Grain-Boundary Ensembles in ЭК-181 Ferritic–Martensitic Chrome Steel

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Ferritic–martensitic chrome steel containing 9–12% Cr is widely used in the manufacture of high-temperature power-plant components such as boilers and turbines. The use of such steel in nuclear and thermonuclear engineering is very promising.

In Russia, readily activated ЭК-181 steel belongs to that class (12% Cr) [1]. To ensure that it is rapidly activated, elements such as molybdenum, niobium, and nickel are added [2]. Thanks to its production technology, this steel is highly resistant to low-temperature radiative embrittlement [3].

The production of specified mechanical properties is based on the creation of optimal microstructure in the steel or alloy. At high operating temperatures ($T \geq 0.4T_m$), diffusion-controlled processes such as recrystallization and the nucleation and growth of secondary phases are activated. Therefore, it is of interest to study the structure of ЭК-181 steel in terms of its stability with respect to such processes.

As we know, practically all stages of recrystallization are associated with the migration of large-angle boundaries. As shown by experimental data, there is a relation between the structure and migration rate of large-angle boundaries [4–8]. For example, in investigating boundaries of special type, lower rates of migration and maximum activation energy are observed in some conditions. Experimental data indicate that, when the mutual orientation of the crystals separated by the boundary ceases to be ideal, special boundaries do not immediately lose their specific properties, but retain them until the disorientation is about 5° [6]. Increase in the inverse density of coinciding points (Σ) is accompanied by decrease in the angular interval within which the special boundaries exist [9]. Another finding is that special boundaries are converted to boundaries of general type as the temperature rises. Only boundaries whose inverse density of coinciding points is three retain their properties until reaching the melting point [9]. Grains with 15°–45° disorientation are characterized by the greatest migration rates in bcc and fcc metals [10].

In addition, it is important to analyze the evolution of the grain-boundary ensemble in terms of the diffusional properties of the boundaries. We know that small-angle boundaries are characterized by lower diffusional permeability than for large-angle boundaries [11, 12]. Hence, the carbide phase deposited at small-angle boundaries will grow relatively little. On the other hand, the formation of small-angle boundaries in packet martensite is undesirable, since they are less stable in the pair coalescence of adjacent martensite crystals during high-temperature annealing [13, 14].

Thus, an important factor determining the structural stability of packet martensite in ferritic–martensitic steel during high-temperature annealing is the relative quantity of large- and small-angle boundaries. The proportion of special boundaries is also significant.

In the present work, we investigate ЭК-181 steel in the following states: after quenching from 1100°C (cooling in air); and after quenching from 1100°C (cooling in air) and 3-h tempering at 720°C.

Disk samples are cut from rod (diameter 12 mm) on an electrospark system. The surface of the sections is mechanically polished.

The microstructure is investigated by the diffraction of back-scattered electrons on a Quanta 600 FEG raster electron microscope, equipped with a Pegasus 2000 integrated system for microanalysis (a Sapphire X-ray detector with an ultrathin window, applicable to elements in the range from beryllium to uranium; energy resolution of the spectrometer 132 eV at the $K_{\alpha Mn}$ line). The crystallographic characteristics of the grains are determined at working magnifications in the range 200–80000.

Analysis of the data for the diffraction of back-scattered electrons (Figs. 1a and 1b) shows that the proportion of small-angle boundaries in the structure of martensite quenched from 1100°C is 39%, while the proportion of large-angle boundaries is 61%. Two maxima of the disorientation are seen in that case (Figs. 2a and 2b). Accordingly, we may distinguish

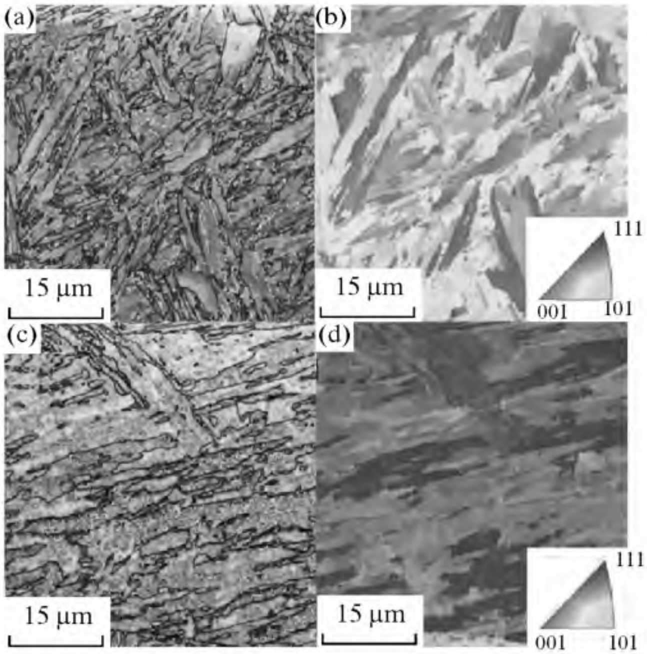

Fig. 1. Electron-microscope images of the structure of ЭK-181 steel after quenching from 1100°C (a) and 3-h tempering at 720°C (c) and the corresponding distributions of crystallographic orientations (b, d). The black and white lines correspond, respectively, to large-angle boundaries and small-angle boundaries.

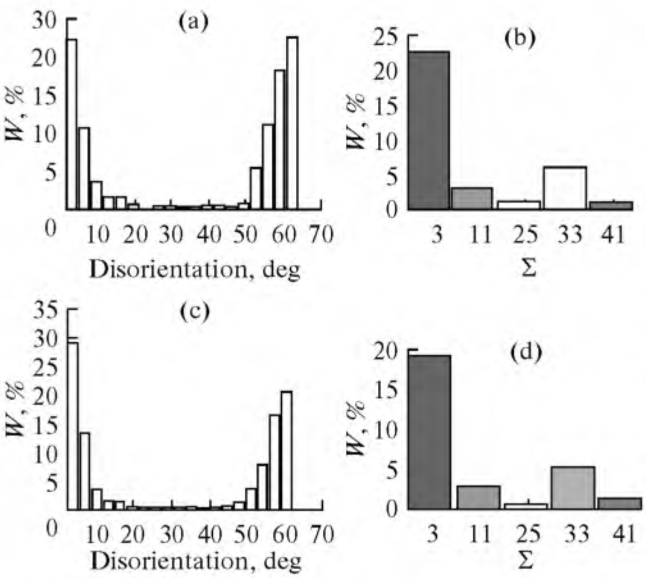

Fig. 2. Distribution of the disorientations (a, c) and proportion of special boundaries (b, d) as a function of the total number of boundaries in ЭK-181 steel after quenching from 1100°C (a, b) and after subsequent 3-h tempering at 720°C (c, d).

three groups: I) disorientation 1.5°–20.0°; II) disorientation 20°–45°; III) disorientation 45°–65°. On recrystallization, the large-angle boundaries in group II are characterized by large migration rates, according

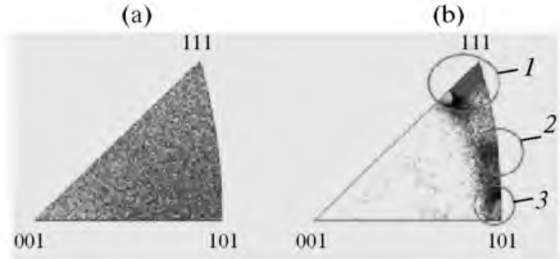

Fig. 3. Distribution of the disorientation axes in the standard stereographic triangle with 0°–15° (a) and 45°–65° (b) disorientation.

to [9]. In quenched ЭK-181 steel, the proportion of such boundaries is tiny.

The content of special boundaries is 34%; these are mainly twin boundaries (Fig. 2a).

In Fig. 3, we show the distribution of the disorientation axes in a standard stereographic triangle. Note that region *1* of the standard stereographic triangle corresponds to boundaries with $\Sigma = 3$ and 33; region *2* to $\Sigma = 11$, 33, and 41; and region *3* to $\Sigma = 25$.

In steel tempered at 720°C for 3 h, grains with a large density of small-angle boundaries are observed (Figs. 1c and 1d), as a result of fragmentation of the individual packet martensite crystals. That process is characterized by decrease in the dislocation density [12]. Specifically, the dislocations are aligned in dislocational walls (small-angle boundaries). If we consider the distribution of the disorientations in the tempered steel, we find that the proportion of small-angle boundaries increases to 47% (Fig. 2c).

Tempering slightly reduces the proportion of special boundaries (to 30%). There is no change in the proportions of boundaries with different Σ (Fig. 2d) nor in the distribution of the disorientation axes in the standard stereographic triangle.

Since the migration rate is less for special boundaries than for general boundaries, the retention of a large proportion of special boundaries in steel after regular heat treatment should help to suppress recrystallization.

After 3-h tempering at 720°C, the proportion of small-angle boundaries is doubled. On the one hand, these boundaries provide sites for the nucleation and deposition of secondary-phase particles, whose growth rate will be limited by the smaller diffusional permeability of small-angle boundaries (relative to large-angle boundaries). On the other hand, in high-temperature creep, small-angle boundaries tend to facilitate the enlargement of the structure by the coalescence of subgrains as a result of the migration of ternary junctions [14].

CONCLUSIONS

On the basis of data obtained by the diffraction of back-scattered electrons, we find that the ratio of large- and small-angle boundaries is 3 : 2 in quenched ЭК-181 steel. That corresponds to a high content of special boundaries (34%), which are mainly twin boundaries ($\Sigma = 3$). In tempering, we observe the fragmentation of the individual packet martensite crystals, with increase in the proportion of small-angle boundaries to 47%. Tempering slightly reduces the proportion of special boundaries (to 30%).

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