STRENGTH ANALYSIS OF NITRIDED LAYERS ON AUSTENITIC STAINLESS STEEL BASED ON ACOUSTIC EMISSION (AE) MEASUREMENTS

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Summary

The aim of this work was to characterize deformation and failure mechanisms of layers formed during nitriding on austenitic stainless steel. The characterization was based on acoustic emission measurements performed during the static tensile test. The experimental work included the plasma nitriding process of stainless steel grade 304 samples, microhardess measurements, microstructural investigation and XRD phase analysis. The main investigation technique used was the acoustic emission measurement. The acoustic characteristics - RMS voltage, and energy, have turned out to be especially useful, as they enable to determine the real yield limit, the exact stress-strain conditions of fracture initiation at the nitrided surface and the dynamics of the fracture progress.

1. INTRODUCTION

Austenitic stainless steels are broadly used in the industry, as they are well known for their excellent corrosion resistance. However, their applicability is often constraint by their relative low mechanical properties, in particular, low hardness (< 215HB) and wear resistance. These properties can be improved by various surface hardening techniques. The Ion nitriding process has been shown in many publications as an effective solution for improving the surface characteristics of stainless steels.

The chemical and phase composition of nitrided layers formed on austenitic stainless steels differs much from typical nitrided layers that are formed on ferritic tool steels. For this reason detailed studies are required in order to characterize their properties and find optimal processing parameters.

It has been shown that the most beneficial surface properties can be obtained by low temperature plasma nitriding (below 500˚C). Stainless steels processed in this way show a significant increase in hardness while keeping their desirable corrosion resistance. This improvement has been attributed to the formation of the so called, single „m-phase" zone at the surface. The „m-phase" is a metastable iron nitride phase with a tetragonal body centred (TBC) crystallographic structure.

This paper focuses on the phenomena taking place during plastic deformation of the material. The main investigation method used was the acoustic emission measurement. This method allows registering *in situ* signals generated by microstructural phenomena taking place in the material under applied load. In a stressed material internal energy is stored, which under certain circumstances is being released by local phenomena (e.g. micro crack propagation, dislocations movement), so called AE origins. That energy generates transient acoustic waves propagating through the material and can be registered by acoustic sensors. Detailed analysis of these signals allows describing the phenomena taking place in the stressed material. The goal of this work was therefore:

- to describe plastic deformation and cracking mechanisms of nitrided layers in correlation with the behaviour of the bulk material
- to define load conditions leading to failure of the nitrided layers

2. EXPERIMENTAL

The base material was AISI type 304 austenitic stainless steel. Flat tensile test samples with the geometry shown in Fig.1 have been laser cut from sheet. The samples have been shot-peened with glass beads. Ion sputtering has been applied to clean and activate the surface. A conventional DC ion nitriding process has been used with parameters shown in Tab.1. The cooling has taken place under vacuum.

The thickness of nitrided layers has been measured on metallographic cross sections. Vickers microhardess tests of the surface have been carried out at 0.2 kgf load (HV0.2) and 15 s indentation time. Qualitative XRD phase analysis has been performed on the Philips PW 1140 diffractometer equipped with a cobalt X-ray source with a wave length CoK_1 equal to 0,178897 nm. The static tensile test has been run on Zwick/Roell Z005 universal testing machine at a test speed of 1.5 mm/min.

Table 1. Nitriding process parameters of AISI type 304 stainless steel

Sample designation	Process	Number of samples	Temperature [°C]	Time [h]	Processing atmosphere	
					Gas proportion (vol.)	Pressure [hPa]
SW	raw sample	3	-	-	-	-
-	ion sputtering	-	550	1	$Ar:H_2$ – 1:4	1.5
430_2h	nitriding	3	430	2	$N_2:H_2$ – 1:1	2.5
430_8h	nitriding	3	430	8		

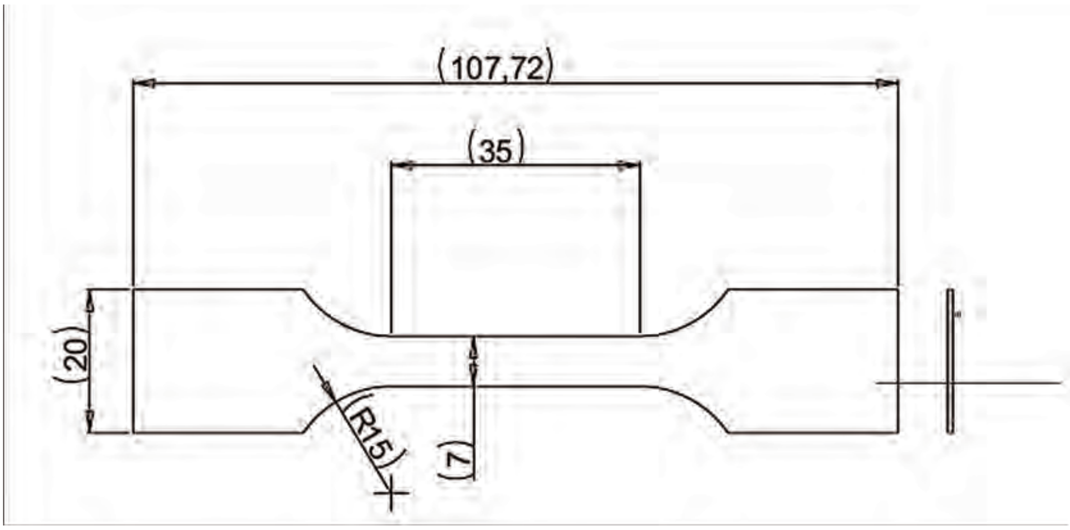

Fig. 1. Geometry of test samples

Acoustic emission measurements have been carried out during the tensile tests, using the AE equipment AMSY-5 Vallen Systeme GmbH with preamplifiers AEP4 (amplification 34dB) and broadband acoustic sensors M31-FUJICERA with a detection range up to 1,5MHz. As frequency filters have been applied, the actual detection range was 95 – 850 kHz. The fractographic examination has been carried out on the HITACHI S-2600N scanning electron microscope.

3. RESULTS AND DISCUSSION

Microstructural characteristics and mechanical properties have been shown on Fig. 2 and Table 2. It could be observed that ion nitriding led to the formation of surface layers with m-phase structure. The layer thickness of samples treated for 8 hours was around 10m and was about 30% thicker than the thickness of layers formed after 2 hours. The influence of the nitriding treatment on tensile properties was negligible, however after nitriding a significant hardness increase to values exceeding 800HV0.2 has been observed.

Table 2. Structure and mechanical properties

	Layer Thickness[μm]	Phase Composition	Microhardness [HV0,2]	Tensile Strength [MPa]	Total Elongation [%]
SW	-	γ, martensite	351 ± 34	709.7	71.3
430_2h	6 – 8	m-phase, γ	864 ± 103	710.0	70.3
430_8h	9 – 12	m-phase, γ	837 ± 83	707.3	71.2

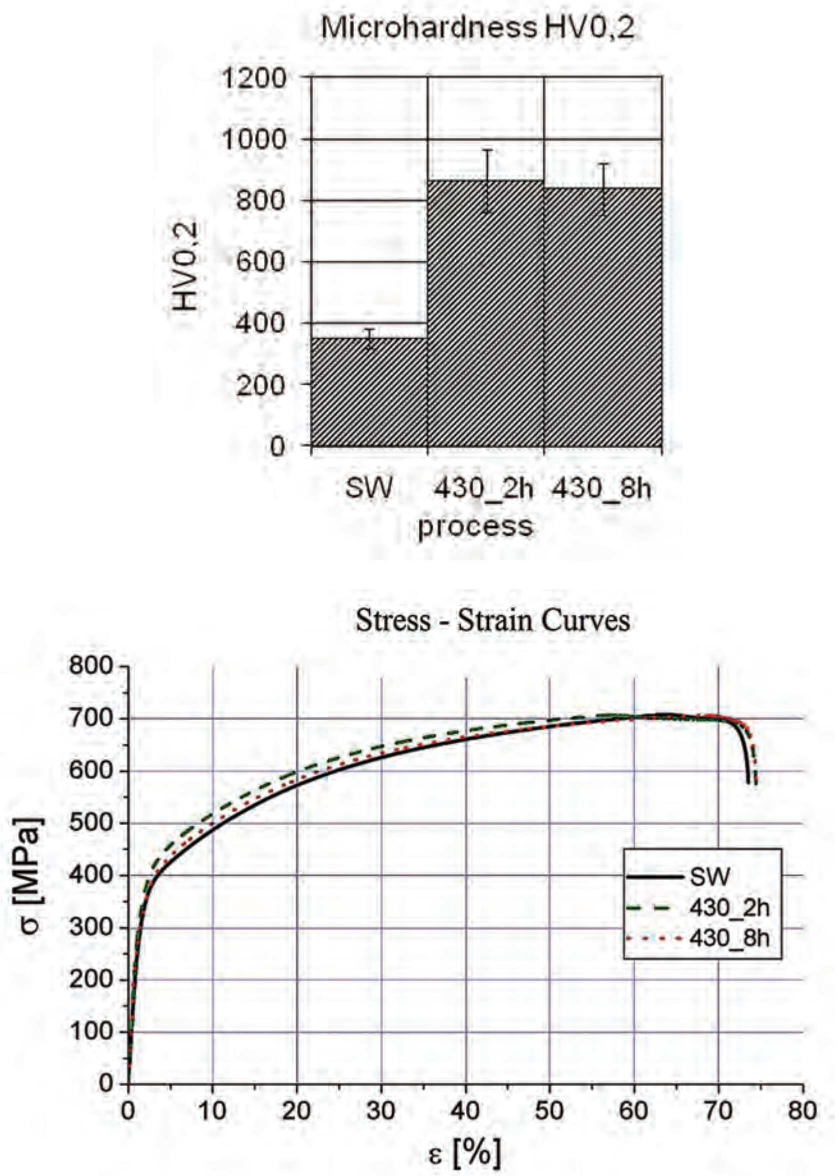

Fig. 2. Mechanical properties of the investigated samples

Fractographic examination of the fracture surface and the free surface of the raw sample has shown a ductile character of the deformation and fracture (Fig. 3.a. and 3.b.). On the other hand, SEM images of nitrided samples have shown a brittle fracture surface of the nitrided layer and a ductile one of the bulk material (Fig. 3.d and Fig. 3.f).

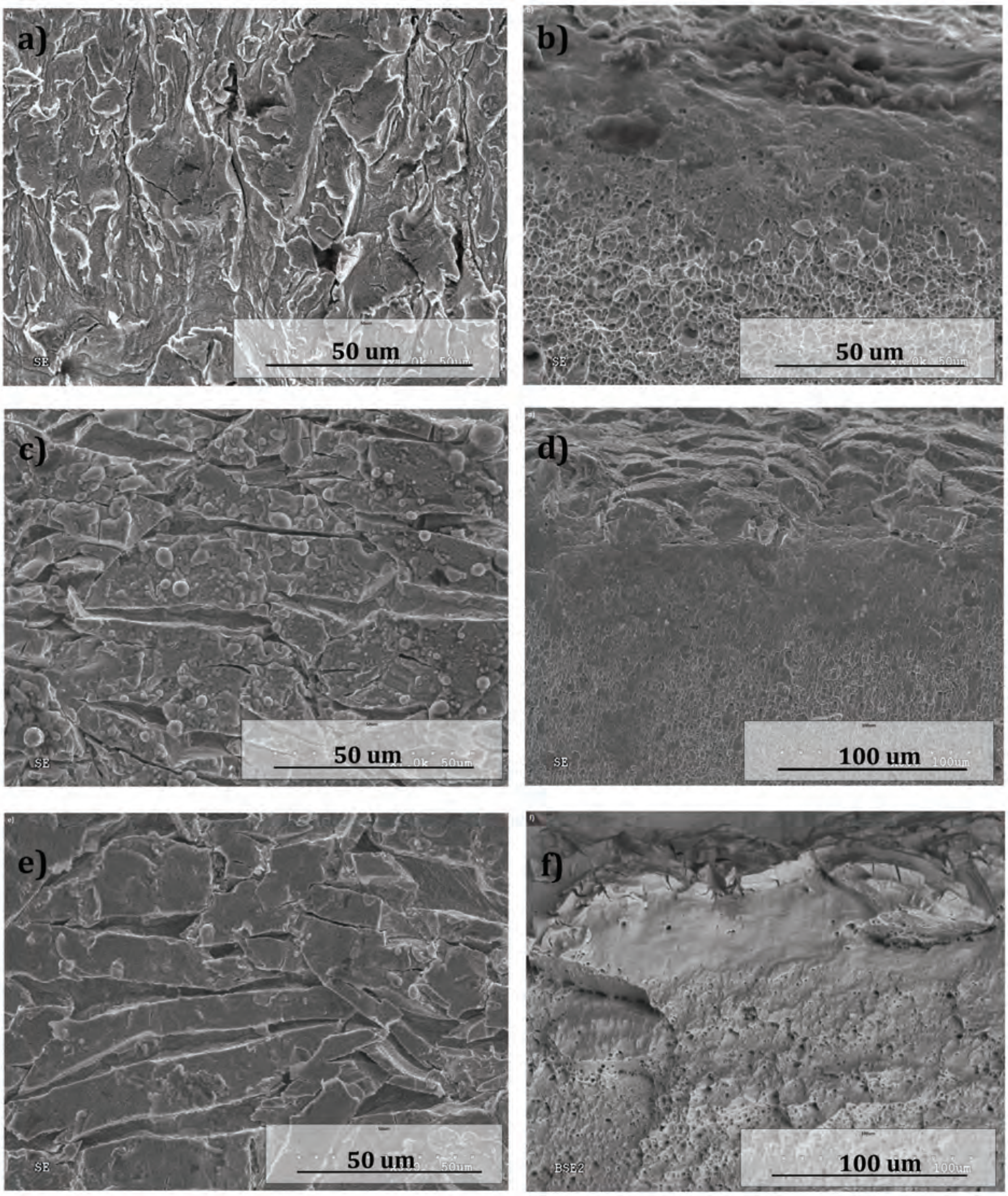

Fig. 3. SEM images after the tensile test: a,b) the topography and fracture surface of the raw sample; c, d) the topography of the layer and the fracture surface of samples nitrided for 2h; e, f) the topography of the layer and the fracture surface of samples nitrided for 8h

The examination of free surfaces of these samples has revealed in the nitrided layer a dense network of microcracks perpendicular to the surface. Worth mentioning is the fact that the SEM images of samples nitrided for 2 hours and for 8 hours looked very similar, which indicated that the deformation processes in both sample series must have been very similar as well.

AE energy and RMS voltage in function of time of the tensile test (Fig. 4) were the most useful acoustic emission characteristics in the analysis of deformation and cracking of the test samples. To allow a confrontation of AE with the load, tensile force vs. time has also been placed on these graphs.

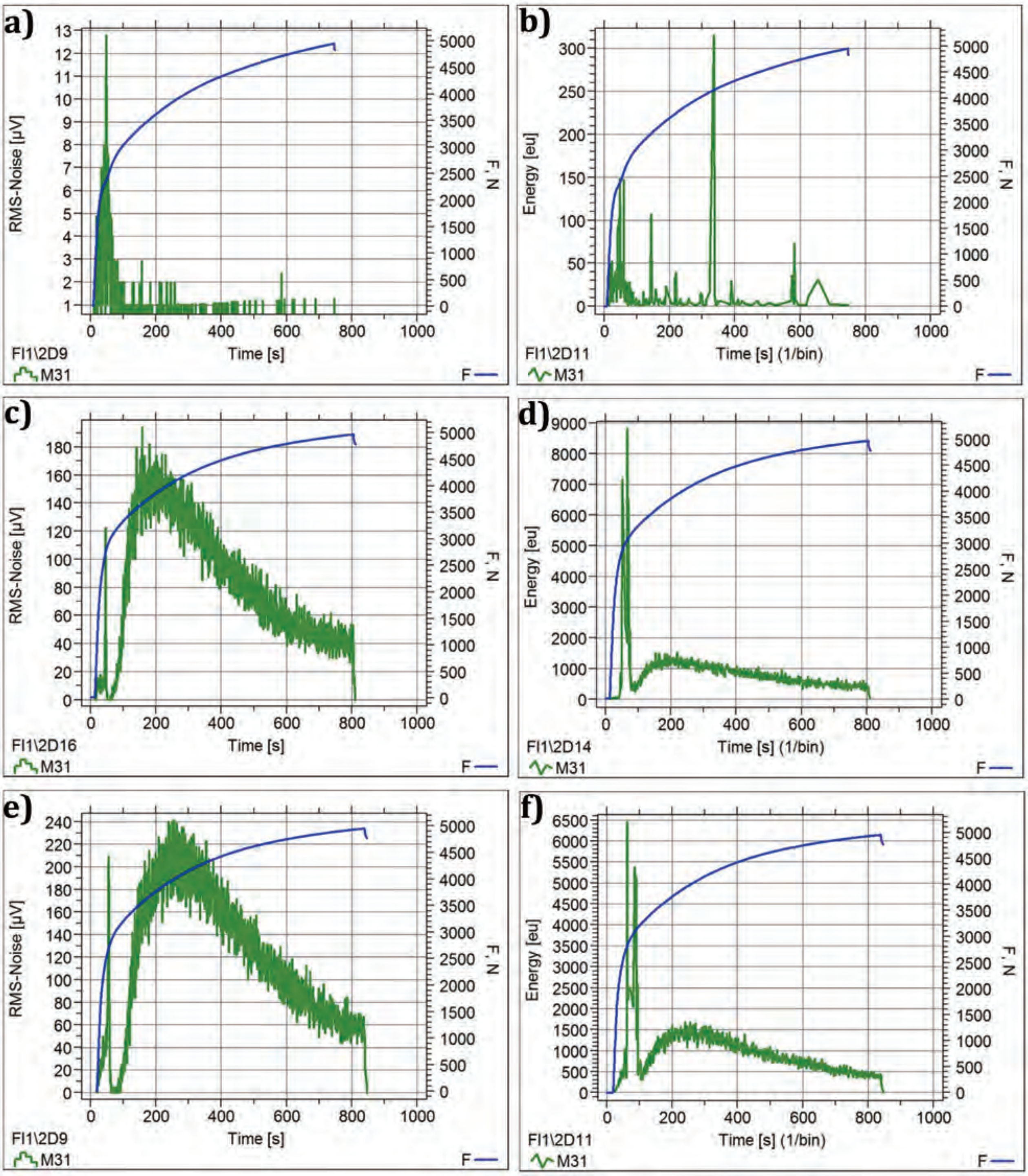

Fig.4 Selected acoustic emission history graphs: a, b) RMS voltage and AE energy in the raw sample; c, d) RMS yoltage and AE energy in the sample after 2 hours nitriding; e, f) RMS voltage and AE energy in the sample after 8 hours nitriding

The RMS voltage of the AE signals, which has been registered for the range of low amplitudes (below the threshold level) has provided valuable information about the general level of acoustic activity, in particular, the level of co called continuous emission, that is usually generated by plastic flow.

On the other hand, the EA energy, that is proportional to the energy released by particular EA origins, has provided useful information about the so called transient emission, which is usually generated by sudden microstructural changes, like crack propagation and delamination.

Test results of the raw sample (Fig. 4.a. and 4.b.) have shown one single maximum of both AE parameters, RMS and energy, close to the yield limit, which has indicated the initiation of plastic flow. During further plastic deformation AE was at a very low level.

The results of the nitrided samples, otherwise than the raw sample, have shown a high level of RMS and energy registered in the plastic region (Fig. 4.c.-4.f.). That has indicated that this emission must have been related with the presence of the nitrided layer and mechanism taking place in that layer. Confronting these results with the fractographic examination has led to the conclusion that the acoustic emission recorded in the plastic region must have been related with the formation of the observed microcrack network, and that the microcracks at the surface formed and propagated in the whole plastic region of the tensile test.

Worth mentioning is also the presence of two maxima of the AE energy at a load close to the yield limit (Fig 4.d. and 4.f.). The first maximum of energy has taken place simultaneously with the maximum of the RMS voltage, which, similar like in the raw sample, has indicated the initiation of plastic flow of the sample material. The second maximum of the energy however has been accompanied by a significant reduction of the RMS voltage. That has indicated the presence of high energy transient signals, thus most probably the initiation of multiple cracking of the nitrided layer. In the sample nitrided for 8 hours this maximum has taken place at a higher strain than in the sample nitrided for 2 hours, which, according to the above interpretation, would indicate a higher strength of the layer formed after 8 hours nitriding.

4. CONCLUSIONS

Based on the results the following could be concluded:

- Low temperature nitriding did not have any adverse effect on the tensile strength of the test samples.
- Cracking of the nitrided surface layer has been initiated beyond the yield limit of the bulk material, and further initiation and propagation of microcracks at the surface has continued till the final break of the sample.
- The most valuable information, that can be obtained via AE measurements for surface hardened materials during the tensile test are:
- The real yield limit of the bulk material and load conditions leading to crack initiation of the hardened layer
- The character of the cracking progress of the hardened layer

5. ACKNOWLEDGEMENTS

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ANALIZA WYTRZYMAŁOŚCIOWA WARSTW AZOTOWANYCH WYTWORZONYCH NA AUSTENITYCZNEJ STALI NIERDZEWNEJ NA PODSTAWIE POMIARÓW EMISJI AKUSTYCZNEJ (AE)

Streszczenie

Celem niniejszej pracy był opis przebiegu deformacji i niszczenia warstw azotowanych wytworzonych na stali austenitycznej w oparciu o badania zjawisk emisji akustycznej (EA) w czasie statycznej próby rozciągania. Warstwy na stali typu (AISI 304) wytworzono metodą azotowania jarzeniowego. Wykonano badania mikrotwardości, badania mikrostruktury oraz składu fazowego. Główną metodę badawczą w tej pracy stanowił pomiar emisji akustycznej. Wykazano, że na podstawie przebiegu charakterystycznych parametrów EA, energii i napięcia skutecznego RMS, możliwe jest określenie granicy plastyczności, początku propagacji pęknięć w warstwie oraz dynamiki przebiegu pękania warstwy.