JOURNAL OF MATERIALS SCIENCE **37** (2002) 2707–2712

Sensitivity of acoustic emission for the detection of stress corrosion cracking during static U-bend tests on a 316L stainless steel in hot concentrated magnesium chloride media

F. FERRER, E. SCHILLE, D. VERARDO, J. GOUDIAKAS
ATOFINA, CRDE, Water and Corrosion Laboratory, BP 61005, 57501 Saint-Avold, France
E-mail: frederic.ferrer@atonfina.com

A lot of laboratory studies have shown that acoustic emission (AE) is a well suited technique to monitor stress corrosion cracking (SCC) during different kind of tests like slow strain rate or constant load tests. In principle, SCC could occur wherever a specific corrodent and sufficient tensile stresses coexist. Even if the medium is low corrosive and the stresses are not very intense, the damage can conduct to rapid and catastrophic damages because there is a synergistic interaction between corrosion process and mechanical effect. In the objective to monitor on-line this phenomenon on real structures, it seems reasonable to characterize acoustic emission during static U-bend tests which are, in term of stress intensity, very representative of what happens on plant. The present study is concerned with static tests conducted on a 316L stainless steel in hot concentrated $MgCl_2$ media. The high sensitivity of AE to detect active cracks during their early stages of propagation is evidenced.

1. Introduction

Stress Corrosion Cracking is one of the most important damaging corrosion problem in a lot of chemical, petrochemical or nuclear industries. Unpredicted failures occur in service which have very important, economic, safety, health and environmental consequences. In fact, a considerable amount of research has been devoted all over the world, during the past three decades to understand the fundamental mechanisms involved in SCC and develop new approaches for monitoring or improving life prediction [1]. A lot of recent studies have shown that AE is very powerful to study corrosion phenomena [1–8]. In the context of SCC study, AE has been used to monitor a large variety of materials including stainless steels, mild steels, high-strength steels, copper, aluminum, titanium, zirconium and uranium alloys [1, 9–15].

The most part of works dealing with AE to monitor SCC phenomenon concern acoustic emission study during slow strain rate tests or static load tests [1]. A lot of results show that in this case, SCC propagation produces a large number of AE bursts which depends on the material tested and on the strain level. The aim of this introduction isn't to give an extensive analysis of this kind of work. Some fundamental aspects about the emissive mechanisms during SCC can be found for example in the paper of Mazille and Rothéa [1].

The objective of the present paper is to show that AE is very powerful to monitor SCC during static U-bend tests. Those kind of tests have been chosen because they are more representative of the industrial reality that slow strain rate tests [16]. The first part of the paper is related to the experimental conditions we used. The second part gives some significant results about the sensibility of AE for SCC detection. A third part concerns the AE signals characterization.

2. Experimental

The experimental device settled for the SCC tests is presented on Fig. 1. It is essentially composed of a 2 liters glass vessel which is equipped with 3 accesses. One for the measure and the control of temperature, one for the insertion of U-bends and one for the fastening of a refrigerant. The contained liquid medium (1 liter) is agitated by the mean of a magnetic stirrer.

In the presented configuration, 3 metal samples can be inserted in the media. These samples are composed of a U-bend on which is welded a special wave guide for the AE monitoring. The upper part of the waves guides is a cone allowing the fastening of an acoustic sensor.

The U-bends are prepared with reproducible conditions. They come all from a same 316L austenitic stainless steel plate of 2 mm thickness (see alloy composition in Table 1). The U part is fastened to the straight part by welding. For the presented work, 3 different metallurgical preparations were used for each test :

– *Reference sample*: after U-bend making and welding with the wave guide, it is stress relieved by heating at 1050°C during one hour and then, water quenched. In order to verify that there were no more significant residual stresses on such prepared

TABLE I Composition of the 316L austenitic stainless steel used in this work

Element	C	Si	Mn	Cr	Mo	Ni
Wt%	<0.03	0.50	1.50	17.80	2.60	12.70

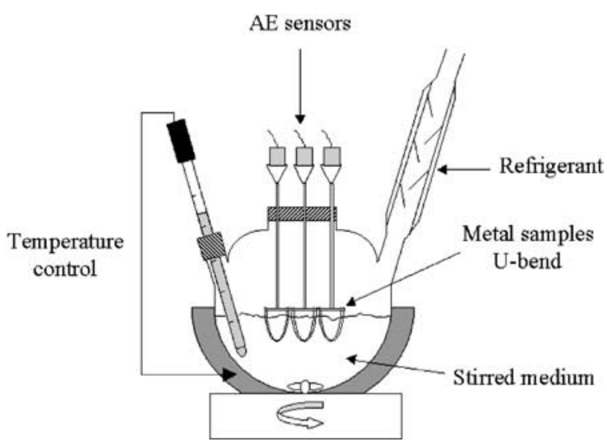

Figure 1 Experimental device.

samples, some of them have been immersed during 3 months in 5% sodium chloride at 70°C. In comparison, not stress relieved U-bend samples were immersed in the same medium. After 3 months, the stress relieved samples were free of cracks while the not stress relieved were completely cracked. It can be concluded that the as mentioned stress relieved sample is very low sensible to SCC.

– *Stressed sample*: not stress relieved.
– *Hyper stressed sample*: the U-bend is made from a part of the 316L plate which had been preliminary shot peened. Then it is not stress relieved. The work of Bouzina have clearly shown that a sample stressed (e.g. U-bended) after shot peening is more sensitive to SCC that a sample stressed without this preliminary mechanic surface modification [17].

Before each test, the surface preparation was the same whatever the metallurgical state. The U-bend was cleaned and passivated in a $HNO_3$15%/NaF2% treatment bath during 20 minutes at 50°C under agitation.

Tests have been conducted in concentrated magnesium chloride ($MgCl_2$) media. Two different concentrations have been studied. A solution of 44% $MgCl_2$ in distilled water and a 33% one, which is less corrosive. In the two cases, temperature has been maintained just under the boiling point. The media were naturally aerated.

The liquid volume was constant during the tests. Only the U part of the samples were immersed in the medium. This precaution has been taken in order to avoid SCC of the U-bend welded parts.

The AE sensors were of PAC R15 type. They were linked, by the way of preamplifiers of a 3 channels VALLEN AMSY4 acquisition system. We started the monitoring just after the insertion of the samples in the media. The insertion was made once the temperature was stabilized at the value we fixed. For all the tests, the AE threshold was fixed at 40 dB.

For each test, channel 1 corresponds to the reference sample, channel 2 to the stressed sample and channel 3 to the hyper stresses sample.

3. Results and discussion

3.1. AE evolution compared to SCC activity

3.1.1. 44%$MgCl_2$ test

Fig. 2 shows the time evolution of the cumulative acoustic activity recorded during a test performed in 44% $MgCl_2$ medium. It can be seen that the more the sample is initially stressed, the greater the total acoustic activity is. If the activity of channel 1 (stress relieved sample) is very low after more than 5 hours of test, the activities of channel 2 and above all of channel 3 are very significant.

At the scale of the Fig. 2 representation, there are two main differences between channel 2 and 3 activities:

The first difference is that for channel or sensor 2, the increase of activity is almost linear. One can only observe some low magnitude variations. On the contrary, for channel 3, it progresses by mixed quiescent and active AE signals.

The second difference is linked to the start of AE detection after the insertion of the samples in the medium. For channel 2 it starts after about 16 minutes although for channel 3, it starts after only 2 minutes. Thus, the more the sample is stressed, the earlier acoustic activity can be detected.

Fig. 3 presents another comparison between channel 2 and 3 activity. In that case, the time scale as been reduced to only half an hour. It confirms that channel 3

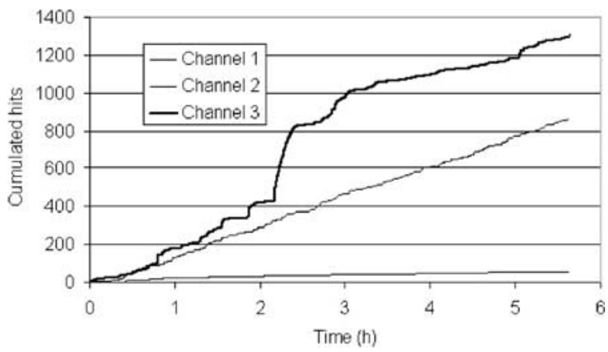

Figure 2 Time evolution of the cumulative acoustic activity recorded during a test in 44% $MgCl_2$ medium. Channel 1 = stress relieved sample. Channel 2 = stressed sample. Channel 3 = hyper stressed sample.

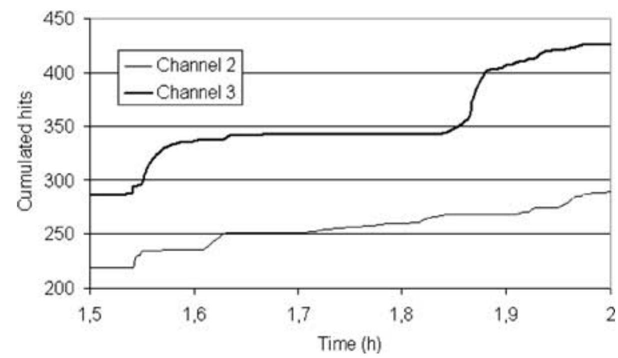

Figure 3 Another view of the time evolution of the cumulative acoustic activity recorded during a test in 44% $MgCl_2$ medium. Channel 2 = stressed sample. Channel 3 = hyper stressed sample.

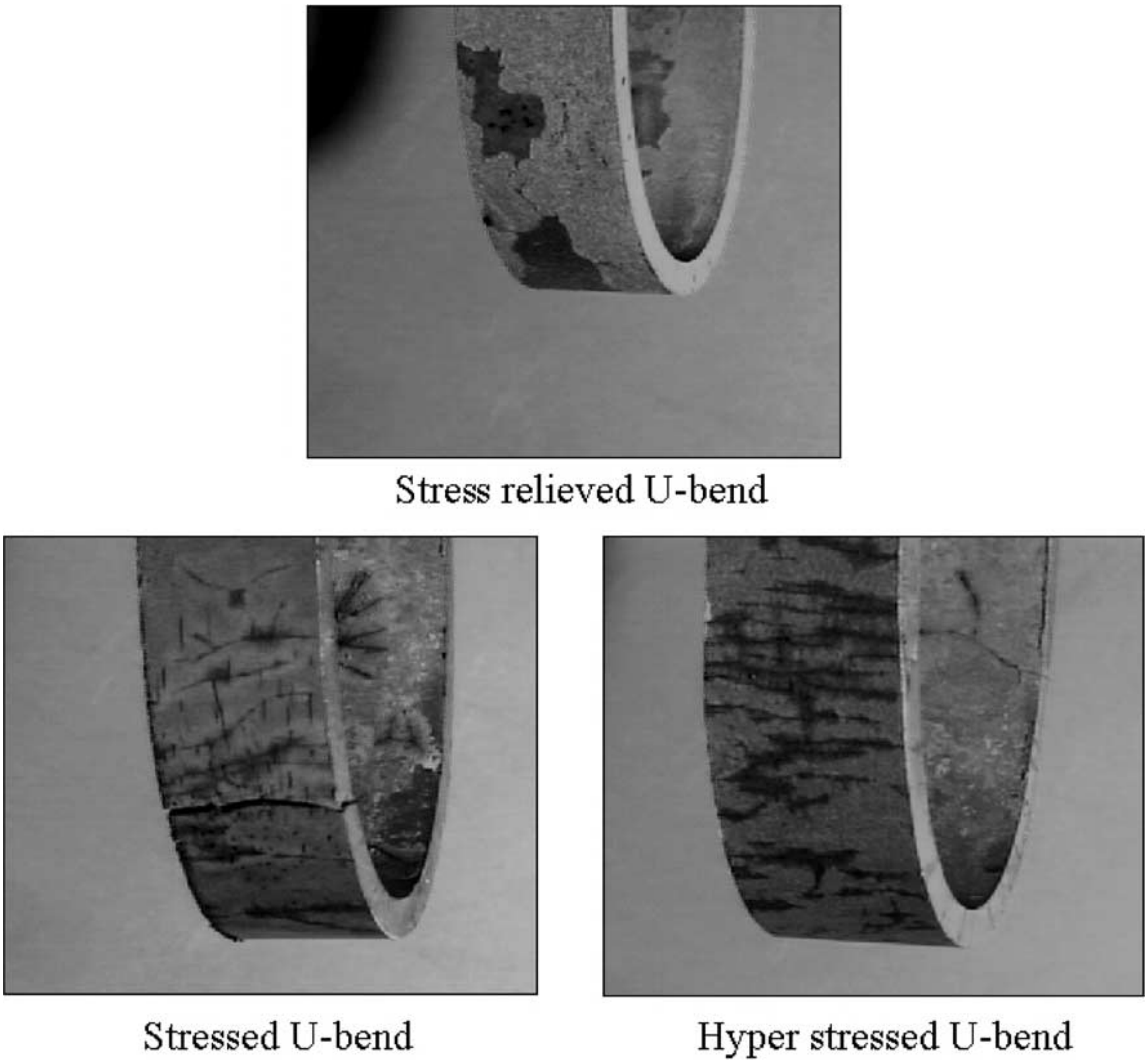

Figure 4 Pictures of three samples after a test of one day in 44% $MgCl_2$ medium.

activity progresses by mixed quiescent and active AE signals and it shows that for this enlarging, channel 2 activity has got a similar behavior. The only difference is that the bonds are less important for the channel linked to the less stressed sample. In the two cases, it can be seen that the periods without AE can have duration of several minutes. During the active AE signals periods, very high rate of AE can be recorded. For example, for the second high bond noted on channel 3 (around $t = 1.85$ h), we recorded more than 50 hits per minute.

Fig. 4 shows pictures of the three U-bends removed from the medium at the end of the test performed in 44% $MgCl_2$. We inspected carefully all the immersed part of the three samples. It is evident that there were no cracks on the initially stress relieved sample. On the contrary, on the two other ones, we observed a lot of cracks. By a quick analysis, we evaluated that the initially stressed sample (linked to channel 2) presented between 2 and 4 cracks per cm^2 although the initially hyper stressed sample (linked to channel 3) presented more than 6 cracks per cm^2.

All the observed cracks were confirmed to be of transgranular type which is typical of austenitic stainless steel SCC phenomenon in chloride containing media. Fig. 5 shows a view of a cross section of the material after metallographic attack revealing the austenitic structure of the material and the presence of numerous welding lines parallel to the sample surface. Two cracks, perpendicular to the U-bend exterior surface, can be observed. One of about 500 μm of length without macrobranching and one of more than 1600 μm of length with hight macrobranching.

The fact that the more the sample is initially stressed, the more it is sensitive to SCC is not a surprise. Most interesting results are on the one hand that AE detected waves in the very first minutes of the test, which tends to demonstrate its high sensitivity to SCC, and on the other hand, that the results concerning the evolution of the cumulative acoustic activity corroborate the corrosion feature observations. We recorded all the more signals that the total amount of cracks at the end of the test is.

3.1.2. 33%$MgCl_2$ test

For a test performed in a less corrosive aqueous media containing 33% $MgCl_2$ the same kind of analysis can be made. Fig. 6 presents the time evolution of the cumulative acoustic activity recorded during this test. As in the previous case, the increase of AE activity depends on the initially stress state of the immersed sample. The

Figure 5 Cross section view of a sample showing two cracks.

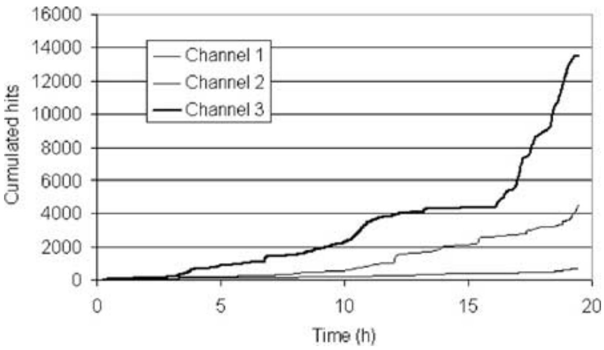

Figure 6 Time evolution of the cumulative acoustic activity recorded during a test in 33% $MgCl_2$ medium. Channel 1 = stress relieved sample. Channel 2 = stressed sample. Channel 3 = hyper stressed sample.

more the sample is initially stressed, the greater the total acoustic activity.

Because the media was less corrosive, the test has been conducted for a longer period than in the 44% $MgCl_2$ medium. During the first 4 hours of the test, acoustic activities of the three channels are almost nil. Then, a first significant activity bound can be noticed for channel 3. It prefigures the start of a high activity since after about 6 hours of test, the total amount of hits is quite similar to those noted after the same time in the previous test. For this channel, a series of spectacular bounds can be observed after 16 hours of test. During a period of 3 hours, more than 9000 hits are recorded. For channel 2, the real start of acoustic activity is difficult to evaluate. In fact, the activity increase is quite low between the fifth and the tenth hours of the test. After that period, the evolution is more evident and some significant bounds can be observed for example at times 12 and 15.5 hours.

As at the end of 44% $MgCl_2$ test, after the 33% $MgCl_2$ test, we correlated the acoustic results with the observation of the three U-bends removed from the medium. We recorded more signals the greater the total amount of cracking at the end of the test (Fig. 7).

The results obtained from the two presented tests have been confirmed by a series of other tests which is not necessary to detail in this paper. Taken as a whole, all the tests show that the AE recorded during the tests is essentially due to the progression of SCC cracks in the samples. It confirms the results of previous studies using different tensile tests [1]. Moreover, thanks to the use of three metallurgical states (with different sensitivity to SCC) and of two corrosive media, it has been shown, without external mechanical prompting, that the rate of acoustic activity is really linked to the number of active cracks. At that point, it isn't possible to determine the relative contribution of the different cracks on the overall activity recorded for one sample. Concerning the fact that the activity progresses by mixed quiescent and active AE signals, we do not know if it is due to the apparition of new cracks or to the SCC emitting mechanism itself. However, this work has not been conducted to establish or to confirm the mechanism of the acoustic emitting phenomena during SCC. Several papers are concerned with this point of view [1, 7].

3.2. Signals characterization

Fig. 8 shows the correlation diagram between the number of counts and the amplitude of the signals. This kind of representation is often used to determine the number of phenomena producing AE signals. For example, experience shows that when two phenomena are sources of AE, and if these phenomena generate different kind of signals, the correlation diagram between the number of counts and the amplitude often has two distinct representative areas of points, that is to say, two populations. In the present case, only one population is evidenced, which tends to demonstrate that with the threshold fixed (40 dB), there is only one type of acoustic signal. This analysis seems to be confirmed if we look at the amplitude distribution (Fig. 9). It consists of a deformed gauss curve essentially situated between 42 and 52 dB. The number of signals with amplitudes superior to 52 dB is not very important.

The analysis of the time evolution of the amplitudes observed during a test performed on an hyper stressed sample in a 33% $MgCl_2$ medium shows two distinct periods (Fig. 10). In order to clarify the data reading, only the signals with amplitude superior to 50 dB have been represented. During the first 17 hours, there are very few signals but a significant part of them have amplitudes of up to 70 dB. After the 17 hours time, the total amount of signals becomes very high (evaluated around a mean of 500 hits per hour) but the great majority of their amplitudes below 60 dB. It seems that before

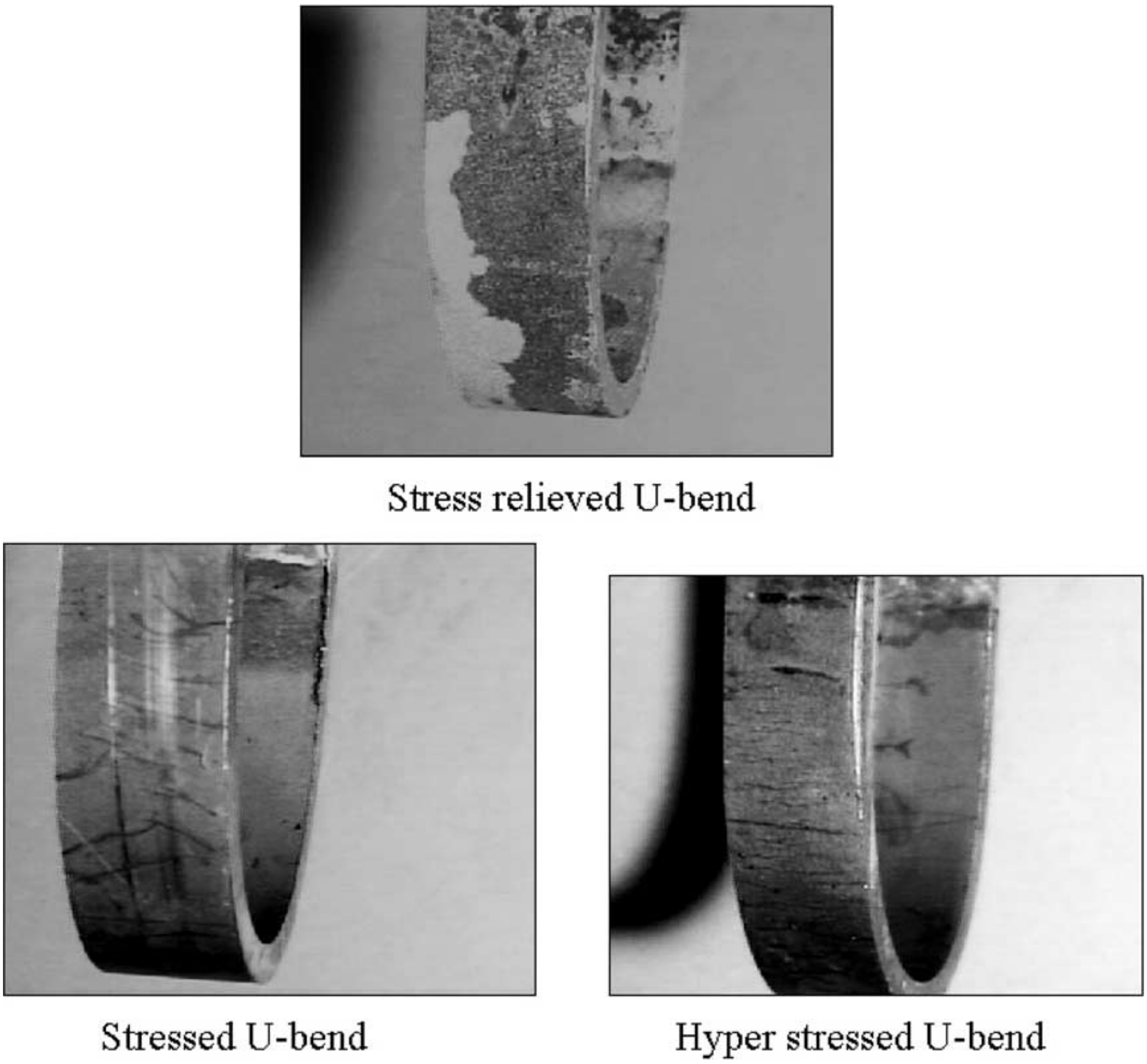

Figure 7 Pictures of three samples after a test of one day in 33% $MgCl_2$ medium.

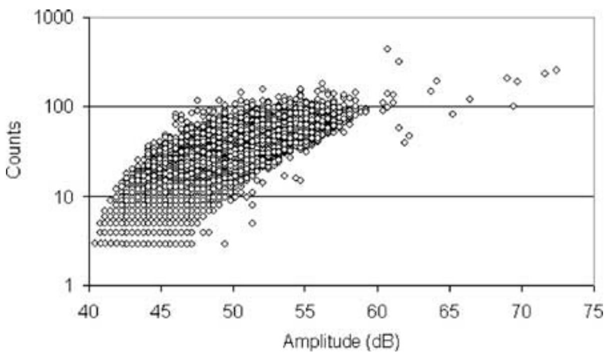

Figure 8 Correlation diagram between the number of counts and the amplitude of signals.

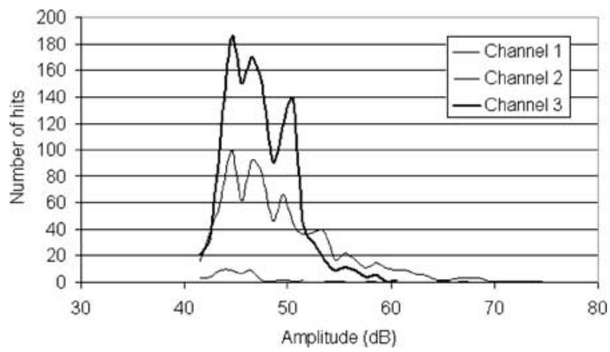

Figure 9 Amplitude distribution of the signals. Channel 1 = stress relieved sample. Channel 2 = stressed sample. Channel 3 = hyper stressed sample.

the real start of crack evolution, the sample is sujected to individual mechanical events generating rather high energy acoustic signals. Once a sufficient amount of cracks is active, this phenomenon becomes less evident. At this state of the study we are unable to say is this reproducible feature is linked to distinct emitting phenomena: for example a crack initiation phenomenon and a propagation phenomenon.

Fig. 11 shows the frequency distributions of the signals recorded during another test performed in 33% $MgCl_2$ medium. The mean frequency is centered around the resonance frequency of the sensors we used. However, if the great majority of frequencies is comprised between 130 and 150 kHz, it is interesting to note that for the channel 2 signals, there are two distinct frequency populations. One centered around 150 kHz and one centered around 125 kHz. It is the second point which tends to demonstrate the existence of two distinct emitting phenomena.

During further investigations, the use of wide band sensors and an extensive analysis of the resulting signals may help to state on that point.

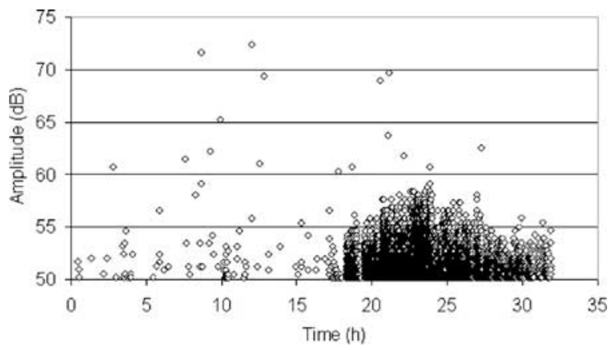

Figure 10 Time evolution of the amplitudes recorded during a test in 33% $MgCl_2$ medium during 17 hours (acoustic threshold = 50 dB).

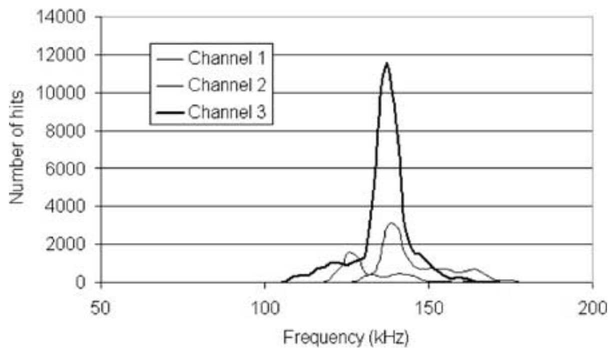

Figure 11 Frequency distribution of the signals. Channel 1 = stress relieved sample. Channel 2 = stressed sample. Channel 3 = hyper stressed sample.

4. Conclusion

The technique of acoustic emission has been chosen to study the SCC evolution of a 316L austenitic stainless steel during static U-bend tests. For that purpose, a specific experimental device has been settled.

In order to make varying the sensitivity of the used stainless steel to SCC, three metallurgical states have been tested: a reference sample totally stressed relieved, a stressed sample and an hyper stressed sample which has been obtained from a piece of metal preliminary shot peened.

Thanks to the use of those three different states, it has been clearly shown that the acoustic activity recorded during the static tests is linked to the evolution of active cracks of transgranular type.

It has been evidenced that:

– The evolution of the SCC phenomenon induces an acoustic activity which progresses by mixed quiescent and active AE signals.
– Even if it isn't possible to say that all the active cracks generate acoustic emission, and even if we can't determine the relative contribution of the different cracks on the overall activity, the rate of acoustic activity is all the more important that to the active cracks are numerous.
– The sensitivity of acoustic emission to detect SCC active cracks is very high.

Further investigations are programmed in order to distinguish the acoustic activity due to SCC initiation from the activity due to the propagation of cracks.

References

1. H. MAZILLE and R. ROTHEA, in "Modeling Aqueous Corrosion," edited by K. R. Trethewey and P. Roberge (Kluwer Academic Publishers, Boston, MA, 1994) p. 103.
2. T. W. RETTIG and M. J. FELSEN, *Corrosion* **32**(4) (1976) 121.
3. K. H. W. SEAH, K. B. LIM, C. H. CHEW and S. H. TEOH, *Corrosion Science* **34**(10) (1993) 1707.
4. H. MAZILLE, R. ROTHEA and C. TRONEL, *ibid.* **37**(9) (1995) 1365.
5. F. FERRER, H. IDRISSI, H. MAZILLE, P. FLEISCHMANN and P. LABEEUW, *Wear* **231** (1999) 108.
6. F. FERRER, E. ANDRES, J. GOUDIAKAS and C. BRUN, *Journal of Acoustic Emission* **18** (2000) 155.
7. S. YUYAMA, in "Corrosion Monitoring in Industrial Plants Using Nondestructive Testing and Electrochemical Methods," ASTM STP 908, edited by G. C. Moran and P. Labine (American Society for Testing and Materials, Philadelphia, PA, 1986) p. 43.
8. S. YUYAMA, T. KISHI and Y. HISAMATSU, *Journal of Acoustic Emission* **2** (1983) 71.
9. A. CAKIR, S. TUNCELL and A. AYDIN, *Corrosion Science* **41** (1999) 1175.
10. W. W. GERBERICH, R. H. JONES, M. A. FRIESEL and A. NOZUE, *Materials Science and Engineering* A **103** (1988) 185.
11. P. F. BUCKLEY, K. BALDWIN, M. J. EHRLICH and J. W. WAGNER, in "Review of Progress in Quantitative Nondestructive Evaluation," Vol. 17A (Plenum, 1998) p. 571.
12. S. YAMASAKI and T. TAKAHASHI, *Tetsu-to-Hagane* (*Journal of the Iron and Steel Institute of Japan*) **83**(7) (1997) 460.
13. B. KNOWLTON, A. M. SIDDIQUI and S. JIHAN, *British Corrosion Journal* **32**(4) (1997) 249.
14. R. H. JONES and M. A. FRIESEL, *Corrosion* **48**(9) (1992) 751.
15. K. Y. SUNG, I. S. KIM and Y. K. YOON, *Scripta Materialia* **37**(8) (1997) 1255.
16. P. COMBRADE, in "Corrosion Sous Contraintes—Phénoménologie et Mécanismes," Les éditions de physiques (Les Ulis, Bombanes, 1990) p. 539.
17. A. BOUZINA, Ph.D. thesis, N° d'ordre: 1998 – 06, 1998, p. 130.

Received 12 June 2001
and accepted 14 March 2002