Tribological Fracture of Ferritic-Pearlitic Steel with Nano and Submicron Structure during Sliding Friction

S P Yakovleva¹, S N Makharova¹, P G Mordovskoy²
¹Russian Academy of Sciences, Siberian Branch, Larionov Institute of Physical and Technical Problems of the North, 1 Oktyabrskaya Street, Yakutsk, 677980, Russia
²Russian Academy of Sciences, Siberian Branch, Yakut Scientific Center, 2 Petrovskogo Street, Yakutsk, 677000, Russia

E-mail: spyakovleva@yandex.ru

Abstract. An urgent task of tribology is to study the wear mechanisms of various materials for the development of stable wear-resistant microstructures and optimal modes of their production. The purpose of this work is to assess the wear resistance of low-alloy steel with a submicrocrystalline ferritic matrix, reinforced with nano- and microspheres of cementite, as well as to reveal the features of its destruction by friction based on the analysis of friction surfaces. Steel nanostructuring was carried out using cold (at room temperature) equal-channel angular pressing and subsequent annealing. The process of steel wear in coarse-grained and nanosubmicron states has been studied at various stages of tribological loading under sliding friction conditions using optical and scanning electron microscopy, profilometry, and analysis of changes in the roughness parameters of friction surfaces. It is shown that the formation in steel of a heterogeneous structure “submicron ferrite matrix - nanoscale carbide phase” provided an increase in wear resistance by more than two times. The features of contact fracture are revealed, which determine the increase in the wear resistance of steel upon refining the structure and indicate significant differences in the nature of the metal tribological strength formation, depending on the level of dispersion of microstructural elements. The results obtained can be used in the development of technological processes for the formation of nanoscale structural components and improving the properties of traditional low-alloy steels.

1. Introduction
One of the most promising ways to create alloys that are optimal in terms of wear resistance is the development of heterogeneous microstructures of the "matrix - finedispersed hardening phases" system [1-3]. Understanding the tribological aspects of the behavior of such microstructures requires not only a quantitative assessment of wear resistance, but also the identification of features of fracture resistance under friction conditions; this is necessary for the development of optimal modes for obtaining stable wear-resistant microstructures. In other words, the study of the mechanisms of wear resistance formation is an urgent task of fundamental and applied research to improve one. The structure and relief of the wear surface of materials, the features of which are associated with their tribotechnical characteristics [4, 5], carry important information about the processes during friction and wear. Accordingly, it is necessary to study the indicators of wear resistance in interrelated to the structure of friction surfaces formed during tribo-tests.
As is known, in recent years, methods have been intensively developed that make it possible to significantly change the physical and mechanical properties of metallic materials (including steels) due to the formation of nano- and submicrocrystalline states in them. This is, first of all, equal channel angular pressing (ECAP) at temperatures usually about 0.3 ... 0.4 of the melting point of the deformed metal [6-8]. As a rule, several pressing cycles (passes) are used. For the industrial application of the ECAP method, it is advantageous to lower the deformation temperatures, but due to the relatively small ductility margin of steels, the workpieces quickly destroyed after several passes. In [9, 10] we have shown the possibility of nanostructuring low-alloy steel by combining two cycles of "cold" (at 20°C) ECAP followed by annealing at 350°C and 450°C. The processing led to the formation of a structure in the form of a submicron ferritic matrix reinforced with nanoscale carbide particles. A multiple increase in the strength of nanostructured steel, as well as its resistance to brittle fracture, was revealed. It should be expected that nanostructured steel will also have good tribological properties. The purpose of this work is to assess the wear resistance of low-alloy steel with a submicrocrystalline ferritic matrix reinforced with nano- and microspheres of cementite, as well as to identify the features of its tribological fracture under sliding friction based on the analysis of friction surfaces.

2. Materials, experiments and research methods

As noted above, a fine-grained structure strengthened by solid micro- and nanophases was obtained in a low-alloy ferritic-pearlitic steel by ECAP. Chemical composition of steel is: Fe–1.34Mn–0.64Si–0.14Cr–0.09N–0.09C, wt. %. The steel was examined in the following states:

- initial coarse-grained state (average grain size ≈ 20 μm);
- after ECAP at 20°C with the number of cycles 2 (mode 1);
- after ECAP at 20°C with the number of cycles 2 and subsequent annealing for 1 h at 350°C (mode 2);
- after ECAP at 20°C with 2 cycles and subsequent annealing for 1 h at 450°C (mode 3).

The main stages of the study of steel samples with different levels of structure dispersion included, in addition to determining the level of wear resistance, analysis of the damage mechanisms and microstructure of friction surfaces at various stages of tribo-testing, as well as their roughness evolution.

The microstructure was analyzed using metallographic microscopes "Neophot-32", "Axio Observer D1m" and scanning electron microscope JSM-6480 LV "JEOL". The JSM-6480LV device was also used to study the microstructure of friction surfaces. To determine the mechanical properties, proportional flat specimens were stretched at 20°C on a UTS 20K testing machine at a deformation rate of 1 mm / min and an ultimate load of 1 ton.

Tests for sliding wear were performed on a serial friction machine SMTs-2 according to the “Pin-on-Disc” scheme, borderline lubrication was machine oil, the average microhardness of the disc material was 9220 MPa. Shaft rotation frequency 5 r/s. Under these conditions, the thermal regime on the surface, which depends on the power of friction, does not lead to a noticeable change in the strength properties of the material. The wear was determined by the weight method. The wear rate was calculated by the formula \( I = \frac{\Delta m}{L} \), where \( \Delta m \) is the mass wear of the sample, equal to \( m_i - m_{i+1} \), i.e. difference in mass before and after testing the sample, \( L \) is the friction path. Profilograms of friction surfaces were obtained using an SJ-201P profilometer along three lines in pre-marked areas of the samples. Mass wear measurements and profilometry were carried out in stages after a certain number of friction cycles (1500 at the beginning of the tests and 4500 later). The friction surfaces were photographed after 4500, 18000 and 36000 cycles. The number of cycles of 5000 corresponds approximately to the end of the wear-in stage and the beginning of steady-state friction.
3. Results and discussion

3.1. Microstructure and quantitative analysis of structural damage to spring steel
The microstructure of the steel in the initial state (figure 1, a) is a ferritic-pearlitic mixture with an average grain size of \( \approx 20 \, \mu m \). ECAP treatment, as it described in the literature [6-8], leads to the grinding of the structural components and the formation of submicrocrystalline structures. The size of ferrite regions free of pearlite and having a submicrocrystalline internal structure is \( \approx 5 \, \mu m \). ECAP caused the fragmentation of pearlite colonies and its "spreading" in the deforming ferritic matrix in the form of small isolated grains (figure 1, b). Similar changes in the structure under cold deformation were observed to a large extent in [11]. With subsequent heating to 350° ... 450°C, the size of the ferritic regions decreases, the transformation of the carbide phase continues (figure 1, b, c). Instead of thin-platy colonies, finely dispersed spherical particles of cementite appear; average size of cementite particles \( \approx 30 \, nm \). A more detailed electron microscopic description of the structural changes in the investigated steel during ECAP according to the indicated modes is given in [12]. The nanostructuring processes developed most efficiently after treatment by mode 2. This mode provided the best in terms of fineness and homogeneity of the structure, as well as the greatest increase in the strength and microhardness of steel: the ultimate strength was 1400 MPa versus 480 MPa for its initial state, the microhardness was 2950 MPa versus 1470 MPa. The ultimate strengths after processing in modes 1 and 3 are equal to 1100 and 1195 MPa, respectively, and the microhardness values are 2430 MPa and 2790 MPa. Such high strength properties are associated both with a significant decrease in the average size of structural elements and with the creation of high internal stresses during ECAP [13].

![Figure 1. The microstructure of low alloy steel: a – in the initial coarse-grained state; b – after processing according to mode 1 (arrows point to the remains of perlite grains); c – after processing according to mode 2; d – after processing according to mode 3.](image)

3.2. Impact of ECAP on wear resistance
The leading role in wear is given to structural factors, and not only the characteristics of the original structure, but also the features of the structural state of the active layer, which is formed during wear and largely determines the tribological properties of the metal. Since the formation of the active layer is associated with plastic deformation occurring under contact loading, wear resistance depends on factors that affect the deformation resistance, such as the hardness of ferrite, dispersion and distribution of the carbide phase particles. Structures of the "submicrocrystalline ferritic matrix reinforced with dispersed carbides" type are characterized by a high level of immunity to local shear instabilities [14]. This is explained by a combination of various hardening factors: due to the small grain size, the presence of structural barriers in the form of sub-boundaries and grain boundaries, and
also due to hardening by a dispersed carbide phase. Therefore, it should be expected that when exposed to a certain tribo-load, the wear resistance of steel processed according to modes 2 and 3 will be higher than that of coarse-grained material or that is in the state immediately after the ECAP. For example, the authors of [15-17] noted an increase in the service characteristics of various materials due to nanostructuring even only near-surface layers.

The results of wear tests of steel samples after three types of processing are shown in figure 2 ((the graph for the initial coarse-grained state is not shown, since the values of its mass wear are ten or more times higher than of the processed samples). The appearance of nanoscale elements in the submicrocrystalline structure obtained by mode 1 has significantly improved the wear resistance indicators: in terms of mass wear – more than 2.5 times; in terms of wear intensity – more than 2 times. The best wear resistance was demonstrated by steel treated according to mode 2, which provided, as shown above, the greatest increase in strength and microhardness. The wear rate of this steel at the wear-in stage has decreased by 3.4 times. It is believed that improving the overall wear resistance by increasing its performance at the wear-in stage is favorable from a tribological point of view (often weight loss during wear-in is the main proportion of the total mass wear).

![Figure 2. Effect of ECAP on mass wear (a) and wear rate (b) of low-alloy steel samples.](image)

3.3. Microstructure of friction surfaces and changes in roughness parameters

The mechanisms of the formation of the friction contact profile have not yet been fully studied because of the complexity and multifactorial nature of the process, which is determined by both periodic factors and numerous random perturbations. The initial roughness consists of a set of microprotrusions different in size and geometry. The most intense loads causing deformation experience to high microprotrusions. Obviously, the features of the process of changing the microroughnesses value depend on the features of the structure and physical and mechanical properties of the conjugating surfaces.

As noted in the methodological part of the work, to analyze the evolution of roughness of friction surfaces, steel samples were profiled after a certain number of friction cycles. Roughness was estimated using well-known formulas [18]:

- The arithmetic mean deviation of the profile Ra

\[
Ra = \frac{1}{N} \sum_{i=1}^{N} |Y_i|
\]

is calculated as the arithmetic mean of the absolute values of the profile deviation (Yi) from the baseline; here N is the number of measurements;

- The standard deviation of the profile Rq within the base length is calculated using the formula

\[
Rq = \left( \frac{1}{N} \sum_{i=1}^{N} Y_i^2 \right)^{1/2}
\]

- The height of the profile microroughness Rz is determined as the average of the 5 largest maxima of the profile and the average of the 5 smallest minima of the profile relative to the midline.
The generalized graphs of changes in roughness parameters with an increase in the number of friction cycles (figure 3) show that the profilograms of all samples differ from each other in the level of smoothness at different stages of testing. This is confirmed by the microstructure features of typical wear surface areas of steel samples with different structures (figure 4). A detailed analysis of macro- and microimages confirms that on the friction surfaces of non-heat-treated steel (that is, after mode 1), a coarser relief of friction tracks with extractions, stickings, delamination, and gallings of the material is formed, indicating the presence of an adhesive wear component. The friction surface of the most wear-resistant material in the state after mode 2 has a less pronounced relief with relatively smooth friction tracks and thinner grooves. The processes of rolling up the grooves formed earlier are noticeable. The appearance of flat-topped microroughnesses means an increase in the support surface between the sample and the counterbody, which reduces contact pressures and deformations and explains the better wear resistance of steel structured according to mode 2. The alternation of successive relief-forming and relief-smoothing processes is confirmed by the oscillatory nature of the change in the values of $R_a$, $R_q$, $R_z$ along the entire friction path for a given state of steel (figure 3, b).

![Figure 3. Influence of the steel processing mode on the change in the roughness parameters along the friction path: a – mode 1; b – mode 2; c – mode 3.](image)

![Figure 4. Typical microstructure of the wear surface of samples with the number of friction cycles 18000 after various processing modes: a – mode 1; b – mode 2; c – mode 3; x1000.](image)

Systemic focal fractures is detected only after 36000 cycles (in the case of treatment according to mode 1 - already with the number of cycles 9000). For samples of two other structural states, the graphs of changes in altitude parameters look more monotonous (figure 3, a, c). A distinctive feature of the wear process of the material treated according to mode 3 is the intensive hardening of the active layer at the wear-in stage.

Thus, the evolution of the friction surfaces microrelief of a nanostructured material during wear differs from the more monotonic evolution of the microrelief of samples with a submicron structure. Additional hardening of the submicron ferrite matrix with relatively uniformly distributed nanoparticles of the carbide phase was a fundamentally significant factor for the multiple growth of the material wear resistance. The presence of micro- and nanoparticles of the carbide phase restrains plastic deformation of the surface layer, increases resistance to cracking and the formation of wear.
particles. As a result, the adhesion and deformation interaction at the contact areas decreases, and the number of prehension centers that cause adhesive destruction of the friction surface decreases. In addition, the abrasive wear caused by the separation of wear particles and their plowing through the friction surface is restrained. The question of the mechanisms of development of nanostructured steel tribodestruction requires further detailed research using a complex of different methods.

4. Conclusion
1. It is shown that nanostructuring of coarse-grained ferritic-pearlite steel and the creation of a heterogeneous structure “submicron ferrite matrix reinforced with nano- and microscale carbide phase” type in it allows more than 2.5 times to increase the wear resistance during sliding friction. The best resistance of steel to wear was provided by structuring according to the cold ECAP mode and subsequent annealing at 350°C.
2. The following features of the steel wear kinetics are revealed, indicating significant differences in the nature of the formation of the tribological strength, depending on the level of microstructural elements dispersion:
   • steel with the lowest wear resistance and submicrocrystalline structure (in the state after ECAP) at the stage of wear-in has the most intense wear; at the stage of steady-state friction, wear is more monotonous;
   • steel with the highest wear resistance, submicron matrix and relatively evenly distributed nano- and microspheres of the carbide phase (in the state after ECAP and annealing at 350°C) at the wear-in stage has the lowest wear rate and most quickly passes from the wear-in stage to stage of steady friction;
   • steel with an intermediate value of wear resistance, a submicron matrix and less uniformly distributed micro- and nanocarbides (in the state after ECAP and annealing at 450°C) undergoes hardening at the wear-in stage, which improves its resistance to wear; as a result, at steady-state friction, the wear rate of steel decreases, and the development of wear processes is stable.

5. References
[1] Marukovich E I, Karpenko M I 2005 Wear-resistant alloys (Moscow: Mashinostroenie) p 428
[2] Gualcoa A, Marini C, Svoboda H and Suriana E 2015 Procedia Material Science 8 pp 934-43
[3] Gadalov V N, Afanasyev A A et al 2011 Vestn. MGTU im. Nosova 4 pp 45-49
[4] Kuksenova L I, Lapteva V L et al 2001 Friction and wear test methods (Moscow: Intermet Engineering) p 152
[5] Chichinadze A V, Berliner E M, Braun E D 2003 Friction, wear and lubrication (tribology and tribo-engineering) (Moscow: Mashinostroenie) p 576
[6] Valiev R Z, Langdon T G 2006 Progress in Materials Science 51(7) pp 881-981.
[7] Furukawa M, Horita Z, Nemoto M et al 2001 J. of Materials Science 36 pp 2835-43
[8] Wang J T, Xu C, Du Z Z et al 2005 Nanomaterials by Severe Plastic Deformation pp 829-34
[9] Yakovleva S P, Makharova S N 2008 Proc. IV Eurasian Symposium on the Problems of Strength of Materials and Machines for Cold Climate Regions (Yakutsk) p 304
[10] Yakovleva S P, Makharova S N, Mordovskoy P G, Borisova M Z 2011 J. Perspektivnye Materialy 13 pp 961-67
[11] Sestri Sh M L, Dobatkin S V, Sidorova S V 2004 J. Metally 2 pp 28–35
[12] Makarov A V, Yakovleva S P, Volkova E G, Makharova S N, Mordovskoy 2016 Diagnostics, Resource and Mechanics of materials and structures 6 pp 39-47
[13] Tereshchenko N A, Yakovleva I L, Chukin M V, Efimova Y Y 2015 Physics of Metals and Metallography 116(3) pp 274–84
[14] Hirth I P, Rigney D A 1983 Dislocation in Solids ed F R N Nabarro (Amsterdam: Elsevier) pp 1–54
[15] Makarov A V, Korshunov L G et al 2010 J. Physics of Metals and Metallography 110(5) pp 1-15
[16] Deng S Q, Godfrey A, Liu W, Zhang C L 2015 *Mat. Sci. Eng.: A* **639** pp 448-55
[17] Unal O, Varol R 2015 *Appl. Surf. Sci.* **351** pp 289-95
[18] Kragelskiy I V, Dobychin N M, Kombalov V S 1977 *Fundamentals of Friction and Wear Calculations* (Moscow: Mashinostroenie) p 526

**Acknowledgments**
The authors are grateful to G.G. Vinokurov, Leading Researcher, Candidate of Technical Sciences and N.F. Struchkov, Senior Researcher Candidate of Technical Sciences (Institute of physical and technical problems of the North) for their assistance in conducting tribological tests and profilometry.