Investigation of the fracture structure of a composite material after bending test by atomic force microscopy

V V Duka, L P Aref’eva, B I Mitrin, V N Pustovoit

Department of Physical and Applied Material Science, Don State Technical University, Rostov-on-Don, Russia

Abstract. The paper presents the results of a study of the surface fracture tested for bending at negative temperatures of a ferrite-martensite composite (FMC) obtained on the basis of K 02704 structural hypoeutectoid steel by atomic force microscopy. Metallographic studies of the sample were carried out on longitudinal sections. Investigations of microstructures were performed on optical microscopes Metam RV-22 and Neophot-21. Impact bending tests were carried out in accordance with ISO 148-1:2016. The quantitative assessment of martensite was carried out using the digital imaging program and amounted to 30%. A three-dimensional image of the fracture of the sample surface was obtained by atomic force microscopy (Nanoeducator II) in the semi-contact mode. An AFM study of the fracture surface showed that the data of optical and atomic force microscopy are in agreement. The three-dimensional AFM image of the surface fracture and its profiles, extracted in different areas, as well as the roughness parameters, show the presence of a dual line structure in steel.

1. Introduction
Improving quality and creating new materials intended for the manufacture of critical structures for various industries are the main tasks of modern production. Structural steels are of great importance in terms of their application. However, it is difficult to find steel that will work reliably in harsh operating conditions, since the requirements for the material are often contradictory. The solution to this problem is the use of a composite material.

The peculiarities of the line structure of the ferrite-martensite composite (FMC) make it possible to obtain higher indicators of cold brittleness, impact toughness, crack initiation and propagation work in comparison with the structural state of steel after complete quenching and tempering. This shows the feasibility of using FMC for high-loaded building and bridge structures, boilers and high-pressure pipes, rails, special-purpose guides, etc [1].

To date, one of the most promising methods for studying and diagnosing the surface of materials of different nature is scanning probe microscopy. The use of diagnostic AFM methods makes it possible to expand the amount of information obtained by obtaining a three-dimensional image of the surface relief, the formation of which is difficult by optical metallography. The advantage of AFM diagnostics is not only the study of the surface topology of steels, alloys and coatings, but also the measurement of physical parameters, the identification of phase components and structural features, investigation of microstructure evolution, to the quantification of nano-precipitates in thermo-mechanically treated microalloyed steels [3-13]. A number of works are aimed at studying the properties of stainless steels [14-18]. For example, in paper [14] the surface morphology of austenitic stainless steel has been investigated with the aim of understanding the passive film property in nitric acid medium by potentiodynamic polarization and electrochemical atomic force microscope.
The aim of this work was to carry out a comprehensive analysis of the FMC fracture using optical and atomic force microscopy.

To carry out a comprehensive analysis, the fracture surface and the smooth surface of the sample tested for bending at negative temperatures were compared by measuring the linear dimensions of the surface relief features. Surface features are defined as bulges or depressions, the extent of which in different directions of the lateral plane is proportional to each other [6].

2. Materials and methods

In this work, a study of the surface tested for bending by FMC, obtained on the basis of structural hypoeutectoid steel K 02704, was carried out. The chemical composition of the steel was determined using a Q8 MAGELLAN optical emission spectrometer (Table 1).

| Grade steel | Chemical compound, % (in mass) |
|-------------|--------------------------------|
|             | C   | Si   | Mn  | Ni  | Cr  | Cu  | S   | P   | others |
| K 02704     | 0.16| 0.32 | 1.53| 0.27| 0.21| 0.23| 0.038| 0.031| As≈0.06 |

The microstructure of steel in the hot-rolled state has a line ferrite-pearlite structure, which, after quenching in the intercritical (A1-A3) temperature range (ICI) and subsequent low tempering (Table 2), forms into a strictly oriented dual ferrite-martensitic structure, which is FMC. (Figure 1).

| quenching temperature, °C | cooling medium | tempering temperature, °C | Resulting structure |
|---------------------------|----------------|---------------------------|--------------------|
| 730                       | water          | 200                       | FMC                |

Metallographic studies of the sample were carried out on longitudinal sections. The preparation was completed on sanding paper with final polishing on a felt wheel using polishing paste (stearin, fine particles of chromium oxide). The sample was etched with 4% solution of nitric acid in ethyl alcohol. Viewing and recording of microstructures (Figure 1) was carried out using Metam RV-22 and Neophot-21 microscopes.

In dynamic bending tests, standard (ISO 148-1:2016) specimens with a U-shaped notch 2 in width and depth and a radius of 1 mm were used. The tests were carried out at a temperature of -70 °C on an MK-30 pendulum hammer with the maximum lifting height of the pendulum. A sample with an FMC structure perceived the load perpendicular to the arrangement of the fibers. The macrostructure of a fracture after impact bending tests is shown in Figure 2 [2].

Probe microscopy does not require special preparation of samples, but limits their overall dimensions to the working volume of the microscope chamber. The samples had linear dimensions of 10 * 10 * 4 mm. Also, the height of the surface features should not exceed 200 µm due to the limitation of the height of the probe rise above the surface. The study of the fracture surface was carried out using a Nanoeducator II atomic force microscope (NT-MDT, Zelenograd, RF) in a semi-contact mode.
The AFM image makes it possible to record small grain sizes, which leads to a deviation from the optical microscopy data, but can be considered more reliable [3-5]. To determine the geometric parameters of structural elements of steel, the Section Analysis and Grain Analysis methods were used. The Section Analysis method allows you to analyze the surface profile, in particular, the shape of particles and their mutual arrangement, and calculate the main statistical parameters of the profile. The Grain Analysis method is intended for geometric parameters of structural elements and surface features such as local height, height measured from the general zero level, linear dimensions, and their area [19].

3. Results and discussion
The volume fraction of phases after quenching from ICI was determined by stereometric analysis of the microstructure using a computer image processing program, the results are shown in table 3.

| Indicator                          | Value   |
|------------------------------------|---------|
| quantity light phase               | 69.82%  |
| quantity dark phase                | 29.86%  |
| Quantity gray (intermediate phase) | 0.32%   |
| average size of particle           | 40.186 μm |

 When carrying out the macrostructural analysis of the fracture surface (Figure 2), three characteristic zones were identified: at the mouth of the notch, the region of the ferrite-martensite interface is fixed, which is formed as a result of shear fracture with delamination along the planes of maximum tangential stresses; then there is a zone of ductile fracture and, finally, a zone of unstable brittle fracture. The macrorelief of the fracture surface of the sample indicates brittle fracture with large cleavage facets surrounded by a network of branching cracks and the formation of micropores [2].

AFM scanning of the fracture surface was carried out in the zone of unstable brittle fracture. Further data processing was carried out in the Gwyddion program [20]. After subtracting the total slope and compensating for the thermal drift, 3D images were obtained, profiles were extracted along lines parallel to each other with a step of 1 μm and going along the X or Y axes. The results of studying the fracture structure on the AFM are shown in Figures 3, 4 and 5.
Figure 3. 3D-image of the FMC fracture obtained by AFM

Figure 4. Profiles of the fracture surface in the direction of the Y-axis of the image
Figure 5. Profiles of the fracture surface in the direction of the X-axis of the image

On the 3D image of the FMC fracture, different areas are clearly visible over the entire surface, within which the shapes of depressions and bulges are repeated, which indicates the heterogeneity of the structure. Table 4 shows the average geometric characteristics of the features (bulges) of the fracture surface.

| Characteristic                        | value         |
|---------------------------------------|---------------|
| average size, nm                      | 222±18.9      |
| average volume, µm* µm *nm            | 3.87±0.57     |
| local height, nm                      | 114.7±12.8    |
| average size X, nm                    | 254±20.5      |
| average size Y, nm                    | 261±21.6      |

As seen from Figures 4 and 5, the surface profiles extracted at different sites show the presence of a line structure with different line widths and a large difference in the heights of their localization, as well as a certain periodicity of features.

As is known, the nature of destruction of ferrite and martensite is different. Consequently, the parameters of the surface roughness of ferrite and martensite grains should differ. As the analysis of the
profiles shows, the height difference between the surface areas is from 450 to 600 μm. Table 5 shows the roughness parameters of the individual phases. The ferrite surface is characterized by a more developed roughness and one and a half times the maximum profile height.

Table 5. Surface roughness parameters of FMC phases after impact bending test

| Roughness parameters, nm | Ferrite       | Martensite   |
|--------------------------|--------------|--------------|
| Average roughness, Ra    | 123,5±24,5   | 76,41±16,01  |
| Mean square roughness, Rq| 153,3±29,1   | 94,1±19,72   |
| Profile height by ten points, Rz | 312,6±32,9 | 208,9±39,51  |
| Maximum profile height, Rt | 506,4±83,2  | 360,6±73,44  |

Features in different parts of the surface have different shapes, which allows us to conclude that there are phases with different physical and physicochemical properties in the structure. The AFM study of the fracture showed that the results of optical and atomic force microscopy are in agreement.

4. Conclusions
The three-dimensional AFM image of the surface fracture and its profiles taken in different areas show the presence of a lineage in the steel structure. The study of the structure by AFM showed that the data of optical and atomic force microscopy are in agreement. Differences in grain sizes and roughness parameters in different parts of the surface make it possible to conclude that the structure contains phases with different physical and physicochemical properties. Despite the fact that this method is a more informative method than optical microscopy and less laborious than electron microscopy, the use of AFM is most expedient in combination with traditional metallography methods.

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