Effect of accumulative high-pressure torsion on the structure of steel 10

I A Ramazanov¹, D V Gunderov¹, ², V V Astanin¹, V D Sitdikov¹,
G T Zakiryanova³, A V Sharafutdinov¹

¹ Ufa State Aviation Technical University, K. Marx str. 12, Ufa, 450008, Russia
² Institute of Molecule and Crystal Physics, Ufa Federal Research Centre RAS,
October Avenue 71, 450054, Ufa, Russia
³ Bashkir State Medical University, Lenin str. 3, Ufa, 450008, Russia

E-mail: ilnar.ramazanov@ya.ru

Abstract. The effect of processing by accumulative high-pressure torsion on the structure of steel 10 is investigated. It is found that after processing by accumulative HPT, a stronger structure refinement takes place in steel 10 as compared to regular HPT processing for the same number of revolutions.

1. Introduction
In the past two decades, there has been a great interest in the new approach for increasing the properties of metallic materials by means of producing ultrafine-grained (UFG) structures in them using severe plastic deformation (SPD) processing [1]. Under UFG materials one means polycrystalline materials with grain sizes below 1 µm having predominantly high-angle grain boundaries [1]. The formation of such structures is possible only when very high strains ($\varepsilon \geq 4 - 6$) are attained at relatively low temperatures ($T_{SPD} \leq 0.4 T_{met}$) under high applied pressures. High-pressure torsion (HPT) enables producing the highest strains [1]. Previously there have been a number of studies on the effect of HPT processing on different steels, in particular [1–3]. Shear strain, $\gamma$, introduced during HPT can be calculated using the following equation: $\gamma = 2\pi nR/h$ [1]. However, it is known that in solid metallic materials the strain that can actually be produced in the process of HPT is much smaller than the expected one calculated using above equation, which is caused by the effect of “slippage” [4,5]. In [6] a new technique called “accumulative high-pressure torsion” (ACC HPT) that enables producing higher strains in hard metallic materials than the regular HPT technique was proposed. In this article, the accumulative high-pressure torsion method was first used for low carbon steel (C 0.1 wt.%, Russian steel 10).

2. Research methods and results
The aim of the present work is to study the structure of low-carbon steel 10 after processing by regular and accumulative HPT.

The chemical composition of the investigated steel 10 is as follows: Fe (basis), C 0.1 wt.%, other substances (Mn, Si, Ni) 0.5 wt.%.
HPT processing was conducted on anvils with a diameter of 10 mm, having a groove 0.3 mm deep, under a pressure of 6 GPa at room temperature. Two disks were subjected to regular HPT for 5 and 11 revolutions. One sample was subjected to accumulative HPT. At the first stage, the sample was subjected to HPT for n=2 anvil revolutions. Then the sample was broken into 4 parts, the parts were stacked onto the anvils, the stack was subjected to compression, and again to HPT for n=2. In this manner, 4 cycles were performed. As a result, the torsional strain was summed with the compression strain (about 75% at each stage). At the final stage, the sample was subjected to HPT for n=3. Thus, the total number of accumulative HPT revolutions was n=11 and an integral disk-shaped sample was obtained.

An analysis of the results of the transmission electron microscopy (TEM) studies for steel 10 after HPT processing for n=5, n=11 and accumulative HPT for n=11 reveals the following. In the electron diffraction pattern of steel 10 after regular HPT processing for n=5, n=11, there are several large blurred reflections on the ring, as well as individual smaller dots (figures 1 and 2). The general view of the electron diffraction pattern indicates the structure refinement. However, in the electron diffraction pattern for accumulative HPT processing for n=11 the reflections are smaller, more numerous and more uniformly distributed on the ring, which indicates a finer structure in the condition after accumulative HPT processing for n=11 (figure 3). The bright-field TEM image with smaller elements also indicates a finer structure in the condition after accumulative HPT processing for n=11.

Figure 1. Structure of steel 10 after HPT for n=5. Bright- and dark-field images, electron diffraction pattern.

Figure 2. Structure of steel 10 after HPT for n=11. Bright- and dark-field images, electron diffraction pattern.
Figure 3. Structure of steel 10 after accumulative HPT for n=11. Bright- and dark-field images, electron diffraction pattern.

The dark-field TEM image of steel 10 processed by HPT for n=11 shows a refined structure with an individual fragment size of about 200 nm. It is difficult to analyze the structure since it is very refined, strongly cold-worked and inhomogeneous. However, in the dark-field TEM image for the condition after accumulative HPT processing for n=11 the grain size is about 50 nm.

The results of the measurements of Vickers microhardness are presented in table 1. The measurements were made on a DuraScan-50 automated microhardness tester under a load of HV 0.025 with the standard exposure time of 10 s.

| Condition          | Values of Hv microhardness |
|--------------------|----------------------------|
|                    | Sample center | Mid-radius | Sample periphery |
| Initial            | 277           |            |                 |
| HPT n= 5 rev.      | 344           | 371        | 492             |
| HPT n=11 rev.      | 425           | 662        | 735             |
| ACC HPT n=11 rev.  | 729           | 736        | 807             |

An analysis of the test results shows that an increase in the number of revolutions leads to an increase in the microhardness values. The implementation of accumulative HPT instead of the classic procedure leads to an overall increase in Hv, and also enables increasing microhardness in the periphery and at mid-radius.

Figure 4 shows the X-ray diffraction patterns of steel 10 in the initial condition, after processing by accumulative and regular HPT.

| Condition          | Lattice parameter, nm | CSR, nm | Lattice distortion, % | Dislocation density, $10^{15}$ m$^{-2}$ |
|--------------------|-----------------------|---------|-----------------------|-----------------------------------------|
| Initial            | 2.86918(2)            | 118     | 0.15                  | 0.8                                     |
| HPT 5 rev.         | 2.86991               | 25      | 0.35                  | 2.8                                     |
| HPT 11 rev.        | 2.87187(8)            | 24      | 0.62                  | 5.6                                     |
| ACC HPT 11 rev.    | 2.87279(7)            | 21      | 0.73                  | 8.7                                     |

According to the XRD data, HPT processing for n=11 leads to structure refinement, coherent scattering region (CSR) reduction, and a considerable increase in dislocation density as compared to the initial condition and that after HPT processing for n=5. ACC HPT processing for n=11 leads to the formation of a CSR with a smaller size (21 nm) and a further increase in dislocation density ($8.7 \times 10^{15}$ m$^{-2}$). The XRD results are presented in table 2.
Hence, the data of X-ray diffraction analysis and transmission electron microscopy indicate that as a result of the accumulative HPT, a higher density of dislocations is accumulated, as well as a stronger grain refinement than in the case of using HPT according to the usual scheme.

3. Conclusions
During accumulative HPT processing a stronger structure refinement takes place in steel 10 as compared to regular HPT processing with the same number of revolutions. The grain (fragment) of steel 10 as a result of accumulative HPT is refined to about 50 nm (according to the TEM dark field), a higher dislocation density and a higher and more uniform microhardness across the disk are achieved than after conventional HPT.

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