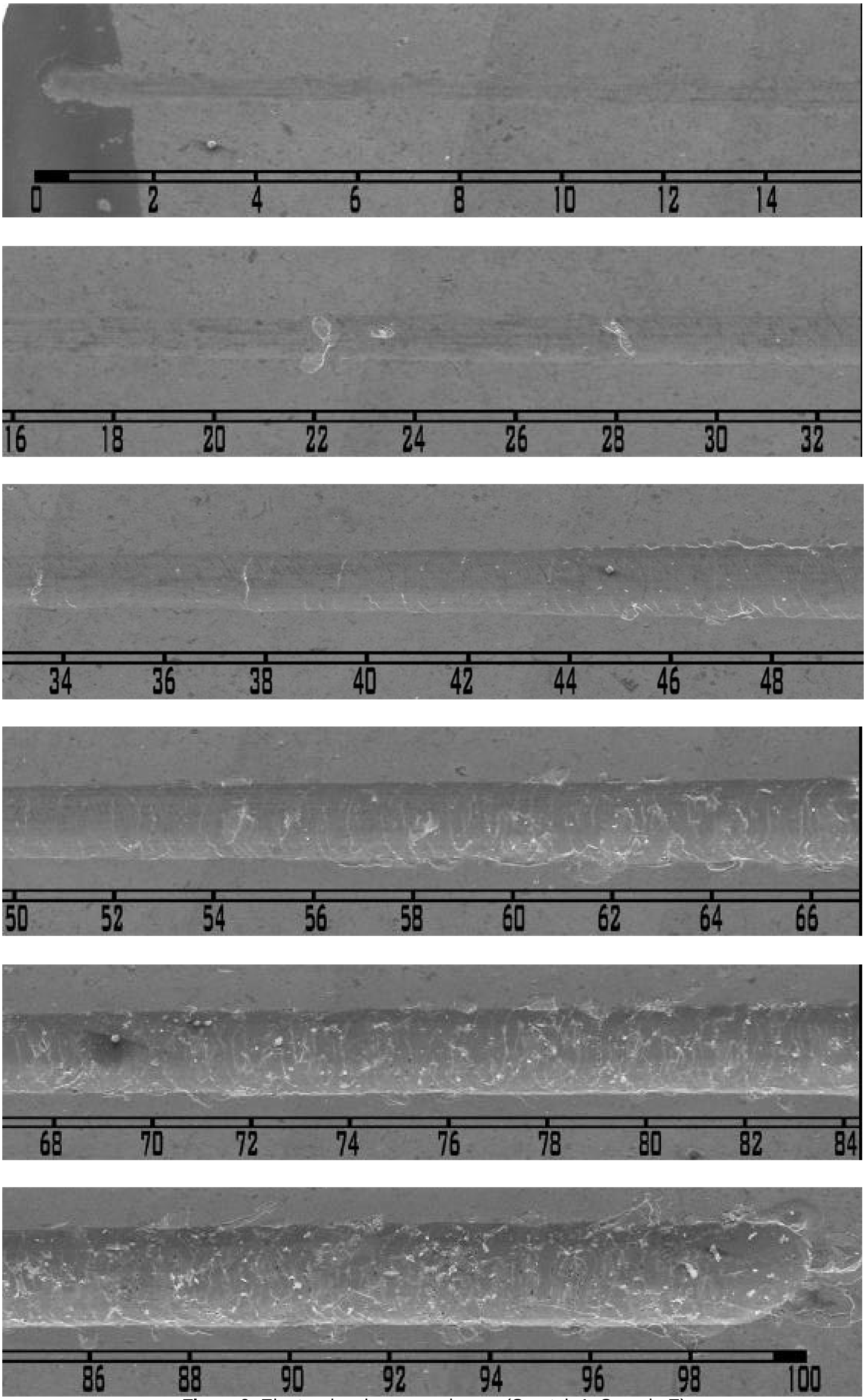

Figure 6: Electronic microscopy image (Scratch 1, Sample E)

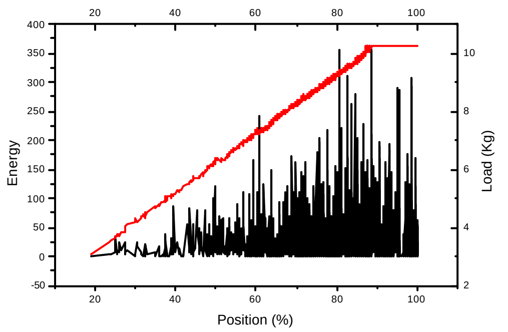

Figure 7: Energy of the AE events (Scratch 1, Sample E)

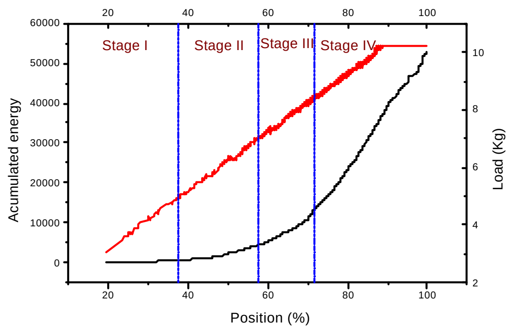

Figure 8: Accumulated energy of the AE events (Scratch 1, Sample E)

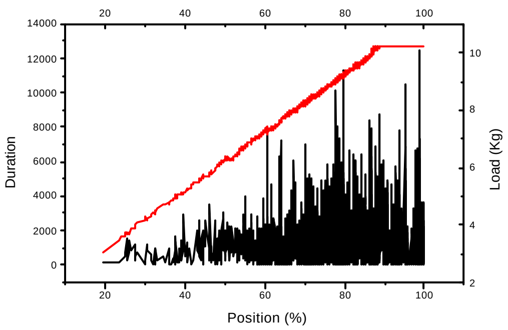

Figure 9 : Duration of the AE events (Scratch 1, Sample E)

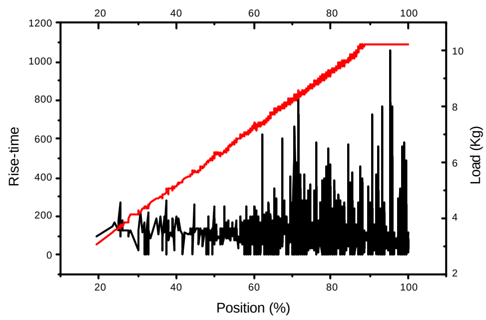

Figure 10: Rise-time of the AE events (Scratch 1, Sample E)

The AE results obtained from one sensor and from the same scratch, are shown in Figures 8-10, where we are representing the energy of events, the accumulated energy, the duration and the rise-time versus the spatial position in %, which is proportional to time. The results correspond to the first scratch of the sample E of Figure 4. We have also obtained the results of AE for the others scratches and samples. However, for shortness, we present here only one of them. These figures show also the load history. First, we can see in Figures 8 and 9 that the first emissions appear about 25%. However, it is not until the 37% when these emissions have a significant energy. It is just at this point where the first transversal microfracture of the film appears (see Figure 6). Thus, we can establish a first stage in which a very-low AE value is observed, because the film is not broken yet or very weak microfractures are produced. In Figures 8 and 9 we can see that the accumulated energy in this stage is flat and the events are very short. Continuing from here, some transversal microfractures appear on the film until approximately the 58% point. A new kind of fracture is presented in this second stage (37%-58%). We can see that the energy and duration of the events is higher than in the previous stage. Moreover, the accumulated energy slowly increases along the scratch. So, we can associate these AE events to emissions produced by transversal microfractures. At the 58% position, an increase of both parameters (energy and duration) is clearly observed in Figures 7 and 9. Similarly, the accumulated energy (Figure 8) increases its rate in a non-linear form until position 72%, approximately. At his point the slope increases strongly and remains constant until the end of the scratch. So, two new stages can be established: Stage III: 58-72% and Stage IV: 72-100%. At Stage III, we can see in Figure 6 that the defects generated by the film detachment (debonding of TiN-steel the interface) are added to transversal microfractures (which continue appearing). Thus, two kinds of fractures generate AE events, which can not be easily separated. Even the change between both stages III and IV is not very clear in Figure 6, an increase of the fracture density (film transversal microfractures and film-substrate detachments) can be observed, showing a very high breakage degree of the film. Finally, from Figure 10 an interesting result can help us to establish the separation between these two stages, because a clear change is appreciated in the rise-time values.

CONCLUSIONS

Stage	Microfractures (with microscopy)	AE Accumulative Energy	Rise-time AE
I (0-37%)	Any or few	Few important	Short
II (37-58%)	Transversal (film)	Slow increase	Short
III (58-72%)	Transversal (film) Debonding (film-substrate)	Non-linear increase	Short
IV (72-100%)	Transversal (film) De-bonding (film-substrate) (High density)	High slope linear increase	High

Table 2: Stages for the sample E (TiN). AE and microfractures.

The surface acoustic waves captured by means of piezoelectric sensors has shown to be a promising method for characterising the adherence of thin films on steel substrates by means of scratch experiments with controlled load. Defects in both the film (transversal microfractures) and the interface film-steel (debonding) originate the failure of the coating. For the case of a ductile film as TiN, using the typical parameters of the non-quantitative AE technique and comparing them with the scanning images, we have demonstrated the existence of four stages. At the first stage, the absence of AE informs us that there are not yet important adherence problems (some or only a few fractures). In the second stage, some film transversal fractures conduce to low levels of AE energy liberation. The third stage is characterised by the presence of both types of fractures, showing thus a loose of film-substrate adherence, manifested by AE: high values of duration and energy of events, a clear non-linearity of the accumulated energy, and low values of rise-time. Finally, at the end of the experiment an increase of the density of fractures is traduced as a change of slope of the accumulated energy with very high values of rise-time. However, this analysis does not yet allow us to separate the events generated by both kinds of fractures in stages III and IV. As a summary, Table 2 shows these four stages for TiN. This is the reason why more research must be done in the near future. Moreover, we have shown by means of optical microscopy images the clear difference of the fracture pattern between TiN (ductile fracture) and CrN (brittle fracture). For shortness, we have not presented in this paper the AE for this second kind of film.

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