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# Evolution of strengthening factors during long-term aging at 650 °C in advanced 10% Cr heat-resistant steel

N Dudova\*, R Mishnev, R Kaibyshev

Belgorod State National Research University, Pobeda, 85, Belgorod, 308015, Russia

\*Corresponding author: dudova@bsu.edu.ru

**Abstract.** The effect of long-term aging for 1000...~40,000 h at 650 °C on the tensile strength at ambient temperature was studied in a low-nitrogen and high-boron 10%Cr martensitic steel. In order to establish the reason for increasing the yield stress and ultimate tensile strength after 10,000 h of aging, the evolution of strengthening factors was analyzed. A decrease in the substructure and solid solution strengthening during long-term aging is compensated by an increase in the dispersion strengthening due to the precipitation of V-rich MX carbonitrides.

## 1. Introduction

New generation fossil power plants with higher thermal efficiency and reduced emission of carbon dioxide require the heat-resistant materials that operate at higher temperatures. The most heated components of the power units will be made of austenitic steels and nickel-based superalloys. Nevertheless, the 9-12% Cr martensitic steels will be the base materials due to their excellent combination of high creep strength, good fatigue and oxidation resistance, and low cost [1]. One of the advanced approaches to improve the 100,000 h creep strength up to a minimum value of 100 MPa at 650 °C is a steel alloying modification by increasing the B content and decreasing the N content [2,3].

The creep resistance of these steels is associated with the stability of non-equilibrium structure, which is called the tempered martensite lath structure (TMLS). The complex TMLS consists of prior austenite grains, packets, blocks and laths with a high dislocation density in the lath interiors [2,3]. The main dispersion strengthening phases are nanoscale boundary  $M_{23}C_6$ -type carbides and MX carbonitrides homogeneously distributed in the laths. 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. It was recently revealed that long-term aging led to a slight increase in the yield strength and ultimate tensile strength [4]. The aim of this work is to examine the effect of long-term aging at 650 °C on the evolution of strengthening factors in advanced 10% Cr steel with low N and high B contents at ambient temperature.

## 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

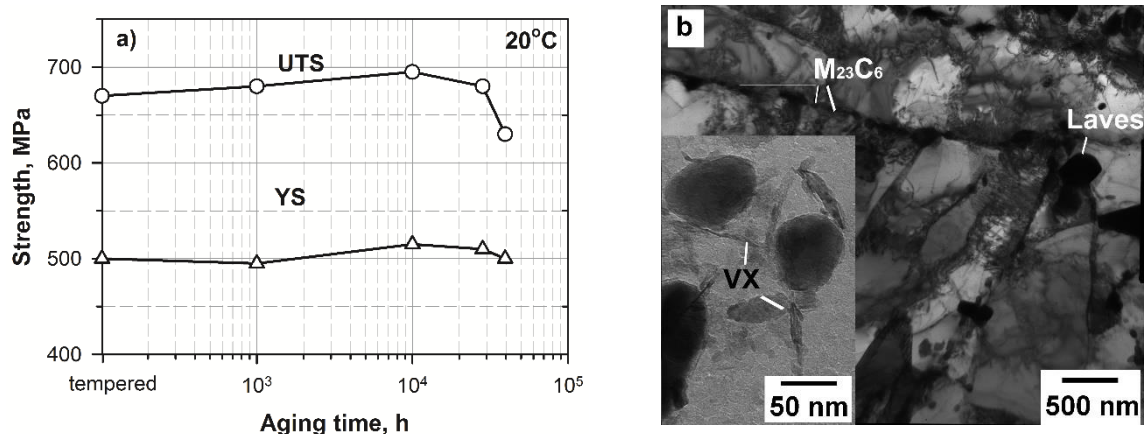


at 650 °C, subjected only to long-term thermal aging for 1000; 10,000; 28,286 and 39,437 hours [4]. Tensile tests were carried out using flat specimens with a gauge length of 4 mm and cross-sectional dimensions of 1 mm x 1 mm using an Instron 5882 testing machine at ambient temperature and a strain rate 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 widths were measured on TEM micrographs by the linear intercept method. 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. Tensile strength and microstructure after long-term aging

Aging of the 10% Cr steel at 650 °C for 1000...~30,000 h leads to a slight increase in the yield strength (YS) and ultimate tensile strength (UTS) (Figure 1a). Maximum rise of YS and UTS (+3...6%) to 520 and 695 MPa, respectively, occurs for 10,000 h aged steel. A ~40,000 h aging results in the UTS of 650 MPa that is 4...6% lower as compared with the tempered condition, whereas the YS returns to the initial level of 500 MPa. Therefore, the 10%Cr steel obviously demonstrates higher tensile strength characteristics after aging at 650 °C for 10,000 h.



**Figure 1.** Effect of long-term aging at 650 °C on the YS and UTS at 20 °C (a). Microstructure of the 10% Cr steel after long-term aging for 10,000 h at 650 °C (b).

The TMLS of the 10% Cr steel is stable under long-term aging condition at 650 °C (Figure 1b) and does not transform into the subgrain structure up to ~40,000 h. Lath width increases less than 2 times (Table 1). Dislocation density in the lath interiors continuously decreases by 4 times.

Secondary phases in the tempered steel are presented by nanoscale  $M_{23}C_6$  carbides (70 nm) located on the boundaries and fine Nb-rich MX carbonitrides (30 nm) homogeneously distributed in the laths. Both  $M_{23}C_6$  and MX precipitates are resistant to coarsening. An insignificant coarsening of  $M_{23}C_6$  carbides from 70 nm to 96 nm after ~40,000 h occurs. Nb-rich MX are highly stable.

Aging leads to Laves phase ( $Fe_2(W,Mo)$ ) precipitation at the boundaries (Figure 1b). Consequently, depletion of W and Mo from the ferritic matrix occurs (Table 1). The onset of the Laves phase coarsening appears after 1000 h and accelerates after 10,000 h.

Between 1000 and 10,000 h of aging, fine V-rich MX carbonitrides precipitate in the lath interiors (Figure 1a). The mean size of these particles estimated by TEM observation of carbon replicas is 26.5 nm. The volume fraction of V-rich MX phase is 0.00862% as calculated by Thermo-Calc. However, structure observations reveals approximately 0.1% of V-rich MX particles.

**Table 1.** Change in the structural parameters of the 10% Cr steel during long-term aging at 650 °C.

Structural parameters	Duration of aging (h)				
	0	1000	10,000	28,286	39,437
Dislocation density, $\times 10^{14}$ ( $m^{-2}$ )	1.70	1.23	1.06	0.61	0.43
Lath width ( $\mu m$ )	0.38	0.409	0.507	0.566	0.614
Concentration of element in the matrix (at. fraction):					
Cr	0.109	0.105	0.104	0.103	0.1004
W	0.0062	0.0034	0.003	0.0026	0.0024
Mo	0.004	0.0034	0.0029	0.0024	0.0018
Co	0.0304	0.0304	0.0304	0.0304	0.0304
Mean size of particles (nm) <sup>a</sup> :					
M <sub>23</sub> C <sub>6</sub>	70	72	81.9	83.6	96
Laves phase	-	145.6	197.6	298.8	319
NbX	30	35	31.3	31.6	35
VX	-	-	26.5	40	58

<sup>a</sup>The volume fraction of M<sub>23</sub>C<sub>6</sub> is 2.048 /2.107%, Laves – 0/1.6%, NbX – 0.05/0.06%, VX – 0/0.1% (before / after aging, respectively.)

### 3.2. Strengthening factors

To reveal the reason for the increase in the steel's strength after long-term aging for 10,000 h, the strengthening factors were analyzed. Estimation was carried out in accordance with a model describing the YS of high-chromium steels [5,6]. The YS is presented as:

$$\sigma_Y = M \sqrt{(\tau_A^2 + \tau_B^2)} \quad (1)$$

where  $M$  is the Taylor factor (2.9),  $\tau_A$  is the strengthening from dislocations,  $\tau_B$  is the strengthening from obstacles.

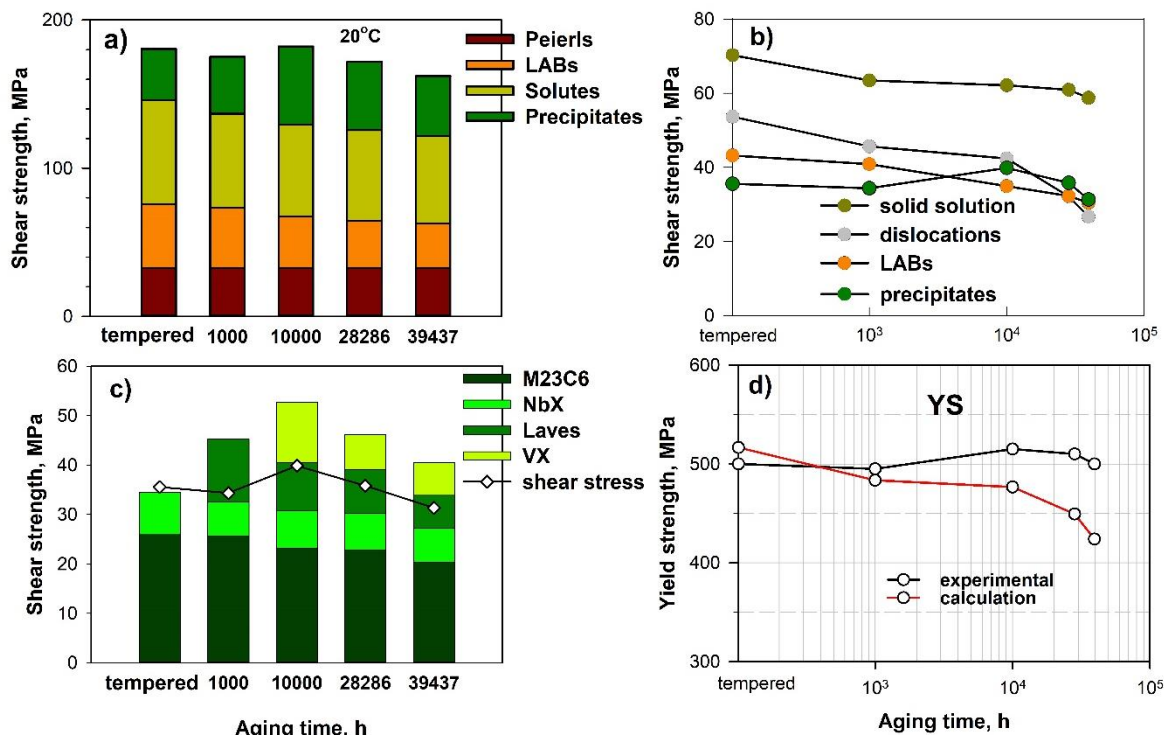
Dislocation strengthening is determined as:  $\tau_A = \alpha_1 \mu b \sqrt{\rho}$ , where  $\alpha_1$  is a constant (0.2);  $\mu$  is the temperature depending shear modulus (at 20 °C it is 83 GPa);  $b$  is the Burgers vector ( $2.48 \cdot 10^{-10}$  m);  $\rho$  is the dislocation density.

Strengthening from the obstacles can be defined as a sum of the strengthening from Peierls-Nabarro barriers ( $\tau_{PN}$ ), low angle boundaries ( $\tau_{BD}$ ), solute atoms ( $\tau_{SS}$ ) and dispersion particles ( $\tau_{prec}$ ) estimated according to Ref. [5,6]:  $\tau_B = \tau_{PN} + \tau_{BD} + \tau_{SS} + \tau_{prec}$ . Dispersion strengthening is determined by the Orowan-Ashbi model as [5,7]:  $\tau_{prec} = 0.045(\mu b / \lambda) \ln(r/b)$ , where  $\lambda$  is the mean distance between particles,  $r$  is the mean radius of particles.

The estimated strengthening factors are shown in Figure 2 (a-c). The evolution of strengthening during long-term aging can be described as follows:

- the dislocation strengthening continuously decreases with aging time, however, in the range of 1000...10,000 h the intensity of decreasing is the smallest;
- the solid solution strengthening by substitutional atoms of Cr, W, Mo, and Co is slightly reduced due to precipitation of W- and Mo-rich particles of Laves phase;
- the substructural strengthening by low angle boundaries of martensitic laths is continuously decreased starting from 1000 h of aging due to the lath widening;
- the dispersion strengthening during aging for 1000...10,000 h is increased on 16% as compared with tempered condition. Although the strengthening from M<sub>23</sub>C<sub>6</sub> and Laves phases is reduced, precipitation of V-rich MX particles compensates for this decrease.

The calculated values of YS (Figure 2d) remain stable between 1000 and 10,000 h of aging and are close to the experimental YS. This fact is resulted from the increasing precipitation strengthening.



**Figure 2.** Change in the strengthening factors (a-c) during long-term aging at 650 °C of the 10% Cr steel. Change in the calculated YS at 20 °C after aging in comparison with experimental data (d).

#### 4. Conclusion

The stabilization of the calculated yield strength of the low-nitrogen and high-boron 10% Cr steel is revealed after 1000...10,000 h aging at 650 °C that is attributed to an increase in dispersion strengthening. Precipitation of fine V-rich MX carbonitrides during aging for 1000...10,000 h can contribute to the strengthening of this steel.

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