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The correlation among microstructural parameter and dynamic strain aging (DSA) in influencing the mechanical properties of a reduced activated ferritic-martensitic (RAFM) steel

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ABSTRACT

The additional effect of the dynamic strain aging (DSA) on the microstructural parameters in influencing the yield strength was analyzed through comparing the difference between the experimental yield strength and the calculated value at room temperature (RT) in 9Cr-1.7W-0.1C reduced activated ferritic-martensitic (RAFM) steel samples treated under two different heat treatments. Different heat treatments leaded to the variation of the microstructural parameters of this steel. Then the contributions of these microstructural parameters and DSA on the tensile properties at elevated temperatures were studied with the modified Crussard–Jaoul (C–J) analysis based on the Swift equation.

1. Introduction

Reduced activation ferritic/martensitic (RAFM) steel has been considered to be one of the reference structural materials for future fusion power reactor, owing to their lower thermal expansion coefficients, higher thermal conductivity coefficients at elevated temperatures and good irradiation resistance [1–8]. These steel possesses typical tempered ferritic/martensitic structure which composed of lath martensite and precipitates ($M_{23}C_6$; M = Cr, W and Fe); MX (M = V, Ta; X = C, N)), it is the main reason for the satisfactory mechanical performance of this steel [2,9–11]. It is explicitly mentioned that the operation temperature range of RAFM steels is about 350–550 °C. The upper rated temperature is limited by the mechanical strength reduction in high temperature region, such as a series of RAFM steels, i.e. F82H, Eurofer97, JLF-1, et. [1,12–14]. In order to enhance the mechanical properties of the RAFM steel at elevated temperatures, a series of strengthening methods have been used, and it also can transfer the upper limit temperature for operation to higher temperature (such as 600 °C) for some RAFM steels, such as in the CLAM steel [15] and INRAFM steel [16].

As previously explored in the aforementioned studies, all the tensile yield stress-temperature and ultimate plastic strain - temperature curves of these RAFM steels exhibit a mild peak (or plateau) and trough corresponding to test temperatures from 200 °C to 450 °C, respectively, which were reported in relation to the dynamic strain aging (DSA) [16]. The edge, screw and mixed dislocations in the matrix are considered to interact dynamically with mobile interstitial or substitutional atoms in this temperature region, causing the “blue brittleness” phenomenon in steels [17]. In our previous study, it has been confirmed that the tensile properties of the 9Cr-1.7W-0.1C RAFM steel samples employed in this work with different heat treatments are assumedly identical as it was described in the above [18].

Considering that different kinds of RAFM steels have different chemical composition, so the range of $(\gamma + \delta)/\gamma$ boundary in phase diagrams of these steels will be different from each other. In view of this, it is easy to understand why different RAFM steels were normalized at different temperatures, such as JLF-1 (1050 °C [19]), CLAM (980 °C [19,20]), F82H (1040 °C [21]), EUROFER97 (980 °C [22]) and INRAFM (980 °C [23]). Also, for balancing the grain size of austenite and homogenization of the solute atoms in the austenitizing process implementing with choosing the suitable normalizing temperature in the single austenite phase, it is the other reason why the normalizing temperature and holding time are different among these RAFM steels as mentioned above. Similarly, tempering treatment is a critical way to refine the carbides size and the dislocation density, and to control the lath martensite thickness in the ferritic/martensitic steels [24,25]. The high-temperature mechanical properties correlate with all the aforementioned microstructural parameters tightly. Many researches have been conducted on studying the effects of these microstructural parameters on the strengthening mechanisms of the lath martensitic steels

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and the oxide dispersion strengthened steels [26–28] at RT or elevated temperatures. In relation to the microstructure and the deformation mechanism changing at elevated temperature complexity, so it is easy to understand why the yield strength evolution at high temperatures is difficult to be described with these microstructural parameters quantificationally. Many models for describing of the evolution of yield strength at elevated temperature, just make the approximation that the contribution of forest dislocations is constant with temperature increasing until a particular temperature reached and then equal to 0. The solid solution contribution follows the same trend but with a higher transition temperature [28], which was discussed in many studies [28–30]. Consequently, there are oversimplifications for revealing the contributions of microstructural parameters on the mechanical properties at elevated temperatures.

The purpose of the present work is to investigate the correlation among microstructural parameter and dynamic strain aging (DSA) in influencing the mechanical properties of a reduced activated ferritic-martensitic (RAFM) steel and then explain the disparity of the mechanical properties of the RAFM steel with different microstructural parameters at elevated temperatures. Here, the different microstructural parameters were achieved through two different heat treatments.

2. Material and methods

An 80 kg ingot of 9Cr-1.7W-0.1C RAFM steel was melted by a vacuum induction furnace. Then it was made into cylinder bars by hot-roll method, with a size of 80 mm in diameter. The chemical composition of the ingot is listed in Table 1. The cylinder bar was normalized at 1000 °C for 30 min followed by air cooling and then tempered at 750 °C for 1.5 h and then by air cooling (N&T 1). The results of mechanical properties of this steel normalized at 1050 °C for 30 min followed by air cooling and then tempered at 750 °C for 1.5 h (N&T 2) were mainly referenced from our previous research [18]. Tensile specimens with a gauge diameter of 5 mm and gauge length of 30 mm were machined with the stress axis aligning to the rolling direction of the cylinder bar as mentioned above. Tensile tests were conducted at an initial strain rate of 1×10^{-3} s^{-1} with the test temperatures ranging from RT to 600 °C.

The specimens for optical microscope characterization were ground and polished finally, then etched by a mixed solution of 5% nitric acid, 2% hydrofluoric acid and 93% water. Transmission Electron Microscope (TEM) samples were ground by 1000# SiC sand paper, followed by twin-jet polishing in a mixed solution (90% ethanol and 10% perchloric acid) at 20 V and −35 °C. TEM examinations were performed by FEI Tecnai G2 F30, operated at 300 kV. The dislocation density of samples before and after tempering treatment were detected by the X-ray diffraction profile analysis using the modified Williamson–Hall equation and counted by the quantitative TEM-micrograph analysis method following the usual procedure [31,32], respectively. The modified Williamson–Hall equation is no longer adequate for the tempered steel, for it is not precise when at low level of the dislocation density, just as the tempering state.

XRD analysis was carried out using a Brucker-D8 advance diffractometer with Cu K_α radiation, radiation at 40 kV and 40 mA. The diffraction lines were recorded from 2θ = 30–120° with a step of 0.02° (2θ). The reflections of {110}, {200}, {211} of the BCC structure were measured. Both of the base line and the $K_{\alpha2}$ were removed from the XRD profiles. The diffraction intensity was plotted versus K using $K = \frac{2sin\theta}{\lambda}$, λ = 0.15405 nm for $K_{\alpha1}$. The diffraction peaks were fitted with a Voigt function. Therefore, the position, full-width at half-maximum (FWHM) of the Lorentzian function (W_E^L) and FWHM of the Gaussian function (W_E^G) can be calculated out by Voigt function [33]. Here, the instrumental broadening was removed by the powders of pure iron with the mesh size of 500, which were vacuum-annealed at 800 °C for 10 h.

Table 1
Chemical composition of the 9Cr-1.7W-0.1C RAFM steel in wt%.

Element	C	Cr	W	Mn	Si	V	Ta	P	Fe
Amount	0.092	8.87	1.71	0.28	0.01	0.19	0.002	< 0.005	Bal.

3. Experimental results

3.1. The effect of normalizing temperature on the prior austenite grain size and block size

In the process of austenitizing, austenite grain growth kinetics will be affected evidently by austenitizing temperature, such as the higher temperature it is, the higher growth rate of austenite grain it will be. Consequently, it is why the mean prior austenite grain size (D_g^{NT2} = 70 μm) of the N&T 2 9Cr-1.7W-0.1C RAFM steel is bigger than that (D_g^{NT1} = 43 μm) of the N&T 1 9Cr-1.7W-0.1C RAFM steel, as illustrated in Fig. 1. Grain-size distribution also changes from concentrating in the lower value region (20–60 μm) to dispersing in the higher value region (40–120 μm), corresponding to the Fig. 1(b) and (d) respectively.

In the martensite transformation process, an austenite grain will be divided into some packets, which are further subdivided into some blocks, and these blocks are consisted of some subparallel lath martensite with small boundary misorientation. A number of low carbon steels, Mar4, Mar5, Fe–0.2C and Fe–0.2C–2Mn, all of which display out that the packet and block size are composition-independent [26,34]. Thus, in the present work, the block size is regarded as obeying a positive linear correlation with of the prior austenite grain size of the 9Cr-1.7W-0.1C RAFM steel, since this steel is one kind of low carbon steels. This linear correlation can be described by the below equation [26]:

$$d_{block} = 0.067D_g \quad (1)$$

The d_{block} and D_g represent the diameter of the block and prior austenite grain, respectively. The results of block size corresponding to the N&T 1 and N&T 2 are listed in Table 2, as one kind of microstructural parameters, which will be used in analysing the contribution of these parameters on the yield strength in the Part 4.1. The block size raises with normalizing temperature increasing.

3.2. The effect of normalizing temperature on the evolution of lath martensite and precipitated carbide particles

The typical TEM microstructures of tempered martensite of the 9Cr-1.7W-0.1C RAFM steel are illustrated in Fig. 2(a) and (b). There is not any relation holds between the thickness of the lath martensite and the normalizing temperature. The precipitated carbide particles mainly distribute at the lath martensite boundaries, as marked in Fig. 2, and some decorate in the lath martensite matrix. These carbides have been verified to be mainly the $M_{23}C_6$, and some is MX (Carbonitride) in our previous study [18]. In order to narrow down the statistical error of the size and the total amount volume fraction of carbides, the carbon extraction-replica technique was used to characterize it, as shown in Fig. 3. And the results of the mean particle size and the mean total volume fraction of it (to provide a reliable statistical analysis, both of the two kinds of data were counted from 5 micrographics for each other) were listed in Table 2. Although both of the N&T 1 and N&T 2 steels experienced the same tempering treatment, but the different normalizing temperature will affect the final precipitation behavior of the carbides, in the follow tempering process. Through comparative analyses, the carbides size in the N&T 1 (see Fig. 3(a)) distribute more uniformly than that in the N&T 2 (see Fig. 3(b)). It is probably because of that the lower normalizing temperature it is, the finer sub-grain it will be, as stated in Part 3.1, and then more compatible sites for carbides to nucleate, since carbides preferentially nucleate at boundaries. As to why the mean volume fraction of carbides in N&T 1 is some bigger