Mechanical behaviour of the St. 20 Steel after equal channel angular pressing

M R Tyutin¹, L R Botvina¹, O V Rybalchenko¹, D V Prosvirnin¹, A A Tokar¹, G I Raab², and S V Dobatkin¹

¹A.A. Baikov Institute of Metallurgy and Materials Science of RAS, Leninsky prospect 49, 119334, Moscow, Russia
²Ufa State Aviation Technical University, Ufa, Russia
³National University of Science and Technology "MISIS", Leninsky prospect 4, 119049, Moscow, Russia

*E-mail: tyutin@imet.ac.ru

Abstract. The mechanical properties of low-carbon (0.2% C) St. 20 steel with conventional and ultrafine-grained (UFG) structures have been studied. The UFG structure of 340-448 nm in grain size was prepared by the method of equal-channel angular pressing (ECAP) for different numbers of passes (N = 4, 8 and 12) and subsequent annealing at 400 and 450°C. The study included static tension, impact bending at room and lower temperatures, fatigue tests, and the examination of the development of plastic deformation zones by replicas. It was established that an increase in the number of ECAP passes increases strength, but decreases plasticity and impact toughness, decreases strain hardening coefficient, and increases yield strength to the level of ultimate tensile strength. The ECAP for 8 passes leads to an increase in fatigue strength by 25%. Annealing does not substantially change mechanical properties. The optimum set of properties is characteristic of the steel after ECAP for 8 passes and annealing at 400°C. The shear instability revealed in the UFG steel upon static loading as the formation of a network of localized deformation bands inclined at an angle of ~ 55° to the loading axis of the specimen depends on the number of passes.

1. Introduction
The possibility of grain refinement upon severe plastic deformation (SPD) has already been reliably established [1]. Such treatment results in the formation of an ultrafine-grained (UFG) structure: nanocrystalline (grain size D ≤ 100 nm) and submicrocrystalline (100 nm ≤ D ≤ 1 μm) structure. The UFG structure formation in low-carbon steels upon SPD has already been demonstrated by the most common SPD methods such as equal-channel angular pressing (ECAP) [2-6] and high pressure torsion (HPT) [7-9]. The novelty of the present work is as follows. First, the unalloyed low-carbon St. 20 steel is taken for the study. Although SPD is conventionally used for alloyed or microalloyed low-carbon steels, the unalloyed low-carbon steels surpass the alloyed ones in relative strengthening caused by grain refinement, and their thermal stability can be improved by varying the initial state. Second, low-carbon steels are used in the initial state after quenching and high-temperature tempering. The low-carbon steels usually are taken for SPD in the ferritic-pearlitic initial state after normalization, although the initial martensitic (bainitic) state provides more intense grain refinement, the formation of a homogeneous UFG structure due to uniform distribution of carbides after quenching, and increased thermal stability of such structure [9,10]. However, deformation of martensite requires increased...
deforming force. The deformation of the ferritic-pearlitic structure does not require increased deforming force, but the resulting structure is inhomogeneous and thermally unstable. Upon SPD of the low-carbon steel taken in the initial quenched and tempered state, a homogeneous UFG structure can be obtained due to uniform distribution of carbides after quenching, and the deforming force can be reduced due to high-temperature tempering of the steel. Third, the mechanical behavior of the low-carbon UFG steel will be studied by both static and cyclic tests. And fourth, the localization of the deformation of UFG steels for the first time will be studied using the replica method by observing the kinetics of the formation of plastic zones at different loading stages.

The aim of the work is to study the mechanical behavior of the St. 20 low-carbon UFG steel containing 0.2% C by static and cyclic tests, including the study of deformation localization at different loading stages.

2. Experimental

The low-carbon St. 20 steel containing (in wt %) 0.19 C, 0.49 Si, 0.21 Mn, 0.03 Ni, 0.04 S, 0.22 P, 0.25 Cr, 0.19 Cu, 0.05 As, and Fe as a balance was used for the study. The steel was subjected to quenching in water from a temperature of 880°C (1 hour) and high-temperature tempering at 600°C (1 hour).

Equal-channel angular pressing (ECAP) of the specimens of 20 mm in diameter and 120 mm in length was carried out at a temperature of 400°C by route Bc (sequential rotation of the specimen about the longitudinal axis by 90° after each cycle). The intersection angle between the channels was 120°, and the numbers of passes N = 4, 8, and 12 resulted in true degrees of deformation of 2.7, 5.4, and 8.1, respectively.

The microstructure was examined by optical microscopy with a Nikon EPiPHOT TME200 microscope and transmission electron microscopy with a JEM-100CX microscope. Specimens 0.5 mm thick were mechanically thinned to a thickness of 0.12 mm on sandpaper and polished in an electrolyte of the following composition: H3PO4/Cr2O3 = 866 ml/10 g.

The microhardness was measured by the Vickers method with a 402 MVD Instron Wolpert Wilson Instruments microhardness tester at a load of 100 g for an indentation time of 10 s.

Uniaxial tensile tests of flat specimens of 7 × 20 × 1.5 mm in dimensions were carried out with an INSTRON 3382 tensile machine (of 10 t in the maximum load) at a tension rate of 2 mm/min. Since the stress-strain curves exhibited yield drop, the yield strength was evaluated at the lower yield point.

Charpy Impact Tests (KCV) were carried out with an Amsler RKP-450 pendulum impact machine with an ISO type striker at a maximum impact energy of 300 J. Specimens of 5 × 10 × 55 mm in dimensions with rectangular cross-section were used for impact toughness tests. The total impact energy (A) evaluated from the deformation diagram was used to determine the impact toughness KCV of the material and the crack initiation (Ai) and propagation (Ap) energies.

Cyclic tests of flat specimens of 7 × 20 × 1.5 mm in dimensions under conditions of repeated tension at a frequency of 30 Hz were carried out with an INSTRON 8801 servo-hydraulic machine (of 10 t in the maximum load).

The side surface of some flat tensile specimens was polished. Upon the test, three to five stops were made, during which silicone replicas were taken from the polished gage part of the specimens. After the tests, the replicas were examined with an optical microscope, and micrographs of the zones of plastic deformation were made at various loading stages. Such micrographs with the help of the functions integrated into the image analysis program were used to determine the parameters such as the number, width, and area of the localized deformation bands, as well as the area of the plastic zones.
3. Results and discussion

3.1. Structure of the steel after ECAP and annealing

The electron microscopic examination of the St. 20 steel after heat treatment by quenching and high-temperature tempering before ECAP revealed the structure consisting of polyhedral ferrite with pearlite colonies and a small amount of acicular ferrite and tempered martensite. The ferrite grain size is 40-50 μm.

The electron microscopic analysis of the steel after deformation by ECAP for a number of passes N = 4 showed a partially submicrocrystalline and, most likely, subgrain structure (figure 1a). The average size of the structure elements is 375 nm (table 1). The high-angle misorientation of the boundaries is judged from the character of the electron diffraction ring pattern with individual point reflections and from banded contrast at the boundaries. With increasing degree of deformation by ECAP to a number of passes N = 8, the fraction of equiaxed structure elements and the fraction of grains with high-angle boundaries in them increase, which is evidenced by an increase in the number of point reflections in the electron diffraction ring pattern (figure 1b). The average size of the structure elements somewhat decreases to 352 nm (table 1). After ECAP for N = 12, the structure becomes more uniform due to spheroidization of cementite plates in the areas of former pearlite colonies (figure 1c). The average grain and subgrain size slightly decreases to 340 nm (table 1). However, the grains and subgrains remain to be somewhat nonuniform in size.

![Figure 1. Structure of the St. 20 steel after ECAP: a. N = 4 passes; b. N = 8 passes; c. N = 12 passes.](image)

Table 1. Average size of structural elements after ECAP and subsequent annealing.

| State            | Annealing   | Average size of structure elements d (nm) |
|------------------|-------------|------------------------------------------|
| ECAP, N=4        | without annealing | 375 ± 12                                 |
|                  | 400°C, 1 h   | 424 ± 11                                 |
|                  | 450°C, 1 h   | 448 ± 13                                 |
| ECAP, N=8        | without annealing | 352 ± 11                                 |
|                  | 400°C, 1 h   | 390 ± 10                                 |
|                  | 450°C, 1 h   | 423 ± 12                                 |
| ECAP, N=12       | without annealing | 340 ± 11                                 |
|                  | 400°C, 1 h   | 362 ± 14                                 |
|                  | 450°C, 1 h   | 381 ± 11                                 |

For further structure perfection, i.e., for the formation of an equiaxed homogeneous structure consisting mainly of submicron grains, it was decided to subject the material to post-deformation heat treatments. The annealing temperatures were selected on the basis of the temperature dependence of microhardness after ECAP (figure 2) as the temperatures, at which there is still no significant reduction in microhardness, and no new grains of ≥1 μm in size are identified metallographically, i.e.,
the structure in the initial deformed grains upon heating remains in the submicron size range. Thus, the post-deformation annealing temperatures were selected to be 400 and 450°C, and the holding time was selected to be 60 minutes.

The following processes of structure formation occur upon heating: transformation of subgrains to grains; coalescence of subgrains in the oriented fragments of deformed acicular ferrite and tempered martensite; spheroidization of carbides in former pearlite colonies and their growth; and the growth of grains and subgrains in the matrix [11]. Such processes lead to an increase in the average size of the structure element upon heating, but also, as well as after ECAP, the average size in the heated specimens decreases with increasing degree of deformation. For example, the average grain size after ECAP for N = 4-12 and heating to 400°C or 450°C ranges between 424 and 362 nm or between 448 and 381 nm, respectively (table 1). Note that, after ECAP and heating, some grain inhomogeneity is retained because the grains are formed in different structure states (polyhedral ferrite, acicular ferrite, tempered martensite, or pearlite).

3.2. Mechanical properties of the St.20 steel upon static tension

Figure 3 shows stress-strain diagrams for the specimens after quenching, tempering, and ECAP.

The yield drop in the stress-strain curves of the specimens after ECAP increases with increasing number of the ECAP cycles. The ECAP for 4 passes increases the strength characteristics such as
yield strength YS and ultimate tensile strength UTS by 24% and 59%, respectively, compared to those of the quenched and tempered state (Table 2). This is caused by structure refinement from a grain size of 40-50 μm to an average size of the structure (grain-subgrain) element of 375 nm. In this case, elongation EL decreases from 21% to 15%. As the number of passes increases from 4 to 8, YS and UTS additionally slightly increase by 8% and 14%, respectively (Table 2). This can be caused by some refinement of structure elements (Table 1). The plasticity in this case is retained at the same level. A further increase in the degree of deformation with increasing number of passes from 8 to 12 leads to an insignificant increase in the strength characteristics, which corresponds to a slight decrease in the size of the structure elements, although it should be taken into account that such effects are virtually within the measurement error (Tables 1 and 2). However, the elongation in this case decreases from 15 to 11%. For the improvement in plasticity and serviceability, the UFG steel was annealed at 400 and 450°C. The tensile test results showed a fairly small effect of heating on both the strength and plasticity of the steel. Nevertheless, the optimal combination of mechanical properties is provided by annealing at 400°C after ECAP for 8 passes. A slight increase in plasticity in this case can be due to a decrease in dislocations density, and a slight increase in strength is due to the precipitation of fine carbides.

Table 2. Results of static tensile tests of the St. 20 steel samples with UFG structure.

| Number of passes | Initial state (after ECAP) | ECAP + annealing (T = 400°C) | ECAP + annealing (T = 450°C) |
|------------------|---------------------------|-------------------------------|-------------------------------|
|                  | YS (MPa) | UTS (MPa) | EL (%) | YS (MPa) | UTS (MPa) | EL (%) | YS (MPa) | UTS (MPa) | EL (%) |
| Initial          | 460      | 600      | 21     | -        | -        | -      | -        | -        | -      |
| ECAP, N=4        | 729±30   | 742±30   | 15±2   | 698±11   | 712±17   | 16±1   | 709±5   | 731±9   | 17±1   |
| ECAP, N=8        | 787±1    | 843±20   | 15±2   | 807±8    | 856±7   | 15±1   | 760±42  | 780±41  | 15±1   |
| ECAP, N=12       | 790±22   | 871±26   | 11±1   | 806±23   | 896±3   | 11±1   | 776±34  | 842±44  | 11±4   |

Thus, the analysis of the results of static tests shows that, with increasing degree of deformation by ECAP, the ultimate tensile strength increases, the yield strength increases to the level of the ultimate tensile strength, the relative elongation decreases, and the yield plateau disappears. Such changes indicate a substantial reduction in the ability of the steel to plastic deformation.

3.3. Impact tests

The impact toughness tested at room temperature and at −40°C after ECAP decreases by a factor of almost two relative to that of the initial state (table 3). Heating somewhat improves the impact toughness characteristics. The best properties are exhibited by the specimens after ECAP for 8 passes and heating to 400°C. This agrees with the data on the tensile properties. Despite the fact that the impact toughness after ECAP decreases, it remains sufficiently high for the St. 20 steel. As follows from the data given in table 3, ECAP causes a significant decrease in the crack initiation energy relative to that of the starting material, and the crack propagation energy upon impact loading remains at an acceptable level, although decreases by factor of two.

Table 3. Results of impact tests of the St. 20 steel with UFG structure.

| State | Annealing | 20°C | −40°C | KCV (J/cm²) |
|-------|-----------|------|-------|-------------|
|       |           | A₁   | A₂   | A₃   | A   | A₁   | A₂   | A₃   | 20°C | −40°C |
| initial | without annealing | 100  | 23   | 77   | 103 | 18   | 86   | 250  | 256  |
| ECAP  | without annealing | 41   | 8    | 33   | 41  | 9    | 32   | 132  | 99   |
| N = 4 | 400°C, 1 h | 49   | 10   | 39   | 50  | 9    | 41   | 136  | 133  |
| ECAP  | without annealing | 47   | 8    | 39   | 57  | 11   | 47   | 141  | 155  |
| N = 8 | 400°C, 1 h | 57   | 11   | 47   | 57  | 11   | 46   | 167  | 155  |
3.4. Fatigue tests
Fatigue tests by the scheme of repeated tension showed that the fatigue strength, which in the initial state of the steel is ~ 300 MPa, after ECAP for 8 passes increases to ~375 MPa, i.e., by 25% (figure 4). The ECAP for 8 passes was found to be more effective not only in the high-cycle fatigue region, but also in the low-cycle fatigue region. This is evident from the comparison of the fatigue curves of the steel specimens with the initial and UFG structures.

3.5. Examination of Plastic Deformation Zones
The specific features of plastic deformation of the St. 20 steel specimens with the initial and UFG structures after ECAP for different numbers of passes have been studied. To this end, surface replicas have been taken from the polished specimens at various deformation stages of static tensile tests. The loads corresponding to the test interruptions for taking replicas are shown by points in the stress-strain diagrams in figures 5a and 5b.

**Figure 5.** Schematic stress-strain diagrams of the St. 20 steel after ECAP and the patterns of plastic deformation on the surface of the samples at different stages of loading after ECAP for N = 4 (a) and N = 12 (b).

It is established that, at the initial stage corresponding to the elastic segment of the stress-strain diagram, parallel bands of localized plastic deformation are formed on the surface of the specimens upon their tension. The bands are inclined at an angle of ~55° to the loading axis of the specimen. Further deformation of the UFG specimens leads to the widening of slip bands with their subsequent coalescence and the formation of a plastic zone. The UFG specimens loaded for 4 and 8 passes exhibit the slip band systems intersecting the initially formed systems and the cross-shaped plastic zones.
(figure 5a). The specimen loaded for $N = 4$ demonstrate a relatively large plastic zone with a small number of localized deformation bands (figure 5a). The character of the development of plastic deformation in the specimen deformed for $N = 12$ is substantially different. In this specimen, the number of localized deformation bands is larger, while the plastic zone is smaller, and it is developed in only one shear direction (figure 5b). The schemes of plastic deformation zones formed upon loading of the St. 20 steel specimens with the initial structure and after ECAP for different numbers of passes are shown in figure 3.

The observed character of the development of plastic deformation zones is not typical of the St. 20 steel with the initial structure and can be associated with the structure changes occurring upon ECAP. A similar pattern of the formation and development of the X-shaped zones of plastic deformation was observed upon the static deformation of a low-carbon 0.09% C-0.08% Mo-0.03% Nb-0.06% V steel specimen after ECAP for 4 passes [12]. In addition, it was shown [13] that the fracture upon deformation of tungsten with an UFG structure occurs along shear deformation bands, although such behavior is not typical of tungsten with a conventional structure.

Note that, with increasing number of ECAP passes, the number of deformation bands grows, and the area of the plastic deformation zone decreases, i.e., the degree of failure localization increases. These results are shown in the graphs of the dependences of the number of plastic deformation bands (Figure 6a) and the area of plastic deformation zones (figure 6b) on the relative deformation defined as the ratio of the current deformation ($\varepsilon$) to the failure deformation ($\varepsilon_f$).

![Figure 6](image)

**Figure 6.** Number of plastic deformation bands NY (a) and the area of plastic deformation zones SY (b) as a function of relative deformation ($\varepsilon/\varepsilon_F$) upon static loading of the St. 20 steel samples after ECAP.

Failure of the UFG specimens after ECAP for 4 and 8 passes occurs at an angle to the loading axis along one of the slip directions in the X-plastic zone. After ECAP for 12 passes, the specimen is failed by shear over the plastic deformation zone (Fig. 5b).

Thus, the UFG structure formed in the St. 20 steel upon ECAP is the cause of the shear instability upon subsequent deformation and failure. Such instability is most pronounced after ECAP for $N = 12$. The results obtained are in agreement with the data [14], in which the grain refinement in compacted specimens from iron powder changes the deformation mechanism from a uniform development of plastic zone to localized shear failure with the appearance of deformation bands forming the X-shaped plastic zone. The transition from uniform plastic deformation to localized deformation with the formation of shear bands can be associated with a decrease in strain hardening coefficient as a result of grain refinement and the transition to local deformation softening.
4. Conclusions
1. ECAP of low-carbon (0.2% C) St. 20 steel for 4, 8, and 12 passes leads to the formation of an ultrafine-grained (UFG) structure of 340-375 nm in grain size. After annealing at 400 and 450°C, the grain size somewhat increases to 381-448 nm.
2. An increase in the number of ECAP passes increases the strength of the steel and decreases its plasticity and impact toughness. In this case, the yield strength increases to the level of the ultimate tensile strength. The optimal combination of strength, plasticity, and impact toughness is ensured by the ECAP for a number of passes $N = 8$ and subsequent annealing at 400°C.
3. The UFG structure of the St. 20 steel after ECAP for $N = 8$ increases the fatigue strength determined by high-cycle fatigue test under conditions of repeated tension by 25% relative to that of the initial quenched and tempered state.
4. Deformation and failure after ECAP exhibit shear instability, which manifests itself in the formation of a network of localized deformation bands inclined at an angle of $\sim 55^\circ$ to the loading axis of the specimen. This effect is most pronounced after ECAP for $N = 12$.
5. With increasing number of ECAP passes, the number of deformation bands increases, and the area of plastic deformation zone decreases, which indicates an increase in the degree of failure localization.

Acknowledgments
The work was supported by the Russian Foundation for Basic Research (project no. 18-08-00321) and the Fundamental Research Program 37 P of the Presidium of the Russian Academy of Sciences. Investigation of crack resistance was carried out within the framework of state task No. 007-00129-18-00.

5. References
[1] Valiev R Z, Zhilyaev A P, Langdon T G 2014 Bulk nanostructured materials: fundamentals and applications (New Jersey-Wiley) p 456
[2] Dobatkin S V, Valiev R Z, Krasilnikov N A et al. 1999 Recrystallization and Related Phenomena - Sendai: JIM 13 913
[3] Shin D H, Kim W-J, Choo W Y 1999 Scripta Mater.41 259
[4] Park K-T, Shin D H 2002 Mater. Sci. Eng. A 334 79
[5] Fukuda Y, Oh-ishi K, Horita Z, Langdon T 2002 Acta Mater. 50 1359
[6] Mayer G G, Astafurova A G, Maier H J et al. 2013 Mater. Sci. Eng. A 581 104
[7] Degtyarev M V, Chashchukhina T I, Voronova L M et al. 2007 Acta Mater. 55 6039
[8] Dobatkin S V, Shagalina S V, Sleptsov O I, Krasilnikov N A 2006 Russian Metallurgy (Metally) 5 445
[9] Astafurova E G, Dobatkin S V, Naidenkin E V et al. 2009 Nanotechnologies in Russia 4 109
[10] Sastry Sh M L, Dobatkin S V, Sidorova S V 2004 Russian Metallurgy (Metally) 2 129
[11] Dobatkin S V, Odessky P D, Raab G et al. 2016 Russian Metallurgy (Metally) 11 1012
[12] Botvina L R, Tyutin M R, Levin V P et al 2008 Mater. Sci. Forum 584-586 281
[13] Wei Q, Ramesh K T, Ma E et al. 2005 Applied Physics Letters 86 (10) 101907
[14] Jia D, Ramesh K T, Ma E 2003 Acta Mater. 51 (12) 3495