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Mechanism detection of stress corrosion cracking by acoustic emission and effect of manufacturing process on AE signals

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Stress corrosion cracking could occur wherever a specific corrodent and sufficient tensile stresses coexist. In the objective to monitor online the SCC on real structures, it seems reasonable to characterize and recognize acoustic emission during static U-bend tests. The present study is concerned with static tests on 304 stainless steels in two different media (5% HCl and 5% H₂SO₄) in order to find a criterion to distinguish, the two different mechanism of SCC (anodic dissolution and hydrogen embrittlement) by characteristic parameters and waveform analysis of AE signals. In the next stage, effect of residual stress on stress corrosion cracking in chloride solution has been studied. Three samples have been prepared: one of them was stress relieved; the second one was made sensitive to SCC by heat treatment and the last one was cold worked by rolling. Cumulative count, amplitude distribution and waveform analysis were selected as AE-parameters. AE with amplitudes ranging from 39 to 65 dB with different counts and energy occurred during SCC of SS-304 at room temperature. According to the results, acoustic emission is able to be used as robust technique for mechanism detection of SCC. In addition it can be used to measure the effect of residual stress in manufacturing process on SCC.

1 Introduction

The effects of residual and applied stresses and corrosive environments in service are closely interrelated. The more highly stressed (higher energy) regions of a metal will become anodic and corrosive cells will be set up due to differences in local stress levels. Cold worked regions, for example tube or sheet bends and cut edges, will be corroded in preference to uniform parts of sections in the same way that grain boundaries are attacked more than grain interiors on the microscopic scale. Stress corrosion cracking is an insidious type of failure as it can occur without an externally applied load or at loads significantly below yield stress. Thus, catastrophic failure can occur without significant deformation or obvious deterioration of the component. Pitting is commonly associated with stress corrosion cracking phenomena. The typical form of stress corrosion cracking is shown in Figure 1.

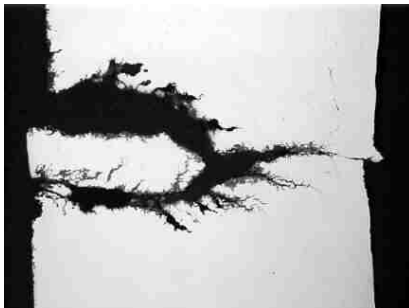


Figure 1. Typical stress corrosion cracking

The periodic inspections of structural materials and components are very important to detect and evaluate their degradation. The stringent inspection regulations necessitate the use of more advanced and reliable non-destructive testing (NDT) methods for assessing structural integrity of parts, specifically to detect the damage at its initiation stage, in other words, much before the defects detected by conventional NDT techniques. The structural integrity of the components is affected by various types of material degradation processes including stress corrosion cracking (SCC), pitting corrosion, thinning, creep and fatigue damage.

Among various structural materials, austenitic stainless steels are most commonly used in a wide variety of industries including power, chemical, petrochemical, process and nuclear. These steels exhibit excellent resistance to general corrosion, adequate mechanical properties and good fabric ability. However, these steels suffer from attack by localized corrosion, such as pitting corrosion, crevice corrosion, intergranular corrosion (IGC) and environment-induced cracking. A number of failures of stainless steel components have been attributed to these localized corrosion processes, particularly to stress corrosion cracking (SCC), in nuclear and petrochemical industries. Most of these failures have been attributed to sensitization of austenitic stainless steels [1].

A number of non-destructive evaluation techniques such as X-radiography technique, ultrasonic technique, eddy current testing technique, magnetic particle inspection technique and liquid penetration techniques are available. Most of these techniques are used off-line i.e. during shut down periods and not during operation of the component and, hence, can be both time consuming and expensive. However, the aspects of economic losses and safety due to corrosion have necessitated the use of NDT techniques for on-line monitoring of corrosion in plants. The acoustic emission (AE) technique presents the most viable tool for on-line monitoring of corrosion. AE has been developed for more than three decades as a non-destructive evaluation technique for structural integrity monitoring and for leakage detection in pressure systems, in addition to being a useful tool for materials research. The acoustic emission phenomenon is the result of transient elastic wave propagation generated by a rapid release of energy within a material due to changes in local stress and strain fields. These elastic waves propagate over a wide range of frequencies ranging from audible frequencies to frequencies in MHz range. The acoustic emission technique (AET) is a passive and non-intrusive technique and remote monitoring is possible. AET is widely used for structural integrity monitoring applications because of its possibility for detection and location of dynamic defects. It provides dynamic information on the mechanism involving the failure. The success of AET, when used as a tool for NDE depends on how well the sources of AE detected during material damage can be interpreted. AE can occur due to atomistic processes like dislocation motion or due to macroscopic processes like deformation, corrosion and

crack growth [2]. Some processes such as film cracking, gas evolution, hydrogen migration, plastic zone formation, discontinuous cracking during SCC and hydrogen cracking produce AE since all these processes strain the lattice[3].

Corrosion processes present an attractive proposition for detection and characterization by AE technique.

Although acoustic emission (AE) technique has been used extensively to study stress corrosion cracking and has been considered a promising technique already some thirty years ago, there are several factors preventing the advancement of this technique. One of these factors is source pattern recognition, as there exist some difficulty in characterizing parameters.

In previous work F.Ferrer et.al studied the SCC of static U-bend on a 316L stainless steel in hot concentrated magnesium chloride media but they could not recognize and distinguish the source of AE signals and SCC phenomena [4].

In this paper, AE monitored SCC tests were performed for U-bend on stainless steel in two different solution (5% HCl and 5% H₂SO₄) in order to find a criterion to distinguish, the two different mechanism of SCC by characteristic parameters and waveform analysis of AE signals.

2 Theory of stress corrosion cracking

There are many models for the mechanism of environmental sensitive cracking, including stress corrosion cracking (SCC) so far. For aqueous solution, crack growth is either controlled by anodic dissolution or hydrogen embrittlement at crack tip [5].

Anodic stress corrosion cracking occurs in the passive condition of material, i.e. when the metal surface is covered by a passive or protective layer. Most model concepts assume that an accelerated anodic disintegration of metal takes place on a crack tip kept free from protective layers by chemical and mechanical effect (see fig1). According to this theory freshly created crack edge must be immediately re-protected by the formation of passive or covering layers because otherwise crack fatigue could occur. In this way stress corrosion cracking spread through the material like an "electromechanical knife"[6].

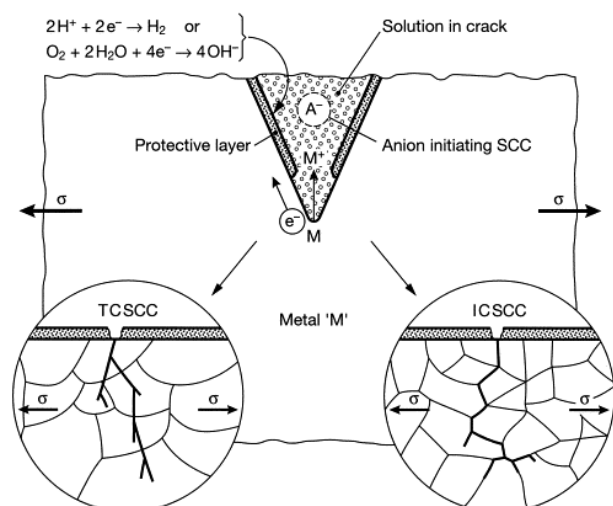
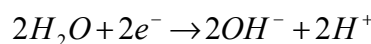
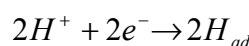
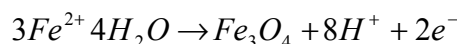


Figure 2. mechanism of common SCC by anodic solution

Hydrogen-induced stress corrosion cracking (HISCC) is transition form between the classical hydrogen embrittlement and stress corrosion cracking. When a building component or system is subjected to corrosion created by development of hydrogen, cathodically isolated hydrogen diffuse into the material and cause local embrittlement. The hydrogen develops either in oxygen-free, neutral or alkaline medium by the reduction of water molecules



or as result of hydrolysis of corrosion products (in pitting location, narrow cracks), for instance :



Molecular hydrogen is less damaging for steel than atomic hydrogen due to its high dissociation constant at room temperature. When kinetic inhibition of recombination exists, as is the case with the presence of, for instance, sulphides, arsenic, selenium and phosphorus compounds, considerable partial pressure can occur due to the accumulation of adsorbed hydrogen atoms on the surface.

The diffusion coefficient of steel is between 10^{-4} and $10^{-5} \text{ cm}^2/\text{s}$ which is comparable with diffusion coefficient in fluids. Therefore as soon as the hydrogen has penetrated through the phase boundary into the metal, it is capable of penetrating to a depth of 100 μm into the material. Thus a lattice deformation takes place and also an accumulation of hydrogen on the boundary surface (e.g. at grain boundaries, pores and inclusions) [7].

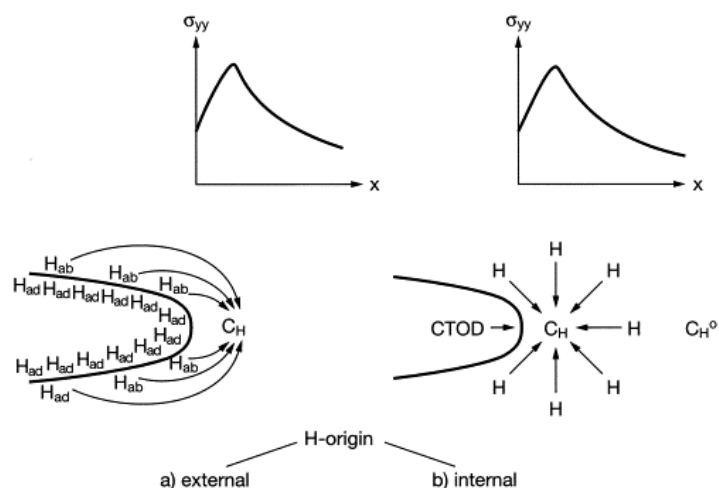


Figure 3 Hydrogen diffusion in a stressed specimen at externally (a) or internally (b) supplied hydrogen

It is now generally accepted that SCC of stainless steel in Cl^- containing aqueous solution is controlled by an anodic dissolution mechanism and SCC of stainless steel in SO_4^{2-} containing aqueous solution is controlled by hydrogen embrittlement but there is no existing criterion to distinguish them [5].

3 Experimental procedure

The U-bends are prepared with reproducible conditions. They come from a same 304 austenitic stainless steel plate of 2mm thickness (see alloy composition in table 1). Samples were prepared without crack and according to ASTM G30. Before starting the test, samples were investigated by magnetic particle method and no crack has been found.

The experiments duration for all samples was 24 hours at room temperature. In first step, the U-bend samples were immersed in two different media (5% HCl and 5% H₂SO₄) in order to find a criterion to distinguish, the two different mechanism of SCC (anodic dissolution and hydrogen embrittlement). The experimental device settled for the SCC tests is presented on Fig. 3.

Table 1. Composition ranges for 304 stainless steel

Grade		C	Mn	Si	P
304	min.	-	-	-	-
	max.	0.08	2.0	0.75	0.045
		S	Cr	Ni	N
304	min.	-	18.0	8.0	-
	max.	0.030	20.0	10.5	0.10

In the next stage, effect of residual stress on stress corrosion cracking in chloride solution has been studied. Three samples have been prepared: one of them was stress relieved; the second one was made sensitive to SCC by heat treatment and the last one was cold worked by rolling. Sample is heat treated at 873 K for 4 hours (table 2).

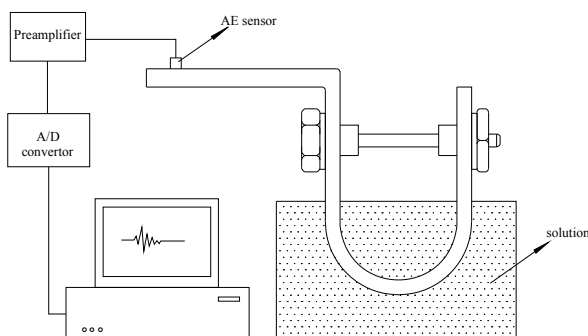


Fig.3. Experimental device.

Table 2. Samples characteristics.

samples	Characteristics	Corrosive solutions %
Reference sample	Not bended, Not heated, Not worked	5% HCl
U-bend	Bended, Not heated, Not worked	5% HCl
Heat treated	Bended, Heat Treated at 873 K for 4 hr	5% HCl
Cold worked	Bended, r =20%	5% HCl

The acoustic emission software used was the MI-TRA (transient recorder package) from Physical Acoustics Corporation 2001. A wide band sensor, model PAC WD, with an operational range of frequencies between 20 and 1000 kHz and a resonant frequency at about 125 kHz was used. The surface of the sensor was covered with silicon grease in order to provide good acoustic coupling between the specimen and the sensor.

A PAC preamplifier, model 1220A, with a 40 dB gain and a 100–300 kHz bandwidth was used for signal conditioning. The main data acquisition board was a PAC AEDSP-32/16 PC based two-channel A/D system with a sampling rate of 8 MHz. PAC AE application software mistras3 was utilized to acquire the raw data and perform basic data processing and the conventional AE analysis. The threshold was 42 dB.

4 Results

4.1 AE evolution compared to SCC activity

4.1.1 U-bend in 5% HCl and 5% H₂SO₄

Fig. 4 shows the time evolution of the cumulative acoustic activity recorded during the tests performed in 5% HCl and 5% H₂SO₄ mediums. It can be seen the acoustic activity of SS 304 in 5% HCl is divided to two separate sections firstly passive zone (approximately 0-20000 s), secondly active zone (after more than 20000 s). The characteristic of passive zone is returning to thermodynamic and kinetic of corrosion reaction. The released activation energy of corrosion reaction in passive zone, prepare activation energy of active zone. According to the theory of anodic dissolution mechanism, the passive film on the surface is unstable and this phenomenon is confirmed by visual inspection as shown in Fig.4. The acoustic activity of SS 304 in H₂SO₄ like HCl has two separate sections but firstly the active zone is occurred. On the other hand, the passive zone is activated at the second stage (after less than 20000 s). In H₂SO₄ solution, the active zone is due to hydrogen migration into plastic deformation zone in lattice of stainless steel. The capacity of lattice is finite so after specific time duration the rate of migration is decreased and the passive zone is appeared. Another reason of appearing the passive zone according to the theory of hydrogen embrittlement is stabilization of passive film and this phenomenon is confirmed by visual inspection as shown in Fig.4.

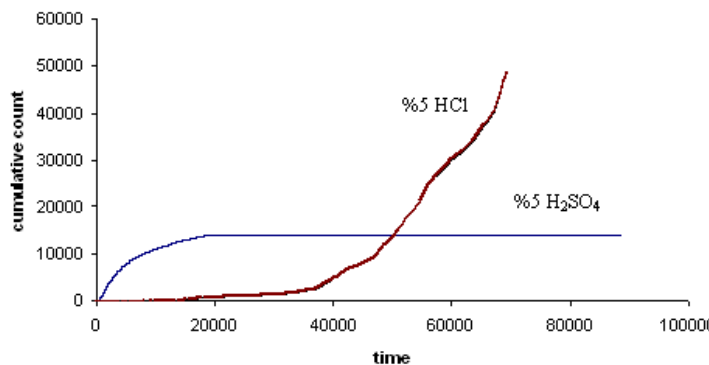


Fig.4. Time evolution of the cumulative acoustic activity recorded during the tests performed in 5% HCl and 5% H₂SO₄ mediums.

Fig 5. Presents the typical wave form of stress corrosion of stainless steel 304 in (a) 5% H₂SO₄ and (b) 5% HCl at the same time respectively. It can be seen the amplitude and energy of waveform of SS 304 in 5% H₂SO₄ are higher than SS 304 in 5% HCl. It means the amount of energy is produced by hydrogen diffusion in hydrogen embrittlement mechanism which is occurred in H₂SO₄ is higher than the energy is produced in anodic mechanism which is occurred in HCl. These results is according with Yuyama et al. researches on amplitude distribution.[8]

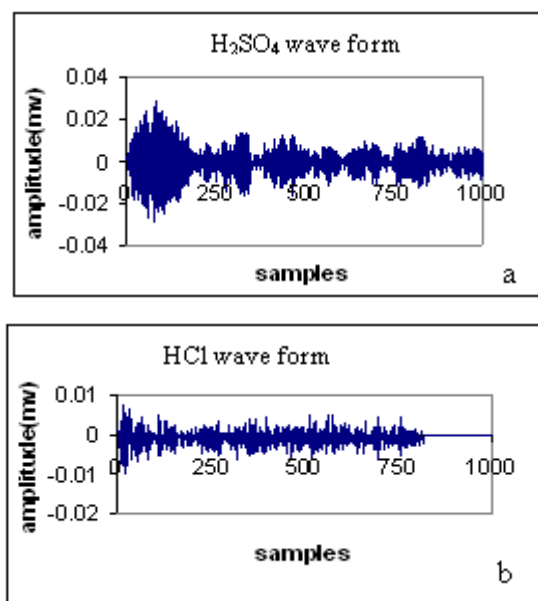


Fig.5. Typical waveform of stress corrosion of stainless steel 304 in (a) 5% H₂SO₄ and (b) 5% HCl

4.2 The effect of manufacturing process

Fig. 6 shows the time evolution of the cumulative acoustic activity recorded during the tests those characteristics shown in table 2. All of the samples in chloride solution have a same behavior it means they have two separate section (passive zone and active zone). The critical point is that the duration of passive zone and the slope active zone in all of the curves are different. The without stress sample has certain passive time and active slope. The

manufacturing processes can be changed the condition and affect on the passive time and active slope. According to Fig.6 the cold worked curve slope in active zone is the sharpest. It is due to increasing of the internal energy of sample by cold work process. In addition heat treated passive zone duration is the smallest. Because of the formation of chromium carbides involves long-range diffusion, it accelerates corrosion process.

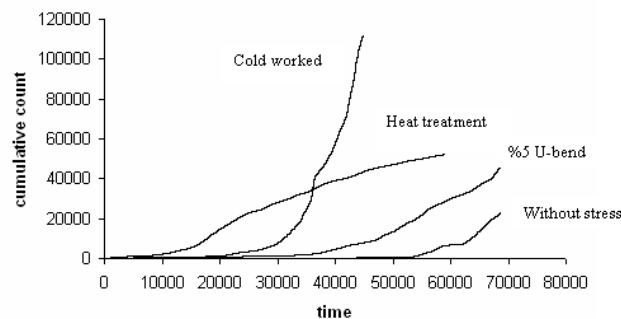


Fig. 6. Effect of manufacturing process on acoustic emission signal

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5 Conclusion

Some important findings obtained are summarized below:

- 1- Both of tests of SS 304 in 5% HCl and %5 H₂SO₄ have two separate zones (passive and active) while in 5% HCl passive film is unstable whereas in 5% H₂SO₄ passive film is stable.
- 2- Energy and amplitude of hydrogen embrittlement mechanism of SS 304 in 5% H₂SO₄ solution are more than anodic dissolution mechanism of SS 304 in 5% HCl
- 3- The cold worked curve slope in active zone is the sharpest and the heat treated passive zone duration is the smallest.
- 4- According to the results, acoustic emission is able to be used as robust technique for mechanism detection of stress corrosion cracking. In addition it can be used to measure the effect of residual stress in manufacturing process on stress corrosion cracking.

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