

## IOP Conference Series: Materials Science and Engineering

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To cite this article: N Dudova *et al* 2019 *IOP Conf. Ser.: Mater. Sci. Eng.* **672** 012062

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# Effect of precipitation under long-term aging at 650 °C on the tensile strength of advanced 10%Cr heat-resistant steel

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**Abstract.** The effect of long-term aging for  $10^3$  to  $3.9 \cdot 10^4$  h at 650 °C on the microstructure, dispersion of secondary phases, and tensile strength properties was studied in a low-nitrogen 10%Cr martensitic steel with 3%Co and 0.008%B additives. Tensile tests of small specimens cut from grip portions of creep tested specimens were carried out at ambient and elevated (650 °C) temperatures. An increase in the yield stress and ultimate tensile strength after 10,000 h of aging was revealed to be associated with the precipitation of V-rich MX carbonitrides, which compensated for the coarsening of the boundary particles and depletion of W and Mo solutes from the ferritic matrix. The effect of changes of the microstructure and dispersion of secondary phases on strengthening of the steel is discussed.

## 1. Introduction

High-Cr ferritic/martensitic steels remain the major part of the materials used for components of fossil power plants operable at ultra-supercritical steam parameters [1]. This fact determines further improvement of their creep strength. The main factor providing high creep resistant of these steels is the stability of the tempered martensite lath structure (TMLS), which consists of prior austenite grains, packets, blocks and laths with a high dislocation density in the lath interiors [2]. The dispersion strengthening of these steels is ensured mainly, by nanoscale boundary  $M_{23}C_6$ -type carbides and MX carbonitrides homogeneously distributed in the laths.

The 9-11%Cr martensitic steels recently developed in accordance with the new approach by the increasing the B content to approximately 0.01wt% and decreasing the N content to approximately thousandths of a percent, demonstrate high creep resistant at an elevated temperature of 650 °C [3,4]. The role of precipitates of secondary phases ( $M_{23}C_6$ , MX, Laves phase) in the high stability of TMLS under creep condition is the subject of research interest.

The aim of this paper is to examine the effect of long-term aging at 650 °C on the tensile strength of advanced 10% Cr steel with low N and high B contents. Specific attention will be paid to the effect of changes in the microstructure and dispersion of the secondary phases on the strength.

## 2. Experimental

A 10% Cr steel with the following chemical composition (in wt.%) 0.1C, 0.06Si, 0.1Mn, 10.0Cr, 0.17Ni, 0.7Mo, 0.05Nb, 0.2V, 0.003N, 0.008B, 2.0W, 3.0Co, 0.002Ti, 0.006Cu, 0.01Al and Fe-balance was examined. The vacuum induction-melted steel was subsequently hot-forged by Lasmets, Chelyabinsk. The steel was subjected to standard heat treatment: normalization at 1060 °C/30 min and tempering at 770 °C/3 h. Small specimens for tensile tests were cut from the grip portions of creep



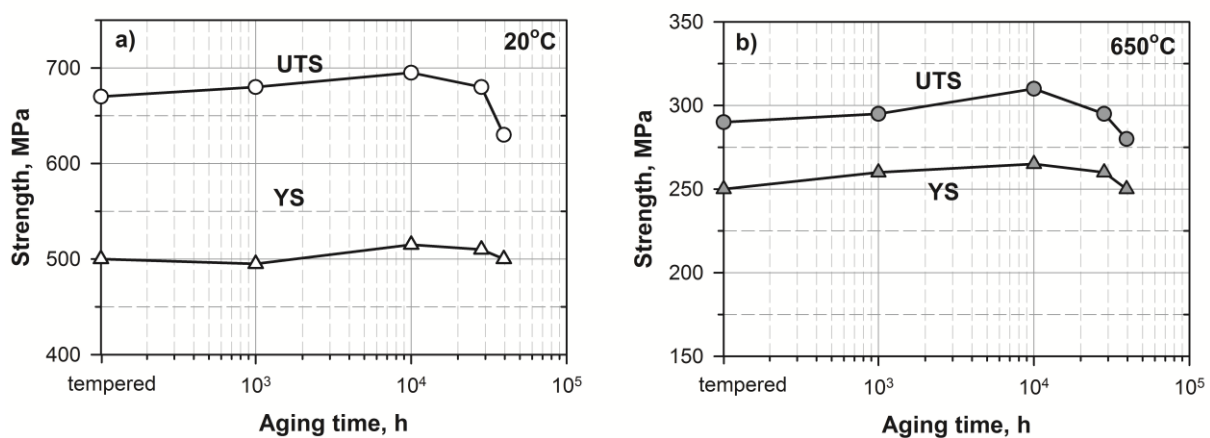
tested specimens at 650 °C, i.e. they were subjected only to long-term thermal aging for 1000, 10000, 28286 and 39437 hours [4]. Tensile tests were carried out using flat specimens with a gauge length of 4 mm and cross-sectional dimensions of 1 mm x 1 mm using an Instron 5882 testing machine at ambient temperature and a temperature of 650 °C and a strain rate of  $\sim 10^{-3} \text{ s}^{-1}$ .

The structural characterization was performed using a Jeol JEM-2100 transmission electron microscope (TEM) with an INCA energy dispersive X-ray spectrometer. The transverse lath/subgrain sizes were measured on TEM micrographs by the linear intercept method, counting all clear visible (sub)boundaries. The dislocation densities were estimated by counting individual dislocations in the (sub)grain/lath interiors per unit area on at least six arbitrarily selected typical TEM images for each data point.

### 3. Results and discussion

#### 3.1. Effect of long-term aging on the tensile properties

Figure 1 shows the effect of long-term aging at 650 °C on the tensile properties of the 10%Cr steel: the yield stress (YS) and ultimate tensile strength (UTS) at 20 and 650 °C. It has been revealed that aging for a long time (up to  $\sim 3 \cdot 10^4$  h) does not lead to a decrease in YS and UTS. Moreover, the slight increase in the YS and UTS values occurs. The maximal increase in the strength of +3...6% is observed after 10,000 hours of aging. The subsequent aging (39,437 hours) results in an insignificant decrease in UTS by 4...6% as compared with tempered condition, while the YS values return to the level of the tempered condition. Therefore, the 10%Cr steel obviously demonstrates higher tensile strength characteristics both at ambient and elevated temperatures after aging at 650 °C for 10,000 h.



**Figure 1.** Effect of long-term aging at 650 °C on the yield stress (YS) and ultimate tensile strength (UTS) at 20 °C (a) and 650 °C (b).

#### 3.2. Evolution of microstructure and dispersion of the secondary phases during long-term aging

The initial microstructure of the 10%Cr steel in the tempered condition is a typical tempered martensite lath structure with the parameters presented in table 1 [4]. The studied steel is characterized by a smaller size of  $M_{23}C_6$  carbides (70 nm) and their higher volume fraction (2.05%) in contrast to conventional high-chromium steels with standard N and B contents. Also, a peculiarity of the tempered 10%Cr steel is the absence of V-rich MX carbonitrides in contrast to conventional steels.

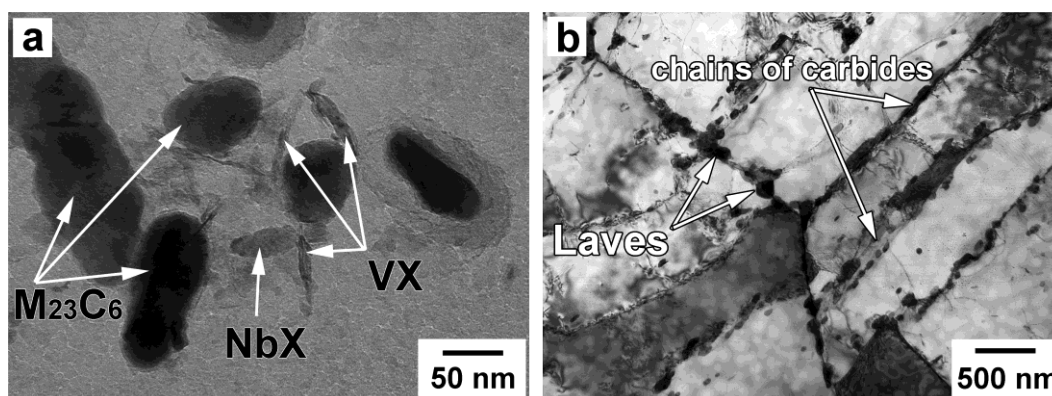
The lath structure of the steel remains almost unchanged during long-term aging. A 60% increase in the lath width and a 4-fold decrease in the dislocation density is observed after  $\sim 40,000$  hours of aging (table 1).

**Table 1.** Parameters of the structure of the 10%Cr steel before and after long-term aging at 650 °C.

| Structural parameters                                     | tempered | After aging at 650 °C |          |          |          |
|---|----------|-----------------------|----------|----------|----------|
|   |          | 1000 h                | 10,000 h | 28,286 h | 39,437 h |
| Lath width (nm)   | 380      | 409                   | 507      | 566      | 614      |
| Dislocation density, $\times 10^{14}$ ( $\text{m}^{-2}$ ) | 1.7      | 1.23                  | 1.06     | 0.61     | 0.43     |
| Mean size of particles:                                   |          |                       |          |          |          |
| $\text{M}_{23}\text{C}_6$ (nm)                            | 70       | 72                    | 81.9     | 83.6     | 96       |
| Nb-rich MX (nm)   | 35       | 35                    | 31.3     | 31.6     | 35       |
| V-rich MX (nm)  | -        | -                     | 26.5     | 40       | 58       |
| Laves phase (nm)  | -        | 145.6                 | 197.6    | 298.8    | 319      |
| W content in the solid solution (wt.%)                    | 2        | 1.1                   | 0.95     | 0.85     | 0.8      |

The precipitation processes occurring in the steel during long-term aging are as follows:

- 1) an insignificant coarsening of  $\text{M}_{23}\text{C}_6$  carbides from 70 nm to 96 nm after  $\sim 40,000$  hours.  $\text{M}_{23}\text{C}_6$  carbides demonstrate a high resistance to coarsening under aging condition (figure 2a);
- 2) the precipitation of the Laves phase particles ( $\text{Fe}_2(\text{W},\text{Mo})$ ) and their growth, which leads to the depletion of W and Mo from the solid solution. The Laves phase particles precipitate at the lath boundaries and high-angle boundaries of the blocks, packets, and PAGs (figure 2b). This process is accompanied by the depletion of W and Mo from the ferritic matrix [4]. The onset of the Laves phase coarsening appears after  $10^3$  h and accelerates after  $10^4$  h;
- 3) as it was revealed [4], during aging for the time between 1000 and 10,000 hours, fine V-rich MX carbonitrides precipitate in the lath interiors (figure 2a). The mean size of particles is 26.5 nm. The following aging for 28,286 and 39,437 hours results in an increase in the mean size to 40 and 58 nm, respectively. Their volume fraction is small and comprises 0.00862% (as calculated by Thermo-Calc). Aging for  $\sim 40,000$  hours does not lead to MX $\rightarrow$ Z-phase (CrVN) transformation due to the low content of N.



**Figure 2.** TEM micrographs of the 10%Cr steel after long-term aging: a) precipitation of V-rich MX particles after aging for 10,000 hours (replica); b) retained lath structure after aging for 39,437 hours (foil).

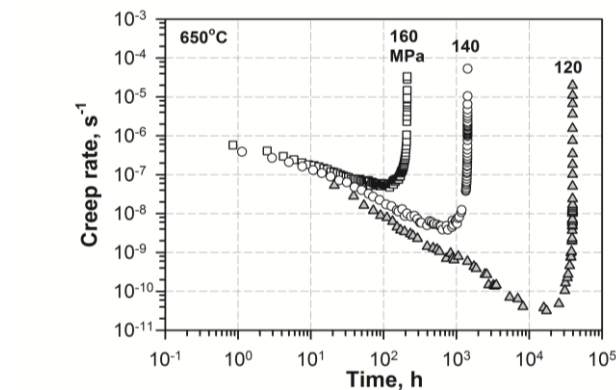
### 3.3. Effect of precipitation on the tensile strength

Usually, a recovery of non-equilibrium tempered martensite lath structure occurs during long-term aging at high temperature that results in the softening of high-Cr martensitic/ferritic steels [5,6]. In contrast, in the studied 10%Cr steel, an obvious stabilization of the tensile strength after long-term aging and a slight strengthening after 10,000 hours of aging is observed.

Recovery of the lath structure of the 10%Cr steel under aging condition leads to the lath widening, a decrease in the dislocation density and the depletion of W and Mo from the solid solution. These processes reduce the dislocation, substructure and solid solution strengthening of the steel. On the

other hand, precipitation hardening is affected by precipitation of the Laves phase particles and their coarsening, precipitation of fine V-rich MX carbonitrides, and retention of stable against the coarsening  $M_{23}C_6$  carbides. The strength of the aged 10%Cr steel retaining at a high level corresponding to that for the tempered condition is indicative of the fact that dispersion strengthening compensated for the lath widening, decrease in the dislocation density and the depletion of the ferritic matrix.

The strengthening after 10,000 hours of exposure seems to be associated with the precipitation of fine V-rich MX carbonitrides, since the precipitated Laves phase particles enlarged by  $\sim 35\%$  during 1000...10,000 hours of aging and did not increase the dispersion strengthening. The strengthening due to V-rich MX phase can explain why the creep rate significantly decreases to  $\sim 10^{-11} \text{ s}^{-1}$  at 120 MPa providing a high rupture time of  $\sim 40,000$  hours for the 10%Cr steel [4] (figure 3).



**Figure 3.** Creep rate vs time curves of the 10%Cr steel at 650 °C.

#### 4. Conclusion

The effect of long-term aging at 650 °C on the tensile strength and the changes in the microstructure and precipitations of secondary phases were studied in advanced 10% Cr martensitic steel with 0.008% B and 0.003% N. The main results can be summarized as follows:

1. Aging for 1000... $\sim 30,000$  hours leads to a slight increase in the yield stress and ultimate tensile strength, reaching the maximum values (+3...6%) at 10,000 hours of aging, whereas subsequent aging for  $\sim 40,000$  hours results in the 4...6% lower ultimate tensile strength as compared with the tempered condition.
2. Strengthening of the steel after 10,000 hours of exposure is associated with the precipitation of fine V-rich MX carbonitrides in the lath interiors.

#### Acknowledgments

One of the authors (N.D.) greatly acknowledges the joint support by DAAD (Deutscher Akademischer Austausch Dienst) under Grant No. 57447934 and the Ministry of Education and Science of Russian Federation under Grant No. 11.13447.2019/13.2 in the Michael Lomonosov Programm. The authors are grateful to the staff of the Joint Research Center, "Technology and Materials", Belgorod State University, for providing the equipment for instrumental analysis.

#### References

- [1] Abe F, Kern T-U and Viswanathan R 2008 *Creep-Resistant Steels* (Cambridge: Woodhead Publishing)
- [2] Hald J 2008 *Int. J. Press. Vess. Piping* **85** 30
- [3] Abe F 2011 *Procedia Engineering* **10** 94
- [4] Mishnev R, Dudova N and Kaibyshev R 2018 *Mater. Sci. Eng. A* **713** 161
- [5] Sahoo K C, Vanaja J, Parameswaran P, Vijayanand V D and Laha K 2017 *Mater. Sci. Eng. A* **686** 54
- [6] Xu Y, Nie Y, Wang M, Li W and Jin X 2017 *Acta Mater.* **131** 110