Corrosion Science 110 (2016) 273–283

Contents lists available at ScienceDirect

Corrosion Science

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

Monitoring of chloride stress corrosion cracking of austenitic stainless steel: identification of the phases of the corrosion process and use of a modified accelerated test

Fabienne Delaunois[a], Alexis Tshimombo[b], Victor Stanciu[a], Véronique Vitry[a,*]

[a] Université de Mons, Faculté Polytechnique, Service de Métallurgie, rue de l'Epargne, 56, Mons, Belgium
[b] Institut Supérieur de Techniques Appliquées, Section Mécanique, Kinshasa, Democratic Republic of the Congo

ARTICLE INFO

Article history:
Received 30 September 2015
Received in revised form 22 April 2016
Accepted 23 April 2016
Available online 25 April 2016

Keywords:
A. Stainless steel
B. SEM
C. Stress corrosion

ABSTRACT

A novel chloride stress corrosion cracking CSCC test was carried out on AISI 304L: the imposition of an anodic potential to shorten the test duration time. A stress is applied to promote cracks. Acoustic emission (AE) methods were used to characterize and monitor the phenomena. The degradation of the material was characterized by coupling of acoustic emission and electrochemical measurements. The evolution of current density and applied load was monitored to make links between the AE results and the various stages of CSCC. The fracture faces and the corrosion were observed by optical microscopy and SEM.

1. Introduction

Stainless steels (SS), singularly austenitic stainless steels ASS, are widely used in the petrochemical, nuclear and chemical industries due to their excellent mechanical properties and corrosion resistance. Under specific stress and environmental conditions, austenitic stainless steels (ASS) are susceptible to chloride stress corrosion cracking (CSCC) despite their excellent general corrosion resistance in water [1]. This susceptibility limits their use in warm and/or oxygenated environments at certain chloride levels [2]. Stress corrosion cracking (SCC) is one of the greatest risks of catastrophic failure when those materials are exposed to aggressive environments and subjected to mechanical loads. Due to this, the early detection of SCC stays an important topic of research with the goal to reduce potential corrosion damage and to increase operational safety.

The acoustic emission (AE) method can be considered as a passive non-destructive technique, because it usually identifies defects only while they develop during the test [3].

The contribution of acoustic emission (AE) to the study of SCC is undoubtedly important because this non-destructive technique is based on the detection of the rapid release of energy from localised sources within a material which can generate an elastic stress wave (between several kHz and a few MHz) [3]. Moreover, AE allows to detect corrosion processes such as electrochemical corrosion [4] and SCC [5–12].

During laboratory tests, AE provides significant and meaningful information on the evolution stages of the SCC phenomena. Actually, when a structure is subjected to an external stimulus (load, temperature, environmental conditions, etc.), it produces a fast energy relaxation at a localized source due to a sudden redistribution of stress in the material, and thus produces transient elastic waves [3]. The influence of various factors such as applied potential [7,8] and load [9,13] have already been considered.

Numerous investigations have already been done to link the evolution of pitting corrosion and the corresponding AE signals [14–19]. During pitting corrosion, the hydrogen-bubble evolution inside the pits is the most widely considered mechanism to be responsible for the AE signal [1,19–21]. The influence of the applied potential has also been evaluated [15,16]. Fregonese et al. [16] showed that the initiation and the propagation steps of the pits could be studied separately using a specific polarization procedure; actually, the initiation step of pitting corrosion was not significantly emissive, whereas the propagation step was characterized by the emission of resonant signals.

Chlorides are known to promote SCC in aqueous environments for austenitic stainless steels because of the aggressive nature of the Cl^- ion. Halide ions initiate cracks via pit formation on the sur-

* Corresponding author.
E-mail addresses: fabienne.delaunois@umons.ac.be (F. Delaunois), alexis.diaka@hotmail.fr (A. Tshimombo), victorioan.stanciu@umons.ac.be (V. Stanciu), veronique.vitry@umons.ac.be (V. Vitry).

http://dx.doi.org/10.1016/j.corsci.2016.04.038
0010-938X/

face [22]. The main mechanism of corrosion can be attributed to a dissolution film-rupture model SCC mechanism [2]. Pits formed on the ASS surface due to the crack of passive film create a localized area of stress concentration. As pits remain active and grow, they reach a critical length that allows a crack to propagate through the metal. Actually, the threshold stress needed to initiate cracking decreases until the applied stress is able to cause a crack to form and grow at the surface of the sample. To summarize, the mechanism can be described in terms of initiation stage, dominated by electrochemical mechanisms, and crack propagation stage in which both electrochemistry and metal cracking are involved [1].

The susceptibility of ASS to CSCC depends on a range of environmental variables that include chloride concentration, temperature and pH. Other variables are stress level (applied or residual stress), surface finish and metallurgical condition of steel [1]. Those variable can affect the initiation of CSCC by localized corrosion, and crack propagation when the rate of CSCC exceeds the rate of localized corrosion. Classical accelerated tests that promote cracking are generally used to assess the resistance to CSCC. The three most common tests are boiling acidified sodium chloride, evaporation of sodium chloride and boiling magnesium chloride, in order of increasing severity [1].

Jomdecha et al. [23] reported that various corrosion types, uniform, pitting, crevice, and stress-corrosion cracking, were identified by AE for AISI 304 stainless steel in NaCl environments. They showed that during the SCC test, the AE rate was high at the beginning of the test decreasing to the middle of the test and increasing again before the end of the test. Ramadan et al. [11] studied the SCC phenomena for AISI 316LN stainless steel in $MgCl_2$. They found that AE counts and energy parameters indicated the initiation of SCC, and bursts of AE events occurred prior to crack growth. During the propagation stage, the major source of AE was plastic deformation ahead of the crack tip.

The AE signal parameters generally considered for damage prognosis in SCC test are energy, amplitude, rise time, duration and counts. Moreover, other parameters are interesting to describe the type of damage: signal strength and number of detected hits. Recent AE systems are so powerful that AE signal waveforms can be recorded in real time, and frequency parameters are also interesting to study (from Fast Fourier Transformation FFT): the frequency centroid (FCOG that is the FFT center of gravity in kHz) and the peak frequency (FMXA that is the FFT peak frequency in kHz) [3].

Despite many advantages of the use of classical AE techniques such as the high recording and data storing speeds that facilitate fast visualization of the data (that makes the technique very economical), reducing a complicated signal to only a few parameters can be a significant limitation that could lead to errors in the interpretation of signal. It can be difficult to discriminate an AE signal from noise after the signal has been reduced to a few parameters. And in many experiments the parameters of AE signals are strongly related to the material and the geometry of the structure [3].

Fracture mechanisms testing has demonstrated that crack initiation can be described in terms of a critical stress intensity factor K_{ISCC} at a low crack propagation rate (i.e. 3.10^{-11} $m.s^{-1}$) [1].

This study introduces a new accelerated CSCC test where a load is applied to the sample in combination with an imposed anodic potential in order to shorten testing time. The damage process is followed simultaneously by chronoamperometry and AE. The AE signals of SCC of AISI 304L steel in hot chloride environment are detected and characterized using time and energy parameters (bursts counts, amplitude, rise time, acoustic activity, duration, peak frequency, frequency centroid, etc.). The presence of the different stages of corrosion damage occurring during CSCC was checked. The various stages were characterized and acoustic parameters were attributed to discriminate each emission phenomenon. To improve the identification of the various phases by the study and discretisation of the AE signals, and to avoid wrong interpretation of the recorded AE signal, the evolution of current and applied load during the accelerated test is recorded and analysed.

2. Experimental methods

2.1. Materials and sample preparation

CSCC tests were performed on two types of samples depending on the type of experiments: austenitic stainless AISI 304L steel wires with a diameter of 3 mm and length of 300 mm, and austenitic stainless AISI 304L steel rods with a diameter of 6 mm and an active length of 40 mm (Fig. 1). The choice of a specific geometry of sample for testing was linked to the experiments realized. In fact, the accelerated CSCC test applied in this study is derived from studies performed on steel wires [8,11]. Thus for AE measurements, wires were chosen to allow comparison with results from literature. However, normalized specimen used in usual CSCC tests are machined rods. Thus experiments were also performed on steel rods. Finally, for the recording of current density and stress evolutions, specimen with a sufficient work surfaces were needed to avoid measurement of artefacts and to increase precision.

All the specimens were wet ground up to 4000 grit with silicon carbide paper, cleaned with de-ionized water and then rinsed with

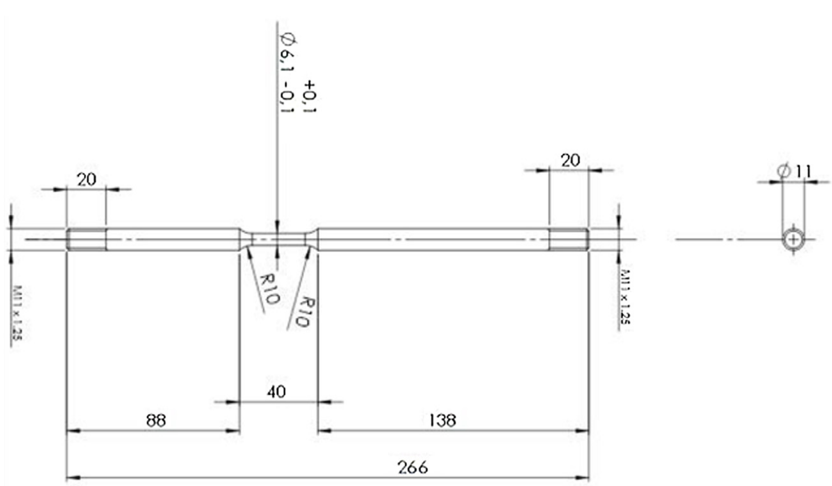

Fig. 1. Geometry of the rods used for SCC tests with current and load evolution (all lengths are given in millimeters).