CONDITIONS OF THE "FISH EYES" FORMATION IN THE PROCESS OF HYDROGEN INDUCED DESTRUCTION

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Summary: The purpose of this paper is to present the results of structural and mechanical investigations of A508.3 and 10H2M steels saturated with hydrogen. The influence of hydrogen concentration on the mechanical behaviour of a material during destruction in a static tensile test is discussed. The results of investigations of J-R resistance curves of 10H2M steel are presented. An attempt has been undertaken to correlate geometrical features of the so called "fish eyes" with crack resistance of the investigated materials.

INTRODUCTION

A determination of mutual quantitative relationships between mechanical properties, hydrogen concentration in a microstructure and stereological properties of fractures is one of the basic issues on the way to evaluate the susceptibility of steel to hydrogen evoked destruction [1, 2]. In order to determine these relations, an attempt was undertaken to correlate the results of investigations of the basic mechanical properties obtained in a static tensile test for two grades of steel, with selected parameters describing the morphology of fracture surfaces, by means metallography and quantitative fractography methods [3÷6]. Afterwards, the methodology developed was verified by statistical methods. A further analysis of the correlations thus obtained created a possibility of describing hydrogen induced cracking in the investigated material, by means of a model based on a fracture mechanics criterion and determining the influence of hydrogen concentration on the cracking micromechanism.

The subject of detailed investigations was the hydrogen interaction in A508.3 [7÷9] and 10H2M steels. In those materials, hydrogen was the cause of formation of defects of a special type, called "fish eyes", on the fracture surfaces of the test specimens. The "fish eyes" are the regions of a quasi-brittle transcrystalline fracture, rosette-shaped, which contain in their centres one or a few non-metallic inclusions [10, 11]. The "fish eyes"' quantitative parameters were used to develop a mathematical model of cracking based on distortion interactions between "hydrogen traps" which constituted the non-metallic inclusions together with the surrounding hydrogen. Since the fractographic investigations revealed that the cracks of "fish eye" type are accompanied, in the initial stage, by a quasi-brittle fracture together with a plastic strain zone, in numerical calculations the plastic and elastic properties of a material with linear hardening were taken into account. Those calculations aimed at determining the state of stress during a simultaneous influence of hydrogen traps and external load. At the same time, they created a possibility of kinetics description of the plastic zone growth, the zone being responsible for the process of "fish eyes" formation and coalescence [9].

Due to a revealed nature of a fracture in the environment of hydrogen traps, for the description of the critical state of the specimen's material, the resistance curve J-R was used.

In the paper, an attempt is undertaken to explain the formation mechanism of microcracks of a quasi-brittle nature - "fish eyes" against the background of brittle fracture. Additionally, quantitative relations are defined between the material's crack resistance and "fish eyes" dimensions.

MATERIAL INVESTIGATIONS

The results of investigations of A508.3 steel were presented in earlier papers [1,2,7,8,9]. Similar investigations were performed for the heat-resisting steel 10H2M, applied in traditional power engineering, among others for steam superheaters.

Mechanical properties investigations

The investigations of the basic mechanical properties were performed on a testing machine MTS 20/M. The investigations results are presented in Table 1. The presented numerical values are average values obtained from the investigations of 3÷5 specimens. In respective columns, information is given on the kind of material and hydrogen saturation conditions in time of 24 h. Next, the values of the yield stress $R_{0,2}$, resistance R_m, elongation A_5 reduction of area Z, strain ε_m corresponding to the maximum force P_m and the embrittlement coefficient El = $[(\varepsilon_{m(air)} - \varepsilon_{m(hydr.)}) / \varepsilon_{m(air)}]*100\%$ are quoted.

TABLE 1. Investigation results of basic mechanical properties of 10H2M steel in the initial state and after hydrogen saturation at a room temperature for 24 h.

Kind of specimen	$R_{0,2}$ (MPa)	R_m (MPa)	A_5(%)	Z%	ε_m (%)	El (%)
10H2M - air	383	555	18,6	58,9	13,9	-
10H2M-0,5 mA/cm^2	502	551	13,9	9,8	13,4	3,6
10H2M-5 mA/cm^2	409	536	6,1	8,8	5,7	59,0
10H2M-10 mA/cm^2	398	531	7,9	5,4	7,5	46,0

Fractography investigations

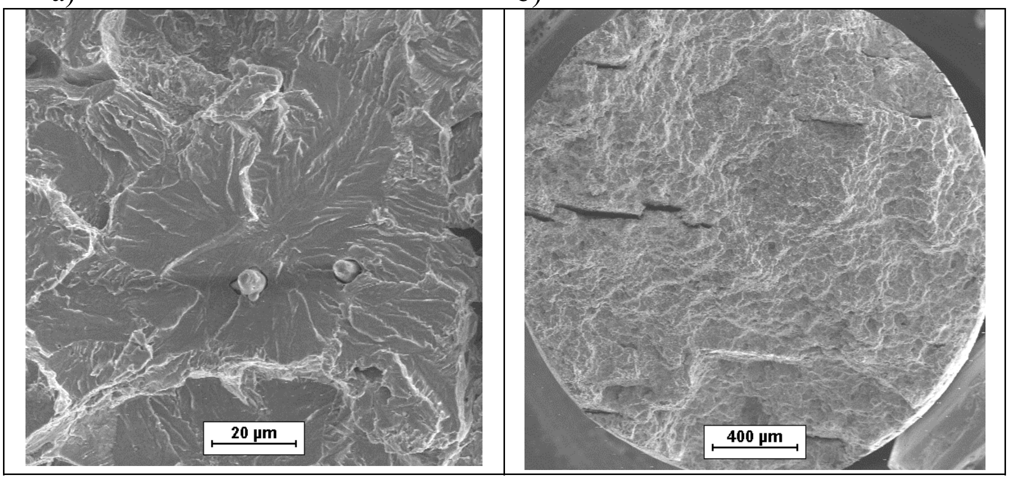
The fractographic investigations were aimed at making an observation of the fracture morphology of specimens in the initial state and after their saturation with hydrogen. Saturating of the specimens with hydrogen was performed electrolytically in a water solution of 1M HCl with an addition of potassium thiocyanate KSCN (1g/l) and hydrazine N_2H_4 with current densities in the range from 0,5mA/cm^2 to 50mA/cm^2, during 24h.

The examinations of fracture surfaces were performed on a scanning electron microscope with a field emission FE SEM S-4200 HITACHI cooperating with an X-ray spectrometer VOYAGER 3500 of NORAN INSTRUMENTS.

The observations showed that in the strength tests, the fracture of specimens without hydrogen is ductile. In specimens saturated with hydrogen, on the fracture surface the oval regions of a quasi-brittle nature were observed, with a non-metallic inclusion in the centre, so-called "fish eye". Those regions are separated from one another by ductile fracture bands, so-called bridges. At the highest current intensities applied for hydrogen saturation, secondary cracks were observed on the fracture surfaces. Exemplary scanning pictures are presented in Figure 1.

Figure 1: Specimen fractures after hydrogen saturation at a current density of 0,5 mA/cm^2; steel 10H2M, test temperature 293K; a) "fish eyes" with non-metallic inclusions, b) secondary cracks.

a) b)



Fracture toughness investigations

The investigations were performed for the 10H2M material in the initial state. The specimens were taken from a 35 mm thick pipe section after toughening. The cracking plane was located perpendicularly to the pipe axis. The specimens' dimensions are presented in Table 2.

TABLE 2 Specimens' dimensions SEN(B) in the investigations of J-R resistance curve.

Material – specimen	B (mm)	W (mm)	a_0 (mm)	Δa (mm)	a_0/W
10H2M – 2	18.02	35.84	18.393	5.001	0.51
10H2M – 3	18.04	35.88	18.776	4.298	0.52
10H2M - test	18.00	35.88	19.210	5.000	0.53

The investigations were carried out on a MTS hydro-pulsate machine, controlled by means of a MicroProfiler 458.91 controller. A detailed description of the research methodology is included in papers [12,13]. In the experiments, the compliance method as well as the method of electric potential drop of the J_{Ic} integral value determination was applied. The research was performed according to PN 88/H-04336 and ASTM E 1820-99a standards.

To determine the J-R curves, for three-point bent specimens SEN(B), Rice's and Ernst's formulas were used, Eq. 1 and Eq 2 respectively:

$$J_0 = \frac{\eta \cdot A}{B \cdot b_0} \tag{1}$$

$$J_{(i+1)} = \left[J_i + \left(\frac{\eta}{b} \right)_i \cdot \left(\frac{A_{i,i+1}}{B} \right) \right] \cdot \left[1 - \left(\frac{\gamma}{b} \right)_i \cdot \left(a_{i+1} - a_i \right) \right] \tag{2}$$

where $\eta = 2$ and $\gamma = 1$ for three-point bend specimens, if the relative length of crack a_0/W is greater than 0.282, $A_{i,i+1}$ is a surface section under the P-Δ curve (force – actuator displacement) between selected steps Δ_i and Δ_{i+1}, *B* is the specimen thickness, b = W-a, W is the specimen width, a is the crack length. The indices i and i+1 determine a consecutive measuring step in which the J integral was determined.

The results of J-R curve investigations, based on Eq. 1 and Eq. 2 formulas, are presented in Figure 2 and Table 3. The investigations were carried out at a room temperature.

For the A508.3 steel, the value of the J integral was evaluated on the basis of investigations of the critical stress intensity factor K_{Ic}, performed by

Beremin [14] for a temperature range from 77 K to 273 K. The values of J_{Ic} were derived from Eq. 3:

$$J_{Ic} = (1-\nu^2)\frac{K_{Ic}^{\ 2}}{E} \tag{3}$$

The results of calculations and of experimental investigations [9] are presented in Table 4.

Figure 2: J-R curves determined by the electric potential drop method according to Rice's model.

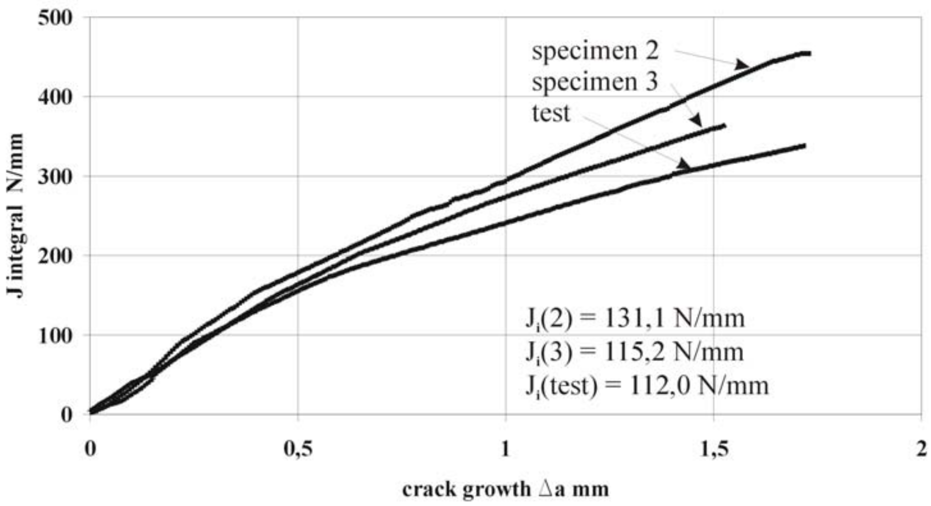


TABLE 3. Values of $J_i = J_{Ic}$ integral for 10H2M steel at a room temperature.

Model	Rice [kN/m]	Ernst [kN/m]
Average value J_{Ic}	123,6	121,8

TABLE 4. Selected mechanical properties of A508.3 steel.

Test conditions	K_{Ic} MN/m$^{3/2}$	J_{Ic} kN/m	$R_{0,2}$ MPa	R_m MPa
293 K (air)	109	54	574	661
293 K (hydrogen)	-	-	554	670

MODEL OF THE "FISH EYES" FORMATION

As the fractographic investigations revealed, (see Figure 1), the fracture surface in a direct neighborhood of a hydrogen trap is of a quasi-brittle nature and differs from the remaining part of the final fracture. This phenomenon occurs for both investigated materials. Relying on a hypothesis that this surface is created during subcritical cracking of the material in the stage of a so-called stable crack growth, an attempt was undertaken to estimate the value of the J integral, at which "fish eyes" are formed of dimensions revealed in the material investigations. Figure 3 illustrates the applied methodology.

The diagram presented in Figure 3 is a fragment of the J-R curve presented in Figure 2 and is related to the initial stage of cracking, whereas the picture of a crack illustrates, in a visual way, the method of finding the J_c integral value for a single "fish eye".

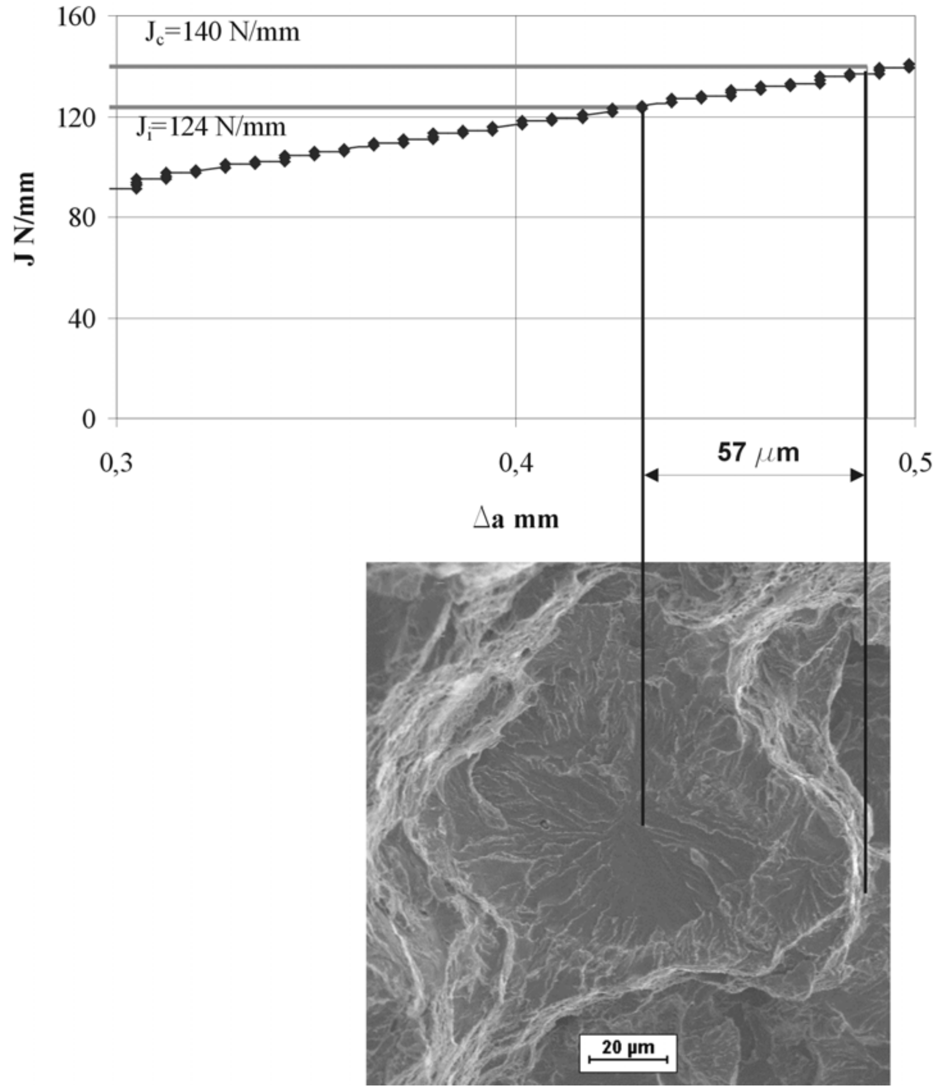
DISCUSSION AND RESULTS

In the paper an attempt was undertaken to use the J-R resistance curve determined on the basis of laboratory investigations for an evaluation of the mechanical condition of a material, in the volume of which spatial internal defects are formed, around which an internal stress field caused by hydrogen pressure is generated. On the basis of the stress state similarity in the specimen crack tip and on the front side of a "fish eye", a hypothesis is postulated that the additive interaction of all hydrogen traps revealed in the critical plane (fracture surface) will decide on the limit state of the material of a given object.

In the case of the investigated specimens, a linear summation of the formed "fish eyes" surfaces is proposed. By measuring the total "fish eyes" area on the fracture surfaces of the investigated specimens, the surface fraction of "fish eyes" can be determined in relation to the total fracture area, and on this basis, a substitute radius of a so-called "fish eyes" colony can be calculated. Afterwards, using the J-R resistance curve plot, the value of a reduced $J_{c\text{-}red}$ integral can be determined, at which destruction of the specimen takes place.

The investigations carried out on the steel A508.3 [2,7,8,9] showed that a hydrogen content of 7,2 ppm concentration introduced by means of a cathodic saturation method at a 5 mA/cm^2 current density in 24 hours, did not bring about significant changes in strength properties of that material. However, in spite of a relatively low value of the J_{Ic} integral, see Table 4, the material showed good ductility that is proved by 37% reduction of area (after hydrogen saturation by 5 mA/cm^2 current density) as well as by big dimensions of the "fish eyes".

Figure 3: Determination of the J_c integral value.



For example, at room temperature the diameter of a "fish eye" was $\bar{D}$=0.294 mm and that of the hydrogen trap $\bar{d}$=0.033 mm. In case of the 10H2M steel, for the same conditions of hydrogen saturation, the following results were obtained: $\bar{D}$=0.0300 mm and $\bar{d}$=0.0045 mm, at a fracture resistance of the material in the initial state J_{Ic}=124 N/mm, which is more than twice as great as in case of the A508.3 steel. Moreover, for 10H2M steel a decrease of plasticity reserve was found as well as a significant decline in ductility (about 59%), a measure of which can be the embrittlement coefficient El, see Table 1. This fact shows the great sensitivity of this material to hydrogen presence and its disadvantageous influence on the mechanical properties. The destructive influence of hydrogen was most evident from specimens' fractures by a very high dispersion of "fish eyes", the dimensions of which, $\bar{D}$ and $\bar{d}$, are smaller by almost one order of magnitude than in the A 508.3 steel. This also shows that the steel 10H2M is less susceptible to "fish eyes" colony formation and, at the same time, the stadium of subcritical cracks extension is shorter. In these cases an evaluation of the material's crack resistance should be done on the basis of a resistance curve obtained for a material with a similar hydrogen concentration.

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