Corrosion Science 55 (2012) 5–9

Contents lists available at SciVerse ScienceDirect

Corrosion Science

journal homepage: www.elsevier.com/locate/corsci

Analysis of acoustic emission signals generated from SCC propagation

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ARTICLE INFO

Article history:
Received 9 May 2011
Accepted 5 August 2011
Available online 9 September 2011

Keywords:
A. Alloy
A. Silver
C. Stress corrosion
Acoustic emission

ABSTRACT

This paper presents a comparison between the acoustic emission (AE) signals generated by the transgranular stress corrosion cracking (TGSCC) of Ag–10Au (at.%) single crystals and those originated by the intergranular stress corrosion cracking (IGSCC) of a polycrystalline silver–gold alloy under the same experimental conditions. No significant difference is found between the mean amplitude and rise-time of the AE signals registered during the propagation of TGSCC and those measured for IGSCC propagation. Results also show that the AE signals generated by either TGSCC or IGSCC propagation in three different alloys such as AISI 304 SS, α-brass and Ag–Au alloys show similar AE parameters and similar amplitude distribution.

1. Introduction

The acoustic emission (AE) technique is based on measurements of elastic waves, which are generated by a rapid release of energy within a material due to changes in local stress fields. This technique is used as a non-destructive evaluation technique for structural integrity monitoring and is considered a useful tool for materials research. Several studies have been reported during the last two decades concerning the development of AE for detection and characterisation of stress corrosion cracking (SCC) processes [1–16]. Cakir et al. [1], in their study on the SCC of type 316L stainless steel (SS) in Hank's solution, concluded that the AE activity in the plastic region is caused by the rupture of an oxide film or salt cap formed over active pits, by plastic deformation or by the pressure built up in the pit by trapped hydrogen bubbles. Similarly, Jones and Friesel [2], concluded that the AE activity during pitting and transgranular stress corrosion cracking (TGSCC) of type 304 SS is correlated with the pitting corrosion and not with TGSCC, dislocation motion or gas bubble formation. Jones and co-workers [3] studied the initiation of intergranular stress corrosion cracking (IGSCC) in type 304 SS using acoustic emission. They found that cracks of approximately 200 μm long by 100 μm deep could be reliably detected by acoustic emission. Proust et al. [4] investigated the possibility of detecting and characterising AE signals produced by the propagation of SCC in austenitic, ferritic and duplex stainless steels in hot chloride environment. They found a significant correlation between AE signals and SCC damages and proposed a discontinuous mechanism of cracking including a high velocity cleavage step. Cox [5] compared the acoustic emissions during the SCC of Zircaloy specimens with the metallographic and fractographic evidence of the events occurring at different stages of cracking. Two types of acoustic emission (continuous and discontinuous) were readily distinguishable, but no unique explanation of either was possible. Yuyama et al. [6] reported that several processes are responsible for acoustic emissions during SCC and corrosion fatigue. These include metal dissolution, hydrogen gas evolution, breakdown of thick oxide film, fracture or decohesion of precipitate and inclusions, plastic deformation by slip or twin, martensitic transformation, and micro/macro-cracking. Each of these can be distinguished by the amplitudes and frequencies of occurrence. Gerberich et al. [7] reported that during crack extension by mixed TGSCC + IGSCC mode by anodic dissolution in type 304 SS, AE was produced due to the transgranular fracture. They found a decrease in AE event rate with increasing area of IGSCC. Jones et al. [8] postulated that AE emissions during transgranular fracture were the result of ligaments that fracture behind the advancing crack front. Fujimoto et al. [9] analysed the fracture dynamics of chloride-SCC of type AISI 304 SS by AE source simulation method. They monitored no AE from the TGSCC, although a large amount of AE signals were monitored during the initiation and propagation of IGSCC. Sung et al. [10] studied the initiation of IGSCC in Inconel 600 by AE. They found that the minimum crack size detectable with AE is approximately 200–400 μm in length and below 100 μm in depth. Shaikh and co-workers [11] analysed the AE signals originated during the TGSCC of type 316LN SS in a $MgCl_2$ solution. Their results show that a surge in the AE counts and energy indicated initiation of SCC and that AE events occurred in bursts during crack growth.

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0010-938X/$ - see front matter © 2011 Published by Elsevier Ltd.
doi:10.1016/j.corsci.2011.08.014

Since SCC is a combination of electrochemical and mechanical processes, in some studies electrochemical noise (EN) and AE techniques have been simultaneously implemented. Newman and Sieradzki [12] performed a fundamental investigation of the TGSCC of brass by the combined use of EN and AE techniques. Intergranular SCC of sensitised type 304 SS exposed to aqueous sodium thiosulphate and sodium thiocyanate solutions was monitored simultaneously for EN and AE by Legat and co-workers [13–15]. According to these authors, the detection of simultaneous transients in the electrochemical current noise and acoustic emission bursts indicated the discontinuous nature of the monitored IGSCC processes. Zhang et al. [16] used the simultaneous application of localised corrosion monitoring, EN and AE techniques for the detection of SCC initiation and propagation in sensitised 304H SS in dilute tetrathionate solutions at ambient temperature.

This paper is a continuation of our previous work on characterisation and comparison of AE signals generated by the propagation of transgranular and intergranular stress corrosion cracks. Results obtained during the occurrence of SCC in α-brass and type 304 stainless steel showed that the AE activity during the propagation of TGSCC is generally one order of magnitude higher than the AE activity during the IGSCC propagation [17,18]. On the other hand, the mean values of the amplitude and rise time of the AE signals registered during both processes were found to be similar. An important result was to discover that the AE signals generated by either TGSCC or IGSCC propagation in two different alloys such as AISI type 304 SS and α-brass show similar AE parameters and similar amplitude distribution [18]. The aim of the present work is to further confirm such findings by making a comparison of the AE signals generated by the TGSCC and IGSCC of the same alloy in the same solution and in the same experimental conditions. Galvele et al. [19–21] have shown that polycrystalline silver–gold alloys, with gold contents from 2.2 up to 50 at.%, are susceptible to purely IGSCC in 1 M HClO4, 1 M KCl, 1 M KBr and 1 M KI aqueous solutions. Single crystals of Ag–Au alloys were found to be susceptible to TGSCC in 1 M HClO4 and 1 M KCl solutions under conditions similar to those at which IGSCC susceptibility was observed in polycrystalline alloys. In this investigation, the AE response of Ag–10Au (at.%) single crystals and a polycrystalline Ag–15Au (at.%) alloy during slow strain rate tensile tests (SSRT) performed under potentiostatic polarisation in a 1 M KCl solution has been measured and compared with those previously found for the SCC propagation of AISI 304 SS and α-brass.

2. Experimental methods

2.1. Materials and equipment

Experiments have been performed on Ag–10Au (in at.%) single crystals, obtained by the Bridgman technique. The single crystals had the shape of a bar, 70 mm long and a square cross-section with 3.3 mm side. Two 7 mm diameter spheres were grown at both ends of the bar to contain the specimens into the grips of a tensile machine [22]. For the experiments with polycrystalline alloy, the samples used were 0.8 mm diameter Ag–Au wires with a nominal composition of 15 at.% gold. The wires were annealed for 1 h in an argon atmosphere at 800 °C and water-quenched. The use of Ag–15Au polycrystalline wires to be compared with the Ag–10Au single crystals is valid because according to Maier et al. [20] polycrystalline Ag–*x*Au alloys with compositions ranging from Ag–2.4Au up to Ag–15Au show the same crack propagation rate (CPR) values. Immediately before the experiments the samples were degreased with acetone, washed with distilled water, rinsed with alcohol and dried with hot air.

Experiments were performed in a 1 M KCl solution at an anodic potential of 0.50 V_{SHE}. The selected potential value was based on results from a previous publication [22]. The solutions were prepared with analytical grade reagents and distilled water. All the tests were performed at room temperature. Constant potential slow strain rate experiments were performed with an Instron-1130 machine. The strain rates used were 3.5×10^{-5} and $3.5 \times 10^{-6}\ s^{-1}$. The cells used in these experiments have been described in previous publications [22,23].

Potentials were kept constant with a Lyp Electronica Potentiostat and measured with a saturated calomel reference electrode (+0.244 V_{SHE} at 25 °C) through a Luggin capillary. All potentials in the present paper are reported in the standard hydrogen electrode (SHE) scale. Before straining, the specimens were allowed to reach a stationary corrosion potential by a 15 min exposure to the solution. Afterwards, samples were exposed at the chosen potential, waiting for the current to reach a stationary value. Finally, the specimens were strained to rupture, while the load and the current were recorded and stored through an Advantech Data Acquisition Card model PCL-818H. After rupture, both the corroded surfaces and the fracture surfaces were observed with a Philips SEM 500 scanning electron microscope.

The system for recording and analysing the AE signals was composed of a two channel data acquisition instrument, storage media, sensors, and amplifiers. The data acquisition instrument was designed and built by the Instrumentation and Control Department, CNEA. Two piezoelectric 100–1000 kHz wide band sensors from the Physical Acoustic Corporation were used. The sensor output was amplified by a gain of 40 dB. One EA sensor was placed on the upper end of the tensile specimen while a second sensor, which acted as "guard" sensor, was attached to the upper grip of the tensile machine. In the experiments performed with wire specimen, the sensor was attached to the external wall of the corrosion cell. The dual detectors allowed discrimination between signals generated within the sample from those generated from the surroundings. The characteristic parameters of the AE signals measured as a function of time were the number of events and the event amplitude and rise-time. The number of events is the number obtained by counting each discerned acoustic emission event once. The amplitude is the peak voltage of the largest excursion attained by the signal waveform from an emission event. Signals with amplitudes below the selected threshold voltage are not recorded. The rise time is the time interval between the first threshold crossing and the signal peak.

2.2. Selection of the threshold voltage

In order to detect exclusively the AE signals caused by the propagation of the cracks, in another set of experiments the AE emissions resulting from other possible sources of AE were identified and its contribution to the overall AE activity was limited to less than 0.1 event s^{-1} by selecting a convenient threshold voltage value.

The mechanical noise was characterised by recording the AE signals generated during the crosshead motion of the tensile machine as a function of the threshold voltage. For the strain rates used in the present work, negligible AE activity has been detected for threshold voltage values higher than 190 mV.

The corrosion-associated AE signals were characterised by exposing samples of polycrystalline Ag–15Au alloy to a 1 M KCl solution in static conditions at the same potential value used in the SCC experiments. Simultaneously, the AE signals generated during the exposition were recorded as a function of time, using different threshold potential values. Fig. 1 shows the changes in AE activity measured using a threshold potential of 200 mV when a potential value of 0.50 V_{SHE} was applied to a Ag–15Au sample immersed in a 1 M KCl solution. Changes in current density have also been included. As soon as the potential was applied, a sudden increase in current density as well as a significant increment in AE