IN-SITU OBSERVATION OF SULPHIDE STRESS CRACKING (SSC) INITITATION AND PROPAGATION IN 34CrMo4 STEEL BY USE OF ACOUSTIC EMISSION (AE)

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Abstract

Steel 34CrMo4 is commonly used for a variety of high strength application in many industrial areas. Some of such applications are exposed to environment containing H_2S and high humidity that can cause the SSC issue. Among such applications, high strength tubes, pipes and also high pressure steel cylinders belong. To prevent any possible failure of above mentioned applications, variety of destructive methods can be applied e.g. tensile testing, CVN testing and microstructural observations. However, such procedures demand the component extraction with subsequent samples preparations. All of these methods are highly cost inefficient and take significant amount of time. As an alternative method to detect a possible initiation and propagation of SSC, the AE testing and observation takes place. This method is one of a few possible in-situ methods to precisely distinguish early phases and propagation phases of such degradation process. This paper is focused on in-situ observation of entire SSC during simulated accelerated test on 34CrMo4 of strength reaching to 1215 MPa.

Keywords: 34CrMo4, sulphide stress cracking, mechanical properties, fracture surface, acoustic emission

1. INTRODUCTION

Many components operate in highly corrosive conditions such as high H_2S, humidity and chlorides contents or in highly demanding environment, e.g. high temperature. The in-situ detection of early phases of all potential degradation processes (SSC, stress corrosion cracking, hydrogen induced cracking, creep, etc.) is an essential to prevent any crucial damages or catastrophic failure of pressure components such as oil pipelines, steam pipelines, welded pressure vessels and seamless high-pressure steel cylinders. Acoustic emission in-situ detection of variety of pressure applications is only one of few known and functional nowadays. Such in-situ detection is an approach how to significantly reduce operating costs of above mentioned applications compared to a standard definition of a degradation level or extent that is based on the samples extraction and their analyzing. There is not much of evidence regarding the in-situ observation of SSC or other degradation processed by AE, however Smanio [1] analysed low alloyed steel and Tsai [2] used a very similar approach however in case of a high carbon steel. To set this experimental procedure, samples from specially heat treated seamless high-pressure cylinders (tensile strength up to 1215 MPa) were extracted and especially customized-machined for an accelerated SSC-A test. This paper is based on the 6 steps of an investigation - chemical analysis of an experimental material, testing of mechanical properties, micro-purity and grainsize observation, microstructural analysis, in-situ acoustic emission observation during the SSC-A test, fractures analysis. Results of all above analyses are evaluated and the potential use of use the AE in industrial applications is proposed.

2. EXPERIMENTAL PROCEDURES AND RESULTS

Experimental procedures started with chemical composition analysis of the 34CrMo4 steel after the heat treatment based on the austenitization at 890 °C with subsequent tempering showed following values (wt. %): 0.35 C, 0.31 Si, 0.65 Mn, 1.05 Cr, 0.018 Ni, 0.20 Mo, 0.004 S and 0.08 P.

Following, second step was based on the mechanical properties examination that started with an extraction and precise machining of round bars for the tensile properties, yield strength (YS), tensile strength (TS) and elongation (El.) testing. Subsequently square shaped specimens were extracted and precisely machined for CVN and Brinell hardness (HBW) testing. For the tensile properties the Zwick / Roell Z 250 machine was used according to the EN ISO 6892-1. The hardness testing machine M4U750 was used to obtain hardness values according to the EN ISO 6506-1. For the notch toughness testing, the RKP 450 Charpy Impact Testing Machine was used. This procedure was carried according to the ISO 148-1 at -50 °C in transverse and longitudinal direction. All mechanical properties results are shown in **Table 1.**

Table 1 Mechanical properties after HT (average values from 2 samples)

Spec.	YS [MPa]	TS [MPa]	El. [%]	CVN $_{\text{transverse -50 °C}}$ [J.cm^{-2}]	CVN $_{\text{longitudinal-50 °C}}$ [J.cm^{-2}]	HBW (2.5 / 187.5) [-]
34CrMo4	1110	1215	14.4	36	40	390

The third step part of an investigation procedure was metallographic observation. This was focused on microstructure after the heat treatment, micro-purity and grain size evaluation using the light microscopy (Olympus IX70). Metallographic sample of the cylinder after the heat treatment was prepared in transverse direction by the grinding; polishing and etching in Nital and/or in sodium hydroxide. **Figure 1** reveals the presence of very fine microstructure of tempered martensite together with the supressed segregation bands.

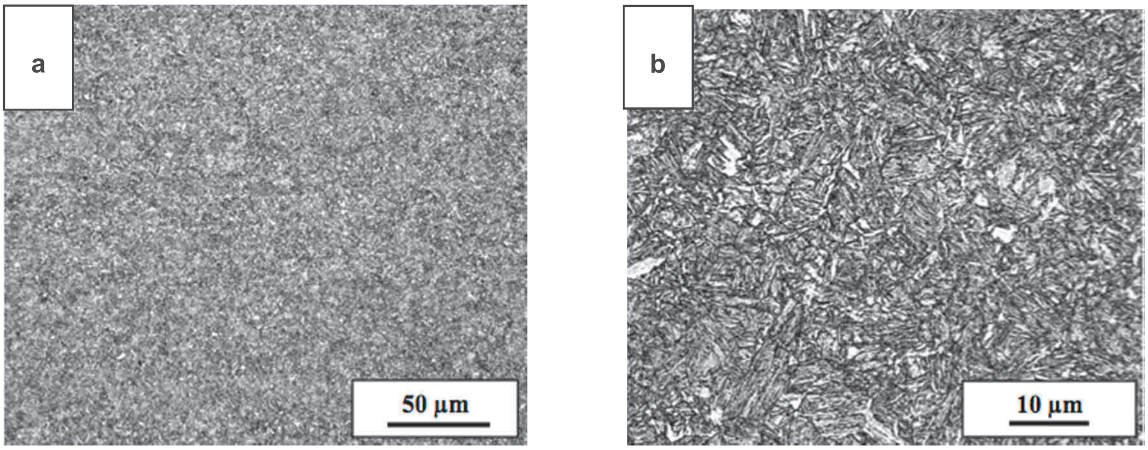

Figure 1 Tempered martensite in central region of wall thickness (transversal direction) a) general view, b) detail

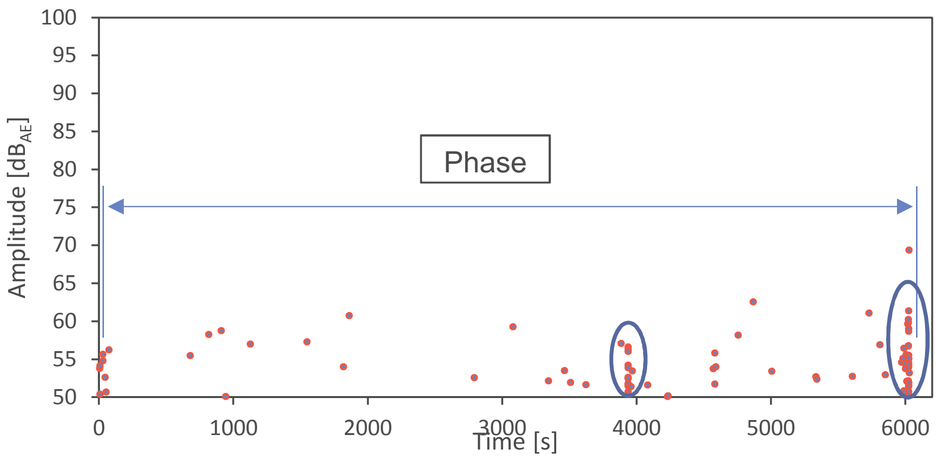

Figure 2 Acoustic emission signals (events) during the exposure of specimen number 1

Second stage of the metallographic observation was the material integrity investigation of sample number 1. This sample was exposed to the corrosive environment and unloaded when first AE signals were detected (see **Figure 2**). **Figure 3** shows cavities and cracks that occurred during the exposure time. **Figure 4** presents data obtained during the loading of the sample until the final fracture. Grain size according to EN ISO643 and micro purity according to ISO 4967 evaluations are shown in **Table 2.**

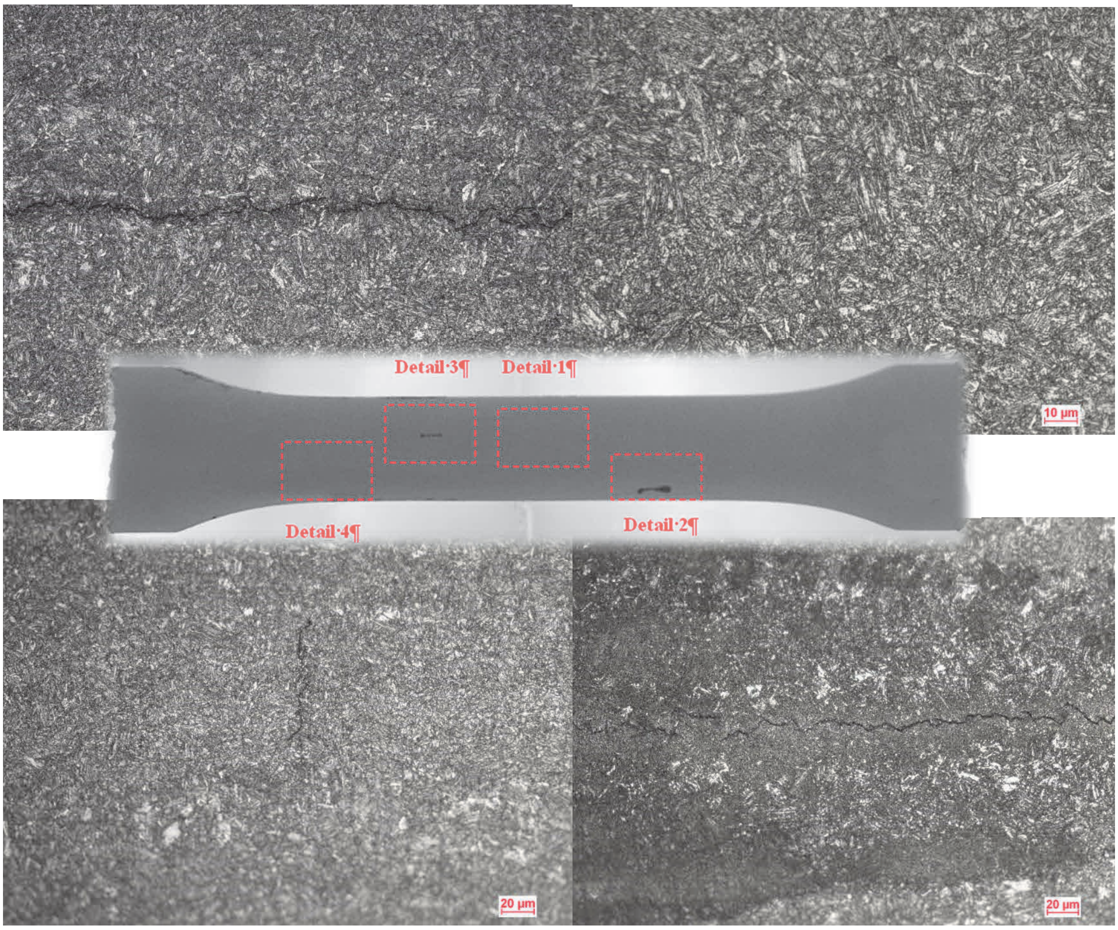

Figure 3 Cavities and cracks in SSC-A sample after before final crack

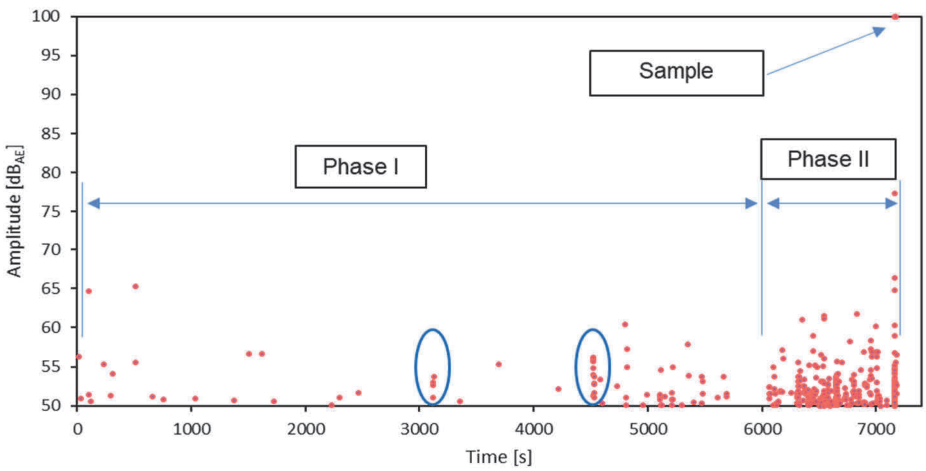

Figure 4 Evaluation of the cracking stages by AE until the final crack stage of specimen number 2

Table 2 Evaluation of micro purity and grain size of final products

Spec.	Sulphides fine/coarse	Oxides banded fine/coarse	Oxides formable fine/coarse	Globular oxides fine/coarse	Coarse globular oxides fine/coarse	Grain size
34CrMo4	-	0.2/0.1	0.1/0.1	0.2/0.1	0.2	10

Fourth step of the investigation process (accelerated SSC-A testing) was based on the designing of special extended SSC specimen for the method A according to NACE TM0177-96. Specimen had to fulfil all requirements of mentioned standard plus the upper cylindrical part had to be extended for the AE sensors connection**.** The overall setup of adjusted specimens in chambers is shown in **Figure 5**. Three machined samples were subsequently exposed to the solution of distilled water buffered with 0.5 % sodium acetate tri-hydrate and continuously saturated by the 100 %. The applied load was equal to 0.6 of minimum prescribed YS value of tested cylinder material and pH of corrosion solution was set up exactly to 4.0. This procedure is a proposed combination of two NACE TM0177-96 and ISO 11439 standards to simulate most severe corrosive conditions for seamless high-pressure steel cylinders. Since the beginning of an exposition process, both samples were continuously monitored by use of Vallen AMSY-6 multi-channel AE system, equipped with a passive piezoelectric AE sensor Vallen VS30-V, which is especially suited for tank floor corrosion, leak detection or for partial discharge detection thanks to its flat frequency response within the 20÷80 kHz interval. The AE signal was subsequently amplified using the Vallen AEP5 preamplifiers with 34 dB gain. AMSY-6 AE unit is capable of processing both types of AE signal, namely the burst and the continuous acoustic emission. In this case, the continuous data acquisition approach has been used in order to record any AE activity, whose amplitude exceeded the 40 dB_{AE} threshold. In addition, for each AE hit, there has been created a transient record including the parametric analysis (hit duration, rise time, number of counts etc.). This information has helped us to perform and effective filtering process, which was subsequently applied to the measured dataset.

For the micro-fractography investigation that was a fifth step of entire investigation process, the SEM (scanning electron microscope) SEM JEOL JSM-6490 LV equipped with X-ray analyser EDA) was used. Crack surfaces of SSC specimens after the accelerated SSC test were only cleaned by demineralized water and subsequently cleaned by use of an ethanol.

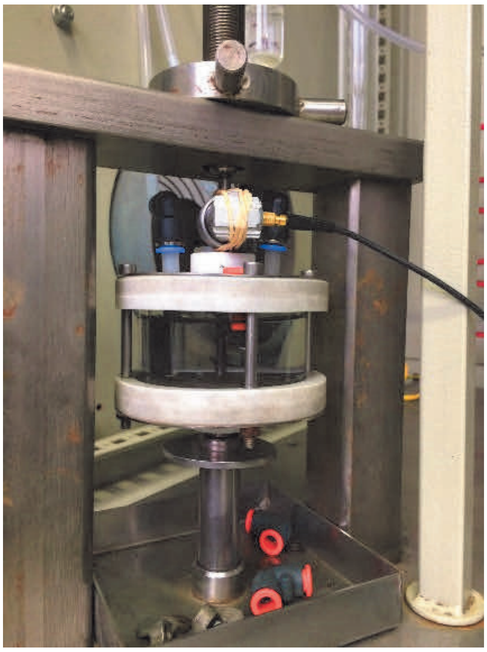

Figure 5 AE Setup of adjusted specimens during the SSC-A exposure

Crack surface of an investigated specimen (number 1) revealed typical cracks distribution and fracture surface morphology of specimens after SSC-A test exposure. **Figure 6** shows general fracture surface (SEM) together with details of defects that were revealed.

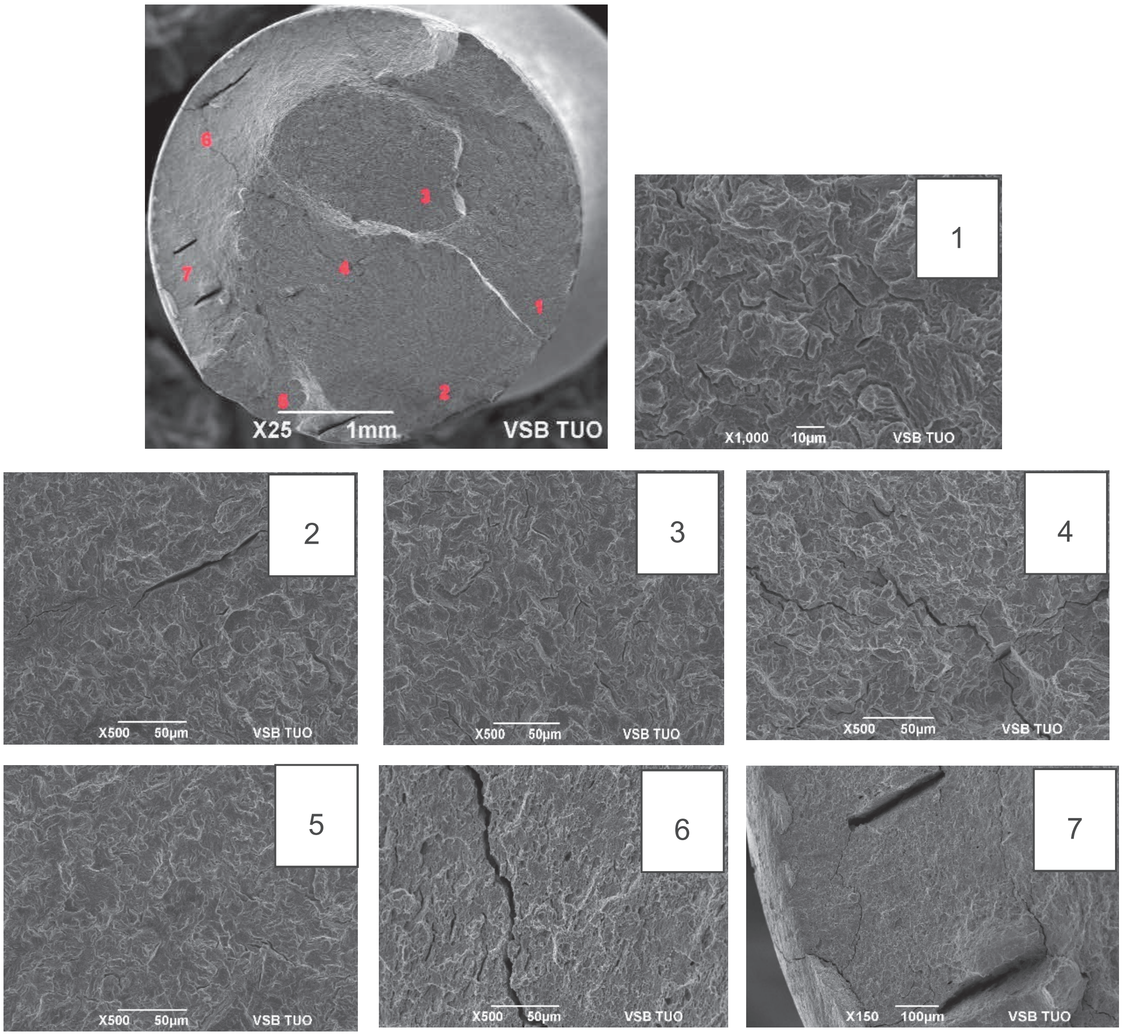

Figure 6 Fracture surface of specimen number 2

3. DISCUSSION OF ACHIEVD RESULTS

Presented material 34CrMo4 (high strength low alloyed steel) very similar by properties to the one investigated by Tsai [2] revealed highly consistent results of degradation process (SSC) detected by AE as **Figures 2 and 4** revealed. The crack initiation and propagation stages were clearly distinguishable and were supported by **Figure 3** that clearly revealed certain defects that were previously detected by AE during the accelerated SSC test that highly complies with results obtained in [1,3]. Presented findings (**Figure 6)** of crack initiation and subsequent propagation was also supported by the SEM fracture analysis that showed also the presence of crack initiation stage, propagation and clearly also position where the final crack took place. These finding highly correlate with the work of Calabrese [4] and Filippov [5].

4. CONCLUSION

This paper clearly describes the in-situ SSC degradation process monitoring by use of an AE. Entire procedure was based on the evaluation of the AE signals (hits) obtained during the accelerated SSC-A test and confronted with the metallographic analysis to determine feasibility of such method to be applied in industrial sphere for continuous in-situ monitoring of high pressure components exposed to the corrosive environment containing H_2S and high humidity. Obtained results fully support the hypothesis that this method may be used in industrial field based on the positive findings of correlation of found cracks by metallographic and SEM analysis and detected AE events (hits) during the test.

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