Analysis of the distribution of alloying elements in ultrafine-grained steel 05G2MFBT by atom-probe tomography

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Abstract. It is shown that in low carbon steel, the structure after the controlled rolling comprises grains of ferrite and carbides. The average grain size of ferrite was 2.5 µm, the carbide size varied from 1 to 5 µm. Additional warm rolling led to the formation of a fibrous ultrafine-grained (UFG) structure with an average grain/subgrain size about 0.3 µm, the carbide size did not exceed 100 nm. The mechanical tests carried out under the uniaxial tension scheme at room temperature showed that the ultimate strength after the controlled rolling is 600-700 MPa, with a relative elongation of 21% while the impact strength at room temperature is 3.15 MJ/m². In the state after warm rolling, the ultimate strength increased to 1000-1050 MPa, the elongation decreased from 21% to 17%, the impact strength at room temperature increased from 3.15 to 3.38 MJ/m². One of the important factors leading to an increase in the mechanical properties of alloys is solid-solution hardening. To confirm this, a tomographic atomic-probe analysis of steel samples in both structural states was carried out, which made it possible to detect an increase in the concentration of carbon in steel ferrite by a factor of two in the state after warm rolling, compared with the state after the controlled rolling.

1. Introduction
In materials subjected to deformation to high degrees, significant structural changes occur, accompanied not only by a sharp increase in the level of mechanical properties [1, 2], but, in some cases, by the manifestation of anomalies in the change of properties. In [3], for example, an anomalous temperature dependence of the toughness in rolled low-carbon steel was found. To understand the nature of such effects, traditional techniques for structure analysis, such as optical metallography, X-ray diffraction analysis, and electron microscope studies, are sometimes not sufficient and it is necessary to handle new high-resolution methods of analysis. One of such progressive techniques is the method of atom-probe tomography, which allows to carry out chemical analysis and evaluate local inhomogeneities at the atomic level.

In the present work, the microstructure and mechanical properties of pipe steel 05G2MFBT were studied after the controlled rolling with accelerated cooling and after additional warm rolling.
2. Experimental
Material of the study was low-carbon steel 05G2MFBT (Fe-1.61% Mn-0.03% Cu-0.192% Mo-0.26% Si-0.236% Cr-0.025% V-0.22% Ni-0.054% C-0.059% Nb-0.009% P-0.0012% S) in two different structural states. The first one was obtained by the controlled rolling at an accelerated cooling rate, and the second one was subjected additionally to plastic deformation by means of intensive warm rolling. Rolling was carried out at a temperature of 550 °C by several passes to the final cross-section of 10 mm x 10 mm rods with a relative narrowing of e = 10-15% in each pass. The accumulated degree of deformation was equal to e = 2.7.

Microstructure analysis was conducted using a Tescan Mira 3LMH scanning electron microscope (SEM) with a back scattered electron (BSE) detector. Tensile tests of flat samples were carried out on an "INSTRON-1185" universal dynamometer at room temperature with an initial strain rate of 10^-3 s^-1.

Impact tests of standard size specimens with a V-shaped notch according to GOST 9454 were carried out on an instrumented pendulum tester "Tinius Olsen IT542M" in the temperature range from +20 to -80 °C. Tomographic studies were performed on an atomic-probe tomography (APT) with laser evaporation (PAZL-3D), using the equipment of the CAMIX Center for Collective Use (http://kamiks.itep.ru/) of the Scientific Research Center "Kurchatov Institute" developed at ITEP [4].

3. Results and discussion
Structural studies of steel after the controlled rolling revealed a grained structure, the average grain size of ferrite was 2.5 µm, the carbide size varied from 1 to 5 µm (figure 1). Warm rolling led to the formation of a fibrous ultrafine-grained (UFG) structure with an average grain/subgrain size of 0.3 µm, while the carbide size did not exceed 100 nm (figures 2,3).

So, the toughness increased from 3.15 MJ/m² to 3.38 MJ/m². Mechanical tests carried out under the uniaxial tension scheme at room temperature showed that the ultimate strength after the controlled rolling is 600-700 MPa, with a relative elongation of 21%. After warm rolling, the strength properties increased to 1000-1050 MPa, while elongation decreased from 21% to 17%. The results of mechanical tests are shown in table 1.

![Figure 1. Microstructure of steel 05G2MFBT subjected to the controlled rolling with accelerated cooling](image-url)
Figure 2. Microstructure of steel 05G2MFBT subjected to warm rolling in transverse section.

Figure 3. Microstructure of steel 05G2MFBT subjected to warm rolling in longitudinal section.

Table 1. Mechanical characteristics of steel 05G2MFBT.

| Treatment conditions | $\sigma_{0.2}$, MPa | $\sigma_u$, MPa | $\delta$, % | KCV, MJ/m² |
|----------------------|---------------------|----------------|-------------|------------|
| Controlled rolling    | 612                 | 685            | 21          | 3.15       |
| Warm rolled           | 1017                | 1080           | 17          | 3.38       |

Tomographic analysis of low-carbon steel after two different deformation-thermal treatments showed that after the controlled rolling, the distribution of alloying elements in the ferrite matrix is uniform, while warm rolling resulted in carbides grinding, and an increase in the concentration of carbon by two times as compared to the state after the controlled rolling. There is also an increase in the concentration of manganese and nickel at the interface between the matrix of iron and carbides, while the distribution of chromium remains homogeneous (figure 4). It is important to note that if carbon and nickel in the particle are uniformly distributed, then the concentration of manganese increases twice exactly on the boundary by substituting iron, wherein in the center of the particle the content of manganese corresponds to its content in the matrix. The revealed differences in the supersaturation of the solid solution and the increase in the concentration of the alloying elements at the carbide boundary contribute to an increase in the strength properties and impact strength of steel 05G2MFBT after warm rolling compared to the state after the controlled rolling followed by accelerated cooling.
Figure 4. a) Representation of a three-dimensional reconstruction of the atomic map of UFG steel with distribution of Cr, Ni, Mn and C atoms in the investigated volume of steel 05G2MFB K65 subjected to warm rolling; b) linear concentration profiles along the direction of research.

4. Summary
As a result, one of the factors leading to an increase in the impact and tensile strength is an increase in the degree of supersaturation of the solid solution of steel ferrite and an increase in the concentration of the content of alloying elements on the boundary of carbides after deformation, as confirmed by atom-probe tomography.

References
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