

Acoustic Emission during Hydrogen Charging of Alloys

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Abstract

There were realized by acoustic emission (AE) measurements of corrosion processes that relatively intensive AE is generated during hydrogen charging. Experiments carried out to study this phenomenon showed that there are several possibilities to explain the results, especially to identify the sources of AE. Set of other experiments enable us to eliminate several considered sources of AE. Large differences were found in AE behaviour of various alloys during hydrogen charging. AE response of two alloys will be presented. One possible explanation of these differences is given.

The second part of the work concerns the relation between exposed surface and exposed volume. Specimens with various specially designed geometries were measured and results showed that there is dependence on volume; it means that at least important part of measured AE is created inside.

There are still many questions connected with the hydrogen charging and AE, it means, the work is continuing.

Introduction

Acoustic emission (AE) technology is a non-destructive testing method which has a high sensitivity for detecting active microscopic events within a material. These events give rise to elastic waves which propagate out into the material and result in detectable AE signals. Our practical interest of this technique lies in its applicability for detection of stress corrosion cracking (SCC) and hydrogen embrittlement (HE). During the process of hydrogen absorption, emission of acoustic waves is due to several reasons, viz. crack initiation and growth induced by hydrogen, evolution of hydrogen gas bubbles during electrochemical charging of hydrogen, the breakdown of oxide film, etc. The phenomena associated with the absorption and desorption of hydrogen could also lead to detectable AE signals. It is known that hydrogen can enter steels in many ways, causing a loss of ductility which may result in catastrophic failure of structures in service. [1, 2]

The original aim of this study is to investigate SCC, corrosion fatigue (CF) and other possible AE sources which could be present during corrosion phenomena.

Experiments

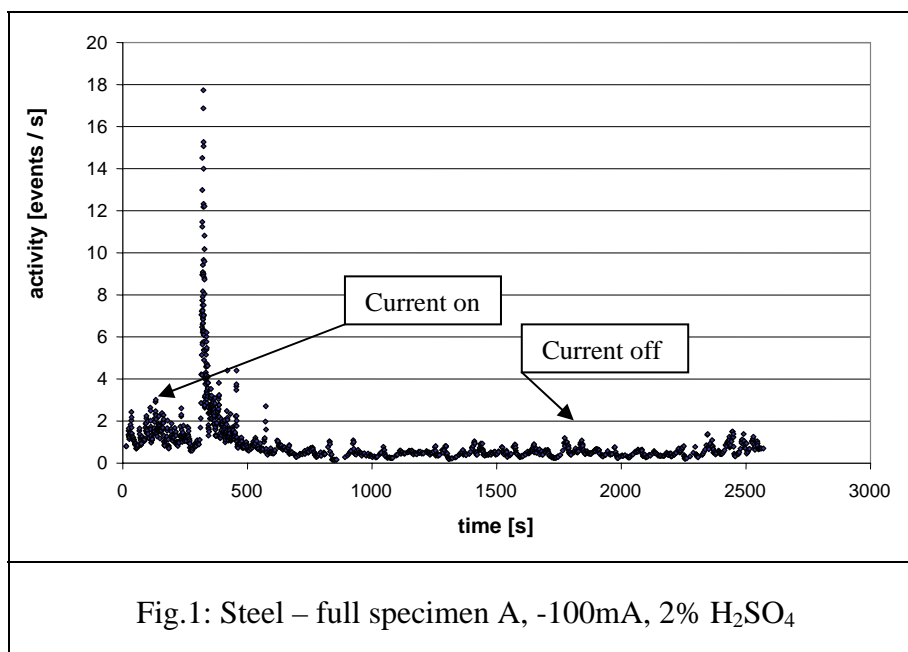
The main part of the used AE apparatus is two piezoelectric transducers working on frequency 100-300 kHz. Transducers are connected to SF41 AE measuring PC card via two preamplifiers with gain 40dB. One transducer was attached on specimen and the other to the glass bar. Data from that second reference channel was used for separating noise background in following interpretation. Some experiments were carried out under galvanostatic control, where the sample was used as working electrode, platinum gauze as auxiliary electrode and calomel half-cell as reference electrode.

Specimens A - F were prepared from carbon steel EN 10083/2-91 with circle profile dia.16x150 mm. Some specimens (B, D, F) were made with cavity dia.12x50 mm from the bottom and then they were filled by silicon paste. The purpose of this arrangement is to measure on the same material with the same surface and different volume. Size of specimens G – I was 200x20x3 mm and material EN 10025-94 carbon steel was chosen. Specimen J was made from brass EN 1652 and size was also 200x20x3 mm. The surface of specimens H, I, J was coated by homogenous nickel layer. This modification helps to reach the same surface conditions on different materials. Each specimen was grinded and than rinsed with ethanol and DEMI water.

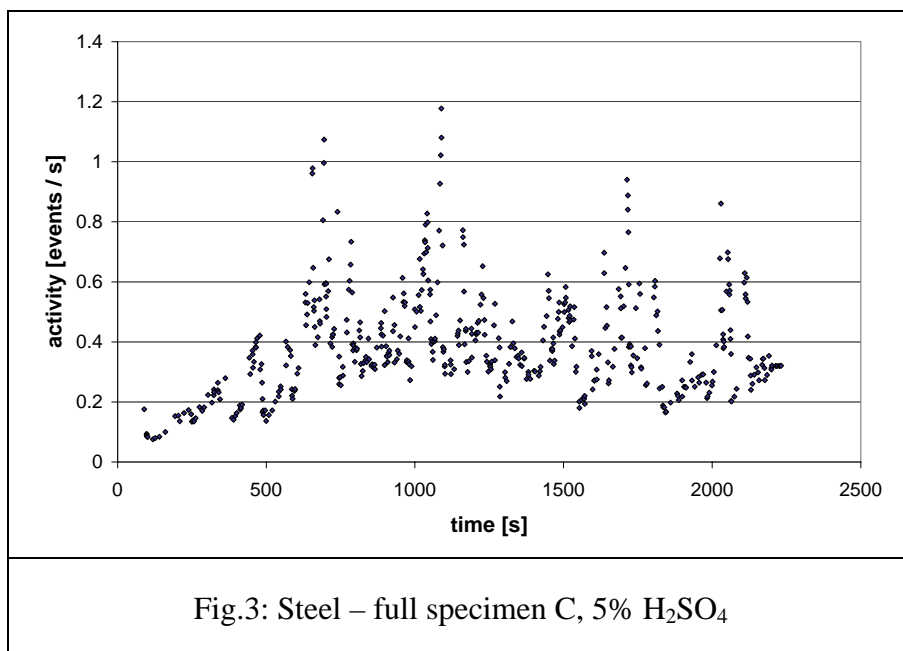
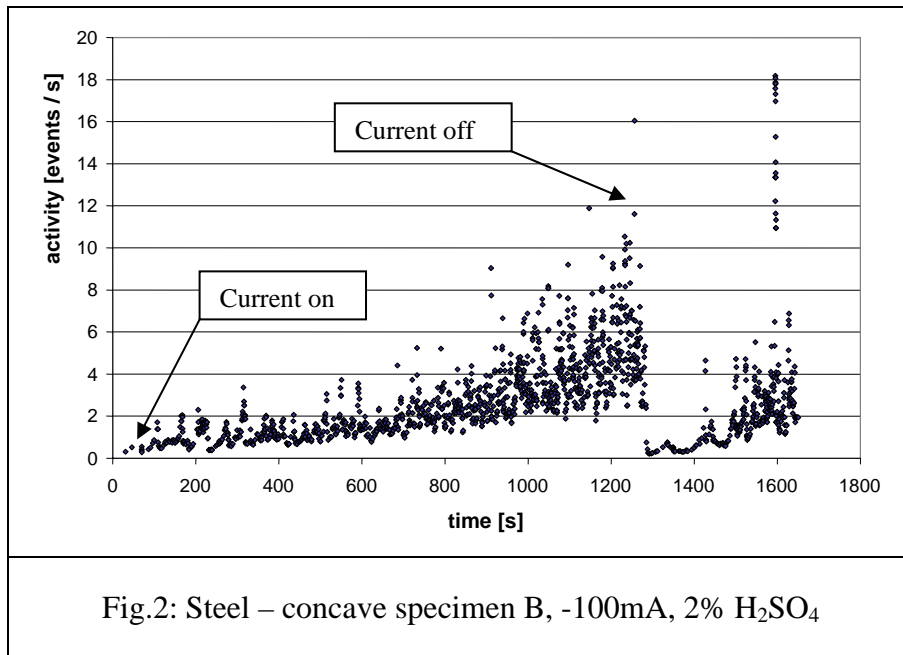
Experiments were carried out in sulphuric acid (concentration 2wt.%, 5wt.% and 10wt.%) and in DEMI water alkalized by Na_3PO_4 to pH=11.

Results

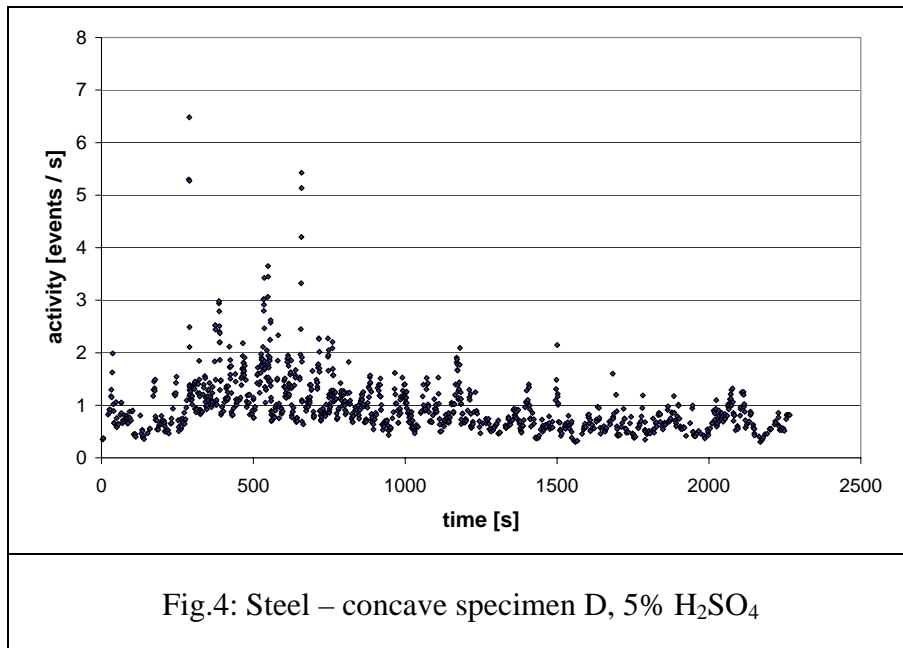
Experiments shown on fig. 1, 2 were performed on specimens A and B at the same conditions.



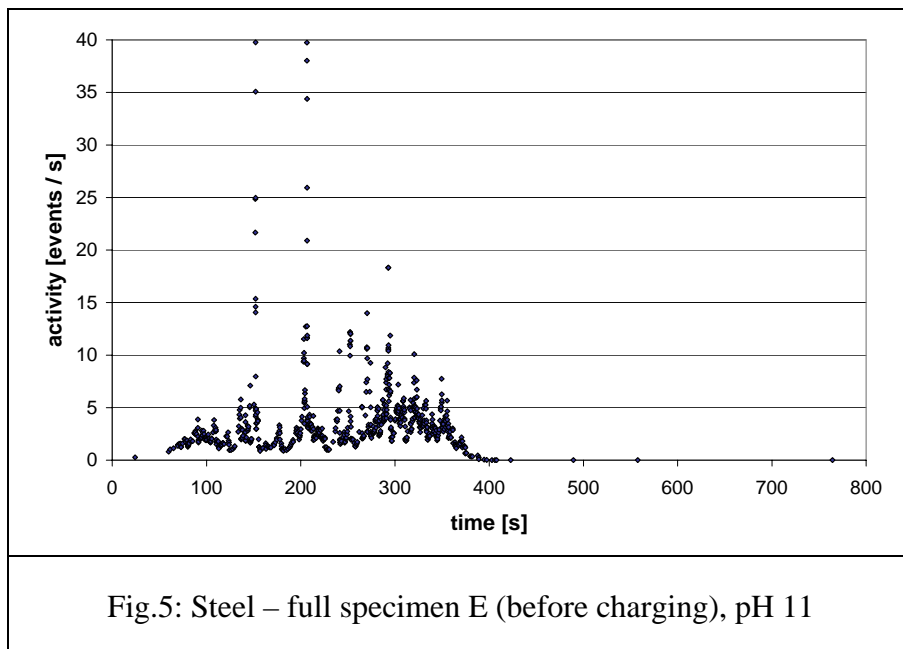
Next two experiments (fig. 3, 4) were carried out on the same type of samples, but without influence of electric current.



On fig. 5 - 8 are shown results of measurement conducted also on full/concave steel specimens, but in that case the hydrogen charging by current flow and AE measuring were not done in the same time. Specimen E (and in following experiment specimen F) with attached AE sensor was immersed in the alkalinized water on the temperature 90°C. Solution with sample cooled gradually about one hour to the temperature 40°C and AE was measured (fig. 5, 7). Then was the specimen immersed into the 5% sulfuric acid and connected with galvanostat. Next 24 hours was the specimen charged by current -100mA without AE measuring. Immediately after this part was repeated the experiment with cooling of the solution from 90°C to 40°C (fig. 6, 8). In all cases the AE activity vanished after the temperature reached approximately 80°C.



Last experiments were carried out on flat samples under galvanostatic control. There are four diagrams (fig. 9 – 12) to compare between steel, nickel coated steel and nickel coated brass at several conditions. Figures of steel characteristics (fig. 9, 10, 12) are not displayed complete, only the most important first part with beginning of electric current pass is shown.



Discussion

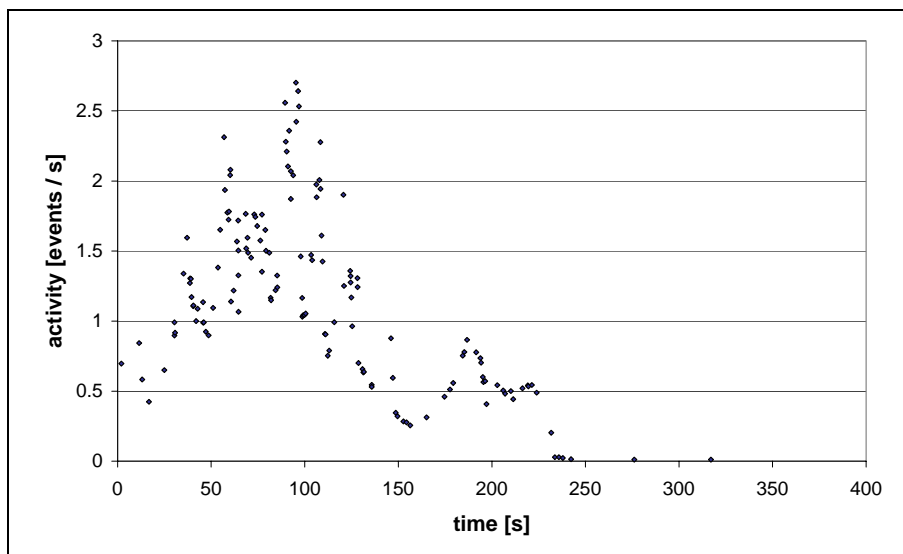
Time to AE initiation

There are clearly visible a time to initiation in experiments results of which are in Fig.1, 2.

The times to initiation are much longer than time which is needed to create the first bubbles of hydrogen on the specimen surface. The bubbles are created almost immediately after the current switch on. It is possible to reason out that the bubbles are not the main source of AE. The similar situation is in the case of a drop of nitric acid dropped on a steel specimen [4]. Following AE has different source, but the relation to bubbles gives the same result.

The measured AE is created by different source in these occasions, but the times to initiation have similar behaviour. The processes which lead to AE are connected with changes in surface layer of material in the first stage. In the case of hydrogen charging the following AE originates not only from the surface layer but with the continuing diffusion from the bulk as well.

The time to initiation shows clearly that the measured emission is caused by processes inside material, not by bubbles creating on the surface of specimen.



Fig,6: Steel – full specimen E (after charging), pH 11

Influence of specimen volume

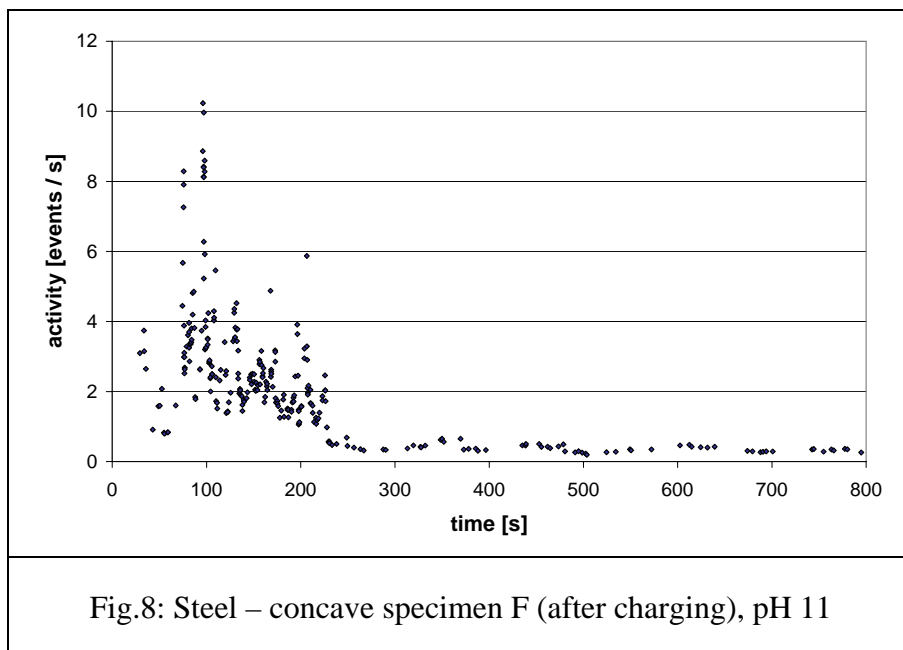
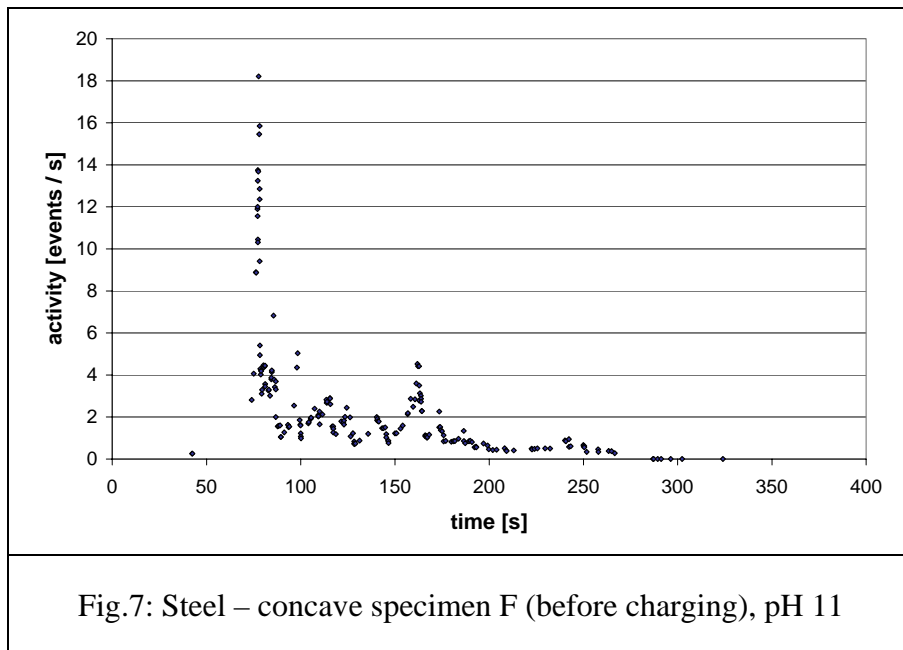
There were measured two sets of specimen from the same steel but with different geometry. The first set contained full cylindrical bars the second set contained cylindrical bars with cylindrical holes. It means that the exposed surfaces of all the specimens were the same but volumes were in ratio 2.29. The AE activity was much higher on concave specimens under the same other conditions (current, temperature, composition of the solution). It means that AE activity is highly dependent on the concentration of dissolved hydrogen in the metal. This piece of knowledge is in agreement with the facts connected with the time to initiation.

Influence of the nickel layer

The nickel layer caused the very high AE activity localised in time into very short intervals (see Fig. 10, 11). There is long time to initiation of this effect. It means that there is relatively high concentration of hydrogen in the nickel layer which is needed to start the process generating the high AE activity. This process seems to be similar to a breaking of some barrier or for example creation of pits.

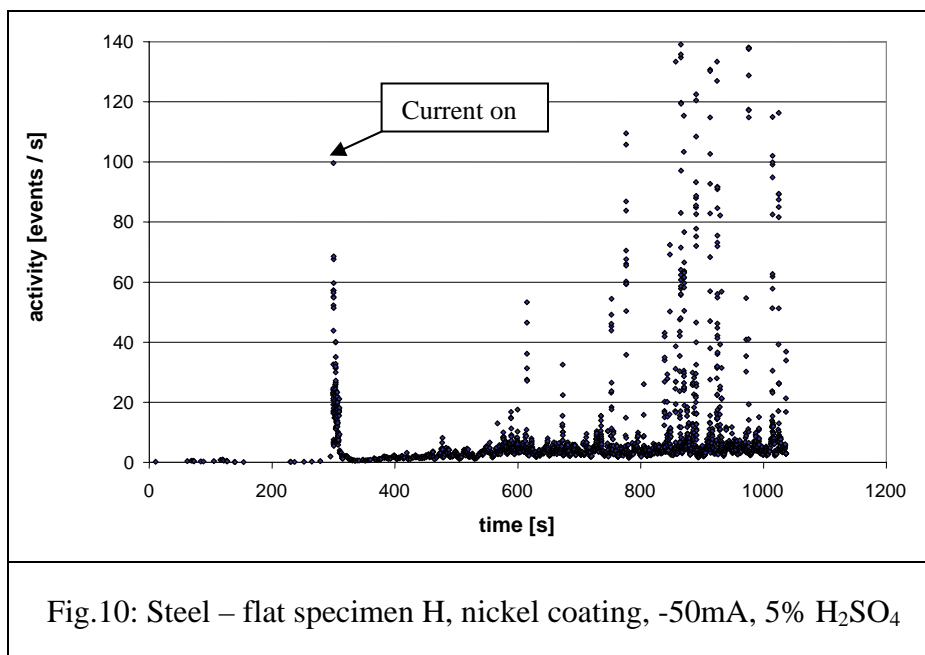
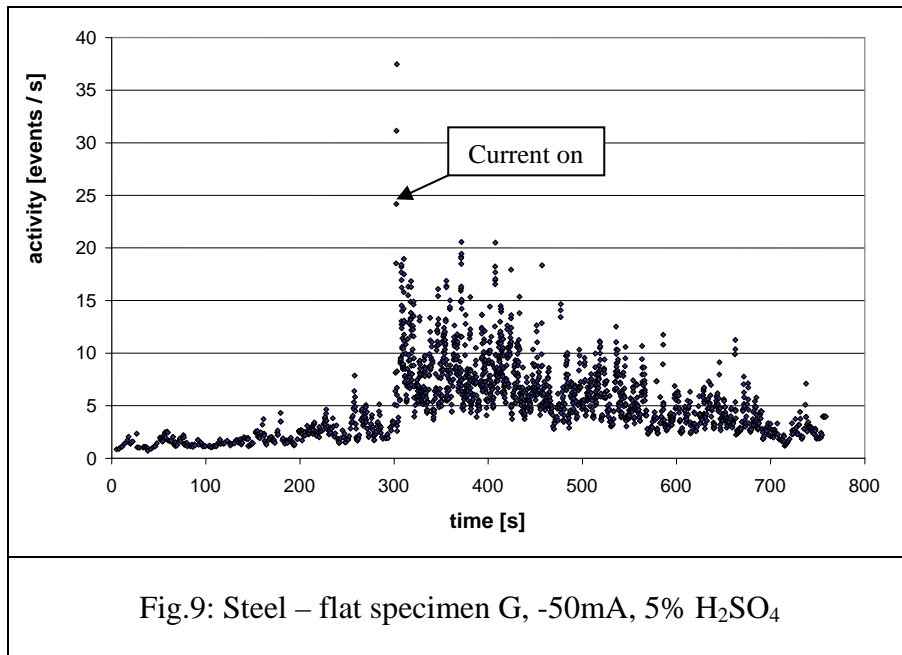
Source of the localised AE is probably related to transfer of hydrogen atoms between nickel layer and the alloy of the specimen. Similar character can be observed on steel

specimens as well as on the brass specimen. Detail explanation of the phenomenon will need some additional measurement and extended theoretical work.



AE during specimen cooling

Very specific part of the measurements is study of AE from specimen during cooling from the temperature 90°C. The specimens were cooled in non corrosive solution. In every case the AE response starts after several seconds after immersion, it means after the specimen got hot. The following course of AE shows activity during certain time (temperature) interval and the AE cease to exist somewhere about 80°C. Relation between full and concave specimens keeps the same character as quoted above; it means the concave specimen had higher AE activity.

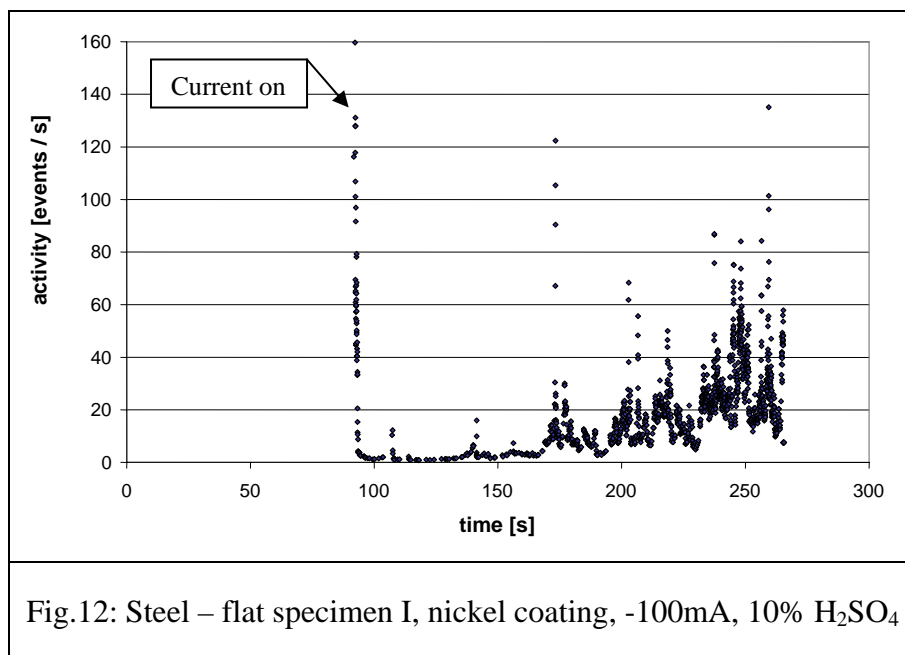
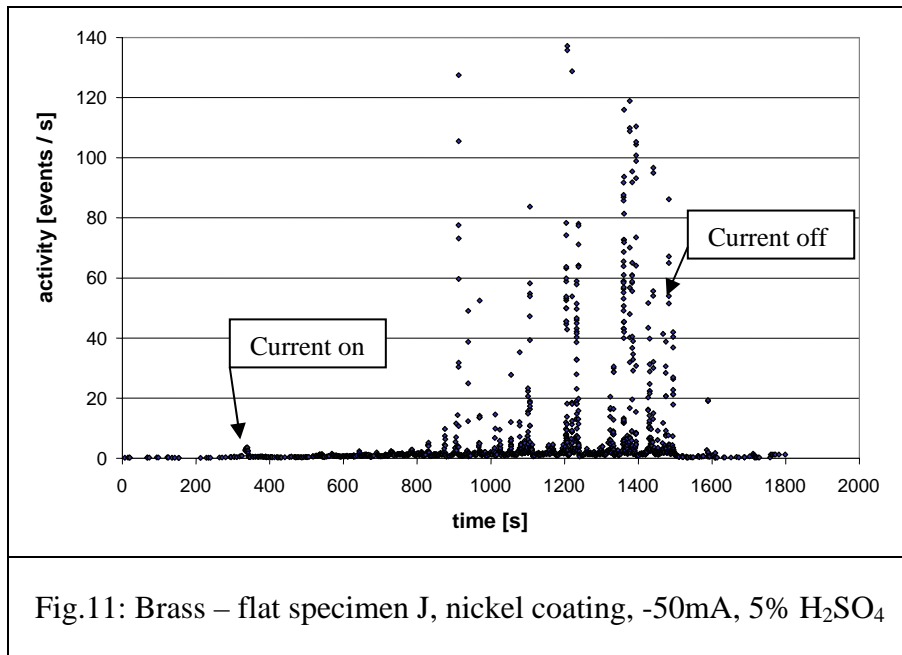


Conclusions

1. Measured AE is generated by processes inside material, not by bubbles.
2. AE depends on current density and hydrogen concentration. This follows from times to initiation and relation between AE of full and concave samples.
3. Nickel layer induces high AE which is concentrated into groups divided in time.

Questions

1. What are the real sources of AE in the case of hydrogen charging?
2. What is the mechanism of Ni-layer influence?



References

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