Microstructure, properties, and failure characteristics of medium-carbon steel subjected to severe plastic deformation

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Abstract. The paper deals with two-stage processing of medium-carbon steel 45 (0.45 % C; 0.27 % Si; 0.65 % Mn) via quenching and high pressure torsion. Such processing combination allowed producing a nanocomposite microstructure with a ferrite matrix and high-dispersed carbides. The ultimate tensile strength of the nanostructured steel is over 2500 MPa. The processing effect on the structure, mechanical properties and failure mechanisms of steel 45 samples is studied. The peculiarities of static fractures in the samples after HPT are demonstrated in comparison with those after quenching.

1 Introduction

Severe plastic deformation (SPD) is one of innovative directions that allows producing unique properties in bulk materials. One can control microstructure parameters (and thus the properties) by varying SPD regimes in combination with thermal treatment [1]. Cold deformation of low-carbon steels transforms a martensite structure to a submicrocrystalline one at much lower plastic stain degree values in comparison with deformation of initial ferrite-cementite microstructure [2, 3].

The produced structure is characterized by smaller grain size and much higher hardness values. Deformation of martensite in medium- and high-carbon steels is practically impossible at room temperature, as the flow stresses of steel are very high, and the martensite ductility is extremely low [4, 5]. Deformation at elevated temperatures can be effective for this class of materials, when tempering occurs simultaneously with work hardening [4, 6]. However, there are no systematic data on preliminary thermal treatment influence on the properties of medium- and high-carbon steels after SPD.

The aim of this work is to study the microstructure features and mechanical properties of medium-carbon steel after SPD.

2 Experimental procedure

Steel 45 (0.45%C; 0.27% Si; 0.65% Mn) was investigated. Water quenching of samples at 800 °C for 1 hour was applied prior to deformation.

SPD was fulfilled by high pressure torsion (HPT) at an elevated temperature of 350 °C, a pressure P=5 GPa, a number of rotations was N=5. Samples with a diameter of 10 mm and 0.2 mm thick were subjected to deformation [4].

Microstructure was studied with the help of a scanning electron microscope JSM 6390. A fine microstructure was examined by a transmission electron microscope (TEM) JEM-2100 at an
accelerating voltage of 160-200 kV using diffraction regimes and image formation in bright and dark fields. Microdiffraction patterns were taken from the areas with a size up to 1.2 µm.

A lattice parameter was determined by X-ray analysis on a DRON 4M diffractometer with CoKα radiation with a graphite monochromator on a diffracted beam through three main maxima of the X-ray pattern. The range of 2Theta was 40-140° with a step 0.1° and exposure of 20 sec.

The microhardness was measured on a microhardness tester MICROMET 5101 with a load of 0.1 kg for 10 sec. In order to evaluate the influence of preliminary heating prior to deformation, the microhardness of the samples heated up to the deformation temperature and held for 5 minutes (holding time prior to deformation) was measured.

Mechanical tests were performed on a special set for precision testing of small samples with a gauge length of 2 mm and 0.1 mm thick. The samples for mechanical tests were cut at a distance of 1/2 radius from the sample center [4].

3 Research results and discussion

The steel in the initial state (a hot-rolled rod) had a ferrite-pearlite microstructure (Fig.1a). The ferrite lattice parameter was \(a = 2.866 \pm 0.00021\) Å. The microhardness in the initial state was 3520 MPa (Fig.2).

After quenching the steel microstructure is lath martensite (Fig.1b). The average size of martensite lathes was 8±0.5 µm, the average width of plates was 0.3±0.1 µm. A well developed dislocation substructure is observed in the volume of martensite crystals in TEM images. The lattice parameter was \(a = 2.868 \pm 0.00025\) Å, which confirms the martensite solid solution saturation with carbon. The microhardness increases up to 8690 MPa as a result of quenching (Fig. 2).

The microstructure studies of steel after quenching and heating for 5 minutes (holding time before deformation) showed that thin lath-like particles of cementite 10…20 nm thick and up to 500 nm long appeared in the martensite lathes by the beginning of deformation (Fig.1c). This testifies to the beginning of martensite tempering. The ferrite lattice parameter is \(a = 2.867 \pm 0.00013\) Å, which is higher than that in the initial state. This testifies to the fact that saturation of the ferrite solid solution with carbon retains by the beginning of deformation.

The microhardness decreases two times to 5000 MPa, but retains at a higher level than that in the initial state after holding of samples at an elevated temperature prior to deformation (Fig. 2). This is connected with retention of some carbon in the solid solution, on the one hand, and, on the other hand, with precipitation of more dispersed particles of cementite during tempering as compared to the initial ferrite-pearlite state.
The strength of tempered steel reduces two times in comparison with the quenched state, whereas the ductility increases more than 5 times (Fig. 3).

The microhardness of steel 45 after HPT changes from 9000 MPa in the sample center to 10000 MPa on its periphery, where the maximum strain is registered [4]. The microhardness values across the section are higher than those in the quenched steel and are characterized by quite a uniform distribution across the diameter, which testifies to the microstructure homogeneity in the specimen volume.

The tensile test results are displayed in Fig. 3 and listed in Table 1.

Table 1. Mechanical properties of steel 45

| State                                                        | $\sigma_y$, MPa | $\sigma_{ult}$, MPa | $\delta$, % | HV, GPa |
|--------------------------------------------------------------|-----------------|--------------------|------------|---------|
| Initial rod                                                  | 863             | 770                | 7          | 3.03    |
| T=800 °C, 60 minutes, water quenching                        | 2192            | 2000               | 1          | 8.68    |
| T=800 °C, 60 minutes, water quenching + HPT, T=350 °C, 5 minutes | 1665            | 1350               | 12         | 5.46    |
| T=800 °C, 60 minutes, water quenching + HPT, T=350 °C, 5 rot., 5 GPa, 1 rot/min | 2649            | 2397               | 3          | 10.63   |
Quick failure at a high ultimate tensile strength and relative elongation of about 1% is typical of the quenched state.

A short range of strain hardening just before softening (Fig. 3) is typical of tensile curves after SPD and indicates at the start of macroscopic plastic instability (necking of a sample) at low plastic strain values. Nevertheless, the relative elongation of samples after HPT exceeds the ductility of the quenched steel. The strength characteristics of the steel after HPT increase in comparison with both a tempered sample and a sample in the quenched state, as shown in Table 1. After the tensile tests significant difference is observed between the fracture structure of steel 45 in the quenched state and after HPT (Fig. 4).

The fractures of the samples with a martensite structure are homogeneous (Fig. 4a). Mainly brittle failure through a cleavage mechanism is observed in the section central part (Fig. 4c).

The fracture analysis of HPT processed samples (Fig. 3b) testifies to non-uniform deformation. In the section central zone the fractures split into separate fragments 2-5 µm wide (Fig. 4d) oriented mainly along the larger sample part and, therefore, radially towards the deformed sample. Ductile failure with a developed dimpled microrelief (Fig. 4d) is observed within these fragments. Shear dimples are observed in the periphery of the failure zone.

The fractures of samples in the quenched state (a, c) and after HPT (b, d): a, b – general view of the fracture, c, d – central zone

The microstructure after HPT is sophisticated and multi-level. On SEM images (Fig. 5a) one can distinguish irregular boundaries of initial austenite grains and blocks of martensitic plates that are strongly curved locally. The causes of grain boundaries retention in the martensite structure after SPD are not clear enough. One may assume that misorientation angles are higher in these boundaries than those between grains newly formed during deformation or these boundaries have more impurities. TEM images demonstrate a typical poorly-defined banded structure (Fig. 3b). A band width is 120±30 nm, which allows assuming that every martensite crystal is divided into 2-3 bands [5]. Cross boundaries dividing it into fragments are observed within each band (Fig. 5b), as a result the band boundaries become blurred and are practically not distinguishable at high-power magnifications (Fig. 5d). The average grain size estimated via dark-field images (Fig. 5c) was 120±20 nm. Selected-area electron diffraction shown in Fig. 3d indicates at high-angle misorientations of submicrocrystalline
grains. The ferrite lattice parameter was $a = 2.867 \pm 0.00016$ Å, which is higher than that in the initial state, which indicates an enhanced content of carbon in the matrix.

Plate-like cementite precipitates formed during tempering caused by heating before deformation are not observed after deformation. They are divided by shear bands into fragments. During fragmentation they are distributed along boundaries of newly formed grains of the matrix due to diffusion activation in the course of SPD. Thus, high-dispersed cementite particles are observed in the microstructure. The particles are located mainly along grain boundaries of the $\alpha$-phase (Fig. 5d, e). The average particle size is $15 \pm 5$ nm. This process is accompanied with deformation-induced dissolution of the cementite in the matrix volume [7] and precipitation of carbon along boundaries of newly formed grains. This explains carbon segregations in grain boundaries [8, 9] and a reduced volume fraction of the cementite in the structure. Thus, the structure can be classified as nanocomposite.

One may assume that the sample fracture during tensions reflects complex structural changes during HPT. The fragments in the fracture are parts of primary lathes of martensite plates. Failure starts during sample loading along straight lath boundaries situated along the axis of a tensile sample. These boundaries have an extended length as the lath sizes are about 10 µm. Therefore strain accommodation on them is complicated, and the material splits along the boundary. As a result deep pits form on the fracture surface. Not only grain boundaries but also carbide particles in original grain boundaries as well as in boundaries of new SMC grains formed during deformation can be points of crack initiation. However, a homogeneous SMC structure with non-equilibrium boundaries formed within original martensite lathes, which is a ductile component of the structure. After the splitting starts along lath boundaries, resistance to failure could increase due to the ductile mechanism of formation and coalescence of microvoids formed in dispersed cementite particles along the boundaries formed during deformation of grains, which is testified by a dimpled fracture within the split fragments (Fig. 4d).

Thus, the analysis of the microstructure studies and static fractures testifies to inheritance of the martensite structure in steel 45 after HPT. Lack of coarse cementite particles in the initial structure...
provides high-dispersed cementite particles uniformly distributed in grain boundaries due to simultaneous processes of work hardening and dynamic tempering.

4 Conclusions
As a result of combination of phase transformation and severe plastic deformation during SPD at 350 °C of steel 45 with the initial martensite microstructure, a nanocomposite microstructure forms consisting of the ferrite matrix with a grain size 120 nm and high-dispersed carbide particles with a size 10-20 nm uniformly distributed in the matrix. Such a structure in steel provides extremely high strength properties. The ultimate tensile strength is over 2500 MP and the relative elongation is 3%, which is sufficient for principal possibility for innovative application of steel as a structural material.

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References
[1] Valiev Ruslan Z., Zhilyaev Alexander P., Langdon Terence G. Bulk Nanostructured Materials: Fundamentals and Application Wiley 2013 456 P.
[2] Tsuji N., Ueji R., Minamino Y., Satio Y. Scripta Materialia 46 (2002), pp.305-310.
[3] Tsuji N. Advanced Ing. Mat., 2010, 12, № 86 pp.701-707.
[4] Karavaeva M. V., Nurieva S. K., Zaripov N. G., Ganeev A. V., Valiev R. Z. Metal Sci. and Heat Treatment, 2012, № 4, pp.1-5.
[5] S. Dobatkin, J. Zrnik, I. Mamuzic. Metallurgija 47 (2008) 3, 181-186.
[6] J.Zrnik, R. Pippan, S.Scheriau, L.Kraus, M.Fujda. J. Mater.Sci (2010) 45, pp.4822-4826.
[7] Yu. Ivanisenko, W. lojkwski, R.Z. Valiev, H.-J. Fecht. Acta Mat. 51 (2003), pp.5555-5570.
[8] Sauvage X., Queleennec X., Malandain J.J., Pareige P. Scripta Materialia 54 (2006), pp. 1099-1103.
[9] Sauvage X., Ganeev A., Ivanisenko Y., Enikeev N., Murashkin M., Valiev R., Adv. Eng. Mater., 2012 Vol 14 (11) pp968-974.