DETERMINATION OF INFLUENCE OF THERMO-CYCLES ON HYDROGEN CRACKING OF STEEL

Valentyn SKALSKY, Denys RUDAVSKYY, Yurij MATVIJIV

H. V. Karpenko Physico-Mechanical Institute of National Academy of Sciences of Ukraine, Lviv,
Naukova Str5., Lviv, 79601,Ukraine, skal@ipm.lviv.ua

Summary

Influence of thermo-cycles "heating-cooling" on the hydrogen induced cracking of structural steels, which are widely used in the heat-power equipment is investigated in this paper. An acoustic emission method of diagnostics of specimens made of exploited and non-exploited material of steam pipelines is described. Effective diagrams of dependence of number of events on heat cycle number for the all groups of specimens are plotted.

Keywords: heat cycling, hydrogen concentration, acoustic emission.

1. INTRODUCTION

For express-evaluation of material liability to hydrogen degradation, it is heat cycled in hydrogen of pressure higher then the working one, from working temperature that is characteristic for concrete technological process to the room temperature [1]. Then the nucleation and growth of crack type defects is conducted by radiation of elastic waves known as acoustic emission (AE). Its parameters allow estimating an intensity of initiation and equivalent areas of mentioned defects during heat cycling. The results obtained in this experimental research are described below.

It is generally known that the amount of hydrogen, which can dissolve in a metal, increases with temperature. This phenomenon and high diffusive mobility of hydrogen atoms in α-Fe at high temperature could be the ground of the laboratory express-method of accelerated high temperature hydrogen steel degradation [1]. The abrupt temperature decrease at the cooling stage considerably retards diffusive processes (diffusion coefficient of hydrogen in α-Fe decreases from $1.8\cdot10^{-4}$ to $4.4\cdot10^{-5}$ sm^2/s with decrease of temperature from 540 to 100°C [2-4]) and is accompanied by localization of dissolved hydrogen in metal at lower temperature, which concentration is over-equilibrium (from 0.1 to 1.0 sm^3/100 g of metal, according to [5]). In consequence of cooling, approximately the same concentration of hydrogen as an equilibrium one for temperature of 540 °C is observed inside a specimen. Obviously, that this hydrogenation assists in active migration of hydrogen to the nearest free surfaces, regardless of their location (internal or external). Thus residual hydrogen either goes out from a metal (from the subsurface layers of specimen) or gathers in traps at structural defects, grain or phase boundaries, which from the view point of energy expenditures are the advantageous places of its location (inside the specimen). As hydrogen fills any defects, its migration assists in moving of carbon atoms and alloying elements. It occurs because of degrees of energy barrier (affected by hydrogen) to moving of atoms of any element [6]. It is clear that such redistribution of elements accelerates the redistribution of carbides and formation of net of the fine-dispersed special or alloyed carbides in places with high concentration of alloying elements.

During the cooling, the redundant hydrogen goes to the traps at grain, phase boundaries or structural defects. Migration of hydrogen creates a track of high-gradient tensile field, assisting in the redistribution of carbon and alloying elements in a metal and, therefore, intensifies microstructure transformation of steel. As hydrogen localizes at these defects, it creates additional tension at interphases. At the frequent reiteration of this cycle pre-conditions for the formation of microscopic damages as micro-voids and micro-cracks are created. After each heat cycle the pressure of hydrogen grows, deformation of linkages between micro-voids and micro-cracks and their creep growth as result of the combined action of high temperature and loading becomes possible. These processes are conducted by the radiation of elastic AE waves [7].

In addition, such heat cycling of specimens in hydrogen creates pre-conditions for cyclic deformation of metal near the defects, which accumulate hydrogen. Amplitude of deformation is determined by many factors: distribution of defects by their sizes and amount, temperature range of heat cycling, pressure of hydrogen in a chamber, number of heat cycles etc.

Thus, the abrupt cooling of hydrogenated metal, at first, causes an increase of internal stresses in metal as a result of over-equilibrium concentration of hydrogen in metal, secondly, assists in the initiation of micro voids filled by hydrogen, thirdly, intensifies diffusion, in particular of carbon, assists in transformation of microstructure. All these processes assist in intensive crack formation in steels and can be effectively detected by AE signals.

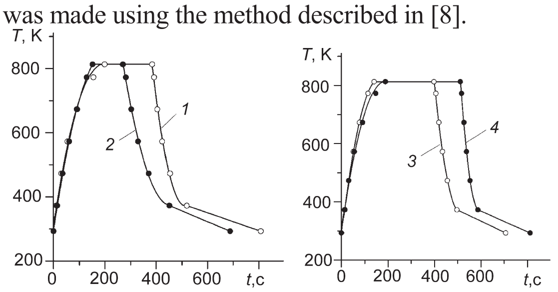
2. METHOD OF AE TESTING

Experiments were carried out for the four groups of smooth beam specimens of 12H1MF steel of sizes 12×18×180 mm^3. Chemical composition (%) of steel is: S - 0,1; Cr - 1,1; Mo - 0,26; V - 0,17; Mn - 0,54; Si - 0,26; S - 0,019; P - 0,015. The specimens of first and third groups were made of basic (not operated) material and second and fourth groups - of material, which operated in steam pipes of power station about 150 thousand hours (540 Ms). Specimens of first and second groups were heat cycled in air, third and fourth - in the environment of gaseous hydrogen. "Heating - holding – cooling" cycles are shown in Fig. 1 for the specimens of all four groups (pressure of hydrogen environment for the 3rd and 4th groups of specimens is shown in the Table 1). Heating of specimens up to the temperature of 813 °C and its maintenance was carried out by an alternating electric current. Its value is shown in the Table 1.

Table 1. Characteristics of specimen heating modes

Group of specimen	Heating current, кA	Supplied energy, MJ	Pressure in autoclave, MPa
1	3,6	1,017	atmospheric
2	2,88	0,587	atmospheric
3	3,84	1,615	0,14
4	2,58	0,334	0,4

AE signals were recorded using waveguide. The specimen and one end of the waveguide were placed in the autoclave. The other end of waveguide was placed outside of autoclave through the special airtight knot. On this end, the primary piezoelectric AE transducer (AET) was located. Calculation of geometry of waveguide and matching of its response characteristic with the similar characteristics of AET was made using the method described in [8].

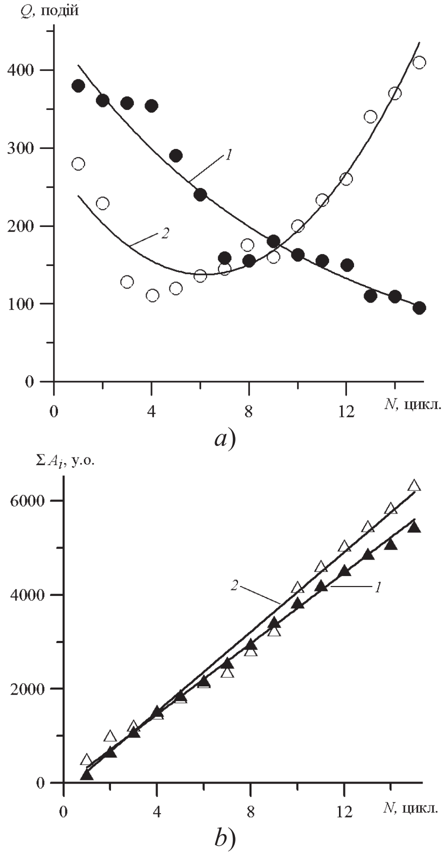


a) *b*)

Fig. 1. Time variations of temperature in cycles of heating for four groups of specimens (the numeration of curves corresponds to the numbers of groups of specimens)

2. RESULTS OF AE RESEARCHES

In the temperature-cycle testing of the first group of specimens AE was emitted, mainly, during the specimen cooling. A tendency of its activity decaying with growth of temperature-cycles number was observed. The results of tests are shown in Fig. 2.



a)

b)

Fig. 2. Dependence of the number of events Q (*a*) and the sum of amplitudes ΣA_i of AE signals (*b*) on the number of heat cycles N for the specimens of first and second groups

Fig. 2a shows that for 1st and 2nd groups of the specimens the number of events Q, which was recorded during 15 cycles in air (specimens of steel 12H1MFA of base material), decreases with the number of cycles increase, starting from the first cycles. This experimentally obtained dependence is well approximated by exponential function of the form $Q = a{\cdot}\exp(bN+c)$ with the correlation factor $r = 0,912$. The coefficients of approximation are resulted in Table 2a. The second group of specimens made of the operated steel has shown complicated character of AE events number variation depending on the number of heat cycles in air. For the first cycles a tendency to their decreasing was observed, then the AE activity increased with growth of the number of heat cycles (curve 2 in the Fig. 2a). Experimental points for this group of specimens are approximated by expression $Q = aN^2+bN+c$ (the parameters of approximation are given in Table 2*a*).

In relation to the sum of amplitudes, which contains an information on bulk damaging of material [9], we notice that for the specimens of the specified 2 groups it varies linearly (see Table 2b and Fig. 2b) with the number of heat cycles. The absolute values of this index for specimens made of the operated material are higher, starting from 10th heat cycle.

Table 2*a*. Parameters of approximation of experimental results

Group of specimens	Parameter of approximation			
	a	*b*	*c*	*r*
1	210,863	-0,102	0,758	0,912
2	3,808	-46,941	282,235	0,903
1	376,307	-43,39	--	0,997
2	423,907	-178,89	--	0,983
3	0,31	1,344	2,794	0,901
4	23,607	76,06	-60,25	0,99

Table 2*b*. Dependences of approximation of experimental results

Group of specimens	Type of dependence	Note
1	$Q = a\exp(bN+c)$	Fig. 4,a
2	$Q = aN^2+bN+c$	
1	$\Sigma A_i = aN+b$	Fig. 4,б
2	$\Sigma A_i = aN+b$	
3	$\Sigma A_i = aN^2+bN+c$	Fig.10,б
4	$\Sigma A_i = aN^2+bN+c$	

Similarly to the previous, specimens of third and fourth groups were tested in the environment of gaseous hydrogen. Time character of change in temperature of cycles heating - holding - cooling is shown in Fig. 1b. Having this data, firstly, we calculated the number of cycles to complete saturation of specimens of third and fourth groups by numerical methods for parameters of hydrogen environment indicated in Table 1. A mathematical model of temperature condition changing for each cycles was developed for this purpose. In this model the time interval of heating is described by the function $T = at^2+bt+c$, the interval of specimen holding for temperature 813 K was approximated by constant $T = \mathrm{B} = const$ (where B = 256,6 s for the specimens of third group and B = 323,3 s for the specimens of fourth group). For the time interval of cooling two intervals of approximation were defined: for the temperature range 813...373 K the dependence $T = at^2+bt+c$ was used and for the temperature range 373...293 K $T = at+c$.

Using the data collected in Tables 1 and 3 and calculation method developed in paper [10], we determined the number of cycles for attaining equilibrium saturation of specimens of third and fourth groups with hydrogen. They were, respectively, 7 and 6 heat cycles [10]. Basing on results of these computations, we conducted heat cycling of specimens of these groups in gaseous hydrogen. The results of these tests are shown in Fig. 3, in Tables 1 and 2.

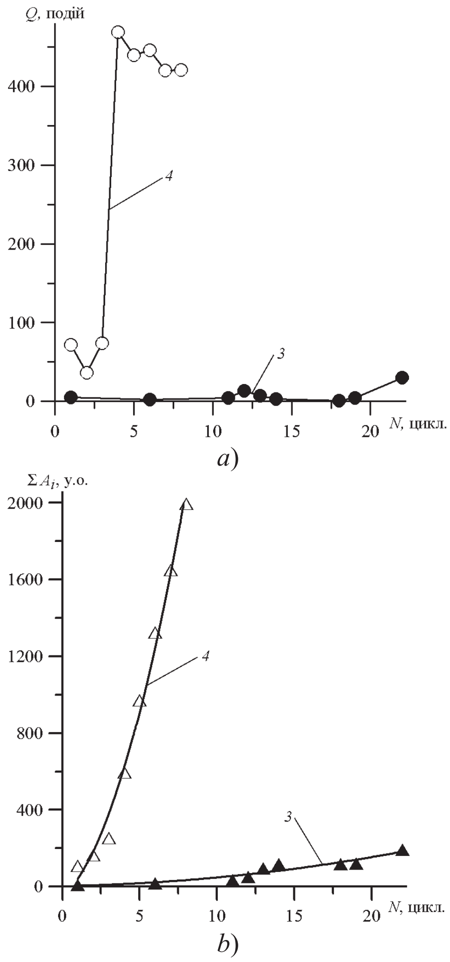


Fig. 3. Dependence of number of events Q (a) and sum of amplitudes $\sum A_i$ of the AE signals (*b*) on the number of cycles N of the heating and cooling of specimens of third and fourth groups

As it is shown in Fig. 3 AE for the specimens of third group sporadically observed only at the 2nd and 6th cycles with negligible indexes: 5 and 2 AE event, respectively, with approximately the same sum of amplitudes of 9 a. u. Starting from 11th cycle AE increased slightly: the activity increased to 7...13 events (for 13th and 12th cycles, respectively). The sum of amplitudes increased to 16...44 a. u. for these cycles. Later with growth of number of heat cycles, the radiating of AE was interrupted, which finished mainly, at the 20th cycle and monotonous growth of mentioned indexes of AE activity was observed. Thus, AE activity during heat cycling of initial material in the hydrogen environment is insignificant that confirms insignificant damaging of material.

The activity of AE in the tests of specimens of 4th group in the hydrogen environment is in orders higher. At the first 3 cycles it slightly decreases (by analogy with heat cycling of this material in air, see Fig. 2*a*; curve 2), and later abruptly increases showing a tendency to smooth decaying with the increase of cycles number. For 5th cycle the dependence between the variation of number of events and the number of cycles is approximated by straight line: $Q = aN + c$, where $a = -11{,}6$; $c = 508{,}8$ and the coefficient of correlation is $r = 0{,}823$. The sum of amplitudes depends on the number of heat cycles. The approximation parameters of this dependence are shown in Table 2. Having these distinctions in AE generation, we decided to test the specimens of 4th group on the base of 8 cycles, because the tendency of their AE activity in these experiments is determined precisely from the test start.

As it follows from the obtained results, the used material taking substantially less energy (see Table 1) generates during the heat cycling in hydrogen and air considerably more of the AE signals. Therefore, it means it is more damaged.

Thus, AE method gives an effectively assessment of heat cycling effect in various environments on the bulk damaging in steels of heat-power equipment depending on the level of their degradation.

5. CONCLUSIONS

Heat cycling of steels both in air and hydrogen environment leads to growth of bulk damaging, which kinetics well correlates with activity of AE signals.

It is stated using the parameters of AE signals that the used material is more disposed to the micro- and macro crack initiation under influencing of temperature factor and service environment.

The dynamics AE signals development has showed that heat cycling of steels in the environment of gaseous hydrogen can be used for the accelerated obtaining of the prescribed index of their degradation during exploitation of steels operating conditions.

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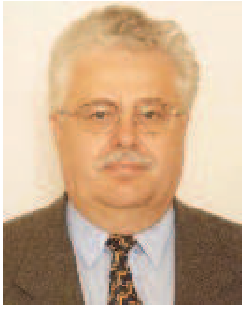

Valentine SKALSKY, Dr. Sci (Engineering Sciences), the head of department "Acoustic emission diagnostics of structural elements" of Karpenko Physico-mechanical Institute of National Academy of Sciences of Ukraine. He is the author over 230 papers in area of technical diagnostics and non-destructive testing of materials and structural elements.