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Microstructure and Mechanical Properties of Heat-Resistant 12% Cr Ferritic-Martensitic Steel EK-181 after Thermomechanical Treatment

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Abstract. The effect of high-temperature thermomechanical treatment (TMT) with the deformation in the austenitic region on the features of microstructure, phase transformations and mechanical properties of low-activation 12% Cr ferritic-martensitic steel EK-181 is investigated. It is established, that directly after thermomechanical treatment (without tempering) the sizes and density of V(CN) particles are comparable with those after a traditional heat treatment (air quenching and tempering at 720°C, 3 h), where these particles are formed only during tempering. It causes the increasing of the yield strength of the steel up to ≈ 1450 MPa at room temperature and up to ≈ 430 MPa at the test temperature $T = 650^\circ\text{C}$. The potential of microstructure modification by this treatment aimed at improving heat resistance of steel is discussed.

INTRODUCTION

The ferritic-martensitic steels (9–12% Cr) are the main candidates for applications as structural materials in the advanced nuclear and thermonuclear reactors. To extend the operating temperature range of these steels, one has to fulfill two requirements: to reduce the ductile-brittle transition temperature and to increase their high-temperature strength [1–5].

The potential of improving heat resistance of the Russian low-activation ferritic-martensitic steel EK-181 (RUSFER-EK-181) is associated with a possible control of its heterophase structure by heat or thermomechanical treatments.

Traditional heat treatment (THT) of steel EK-181 consists of air quenching from $T = 1100^\circ\text{C}$ (for 3 h) and tempering at $T = 720^\circ\text{C}$ (for 3 h). According to [1, 3, 4], this treatment leads to the formation of a ferritic-martensitic structure dispersion-strengthened by nanoscale (5–10 nm) particles of the MX type (M—V, X—C, N) and relatively coarse (diameter $d \approx 110$ nm) $M_{23}C_6$ carbides (M—Cr, Mn, Fe).

Our earlier investigations have shown that a thermo-mechanical treatment (TMT) of steel EK-181 with the deformation in the ferritic-martensitic range leads to a significant acceleration of tempering, an increase in the ferrite volume fraction and faster coagulation of carbide particles [5]. However, it is known [6] that one of the versions of thermomechanical treatments of steel is a TMT with the deformation in the austenitic region—high-temperature thermomechanical treatment. The potential promise of this method has been shown for many steels [6]. So far, it has not been applied to treat the reactor ferritic-martensitic steels, except for a few studies [7, 8] performed on 9%-chromium ferritic-martensitic steels.

TABLE 1. Elemental composition of steel EK-181 ([wt %], Fe-base)

C	Cr	Mn	Mo	Nb	V	W	Ni	N	Si	Ta	Ce	Ti	B	Zr
0.15	11.17	0.74	0.01	0.01	0.25	1.13	0.03	0.04	0.33	0.08	0.15	0.05	0.006	0.05

The main idea of the proposed high-temperature thermomechanical treatment is, firstly, to reduce the size of austenite grains and to increase the density of defects to form highly dispersed martensitic structure after quenching; secondly, to increase the efficiency of dispersion strengthening by forming a large volume fraction of nanoscale V(CN) particles.

According to the differential scanning calorimetry (DSC) data and *in-situ* X-ray analysis of steel EK-181, during rapid cooling (quenching) the martensitic transformation occurs at the temperatures below 400°C [9]. Diffusion-induced ferrite → austenite (austenite → ferrite) transformations are observed in the temperature range ≈850–950°C (890–910°C or slightly lower (≈570–600°C) at higher cooling rates) [9]. Thus, it is possible to carry out plastic deformation in the austenitic region (in supercooled austenite region) at the temperatures ≤1000°C, followed by rapid cooling to prevent the austenite → ferrite transformation. This paper presents the results of a high-temperature thermomechanical treatment application for steel EK-181.

TEST MATERIAL AND EXPERIMENTAL PROCEDURE

The elemental composition of the steel under study is presented in Table 1. Thermomechanical treatment consisted in heating to $T = 1000^\circ\text{C}$, holding for 40 min at this temperature, plastic deformation by rolling at 25% per pass (the rolling mill was at room temperature) followed by water quenching. The initial sample size was 50×20×4 mm. Some of the samples were tempered at 720°C (for 3 h) and 620°C (for 1 h) + 720°C (for 1 h). The efficiency of stepwise tempering in combination with the increased cooling rate (quenching in water) was shown elsewhere [3, 5].

Structural studies were performed using the Philips CM30 (300 kV) and Philips CM12 (120 kV) transmission electron microscopes. Thin foils for TEM examinations were prepared by electrolytic polishing in a solution of CrO₃ in H₃PO₄. Mechanical tests were carried out in a vacuum of $\approx 2 \times 10^{-3}$ Pa by the method of active tensile deformation at a strain rate of $\approx 2 \times 10^{-3}$ s⁻¹ using the proportional cylindrical samples with a diameter of the gage section of 3 mm and a length of 15 mm.

RESULTS

An electron microscopy examination revealed that a high temperature thermomechanical treatment leads to a multiple increase in the density of dislocations and carbonitride V(CN) nanoparticles pinned on them, compared to quenching from $T = 1100^\circ\text{C}$ (Figs. 1a and 1b). The average width of the martensitic lamella remained the same (Fig. 1c). Certain round-shaped M₂₃C₆ particles (Figs. 1c and 1d) and individual plates of M₃C carbides were observed.

After the thermomechanical treatment, the sizes and density of V(CN) particles were found to be comparable with those after a traditional heat treatment (air quenching (normalization) and tempering at 720°C, 3 h). The result of this treatment is partial autotempering of the martensitic structure followed by precipitation of V(CN) nanoparticles. Note that the density of M₂₃C₆ particles is comparable with that after quenching. Both after TMT and quenching, some plates of M₃C carbides are observed. Thus, the essential difference between the structural conditions after TMT and quenching is a high density of dislocations and nanoparticles of vanadium carbonitride formed on them. It was shown earlier [5] that a high density of stable V(CN) nanoparticles is an important factor of increasing the strength of ferritic-martensitic steels.

The electron-microscopy investigations have shown that a subsequent (after TMT) tempering at $T = 720^\circ\text{C}$ for 3 hours gives rise to the formation of a structural condition with lower density of coarse M₂₃C₆ carbides and their preferential precipitation inside the grains, in contrast to the traditional heat treatment. The density of V(CN) nanoparticles after TMT and tempering at $T = 720^\circ\text{C}$ (for 3 h) is comparable with that after the traditional heat treatment. These particles are mainly formed on dislocations, pinning the dislocation structure. The particle size of vanadium carbonitride, precipitating during quenching, does not change significantly during tempering at 720°C (for 3 h). In other words, no intensive particle coagulation occurs in the process of tempering after TMT.

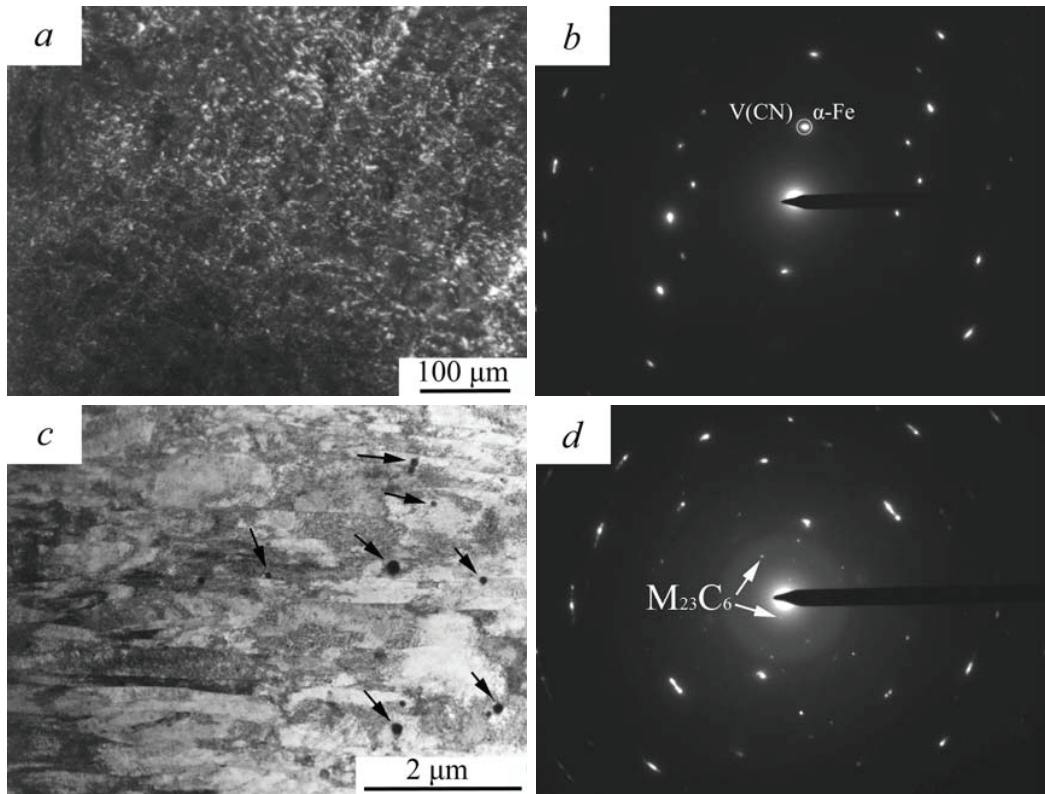


FIGURE 1. The microstructure of steel EK-181 after the high-temperature thermomechanical treatment: a, b—dark-field image of V(CN) nanoparticles and respective microdiffraction pattern (circle selects combined reflex of α -Fe + V(CN)); c, d—martensitic structure (arrows indicate $M_{23}C_6$ carbides) and respective microdiffraction pattern

During tempering after TMT, the density of nanoparticles responsible for the precipitation hardening remains the same. It should be noted that after TMT and tempering at 720°C (for 3h) the volume fraction of the tempered martensite in the steel structure becomes quite high.

In order to change the conditions of precipitation of carbides after TMT, in this study in addition to tempering at 720°C we carried out a stepwise tempering treatment at 620°C, 1h + 720°C. This tempering in combination with an increased quenching rate was reported to be highly efficient [3, 5]. Using the stepwise heat treatments, including stepwise tempering combined with a high-rate quenching (water quenching), it is possible to reduce the density of coarse $M_{23}C_6$ carbides and increase that of nanoscale V(CN) particles. Moreover, a high volume fraction of the tempered martensite is thus maintained [3, 5].

An electron microscopy examination of the steel microstructure after TMT and stepwise tempering at 620°C, 1 h + 720°C, 1h has shown that this tempering modifies its carbide subsystem. In particular, the density of coarse $M_{23}C_6$ precipitates becomes lower and their morphology and distribution in the matrix change.

The $M_{23}C_6$ carbides are, with the same probability, distributed both at the grain boundaries and inside the grains rather than primarily at the lamellae boundaries. After stepwise tempering, a large number of rounded particles appears, while after tempering at 720°C elongated particles are formed. In spite of qualitative and quantitative changes in the morphology of coarse carbides, the density and size of nanoparticles of vanadium carbonitride after TMT and stepwise tempering did not significantly change. It should also be noted that after this treatment a substantial volume fraction of the tempered martensite is observed.

The mechanical tests have shown that TMT with deformation in the austenitic region (with subsequent water quenching) increases the yield strength of the steel under study up to ≈ 1450 MPa at room temperature. The ductility

is decreased to $\approx 5.5\%$. At the test temperature $T = 650^\circ\text{C}$, the yield strength reaches about 430 MPa and the ductility is as high as $\approx 7\%$.

Tempering of steel at $T = 720^\circ\text{C}$ (for 3h) after TMT leads to a significant decrease in the yield strength to $\approx 760\text{--}780$ MPa at room temperature and the ductility is 5–7%. At the test temperature $T = 650^\circ\text{C}$, the yield strength reaches ≈ 300 MPa and the plasticity is about 15%.

Stepwise tempering of steel after TMT at 620°C , 1 h + 720°C , 1h was also observed to give rise to a decrease in the yield strength both at room temperature and at 650°C . In the former case, the yield stress is found to be about 775–810 MPa and the ductility is $\approx 6.6\text{--}7.4\%$. At the test temperature $T = 650^\circ\text{C}$, the yield stress was found to be $\approx 290\text{--}300$ MPa and the ductility $\approx 11.5\text{--}15\%$.

Thus, the highest yield strength values of steel EK-181 both at room temperature and at $T = 650^\circ\text{C}$ are observed after a high-temperature thermomechanical treatment. These values are however reduced during tempering and become comparable with or slightly lower than those in the case of traditional heat treatment ($\sigma_{0.1} = 827$ MPa, $\delta = 8\%$ at $T = 20^\circ\text{C}$; $\sigma_{0.1} = 390$ MPa, $\delta = 8\%$ at $T = 650^\circ\text{C}$) [5]. In our opinion, a reduction in strength may be caused by the structural-phase condition of the material after TMT and tempering. Despite a significant increase in the density of nanoscale V(CN) particles after TMT, there is no further increase in their density during tempering. Moreover, since the material after TMT has a higher level of internal stresses than after traditional quenching, this can cause acceleration of the tempering processes and result in faster structural changes within the same time periods (3 hours).

SUMMARY

In addition to the factors controlling precipitation hardening (size, density, volume fraction of particles), such factors as substructural hardening by large- and small-angle grain boundaries and by dislocation substructure have to be taken into consideration. The strength characteristics are affected by the relative content of the tempered martensite. The ratio between such structure components as tempered martensite and ferrite, whose mechanical properties differ, is determined by the mode of tempering. Thus, a change in the tempering mode can change the strength properties of steel after the thermomechanical treatment discussed in this work.

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