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# Effect of tempering temperature on the microstructure and creep resistance of a 10%Cr martensitic steel

# Effect of tempering temperature on the microstructure and creep resistance of a 10%Cr martensitic steel

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**Abstract**. The tempered martensite lath structure of a 10% Cr martensitic steel after normalizing and tempering at 750 °C was studied. The effect of reducing the tempering temperature from 770 by 20 °C on the microstructural parameters in steel was shown. Tempering at a lower temperature of 750 °C provides thinner laths and finer $M_{23}C_6$ particles (55 nm) than tempering at 770 °C. Such structural changes do not affect the creep rupture time at 650 °C and applied stresses of 180 and 160 MPa, whereas the positive effect occurs at 140 MPa, the time to rupture increases by more than two times from 1426 to 3909 hours.

## 1. Introduction

High-chromium creep-resistant martensitic steels are widely used for critical components of fossil power plants that operate at temperatures up to 620 °C due to their excellent creep resistance [1]. The newest generation of 9–10% Cr steels with 3 wt.% Co addition, an increased B content (≥0.008 wt.%) and a highly reduced N content (<0.003 wt.%) was developed to increase the stability of the tempered martensite lath structure and, therefore, to enhance the creep strength [2,3]. A ten-fold increase in the creep rupture time of these steels as compared with that of the P92-type steels was attained [4]. This fact is associated mainly with improving the coarsening resistance of $M_{23}C_6$ boundary carbides during tempering and creep. Thus, the size of carbides after tempering at 770 °C in this steel is ~70 nm [4]. It can be suggested that steel tempered at a lower temperature due to the smaller $M_{23}C_6$ carbide size could exhibit a higher creep rupture time. The aim of this work is to study the effect of lowering the tempering temperature from 770 to 750 °C on the microstructural and creep characteristics at 650 °C. The results of studying the steel tempered at 750 °C are compared with those for 770 °C published earlier [5].

## 2. Material and methods

A 10% Cr steel with the chemical composition (in wt.%) 0.1 C, 0.06 Si, 0.1 Mn, 10.0 Cr, 0.17 Ni, 0.7 Mo, 0.05 Nb, 0.2 V, 0.003 N, 0.008 B, 2.0 W, 3.0 Co, 0.002 Ti, 0.006 Cu, 0.01 Al and Fe-balance was examined. The steel was subjected to normalizing at 1060 °C for 30 min and tempering at 750 °C for 3 h. The microstructure was examined using a JEOL JEM-2100 transmission electron microscope (TEM) and a Quanta Nova scanning electron microscope (SEM) equipped with an electron backscatter diffraction (EBSD) pattern analyzer incorporating an orientation imaging microscopy (OIM) system. Flat specimens with a gauge length of 25 mm and cross-sectional dimensions of 7 mm×3 mm were subjected to creep tests until rupture under applied stresses of 140, 160 and 180 MPa at 650 °C.

## 3. Results and discussion

### *3.1. Tempered martensite lath structure*

The structure of the 10% Cr steel after tempering at 750 °C is characterized as a tempered martensite lath structure with an average size of prior austenite grains (PAG) of about 35 μm as well as after tempering at 770 °C. On the other hand, the lath structural and precipitation parameters of 750 °C tempered steel Figure 1 sufficiently differ from those of 770 °C tempered as can be seen from Table 1. The average width of martensitic laths is smaller and comprises 275 nm (Figure 1a). The dislocation density is slightly higher, comprises about $2.3\times10^{14}$ m$^{-2}$. Finer $M_{23}C_6$ – type carbides with a mean size of 55 nm are located at the high-angle boundaries (HABs) of PAGs, packets, and blocks and low-angle boundaries (LABs) of laths nearly in the equal proportion. The average size of Nb-enriched M(C,N) carbonitrides is also smaller (20 nm). They are uniformly distributed within the martensitic laths. It should be noted that V-rich M (C,N) particles, as after tempering at 770 °C are not observed [5]. Scarce, fine W-rich $M_6C$ carbides with the same average dimension of 25 nm are found whereas no Laves phase precipitates are observed.

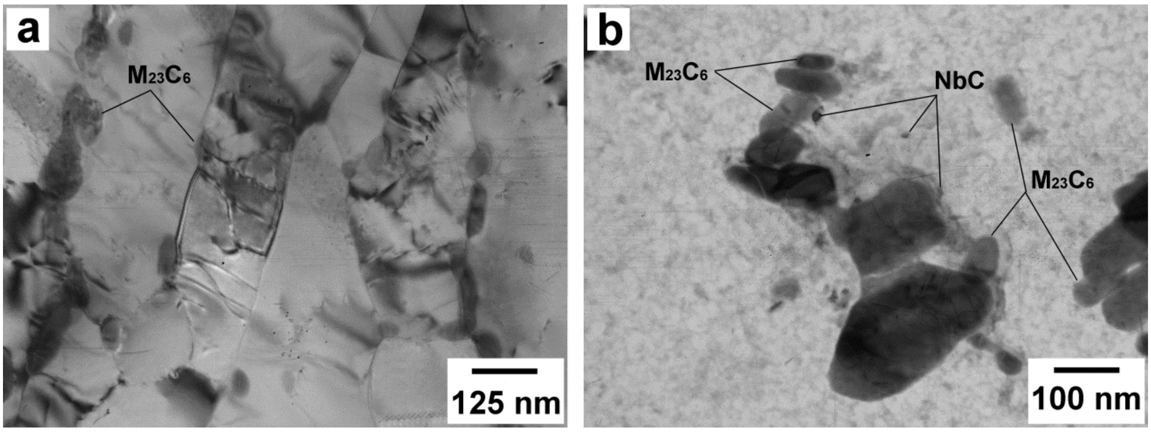


**Figure 1.** Microstructure of the 10% Cr steel after tempering at 750 °C: TEM micrographs show precipitated phases on foil (a) and replica (b).

Figure 2 demonstrates the data from EBSD analysis. ''Bain circles" appeared on the {001} pole figure (Figure 2d) reconstructed from a separate PAG (Figure 2a), indicating that an austenite→martensite transformation followed the Bain, Kurdjumov – Sachs and Nishiyama – Wassermann orientation relationships [5] took place in the steel. The meso-scale strain distribution indicated by the kernel average misorientation (KAM) map (Figure 1(a)) is non-uniform. Highly-misoriented areas of fine laths alternate with low-misoriented coarse laths within the packets. The KAM value after tempering at 750 °C is ~0.73° that is somewhat higher than after tempering at 770 °C (~0.67°) [5].

**Table 1.** Effect of tempering temperature on microstructural parameters.

| Tempering temperature, °C | Mean size of PAGs, μm | Lath width, nm | Dislocation density, $\times10^{14}$ m$^{-2}$ | Mean size of $M_{23}C_6$, nm | Mean size of Nb(C,N), nm | Mean size of $M_6C$, nm |
|---|---|---|---|---|---|---|
| 750 | 35±5 | 275±30 | 2.3 | 55±5 | 20±5 | 25±5 |
| 770 | 35±5 | 380±30 | 1.7 | 70±7 | 30±5 | 25±5 |

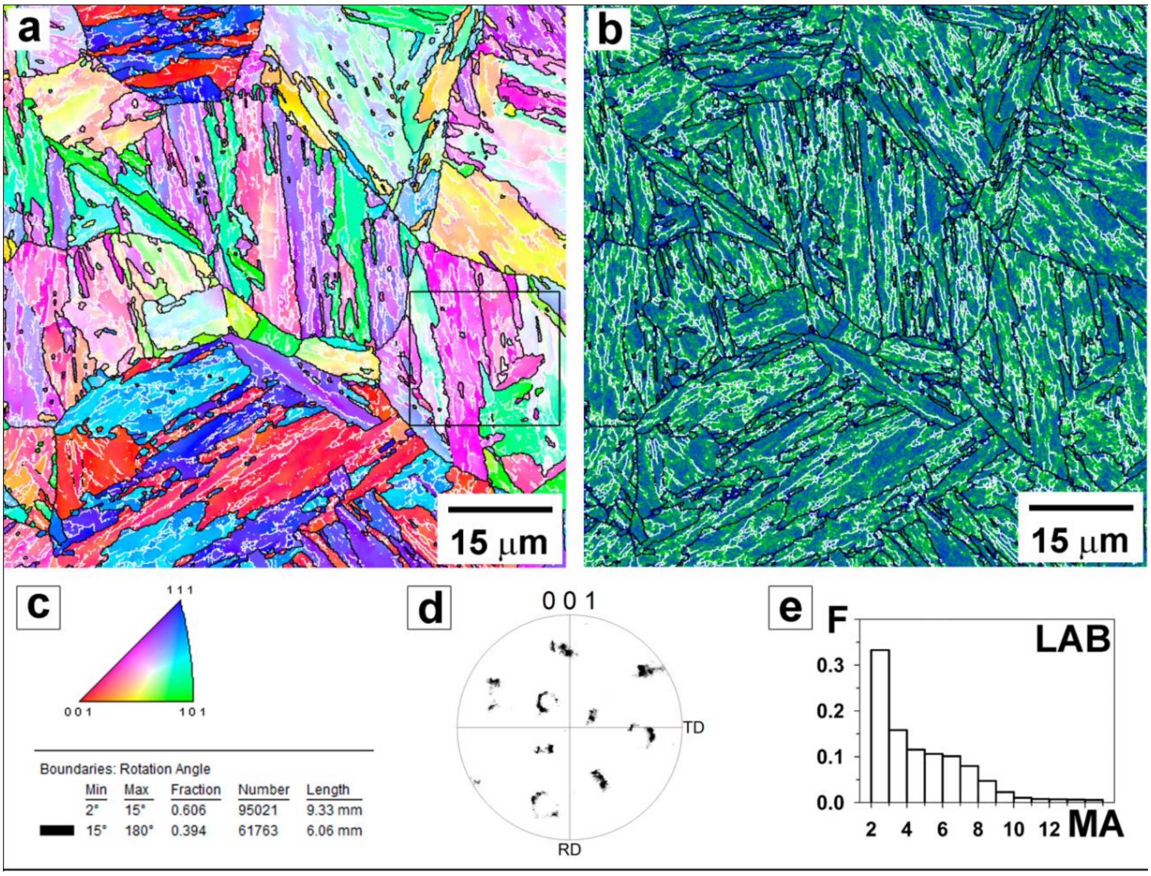


**Figure 2.** Microstructure of the 10% Cr steel after tempering at 750 °C: OIM image with {001} pole figure and distribution of LAB misorientations (F – fraction, (%), MA – misorientations angle, (degree)) (a,c,d,e); kernel average misorientation map (b).

### *3.2. Creep behavior*

As can be seen from Table 2, structural changes associated with a decrease in the tempering temperature had a positive effect on the creep resistance of the 10% Cr martensitic steel. In the short-term region (180-160 MPa), the time to rupture does not depend on the studied tempering temperatures. This fact can be associated with an insignificant contribution of the structure stability to the creep resistance, while the localization of deformation has a large effect on the creep behavior at high applied stresses [6].

A noticeable positive effect of tempering at 750 °C was revealed at an applied stress of 140 MPa. The time to rupture is about 2.7 times higher after tempering at 750 °C as compared to tempering at 770 °C.

**Table 2.** Effect of tempering temperature on creep rupture time (h) at 650°C.

| Tempering temperature, °C | 180 MPa | 160 MPa | 140 MPa | 120 MPa |
|---|---|---|---|---|
| 750 | 32.95 | 203.6 | 3 909 | - |
| 770 | 18.7 | 211 | 1 426 | 39 437 |

The time and strain dependencies of the creep rate at different stresses of the 10% Cr steel after tempering at 750°C are shown in Figure 3(a) and (b), respectively. The minimum creep rate is reduced by approximately one order of magnitude with a decrease in the applied stress from 180 to 160 MPa. It is interesting to note that a more significant reduction in the minimum creep rate by approximately 2 orders of magnitude occurs when the applied stress decreases from 160 to 140 MPa.

Taking into account that the 10% Cr steel shows a satisfactory impact toughness of 70 J/cm$^2$ [7] at ambient temperature after tempering at 750 °C, then temperatures of 750…770 °C can be recommended as the feasible tempering interval for this heat-resistant steel, which provides high creep rupture strength during 100,000 h at 650 °C.

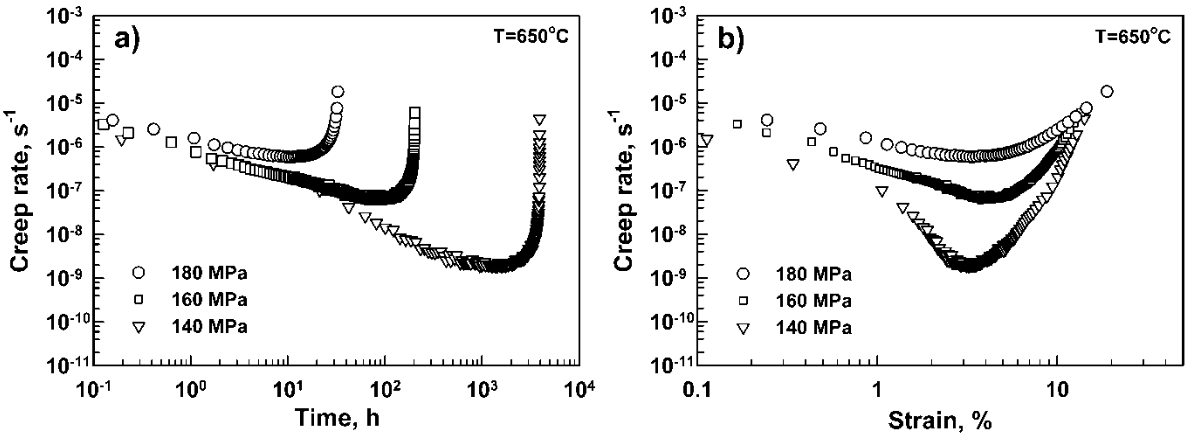


**Figure 3.** Creep rate vs. time (a) and strain (b) curves at 650 °C and different applied stresses from 140 to 180 MPa of the 10% Cr steel tempered at 750 °C.

## 4. Summary

A decrease in the tempering temperature from 770 to 750 °C ensures the formation of a tempered martensite lath structure with thinner laths and finer $M_{23}C_6$ carbides with a mean size of 55 nm in the 10% Cr steel with high B and low N contents. Such structural changes do not affect the creep rupture time at 650 °C and applied stresses of 180 and 160 MPa, whereas at 140 MPa the positive effect on increasing creep rupture time by more than two times from 1426 to 3909 hours occurs as compared to steel tempered at 770 °C.

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