Effect of the UFG structure on the fatigue properties of ferritic/martensitic steel

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Abstract. This paper presents the results of investigation of the effect of an ultrafine-grained structure on the fatigue properties of ferritic/martensitic steel. Special attention is paid to the study of structural changes after fatigue tests. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were employed to study the features of fatigue fracture and microstructural evolution, depending on the distance from the fracture surface. Using X-ray diffraction, we determined the lattice parameter, coherent scattering region and microstructural evolution, depending on the distance

1. Introduction
It is known that in 9-12%Cr steels during low-cycle fatigue tests at elevated temperatures, softening occurs due to the instability of the dislocation structure, which leads to a change in the dislocation density and carbide coarsening [1, 2]. Besides, the fatigue strength is greatly influenced by the structural features formed after quenching and tempering. For example, it was found that carbide in the form of chains at the boundaries of former austenite grains are the spots for the crack nucleation in ferritic/martensitic steels [3]. Among the methods that have been actively developed in the past two decades and enable a considerable enhancement of the strength properties of metallic materials are severe plastic deformation (SPD) techniques. It is known that the UFG structure formation by means of equal-channel angular pressing (ECAP) leads to an increase in microhardness and the ultimate tensile strength in various metals and alloys [4], and could result in an increase of the fatigue endurance limit. For instance, in [5] it was demonstrated that the UFG structure formation occurring in the process of ECAP, alongside with a more uniform distribution of strengthening particles, also contributed to an increase in fatigue endurance limit in a number of Al-, Mg- and Ti-based light alloys.

Ferritic/martensitic steel 12Cr-2W-2Ni-0.5Mo, used in power mechanical engineering and operating at temperatures of up to 600 °C, contains a large number of carbide particles. It was found that in the process of SPD, these particles become refined and their volume fraction increases [6, 7]. In [8], it was shown that the particles of MC carbides precipitating at grain boundaries inhibit the dislocation motion, enhancing the low-cycle fatigue behavior. However, currently there is little data from the study of the fatigue properties and the effect of carbide particles in UFG ferritic/martensitic steels. The aim of the present paper is to study the features of fatigue fracture in the UFG ferritic/martensitic steel 12Cr-2W-2Ni-0.5Mo.
2. Experimental procedure
As the initial material, we used samples of ferritic/martensitic steel 12Cr-2W-2Ni-0.5Mo (table 1). Prior to ECAP processing, we performed a standard treatment – quenching from a temperature of 1050 °C in oil and tempering at 800 °C. Processing by ECAP was performed using 6 passes on the route B, with the angle of intersection of the channels equal to 120 degrees at a temperature of 550 °C on samples with a diameter of 20 mm and a length of 100 mm [6,7].

Table 1. Chemical composition, wt.%.  

|   | C    | Si  | Mn  | Ni | S  | P  | Cr   | Mo  | W   | V  |
|---|------|-----|-----|----|----|----|------|-----|-----|----|
|   | 0.14 | 0.31| 0.32| 1.63 | 0.009 | 0.02 | 11.53 | 0.45 | 1.66 | 0.18 |

To find the fatigue endurance limit, we used cylindrical samples with a gage diameter of 3 mm. The fatigue tests of the standard samples were performed under a symmetric loading cycle at room temperature with 12 samples per each condition. The cycle asymmetry coefficient \( R = \sigma_{\min}/\sigma_{\max} \) was considered as equal to -1.

For X-ray and TEM analysis, disks with a diameter of 3 mm were cut near the fracture surface. The first disk was cut at a distance of 0.3 mm from the fracture surface, followed with a step of 0.5 mm. To calculate the lattice parameter and the sizes of the coherent scattering regions, we used diffraction patterns obtained with a Rigaku Ultima IV diffractometer employing the Bragg-Brentano focusing method (goniometer geometry). The microstructure was characterized using a JEM-2100 transmission electron microscope. Thin foils for TEM investigations were punched from the slices of the samples. Then they were mechanically ground to a thickness of 0.15 mm, and finally double-jet electropolished to perforation using an electrolyte based on n-butyl alcohol. To study the relief surface of the fractured samples, and also to further study the structure, a JSM-6390 scanning electron microscope with an accelerating voltage of 30 kV was employed [7].

3. Results
The microstructural study by TEM revealed that the structure after quenching was martensitic, with an average band width of 520 nm and a length of 3-4 μm. Tempering resulted in the precipitation of the second-phase particles about 40-60 nm in size, predominantly at the boundaries of former martensite plates [6,7]. ECAP treatment led to the formation of an equiaxed UFG structure with an average grain size of up to 0.6 μm with high-angle misorientations and an average particle size of up to 90 nm. In this case, a uniform arrangement of particles is observed both at the boundary and in the body of the grain. The analysis revealed the presence of \( \text{Cr}_2\text{C}_6, \text{Fe}_2\text{C}, \text{WC}, \text{Ni}_3\text{C} \) particles [7]. The X-ray phase analysis demonstrated that SPD leads not only to a decrease in the size of coherent scattering regions (CSR) and an increase in the dislocation density, but also a decrease in the lattice parameter and an increase in the volume fraction of carbide particles [7]. After ECAP, in comparison with the tempered sample, one can observe an increase in the ultimate tensile strength from 950 MPa to 1100 MPa and the yield stress from 790 MPa to 1040 MPa without significant changes in ductility [7]. Figure 1 shows the fatigue curves in the initial state (tempering) and after ECAP processing. It can be seen that after ECAP processing, the fatigue endurance limit at \( 10^7 \) cycles increases by 20% from 472 MPa to 570 MPa. The enhancement of the fatigue properties in the samples with a UFG structure is related to grain refinement, as well as with an increase in the volume fraction of second-phase particles [7]. In the low-cycle region (up to \( 10^5 \) cycles), there are practically no differences in the fatigue curves between the tempered and the UFG samples. However, in the high-cycle region, the larger the number of cycles to failure, the more significant is the advantage of the UFG material.
Figure 1. (a) Dependence of the number of cycles to failure, $N$, on the stresses in a cycle; microrelief of the fracture in the zone of stable crack growth: (b) in the UFG state, (c) in the CG state.

The basic difference in the fracture behavior, based on the data of fractographic analysis, is that in the ECAP-processed samples, the zone of stable crack growth is practically twice as large as the similar zone in the samples after tempering. In the UFG state, we observe the formation of wider (up to 50 µm) fatigue striations where the inhibition of secondary cracks occurs (figure 1 b, c).

In the samples after the standard treatment (tempering), we observe a line arrangement of carbide particles along the boundaries of former austenite grains (figure 2), which is a factor that reduces the fatigue endurance limit in ferritic/martensitic steels [3]. In the ECAP-processed samples, in the immediate vicinity of the fracture zone, we observe finer carbide particles. Besides, the dislocation density immediately in the fracture zone grows by a factor of 1.5, but with distance from the fracture zone, the values gradually decrease to the initial state before testing.

Figure 2. Microstructure (a, b) of the tempered samples and (c, d) of the ECAP-processed samples after fatigue tests: (a, c) – in the fracture zone, (b, d) at a distance of 0.5 mm from the fracture zone.

X-ray diffraction analysis shows that in the fracture zone in the UFG state a dynamic aging process takes place, as a result of which the lattice parameter decreases (table 2).
Table 2. XRD and TEM results after fatigue testing of the tempered and the ECAP-processed samples.

| State          | Lattice parameter, A | Lattice strain, % | Crystallite size, nm | Disl. dens., 10^15 m^2 | Precipitate size, nm (TEM) |
|----------------|----------------------|-------------------|----------------------|-------------------------|---------------------------|
| Tempering      |                      |                   |                      |                         |                           |
|                | 2.87801(5)           | 0.22(9)           | 122(31)              | 0.96(9)                 | 250±30                    |
| Distance from  | 0.3                  | 2.878206(6)       | 0.166(9)             | 81(6)                   | 2.11(8)                   | 150±14                     |
| the fracture   | 0.8                  | 2.878180(7)       | 0.154(6)             | 96(8)                   | 2.04(7)                   | 160±12                     |
| zone, mm       | 1.3                  | 2.878159(8)       | 0.131(8)             | 114(11)                 | 1.43(9)                   | 200±14                     |
|                | 1.8                  | 2.878148(9)       | 0.123(5)             | 127(15)                 | 1.12(6)                   | 220±20                     |
|                | 2.3                  | 2.878133(9)       | 0.112(7)             | 131(18)                 | 1.06(11)                  | -                          |
| Ultrafine-     |                      |                   |                      |                         |                           |
| coarse-grained |                      |                   |                      |                         |                           |
| sample         |                      | 2.87409(4)        | 0.13                 | 36(4)                   | 3.87(16)                  | 90±8                       |
|                | Distance from        | 2.874197(5)       | 0.227(5)             | 24(4)                   | 5.72(6)                   | 60±7                       |
| the fracture   | 0.8                  | 2.874184(8)       | 0.219(8)             | 27(3)                   | 5.21(5)                   | 80±9                       |
| zone, mm       | 1.3                  | 2.874146(9)       | 0.211(7)             | 31(2)                   | 4.86(5)                   | 90±10                      |
|                | 1.8                  | 2.874122(6)       | 0.205(8)             | 34(2)                   | 4.27(4)                   | 95±12                      |
|                | 2.3                  | 2.874110(7)       | 0.193(9)             | 37(3)                   | 3.94(5)                   | -                          |

4. Conclusions
The formation of the UFG structure enabled increasing the fatigue endurance limit by 20% as compared to the standard treatment (from 472 MPa to 570 MPa). Smaller second-phase particles and their uniform distribution along the grain boundaries and in grain interiors inhibited crack propagation, thereby increasing the fatigue endurance limit. The main differences in the fracture behavior of the 12Cr-2W-2Ni-0.5Mo steel in the ultrafine-grained and coarse-grained samples were, first, the different areas of the crack nucleation zone (in the CG state, the zone width was 0.7 mm, and in the UFG state – 1.4 mm), and second, the size and distribution of carbide particles.

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