ON ACOUSTIC REGIME AT FRACTURE OF STEEL SPECIMENS AFTER HOLDING IN CORROSION MEDIUM

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ABSTRACT

Acoustic emission parameters characterizing the fracture process at tension of double beam specimens from the low-alloy steel were estimated and fracture mechanisms at different stages of crack development were studied. The steel was embrittled in result of holding in corrosion medium. The amplitude distributions of AE signals were obtained and the cumulative distribution curves of acoustic emission events by their magnitude were plotted. The value of the $b_{AE}$ parameter was evaluated by the slope of distribution curve which is described by the relation $\log N = C_{AE} - b_{AE}M$, where $M$ is the signal magnitude measured in decibels, $N$ is a cumulative number of the AE signals, $C_{AE}$ is a constant.

It was shown that the increase in holding time in corrosion medium changes the form of loading curves and leads to appearance of several sharp drops of load for the embrittled specimens. Fractographic investigation showed the presence of the ductile dimples on the fracture surfaces of the as received material and the marks of crack jumping and cleavage facets on the fracture surfaces of embrittled one, indicating two different mechanisms controlling the fracture process.

The relationship between the character of the time changes in a load, the $b_{AE}$-parameter and the fracture mechanism was established. It was shown that in the most cases, the decrease in the $b_{AE}$-parameter precedes the crack jump as a result of appearing of acoustic events with a large magnitudes, so that the $b_{AE}$ parameter may serve as a precursor of a localized fracture. The properties of the form and spectrum of AE signals for the different fracture mechanisms were investigated and suggested as the informative characteristics of a localized fracture.

INTRODUCTION

Hydrogen induced cracking occurring in pipe steels under the conditions of hydrogen sulfide environment considered to be a serious problem of the oil and gas industry. As the hold time in this medium is increased, multiple origins of fracture initiate near structural stress concentrators (oxides, sulfides, carbides, grain boundaries and dislocation piles up), which play the role of hydrogen traps. At loading these multiple scattered fracture sources develop and lead to localization of fracture, i.e. to main crack formation. Kinetics of this process may be studied using acoustic emission method, allowing to observe the process in a real time regime. This paper deals with analysis of fracture stages in the low alloyed steel 30G2 after holding in hydrogen sulfide containing medium using the acoustic emission and fractographic methods.

Material and experimental details

For analysis of acoustic emission changes during fracture, double beam specimens from the low-carbon steel 30G2 (0,30-0,35%C, 1,1-1,35%Mn, 0,17-0,37% Si, 0,035% P, 0,035% S, 0,010%Cr, 0,30% Ni, 0,035% Ni, 0,010%Cr, 0,30% Ni,
0.30%Cu, 0.08%Al, Fe balance) were tested at tension conditions with the loading rate equal to 0.5 and 1 mm/min. This steel had a ferritic-pearlitic structure with sulfides whose mean statistical dimensions were 600x30x5 µm and the following mechanical characteristics: ultimate tensile strength 655 MPa, yield strength 380 MPa, elongation 16%, reduction in area 50%, hardness HRC 18. The specimens were precracked and hydrogen charged by holding for 360 and 2160 hours in a NACE medium causing the material embrittlement [1]. After that the specimens (14 pieces) from as received and embrittled material were tested at tension with measuring the acoustic emission (AE) parameters. The amplitude threshold level was set up using the results of a preliminary testing a specimen without a notch in order to exclude acoustic signals, coming from the loading device captures. The AE information obtained during testing included a sequence of AE signals (digitized with sample rate equal to 2 MHz), the arrival time of signals for each piezoelectric sensor and corresponding loading value. Data processing was done using both the form of signals and the set of parameters, extracted from the signal, namely, peak amplitude, rise time, energy, counts, etc.

In result of experiments, the amplitude distributions of AE signals were obtained. The characteristic of the cumulative distribution function, $b_{AE}$, was evaluated as a tangent of the slope angle of straight, obtained by means of the line approximation of the distribution curve using the mean least-squares method. Due to its importance, $b_{AE}$-parameter was included in American Standards on estimation of AE characteristics [2] as a parameter most fully reflecting a stochastic nature of the AE signals waves during material fracture.

The linear relation describing this cumulative distribution curve is the following:

$$\log N = C_{AE} - b_{AE} M,$$

where $M$ is the signal magnitude measured in decibels, $M = 20 \log (A/A_0)$, $A$ and $A_0$ are the current and threshold amplitudes, correspondingly; $N$ is the number of the AE signals with an energy lower than $E \sim A^2$; $C_{AE}$ is the constant.

The $b_{AE}$-parameter was evaluated by two methods. In the first case, the dependence of logarithm of the cumulated number of AE events on the magnitude was plotted for all acoustic events, obtained during specimen fracture. In the second case the temporary dependence of the $b_{AE}$-parameter was analyzed. Each meaning of $b_{AE}$-parameter for latter case was estimated in a sliding window using formula (1). The size of the window, which depends on AE activity, was set up to 50 points. Every next point of the $b_{AE}$-time dependence was obtained by shifting the window with a step equal to 3-5 points. The linear smoothing of the curve was made at the termination of the processing procedure.

Fracture surfaces of the specimens were studied using optical and electron microscope SEM-505.

**Study of fracture surfaces of specimens**

In Figure 1 the typical loading curves and the time dependencies of $b_{AE}$ for two specimens with different degree of embrittlement are presented. As follows from these graphs, the increase in holding time in corrosion medium changes the form of loading curves and leads to appearance of several sharp drops of load (Figure 1b). These drops are absent on the loading curve corresponding to as-received material (Figure 1a). Fractographic investigation shows that the load drops are connected with the jumps of a brittle crack which can be seen on the specimen fracture surfaces. In the jump region, crack grows quickly by mechanism of transgranular cleavage (Figure 1c, right); in the other regions it develops slowly by a ductile mechanism with formation of dimples (Figure 1c, left). The ductile fracture is also found at fracture surfaces of as-received material. It is established that the fracture process of latter is controlled only by the ductile mechanism so that the marks of crack jumping are absent on its fracture surfaces.

**Analysis of time dependencies of $b_{AE}$ parameter and cumulative magnitude-frequency distributions.**

As follows from these graphs in Figure 1, the changes in $b_{AE}$-parameter correspond to those in loading curve. The first reduction in the $b_{AE}$-parameter is connected with beginning a plastic flow near a crack tip. Other reductions of this parameter precede the load drops in result of crack jumping.

It is related with the appearance of the AE events with high magnitudes probably due to the coalescence of microcracks in the plastic zone at the main crack tip before its growth. It shows that the decrease in $b_{AE}$-value may be used as the precursor of the localized fracture. The decrease of $b_{AE}$ is preceded by its increase...
as a result of generating of many signals with the low magnitudes connected to the plastic deformation mechanisms. The comparison of graphs in Figure 1 shows that the material embrittlement leads to appearance of periodic changes of $b_{AE}$–parameter due to the crack jumps.

Figure 1: (a) The time dependencies of load and the $b_{AE}$-parameter and corresponding fractograms, obtained for two kinds of tested specimens: (a) as received ductile material; (b) embrittled material; (c) microrelief of fracture in different regions on the fracture surfaces.
In Figure 2 the cumulative frequency-magnitude distributions obtained for three periods of loading are shown. The distribution curve corresponding to the initial stage of deformation curve (without crack jump) can be described by the linear dependence in logarithmic scale, Figure 2b. Such curves for time periods including crack jumps have a knee in the high magnitude region, Figure 2c, d. The $b_{\text{AE}}$-values corresponding to the linear part of curves decrease with the growth of crack jump number.

Figure 2: The time dependence of AE energy (a) and the cumulative magnitude-frequency distributions for three time periods: (b) $T_1 = 320$ sec; (c) $T_2 = 480$ sec; (d) $T_3 = 1870$ sec.

**Study of AE signal form**

Study of the form features of AE signals accompanying fracture process may serve as a sensitive instrument in analysis of deformation and fracture mechanisms. For this purpose several informative parameters were extracted from the form and spectrum of AE signal, such as peak amplitude, time rise defined by time to amplitude peak, signal duration, partial energies characterizing the part of a signal energy in different frequency bands and others. Using these parameters allows to observe two types of AE signals, corresponding to the ductile fracture mechanism and the brittle one.

The first type of the signals is characterized by large amplitudes, high frequencies and short rise times. On the contrary, the second type is related to comparatively low amplitudes and frequencies and large rise times. It was shown that the signals of the first type dominated on the initial region of loading curve connected with plastic deformation, and also on the stage of ductile fracture, while the number of signals of the second type increased before the brittle crack jumps. Therefore the ratio of the parts of signals belonging to the indicated types may considered as useful diagnostic criterion of a localized fracture.
Discussion

Analysis of fracture processes on various scale levels allows to suppose that the time dependence of $b_{AE}$ value and the cumulative number of acoustic events are universal. So, in [3] was shown that cumulative number of fatigue microcracks in specimens of carbon steel is connected with their length ($L$), which is proportional to acoustic emission magnitude, by the following relation similar to Eqn. 1:

$$\log N = A_C - b_C L, \quad (2)$$

where $N$ is the cumulative microcrack number with the length equal or lower than $L$, $A_C$ is the constant. Coalescence of microcracks and transition to localized specimen fracture lead to almost twofold decrease in $b_C$-parameter.

Estimation of acoustic emission in tests of rock specimens used for modeling seismic activity before earthquake [4, 5] found the time change in $b_{AE}$ analogous to that observed in current study. Magnitude dependence of cumulative number of acoustic events was similar to Eqn. 1 and to well-known relation of Gutenberg-Richter describing magnitude relationship of cumulative number of seismic events:

$$\log N = B_S - b_S M, \quad (3)$$

where $N$ is the cumulative number of seismic events with an energy equal or lower than $E$, $M$ is the magnitude, $M = \log E$, $B_S$ is the constant.

Moreover, as follows from the studies conducted in [3–6], the $b_S$-parameter decreases in result of crack coalescence before formation of main fault. The time dependencies of $b_{AE}$-values evaluated at testing rock and metal specimen are also similar and their values are close.

The analogy of the relations noted above (Eqns. 1-3) and the time dependencies of $b$-parameters means that fracture processes in solids are characterized by the universal features which may be consequence of self-similarity of fracture kinetics on various scale levels.

Conclusions

1. The influence of hydrogen sulfide medium on mechanical and acoustic properties of the low alloy pipe steel was studied.
2. The results of fractographic and AE studies indicated that the crack growth in steel after holding in corrosion medium was discontinuous in nature and may be characterized by change in the $b_{AE}$ parameter.
3. The AE cumulative frequency-magnitude distributions for the different time periods of loading were obtained and the changes of $b_{AE}$-values as a function of time were evaluated. The interrelationship between the changes in the $b_{AE}$ parameter, the load level and fracture mechanism is established.
4. It is shown that the form and spectrum of AE signals may be used for identification of fracture mechanism of steels after holding in corrosion medium.
5. It is established that the $b_{AE}$ parameter may serve as a criterion of a degree of hydrogen embrittlement.
6. It is supposed that the regularities of changes of the $b_{AE}$ parameter with time and also cumulative acoustic events with magnitude have the universal character and are similar to those observed on various scale levels.

REFERENCES