Influence of thermo-mechanical treatment in ferritic phase field on microstructure and mechanical properties of reduced activation ferritic-martensitic steel

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Abstract: The present study focuses on the evaluation of microstructure and mechanical properties of reduced activation ferritic-martensitic (RAFM) steel (9Cr-1W-0.06Ta) subjected to thermo-mechanical treatment (TMT) in ferritic phase field. The results obtained were compared with the steel in conventional normalised plus tempered (N+T) condition. The microstructure of the steel in N+T and TMT conditions was assessed by optical and scanning electron microscopes. Hardness, tensile and creep studies were carried out and the results were correlated with the microstructural studies. While the TMT processed steel resulted in coarser prior austenite grains and exhibited ferritic microstructure with large distribution of fine M23C6 and MX precipitates, the N+T steel reveals tempered martensitic structure with finer prior austenitic grains with coarser M23C6 and MX precipitates. Although ferritic structure is present in TMT processed steel, it exhibits better tensile and creep rupture strengths than N+T steel due to the presence of increased dislocation density and finer distribution of precipitates.

Keywords: Reduced activation ferritic-martensitic steel; Thermo-mechanical treatment; Electron microscopy; Tensile and creep strengths.

1. Introduction

Reduced activation ferritic-martensitic (RAFM) steels having 9Cr-1W-0.22V-0.06Ta as the major alloying elements are potential candidate materials for the test blanket module (TBM) compared to austenitic steels due to their attractive properties, for example, low thermal expansion coefficient, high thermal conductivity and favourable void swelling resistance [1]. The steel in normalized and tempered condition consists of tempered martensite with laths, blocks, and packets inside the prior-austenite grains. The laths, blocks and packets contain high concentration of transformation induced dislocation network, decorated with fine intra-granular MX type (where M=V, Ta; X=C, N) precipitates, and chromium-rich M23C6 carbides along the boundaries [2-4]. The microstructure of the ferritic-martensitic steel depends on the heat treatment temperatures especially those inducing phase transformation [5]. The precipitates coarsen on thermal aging and also during creep exposure at elevated temperatures by Ostwald ripening mechanism. The coarsening rate of M23C6 carbides is much faster than the MX carbo-nitrides [6]. The coarsening of M23C6 carbides present along the prior austenitic grain boundaries promote faster microstructural recovery in the vicinity of grain boundaries [7], thus resulting in increase in minimum creep rate, early onset of tertiary stage of creep deformation and hence reduce the creep rupture life. To increase creep rupture strength of the steel, it is desirable either to (i) decrease coarsening kinetics of M23C6 precipitates or (ii) eliminate the presence of unstable M23C6 carbides from martensitic microstructure and promote the precipitation of very fine and thermally more stable MX carbo-nitrides not only within the matrix but also along the prior austenitic grain boundaries.

In the present work, an attempt has been made to understand the effect of thermo-mechanical treatment on microstructure and mechanical properties of RAFM steel. The tempered martensitic
structure of RAFM steel in normalized and tempered condition is transformed into ferritic (α) phase by prolonged soaking. The steel in ferritic phase field was subjected to thermo-mechanical treatment by warm rolling. The microstructural stability, tensile and creep properties of thermo-mechanically processed steel in ferritic phase condition were compared with those obtained in normalized and tempered condition.

2. Experimentation

2.1 Materials and Methods

The RAFM steel (9Cr - 1W - 0.06Ta), in the form of plate, was received in normalised and tempered condition. The normalizing was done at 1253 K for 30 min followed by tempering at 1038 K for 90 min. The major alloying elements (in wt.%) present in the steel are given in Table 1. The received plate was subjected to thermo-mechanical treatment (TMT) by warm rolling. Prior to TMT, the steel was austenitized at 1423 K for 10 min followed by air cooling up to 973 K and then holding for 2 h at the same temperature in a furnace to transform into fully ferritic phase. Subsequently, warm rolling was carried out at 973 K using a 200 ton capacity, two-high rolling mill (Make: DEMAG) with a roll diameter of 300 mm. During warm rolling, a reduction of 25 % in thickness was imparted in five passes. This was followed by tempering at 1038 K for 150 min. Representative samples of steel from normalized and tempered condition and TMT condition were cut and polished to determine their microstructures using optical and electron microscopes. The samples were immersion etched with Villella's reagent (1 g picric acid and 5 ml hydrochloric acid in 100 ml ethanol). Hereafter, the steel in normalized and tempered condition is referred to as "N+T" and the steel with 25 % reduction in thickness by warm rolling followed by tempering is referred to as "FF-TMT25". A Leica metallurgical microscope (Model: DMi8A) equipped with image analysis software (Leica Application Suite (LAS), version 4.8) was used for obtaining optical microstructures of steel in N+T and FF-TMT25 conditions. High magnification images of microstructural features of steel in N+T and FF-TMT25 conditions were obtained by using scanning electron microscope (SEM) (Make: TESCAN, Model: VEGA 3 LMU). The hardness of the steel in N+T and FF-TMT25 conditions was determined by using Vickers macro hardness tester (Make: Matsuzawa, Model: VMT-X) using 10 kgf force and 15 s dwell time. At least ten measurements were made in each condition and their average and standard deviation values were reported. Threaded cylindrical tensile specimens were prepared from N+T and FF-TMT25 steel plates whose gauge length is parallel to the rolling direction. Tensile properties were evaluated in the temperature range of 300 - 923 K and at nominal strain rate of 3 × 10⁻⁴ s⁻¹. The creep rupture life of the steel in N+T and FF-TMT25 conditions was assessed by carrying out constant load creep tests at 823 K in ambient air at 240 MPa.

Table 1. Chemical composition of RAFM steel (in wt. %) (Major elements)

| Element | C   | Cr  | W   | V   | Ta  | Mn  | N   | Fe |
|---------|-----|-----|-----|-----|-----|-----|-----|----|
| wt.%    | 0.08| 9.04| 1.01| 0.22| 0.06| 0.55| 0.0226| Rest |

3. Results and Discussion

3.1 Microstructural analysis

Figures 1a and 1b shows the optical microstructures of steel in N+T and FF-TMT25 conditions respectively. The steel in N+T condition revealed tempered martensitic structure (Figure 1a) and that in FF-TMT25 condition revealed fully ferritic phase (Figure 1b). The existence of ferrite phase was also confirmed through macro hardness measurement and is discussed in the next session. The average prior austenitic grain (PAG) size of steel in N+T and FF-TMT25 conditions was found to be 15±2 μm and 150±20 μm respectively. As the PAG size in FF-TMT25 condition is very large, a lower magnification image of the steel is shown in Figure 1b to cover more PAGs. Coarser PAG size by TMT was also reported by Sakthivel et al. [8] for modified 9Cr-1Mo steel (T91 steel). The coarsening of PAGs in FF-TMT25 steel is due to the reaustenitising at high temperature where most of the precipitates dissolved.
SEM images of RAFM steel in N+T and FF-TMT25 conditions are shown in Figures 2a and 2b respectively. In N+T condition the PAGs are seen clearly and are decorated by M$_{23}$C$_6$ precipitates along the boundaries, whereas in FF-TMT25 condition the PAG structure is refined and the precipitates are formed along the sub grain boundaries too and found occasionally within the grains. Based on the previous studies [4], the larger precipitates are of M$_{23}$C$_6$ and the finer one are of MX type. It can also be clearly seen that the number density of precipitates in TMT steel is higher than those in the N+T condition. This can be attributed to the fact that the warm rolling of steel develops higher dislocation density. These dislocations serve as preferential nucleation sites for the precipitation of M$_{23}$C$_6$ and MX precipitates. Thus resulting in the formation of large number of precipitates in TMT steel compared to N+T steel. Further, it can be observed that the sizes of M$_{23}$C$_6$ and MX precipitates in FF-TMT25 condition is finer than those observed in N+T condition. While the size of M$_{23}$C$_6$ and MX precipitates are in the range of 50-160 and 20-30 nm respectively in N+T condition, their size was refined and found to be in the range of 30-120 and 5-30 in TMT condition.

Figure 1. Optical micrographs of RAFM steel in (a) N+T (b) FF-TMT25 steel condition.

Figure 2. Scanning electron microscope images of RAFM steel in (a) N+T (b) FF-TMT25 steel condition.

3.2 Hardness

The Vickers hardness values of the steel in N+T, TMT in ferritic phase field condition (without tempering), and TMT processed plus tempered (FF-TMT25) conditions are shown in Figure 3. The
hardness of steel in N+T condition is found to be 206±2 HV, which is increased to 409±2 HV upon TMT in ferritic phase field. This sudden rise in hardness can be attributed to the development of high density of dislocation structure resulted due to deformation and the existence of large number of fine precipitates [8-10]. A drastic reduction in hardness can be observed after tempering (i.e. in FF-TMT25 condition), which could be due to the presence of fully ferritic phase and annihilation of dislocations.

Figure 3. Macro Vickers hardness of RAFM steel in different conditions.

3.3 Tensile analysis

Engineering stress-strain curves of RAFM steel in N+T and FF-TMT25 conditions tested in the temperature range of 300 K - 923 K at strain rate of $3 \times 10^{-4}$ s$^{-1}$ are shown in Figure 4. The curves show that the steel in both conditions follow monotonic behavior at all test temperatures. Further, there is no evidence of serrated flow in both conditions of the steel, which commonly noticed in ferritic steels in the intermediate temperatures [11, 12]. While the strain to failure is reduced as the temperature is increased from 300 K to 773 K, and it is increased beyond 773 K in both N+T and FF-TMT25 conditions.

Figure 4. Comparison of engineering stress - engineering strain plots of RAFM steel in N+T and FF-TMT25 conditions at selected temperatures.

Figure 5. Variation of tensile strengths with test temperature.
The variation of yield strength (YS) and ultimate tensile strength (UTS) of RAFM steel in N+T and FF-TMT25 conditions with temperature is shown in Figure 5. The YS and UTS were found to decrease with increase in the test temperature. TMT of RAFM steel in ferritic phase field is found to impart slight improvement in YS and UTS than the steel in N+T condition. However, plateau region is observed in the YS in the intermediate temperature range (473 - 573 K) for N+T condition and similar feature is observed at (623 K-723 K) for FF-TMT25 condition, for the UTS plots the plateau region for N+T condition is found for temperature range of (473 K- 523 K) and no plateau region found for FF-TMT25 condition. A rapid decrease in YS and UTS was observed beyond 773 K for both conditions of steel. The yield stress of FF-TMT25 steel was higher than the N+T steel up to 573K beyond which close fluctuations are observed in both conditions. Higher UTS values are observed in TMT condition, except at 523 - 573 K, than N+T condition. On a whole slight improvement in YS and UTS are observed in FF-TMT25 condition than the N+T condition. This slight increase in tensile strengths of FF-TMT25 steel over N+T condition at higher temperatures can be attributed to the presence of fine and homogeneous distribution of precipitates, which assisted in delaying the dynamic recovery of the dislocation structure; where the dominance of dynamic recovery has been observed in the steel generally at higher test temperatures. This behaviour can be correlated with the microstructural features discussed in Figures 2a and 2b.

Figure 6 shows the percentage elongation and percentage reduction in area of RAFM steel in N+T and FF-TMT25 conditions. Percentage elongation is lower at room temperature for FF-TMT25 condition than N+T condition and small dip is observed at 523 K- 773 K in both conditions with closer values; higher elongations are observed for N+T condition beyond 773 K. Percentage reduction in area is lower for FF-TMT25 condition than the N+T condition over all the temperature range. Slight decrease at intermediate temperatures and rapid increase at higher temperatures for both conditions of the steel are observed. The appearance of mild plateau in YS and UTS as well as dip in elongation curves in the intermediate temperature range is an indication of the occurrence of dynamic strain aging (DSA) [11-13].

3.4 Creep properties
The creep curves of RAFM steel in N+T and FF-TMT25 conditions at stress level of 240 MPa and temperature of 823 K are compared in Figure 7. From these curves, it is evident that TMT processing of steel in ferritic phase field has significantly lowered the creep deformation and greatly improved the rupture life. The reduced creep deformation and improved rupture life can be attributed to the presence
of large number density of nano sized M$_{23}$C$_6$ and MX precipitates. The presence of these nano size precipitates significantly delays the onset of tertiary stage of creep, there by leading to the enhancement of rupture life than the same steel in N+T condition.

4. Conclusions

In the present work, the effect of TMT in ferritic phase field on tensile properties and creep rupture life of RAFM steel was studied and the data obtained was compared with the steel in N+T condition. The following are the salient conclusions:

(i) The microstructure of the RAFM steel was refined through TMT. Coarser prior austenite grain sizes, finer sub-grains, dense and homogeneous distribution of precipitates were achieved in the TMT steel as compared to the N+T steel. The number density of M$_{23}$C$_6$ and MX type precipitates are more in FF-TMT25 condition than N+T condition.

(ii) Vickers hardness value of 198 HV$_{10}$ at the end of TMT followed by tempering proved that the transformation of tempered martensitic structure (in N+T condition) to ferritic phase (in FF-TMT25 condition).

(iii) TMT of RAFM steel in ferritic phase field was found to impart slight improvement in YS and UTS than the steel in N+T condition at all test temperatures and at strain rate of 3$\times$10$^{-4}$ s$^{-1}$.

(iv) Creep tests performed at 240 MPa and 823 K on N+T and FF-TMT25 steels showed significant improvement in rupture life of TMT steel over N+T steel.

References

[1] Zinkle S J and Busby J T 2009 Mater. Today 12 pp12-19.

[2] Jayaram R and Klueh R L 1998 Metall. Mater. Trans. A 29 pp1551-1558.

[3] Klueh R L, Alexander D J and Sokolov M A 2002 J. Nucl. Mater. 304 pp139-152.

[4] Fernandez P, Lancha A M, Lapena J and Hernandez-M 2001 Fusion Eng. Des. 58–59 pp787-792.

[5] Saroja S, Dasgupta A, Divakar R, Raju S, Mohandas E, Vijayalakshi M, Bhanu Sankara Rao K and Raj B 2011 J. Nucl. Mater. 409 (2) pp131-39.

[6] Ravikiran, Mythili R, Raju S, Saroja S, Jayakumar T, Rajendrakumar E Mater. Charact. 84 pp196-204.

[7] Chandravathi K S, Sasmal C S, Laha K, Parameswaran P, Nandagopal M, Vijayanand V D, Mathew M D, Jayakumar T and Rajendra Kumar E 2013 J. Nucl. Mater. 435 pp128-136.

[8] Sakthivel T, Shruti P, Parameswaran P, Nageswara Rao G V S, Laha K and Srinivasa Rao T 2016 Trans Indian Inst Met 47 pp204-209.

[9] Benjamin A, Shassere, Yamamoto Y, Suresh Babu S 2016 Metall. Mater. Trans. A 47A 2190.

[10] Matthias Nohrer, Walter Mayer, Sophie Primig, Sabine Zamberger, Ernst Kozeschnik, and Harald Leitner 2014 Metall. Mater. Trans. A 45A pp4210-4219.

[11] Vanaja J, Laha K, Shiju Sam, Nandagopal M, Panneer Selvi S, Mathew M D, Jayakumar T, Rajendra Kumar E 2012 J. Nucl. Mater. 424 pp116-122.

[12] Vanaja J, Laha K, Nandagopal M, Shiju Sam, Mathew M D, Jayakumar T, and Rajendra Kumar E, 2013 J. Nucl. Mater. 433 pp412-418.

[13] Palaparti D P, Choudhary B K, Isaac Samuel E, Srinivasan V S and Mathew M D 2012 Mater. Sci. Eng. A 538 pp110-117.