Effects of grain refinement by HPT processing in carbon steel with various cementite morphology

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Abstract. This article reports on the effect of the initial phase composition (martensitic, patented, spheroidized) of the middle carbon steel C45 (0.45 wt.% C) on grain refinement during high-pressure torsion (HPT) at elevated temperature. It is established that the cementite dissolution and the final grain size after HPT strongly depend on the processing regime, as well as on the cementite morphology in the initial state of the steel. As result of this study, new approaches to obtain a very high-strength state in carbon steels have been proposed.

1. Introduction
Studies of the influence of the structural phase state of the starting metal materials on the formation of a nanostructure in them during processing by the methods of severe plastic deformation (SPD) are of great interest [1]. In particular, it was established that the formation of the initial martensitic structure leads to a stronger refinement of the structure in low-carbon steels using SPD [2]. In [3], in industrial pearlite steel (~ 0.7% C), the formation of a nanostructure with a grain size of about 10 nm was observed, as well as the complete dissolution of cementite plates during HPT at room temperature. Obviously, the initial structure has a significant effect not only on the formation of the nanostructured state and their mechanical properties, but also on the choice of modes of severe plastic deformation in steels. This topical issue was the subject of study in this paper.

2. Material and methods
To assess the influence of the initial structure on the processes of nanostructuring, we used the high pressure torsion (HPT) technique. The work was carried out on high-quality carbon steel C 45 (C - 0.44, Mn - 0.65, Si - 0.18, P0.01, S - 0.0006, Cu - 0.05 wt.%). Several variants of the initial structure states were proposed: stable, metastable and unstable. The stable ferritic-pearlite structure of steel C45 was obtained by austenitization at 800 °C, quenching in water and subsequent high tempering for 1 hour at 650 °C (spheroidized state). A metastable bainitic structure was obtained by heating at 800 °C for 1 hour followed by isothermal conversion to salt melting at 450 °C and cooling with water (patented state). An unstable martensitic structure was obtained by austenitization at 800 °C for 1 hour and quenching in water (martensitic state).

HPT was performed at 350 °C for 10 revolutions under a pressure of 6GPa and speed of 0.2 rpm. Specimens after HPT were disk-shaped with a diameter of 20 mm diameter and a thickness of1.1 mm.
Microstructural, mechanical and electrical data were collected from the specific location in the middle of the radius of the sample \( (r = 10 \text{ mm}) \). Tensile tests were carried out on an Instron 5982 machine at room temperature with a strain rate of \( 10^{-3} \text{ s}^{-1} \). Yield stress (YS), ultimate tensile stress (UTS) and ductility (el.%), measured as elongation-to-failure, were obtained on the small plane samples with a gauge dimensions of 1.0 x 1.0 x 4.0 mm prepared by the spark wire-cutting technique. 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 0.12 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, as well as to further study the structure, a JSM-6390 scanning electron microscope with an accelerating voltage of 30 kV was employed.

### 3. Results and discussion

An analysis of the structure by SEM showed that after HPT in steel C45 with a different initial state, the grain size is refined in all cases. So, for a spheroidized state, the average grain size decreased from 13 \( \mu \text{m} \) to 1 \( \mu \text{m} \). The size of the globule carbide decreased from 2 \( \mu \text{m} \) to 0.5 \( \mu \text{m} \). The fraction of carbides observed in SEM increased from 10 to 26\%. This value significantly exceeds the equilibrium value of 7\%, which, apparently, is associated with the etching effect. In the patented state, the initial structure of bainite after HPT changes not so noticeably at low magnification. In SEM images, some dissolution of the cementite lamellas is observed. The distance between the lamellas after HPT changed from 0.3 \( \mu \text{m} \) to 0.2 \( \mu \text{m} \). Finally, in steel C45, after the deformation of the martensitic state, a dispersed ferrite-cementite mixture is observed in the structure. At a small magnification in SEM, a metallographic texture is visible, which is characteristic of the microstructure of steels obtained by deformation according to the simple shear pattern.

![Figure 1](image.png)

**Figure 1.** The evolution of the structure of steel C45 during HPT at 350 °C, SEM: initial heat treatment (a) spheroidized state; (b) - patenting state; (c) martensitic state; after HPT: (d) spheroidized state, (e) - patenting state; (f) - martensitic state.

Analysis of the microstructure of steel C45 in the spheroidized state showed that, despite a significant degree of deformation, the dissolution of coarse carbides practically does not occur. If the size of the ferrite grains decreases by 13 times, then the size of carbides changes only by 4 times. Apparently, this is due to the excessively high hardness of cementite (more than 800 HB).
The evolution of the structure of the patented state of steel C45 in the process of HPT at elevated temperature was considered in detail in a preliminary study [4]. It was shown by TEM that the initial cementite plates fragment during processing. The particles in this case are in most cases along the grain boundaries. The microstructure is formed by grains with an axial axis and elongated along the shear direction with grains with an average size of about 40±10 nm in the cross section (figure 2a).

The initial martensitic structure with lamellar morphology of the matrix phase accelerates the fragmentation of the structure due to the large extent of the boundaries of martensitic origin [5]. Since the HPT processing occurred at an elevated temperature of 350 °C, a partial decomposition of martensite already has time to pass in the structure of steel C45. The absence of large cementite particles in the initial structure ensures the release of highly dispersed cementite particles uniformly distributed over the grain boundaries as a result of deformation. The average grain size calculated from dark-field images was 120 ± 20 nm. Dispersed carbides were also observed, which are located mainly along the grain boundaries of the α-phase. The average particle size of carbides is 15 ± 5 nm. The distance between cementite particles is about 100 nm [5]. An analysis of the microstructure of steel C45 in the cross section of the disk after HPT obtained by STEM showed that grains elongated along the shear axis are formed with an average length of 130 nm and a width of 25 nm (average grain size is 50±10 nm) (figure 2b).

Figure 2. The structure of steel C45 during HPT at 350 °C in cross section: (a) patenting state [4]; (b) martensitic state, STEM.

The difference in the structure of steel C45 after HPT from the initial state can be explained by the difference in the share of the ferrite/cementite interfaces. Smart analysis shows that the length of the interphase boundaries in the patented state increases by 11 times, compared with the spheroidized state. In addition, a thin lamellar structure actively dissolves during HPT [3,4] and dispersed carbides stabilize the ferrite structure, which leads to the formation of a metallographic texture along the shear direction (figure 2). An analysis of the structure in the cross section for the patented and martensitic states shows that the grain shape and carbide size have a significant coincidence. This is explained by the fact that at 350 °C the decomposition of martensite already begins and thin lamellas of cementite are formed [5].

However, despite the similarity, these states after the HPT have differences in mechanical behavior. The results of the tensile tests are presented in table 1.
Table 1. Structure parameter & mechanical properties of steel C45 after heat treatment and HPT.

| State            | Grain size/ plate width, nm | Cementite size/ plate width, nm | YS, MPa | UTS, MPa | EP%, % | Ref. |
|------------------|-----------------------------|---------------------------------|---------|----------|--------|------|
| Quenching        | 120                         | -                               | 2000    | 2190     | 1      | [5]  |
| Patenting        | 300±20                      | 150±50                          | 350     | 800      | 16     |      |
| Spheroidized     | 13000±800                   | 2000                            | 320     | 620      | 22     |      |
| Quenching + HPT 350 °C | 50±10                      | 10±5                            | 2390    | 2650     | 3      | [5]  |
| Patenting + HPT 350 °C | 40±10                      | 10±5                            | 1390    | 2180     | 4.5    | [5]  |
| Spheroidized + HPT 350 °C | 1000                      | 500±50                          | 800     | 1200     | 12     |      |

In order to understand the nature of the high-strength state of the UFG steel under study, the contribution of different structural parameters to the yield strength following our previous investigations was estimated using a superposition of the contributions of structural components [5]. Where \( \Delta \sigma_0 \) is the friction stress of the \( \alpha \)-iron lattice; \( \Delta \sigma_{hp} \) - grain boundary; \( \Delta \sigma_{ss} \) - solid solution; \( \Delta \sigma_{disp} \) - dispersion; \( \Delta \sigma_d \) - dislocation hardening. The obtained values are listed in table 2. It can be noted that grain boundary hardening in all cases makes the main contribution to the strength. The maximum hardening in steel 45 is achieved due to the formation of the UFG structure and the incomplete decomposition of the supersaturated solid solution.

Table 2. Contributions of various hardening mechanisms to the tensile strength of UFG C45 steel.

| State                | \( \Delta \sigma_0 + \Delta \sigma_{ss} \), MPa | \( \Delta \sigma_{hp} \), MPa | \( \Delta \sigma_{disp} \), MPa | \( \Delta \sigma_d \), MPa | \( Y_{calc} \), MPa | \( Y_{exp} \), MPa |
|----------------------|---------------------------------------------|----------------------------|------------------------------|--------------------------|-------------------|-------------------|
| Quenching + HPT 350 °C | 560                                        | 1350                       | 190                          | 110                      | 2200              | 2390              |
| Patenting + HPT 350 °C | 102                                        | 1373                       | 190                          | 102                      | 1760              | 1400              |
| Spheroidized + HPT 350 °C | 100                                        | 475                        | 332                          | 90                       | 994               | 800               |

4. Conclusions
The influence of the initial state (martensitic, patented, spheroidized) of carbon steel C45 on the formation of the UFG structure during high-pressure torsion at elevated temperature is determined. It is shown that coarse carbides practically do not deform during HPT; on the contrary, the thin lamellar form of cementite or its partial dissolution in a solid solution contributes to a significant refinement of the grain structure of the steel and made it possible to obtain a high-strength state in carbon steels. Thus, for the first time in steel 45, record values of the yield stress - 2390 MPa, and the ultimate tensile strength - 2650 MPa were achieved.

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