Nuclear Materials and Energy 9 (2016) 331–337

Contents lists available at ScienceDirect

Nuclear Materials and Energy

journal homepage: www.elsevier.com/locate/nme

Precipitation of carbides in F82H steels and its impact on mechanical strength

S. Kano[a,*], H.L. Yang[a], R. Suzue[b], Y. Matsukawa[a], Y. Satoh[a], H. Sakasegawa[c], H. Tanigawa[c], H. Abe[d]

[a] Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba, Sendai, Miyagi 980-8577, Japan
[b] Graduate School of Engineering, Tohoku University, 6-6-04, Aramaki, Aoba, Sendai, Miyagi 980-8579, Japan
[c] Fusion Research and Development Directorate, Japan Atomic Energy Agency, 2-166 Omotedate, Obuchi, Rokkasho, Aomori 039-3212, Japan
[d] Nuclear Professional School, School of Engineering, The University of Tokyo, 2-22, Shirakata-Shirane, Tokai-mura, Ibaraki 319-1188, Japan

ARTICLE INFO

Article history:
Available online 18 November 2016

Keywords:
F82H steel
Carbide
TEM
Extracted residue test (ERT)
Precipitation strengthening

ABSTRACT

The precipitation of carbides in F82H steel and its model steel (Fe-0.2TaC) were investigated by transmission electron microscopy (TEM) and extracted residue tests (ERT). The effects of tempering on the precipitation in F82H steels were elucidated on the basis of the characterization of carbides, as well as the quantitative estimation of precipitation strengthening at room temperature. Firstly, the number density of precipitates was measured by extracted residue test in Fe-0.2TaC and tempered F82H steel, and compared with the TEM observation. It was found that the ratio of volume fraction between the TEM and the ERT was respectively 1.84 and 0.54 for Fe-0.2TaC and tempered F82H steel, revealing that the collection probability of ERT strongly depends on the precipitate features, size and number density. Effects of annealing on the precipitation in F82H steels were investigated by ERT. The amount of carbide showed a non-linear relationship to tempering parameter, $TP = T(20 + \log t)$. It steeply increased in the TP range from 15.3 to 16.2. The precipitation strengthening in F82H steel was estimated to be about 5–10% relative to its proof strength, suggesting that the carbides in F82H steel have a minor role on the tensile strength at room temperature, though these precipitates are greatly beneficial for improving the creep and radiation resistance at elevated temperatures.

1. Introduction

Among the various reduced activation ferritic/martensitic steels (RAFMs), F82H (Fe-8Cr-2W-V-Ta) steels have been considered as one of the promising candidate materials for the Japanese test blanket module (TBM) system in ITER, because of their excellent heat resistance and good irradiation resistance [1,2]. The satisfactory mechanical performance of this material in terms of strength, creep and toughness, is due to their unique microstructure. The microstructural of F82H steel is described as tempered ferritic/martensitic structure composed of lath martensite and precipitates such as $M_{23}C_6$ (M = Cr, W and Fe) and MX (M = Ta, V; X = C, N). These carbides are apparently of great importance to influence the mechanical properties, therefore, many studies on the precipitates in F82H steel have been investigated [3–7].

On the other hand, it is easy to notice that the characterizations of the carbides, including particle size, number density, chemical composition and so on, were carried out via transmission electron microscope (TEM) observations in most of the investigations [3,4]. Owing to the very high resolution and unique crystallographic structure determination ability, TEM is a very powerful tool to characterize the fine particles in a wide variety of materials. In terms of macro-scale characterizations, however, the shortage of view field and less efficiency are the weaknesses of TEMs. It is not suitable to measure coarse sized particles such as inclusions, which generally possess the size of several μm [5]. Further, TEM has difficulty in the quantitative estimation of precipitate and dislocation densities in F82H steels, because of the complicated and relatively large scale microstructure due to tempering and martensitic transformation [1]. This results in a large margin of error in the estimated data. Thus, TEM observation is insufficient to well build the microstructure database for F82H steel. A more precise estimation of carbide density is expected by macroscale experiments like extracted residue test (ERT) combined with other techniques.

* Corresponding author. Fax. +81-22-215-2066.
E-mail address: kano.sho0215@gmail.com (S. Kano).

http://dx.doi.org/10.1016/j.nme.2016.09.017

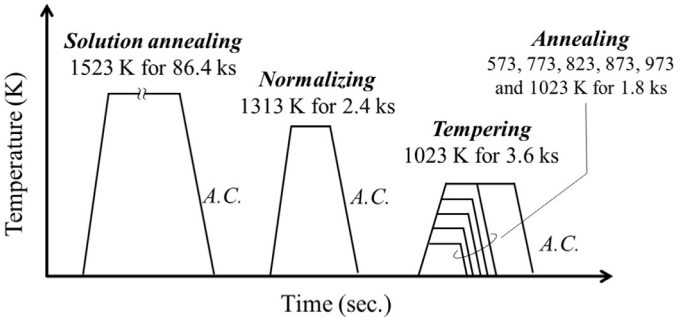

Fig. 1. Thermal history of F82H steels. A.C. denotes air cooling. Heating and cooling rates were 2 and 3 K/s, respectively.

Nagasaka et al. [6] proposed a combination of ERT, X-ray diffraction (XRD) and inductively coupled plasma (ICP) analyses to analyze the precipitation in the thermally aged F82H steels. Since the average sizes of MX and $M_{23}C_6$ in the steel were 60 and 118 nm [3,7], respectively, they were successful in filtering the carbides. However, they underestimated the amount of carbides, especially MX, which our range size is less than 50 nm [8–10]. The purpose of the present study is, therefore, to investigate the precipitation of carbides in F82H steels by TEM and ERT. The precipitation will be investigated tempered and annealed F82H steels and its model alloys to get insights into the corresponding precipitation strengthening.

2. Experimental procedure

Two kinds of experimental materials were used in the present study. One is F82H steel (Fe-0.098C-7.81Cr-1.88W-0.44Mn-0.19V-0.037Ta), and the other is the ternary model alloy (Fe-0.015C-0.19Ta), which will be called Fe-0.2TaC hereafter. The carbon concentration of F82H steel is slightly lower than that of the original F82H-IEA (0.1 wt.%C) [5]. In order to prevent carburizing and decarburizing, the specimens were acidized using a solution of 20% hydrochloric acid and balanced methanol for 43.2 ks. Then, the specimens were capsuled into clean quartz tubes with high purity argon gases. Thermal history of the three-step heat treatment of F82H steel is shown in Fig. 1. The specimens firstly underwent the solution annealing at 1523 K for 86.4 ks to homogenize the alloying elements. Subsequently the specimens were normalized at 1313 K for 2.4 ks. Afterwards, the specimens were tempered at 573, 773, 823, 873, 973 and 1023 K for 1.8 ks, or 1023 K for 3.6 ks, respectively. In all of the heat treatments, the heating and cooling rate were respectively set as 2 and 3 K/s, also, the occurrence of martensitic transformation in this cooling rate was confirmed from the thermal expansion measurement. For the F82H steels, the one tempered at 1023 K for 3.6 ks is named as tempered F82H, as this heating regime is the same with F82H-IEA tempering process; the rest of the specimens (annealed at 573, 773, 823, 873, 973 and 1023 K for 1.8 ks) are named as annealed F82H. For the Fe-0.2TaC, the specimens were tempered at 1123 K for 3.6 ks, after a solution annealing at 1523 K for 43.2 ks followed by water quenching.

The ERT was conducted using an AA solution, the mixture of 10% acetyl acetone, 10% tetra methyl ammonium chloride and balanced methanol, with keeping the electric current of roughly 100 mA at room temperature [6,7]. At this electric current, it was examined that the matrix could be electrochemically dissolved and precipitates were stably remained in the etchant solution [11,12]. The remained precipitates were collected on a membrane filter with 50 nm pore size by vacuum percolation. The extracted residues were, then, weighed using an electronic balance with an accuracy of ±0.1 mg, which was roughly equivalent to

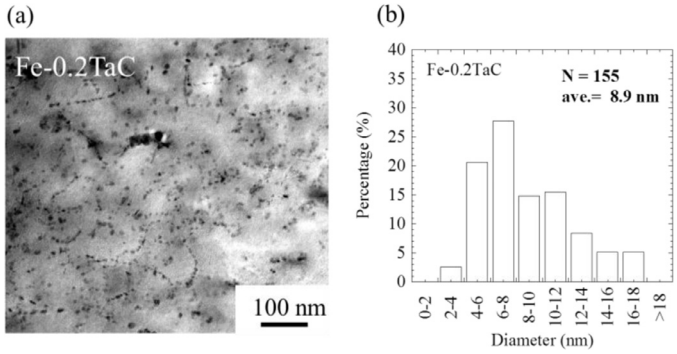

Fig. 2. (a) TEM bright field image of Fe-0.2TaC model alloy, and (b) the size distribution of TaC. N and ave. stand for the number of measurement and the averaged size.

Table 1
Weight percentage of extracted residue in Fe-0.2TaC model alloy.

Test No.	Extracted residue, *W* (%)
1	0.242
2	0.234
3	0.251
AVE	0.242

0.1% of the total weight of residue obtained in each test. Afterwards the XRD analysis was performed on the extracted residues on an X-ray diffractometer (Rigaku, Ultima IV) with a Cu target at 40 kV and 40 mA. The size and number density of carbides were also investigated by TEM, so as to compare to the results attained from ERT. Twin-jet electro-polishing to achieve electron transparent F82H specimens was carried out in a solution containing 8% perchloric acid and acetic acid at room temperature.

3. Results and discussion

3.1. Comparison of particle volume fractions obtained by TEM observation and ERT

3.1.1. Fe-0.2TaC

Fig. 2 shows the bright field (BF) image of Fe-0.2TaC and the size distribution of carbides. It is noticed from the BF image (Fig. 2(a)) that numerous fine TaC particles were dispersed in the matrix, appearing in either spherical or thin foil shape, which is consistent to a previous study [13]. The thickness of the observed region was determined by the thickness fringe technique, and the number density of carbides was estimated as 7.9×10^{21} m^{-3}. As shown in Fig. 2(b), the unimodal size distribution was observed ranging from roughly 2 to 20 nm with a peak at around 6–8 nm. Also, average particle size, which is defined as the length of the major axis, was estimated as roughly 8.9 nm, which is consistent with our previous study [14]. Thereby, the volume fraction of TaC was calculated as 0.289%, assuming the particles were spherical.

The number density of TaC was also examined by the ERT. The results are listed in Table 1. Weight percentage of extracted residue, *W*, is calculated according to the following equation [11]:

$$W = \frac{W_{PPT}}{\Delta W} \times 100\% \quad (1)$$

where, W_{PPT} is the weight of precipitates and ΔW is the weight difference of specimen between before and after ERT. The ERT was repeated three times, and the average weight percentage of the extracted residue was 0.242%, corresponding to a volume fraction of TaC of 0.157%. The volume fraction is obtained by multiplying the weight fraction with the density ratio (D_{matrix}/D_{ppt}),