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Veronique Smanio, Marion Fregonese, J. Kittel, Thierry Cassagne, Francois Ropital, et al.. Evaluation of hydrogen induced cracking damages in steels using acoustic emission. Eurocorr 2007, Sep 2007, Freiburg, Germany. hal-02475581

HAL Id: hal-02475581

<https://ifp.hal.science/hal-02475581>

Submitted on 12 Feb 2020

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Evaluation of Hydrogen Induced Cracking damages in Steels using Acoustic Emission

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Summary

In the oil & gas industry, it is known that carbon steel equipments which operate in wet hydrogen sulphide (H₂S) media can be subjected to damages like Hydrogen Induced Cracking (HIC) and sulphide stress cracking (SSC).

For material selection operating in sour conditions, standard tests were developed, like NACE TM0284 for HIC or NACE TM 0177 for SSC. These tests are usually used by operators and give a pass or fail response in a short time of testing. However, they give only limited information on crack mechanisms, nor on the evolution with time of cracks.

In this study, Acoustic Emission (AE) technique is used to monitor the cracking of steels immersed in sour media. The aim is to get more detailed information on the cracking mechanism of steels during standard tests. The main focus of this paper is HIC. The preliminary identification of the different AE sources involved is carried out in order to establish a methodology for HIC detection. The identification of the different AE signals and the monitoring of HIC tests on different steel grades are described. The AE data are compared with the standard HIC criteria. The results indicate that AE can provide an early detection of cracking when the various AE sources can be identified.

Introduction

Production waters containing hydrogen sulphide can lead to different types of Hydrogen Embrittlement (HE) in steels, like Hydrogen Induced Cracking (HIC) and Sulfide Stress Cracking (SSC).

Hydrogen Induced Cracking (HIC) is a planar mode of cracking that occurs in carbon and low alloy steels when atomic hydrogen diffuses into the metal and then combines to form molecular hydrogen at trap sites. Cracking results from the pressurization of trap sites by hydrogen. No external applied stress is needed for HIC development. Trap sites involved in HIC are commonly found in steels that have a high density of planar inclusions like MnS and/or regions of anomalous microstructure (e.g. pearlitic structure) produced by segregation of impurities and alloying elements.

Sulphide Stress Cracking (SSC) leads to the early failure of the metal due to the combined effect of both hydrogen penetration and tensile stress (residual and/or applied) in wet H₂S environment. High strength metallic materials and hard welded zones are prone to SSC.

The present work is focused on HIC, but study of SSC by acoustic emission is one of the future prospects of the study.

The selection of HIC resistant steels is mainly based on HE standard tests (NACE TM 0284 [1] or EFC 16 [2]) in sour media. In these tests, material damage is evaluated from cracking ratios measured in three equidistant cross-sections of the tested specimens (Crack Sensitivity Ratio, CSR, Crack Length Ratio, CLR and Crack Thickness Ratio, CTR). Information provided by these ratios is very local because it represents only the cracking state into 3 sections. Depending on the metallographic procedure used to observe cracks randomly

distributed within the material, there is a marked deficiency in standard test sensitivity. Furthermore, metallographic examination is solely representative of the cracking that is achieved after the total duration of the test. Other important information are missing, e.g. the incubation time necessary for the initiation of cracking, or the evolution of cracking with time.

On the other hand, the Acoustic Emission (AE) technique is a non destructive evaluation method allowing the detection of active defaults within materials on a real time and continuous basis during the test. Each acoustic emission event gives rise to elastic waves which propagate into the material and results in detectable AE signals [3]. Materials can spontaneously generate transient elastic waves, called acoustic emission (AE), when they undergo solicitations. Among the variety of mechanisms of deformation and damage that can be sources of acoustic emission, corrosion processes take a particular position, as the acoustic source is often not the electrochemical corrosion driving force itself, but its consequence: bubbles evolution, cracks propagation, modifications of corrosion products deposits [4-5], whether corrosion is localized or uniform. Indeed, good correlations were recently obtained between specific AE parameters and amplitude of corrosion damage in various cases: stress corrosion cracking [4-7], abrasion or erosion corrosion [8], pitting corrosion [9-12], crevice corrosion [13-14], exfoliation corrosion [15], uniform “acidic” corrosion [16].

In the past, Acoustic Emission technique has been applied to monitor Hydrogen Embrittlement in several studies [17-22]. Cayard and al. [19] and also Gingell and al. [21] showed that during SSC tests leading to the failure of the tested metal, a higher AE energy build-up rate was recorded compared to no-failure tests. This critical energy build-up rate associated to SSC failure remains dependant on the tested steels and the applied stress level. Weng and al. [18] found out a correlation between HIC damage evaluated by Crack Length Ratio and the AE energy level. Moreover, Gingell and al. [21] showed that AE can be used to discriminate initiation and propagation stages of SOHIC (Stress Oriented Hydrogen Induced Cracking) development during NACE TM0177-96 [23] method A tensile tests. In all these studies, AE data were treated in a global manner, i.e. considering simultaneously all the AE sources involved during the test. The objective of our work is to discriminate the different involved AE sources in order to allow an early detection of HIC, but also to monitor the different physical phenomena occurring during the test.

Experimental Procedure

Materials

Two API X65 grade pipeline steels were used as reference materials. One was identified as X65 Non Sour Service (NSS) grade because of its poor HIC resistance and the other one was identified as X65 Sour Service (SS) grade.

The chemical compositions of both X65 grade steels were analyzed by optical emission spectroscopy (OES), except for carbon and sulphur, analyzed by a chemical method (Table 1).

Table 1: Chemical composition (wt %) of X65 Sour Service and Non Sour Service grades

Steel	C	Mn	Si	P	S	Cr	Ni	Mo	Cu	Al	Ti	Nb
X65 SS	0.046	1.36	0.322	0.008	0.001	0.041	0.036	0.008	0.047	0.036	0.019	0.045
X65 NSS	0.09	1.56	0.28	0.014	0.001	0.05	0.03	0.01	0.02	0.030	0.000	0.040

For microstructure observation, the specimens were ground with silicon carbide (SiC) paper down to 1200 grade and polished down to 0.05 μ m diamond suspension and then etched with a Nital solution (2%vol. nitric acid and ethanol). The microstructure was observed in the

long transverse direction (LT) using a DM4000 Leica optical microscope. The different microstructures at the mid wall location are presented in Figures 1 and 2.

The major phases observed in the sour service grade are pearlite and ferrite. The microstructure is not oriented and homogenous, which is the principal reason for its good HIC resistance.

The non sour service grade presents a typical ferrite-pearlite microstructure. The microstructure is highly oriented in the rolling direction, which explains its poor HIC resistance.

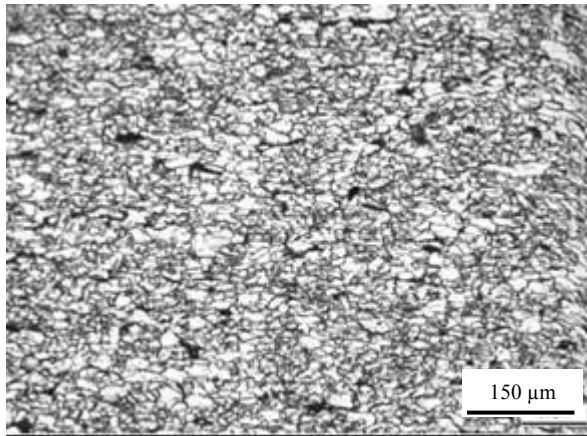


Figure 1: Microstructure of X65 Sour Service grade (LT plane)

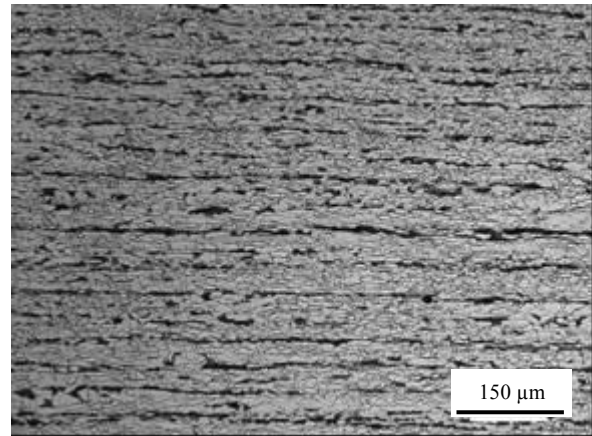


Figure 2: Microstructure of X65 Non Sour Service grade (LT plane)

The tensile properties of both steels in the longitudinal direction parallel to rolling are gathered in Table 2. The inclusion content was analyzed according to ASTM E45 method A. The results are presented in Table 3.

Table 2: Mechanical properties of X65 Sour Service and Non Sour Service grades (tests conducted according to NF EN10002-1 standard)

Steel	$R_{p0.2} \pm 2\%$	$R_m \pm 1.5\%$	$A\% \pm 2.5\%$
X65 SS	529 MPa	571 MPa	47
X65 NSS	523 MPa	649 MPa	24

Table 3: Comparison of inclusion content of tested steels, according to ASTM E45 method A

Steel	Type A (sulfides)		Type B (aluminates)		Type C (silicates)		Type D (oxides)	
	Thin	Heavy	Thin	Heavy	Thin	Heavy	Thin	Heavy
X65 SS	0	0	0	2	0	0	0.5	0.5
X65 NSS	0	0	0	0	0	0	0.5	0.5

No significant difference is observed between both grades in terms of inclusion content, which confirms that the difference of HIC sensitivity between the selected steels is mainly linked to ferrite and pearlite distribution and grain orientation.

Experimental Set up

The experimental set up (Figure 3) was based on the SSC standard test NACE TM0177-96 [23] device. The specimen was placed in a proof ring, yet no load is applied when HIC test were performed. Only the gauge section of the sample was immersed in the corrosive solution. Tests were conducted in buffered solution recommended by EFC 16 [2] (5 wt%

NaCl and 0.4% CH₃COONa in distilled water) under 1 bar H₂S. The solution was deaerated by nitrogen bubbling to less than 15ppb dissolved oxygen before each test. The pH of the solution was controlled and adjusted every day through HCl or NaOH additions.

A circulation of the corrosive fluid (sour media) was ensured between the test cell and a vessel, in which gas bubbling was maintained. This procedure allowed to avoid any interfering AE noise detection (e.g. gas bubbling in the test cell would result in AE) and to maintain constant electrochemical conditions in the cell during the test (easier pH control).

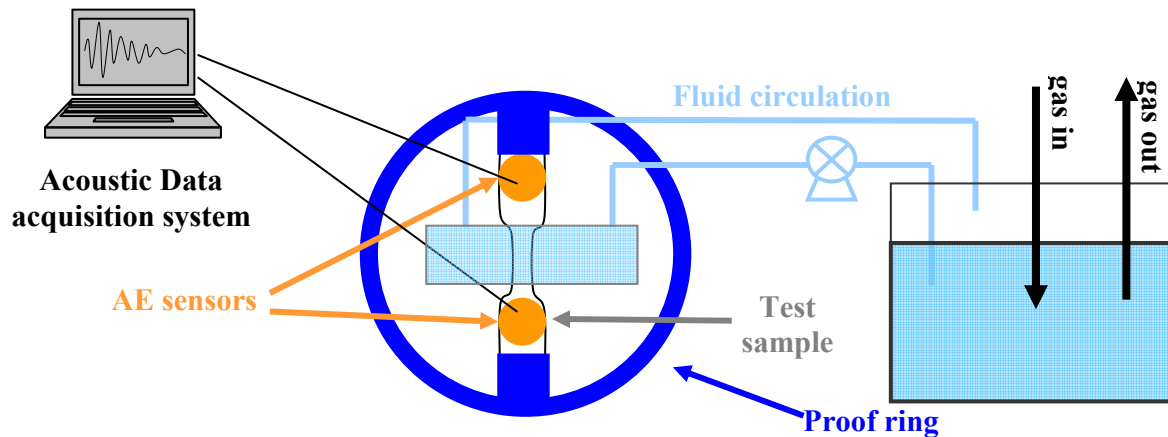


Figure 3 Experimental set up

Acoustic emission technique has been adapted to this test: two AE sensors were coupled to the top and bottom of the specimen (Figure 3) in order to detect AE signals.

AE instrumentation consisted of a transducer, a preamplifier and an acquisition device (MISTRAS with AEWIN software from Physical Acoustic Corp.). The gain of the preamplifier was set to 40dB. A band pass filter with frequency range from 100 to 300 kHz was used. The transducers were Nano 30 type from PAC (piezo-electric disks). The acquisition system was completely computer controlled. The waveforms, the events number and the characteristic acoustic parameters were stored on a hard disk as soon as detected, and were available for post-treatment. For each detected AE signal, following acoustic parameters can be studied:

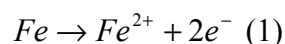
- Events number : number of AE signals detected (discontinuous emission),
- Amplitude : maximal amplitude of the considered AE event (peak),
- Rise time : time between the first overshoot of the defined threshold and the peak,
- Counts number : number of times that the threshold is overshoot for a given AE event,
- Duration: time between the first and the last overshoot of the define threshold,
- Energy: envelop of the signal.

In this study, the threshold was set to 28 dB.

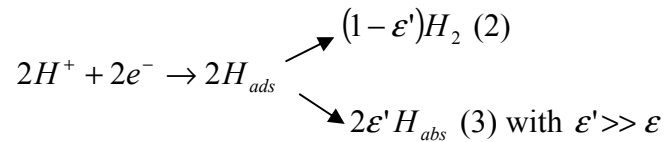
Corrosion reactions in sour media

Behavior of steel immersed in an acidic media can be described by the following corrosion reactions [24]:

- anodic reaction,



- cathodic reactions,



Furthermore, in presence of H₂S, a sulphide layer is created at the steel surface. Hydrogen uptake into the material is also enhanced by H₂S, leading to Hydrogen Embrittlement.

Methodology

The first step of this study was to identify the different acoustic emission sources involved when a steel sample is placed in acidic media containing H₂S.

Under these conditions, the potential physical phenomena leading to acoustic emission are linked to:

- H₂ evolution resulting from corrosion reaction,
- presence of the sulphide layer on the sample,
- cracking resulting from the absorption, the diffusion and the trapping of hydrogen.

Then, by varying the experimental conditions and by comparing between sour service and non sour service steels, it was possible to discriminate the acoustic emission sources. Table 4 summarizes the successive experimental steps that were applied for identification of acoustic signature of individual sources. A first tests batch was conducted without H₂S under cathodic polarization in order to identify AE signals resulting from H₂ evolution. Then, a second tests batch was conducted under 1 bar H₂S at pH 4.5 on both sour service and non sour service steels to discriminate sulphide layer induced AE signals from HIC induced AE signals.

Table 4: Active Acoustic Emission sources as a function of experimental conditions and tested materials

Experimental conditions				Acoustic emission sources		
Gas	pH	Steel	Polarization	H ₂ evolution	Sulphide layer	HIC
N ₂	5.5	X65 SS	cathodic	X	-	-
H ₂ S	4.5	X65 SS	no	X	X	-
H ₂ S	4.5	X65 NSS	no	X	X	X

After the tests, evaluation of HIC damage was performed according to the standard NACE TM0284-03 practice, with three equidistant cuts along the length and metallographic examination of the faces. CLR and CSR were then calculated. However, it has to be recalled at that point that the geometry was similar to SSC tensile specimens as per NACE TM0177, with a 6.25mm diameter section. This dimension is rather low when compared to standard HIC specimens which are 20mm wide. It is therefore expected that the experimental error and scattering of crack ratios determined from the small SSC shaped specimens might be important.

Results and discussion

Identification of the different AE sources

The first tests batch consisted in a cathodic polarization of the X65 SS grade immersed in the test solution under 1 bar N₂ at pH 5.5. The applied current was -280 μA/cm², corresponding approximately to a cathodic potential of -1231 mV/Eoc and the tests lasted 8 hours. Cathodic reaction leading to H₂ evolution is enhanced by the imposition of a cathodic

potential to the sample; at the same time anodic reaction resulting in iron dissolution is limited.

Under these conditions, only one AE source is active: the evolution of hydrogen gas resulting from the cathodic reaction. No HIC resulting from cathodic hydrogen charging was expected for the sour service steel. AE results are presented in a correlation chart representing the absolute energy of the signals as a function of their duration (Figure 4). On this figure each point represents an AE event. AE related to H₂ evolution exhibits the following characteristics:

- signal duration < 1500 μS,
- signal absolute energy < 100 aJ.

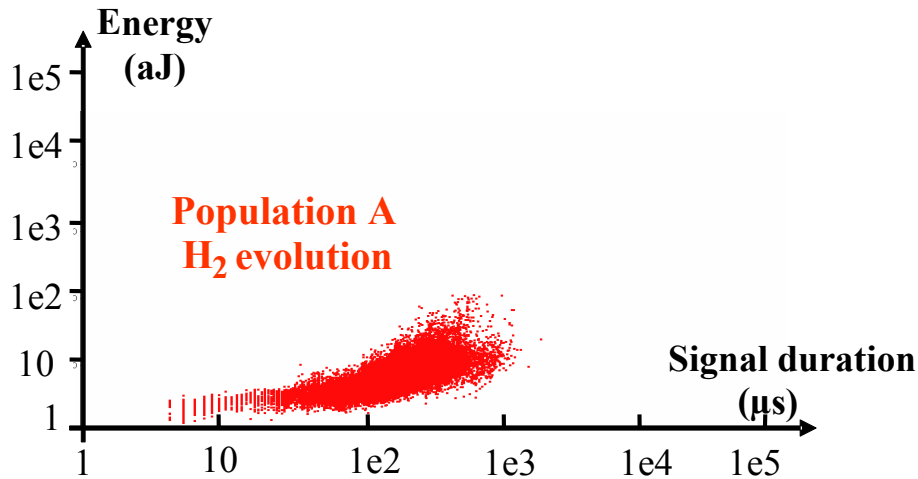


Figure 4: Correlation chart representing absolute energy as a function of signals duration for a test conducted in the EFC 16 solution at pH 4.5 under 1 bar H₂S, on a X65 SS specimen

In order to identify the AE signals related to the sulphide layer and HIC, further tests were performed on sour service and non sour service steels at pH 4.5 under 1 bar H₂S. Evaluation of HIC damage after these tests is given in Table 5.

Table 5: HIC damage evaluation on sour service and non sour service steel grades at pH 4.5 and 1 bar H₂S

	X65 NSS	X65 SS
Metallographic observations	CLR ~ 30% CSR ~ 2%	No cracks

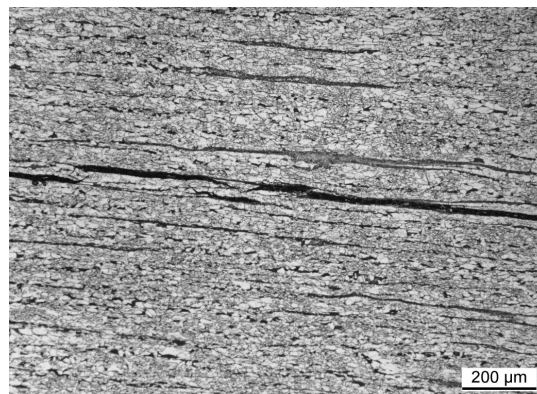


Figure 5: HIC detected in X65 NSS after a test performed in solution EFC 16 at pH 4.5 under 1 bar H₂S

Cracks were only observed in the non sour service steel grade. They are localized in the bands of pearlite at mid-wall location (Figure 5).

From these observations, it can be assessed that:

- for the sour service steel tested at pH 4.5 and under 1bar H₂S, the emissive phenomena are only H₂ evolution and the presence of the sulphide layer,
- for the non sour service steel tested at pH 4.5 and under 1bar H₂S, the emissive phenomena are H₂ evolution, the presence of the sulphide layer together with HIC development.

The characteristics of the AE signals recorded during the former test (pH 4.5, 1 bar H₂S) performed on the sour service steel are presented in Figure 6. Population A, previously attributed to H₂ evolution, is still present, in agreement with the fact that this AE source is still active with this test procedure. As compared with the AE diagram obtained for H₂ evolution under cathodic polarisation (Figure 4), a new AE population is detected, with signals absolute energy higher than 100 aJ. As no cracks are detected for these experimental conditions, it can be assumed that this population B is associated to the presence of the sulphide layer at the sample surface.

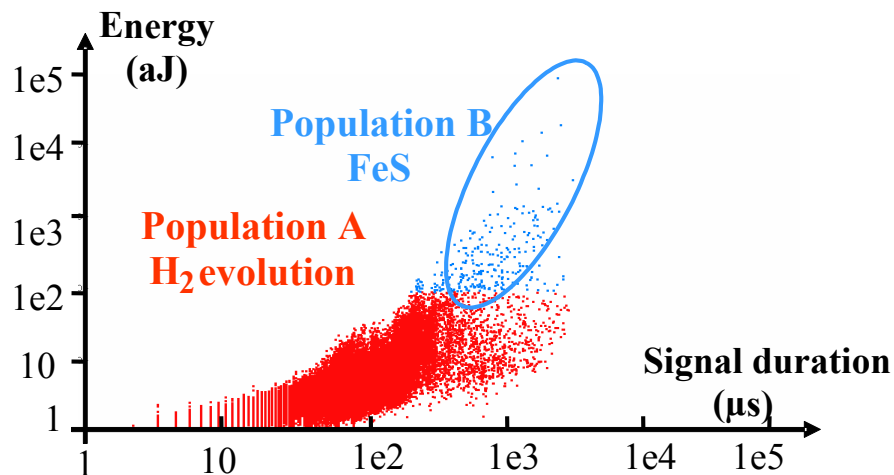


Figure 6: Correlation chart representing absolute energy as a function of signals duration for a test conducted in the EFC 16 solution at pH 4.5 under 1bar H₂S on a X65 SS specimen.

AE results obtained during tests performed on the non sour service steel grade are presented in figure 7. As previously, H₂ evolution and sulphide layer populations can be identified again. But a third AE population exhibiting signals duration higher than 1500 µs is detected. Thanks to metallographic observations evidencing the presence of cracks in the sample after the test, this AE population can be associated to HIC development within the material.

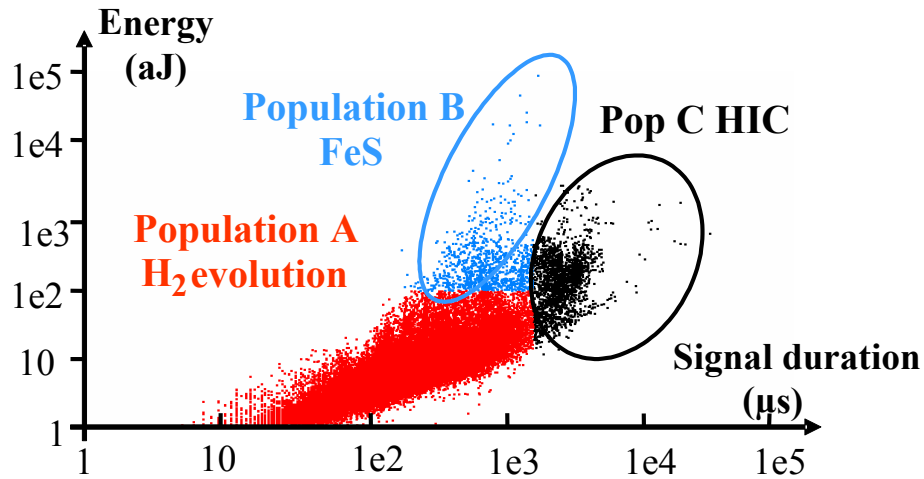


Figure 7: Correlation chart representing absolute energy as a function of signals duration for a test conducted in the EFC 16 solution at pH 4.5 under 1 bar H₂S on a X65 NSS specimen.

Therefore, the experimental method proposed in the present study enables the discrimination of the AE sources involved in the HIC testing procedure, i.e. H₂ evolution, the presence of a sulphide layer at the surface of the metal and HIC development.

Kinetics of the different physical phenomena involved in HIC testing procedure

The identification and the discrimination of the different AE signals, related to the different physical phenomena associated to HIC development within steels tested in sour media, now allow studying the evolution of these phenomena during the test. Figure 8 presents the evolution with time of the cumulative energy of each AE population, recorded all along a test performed on a non sour service specimen at pH 4.5 under 1 bar H₂S.

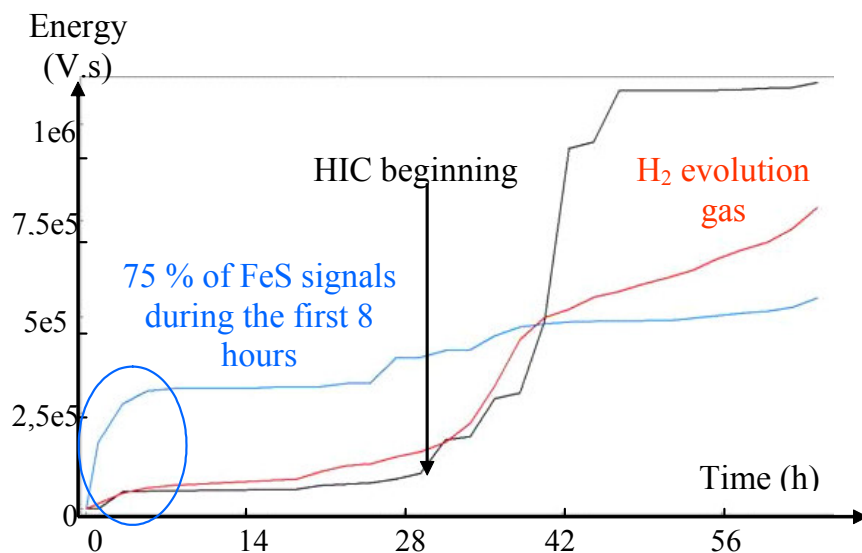


Figure 8: Evolution of cumulative energy of the different acoustic sources during a test performed on the X65 NSS steel grade at pH 4.5 under 1 bar H₂S

The main features are the following:

- the most important part of AE associated with sulphide layer is detected during the first 8 hours of the test. According to visual observations, this time corresponds to the formation of the sulphide layer. This result is in good agreement with literature data. Tsai and al. [20] have observed that the formation of a dense iron sulphide film lasted about 8 hours in a H₂S containing solution, whereas an outer porous sulphide layer was observed after 24 hours of test. The authors proposed that sulphide film formation and/or breakdown could be responsible for AE signals.
- An increase in AE energy induced by HIC development is observed after 28 hours of test, which can correspond to the time necessary for trapped hydrogen to reach pressure level sufficient to initiate HIC. It could also be inferred that crack propagation lasts until 47 hours and then stops. AE measurements give therefore real time information about the incubation period required for HIC development, but also on the evolution of cracks.
- Yet, it is worth noting that, even if H₂ population evolves more regularly, which is in agreement with the evolution of the physical AE source itself (proton reduction occurs continuously), an increase of population A is also observed after 28 hours of test. This increase can be due to the fact that populations A and C are certainly mixed in the junction domain of the three populations of AE signals. Further post-treatments of the signals are necessary to more accurately separate the different active AE sources.
- Moreover, HIC process is the physical phenomenon that induces the highest cumulative AE energy (> 1e6 V.s). This result is in a good agreement with previous studies in which the global AE energy detected during the test was chosen as a criterion for detecting the formation of cracks [18-19, 21].

Conclusions and Outlook

AE monitoring was carried out during standard tests devoted to characterize Hydrogen Embrittlement sensitivity of steels. An experimental procedure was proposed to discriminate the different AE sources (H₂ evolution, FeS layer, HIC). This discrimination provides information about the physical phenomena at the origin of Acoustic Emission: the formation of the sulphide layer occurs during the first hours of the test, whereas HIC is a delayed phenomenon. Early detection of HIC is found to be possible by the use of AE technique, by discriminating data in terms of duration of the signals and acoustic energy. In the future, this technique will be applied to the early detection of HIC in mildly sour environments, but also to quantify HIC damage in a sample. SSC development will also be investigated by Acoustic Emission.

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