

EFFECT OF AUSTENIZATION TEMPERATURE ON CREEP RESISTANCE OF STEEL 10Kh9V2MFBR¹

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The effect of austenization temperature on the microstructure and on the time to failure due to creep of steel 10Kh9V2MFBR is studied. The microstructure and the mechanical properties of the metal are determined after tempering at 730°C preceded by normalizing from 1050 and 1150°C. It is shown that in order to attain the highest creep resistance the austenization temperature of steel 10Kh9V2MFBR should not exceed 1050°C.

INTRODUCTION

High-chromium steels of martensitic class have been used as structural materials in aircraft engines and stationary power plants from the middle of the last century. Intense study of the physical metallurgy of these steels performed in the last 25 years in the USA, Japan, and countries of the European Community has made it possible to start commercial production of a new generation of these materials with operating temperatures 70–100°C higher than those of steels ÉI961, ÉP517, ÉP505, etc. [1–4]. The creep resistance of new-generation high-chromium steels is determined by precipitation hardening due to uniformly distributed carbides of type MeC with a size of less than 100 nm [3–7]. In addition, these dispersed carbides hinder effectively the motion of dislocations and thus prevent the development of cell formation and recrystallization in the dislocation structure [3–6]. This combination of precipitation hardening and strengthening due to the dislocation structure of troostomartensite allows new-generation high-chromium steels to serve at higher temperatures as compared to their predecessors. The efficiency of the precipitation hardening depends on the size of the particles and their specific volume and distribution [7]. These parameters can be controlled by varying the modes of heat treatment.

Standard heat treatment of these steels includes normalizing and subsequent medium-temperature tempering, which is still called high-temperature one by analogy with the steels of the previous generation despite the fact that in new-generation steels such tempering does not result in cell formation, to say nothing of recrystallization of the dislocation structure of martensite [6–9]. Standard tempering (a hold at 730–780°C for 3 h or for a shorter time) causes decomposition of martensite and lowers the dislocation density by a factor of 1.5–2 [6–9], yielding the structure of troostomartensite. Data on the effect of the temperature and of the duration of tempering on the microstructure and creep resistance of new-generation high-temperature high-chromium steels have been published [7–9]. At the same time, published sources contain no information on the effect of the austenization temperature on the microstructure and properties of the steels in question.

The aim of the present work consisted in determining the effect of austenization temperature on the parameters of microstructure and creep resistance of steel 10Kh9V2MFBR. In order to understand the nature of the effect of austenization temperature on the time to failure in creep, we analyze in detail the distribution of secondary phases after tempering, aging (in the heads of the specimens), and creep (in the functional parts of the specimens).

METHODS OF STUDY

We cut the specimens for the study from a hot-deformed steam pipe from steel 10Kh9V2MFBR (0.1% C, 0.17% Si, 0.54% Mn, 8.75% Cr, 0.21% Ni, 0.51% Mo, 1.60% W,

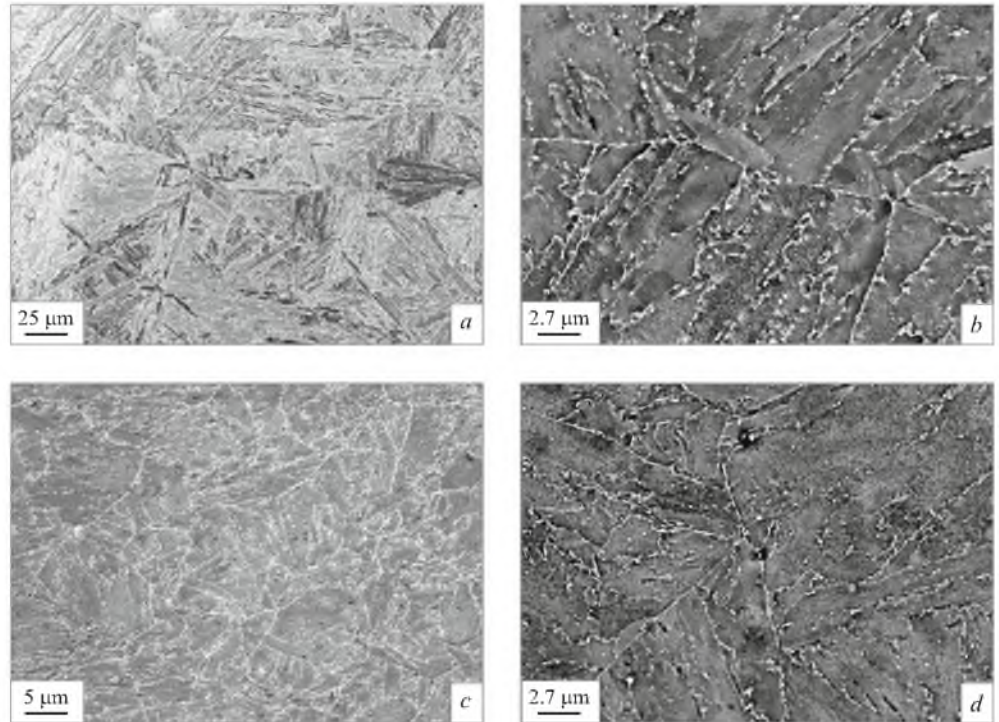


Fig. 1. Microstructure of troostomartensite in steel 10Kh9V2MFBR after normalizing from 1150°C (*a*) and 1050°C (*b*) and 3-h tempering at 730°C.

0.23% V, 0.07% Nb, remainder Fe) produced at the Chelyabinsk Tube-Rolling Plant. They were subjected to normalizing from 1050 and 1150°C and then to 3-h tempering at 730°C. Specimens with a diameter of 10 mm and a length of the functional part of 50 mm were tested for creep at 650°C at a stress of 118 MPa until failure in a lever-type machine designed at TsNIITMASH.

The specimens for analysis of structural changes after creep were cut near a neck in the longitudinal direction. The structure of the martensite was analyzed using a high-resolution FEI Quanta FEG scanning electron microscope with autoemission cathode. The microscope was equipped with an analyzer of diffraction of back-scattered electrons, a system for plotting off-orientation maps, and an EDAX system for microscopic energy spectrum analysis. The off-orientation maps were corrected with minimum reliability parameter of 0.1. The fine structure was analyzed using a JEOL JEM-2100 transmission electron microscope (TEM) with an attachment for energy dispersive microanalysis (Oxford Instruments). The foils for the TEM were prepared by the method of jet electropolishing using a solution of 10% perchloric acid in acetic acid in a Struers TenuPol-5 device. The sizes of subgrains were measured on TEM photographs by the method of random secants with allowance for all clearly seen (sub)boundaries. The dislocation density was determined by counting individual dislocations inside laths/subgrains on characteristic TEM images. The microhardness was measured at a load of 5 N using a Wolpert 402MVD digital microhardness meter.

RESULTS AND DISCUSSION

Microstructure of the Steel after Tempering

The microstructure of steel 10Kh9V2MFBR after normalizing and subsequent tempering is presented in Figs. 1 and 2. It can be seen that the austenization temperature affects the type of the formed martensite and its structural characteristics. After normalizing from 1150°C the austenite grains are comparatively large and have a size of about 80 μm; lath martensite is accompanied by lamellar martensite (Fig. 1*a*). The transverse size of martensite laths is about 340 nm, and the density of dislocations inside them is $4.3 \times 10^{14} \text{ m}^{-2}$ (Fig. 2*a*). The boundaries of laths and blocks are decorated by segregations of Me₂₃C₆ carbides with a mean size of about 136 nm (Fig. 3*a*). Fine particles of V(C, N) are homogeneously distributed inside laths. The size of dispersed carbonitrides is mostly 5–10 nm, though larger particles 20–30 nm in size are encountered. Martensitic transformation is accompanied by formation of special boundaries, the specific fraction of which amounts to 13%.

After normalizing from 1050°C the size of the initial austenite grains in the metal is 20 μm; virtually the whole of the martensite is of a lath type (Fig. 1*c*). Decrease in the size of austenite grains causes a decrease in the size of martensite packets and growth in the fraction of special block boundaries to 17%. The size of the martensite blocks is 330 nm, and the dislocation density inside them is $6.2 \times 10^{14} \text{ m}^{-2}$ (see Fig. 2). It should be noted that when the austenization temperature is lowered to 1050°C, the size of the Me₂₃C₆

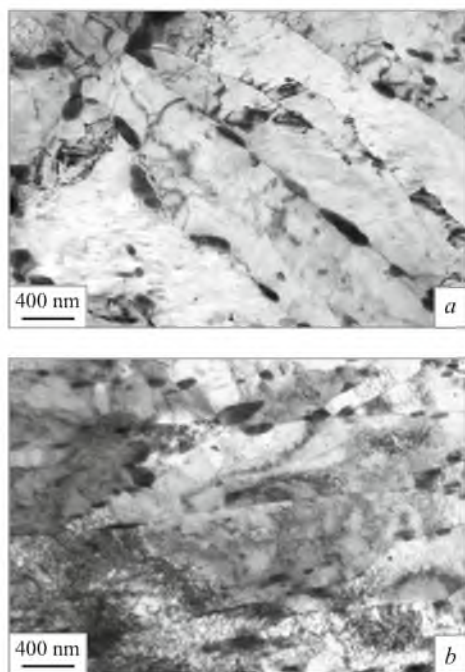


Fig. 2. Substructure of steel 10Kh9V2MFBR after normalizing from 1150°C (*a*) and 1050°C (*b*) and 3-h tempering at 730°C.

carbides segregated on the boundaries of initial austenite grains and on the boundaries of martensite packets decreases considerably. The mean size of the comparatively coarse Me_{23}C_6 segregations is about 85 nm (Fig. 3*b*), i.e., it is almost halved upon decrease in the austenization temperature. The distribution of these carbides becomes more uniform over the volume of grains. The austenization temperature does not affect the distribution of fine particles of type $\text{Me}(\text{C}, \text{N})$ inside martensite crystals. Thus, the decrease in the austenization temperature from 1150 to 1050°C results in a fourfold decrease in the size of the initial austenite grains and in segregation of finer Me_{23}C_6 carbides over boundaries of martensite crystals. This results in considerable increase in the density of segregations of second phases (the distance between the particles decreases) and may cause substantial growth in the creep resistance, especially in the time to failure, with allowance for the fact that the rate of formation of pores on the boundaries of former austenite grains near fine particles of Me_{23}C_6 carbides is substantially lower than near similar particles of greater size [6].

Tests for Long-Term Strength

The difference in the conditions of the prior heat treatment of the steel results in different creep resistance. The time to failure of specimens subjected to normalizing from 1150 and 1050°C is 513 and 1271 h, respectively. In order to determine the effect of plastic yielding on the softening in creep, we measured the hardness in the central part along the axis of specimens from the head to the neck at a step of 2 mm. The hardness in the head of a specimen (206 *HV*) de-

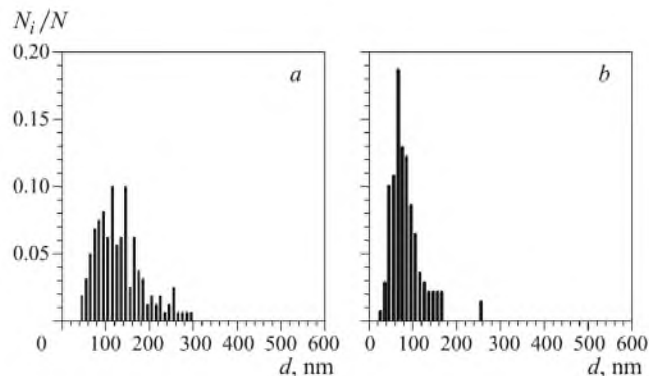


Fig. 3. Size (*d*) distribution of particles of second phases on boundaries in steel 10Kh9V2MFBR after normalizing from 1150°C (*a*) and 1050°C (*b*) and tempering at 730°C (N_i is the number of particles of the specified size range, N is the total number of particles): *a*) $d_{av} = 136$ nm; *b*) $d_{av} = 85$ nm.

creased smoothly (to 176 *HV*) in the functional part and increased to the same value (206 *HV*) in the neck. The decrease in the hardness in the functional part is connected with plastic flow in creep, i.e., with strain-induced softening. Plastic strain determined in terms of contraction in the corresponding part of the specimen is 30%. The strain hardening in the neck may be connected with localization of strain. The contraction in the region of the neck of this specimen attains 90%.

The hardness of the specimen after normalizing from 1050°C and tempering was 263 *HV*; in the process of creep it decreased to 235 *HV* in the head and to 176 *HV* close to the neck of the specimen. The specimen normalized from 1050°C underwent more uniform deformation along the functional part of the specimen; the strain (contraction) increased progressively from 0% in the head to 70% in the neck of the specimen. In other words, the localization of strain, which shortens considerably the time before failure in creep tests, was more intense in the specimens with coarse-grained structure.

A typical substructure formed in the region of a neck of a specimen after creep testing is presented in Fig. 4. The evolution of the structure in creep consists in transformation of the dislocation structure of troostomartensite into equiaxed subgrains, growth in the size of particles of secondary phases, and decrease in the density of free dislocations. In a specimen normalized from 1150°C the transverse size of subgrains and the dislocation density inside them were 930 nm and $1.4 \times 10^{14} \text{ m}^{-2}$, respectively; after normalizing from 1050°C, the average size of subgrains was 740 nm and the dislocation density was 10^{14} m^{-2} . Consequently, in specimens of steel 10Kh9V2MFBR subjected to normalizing from the lower temperature the size of subgrains was lower than after normalizing from the higher temperature. It is known [3–7] that it is the stability of the dislocation structure in creep that determines the time to failure of the new-generation high-chromium steels of martensitic class.

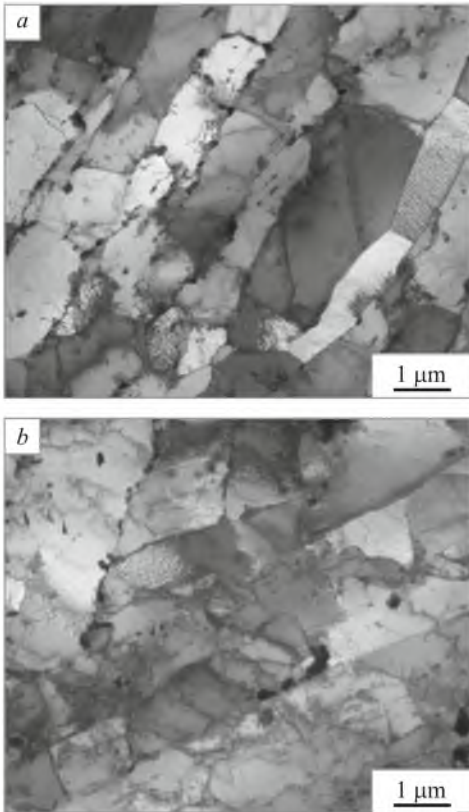


Fig. 4. Substructure in high-temperature steel 10Kh9V2MFBR after creep testing: *a*) $t_{\text{aust}} = 1150^{\circ}\text{C}$; *b*) $t_{\text{aust}} = 1050^{\circ}\text{C}$.

Growth of the components of substructure in specimens subjected to creep is accompanied by growth of carbides and segregation and growth of Laves phases. The size distribution of particles of secondary phases, which have been initially arranged over boundaries of blocks and laths, is presented in Fig. 5. In the specimen normalized from 1150°C the average size of the particles after creep testing is 146 nm, whereas after normalizing from 1050°C it is 211 nm. Analysis of this distribution shows that in the specimens normalized from the lower temperature the average size of the particles of secondary phases is larger due to the presence of coarse particles from 0.3 to $0.55\ \mu\text{m}$ in size. In steel 10Kh9V2MFBR only the $\text{Fe}_2(\text{W}, \text{Mo})$ Laves phases can grow to such a size [3, 4, 6]. It can be seen from Fig. 5 that in the specimens subjected to normalizing from 1050°C the amount and size of the Laves phases are substantially higher than after normalizing from 1150°C . The study of the nature of coarse particles of second phases in the specimens normalized from 1050°C after creep (Fig. 6) has shown that the decrease in the size of the initial austenite grains and in the thickness of the martensite crystals creates favorable conditions both for heterogeneous nucleation and for growth of particles of a $\text{Fe}_2(\text{W}, \text{Mo})$ Laves phase. All the particles identified as $\text{Fe}_2(\text{W}, \text{Mo})$ lie either over the boundaries of former austenite grains or over the boundaries martensite crystals

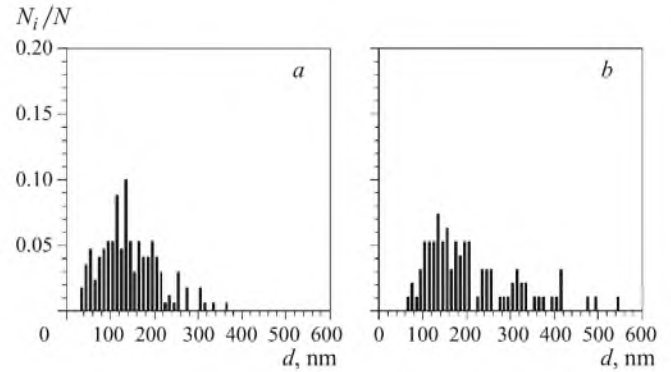


Fig. 5. Size distribution of comparatively large dispersed particles of second phases (d) in steel 10Kh8V2MFBR after creep testing (the neck of a specimen): *a*) $t_{\text{aust}} = 1150^{\circ}\text{C}$ + tempering; $d_{\text{av}} = 146\ \text{nm}$; *b*) $t_{\text{aust}} = 1050^{\circ}\text{C}$ + tempering, $d_{\text{av}} = 211\ \text{nm}$.

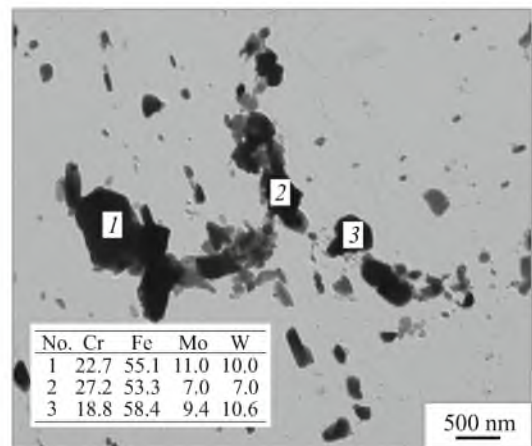


Fig. 6. Laves phases in steel 10Kh9V2MFBR normalized from 1050°C after creep testing (the mass percent of elements in phases 1, 2, 3 is given in the table).

(Fig. 6). Their segregation stabilizes the dislocation structure in creep [10].

In a specimen normalized from 1050°C the sizes of laths/subgrains and of the particles of secondary phases are obviously correlated. The proportion of the sizes of laths/subgrains before and after the creep tests is equal to $740/330$ and is almost the same as the proportion of the sizes of the particles, i.e., $211/85$. This confirms the fact that the growth of subgrains during deformation is controlled by the growth of particles in accordance with the Zener relation of stabilization of microstructure [11]. The variation of the dislocation density (ρ) during creep also matches the size of subgrains (d) in a well-known relation $1/d \sim \rho^{0.5}$. However, the growth in the size of subgrains exceeds substantially the growth in the size of particles in the specimen normalized from 1150°C . It seems that such unpredictable growth of martensite laths is connected with the comparatively fast localization of strain due to creep. The role of the dispersed

particles of secondary phases as stabilizers of structure of troostomartensite in the specimen normalized from 1150°C decreases because the increase in the austenization temperature causes growth of their size and violates the uniformity of their distribution over the volume of the material. The localization of strain provides an additional driving force for the migration of boundaries/subboundaries. Consequently, the resistance to creep drops rapidly upon growth of the initial austenite grains and coarsening of the dislocation structure of the troostomartensite, which leads to comparatively fast failure of the specimen.

We may conclude that the degree of homogeneity in the distribution of particles of secondary phases over boundaries of grains/subgrains in troostomartensite plays an important role in the elevation of the creep resistance of high-chromium martensitic steels of the new generation. A specimen with smaller initial austenite grains is characterized by a more homogeneous distribution of particles of secondary phases. As a result, the time to failure of this specimen more than doubles. In addition, the fraction of special boundaries more resistant to local migration than small-angle dislocation subboundaries and boundaries of a general type is higher in a specimen with smaller austenite grains, which also promotes increase in the long-term strength. Thus, the temperature of austenization of steel 10Kh9V2MFBR affects substantially its creep resistance. The results of our study show that when the size of the initial austenite grains in the steel is decreased and, consequently, the austenization temperature, the creep resistance increases due to more uniform distribution of the Me_{23}C_6 carbides, higher dislocation density, lower size of martensite crystals, and growth in the specific fraction of special boundaries.

CONCLUSIONS

1. The temperature of austenization of steel 10Kh9V2MFBR affects substantially its creep resistance. Decrease in the austenization temperature from 1150 to 1050°C more than doubles the time to failure of the steel in creep at a stress of 118 MPa at 650°C.

2. The growth in the creep resistance of steel 10Kh9V2MFBR after normalizing from 1050°C is connected with a more dispersed distribution of particles of secondary phases over the boundaries of blocks and laths of martensite and with a higher fraction of special boundaries in the structure of troostomartensite. This is explainable by the effect of the size of the initial austenite grains on the structural type of martensite formed during cooling and on the

processes of formation of carbides of type Me_{23}C_6 in subsequent tempering.

3. After normalizing from 1050°C and tempering at 730°C the size of the initial austenite grains in steel 10Kh9V2MFBR is $D = 20 \mu\text{m}$; the size of the secondary segregations $d = 85 \text{ nm}$; the fraction of special boundaries $\gamma_{\Sigma 3} = 17\%$; after normalizing from 1150°C and tempering $D = 80 \mu\text{m}$, $d = 140 \text{ nm}$, and $\gamma_{\Sigma 3} = 13\%$.

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REFERENCES

1. K. A. Lanskaya, *High-Chromium High-Temperature Steels* [in Russian], Metallurgiya, Moscow (1976).
2. F. Abe, M. Igarashi, S. Wanikawa, et al., "New ferritic heat resistant steels for 650°C USC boilers," in: *Proc. 3rd Int. Conf. Advances in Materials Technology for Fossil Power Plants, UK* (2001), pp. 79 – 89.
3. F. Abe, T. Horiuchi, M. Taneike, and K. Sawada, "Stabilization of martensitic microstructure in advanced 9Cr steel during creep at high temperature," *Mater. Sci. Eng. A*, **A378**, 299 – 303 (2004).
4. J. Hald, "Microstructure and long-term creep properties of 9 – 12% Cr steels," *Int. J. Press. Vess. Piping*, **85**, 30 – 37 (2008).
5. M. Taneike, F. Abe, and K. Sawada, "Creep-strengthening of steel at high temperatures using nano-sized carbonitride dispersions," *Nature*, **424**, 294 – 296.
6. R. O. Kaibyshev, V. N. Skorobogatykh, and I. A. Shchenkova, "New steels of martensitic class for the power industry. High-temperature properties," *Fiz. Met. Metalloved.*, **109**(2), 200 – 215 (2010).
7. H. Chilukuru, K. Durst, S. Wadekar, et al., "Coarsening of precipitates and degradation of creep resistance in tempered martensite steels," *Mater. Sci. Eng.*, **A510 – 511**, 81 – 87 (2009).
8. M. Tamura, Y. Haruguchi, and M. Yamashita, "Tempering behavior of 9% Cr – 1% Mo – 0.2% V steel," *ISIJ Int.*, **46**(11), 1693 – 1702 (2006).
9. K. Suzuki, S. Kumai, Y. Toda, et al., "Two-phase separation of primary MX carbonitride during tempering in creep resistant 9Cr1MoVNb steel," *ISIJ Int.*, **43**(7), 1089 – 1094 (2003).
10. F. Abe, "Effect of fine precipitation and subsequent coarsening of Fe_3W Laves phase on the creep deformation behavior of tempered martensitic 9Cr – W steels," *Metall. Mater. Trans.*, **36A**, 321 – 332 (2004).
11. P. A. Manohar, M. Ferry, and T. Chandra, "Five decades of the Zener equation," *ISIJ Int.*, **38**, 913 – 924 (1998).