MATERIALS CHARACTERIZATION 84 (2013) 196–204

Available online at www.sciencedirect.com

ScienceDirect

www.elsevier.com/locate/matchar

Influence of W and Ta content on microstructural characteristics in heat treated 9Cr-reduced activation ferritic/martensitic steels

Ravikirana [a], *R. Mythili* [b], *S. Raju* [b], *S. Saroja* [b,*], *T. Jayakumar* [b], *E. Rajendrakumar* [c]

[a]*Homi Bhabha National Institute, Indira Gandhi Center for Atomic Research, Kalpakkam, India*
[b]*Metallurgy and Materials Group, Indira Gandhi Center for Atomic Research, Kalpakkam, India*
[c]*TBM Division, Institute of Plasma Research, Gandhinagar, India*

ARTICLE DATA

Article history:
Received 18 February 2013
Received in revised form
2 August 2013
Accepted 11 August 2013

Keywords:
RAFM steel
Microstructure
Martensite
Tempering
Carbides

ABSTRACT

This study aims at understanding the microstructural changes due to variation of W and Ta content and heat treatment in 9Cr–W–Ta–0.2V–C reduced activation ferritic/martensitic steels, with W and Ta concentrations varied from 1 to 2 wt.% and 0.06 to 0.14 wt.% respectively. An increase in concentration of W and Ta in the steels refined prior austenite grain size and lath width, though the change was not significant beyond 1.4 wt% W. Increase in W and Ta concentrations also resulted in a decrease in size and increase in number density of carbides on tempering. Microchemical analysis of $M_{23}C_6$ precipitates in tempered steel showed enrichment of W with increase in W content of the steel, whereas addition of Ta showed no significant change in chemistry of $M_{23}C_6$ or MX, but only increased the number density of fine MX precipitates.

1. Introduction

9Cr ferritic/martensitic steels are structural materials for steam generator circuits of power generation industries due to their good mechanical properties at elevated temperatures. Advanced 9Cr–1Mo steels are also considered as candidate materials for core component applications in metal fueled sodium cooled fast breeder reactors [1–3] due to their excellent radiation swelling resistance and compatibility with sodium. Alloying elements like Mo and Nb in 9Cr–1Mo steels are reported [1] to transmute on neutron irradiation and produce long lived radioactive isotopes, complicating post irradiation service and radioactive waste disposal. Hence, replacement of Mo by W and Nb by Ta in the reduced activation ferritic/martensitic (RAFM) steels finds them as attractive structural materials for fusion reactors [1–3]. Continuous efforts are in progress to develop RAFM steels with better mechanical properties than modified 9Cr–1Mo steels by tailoring the alloy chemistry and processing parameters [1,4]. The effect on W and Ta on mechanical properties of RAFM steels is well reported in literature [5,6]. It is also known that the mechanical properties of the steel are dictated by its microstructural stability. A general trend in the microstructural evolution in 9–12Cr ferritic/martensitic steels is fairly known [7], though literature reporting the specific role of these elements on microstructure is scarce. It is reported that addition of W controls coarsening of $M_{23}C_6$ [8], while Ta refines the PAGS significantly [9]. However, a systematic variation of the W and Ta concentrations of the steel and a study on their effect on microstructure are necessary to understand the role of

* *Corresponding author*. Tel.: +91 44 27480306; fax: +91 44 27480202.
E-mail address: saroja@igcar.gov.in (S. Saroja).

1044-5803/$ – see front matter © 2013 Elsevier Inc. All rights reserved.
http://dx.doi.org/10.1016/j.matchar.2013.08.003

these elements, especially the substructural changes and precipitation characteristics like their distribution and microchemistry. A complete characterization of the initial microstructure in the normalized and tempered condition is necessary, since creep strength of the steel to a large extent depends on the initial microstructure and its long term stability at elevated temperatures. This study aims to understand the effect of W and Ta on the microstructure through a detailed investigation of different microstructural parameters on normalizing, subsequently followed by tempering of 4 different RAFM steels with a systematic variation of W and Ta concentrations from 1 to 2 wt.% and 0.06 to 0.14 wt.% respectively.

2. Materials and Methods

The chemical composition of the four 9Cr RAFM steels employed in this study is given in Table 1. The steels were supplied by MIDHANI, Hyderabad, India, in the form of 12 mm thick plates. The steel was produced through vacuum induction melting followed by vacuum arc refining. Concentration of radioactive tramp elements and other elements that promote embrittlement was restricted to ppm level. The steels have been remelted three or four times to ensure a high degree of homogeneity. Plates of 12 mm thickness were obtained through subsequent controlled thermo-mechanical processing including forging, rolling and heat treatments consisting of normalizing for 30 min at 1253 K followed by tempering for 60 and 90 min at 1033 K for the 1W and other steels respectively.

The above steels were normalized for 30 min at 1253 K in the laboratory, followed by tempering at 1033 K for 60 min. After metallographic preparation, the steels were etched with 2% Nital followed by Villella's reagent. Preliminary microstructural analysis was carried out using Optical Microscopy and Scanning Election Microscopy (SEM) and hardness was measured using Vickers hardness tester. Detailed Transmission Electron Microscopy (TEM) investigations were carried out on thin foils prepared by conventional electropolishing and carbon extraction replica to characterize the fine microstructural features. Details of the techniques employed and their operating conditions are summarized in Table 2. Image analysis software ImageJ® [10] was used for measurement of prior austenite grain size (PAGS), lath width, carbide distribution and analysis of electron diffraction patterns. Identification of the type of carbides was carried out using electron diffraction pattern analysis. Further, microchemical analysis of carbides was performed using Cliff–Lorimer method [11] on carbon extraction replica of steels to avoid interference from the matrix of α ferrite. Quantification of the composition of $M_{23}C_6$ carbides in both normalized and tempered steels was carried out using Fe Kα, Cr Kα and W Lα lines using the procedure detailed elsewhere [11,12]. The normalized concentration of the metal atoms alone in the precipitates was determined and no attempt was made to determine the carbon concentration of the carbides. Since the type of carbide is known as $M_{23}C_6$ by SAD analysis and the specific ratio of the content of the elements is determined by EDS analysis, the stoichiometry of the carbide is calculated as follows for Fe, Cr and W.

Table 2 – Details of experimental techniques.

Technique	Instrument used
Optical microscopy	Leica MEF4 A
SEM	Helios 600i, FEG SEM operated at 20 kV
Hardness	FIE VM-50 PC based Vickers hardness tester at 10 Kg load
TEM	Phillips CM 200 ATEM (200 kV) with Oxford X-Max SDD detector

$$XFe = \frac{\text{At.\%Fe in M}}{100} \times 23. \tag{1}$$

3. Results

3.1. Microstructural Characterization of Normalized Steels

Fig. 1(a to d) shows SEM micrographs of the four normalized steels. It is observed that the microstructure is fully martensitic. No evidence for presence of δ-ferrite was observed in any of the steels. Variation of PAGS and hardness with increase in W and Ta is presented in Table 3. It is observed that PAGS decreases from 25 to 11 μm, with increase in both W and Ta content. The maximum value of hardness is observed for the steel with 1.4%W.

For a detailed study of the microstructural features, TEM analysis of the thin foil and carbon extraction replica of the normalized steels was carried out. A lath martensitic structure with high density of dislocations was observed in all the normalized steels. Measurement of lath width showed a decrease from 365 to 240 nm with increase in W, while increase in Ta did not show appreciable change, suggesting higher influence of W than Ta. Fig. 2(a) shows a typical carbon extraction replica micrograph of 1W–0.14Ta steel. Presence of both fine and coarse precipitates with globular and acicular morphologies is observed in all the steels. Fig. 2(b) shows the typical EDS spectra taken from different types of precipitates, which are enriched with Cr or Ta or V. Electron diffraction patterns from these precipitates are shown in Fig. 2(c to e), based on which they were identified to be $M_{23}C_6$ or MX. The

Table 1 – Chemical composition (wt.%) of RAFM steels studied.

Steel	Element (wt.%)									
	Cr	C	Mn	V	W	Ta	N	O	P	S	Fe
1W–0.06Ta	9.04	0.08	0.55	0.22	1	0.06	0.0226	0.0057	0.002	0.002	Bal.
1.4W–0.06Ta	9.03	0.126	0.56	0.24	1.38	0.06	0.03	0.002	<0.002	<0.001	Bal.
2W–0.06Ta	8.99	0.12	0.65	0.24	2.06	0.06	0.02	0.0024	0.002	0.0014	Bal.
1W–0.14Ta	9.13	0.12	0.57	0.22	0.94	0.135	0.033	0.0041	<0.002	0.0015	Bal.