

# Hydrogen Influence on certain mechanical and magnetic properties of a stressed low – carbon steel after corrosion in NaCl - water solution

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## Abstract

Atomic hydrogen produced by corrosion of a low-carbon steel in NaCl- Water solution may markedly affect its certain tensile mechanical and magnetic properties in a complex and peculiar manner. This influence was investigated by employing the intrinsic micromagnetic emission (ME)-response as well as mechanical response of this ferromagnetic material, where relevant processes and parameters of micromagnetic and mechanical activity were implemented. In this fashion, it was shown that an increase in the hydrogen accumulation with corrosion time leads to an associated increase in the pervasive and embrittling influence expressed by a marked loss in ductility of the material. The competitive interplay of cumulative hydrogen, applied stress and plastic strain-induced microstructural damage was related to a specific ME-response parameter by which an increased magnetic hardening tendency of material with corrosion time was established. It was also shown, in an indirect way, that the embrittlement is a product of hydrogen accumulation by which certain mechanical properties are influenced.

## Introduction

There is a considerable evidence in the literature that atomic hydrogen may segregate by rapid diffusion at internal particle-matrix interface grain boundaries, microvoids and stress gradient sites by lowering the cohesive atomic bonding at these sites [1]. This in turn results in formation of extensive regions of voids. In unreformed materials the introduced atomic hydrogen will occupy any interstitial site in the lattice where it appears that in body – centered cubic materials the location is the tetrahedral interstice. Furthermore, it is also evident that in addition to the interfacial site hydrogen resides in traps such as dislocations and point defects, debris left behind the jogs of dislocations as they move through the metal during plastic deformation [2-3]. (During ears) to affect the mechanical properties of metal in a more specific way by controlling the ease by which dislocations nucleate and move through the crystal lattice. Many models suggested at this problem require the redistribution of hydrogen during the test. Especially, during slow – strain – rate tests at ambient temperatures hydrogen transported by moving dislocation, may essentially contribute to this redistribution [4, 5]. In this aspect it has been shown that hydrogen affects the plastic flow by promoting the onset of localized plastic instability and thus the premature fracture [3]. However, today, despite extensive research on these two problem groups a fully understanding of the complexity of hydrogen – assisted microstructural damaging processes is not possible [5-10]. Because of this, new variant aspects and a processes in the direction of mechanical and physical characterization of mechanically loaded steels after their exposure to corrosive hydrogen environment would be of significant importance for many structural steel components of various industries. Today, micromagnetic emission also called Barkhausen noise (BN) is a well-established, versatile and stress- sensitive, non- destructive testing method for a such characterization of steels at microscopic level where by using well known related micromagnetic events certain mechanical and microstructural changes in the material can be detected and analysed [11,12].

## Experimental Procedure

C	Mn	Si	Ni	Cr	Mo	S	P	N
0.05	0.40	0.015	0.017	0.01	0.001	0.014	0.01	0.003

Table 1. The chemical composition of Armeo – type low – carbon steel (%)

A block diagram of the experimental set up used for the measurement of micromagnetic Barkhausen emission (MBE) response is shown in Figure 1. The applied sampling frequency was 100KHz and maximum magnetic induction field of the excitation was about 20G. The MBE signal at 10 KHz magnetic excitation was acquired by a 2 mm ferrite surface probe which had 1000 turns and then amplified to 40dB using a low noise amplifier. The total number of Barkhausen counts (events), and the corresponding rms - voltage level was measured for one cycle of magnetization frequency using the counter processing module of the given apparatus. The specimen had dogbone – type geometry where thickness was 2mm, width 10mm and the effective gauge length 100mm. The samples were subjected to uniaxial tensile test at room – temperature and nominal low strain rate  $10^{-4}$ /s, using a universal testing machine of Instron- type. The ultimate stress was 380 MPa and the yield stress (0.2% offset) was 190MPa. Before testing, the samples were exposed to a corrosive environment produced by a continuously sprayed 3.5%NaCl aqueous solution in a Salt Spray Fog (SSF) apparatus. The produced corrosion layer was removed by dry air blast and soft natural bristle brush revealing underneath a black and strong adhering layer in form of magnetite ( $Fe_3O_4$ ) product. Afterwards, the magnetite layer was removed by appropriate chemical etching procedure. Other this layer may obscure the investigating hydrogen effect. The elemental composition of the used low-carbon steel is given in table 1.

## Results and Discussion

It is known that for the investigation using the Barkhausen ME- response among several measurement parameters the common count rate and corresponding root- mean- square voltage  $V_{rms}$ , signal are mostly used [11-12]. Nevertheless, some apparent ‘problems’ may arise by using these parameters separately. This is because the  $V_{rms}$  single reflects the qualitative behavior activity whereas count rate the quantitative one. To overcome, at least in part, this apparent ‘inherent weakness’ we propose to use an operational variable parameter  $J = V_{rms}/N$ , where the  $V_{rms}$  is the measured root mean – square voltage and N the corresponding detected number of counts (pulses). This means that this parameters, has dimension of energy rate per micromagnetic event detected in form of specific energy number indicative of the strength of a elementary jumping or displacement step of domain wall. Thus, the above parameter can be seen as a measure of the strength of the specific ME- response. It has been observed that an increase (decrease) in the number of pinning sites (N) would lead to an effective domain wall multiplication (reduction) to an associated decrease (increase) in the effective wall velocity and thus in the induced total sequel energy. From these factors one can finally deduce that an increase (decrease) in the number, N, of pinning sites (density), would result in an (increase) in the above ratio of J- parameter. However, this happens only in a stress – free or unloaded state of the material. As it will be shown in the following, this behavior of the J- parameter changes substantially for a stressed material. In this general context, it was observed, in this study, that the J-parameter may be a robust and characteristic experimental variable for the material in the sense that it may remain almost constant by changing the measurement locations on the specimen surface and especially by including the mostly inevitable lift- off effect simulated by varying the probe-surface inclination angle. Further, it was, in the present study, also observed that this parameter is almost independent an electronic setting on the apparatus such as signal gain and / or

threshold levels. In general, because this parameter has a more specific physical meaning and may alter under influence of certain mechanical and physical factors such as cumulative hydrogen as well as stress-strain induced micro structural changes, this parameter in the following will describe the physico-mechanical behavior of steel under these conditions. Moreover, in a more specific magnetic aspect the proposed J-parameter could be an indicator of the current state of the ME –spectrum changing from numerous, low energy wall jumps (small pulses) described by a decreasing J-parameter to view, high energy wall jumps (large pulses), described by an increasing J-parameter. In the context of the above-mentioned it is suggested to presume that an increase in the J-parameter may indicate an associated decreasing (increasing) magnetic hardening tendency of the steel. As such, the magnetic hardening of steel is an important micromagnetic property which as it will be shown later can be related with its hydrogen – assisted embrittlement process.

In the series of fig. 2-5, the physico-mechanical behavior expressed through the change of J-parameter with strain of the corrosion – free and corrosion – related material is presented. For convenience of the following discussion in each figure the corresponding stress – strain curve of the material was inserted. At first, it is interesting to observe the formation of a maximum in the J-parameter curve, similar to that in the stress- strain curve. This fact implies to assume that certain related microstructural changes should occur for both curves at this maximum point. Thus for the stress-strain curve, at this point, the well-known phenomenon of plastic instability in form of a localized necking sets on, which is a precursor of ductile fracture. At the same time, at this point, a state of high triaxial stresses develops by producing various internal microcracks and or microcavities which by coalescence lead to the final – fracture of the material. Because all of these, it seems logical to try to analyse, for example as shown in figure 2 the response behavior by two major characteristic ranges, a prenecking stage I, and a post necking stage II, separated by the critical strain point  $\dot{\epsilon}_c^*$ , corresponding to the ultimate tensile strength value. Thus, it would be helpful and of valuable practice to try to analyse, as shown in the same figures, the mechanical – induced magnetic response behavior on the similar basis of the above- introduced two characteristic mechanical response ranges: prior magnetic stage I\* and posterior magnetic one II\*, separated by the corresponding critical strain point  $\dot{\epsilon}_c^*$ , of maximum signal an evident overall increase in the J-parameter, within the range I\* is observed. This fact can be attributed to a compending influence of applied stress and plastic strain on the magnetic response. Primary, elastic tensile stresses favor an increase in the magnetic energy signal [11]. At the same time, however, plastic strain-induced pinning sites in form of dislocation pile-ups and tangles, exhibit a pronounced suppressive effect on the magnetic energy signal [12]. From the above, the dominating influence of the applied elastic and effective hardening stress over the plastic strain in increasing the J-parameter within the magnetic range precursor I\*, results. Beyond the characteristic strain point  $\dot{\epsilon}_c^*$  and within the posterior magnetic range II\*, the physico-mechanical behavior is characterized by an evident continuous decrease in the J- parameter of specific ME-response. It seems that, this time, within this stage, plastic strain – induced dominate over the applied stress effects in decreasing the specific ME – response. This is because, within the magnetic posterior range, the damage in form of microcracks, voids and other cavities which act as strong additional pinning sites can reduce the specific ME-response. More concretely, the above damage inhomogeneities under applied stress conditions may act as a dense – spaced local triaxial stress raisers which lead to an increase in the magnetoelastic. This afterwards, as earlier explained, results in a decrease in the effective wall velocity and consequently in the rate of magnetic volume change and hence in the induced  $V_{rms}$  energy signal. Furthermore, the interesting fact concerning the total shift of the physico-mechanical J- curves with corrosion time towards lower strains is indicative of occurrence of hydrogen-assisted environmental embrittlement processes. This shift is consistent with the total shift of the related (mechanical) stress-strain curves towards lower strains with time of corrosion as shown the series of in figs. [2-5], where an evident reduction in the ductility of the material can be deduced. It is mentioned that similar shifts due to hydrogen embrittlement have been obtained elsewhere [10]. With respect to this it should be pointed

out that a related convincing evidence of occurrence of hydrogen embrittlement in the present material was gained by direct microscopic indicators of as detailed SEM- microfractographic analysis [6]. Moreover, at the same time an overall shift of the J- magnetic curves to lower magnetic values with increasing corrosion time can be observed. This behavior presents another indirect indicator of magnetic hardening tendency of the steel with hydrogen accumulation caused by corrosion process. This argument may be supported by the effective reduction of the MBE-signal due to magnetic hardening of material observed elsewhere [12].

## Conclusions

Certain physical and mechanical properties of a low-carbon steel, determined by its mutual tensile and micromagnetic (Barkhausen) emission response, may considerable be influenced by cumulative atomic hydrogen produced under corrosive NaCl – water solution environment. The influencing factors can better be revealed, described and analyzed by introducing a relevant specific micromagnetic Barkhausen emission parameter and employing certain intrinsic processes of ferromagnetic activity and mechanical behavior. In this manner one can show that an increase in the hydrogen accumulation with corrosion time leads to an associated increase of the embrittlement of steel, expressed by appreciable loss in its ductility as well as to a parallel increase in the magnetic hardening tendency expressed by a general reduced specific Barkhausen micromagnetic emission parameter.

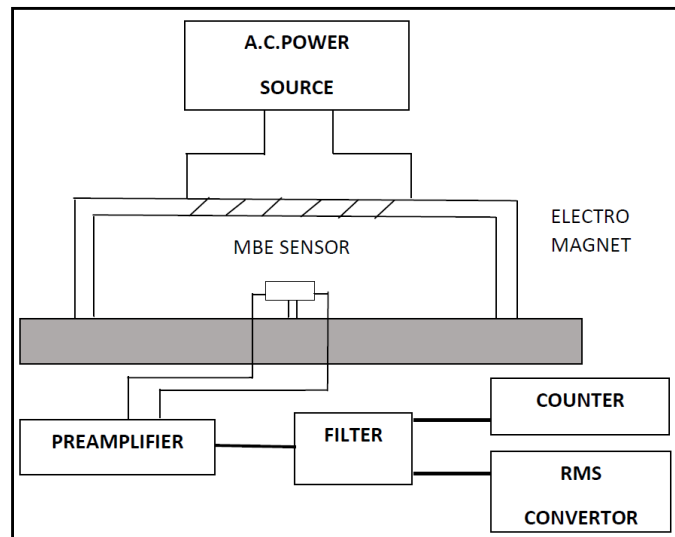


Figure 1: Block diagram of the experimental MBE – setup.

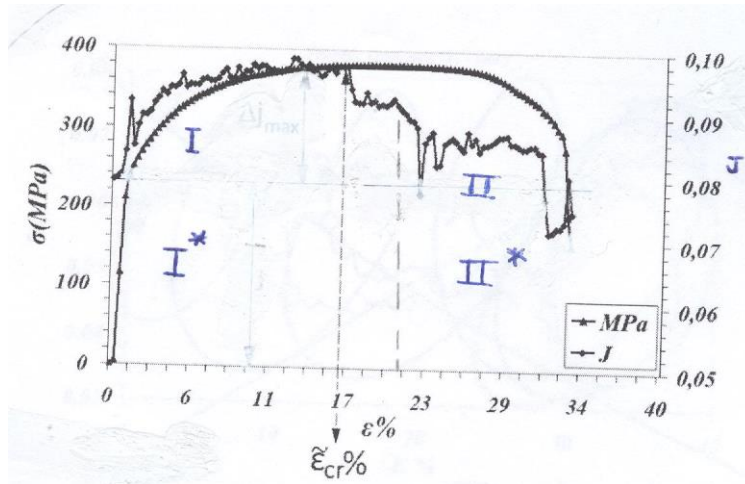


Figure 2: Mechanical stress-strain and magnetic J-response curves for virgin (as received) specimens.

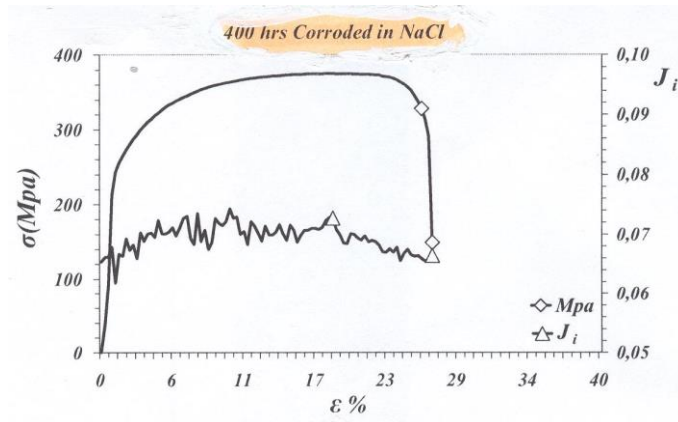


Figure 3: Mechanical stress-strain and magnetic J-response curves for 400 hours of corrosion time.

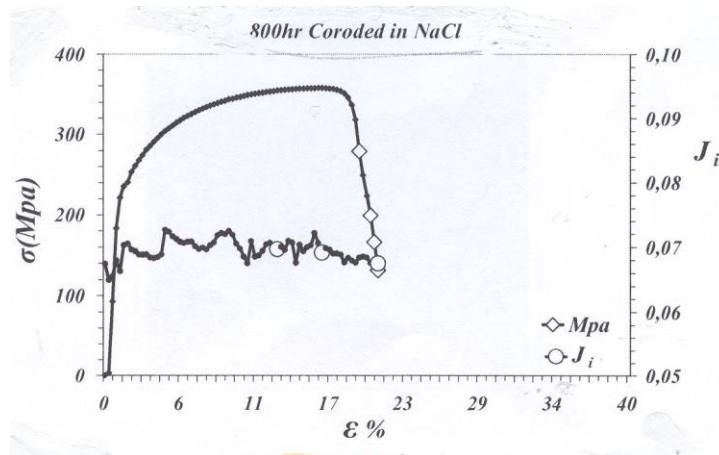


Figure 4: Mechanical stress-strain and magnetic J-response curves for 800 hours of corrosion time.

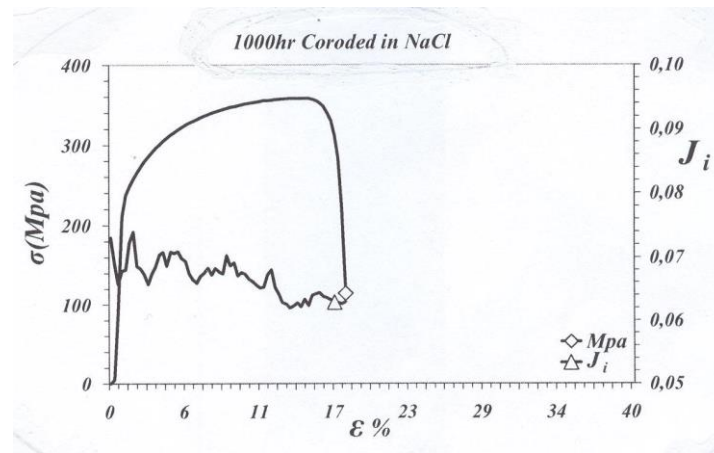


Figure 5: Mechanical stress-strain and magnetic  $J$ -response curves for 1000 hours of corrosion time.

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