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Evolution of creep damage in a modified ferritic heat resistant steel with excellent short-term creep performance and its oxide layer characteristic

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ABSTRACT

By performing short-term creep tests at 923 K, the evolution of creep damage in a modified heat resistant steel and its oxide layer characteristic were investigated. Compared with conventional ferritic steels, the modified steel exhibited excellent short-term creep resistance. And the fracture mode remained transgranular under all test conditions. By analyzing creep data, high creep damage tolerance factors were obtained, indicating that precipitation coarsening should be the pronounced factor causing creep strain accumulation and the onset of tertiary creep stage. Combined with microstructural examination, it is found that the coarse precipitates at the prior austenite boundaries, mainly $M_{23}C_6$, contribute to the void formation and eventual transgranular failure. The oxide layers formed on the specimen surface consisted of Fe-rich $[Fe,Cr]_2O_3$ outer layer and Cr-rich $[Fe,Cr]_3O_4$ inner layer.

1. Introduction

The nuclear energy can be produced without the side environmental effects accompanying the use of coal and petroleum products. And a new generation (Generation IV) of reactor is being developed to produce abundant, reliable, inexpensive energy [1]. Among the structural materials applied to nuclear reactor, ferritic 9–12%Cr steels have usually been considered preferentially, because of their high creep strengths, good ductilities and low swelling properties. When developing advanced ferritic 9–12%Cr steels, it is required to elevate the service temperature (above 873–923 K) and to achieve sufficient creep strength with creep duration up to 100,000 h [2]. Abe at al. have been exploring new alloy design concepts for heat resistant steels, including reducing carbon content to very low amounts, and adding a certain amount of boron without the formation of large boron nitrides [3]. For the fourth generation of steels being developed, like NF 12 and SAVE 12, it is prevailing to increase the content of Cr to 12%, and to add 3% Co aimed at balancing the effect of ferrite stabilizers [4]. High Cr can ensure the oxidation resistance at elevated temperature, but it may also promote the precipitation of Laves phase, deteriorating long-term performance. Considering these aspects, appropriate Cr content is needed to achieve a balance between oxidation-resistance property and creep-resistance property [5]. In this work, a 10%Cr ferritic steel was designed based on the design criteria proposed by Abe at al. [3] and the composition of NF12 and SAVE 12.

It has been demonstrated that extrapolation of short-term creep data may lead to an overestimation of the long-term creep strength, which is properly associated with the metallurgical evolution during creep or damage development [6]. Attention has been paid to the microstructure evolution during creep in ferritic steels [6–8]. Yet, the relationship between the creep damage and creep behavior of heat resistant steel has not been studied in detail. In this work, we study the creep damage evolution in a modified heat resistant steel, and the characteristic of oxide layers formed on the surface of crept specimen has also been investigated.

2. Experimental

The steel used in this study is developed on the design principles for the fourth generation Cr–Mo steels [4], but with a major variation with respect to elements like C, Cr, Co, etc. Table 1 shows the chemical compositions of the experimental steel. The material was supplied by Tianjin piping corporation in the form of hot-rolled pipe 9 mm in thickness. Heat treatments, with normalizing at 1373 K for 20 min to ensure complete dissolution of $M_{23}C_6$ type carbides, subsequent air-cooling and tempering for 40 min at

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Table 1
Chemical compositions of the modified high Cr ferritic steel (wt%).

C	Mn	Cr	Mo	W	V	Nb	N	Co	Ti	B	Ni	Cu
0.06	0.49	9.82	0.35	1.69	0.21	0.07	0.0141	1.46	0.01	0.0044	0.03	0.04

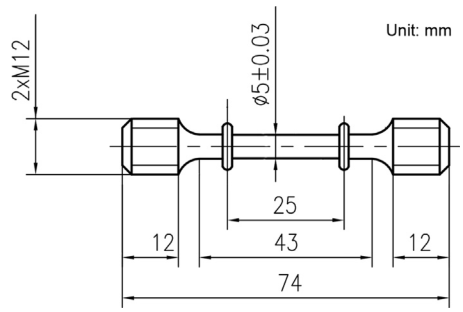

Fig. 1. Dimension of the specimen for creep fracture test.

1023 K, were performed to produce the initial microstructure of creep specimens. Creep tests were carried out at 923 K under stress levels of 190, 210, 225 and 250 MPa. The size of creep specimens is displayed in Fig. 1. Creep specimens are cylindrical form of 25 mm in gauge length and 5 mm in diameter.

The microstructure of the steel after tempering was examined by optical microscopy (OM) to measure the volume fraction of delta-ferrite, and by transmission electron microscope (TEM) to find out the factors related to the improvement of short-term creep strength. To prepare the TEM specimens, mechanical polishing was followed by twin-jet-polishing with a solution of 5 vol% perchloric acid in ethanol. Since the fine MX in heat resistant steel would be obscured due to diffraction contrast in the metal foil, extraction replicas were made. After deep etching and carbon coating, the specimen surface was lightly scored into squares of 2.5 mm side length, and then bulk replicas were liberated in the Vilella's reagent, followed by floating the replicas off in 10% methanol/water. Copper TEM grids were used to dry up the replicas. The samples for creep damage investigation were taken from a region about 3 mm away from the fracture zone, and prepared by mechanical polishing, followed by etching with a mixed solution of hydrochloric acid and ferric chloride. Scanning electron microscopy (SEM) was employed to analyze creep fracture morphologies of the specimens, and to identify the sites where creep damage occurs. The chemical compositions of oxide scales were examined using EDS system.

3. Results and discussion

3.1. Short-term creep properties

Since half yield (0.5 yield stress) is considered to be a macroscopic elastic limit where successive plastic deformation of polycrystalline material initiates, dependence of minimum creep rate and creep rupture life on stress change at half yield [9]. Half yield can be deemed as a critical stress at which stress condition shifts from plastic regime in the short-term to elastic regime in the long-term [10]. The 0.2% offset yield stress of modified ferritic heat resistant steel at 923 K was determined as 360 MPa, higher than that of conventional heat resistant steels, such as T91 (191 MPa), T92 (198 MPa) [11,12]. Obviously, stress levels of 190, 210, 225, 250 MPa in this study are in the high stress regime over half yield. Creep curves and creep rate versus time plots for modified steel crept at different stresses are presented in Fig. 2. The modified

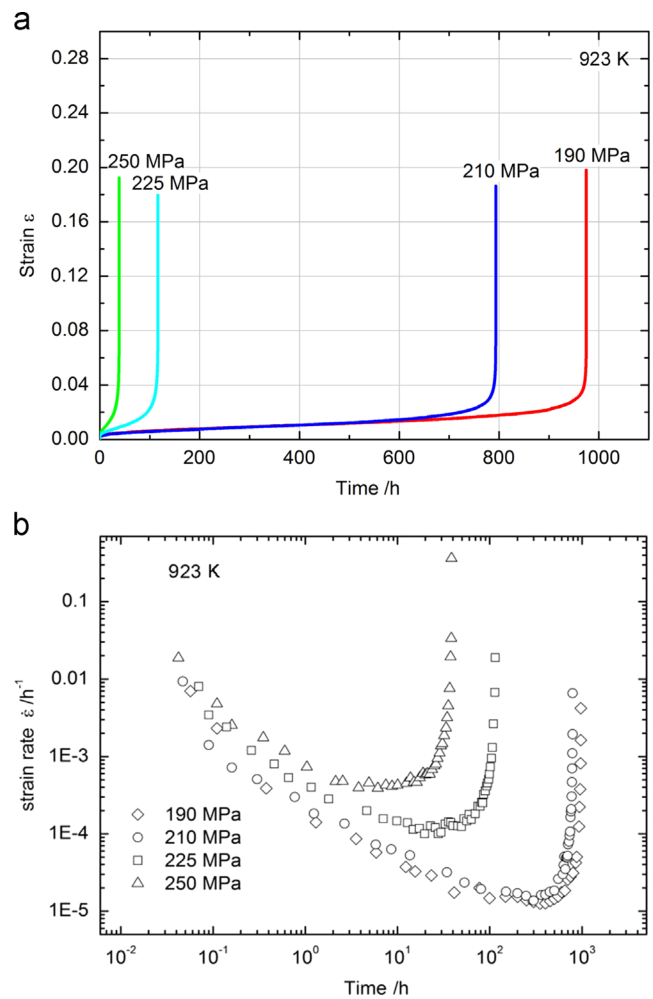

Fig. 2. (a) Creep curve and (b) creep rate versus creep time at 923 K for the modified steel under different stress conditions.

steel seems to exhibit a normal three stage creep behavior, with a little primary creep strain, a well-defined secondary creep stage, followed by tertiary creep stage. However, it should be noted that the secondary creep stage is reduced to a turning point in Fig. 2b. It is clear that the minimum creep rate decreases monotonically with a decrease in the applied stress. Although the shapes of creep rate versus time curve at 210 MPa and 190 MPa is similar to each other, the onset of acceleration creep stage at 190 MPa is delayed, leading to longer creep rupture life.

Fig. 3 shows the relationship between the applied stress and the time to rupture for the modified steel, T91 steel and P92 steel at various stresses. The creep data of T91 steel and P92 steel is from the literature [13]. The modified steel in this study exhibits excellent short-term creep performance, which is superior to the P92 steel at the same level of stress. However, sigmoidal inflection of the relationship between time to rupture and applied stress has been frequently observed in ferritic heat resistant steel [14]. Thus, the prediction of long term creep strength from short term creep data may be inaccurate and unreliable, causing the overestimation of long term creep strength. The long term creep tests of this novel