Strojniški vestnik - Journal of Mechanical Engineering 54(2008)1, 25-36
UDC 620.19

Paper received: 28.6.2006
Paper accepted: 19.12.2007

An Attempt to Detect Various Types of Stress-Corrosion Cracking on Austenitic Stainless Steels by Simultaneous Measurements of Acoustic Emission and Electrochemical Noise

Žiga Bajt - Mirjam Leban - Jaka Kovač* - Andraž Legat
Slovenian National Building and Civil Engineering Institute, Slovenia

This paper discusses the potential use of acoustic emission (AE) and electrochemical noise (EN) for the detection and characterization of different types of stress-corrosion cracking (SCC) in austenitic stainless steels. The discussion is based on the performance of constant load experiments. Three different measurements were performed simultaneously during the experiment: acoustic emission, electrochemical noise, and elongation of the test specimen. The stainless steel was sensitized to increase its susceptibility to SCC. Two different electrolytes and two different load levels were used to obtain different types of SCC. Intergranular stress-corrosion cracking (IGSCC) was investigated in an aqueous solution of sodium thiosulphate at a load level below the yield point, whereas transgranular stress-corrosion cracking (TGSCC) was investigated in an aqueous solution of sodium thiocyanate at a load level beyond the yield point. No simultaneous AE activity increases, EN transients or elongation discontinuities were observed in the case of IGSCC. Except for a few EN transients, the signals were smoother than in the case of TGSCC, which could reflect the more continuous nature of IGSCC processes. This is also probably the reason that no simultaneous events were observed in this case. On the other hand, a significant increase in the DC part of the measured electrochemical current, corresponding in time with increases in elongation and, to some extent, also increases in AE activity, was observed in the second part of experiment. It was assumed that the current shift in the anodic direction was caused by an increase in the dissolving surface area due to the growth of cracks. On the other hand, on the basis of the simultaneous use of the three techniques, several single TGSCC events were detected. Detection was accomplished by the observation of simultaneous increases in AE activity, EN transients, and discontinuities (jumps) in elongation. On the basis of the time resolution of the measurements, it was concluded that the detected electrochemical events were the result of corresponding mechanical events. It was also concluded that the simultaneous use of EN and AE is a promising approach for the detection and characterization of SCC, but that further research and improvements of the measuring system are needed.

Keywords: stress corrosion cracking, electrochemical noise, acoustic emission, austenitic stainless steel

0 INTRODUCTION

Stress-corrosion cracking (SCC) is one of the most dangerous degradation processes to which metals can be subjected, because it can cause unexpected failures of structural elements without any previously visible signs [1] to [4]. The most exposed groups of metals are stainless steels and other alloys that exhibit good resistance to the general corrosion.

Stress-corrosion cracking is a process which causes the initiation and propagation of cracks by the simultaneous action of mechanical stress and corrosion reactions. In general, there are three different conditions that must be satisfied for the development of SCC: a material sensitive to SCC, a corrosion solution that causes localized corrosion, and a large enough tensile load (in most cases it can be below the yield point of material). Regarding propagation, SCC processes are divided into transgranular SCC (TGSCC) and intergranular SCC (IGSCC) modes. It is known that the TGSCC process advances with bigger discontinuous steps, while researchers do not agree about the discontinuity of IGSCC. It is only certain that the individual dissolution and fracture processes (steps) are much smaller than in the case of TGSCC. In the past, several different mechanisms describing TGSCC and IGSCC processes have been proposed. All these mechanisms show relatively good agreement for some special cases, but in general the alternating processes of dissolution and fracture

---

*Corr. Author's Address: Slovenian National Building and Civil Engineering Institute, Dimičeva 12, SI-1000 Ljubljana, Slovenia, jaka.kovac@zag.si

have not yet been precisely explained. One important reason for the insufficient knowledge of SCC processes are the measuring techniques for their detection and monitoring. Electrochemical measuring techniques can only provide the electrochemical components of the SCC process, whereas physical measuring techniques can only provide data about the physical parameters. Also, conventional electrochemical methods for monitoring corrosion processes (different types of potentiodynamic polarization, impedance spectroscopy, etc.) are not suitable for the detection and monitoring of SCC processes, since they can only estimate the susceptibility of the investigated system metal/corrosion solution to a certain type of corrosion. For these reasons, acoustic emission and electrochemical noise are promising techniques, which have been developed for the monitoring and characterization of SCC processes.

1 ELECTROCHEMICAL NOISE AND ACOUSTIC EMISSION, AND PREVIOUS RESEARCH

Electrochemical noise (EN) is a method that measures the fluctuations of current and potential between two electrodes. Three electrodes are needed for such measurements: a working electrode and two reference electrodes. Voltage is measured between the working electrode and one of the reference electrodes, whereas potential is measured between the working electrode and the other reference electrode. In our measurements all three electrodes were made from the same material, which means that not real reference electrodes (i.e. graphite electrodes) but "pseudo" reference electrodes were used.

Corrosion events on a single electrode are reflected in the measured current and voltage. In the case of three equal electrodes no net current flows and there is zero potential between the working and the reference electrode. When local anodic corrosion damage appears at the working electrode (i.e. breakage of the passive film, the initiation of pits, the formation of new surface during SCC), it results in a shift (turn) in the so-called anodic direction of the measured current and potential. Originated electrons then travel from the working electrode to the reference electrodes. If local anodic corrosion damage occurs at the reference electrode for current measurement, it is reflected in a shift of the measured current in the opposite direction – the cathodic direction (electrons travels away from the current reference electrode), and in a shift of the measured potential in the anodic direction. In the case of local corrosion damage occurring on the reference electrode for potential measurements, this is reflected in a shift of voltage in the cathodic direction. In reality it should be taken into account that only a part of the current is measured as described, because part of electrons can be used up at the same electrode where they were produced. This is possible because anodic and cathodic areas can be present on the same electrode. Consequently, only a very small part of the current which is a result of local corrosion damage can be measured, or no current is measured in the case that all electrons are used up on the same electrode. In such situations local corrosion processes are hardly detectable.

The main advantage of the EN method in comparison with the other electrochemical methods is its ability to monitor corrosion in a freely corroding system without any external excitation signals. This feature makes it possible to measure the development of the natural corrosion process. On the basis of fast changes (transients) in the measured current and voltage, the EN technique detects the initiation and development of corrosion processes.

Over last few decades electrochemical noise measurements have become one of the most promising methods for the detection and, to some extend can be used to, characterization of local corrosion processes [5] to [29]. Positive results have been obtained in various cases of localized corrosion. The method proved to be successful in the detection of unstable pitting corrosion [7] to [10], and in the detection of the transition from unstable to stable pitting corrosion [11] and [12]. The method has also been more or less successful in the case of crevice corrosion detection [10], [13] and [14]. Numerous measurements have been performed regarding the study of passive film formation and breakage processes [15], and also regarding the study of the effect of corrosion inhibitors on these processes [12], [16] and [17].

Many more or less successful EN measurements have also been performed in the study of SCC. The beginnings of the use of EN method for the detection of SCC, the first successful detection of SCC by EN measurements, and differentiation between the cracking non-active and the cracking active periods, go back to the 1990's [18] to [20]. Over the next period researchers [21] to [30] succeeded in recognizing single characteristic events (transients) of EN and correlated these events with crack growth. Researchers [21] found that the observed EN transients corresponded in time with sudden drops in the simultaneous measured force on the specimen during tensile tests. In the research the velocity of crack propagation calculated from the measured force drop was compared with the current transients. It was found that the crack propagation velocity is proportional to the measured current. In our previous research [24] to [27], which was performed on stainless steel in thiosulphate solutions, EN measurements were compared with simultaneous measured elongation during SCC processes. A relation between the current transients and the simultaneously generated elongation rate was found. A similar approach was used by some other researchers using pre-cracked compact tension specimens [28] and [29]. In this research periodic fluctuations were observed, and it was assumed that these fluctuations were related to the periodic propagation of the crack.

The second method used was acoustic emission (AE) analysis. This analysis is based on measured acoustic emission, which is a consequence of the fast energy relaxation that occurs at a localized source (i.e. crack development in the material). This relaxation of energy causes a development of elastic waves that propagate through the material and can be detected by piezoelectric sensors. The analysis of acoustic emission, whose purpose is to detect elastic waves, was developed in the 1960's, and since then it has developed into one of the most successful methods for the nondestructive monitoring of the initiation and progress of deformations in materials [31] to [33]. Besides use for the detection and research of mechanical defects, as well as other energy changes in materials [31] to [36], it has also been developed for the characterization and detection of chemical processes (corrosion) [33], and the monitoring of manufacturing processes [33], [37] and [38].

It is characteristic that the AE signal which is captured by the piezoelectric sensor consists of oscillating voltages, which quickly rise to the maximum level, and then drop back slowly to the previous level. Signals or AE events are caused by desired events (i.e. crack propagation) and undesired events (i.e. instrumental noise, external noise). Due to the large number of AE events, a trigger level which is a measure for the intensity of events is defined for each measurement. Only signals with an intensity above the trigger level are captured, whereas other signals are eliminated. The number of AE events is estimated on the basis of the captured signals. During such characterization various problems occur: the summation of signals from different events, and the capturing of undesired signals that are the consequence of reflected waves and noise signals from the surroundings. Not only are some undesired signals captured, but also some useful signals are lost due to the described way of measuring.

Regarding the detection and characterization of SCC, AE analysis can be used for the detection of mechanical events, as opposed to EN, which detects electrochemical events. In the SCC process mechanical and electrochemical events are related to one other, so the combination of AE and EN could be the best tool for the detection and characterization of SCC. The main idea of the combined use of AE and EN is the simultaneous monitoring of SCC using both techniques, and the search for a relation between the mechanical and electrochemical components of crack formation. The aim of the simultaneous use of both methods is also to differ between the transients caused by SCC, and the EN transients caused by other corrosion events (i.e. the initiation of corrosion pits).

Several successful investigations, which used AE for the detection and characterization of SCC, have been realized in the past [39] to [43]. One of the first investigations of SCC by means of AE was performed by Newman and Sieradzki, who are known as pioneers in the field of SCC detection. They looked for correlations between measured AE and EN signals. Experiments were performed on three different systems, and positive results were obtained in the experiment performed on brass [39]. At that time several AE experiments during SCC in different systems showed mixed results [40]. Later Jones investigated different types of cracking by using AE analysis on various materials, and TGSCC was successfully detected in this research [40]. On the other hand, results concerning IGSCC were not so successful. Jones proposed that some transgranular fracture surfaces were the result of ligaments that fracture behind the advancing intergranular crack front, and that most of the detected AE signals were the result of the transgranular part of the fracture. In a continuation of this research it was established that the AE technique can reliably detect intergranular stress corrosion cracks of 200 to 300 µm length and at a depth of 100 to 200 µm [41]. Some other researchers have also agreed with Jones's findings. They also successfully detected TGSCC, whereas the detection of IGSCC was described as problematic. Researchers assumed that the AE signal during IGSCC comes from small individual parts of the crack that have a transgranular nature [42]. In this research it was established that pure anodic dissolution does not generate observable AE signals. Several researchers [43] have found that AE activity due to the SCC process during tensile tests is the highest immediately after the yield point is reached and during final fracture.

In our previous research [27], characteristic EN transients were compared with simultaneous measured elongation events (discontinuous jumps). In order to measure these events even more precisely, and to estimate the sequences of electrochemical and mechanical events, a combination of EN and AE was used in this research. At the same time elongation of the specimen exposed to SCC processes was also measured. Analysis of the results from all three types of measurements was performed with aim of detecting the processes in the case of transgranular and intergranular SCC, as well as to get the basic characteristic of the EN and AE signals which were generated during both types of SCC, and thus to explore the possibility of detecting and characterizing different types of SCC with EN and AE measurements. The main emphasis was on the simultaneous use of the three methods, and on the analysis of the simultaneously measured signals obtained using these methods.

2 EXPERIMENTAL

The experiments were carried out in a corrosion cell that was filled with a corrosion solution (Fig. 1 - left). Three electrodes made from the same material were sealed in the corrosion cell.

The middle electrode had the shape of a tensile specimen and was loaded during the experiment. The other two electrodes were straight, with a constant quadratic cross-section along the entire length of the electrodes. No mechanical stress was applied to them during the experiment. These two electrodes were used as pseudo reference electrodes for the EN measurements. An apparatus with a rigid frame was used for the constant load application (Fig. 1 - right). The tensile specimen was fixed between two movable jaws that were fixed to the rigid frame. An arm with weights was fixed to the bottom jaw. Weights were added or removed so

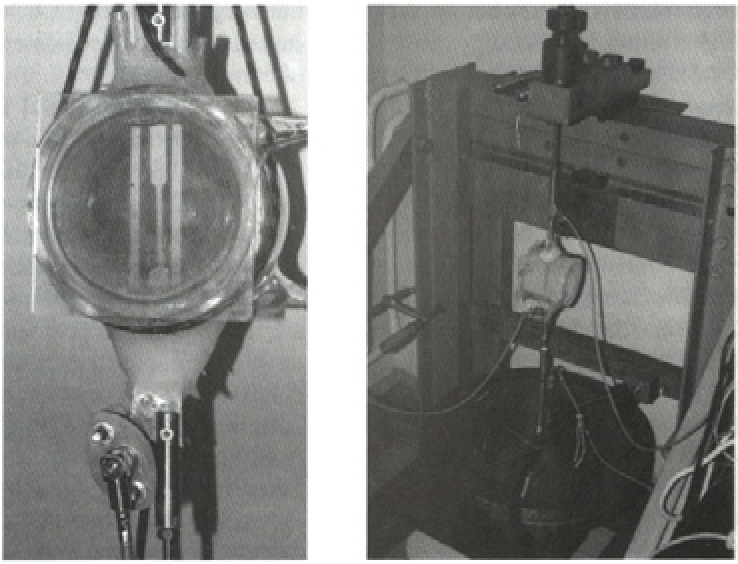

Fig. 1. *Left) The corrosion cell, inside which three electrodes can be seen. The middle electrode with the reduced cross-section is the working electrode. The AE sensor is fixed to the lower end of the working electrode. Right) The tensile testing apparatus. The elongation meter is fixed to the frame of the apparatus.*

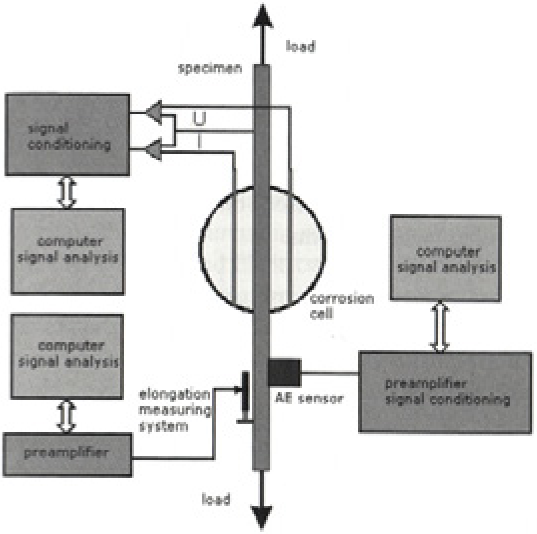

Fig. 2. *Schematic view of the experiment and of all three measuring systems*

that the desired load was achieved. The upper jaw was fixed to the top of the metallic frame. The distance between the lower and upper jaws could be adjusted by means of a spindle, which also transferred the load to the tensile specimen. The apparatus stood on a rubber support plate, which enabled electrical isolation from the neighbourhood, and also damped unwanted vibrations. The tensile specimen was additionally electrically isolated from its surroundings by means of non-conducting plugs. All the mobile elements of the apparatus were tightly screwed into the desired position, so that no unwanted oscillations could disturb the experiment.

Measurements were performed with the three different measuring systems simultaneously (Fig. 2), and the specimens were monitored up until failure. EN was measured in a freely corroding system. The current was measured between the working electrode (tensile specimen) and one of the reference electrodes with a zero resistance ammeter (ZRA), whereas voltage was measured between the working electrode and the other reference electrode with a high-impedance voltmeter. The measured current and potential were amplified and digitalized using a 16-bit A/D converter. The sampling rate for EN data collection was 10 Hz. The resolution of the measured current was 3 nA, and 30 µV for measured voltage.

A piezo-electric sensor, which was fixed to the bottom part of the working electrode outside the corrosive cell, was used for the AE measurements. Amplification of the AE measuring system was 56 dB. The maximum amplitude was 10 V. The signal was captured with a frequency of 250 kHz. In order to eliminate low-frequency disturbances, the signal was filtered by means of a high-pass filter with a cut-off frequency of 50 kHz. Since the duration of the measurements was, in many cases, several tens of hours, selection of data was performed by using a simple auto-detection function: when a signal exceeded a trigger level, the system started to store the signal for 1 s. An instrument-based noise of 0.008 V was determined, and signals higher than 0.01 V (the trigger level) were stored. The stored packets of AE were afterwards analysed using Matlab software, and compared with the simultaneous measurements of EN and elongation.

Elongation of the tensile specimen was measured by means of an inductive probe fixed to the weights that were hung directly on the tensile specimen. Resolution of the elongation measuring system was 1 µm, but only changes larger than 2 to 6 µm (taking into account the presence of noise during individual measurements) were stored. The size of the stored changes was defined for each case individually taking into account the present noise. Two individual experiments are presented in this paper. The minimum size of the stored changes was 2 µm in the first experiment, and 6 µm in the second experiment.

The surfaces of the working and reference electrodes that were exposed to the corrosive medium, as well as the fracture surfaces of fracture, were examined after the experiment was finished. Visual and scanning electron microscope inspection techniques were used.

The specimens were made from austenitic stainless steel type AISI 304. The material was treated before the specimens were produced in the following manner: 1) heated to 1050°C, kept at this temperature for 1 hour - water quenched, 2) heated to 650°C in a nitrogen atmosphere, kept at this temperature for 24 hours, cooled down in air. Such heat treatment causes the growth of austenitic grains, which in our case had a size of approximately 250 µm. Chromium carbides $Cr_{23}C_6$ were precipitated at the grain boundaries. It is known [44] that the described microstructure is very sensitive to SCC processes.

The experiments were performed in two different corrosive solutions, and at two different load levels: 1) in a diluted 0.5 M aqueous solution of sodium thiosulphate ($Na_2S_2O_3$), at a load -corresponding to approximately 85 % of the yield point of the material, 2) in a diluted 1.4 % aqueous solution of sodium thiocyanate (NaSCN), at a load corresponding to approximately 90 % of the tensile strength of the material. In the first case stress-corrosion cracks propagated mainly across the crystal grains – TGSCC, whereas in the second case cracks were propagated along the crystal boundaries - IGSCC.

3 RESULTS AND DISCUSSION

In the interpretation of the results, sudden changes in all three measured signals (fluctuations, discontinuities) were mainly inspected. It was expected that the characteristics of IGSCC and TGSCC could be described on the basis of analyses of simultaneous events.

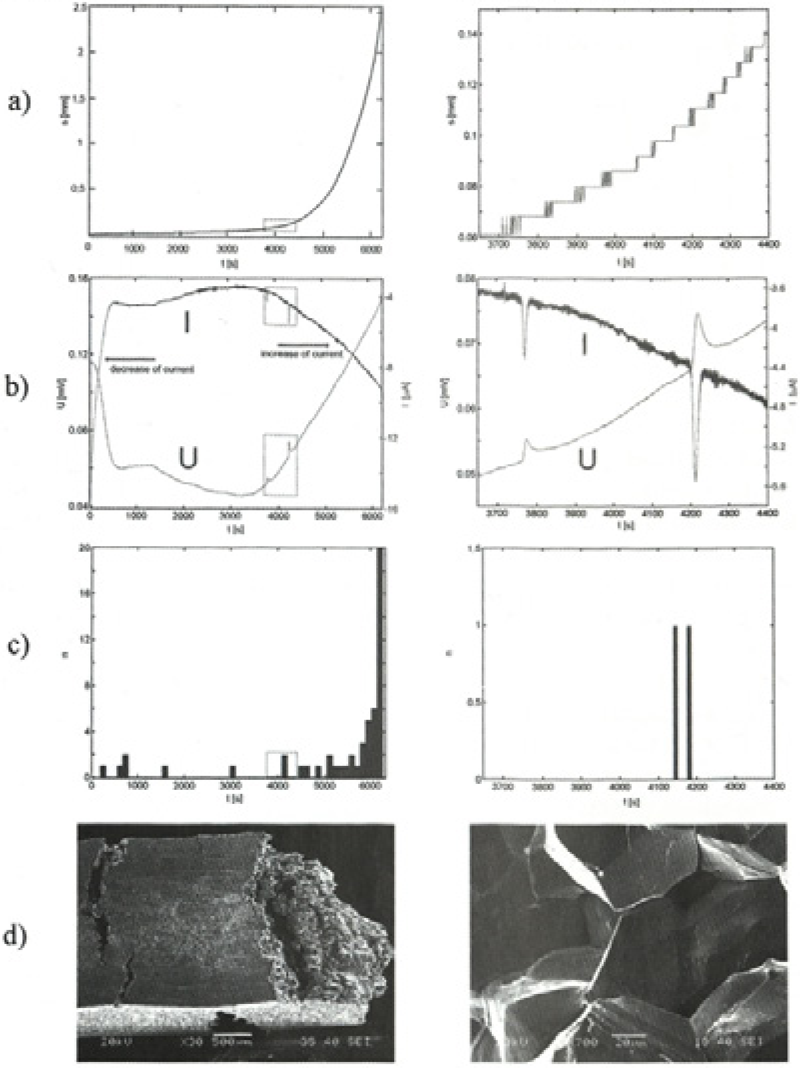

Fig. 3. *Results of measurements during intergranular stress-corrosion cracking of the heat treated stainless steel AISI 304: a – left) entire elongation (l), a – right) enlarged detail from the left-hand diagram (the detail is marked with a frame in the left-hand diagram), b – left) EN: potential and current for the entire measurement, b – right) enlarged detail from left-hand diagram (the detail is marked with a frame in the left-hand diagram) with two events, c – left) AE histogram: number of AE events (n) for the entire measurement, c – right) enlarged detail from the left-hand diagram (the detail is marked with a frame in the left-hand diagram) with the finest division of events in the time intervals (shorter time intervals), d – left) SEM image of the specimen surface with a magnification of 30x, and d – right) SEM image of the fracture surface with a magnification of 700x.*

Typical results of all three measuring techniques during the entire duration of the experiment carried out in a diluted aqueous solution of $Na_2S_2O_3$ are presented in the left-hand diagrams in Figure 3. On the right-hand side of Figure 3 there is an enlarged time detail from the last part of the experiment. The first diagram (Fig. 3a – left) shows an elongation measurement, which was monitored together with AE and EN in order to obtain a better understanding of the deformation processes during the experiment. It can be seen that the elongation increased uniformly, without any sudden jumps that could be expected due to sudden crack propagation or opening. In this case the elongation measurement did not provide useful information about individual events. In the first part (the first 500 seconds) of the measured EN signals (Fig. 3b – left), a large decrease in the measured current can be seen, which was the consequence of passive film growth on the surface of electrodes. The passive film protects the surface of the electrodes against the corrosion, resulting in a smaller corrosion current. In the last part of the measurement a large increase in the DC component of the measured current can be seen. This increase could be related to the initiation and growth of cracks. Also in the last part of the experiment (40 minutes before failure) two typical EN transients (Fig. 3b – right), which could be related to individual SCC events, were detected. However, these transients did not correspond in time with increases in AE activity (Fig. 3c – right), or with any observable sudden increase in elongation. The observed EN transients could have been generated by alternating dissolution at the crack tip (according to the slip-dissolution model [45]), but it is more probable that they were generated by the initiation of pits [6] to [10], which were observed on all electrodes after the experiment was finished. The AE signal did not show any sudden increases in AE activity, but the AE activity increased monotonically and approximately exponentially until failure. For these reasons individual events could not be determined from the presented simultaneous measurements.

In order to determine the prevailing mode of cracking and to characterize the intensity of the process, analysis of the specimen's surface (Fig. 3d – left) and fracture (Fig. 3d – right) were performed using a scanning electron microscope. It was found that a good two-thirds of the fracture was brittle and that less than one third was ductile. Fractographic analysis of the brittle part of the fracture showed that it was caused by propagation of the main intergranular crack. On the other parts of the specimen quite a lot of large cracks propagating perpendicular to the load direction were observed. Some of these large cracks can be seen on the left-hand side of Figure 3d.

From the description above it can be concluded that despite the great number of cracks observed in the case of IGSCC, the detection of individual crack formation or crack growth steps was not successful. This indicates that the process is probably fairly smooth or continuous. On the other hand a relationship between the DC component of the electrochemical current, AE activity and elongation was established. For this reason it can be assumed that these techniques enabled monitoring of the cumulative process of IGSCC.

More information about individual SCC events was obtained by analyzing the three types of measurements during the SCC of specimens exposed to a dilute aqueous solution of NaSCN. Typical results across the entire duration of the experiment are presented in the left-hand diagrams in Figure 4. On the right-hand of Figure 4 is an enlarged time detail from the last part of the experiment, before failure. The characteristics of the measured EN, AE signals and elongation are rather different than in previous case. It can be clearly seen that in this case the signals are much less smooth and more variegated. Also, the duration of the experiment was much longer than in the previous case, although the load level was higher (above the yield point) in this experiment. Two significant increases in the elongation can be seen in the elongation measurements (Fig. 4a – left). The increase in elongation at the beginning of the measurement was probably due to plastic deformation or yielding of the specimen, which was loaded beyond its yield point. The final increase in elongation was the result of final fracture. Two bigger jumps (discontinuities) in the elongation increase can also be seen before the final increase of elongation. In the first part (the first two hours) of the measured EN signal (Fig. 4b – left) a large decrease in the measured current can be seen, which is, as in the previous experiment, a consequence of passive film growth on the surfaces of the electrodes. After the initial decrease, the current stabilized, and did not increase as in case of IGSCC.

Two characteristic events, which are more pronounced in the potential measurement than in the current measurement, can be seen in the last part of the experiment, before fracture. Both events occurred together with the above mentioned discontinuities in the elongation (Fig. 4a) and with the increases in AE activity (Fig. 4c). The highest AE activity was expected to be observed at the fracture, as it was in the case of IGSCC. In Figure 4c an increase in AE activity in the last part of measurement can be seen, but the AE activity was not the highest when fracture occurred. The AE activity was also somewhat higher at the beginning of the measurement. This increase was probably the result of specimen's plastic deformation immediately after loading, which also caused

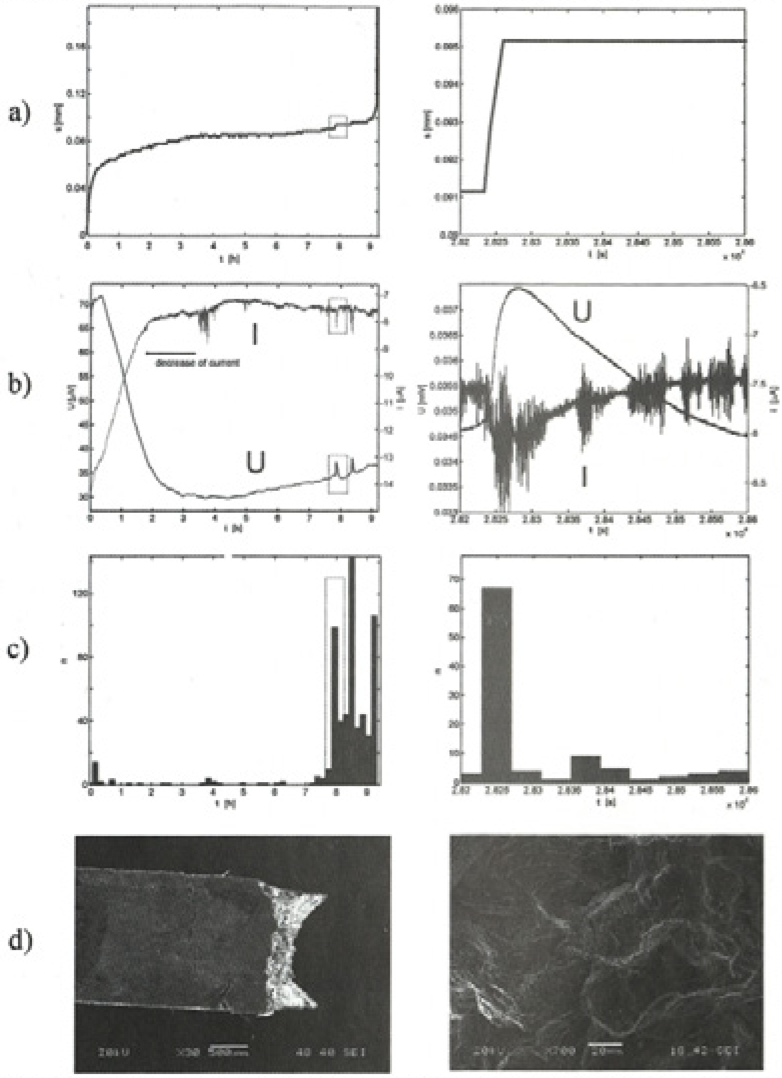

Fig. 4. *Results of measurements during intergranular transgranular stress-corrosion cracking of the heat treated stainless steel AISI 304: a – left) entire elongation (l), a – right) enlarged detail from the left-hand diagram (the detail is marked with a frame in the left-hand diagram), with an elongation jump, b – left) EN: potential and current for the entire measurement, b – right) enlarged detail from left diagram (the detail is marked with a frame in the left-hand diagram) with a characteristic event, c – left) AE histogram: number of AE events (n) for the entire measurement, c – right) enlarged detail from the left-hand diagram (the detail is marked with a frame in the left-hand diagram) with the finest division of events in the time intervals (shorter time intervals), d – left) SEM image of the specimen surface with a magnification of 30x, and d – right) SEM image of the fracture surface with a*

growth of the elongation over the same time interval.

On the right-hand diagrams of Figures 4a, b, c, a typical SCC event detected by all three systems can be seen. On the basis of the measurement results (presented in Fig. 4), and taking into account the resolution of the measurements, it can be concluded that mechanical events occurred first. These mechanical events caused the occurrence of new surface, and were detected as a sudden increase in elongation, combined with a simultaneous increase in AE activity. At the same time a sudden decrease in current, and an increase in potential, followed in both cases by a slower return to the initial levels, was observed. Such a shift of the EN signals in the anodic direction (from the point of view of the working electrode), and its return to the initial levels, is characteristic for the occurrence and passivation of new surface at the crack tip during crack propagation. The analysis showed that not all EN transients were accompanied by increases in AE activity and elongation (Fig. 4). These characteristic events were probably generated by the initiation of pits. The presence of pits on the surfaces of the specimens was confirmed by visual and SEM inspection after each experiment. On the other hand, no AE activity increases and sudden jumps of elongation were found without accompanying EN transients, meaning that, in the case of the selected parameters, no significant noise occurred in the results of the AE and elongation measurements. It needs to be stressed that the measured elongation jumps were very small and close to the limit of the resolution of the measuring system. Thus it would be very difficult to predict the growth or opening of cracks just on the basis of detected elongation jumps, without corresponding increases in AE activity. An important fact is also the reduced resolution due to the elimination of noise, because of which some useful signals were probably not captured. This was most likely in the case of the AE measurements, where it was very difficult to set a proper trigger level above which signals should be stored. This is also probably the most important reason that the number of observed cracks on the specimen was larger than the number of detected events, which were characteristic for the development of cracks.

As in the previous case, analysis of the surface of the specimen (Fig. 4d – left) and of the fracture (Fig. 4d – right) was performed using a scanning electron microscope after the experiment had finished. It was found that a little less than two thirds of the final fracture was completely brittle, caused by the SCC process, whereas a little more than one third was a ductile fracture, caused by specimen overloading due to the reduced cross-section. Besides the main crack that caused failure of the specimen, also two bigger cracks and several small cracks were observed during the analysis (Fig. 4d – left). These cracks propagated perpendicular to the loading direction. From the observations it was assumed that all the cracks, except the three major ones, stayed in the initial phase, and did not propagate further. Fractographic analysis of the fracture surface showed that the larger part of the fracture caused by SCC was transgranular – the crack propagated across the crystal grains. TGSCC on the austenitic stainless steel could be described by the slip-dissolution model. The model consists of two main processes: dissolving at the crack tip, and a mechanical event – slip of the crystal planes [45]. These two processes act alternately, so that the propagation of the crack is probably non-uniform or discontinuous. The results of our measurements indicate this manner of cracking, because from the typical shapes of the signals from all three measuring techniques, a discontinuous process can be assumed.

From the results of the measurements and analysis of the two different experiments a question arises how big is the possibility for the detection of individual event during IGSCC with the simultaneous use of AE and EN. As has been previously mentioned, TGSCC is clearly a discontinuous process, which can be detected by measurements of AE and EN, which has also been shown in the case of our measurements. On the other hand, the results of the measurements indicated that IGSCC is a much more continuous process, although it is believed that it is not completely continuous. This is also in agreement with the results of some other researchers [40] and [41], who are also convinced that IGSCC is not a completely continuous process. However, researchers have stressed that the detection of IGSCC by means of AE is not reliable due to the very small amount of energy that is relaxed during deformation. The relaxed energy could be below the detection limit of the measuring method.

Interpretation of EN results for the IGSCC process is also difficult. As has been mentioned in the introduction cathodic surfaces in long cracks can be located on the crack walls or close to the mouth of crack. In such cases the measured electrochemical current represents only a small proportion of the total corrosion current, so that small events are probably undetectable. Some researchers have tried to avoid this problem by using of so-called compact tension specimens, where most parts of the specimen is protected by means of an anticorrosion coating, and only the active crack is exposed to the electrolyte [29] and [30]. In this case, the coupling current between the CT specimen and the electrode placed directly ahead of the crack mouth is measured. But even in this case cathodic surfaces are not completely eliminated, and the distribution of the current is not completely clear.

Even though our measurements of EN and AE during IGSCC processes did not detect individual characteristic SCC events (Fig. 3), which at the same time proves the statement that IGSCC progresses more continuously than TGSCC, the nature of the IGSCC process can be partly described from the main characteristics of all three measurements. In the last part of this experiment (Fig. 3b) the DC component of the measured electrochemical current increased monotonically, whereas the DC component of the electrochemical current in the TGSCC experiment was more or less constant (Fig. 4b). This observation is in agreement with those of some other authors [28-30], who also found a similar increase in the DC component of the measured current, and an absence of characteristic EN events. The increase of current in the anodic direction was probably the consequence of new anodic surface formation occurring at the crack tip. On the basis of the increasing current, it can be assumed that the rate of new surface formation, and thus the propagation of the crack, increased until the final fracture, or that at the same time the number of cracks, and thus the number of new anodic surfaces at the crack tip, increased. This assumption was additionally confirmed by the approximately simultaneous increase in elongation (Fig. 3a – left), which was a consequence of crack growth or opening, and also partly confirmed by the increase in AE activity in the last part of the experiment, which showed an approximately exponential increase until the failure of specimen. The increased AE activity in the last part of the experiment was probably generated by plastic deformation of the specimen, caused by passing the yielding point due to crack propagation that reduced the cross-section of the specimen. The second possibility for the generation of AE events was mechanical breakage of ligaments at the crack tip. On the basis of our research, it cannot be confirmed that AE signals are a consequences of the first, second or both processes, but based on previous experience it can be assumed that the first mentioned source of AE is more probable. Further research is needed to confirm our observations, to determine the prevailing source of AE during IGSCC, and to eliminate the possibility that the increasing current was caused by the initiation and growth of pits. From the measurements during IGSCC it was additionally observed that the increasing signals (the DC component towards the end of measurement, AE activity and elongation) were fairly smooth. This indicates smaller steps in the IGSCC process, and grater continuity of the process in comparison with the TGSCC process.

4 CONCLUSIONS

The possibility of the combined use of EN and AE for the detection and characterization of different types of SCC was investigated on the basis of experiments performed on austenitic stainless steel. AE and EN were measured simultaneously. Additionally, elongation of the specimens was measured simultaneously. On the basis of the presented results from the AE, EN and elongation measurements, it was established that the successfulness of the used AE and EN methods strongly depends mainly on the type of SCC.

Detection was only partly successful in the case of IGSCC. Some specific EN events were detected, but they did not correspond in time with changes in elongation or changes in AE activity, that these EN events could not be related to individual SCC events. On the other hand, a constant increase in the DC component of the measured current was detected in the second half of the experiment. It is believed that this shift in the current was the result of new anodic surface being formed at the crack tip. This assumption cannot be proved because the EN measurements cannot reliably distinguish between the propagation of intergranular cracks with dissolution, and the growth of stable pits or crevices. However, the above statement can be supported by the simultaneously observed increase in elongation and the approximately simultaneous increase in AE activity. However, the exact source of the AE during IGSCC cannot be determined from the results of the measurements. Based on previous experience, it is believed that the plastic deformations caused by the reduced cross-section are more probable sources of AE than mechanical breakages of ligaments at the crack tip. The main reason that no individual simultaneous events were detected probably lies in the way of progress of IGSCC the processes. This is because these processes advance relatively continuously, which is also reflected in the greater smoothness of the measured signals.

The detection of TGSCC with the simultaneous use of all three measuring techniques was more successful. During the TGSCC processes several simultaneous AE, EN and elongation events that were assigned to the SCC process were detected. Taking into account the time resolution of the measurements, it was concluded that the mechanical events occurred first. These mechanical breakages at the crack tip were detected by a sudden increase in elongation and a simultaneous increase in AE activity, whereas the following rapid fluctuations in current and potential were the consequence of the occurrence and passivation of a new surface at the crack tip.

It can be concluded that the detection of an individual SCC event by simultaneous measurements of EN and AE depends mainly on the discontinuity of the SCC processes. For this reason the detection of the more discontinuous TGSCC process is more reliable than the detection of the more continuous IGSCC process. Simultaneous use of the three techniques in the case of TGSCC makes it possible to obtain information about sequences of electrochemical and mechanical events, as well as information about the characteristics of these events. On the basis of the results so far obtained, it is expected that, with certain improvements to individual measuring systems, it would be possible to detect individual events of IGSCC, besides the monitoring of the cumulative progress of the IGSCC process. On the basis of the measurements some new data about SCC processes could be obtained, and this information would contribute to a better understanding of SCC mechanisms.

5 REFERENCES

[1] Gangloff, R.P., Ives, M.B. (Eds.). *Environmental-induced cracking of metals*. Houston, Texas: NACE Publication, 1990.

[2] Jones, R.H. *Stress-corrosion cracking – Material performance and evaluation*. ASM International Materials Park, 1992.

[3] Baboian, R., et al. (Eds.). *Corrosion tests and standards: Application and interpretation*. Philadelphia, PA: ASTM Publication, 1995.

[4] Liu, A. F. *Structural life assessment methods*, Ohio: ASM international, 1998.

[5] Kearns, J.R., Scully, J.R., Roberdge, P.R., Reichert, D.L., Dawson, J.L. (Eds.). *Electrochemical noise measurements for corrosion applications*, STP 1277. Philadelphia, PA: ASTM Publication, 1996.

[6] Isaacs, H., Bertocci, U., Kruger, J., Smialowska, S. (Eds.). *Advances in localized corrosion*. Houston, Texas: NACE International, 1990.

[7] Frankel, G.S., Stockert, L., Hunkeler, F., Boehni, H. Metastable pitting of stainless steel. *Corrosion*, 1987, 43, p. 429-436.

[8] Burstein, G.T., Mattin, S.P. Nucleation of corrosion pits on stainless steel. *Philosophical Magazine Letters*, 1992, 66, pp. 127-131.

[9] Legat, A., Dolecek, V. Chaotic analysis of electrochemical noise measured on stainless steel. *J. Electrochem. Soc.*, 1995, 142, p. 1851-1858.

[10] Yang, M.Z., Wilmott, M., Luo, J.L. Analysis of the electrochemical noise for localized corrosion of type A516-70 carbon steel. *Corrosion*, 1998, 54, p. 869-876.

[11] Lunt, T.T., Pride, S.T., Scully, J.R., Hudson, J.L., Mikhailov, A.S. Cooperative stochastic behavior in localized corrosion. *J. Electrochem. Soc.*, 1997, 144, p. 1620-1629.

[12] Legat, A., Osredkar, J., Kuhar, V., Leban, M. Detection of various types of corrosion processes by the chaotic analysis of electrochemical noise. *Mater. Sci. Forum*, 1998, 289-2, p. 807-811.

[13] Conde, A., Williams, D.E. Crevice corrosion and pitting detection on 304 stainless steel using electrochemical noise. *Mater. Corros.*, 1999, 50, p. 585-590.

[14] Wharton, J.A., Mellor, B.G., Wood, R.J.K., Smith, C.J.E. Analysis of electrochemical potential noise measurements on type 304 stainless steel in chloride solutions. *J. Electrochem. Soc.*, 2000, 147, p. 3294-3301.

[15] Kolman, D.G., Gaudett, M.A., Scully, J.R. modeling of anodic current transients resulting from oxide Rupture of plastically strained b + a titanium. *J. Electrochem. Soc.*, 1998, 145, p. 1829-1840.

[16] Yang, M.Z., Wilmott, M., Luo, J.L. Crevice corrosion behavior of A516-70 carbon steel in

solutions containing inhibitors and chloride ions. *Thin Solid Films*, 1998, 326, p. 180-188.

[17] Schneider, M., Galle, K., Pohl, H. Inhibitor efficiency inside small crevices investigated by electrochemical noise analysis. *Mater. Corros.*, 2003, 54, p. 966-973.

[18] Newman, R.C., Sieradzki, K., Woodward, J. in: A. Turnbull (Eds.). Current fluctuations during transgranular stress-corrosion cracking. *Proceedings of the Conference on Corrosion Chemistry within Pits, Crevices and Cracks, NPL*, Teddington, Middlesex, 1987, p. 203-212.

[19] Loto, C.A., Cottis, R.A. Electrochemical noise generation during stress corrosion cracking of alpha-brass, *Corrosion*, 1987, 43, p. 499-504.

[20] Loto, C.A., Cottis, R.A. Electrochemical noise generation during stress corrosion cracking of the high-strength aluminum AA 7075-T6 alloy. *Corrosion*, 1989, 45, p. 136-141.

[21] Flanagan, W.F., Wang, M., Zhu, M., Lichter , B.D. A fully plastic microcracking model for transgranular stress-corrosion cracking in planar slip materials, *Metall. Mater. Trans. A*, 1994, 25, p. 1391-1401.

[22] Wells, D.B., Stewart, J., Davidson, R., Scott, P.M., Williams, D.E., SCC of steel in dilute thiosulphate solution. *Corros. Sci.*, 1992, 33, p. 39-71.

[23] Watanabe, Y., Kondo, T. Current and potential fluctuation characteristics in intergranular stress corrosion cracking processes of stainless steels. *Corrosion*, 2000, 56, p. 1250-1255.

[24] Leban, M., Legat, A., Dolecek, V., Kuhar, V., Electrochemical noise as a possible method for detecting stress-corrosion cracking. *Mater. Sci. Forum*, 1998, 289-2, p. 157-161.

[25] Leban, M., Dolecek, V., Legat, A. Comparative analysis of electrochemical noise generated during stress corrosion cracking of AISI 304 stainless steel. *Corrosion*, 2000, 56, p. 921-927.

[26] Leban, M., Legat, A., Dolecek, V. Electrochemical noise during non-stationary corrosion processes. *Mater. Corros.*, 2001, 52, p. 418-425.

[27] Leban, M., Bajt, Z., Legat, A. Corrosion processes of steel in concrete characterized by means of electrochemical noise. *Electrochim. Acta*, 2004, 49, p. 2795-2801.

[28] MacDonald, D.D. Fate of the coupling current in stress corrosion cracking – Mechanistic and corrosion control implications. *CORROSION 2004 Conference*, New Orleans, Paper No. 04570.

[29] Gomez-Duran, M., MacDonald, D.D. Stress corrosion cracking of sensitized type 304 stainless steel in thiosulphate solution. II. Dynamics of fracture. *Corrosion Science*, 2006, 48, p. 1608-1622.

[30] Aballe, A., Newman, R.C., Cottis, R.A. Electrochemical noise study of stress corrosion cracking of sensitized 304H in thiosulfate. *CORROSION 2003 Conference*, San Diego, Paper No. 03403.

[31] Grabec, I. Acoustic emission. *Stroj. vestn.- Journal of Mechanical Engineering*, 1975, 3-4, p. 41-48. (In Slovenian).

[32] Nicholas, R.W. *Acoustic emission*, London: Applied Science Publishers Ltd, 1976.

[33] Miller, R.K., Hill, E., Moore, P.O. *Nondestructive testing handbook*, 3rd ed., Vol. 6, Acoustic emission testing. Ohio: American Society for Nondestructive Testing, Inc., 2005.

[34] Grabec I. Relation between development of defects in materials and acoustic emission. *Ultrasonic*, 1980, 18 (1), p. 9-12.

[35] Možina, J. *Acoustic emission induced by energy changes on metal surface*, PhD Thesis. University of Ljubljana, Faculty of Mechanical Engineering, 1980. (In Slovenian).

[36] Bajt, Ž. *Detection of cracking of construction elements with help of acoustic emission technique*, Master's Thesis. University of Ljubljana, Faculty of Civil and Geodetic Engineering, 1980. (In Slovenian).

[37] Grabec, I., Govekar, E., Susič, E., Antolovič, B. Monitoring manufacturing processes by utilizing empirical modeling. *Ultrasonics*, 1998, 36 (1-5), p. 263-271.

[38] Govekar, E., Gradišek, J., Grabec, I. Analysis of acoustic emission signals and monitoring of machining processes. *Ultrasonics*, 2000, 38 (1-8), p. 598-603.

[39] Newman, R.C., Sieradzki, K. Corrosion of acoustic and electrochemical noise in the stress corrosion cracking of a-brass. *Scripta Metall. Mater.*, 1983, 17, p. 621-624.

[40] Jones, R.H., Friesel, M.A., Gerberich, W.W. Acoustic Emission from intergranular subcritical crack growth. *Metall. Trans.* A, 1989, 20, p. 637-648.

[41] Jones, R.H., Friesel, M.A., Pathania, R. Evaluation of stress corrosion cracking initiation using acoustic emission. *Corrosion*, 1991, 47, p. 105-115.

[42] Munch, E., Duisabeau, L., Fregonese, M., Fournier, L. Acoustic emission detection of environmentally assisted cracking in zircalloy-4 alloy. *EUROCORR 2004 Conference*, Nice.

[43] Van Nieuwenhove, R., Bosch, R.-W. Acoustic emission detection during stress corrosion cracking at elevated pressure and temperature. *J. Acoustic Emission*, 2000, 18, p. 293-299.

[44] Vehovar, L. *Corrosion of metals and corrosion tests*. Ljubljana: self-publishing house, 1991. (In Slovenian).

[45] Newman, R.C. Developments in the slip-dissolution model of stress corrosion cracking. *Corrosion*, 1994, 50, p. 682-686.