Fatigue crack growth in 0.15C-2Cr-Mo-V steel after its high temperature hydrogen degradation

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ABSTRACT: Prediction of residual lifetime of the power plants and petroleum refinery objects is a very important problem. For its solution it is necessary to taking into account material degradation during service. There is an opinion, that hydrogen that discharges from the metal surface during manufacturing, is one of the most important factors of the degradation process. Hence, the important role in residual life prediction is attributed to the development of in-laboratory methods for simulation of in-service degradation of materials.

The aim of this work is to investigate the effect of the exposure specimens to gaseous hydrogen on the change of the microstructure and threshold parameters of fatigue crack growth in 15Kh2MFA steel.

Two methods of the in-laboratory ageing in hydrogen environment were described. In the first one the beam specimen was subjected to the long-term uniaxial tension in hydrogen environment at temperature 450 °C. In the second method the beam specimen was subjected to thermocycling in hydrogen. The thermocycling itself does not cause any essential internal stresses in the unrestrained specimen.

It was shown that the effective threshold parameter is sensitive to the changes of microstructure in metal after isothermal holding and thermocycling in hydrogen. These changes depend on stress level during isothermal holding and a number of thermocycles in hydrogen. It was concluded that just hydrogen is responsible for the acceleration of diffusion process, which is the necessary condition for the observed redistribution of carbides in such ageing conditions.

INTRODUCTION

A heat resistance steel 15Kh2MFA is widely used in nuclear reactors equipment [1]. At present, this steel is used for manufacturing of the reactor body for petroleum hydrocracking. The service temperature for petroleum hydrocracking process reaches 450 ^{O}C. The combined action of high temperature, hydrogen-containing environment, mechanical load causes to metal degradation and reduce its strength properties. In order to predict the residual lifetime of the equipment is needed to take into account the whole range of the service factors and create the rapid method of material degradation.

The main goal of this work is to investigate the effect of specimens exposure to gaseous hydrogen on the change of the microstructure and threshold fatigue crack growth parameters of 15Kh2MFA steel.

EXPERIMENTAL PROCEDURE

Chemical composition and tensile mechanical properties of low alloyed 15Kh2MFA reactor steel are presented in Tables 1 and 2, respectively.

TABLE 1: Chemical composition of the steel, %

Steel	C	Cr	Ni	Mo	V	Mn	Si	S	P
15Kh2MFA	0.15	2.8	0.4	0.70	0.27	0.50	0.30	0.015	0.012

TABLE 2: Tensile properties of the steel

Steel	σ_{YS}, MPa	σ_{UTS}, MPa	δ, %	ψ, %
15Kh2MFA	470	610	25	79

The specimens were cut out from the metal in virgin state and also after in-laboratory ageing, which was performed using two methods. According to the first one the beam specimen of length 320 mm was placed in a vacuum test chamber. The chamber design permits to subject a specimen to the long-term uniaxial tension in hydrogen environment. The gas pressure in the chamber was controlled by a built-in pressure gauge. The chamber with a specimen inside was placed in an electric furnace. The specimen temperature was controlled by a thermocouple, which was dot welded to the external wall of the chamber at a level of the specimen length middle. Its indications were previously graduated relatively to the thermocouple, which was caulked directly on the specimen (in its middle section). This allowed us to keep a temperature regime of ageing with an accuracy of ±5 ^{o}C. The specimen loading up to the required level was started at the hydrogen pressure of p_{H2} = 0.5 MPa and temperature was 450 ^{o}C. The elongation rate of 0.18 mm/hour was kept. The specimens were subjected to the same loading, which was kept with an accuracy of ±1%. The cross sections of the specimens were 10x25 mm. Two levels of tensile stress (120 MPa and 240 MPa) were used. The accepted load level provided the absence of material creep. Ageing period was 2200 hours.

In the second method the 180x10x25 mm beam specimen was placed in a hermetic chamber, filled with hydrogen of pressure 0.3 MPa. The sample was heated by electric current of 1800 A and a frequency of 50 Hz. This allowed us to heat up the specimen to the service temperature (450°C) at a rate of 2 °C/s. The temperature in the working part of the specimen was kept constant with an accuracy of ±5°C with the help of the automatic control system of the signals from the thermocouple and was caulked on the middle of the specimen. The specimen was being kept in such conditions during 0,5 hour. After that occured a sharp decrease of temperature down to 100°C at a rate of 3 °C/s, that was provided by cooling of the current leading blocks with running water (they were made empty, that allows to utilise them both for heating and cooling). Since in the proposed ageing method the specimen was not restrained in grabs, the change of its volume during the heating and cooling did not cause any significant mechanical stresses. Moreover the distance between the current leading blocks several times exceeded the specimen cross-section. It ensured the absence of temperature gradients in the middle part of the specimen, and the probability of appearance of thermal stresses during heating and cooling was very low [2]. Thus, the thermocycling itself does not cause any essential internal stresses in the unrestrained specimen.

The specimens in a virgin state and after ageing were subjected to fatigue cantilever bending tests in air. The load frequency and ratio were f= 15 Hz and R=0, respectively. The crack size was measured on both lateral sides with optical microscopes of 0.01 mm tolerance. The fatigue crack closure was evaluated using the compliance technique described in [3]. Strain-gauge transducer was fixed at the crack tip level and was periodically transferred during the crack growth. Material serviceability was evaluated by using the linear fracture mechanics approach. The values of the nominal ($\Delta K = K_{max} - K_{min}$) and the effective ($\Delta K_{eff} = \Delta K - \Delta K_{cl}$) stress intensity factor ranges were determined. Here ΔSYMBOLK_{cl} denotes the range of SIF from zero load to the loading level, when metal at the crack tip begins to deform cyclically.

EXPERIMENTAL RESULTS

The kinetic diagrams of fatigue crack growth are presented in Fig. 1 for the steel after holding the specimens under different loading (Fig. 1a) and after different number of thermocycles (Fig. 1b) in gaseous hydrogen. The specimens were tested immediately after treatment in hydrogen, therefore they contained a certain amount of hydrogen dissolved in metal which effects significantly enough the fatigue threshold parameters. The test results showed that the main effects of steel degradation as a result of thermocycling in hydrogen or holding under different loading on fatigue crack growth are observed for the threshold level of loading. A part of kinetic diagram in the rate range from 10^{-9} to 10^{-7} m/cycle is insensitive to changes in the metal state.

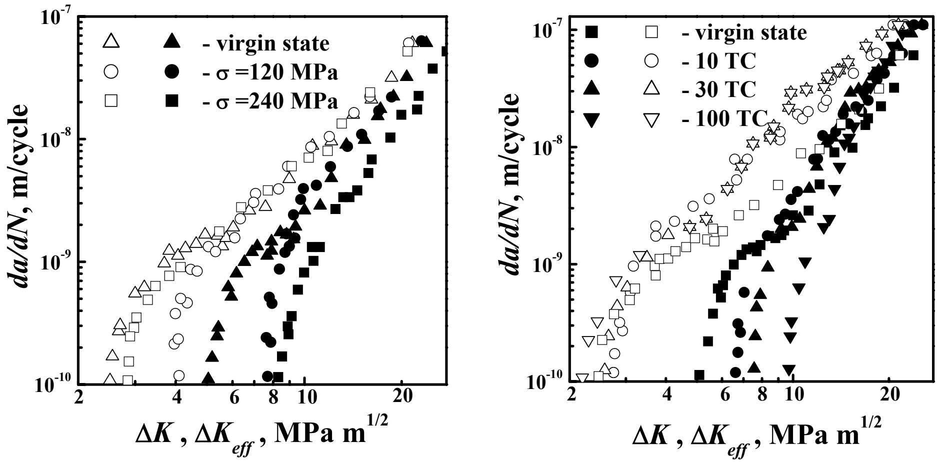

Figure 1: The effect of in-laboratory ageing of the 15Kh2MFA steel using the first (a) and second methods (b) on fatigue crack growth rates.

The regularities of the change of the threshold fatigue crack growth parameters in dependence on the ageing steel state as a result of isothermal holding of the specimen in hydrogen and on the thermocycles number in hydrogen are presented in Fig. 2. It is found that the threshold parameters ΔK_{th} and $\Delta K_{th\ cl}$ increase monotonically with the stresses increase during isothermal holding in hydrogen (Fig. 2a) and thermocycles amount (Fig. 2b). At first sight it testifies to improvement of metal serviceability after ageing. However the analysis of the effective fatigue threshold reveals an ambiguous change of the metal state with the stress and thermocycles number increase. In particular, the effective threshold of the metal increases from 2.5 MPa $m^{1/2}$ in the initial state up to 4.1 MPa $m^{1/2}$ after metal holding in hydrogen at stresses σ = 120 MPa and decreases to 2.8 MPa $m^{1/2}$ after its holding at stresses σ = 240 MPa. After thermocycling the $\Delta K_{th\ eff}$ level rises slightly after 10 cycles but decreases notably after 30 and 100 thermocycles in hydrogen.

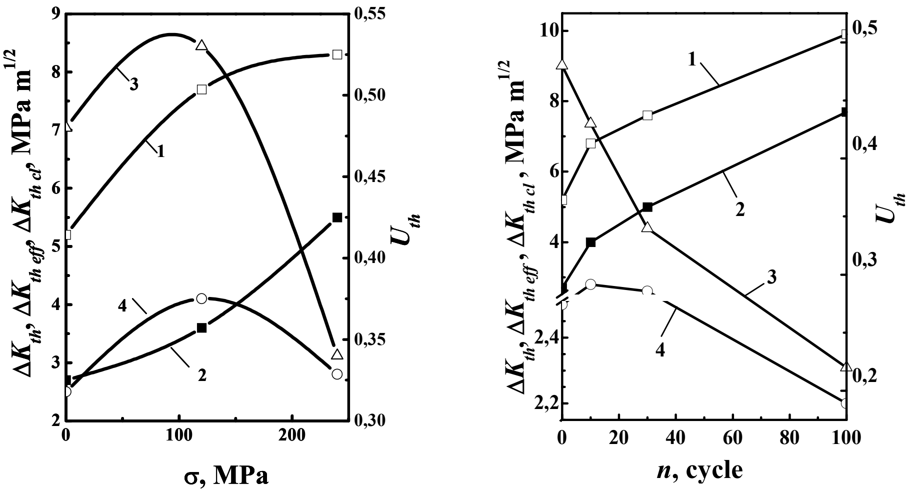

Figure 2: The effect of stress level σ during isothermal exposure (a) and thermocycles amount n (b) in hydrogen environment on the parameters of threshold crack growth resistance of 15Kh2MFA steel: 1 - ΔK_{th}, 2 - $\Delta K_{th\ cl}$, 3 - U_{th} 4 - $\Delta K_{th\ eff}$.

The microstructure of 15Kh2MFA steel in the virgin state and after 3 months holding of specimens in hydrogen under stresses is presented in Fig. 3. It is evident that holding in hydrogen causes a coagulation of carbides with the corresponding average size of carbides increase and their number decrease. Furthermore, the typical for metal in a virgin state the strip orientation of carbide along boundary of the bainitic plates disappears after in-laboratory ageing. The greater number of grains lose their typical orientation of carbide strips in the more rigid ageing conditions. No similar changes were recorded in air in the same conditions of degradation.

DISCUSSION

Hydrogen effects positively the nominal fatigue threshold value with the increase of the stress level during isothermal holding of specimens in hydrogen, on the one hand, and with increase of the number of thermocycles in hydrogen, on the other. However one can draw a fallacious conclusion if only the nominal thresholds analysis is taken into consideration. Since cyclic deformation of the material at the crack tip occurs only during the effective part of the cycle $\Delta K_{th\ eff}$ it is obvious that only the effective fatigue threshold determines the real ability of the damaged material to resist fatigue crack growth. Hence hydrogen absorbed by metal increases the effective fatigue threshold during loading of the specimen under holding or thermocycling in hydrogen up to 10 cycles. However loading above 120 MPa or thermocycling more than 10 cycles decreases $\Delta K_{th\ eff}$ and after ageing by the second method it decreases below the level of the virgin state of the metal. Usage of the described methods of in-laboratory ageing as well as ageing due to service [4] causes the decrease of the effective threshold of fatigue crack growth, and it gives the deterioration of workability of a metal due to degradation of its microstructure and accumulation of damages.

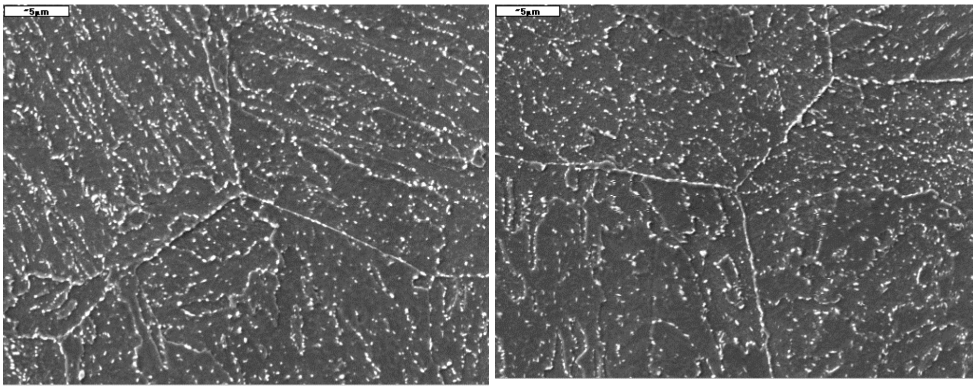

Figure 3: The microstructure of 15Kh2MFA steel in a virgin state (a), and after isothermal ($T = 450\ ^oC$) exposure at $\sigma = 240$ MPa in hydrogen during 3 months (b). The length of the white marks is equal to 5 μm.

The quantitative analysis showed that as a result of hydrogen degradation the coagulation of carbides with a corresponding their number decrease and average diameter increase were occured (Fig. 4).

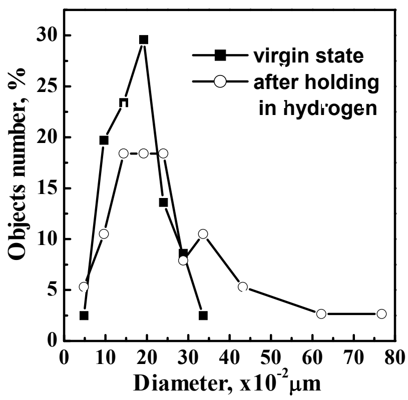

Figure 4: Average distribution of the diameter particles vs. their amount after isothermal holding in hydrogen.

Moreover the carbide orientation along boundary of the bainite plates typical for metal in virgin state disappears after in-laboratory ageing. The greater number of grains lose the typical carbide orientation with the increase of stresses during isothermal holding in hydrogen.

The similar microstructure change was not observed in air under the identical temperature-force holding conditions. Thus, one can conclude that just hydrogen is responsible for the acceleration of diffusion process, which is necessary precondition for the observed redistribution of carbides.

CONCLUSION

The experimental investigations of in-laboratory ageing effect on the parameters of fatigue crack growth in 15Kh2MFA showed that $\Delta K_{th\ eff}$ parameter is sensitive to the changes of metal microstructure. These changes depend on the stress level during the isothermal holding and the number of thermocycles in hydrogen.

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