The use of the atomic force microscopy to investigate the structure of steel 14G2

V V Duka, V N Pustovoit, D A Ostapenko, L P Aref‘eva and Yu M Dombrovskij
Department of Physical and applied material science, faculty «Machinery engineering and equipment», Don state technical university, Gagarin's square 1, Rostov-on-Don 344000, Russia
E-mail: valentina.duka.92@mail.ru

Abstract. In this work, we investigated the structure of doeutectoid steel 14G2, which is mainly used for welded structures. Two methods were used to analyze the structure of this steel: optical microscopy (Metam RV -22 and Neophot - 21) and atomic force microscopy (PHYWE Compact AFM) using the semicontact mode. The investigation of the microstructure of steel was carried out for two samples obtained by different heat treatment. The first sample was quenched in water and subsequent low-temperature tempering and had a uniform troost-martensitic structure. The second sample was quenched from the intercritical interval with subsequent low-temperature tempering and had a stitch structure. The results of studying the microstructure of both samples of steel 14G2 are presented. The analysis of AFM images made it possible to establish the geometric parameters of the structural components, to estimate their shape, relief, roughness, and fractal dimension of surface.

1. Introduction
The most important problem of modern materials science is the creation of new and improving the quality of existing materials, the development of methods for analysis and diagnostics of materials. To obtain the necessary properties, the steel is subjected to various types of processing (thermal, thermomechanical, chemical-thermal, etc.), during which the phase composition and microstructure change. Traditionally, structural changes are studied using optical microscopy, which studies the substructure with sizes up to 1 μm. One of the actual directions for improving research methods is to increase the resolution of tools. As was shown in a works [1–9], scanning probe microscopy allows one to obtain information not only about the morphology and fractal properties of thin films and nonmetallic materials, but also about the micro- and nanostructure of steels. For example, in [6], the AFM method was used to investigation the structure of structural steels before and after prolonged exposure to hydrogen-containing media at elevated temperatures and pressures and showed a change in the micro- and nanostructures. In [9], AFM images of steel 45 samples were obtained in the delivery state and after long-term exploitation, and an analysis was made of the change typical of steel 45 in the delivery state of a ferrite-pearlite structure to a structure consisting of a pearlite component surrounded by a ferrite network from a disk-shaped polyhedral ferrite which the Widmannstätt ferrite plates extend.

The aim of this work is to analyze the use of the atomic force microscopy method to determine the state of steel according to microstructure data. To achieve this aim, the following problems were successively solved: 1) preparing the surface of the thin section, 2) obtaining AFM images, 3)
determining the geometric parameters and fractal dimension of the microstructure of the surface of the samples, 4) comparing the parameters of different samples.

2. The morphology and fractal analysis of steel samples
Metallographic researchs were carried out on longitudinal sections. The preparation was completed on sanding paper with final polishing on a felt wheel using GOI paste. Samples were etched with a 4% solution of nitric acid (HNO₃) in alcohol.

To analyze the state of the steel structure of two samples of structural steel 14G2 with the initial stitch ferrite-pearlite structure, which were processed at different temperature conditions, were investigated using atomic force microscopy. The first sample was quenched at 860°C in water and subsequent low-temperature tempering (200°C) and had a uniform troost-martensitic structure (figure 1a). The second sample was quenched from the intercritical interval at 730°C with subsequent low-temperature tempering (200°C) and had a stitch ferrite-martensitic structure, natural ferrite-martensitic composite. With this processing mode, it becomes possible to control the structure and properties of the composite. By changing the heating temperature for quenching from the intercritical temperature range, it is possible to change the volume ratio of the phases of the composite — ferrite and martensite. The carbon content in martensite and, consequently, its mechanical properties also change. A quantitative assessment of the volume fraction of the hardening phase in sample 2 was carried out using the K0I-1 program (figure 1b). Stitch structure on the one hand, is a defect in metallurgical production. On the other hand, samples with stitch structure can be considered as a natural composite, which has anisotropy of mechanical properties [10, 11] and can be used for highly loaded welded building structures, boiler plants, high pressure pipes, steel armor, etc.

![Figure 1. The microstructures of the sample 1 (a) and sample 2 (b).](image)

Microstructural analysis of metals and alloys is carried out on the basis of the analysis of the sizes of structural components and their volume fraction using Euclidean dimension. The method for describing microstructures based on defect densities has significant drawbacks - on the one hand, for many microstructures, the defect density does not determine the behavior of the material under load, and on the other hand, in the process of changing the system, the dimension and energy of defect change. Fractal geometry studies rough surfaces, which are difficult to describe using traditional methods. Fractal dimension allows to quantitatively describe the microstructures and their constituent elements, to determine the structural parameters associated with the properties of the material [12].

Figures 2 and 3 show three-dimensional AFM images of the surface of both samples. We found that at the nanoscale the structural surface formations of both samples are located fairly uniformly and are smaller than the nanotechnological boundary of 100 nm in all directions. The structural formations of the surface of sample 2 in all directions are smaller than those of sample 1.
Figure 2. Three-dimensional AFM image (a) and surface roughness (b) of the microstructure of steel 14G2 after quenching at 860°C and low tempering.

Figure 3. Three-dimensional AFM image (a) and surface roughness (b) of the microstructure of steel 14G2 after quenching at 730°C and low tempering.

A comparative analysis of the roughness and waviness of the surface of the samples was held (table 1). The data show that sample 1 has separate small areas that differ in size, and also quite large areas of the "plateau" type (figure 2b). Sample 2 has an almost smooth surface.

Table 1. Roughness and waviness parameters of the studied samples.

| Parameters (nm)                      | Sample 1 | Sample 2 |
|--------------------------------------|----------|----------|
| Average roughness                    | 2,686    | 0,686    |
| RMS Roughness                        | 3,45     | 0,855    |
| Maximum roughness height             | 21,371   | 5,400    |
| Maximum roughness depth              | 10,999   | 3,057    |
| Maximum roughness peak height        | 10,373   | 2,881    |
| Average maximum roughness height     | 14,244   | 3,743    |
| Average distance between profile      | 957,857  | 878,820  |
| irregularities                       |          |          |

Both samples are of interest from the point of view of detecting a fractal structure. The fractal dimension of the surface was determined by the method of counting cubes. Figure 4 shows graphs of
The fractal dimension on a logarithmic scale. As you can see, both graphs are linear, that is, the structure is fractal. The slope of the graph directly gives the fractal dimension $D_f$. For sample 1, $D_f = 2.30433$, for sample 2, $D_f = 2.31783$. According to [4, 12], values of fractal dimension tending to 2 are characteristic for a fairly smooth surface, values tending to 3 speak of a more complex surface structure. A comparison of the results shows that the fractal dimensions of the samples differ slightly and the samples have a fairly smooth surface. Since both samples were polished, this conclusion is valid.

![Graph](image1.png)

**Figure 4.** The fractal dimension of sample 1 (a) and sample 2 (b).

### 3. Conclusions

Thus, researches of structural steel 14G2 with troostro-martensitic and line-wise ferritic-martensitic structures showed that atomic force microscopy is an informative method along with optical microscopy and less time consuming than electron microscopy. Atomic force microscopy gives a three-dimensional image of the structural elements of the surface of the material and allows us to evaluate their fractal dimension. At the same time, AFM does not require additional sample preparation. On the other hand, the combination of optical microscopy and AFM is optimal for achieving the main goals of metal science.
References

[1] Memnonov V P, Ul'yanov P G, 2011 Technical Physics. The Russian Journal of Applied Physics. 56 12 1802

[2] Aref'eva L P, Blinov A V, Kravtsov A A, Shebzukhova I G, Serov A V Matec Web of Conferences 2018 226 03009

[3] Aref'eva L P, Shebzukhova I G 2018 Physical and chemical aspects of the study of clusters, nanostructures and nanomaterials 10 27

[4] Sdobnyakov N Yu, Zykov T Yu, Bazulev A N, Antonov A S, 2009 Vestnik Tverskogo gosudarstvennogo universiteta. Seriya «Fizika» 6 112

[5] Brylkin Yu V, Kusov A L 2013 Physical and chemical aspects of the study of clusters, nanostructures and nanomaterials 5 33

[6] Ulyanov P.G., Usachov D. Yu., Senkovskiy B.V., Borygina K.L., Nikolaev Ph. A., Adamchuk V.K., Pushko S.V., Maltsev A.A., Balij K.S. 2012 Vestnik of Saint Petersburg University. Physics and Chemistry 4 43

[7] Zuev L B, Shlyakhova G V 2014 Materials Science 7 7

[8] Barannikova S A, Shlyakhova G V, Zuev L B, 2016 Vestnik Tambovskogo universiteta. Seriya Yestestvennyye i tekhnicheskiye nauki. Prilozheniye k zhurnalu 21 3 882

[9] Shlyakhova G V, Zuev L B, Popova E A Vestnik 2018 Tambovskogo universiteta. Seriya Yestestvennyye i tekhnicheskiye nauki. Prilozheniye k zhurnalu 23 123 581

[10] Pustovoit V N, Dolgachev Yu V, Aref'eva L P, Duka V V, Fedosov V V, Salynskih V M, 2018 MATEC Web of Conferences Vol. 226. 03006.

[11] Pustovoit V N, Dolgachev Yu V, Duka V V, 2017 Izvestiya Volgogradskogo gosudarstvennogo tekhnicheskogo universiteta 10 (205). 118

[12] Ivanova V S, Balakin A S, Bunin I Zh, Oksogoev A A 1994 Sinergetika i fraktały v materialovedenii (Moskow: Nauka) p 383