Creep behavior and microstructure of a Ta-added 9%Cr steel with high B and low N contents

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Creep behavior and microstructure of a Ta-added 9%Cr steel with high B and low N contents

E Tkachev*, A Belyakov
Belgorod State National Research University, Pobeda, 85, Belgorod, 308015, Russia

*Corresponding author: tkachev_e@bsu.edu.ru

Abstract. The creep behavior and microstructure of an advanced a Ta-added 9%Cr steel were investigated. The steel is characterized by increased strength in the long-term creep regime, which can be associated with fine dispersion of (Nb,Ta)(C,N) particles. Strengthening of the studied steel due to the fine MX particles was estimated using the Orowan particle strengthening model.

1. Introduction
High-chromium martensitic steels are the primary material for the manufacture of high-temperature elements of modern coal-fired power units. The increasing demands to the energy efficiency of thermal power plants require increasing the temperature and pressure of the steam entering the steam turbine, thus such elements as superheater and reheater tubes have to operate at more stressed conditions.

It is known that the creep strength of martensitic steels can be significantly increased by reducing the initial size and coarsening rate of the second phase particles [1-3]. The \( \text{M}_2\text{C}_6 \) and MX (where M is a metal and X is C and/or N) precipitates are the most important in terms of increasing the stability of the tempered martensite lath structure and long-term creep properties of these steels [1-3]. In recent works, it was shown that an increase in the boron content and additional alloying with tantalum can increase the dispersion of \( \text{M}_2\text{C}_6 \) and MX particles, and thereby increase the creep strength of the steel [3]. However, so far no studies have been carried out on the creep behavior and the distribution of secondary particles in Ta-alloyed steels with a low N content.

This study examines the microstructure and creep behavior of a 9% Cr martensitic steel with an additional amount of boron and tantalum.

2. Materials and methods
Steel Fe-0.1C-9Cr-1.8W-0.6Mo-3Co-Nb-V-0.0012B-0.007N-0.085Ta was produced by vacuum induction melting and subjected to solution treatment at 1200 °C. Then the steel was austenized at 1050 °C for 0.5 h and tempered at 780 °C for 3 h.

The round-bar creep specimens of 6-mm in diameter and 60-mm in gauge length were subjected to tensile creep tests in air under initial applied stresses of 160, 120, and 110 MPa at 650 °C using ATS2330 lever arm machines.

Crept microstructures and dispersed particles of the studied steel were analyzed by transmission electron microscopy (TEM) using a Jeol JEM-2100 microscope operating at 200kV, equipped with an INCA energy-dispersive X-ray (EDX) spectrometer. The dislocation densities were estimated by counting individual dislocations in the lath interiors per unit area on arbitrarily selected TEM images. Extraction carbon replicas were prepared to determine the size and chemical composition of different...
types of precipitates. The volume fractions of secondary (Nb,Ta)(C,N) and V(C,N) particles were estimated using ThermoCalc TCFE7 software.

3. Results and discussion
The creep curves at 650 °C (Figure 1) show that the studied steel is characterized by increased strength in the long-term creep regime, while the creep strength at a relatively high stress of 160 MPa is close to that for standard P92 steel [4]. So that the creep test with an applied stress of 110 MPa is still in progress after about 10 000 hours without rupture.

![Figure 1. Creep rate versus creep time (a) and creep rate versus strain (b) curves for the studied steel subjected to creep tests at 650 °C.](image)

It is seen that a decrease in the applied stress leads to a significant decrease in the minimum creep rate from $9.0 \times 10^{-8}$ s$^{-1}$ at 160 MPa to about $1.3 \times 10^{-10}$ s$^{-1}$ at 110 MPa. Such a rapid decrease in the creep rate with decreasing the applied stress is inherent to the threshold creep behavior and is associated with the presence of dispersed particles.

The microstructure of the studied steel after tempering was described in detail elsewhere [5]. A typical tempered martensite lath structure with particles of M$_{23}$C$_6$ and MX precipitates was observed. The examination of crept microstructures (Figure 2) reveals precipitation of chains of relatively coarse (Fe,Cr)$_2$(W,Mo) Laves phase particles.

![Figure 2. TEM microstructures of the studied steel after creep under 160 MPa for 158 h (a) and under 120 MPa for 3738 h (b).](image)
The microstructural parameters are summarized in Table 1. The creep for 3738 h at 650°C is accompanied by an increase in the lath/subgrain width by ~80%. It is seen in Figure 1 that small subgrains remain in the grain interiors after creep, but there are also large recovered regions without sub-boundaries that have been formed between the packet/prior austenite grain boundaries. In such recovered regions, the uniformly distributed particles of MX carbonitrides are the main obstacles for the moving dislocations.

Table 1. Parameters of tempered and crept microstructures of the studied steel.

|                        | Tempered at 780°C | 160 MPa / 158 h | 120 MPa / 3738 h |
|------------------------|-------------------|-----------------|------------------|
| Lath/subgrain width, nm| 355 ± 35          | 460 ± 20        | 630 ± 45         |
| Dislocation density, ×10^14 m^-2 | 2.4 ± 0.2    | 0.9 ± 0.2      | 0.6 ± 0.1       |
| Mean size of M_23C_6, nm | 75 ± 4            | 78 ± 4          | 85 ± 5           |
| Mean size of (Fe,Cr)_2(W,Mo), nm | –                | 184 ± 20       | 239 ± 20        |
| Mean size of MX, nm    | 23 ± 3            | 35 ± 2          | 39 ± 3           |

It should be noted that the MX carbonitrides formed in the studied steel are very stable against coarsening, and their mean size after 3,738 h of creep is less than 40 nm (Table 1, Figure 3). Increasing the dispersion of carbonitride particles in high-chromium martensitic steels with a low nitrogen content is particularly important, since it compensates the decreased volume fraction of VN particles.

Figure 3. Fine precipitates of (Nb,Ta)(C,N) in the studied steel after tempering at 780 °C (a) and after creep for 3738 h (b).

To determine the strengthening contribution from the MX particles in the as-tempered conditions, the well-known Orowan particle strengthening model was used [4]:

\[ \sigma_{Or} = 0,3 M G b / 2(1 + \nu) \lambda \]  \hspace{1cm} (1)

where \( \lambda _{i} \) – interparticle spacing, \( M \) - Taylor's factor (= 3), \( G \) - shear modulus (~59 GPa at 650 °C), \( \nu \) – Poisson's ratio, \( b \) - interatomic distance in slip direction (= 0,25 nm). The following equation correlates \( \lambda \) to the number density \( N_s \):

\[ \lambda_{MX} = 0,5 N_s^{-1/2} \]  \hspace{1cm} (2)

The number density \( N_s \) was calculated as

\[ N_s = \frac{3f_p}{2\pi r^2} \]  \hspace{1cm} (3)
where $f_v$ is the volume fraction and $r$ - mean radius of MX precipitates. The obtained value of strengthening from the MX carbonitrides particles in the studied steel was compared with that in steel P92 and 9\%Cr steel with high B and low N contents (Table 2).

Table 2. Nitrogen content, volume fraction, diameter and spacing of MX carbonitrides in 9\%Cr steels, together with Orowan stress estimated form the values of interparticle spacing.

|                        | Nitrogen content, wt.\% | $f_v$ of MX | Mean radius of MX, nm | $\lambda_{MX}$, nm | $\sigma_{Or}$, MPa |
|------------------------|-------------------------|-------------|-----------------------|---------------------|-------------------|
| P92 steel [4]          | 0.05                    | 0.00310     | 20                    | 260                 | 148               |
| 9\%Cr steel with high B and low N contents [6] | 0.007                   | 0.00086     | 17.5                  | 431                 | 89                |
| Studied Ta-alloyed 9\%Cr steel with high B and low N contents | 0.007                   | 0.00146     | 12.5                  | 237                 | 162               |

It can be seen that the estimated strengthening from MX particles for the studied Ta-alloyed steel is almost twice that in 9\%Cr steel with the same nitrogen content. This indicates that addition of Ta to high-chromium martensitic steels with a low N content is a promising approach to improve precipitation hardening due to fine MX carbonitrides particles.

4. Conclusions
The creep behavior and microstructure of advanced Ta-added 9\% Cr steel with high B and low N contents have been studied. The main results are as follows:

1. Fine precipitation of (Nb,Ta)(C,N) particles during tempering effectively decreases the minimum creep rate at the long-term creep regime.
2. Microstructural observations of the crept specimens showed additional precipitation of relatively coarse (Fe,Cr)$_2$(W,Mo) particles while M$_{23}$C$_6$ and MX particles exhibit a low coarsening rate during creep.
3. The Orowan strengthening model suggests a significant increase in precipitation hardening of Ta-alloyed steel due to the precipitation of fine (Nb,Ta)(C,N) particles.

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