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Cyclic hardening and dynamic strain aging during low-cycle fatigue of Cr-Mo tempered martensitic steel at elevated temperatures

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A B S T R A C T

Dynamic strain aging (DSA) has a noticeable impact on low cycle fatigue behavior of Cr-Mo tempered martensitic steel at elevated temperatures. At 350 °C, the stress response presented evidently secondary hardening phenomenon, and the fatigue life is significantly higher than room temperature which is contrary to the usual understanding. The analysis shows that the reason for the secondary hardening is mainly because of DSA which affects the evolution of dislocations. The influence process of the DSA effect on the dislocation evolution in low-cycle fatigue was first observed, which is from lath substructure to a smaller cell substructure at high temperature. Fracture observation indicates that the new dislocation structure promotes the crack propagation resistance of the material. The effect of DSA on the low cycle fatigue properties of high temperature provides a new idea for the study of fatigue resistance of materials.

1. Introduction

High strength Cr-Mo tempered martensitic steel such as Cr-Mo, 2Cr-Mo and 9Cr-Mo steel have been developed for application under high-temperature and high-loading conditions in ultra-supercritical thermal power plants and petrochemical industries [1,2]. The components of high temperature environment are usually subjected to cyclic thermal stresses due to the operating mode of temperature gradients, especially at the process of steam injection, when the temperature changes rapidly. In previous studies, Cr-Mo tempered martensitic (HS80H) steel exhibited a softening – hardening – softening fracture process under low-cycle fatigue (LCF) at room temperature, and the stress response feature remains constant when the strain amplitude and strain ratio are changed [3]. However, this feature varies with the elevated temperature. The stress response feature shows two times hardening when the temperature is above 250 °C. In the study of high-temperature low cycle fatigue of TP347H austenitic stainless and 9Cr-1Mo steel, evident secondary hardening phenomena usually occur at the temperature of more than 500 °C [4,5]. For HS80H steel with low Cr-Mo content, the secondary hardening occurs at 350 °C, which needs further investigation.

The LCF behavior of Cr-Mo tempered martensite steel at room or high temperature has been investigated extensively. The reason for the difference in low cycle fatigue performance at room or high temperature is usually attributed to the influence of dynamic strain aging (DSA) [6,7]. DSA is mainly caused by the pinning effect of solute atoms and slip dislocations, and leads to serrations stress-strain curve on the macroscale [8–13]. Dislocation movement and accumulation state change due to the influence of DSA, thus affecting the macro fatigue properties [14]. Extensive studies indicated that the DSA phenomenon exists generally in the tempered martensitic steel within a specific temperature range, and HS80H steel also has similar properties [15–17]. Gentet [18] showed that C, Cr, Mo and other solid solution elements had remarkable DSA effect when the temperature is above 200 °C. The dislocation substructure changes when the DSA effect is significant. For ferrite martensitic steel, the dislocation structures are formed by a large number of dislocation cells by DSA [19,20]. However, the process and mechanism of formation are not clear. The formation process of dislocation cells is derived from inference and there is no clear evidence of dislocation evolution.

The microstructure change caused by DSA is accumulated due to the repeated action of tension and compression strain, thereby affecting the macroscopic mechanical behavior and changes the fatigue life. Some studies have shown that DSA has a significant effect on the low cycle fatigue properties of materials, such as DSA pre-treatment reduces the fatigue life [4,5]. It is considered that DSA-induced high peak tensile stress causes rapid crack propagation, resulting in the reduction of fatigue life. And related research pointed out that DSA reduced fatigue life in LCF, which comes from the DSA-induced inhomogeneity of

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deformation, and rapid crack propagation [21]. In a similar study, such as on Cr-Mo ferritic martensitic steel, the fatigue life increased significantly from room temperature to 500 °C under the influence of DSA [15,22]. It is pointed that DSA caused crack propagation to be retarded by crack branching and suppression of plastic zone. Then, this is entirely contrary to the conclusion that DSA reduces fatigue life. These two viewpoints analyze the influence of DSA on fatigue life only from mechanical behavior. The relationship between microstructure and macroscopical properties under DSA is not clear. The fatigue life for HS80H steel increased obviously at 350 °C. The reasons for the improvement of fatigue resistance are explained from three aspects of macroscopic mechanical behavior, fracture morphology and dislocation evolution. In this study, the dislocation substructure of different low cycle fatigue stages was first observed, providing strong evidence for the inference of dislocation evolution in the Cr-Mo ferrite martensitic steel due to the effect of DSA. The relationship between microscopic substructure and macroscopic mechanical behavior is explained.

2. Experiment

The investigated materials were HS80H tempered martensitic steel casing for thermal recovery well. It was treated by tempering treatment which is quenched at 890 °C for 30 min and then tempered at 650 °C for 2 h. The chemical components are listed in Table 1.

The specimens used for LCF tests were cylindrical with a diameter of 6 mm and a gauge of 12 mm, as shown in Fig. 1. Strain-controlled fatigue tests that employ tension–compression fatigue loading were conducted with a closed-loop servo hydraulic testing machine (Instron 8862). A symmetrical triangular strain time waveform was employed at a constant strain rate of 4×10^{-3} s^{-1}. Strain amplitude and R values were constant (strain amplitude = 0.7%, R = − 1). The following temperatures selected by LCF test were used: 20 °C, 250 °C, 350 °C, and 450 °C. The temperature was controlled by three temperature sensors near the sample in the heating furnace, and the temperature variation did not exceed ± 1 °C. The samples were defined as failures when the cyclic loading dropped to 75% from the maximum.

The fracture surface investigations were performed using scanning electron microscope (SEM) with an acceleration voltage of 30 kV. Transmission electron microscopy (TEM) was used to assess the evolution of dislocation structures and deformation behavior under LCF conditions at 20 °C and 350 °C. Under the same LCF test conditions, the test was stopped at initial stage, middle stage and end stage respectively. The three samples were sampled by TEM to observe the dislocation substructure evolution process. The samples used for TEM were obtained through wire-electrode cutting at a distance of 3 mm away from the fracture surface. Then the samples were observed under TEM after twin-jet electropolishing.

3. Results

3.1. DSA during stress-strain curve

The stress-strain curves in Fig. 2 show that serrated flow appeared in the temperature range of 250 °C and 450 °C, indicating that HS80H steel had DSA effect in this temperature range. The observed serrations were classified as Type A serrations [23]. Type A serration with lower frequency occurred in the stress-strain curve. When the temperature was 350 °C, the type A serrations can be clearly observed. As the temperature was increased, the amplitude of type A serration increased significantly, especially at 450 °C. Type B serrations with a high frequency appeared between two type A serrations. The deformed flow stress serrations were possibly due to the interaction between the diffused solid solution atoms (C, N, and Cr) and mobile dislocations [24,25], which lead to dislocations multiplication and a higher dislocations density.

Table 1
Chemical compositions (wt%).

C	Si	Mn	P	S	Cr	Mo	Ni	V	Ti	Cu
0.17	0.24	0.98	0.011	0.0034	0.99	0.33	0.059	0.029	0.013	0.021

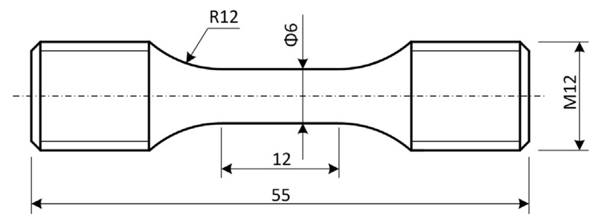

Fig. 1. Fatigue specimen geometry.

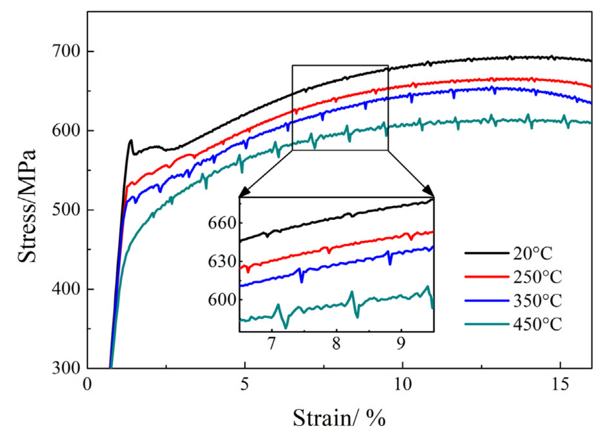

Fig. 2. Stress–strain curve of tensile tests at different temperature.

3.2. Cyclic stress response

The cyclic stress response of the samples at different temperatures is shown in Fig. 3. Cyclic numbers are represented by logarithmic coordinates in Fig. 3a, and cycle processes are represented as a percentage of the failure number in Fig. 3b. The two typical stage for all conditions include an initial cyclic hardening that appeared at several cycles (failure number < 10%), followed by a continuous cyclic softening until 20% failure number. After 20% failure number, the cyclic stress response showed two completely opposite trends at different temperatures. At test temperatures of 20 °C and 250 °C, the samples showed a softening trend. The softening rate of the sample at 250 °C is less than that at 20 °C in the stable softening stage (200 cycles to 450 cycles). At the test temperature of 350 °C, the sample showed evident hardening after the initial softening. The sample then rapidly softened and became unstable when hardening reached maximum. At 450 °C, the sample showed significant cyclic hardening. The sample sharply hardened in the final stage of fatigue (320 cycles to 340 cycles), which resulted in abrupt breaking.

According to the trend of cyclic stress response, the temperature can be divided into three sections, namely, the no-secondary hardening sections (20–250 °C), transitional sections (250–350 °C) and secondary hardening sections (350–450 °C). The secondary hardening occurs as the temperature changes, indicating that some microstructures have been changed, such as subgrain structure and dislocation. This change could result in a primary hardening, then softening, and finally a secondary hardening.

The secondary hardening also has some interesting features, that is, the fatigue life is evidently higher than the others when the temperature is at 350 °C, thereby indicating that secondary hardening improves fatigue life under certain conditions.