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Article

Evolution of Cementite Substructure of Rails from Hypereutectoid Steel during Operation

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Abstract: Transmission electron microscopy methods were used to analyze the cementite substructure in the head of special-purpose long rails of DT400IK category made of hypereutectoid steel after long-term operation on an experimental track on the Russian Railways ring (the passed tonnage is 187 million tonnes). It is noted that the study of various aspects of cementite – its structure, morphology, chemical composition, crystal lattice defects is relevant. The steel structure is represented by three morphological components at a distance of 10 mm from the sample surface: lamellar perlite, fractured and fragmented perlite. The volume fraction of lamellar perlite in the material is 65%. It is shown that after operation, the cementite plates are bent and separated by ferrite bridges. In the plates of ferrite and cementite, a dislocation substructure is formed which is of chaotically distributed and network type in ferrite and of ordered type in cementite. An increased density of dislocations at ferrite-cementite interfaces compared to the volume of ferrite plates was noted. Two possible mechanisms of deformation transformation of lamellar perlite grains are indicated: fracture of cementite plates and carbon pulling out from the lattice of the carbide phase. It is indicated that in the dissolution of cementite plates, the interfacial boundaries “ α -phase-cementite” play an important role. The removal of carbon from cementite plates occurs most intensively near defects in ferrite and cementite. The formed nanosized particles of tertiary cementite are unevenly distributed in the ferrite plates, most of them are observed at the locations of ferrite subgrains and interfacial boundaries. This results in non-uniform diffraction contrast in dark-field images of cementite plates. Nanosized particles of cementite can be taken out into the interlamellar space of pearlite colonies in the process of dislocation slip, or they are formed as a result of deformation decomposition, which is less likely. Fragmentation of ferrite and cementite plates is revealed and azimuthal components of total misorientation angles are estimated. The mechanisms of mass transfer of carbon atoms over interstitial sites, deformation vacancies, dislocation tubes, grain boundaries and fragments are considered. According to all the established patterns of the cementite substructure transformation, a comparison with the results for rails made of hypoeutectoid steel was performed.

Keywords: cementite; hypereutectoid steel; electron microscopy; dislocation substructure; ferrite; fragmentation

1. Introduction

Obtaining information about the cementite behavior in steels under long-term deformation action is relevant and is determined both by the fundamental nature of the problem and its practical significance. In recent years, the attention of researchers in the field of physical materials science has been focused on the traditional study of rail steels. Development of special-purpose rails is conditioned by the intensive use of long-distance transport and an increase in the speed of movement.

In 2013 the production of 100-meter differentially hardened rails of general-purpose made of DT350 hypoeutectoid steel was started in Russia to solve this problem, and in 2018 – rails of special purpose with increased wear resistance and contact endurance made of DT400IK hypoeutectoid steel. DT400IK rails are used for tangent railway tracks with speeds up to 200 km/h and curves without tonnage restrictions.

During long-term deformation, complex processes occur in rails that deteriorate their mechanical properties. In this area, obtaining information is determined both by the fundamental character of the problems as well as their practical significance. The development of patterns of evolution of structural phase state parameters and dislocation substructure during long-term rail operation can only be studied through the use of highly informative methods of physical materials science, particularly transmission electron microscopy (TEM). By using TEM methods, we are able to quantify the contribution of structural components and defective substructures to rail hardening. [1,2].

Cementite (or Θ – Fe_3C iron carbide) is the strengthening phase of carbon steels [1,2]. At temperatures below 727°C , the structure of these steels contains only ferrite and cementite. The solubility of carbon in ferrite is very low. At 727°C it is 0.0218%, but at 650°C it drops to 0.010%, and at 400°C it is less than 0.001%. If we neglect the carbon atoms that can be located in the defects of ferrite crystal lattice, then in steels at room temperature, almost all carbon will be concentrated in cementite [1,2]. It is known from statistical data that from 1.3 to 1.5 billion tonnes of iron alloys are smelted nowadays in the world. In Russia alone, more than 50 million tonnes of steel are smelted annually. Of course, a significant part is made up of low-carbon, mild steels in which the cementite content is low. For rails, rail fastenings, reinforcing bars and many other products, steels containing a noticeable amount of cementite are used. It can be roughly estimated that in Russia alone more than one million tonnes of cementite is produced per year. Since steel products can serve for decades, the amount of cementite in Russia exceeds ten million tonnes. So, it is reasonable to study the features of cementite structure. At the same time, its various aspects can be studied: the morphology of precipitated or already existing crystals, the chemical composition of carbide, the exact type of cementite crystal lattice and defects in it. All these aspects are important and require discussion [1,2].

As a rule, cementite increases the wear resistance of steel, acting as an obstacle to the movement of dislocations. In rail steels subjected to cyclic rolling-sliding loads, the microstructure is initially responsible for deformation hardening and increased wear resistance of the material. Transformation of rails is observed during operation even with a relatively small tonnage passed [3,4]. The strength and plastic properties of cementite crystallites are determined by the state of its defective substructure. Therefore, it is important to know about the evolution of the structure of cementite during operation to predict the behavior of rail steel in rolling-sliding conditions and to ensure reliable operation of railways. There are a number of factors that determine the evolution of cementite. Contact fatigue, for example, depletes carbon in cementite plates by 20%, causing the formation of nonstoichiometric iron carbide [5], which may be related to the removal of carbon from cementite lattices and plate cutting mechanisms. Pearlite steel's deformability is correlated with its morphology. It has been found that the length of cementite plates is the principle factor affecting deformability [6]. According to [7], dissolved cementite particles are found around crushed ferrite boundaries in pearlitic steel surfaces under dry sliding wear.

In the works of Russian [8,9] and foreign researchers of recent years [10–15], the evolution of structural-phase states, mechanical properties, and defective substructure in the rail head under various operating conditions has been analyzed and the mechanisms of cementite fracture have been revealed in model experiments. This made it possible to analyze the mechanisms of wear, degradation of rails properties and the reasons for their failure. The formed data bank, as well as the results of recent studies [16–21] of the patterns of structure formation, phase composition, dislocation substructure can be considered as achievements in the physics of hypoeutectoid pearlite steels. New steel grades have been sought for several years in order to limit contact fatigue and wear. In recent years, improved rail materials with good mechanical properties have been developed through increasing the carbon content to hypereutectoid level [22]. It was found that the hypereutectoid rail has better wear resistance and significantly lower wear losses compared with the eutectoid rail [22]. In addition, the hypereutectoid rail has a smaller interlamellar distance, and the outer layer has a subgrain structure with a higher content of low-angle grain boundaries in the steel matrix and almost no cementite along the boundaries. For rails made of hypereutectoid steel, which include DT400IK rails, such information is practically absent in the literature [22]. They are of undoubted interest for

the formation of high operational properties of such rails and the improvement of differentiated hardening modes.

The purpose of this work is to analyze the cementite substructure of hypereutectoid steel formed during differentiated hardening of rails and their subsequent long-term operation.

2. Materials and Methods

The material of the study is samples of differentially hardened DT400IK rails made of E90KhAF steel produced by JSC Evraz-ZSMK after a passed tonnage of 187 million gross tonnes during ground tests on the experimental ring of JSC VNIIZhT (Shcherbinka, track length 6 km, track width 1520 mm, curvature 965 m, without slopes). The elemental composition is regulated by the Russian State Standard GOST 51685-2013 and TU 24.10.75111-298-057576.2017 of Russian Railways.

Table 1. Main elements in the chemical composition of E90KhAF rail steel (wt.%).

C	Si	Mn	Cr	V	Fe
0.92	0.40	1.0	0.3	0.14	base

The morphology of the structure and phase composition, dislocation substructure were determined by transmission electron microscopy (TEM, JEOL JEM2100F device) [23–25]. Studies were carried out in the rail head along the central axis at a distance of 10 mm from the surface.

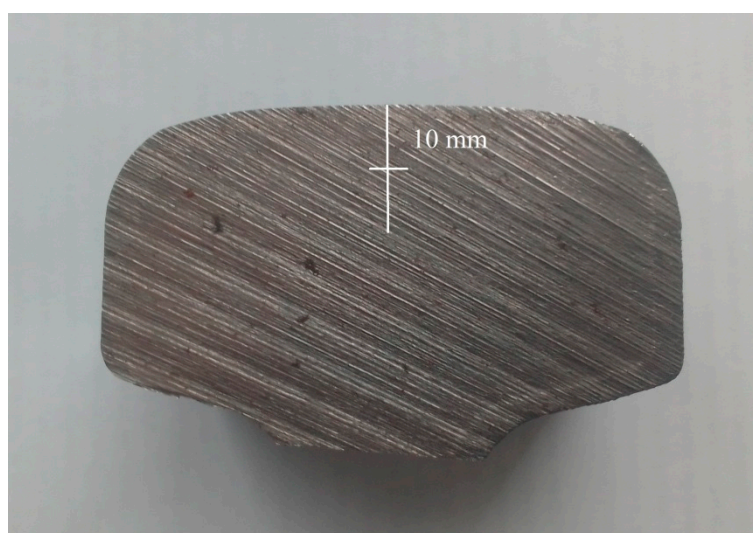


Figure 1. View of the rail sample after the passed tonnage of 187 million tonnes and the preparation scheme of the test object.

3. Results

The conducted studies showed that the steel structure is represented by three morphological components at a distance of 10 mm from the sample surface. The first morphological component is lamellar perlite, which is a group (colony) of alternating parallel plates of ferrite (α -phase) and cementite (Fe_3C). The TEM image of lamellar perlite is shown in Figure 2. It can be seen from the figure that the cementite particles have a lamellar shape and are located almost parallel to each other. The width of the α -phase plates (the distance between the cementite plates) is on average 125 nm, the average thickness of the cementite plates is 25 nm, that is, the steel under study is thin-lamellar pearlite. The volume fraction of lamellar perlite in the material is 65%.

Quite often, the plates of cementite in perlite are curved and separated by ferritic bridges (Figure 3). Thus, it is believed that ferrite is solid in perlite, and cementite is an intermittent phase consisting of separate plates [26,27]. The contrast along the cementite plate is heterogeneous, which indicates the disorientation of one part of the plate relative to the other.

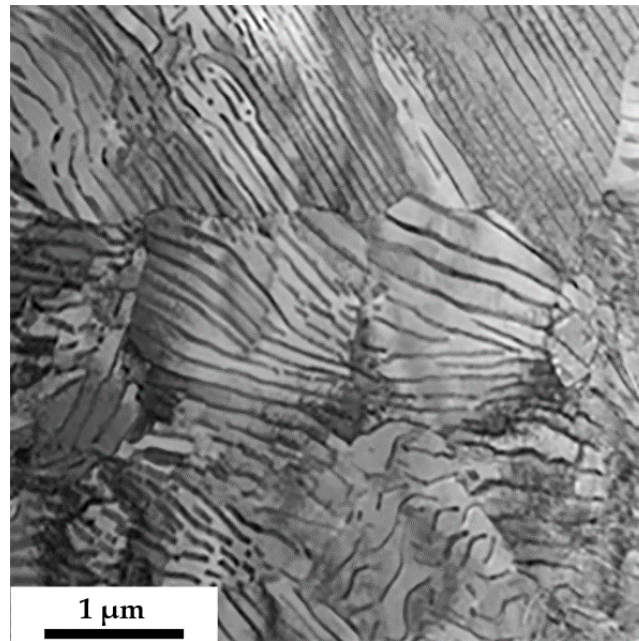


Figure 2. STEM image of the rail steel structure of hypereutectoid composition.

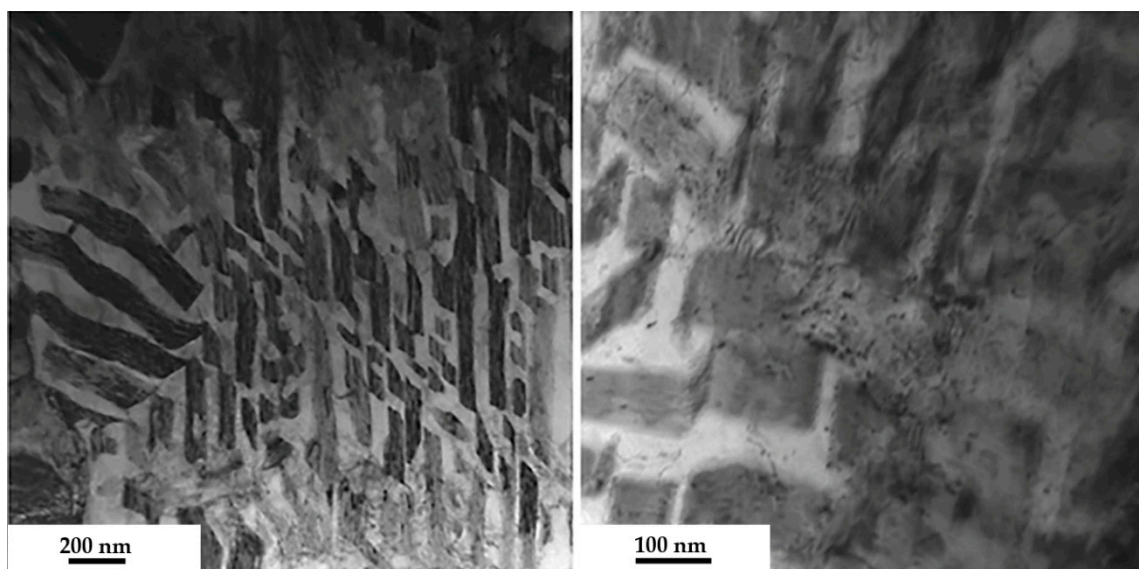


Figure 3. TEM image of cementite plates with ferrite bridges.

The methods of bright-field, dark-field and micro-electron diffraction analysis were used to study the defective substructure of cementite plates. It is established that long-term operation of rails leads to plastic deformation of steel, accompanied by the formation of dislocation substructure both in ferrite plates (Figure 4, a) and in cementite plates (Figure 4, b, d).

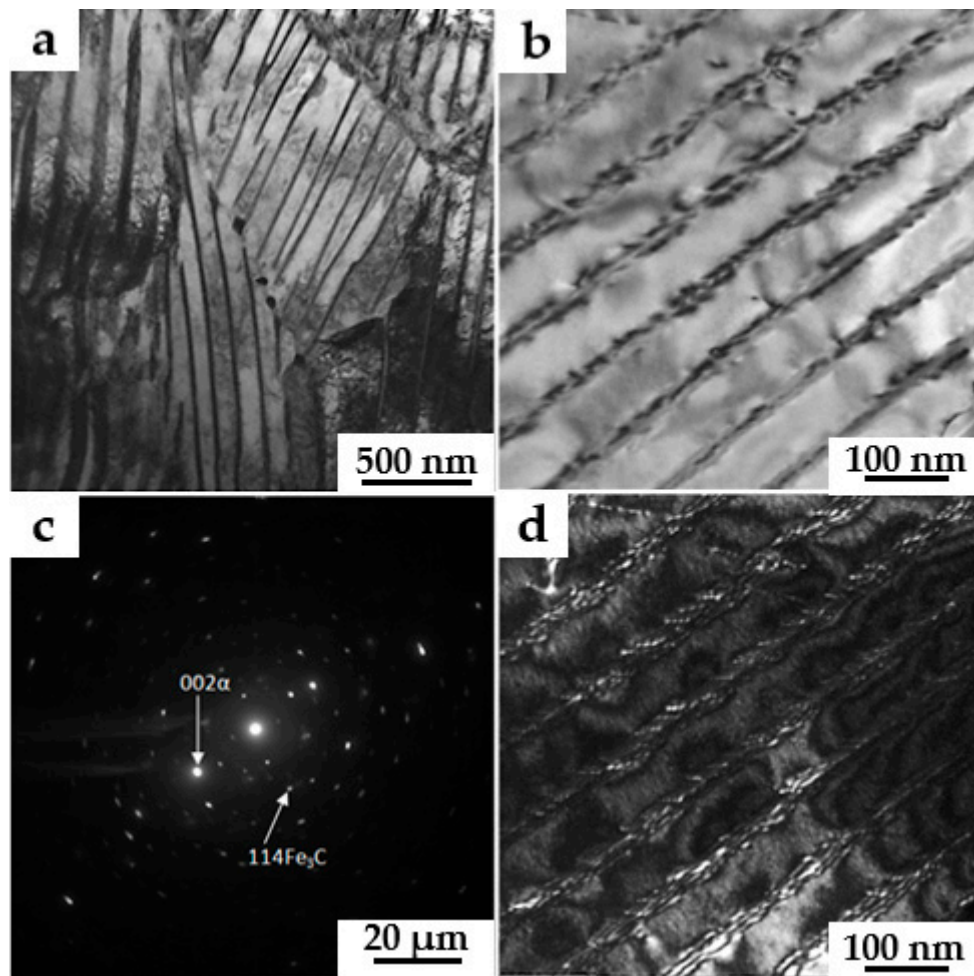


Figure 4. TEM image of the dislocation substructure of lamellar pearlite colony; a, b – bright fields; (c) microelectron diffraction pattern (b); (d) dark field obtained in 114Fe₃C reflection.

Dislocations in ferrite plates are distributed randomly or form dislocation substructure of a mesh type. In the cementite plates, dislocations are arranged in a more orderly manner, forming parallel rows oriented both across (Figure 4, b, d) and along the cementite plates. It can be assumed that the dislocation substructure in cementite plates is formed along the interface, thereby minimizing the elastic stresses arising in the material due to the difference in the mechanical and thermal characteristics of ferrite and cementite. The value of the average scalar density of dislocations is $\rho = 4.8 \cdot 10^{10} \text{ cm}^{-2}$. The dislocation structure in pearlite is polarized, as evidenced by the presence of bend extinction contours in it, the sources of which are the boundaries of pearlite grains, as well as cementite plates in pearlite colonies.

The second morphological component of the material is fractured lamellar perlite – it is areas or colonies of lamellar perlite with curved, cut and refined cementite plates. The volume fraction of this morphological component is 25% of the total volume of the material. The average particle size of cementite is $60 \times 230 \text{ nm}$, and their volume fraction is ~9%. The dislocation structure in destroyed pearlite is also mainly represented by dense dislocation networks. The average scalar density of dislocations is $3.9 \cdot 10^{10} \text{ cm}^{-2}$. The dislocation structure is also polarized.

The third morphological component is fragmented lamellar perlite. All fragments formed in lamellar perlite have a non-equiaxed shape – they are elongated along the cementite plates. From the sides, the fragments are limited by lamellar cementite precipitations, the average size of which is $25 \times 355 \text{ nm}$. The width of the fragments corresponds to the distance between the cementite plates, that is, it is equal to the width of the α -phase plate in lamellar pearlite. The length of the fragments is limited by the formed, fairly clear, dislocation walls, mainly oriented across the direction of the α -

phase plates. The average fragment size is 125×550 nm. The volume fraction of fragmented pearlite is 10% in the material volume.

At the ferrite-cementite interface, the dislocation density is higher than in ferrite plates. The analysis of TEM images shows that dislocations, by the nature of their distribution, can be conditionally divided into two systems. As in [26], it is noted that the dislocations of the first system are elongated within the ferrite layer in the direction from one interphase boundary to another, and for the second system, the dislocation sliding plane is perpendicular to the plane of the interphase boundary. Traces of the sliding plane of these dislocations can pass through the cement plate and penetrate into the adjacent ferritic.

As for rails made of hypoeutectoid steel [3,4,8,9], long-term operation is accompanied by the deformative transformation of plate pearlite grains, namely, the fracture of cementite plate. One of the main fracture mechanisms in cementite is its cutting by sliding dislocations. This mechanism for carbon steels was studied in detail in the works of the Ukrainian school of metallophysics [28–31]. In pearlite columns, the distribution of deformations is heterogeneous: the lamellar character of the structure is preserved in the areas where the local deformation is small.

The second mechanism of destruction of cementite plates consists in pulling carbon atoms out of the carbide phase lattice by dislocations during plastic deformation with the formation of Cottrell atmospheres due to a noticeable difference in the average binding energy of carbon atoms with dislocations (0.6 eV) and with iron atoms in the cementite lattice (0.4 eV). Carbon diffusion takes place in the stress field created by the dislocation substructure that forms around the cementite plate. In this case, the degree of decomposition of cementite should be determined by the magnitude of the dislocation density and the type of dislocation substructure.

At the initial stage of transformation, the cementite plates are enveloped by sliding dislocations, which is accompanied by splitting the plates into separated poorly oriented fragments. Then, a change in the structure of the carbide can occur because of the pulling the carbon atoms from the cementite lattice. This is due to the penetration of sliding dislocations from the ferrite crystal lattice into the cementite crystal lattice. At this stage of dissolution of cementite plates, the interfacial boundaries “ α -phase – cementite” play a special role. A coherent or semicoherent boundary [28–31] facilitates the penetration of dislocations from the α -phase into cementite and vice versa, and thereby contributes to the fracture and dissolution of cementite. An incoherent high-angle interphase boundary stabilizes the carbide structure and leaves only diffusion mass transfer possible. That is why the cementite plates in the pearlite colony are fractured, while the spherical particles of cementite at the boundaries of grains and subgrains are preserved. At the next stage of cementite dissolution, the entire volume previously occupied by the cementite plate is filled with nanoscale particles. The process of mass transfer of carbon atoms can be carried out by several mechanisms. First, it is interstitial diffusion [28–31]. This is a directed movement of a large number of vacancies between regions with different signs of internal stresses. Diffusion of carbon atoms over deformation vacancies is the second transfer mechanism. Under long-term operation of rails, the dislocation density goes up, fragmented structure develops intensively, misorientation of fragments and density of crystal lattice defects increase in the near-boundary regions and at grain boundaries [3]. Diffusion of carbon atoms can be accelerated over dislocation tubes, grain boundaries, and fragments. This will be the third transfer mechanism. It is known that the activation energy of diffusion over cores of dislocations is much less than that over the bulk of the material [28–31]. Of course, the specific gravity of all mechanisms in the process of cementite dissolution will depend on its structure, deformation conditions, and the degree of steel alloying. Dislocations can “lose” carbon atoms, most likely in the areas of solid solution with significant curvature-torsion of the crystal lattice. These areas are fixed after carbon gets there. Otherwise, the process should happen in the opposite direction – carbon should go from the solid solution to dislocations. In addition, nanosized particles of the carbide phase are also observed in the ferrite matrix, which occupies the interlamellar space of pearlite colonies. These particles can be introduced there in the process of dislocation slip, or, which is less likely, they were formed as a result of deformational decomposition of carbon solid solution in the iron crystal lattice.

Under severe plastic deformation of pearlitic steel at the last stage of cementite plates evolution, the formation of a misoriented quasi-strip structure based on α -Fe is possible [3]. At a high dislocation density in the α -matrix, Fe_3C carbide particles turn out to be the most stable. Aspects related to the movement of carbon atoms under conditions of intense deformation action during long-term operation of rails are of particular interest and require special mechanisms to explain them. These include the idea of the mechanism of plastic distortion, when due to the formation of the curvature of the crystal lattice, the material is able to experience significant shape changes without discontinuity [32].

Within the framework of physical mesomechanics, such a mechanism is interpreted as nanoscale mesoscopic structural states. This is a new approach in the mechanics of strength and plasticity of solids [32–34]. Since the rotational modes of plastic deformation are associated with the formation of local curvature of the lattice [7], it can be assumed that the development of this effect facilitates the movement of carbon atoms. Due to the cyclic nature of the load application, such a mechanism can develop back, which allows the elements of the internal structure to be rebuilt without the formation of discontinuities. It should be specially noted that this process does not have a diffusion character, since it develops at low temperatures, and the load is applied cyclically. On the other hand, a critical defect density accumulates in the surface layer of rails, which hinders the development of reversible elastic deformation and the involvement (development) of the plastic distortion mechanism. The formation of such a “critical” structure will end with the initiation of microcracks through the fatigue mechanism and failure of rails. For this reason, an increase in the service life of rails can be achieved by preserving the structure for as long as possible, capable of developing reversible deformation processes that exclude the cementite plates fracture.

The phenomenon of cementite dissociation during plastic deformation of high-carbon steels was observed in [35]. Carbon removal from cementite plates occurs most intensively near defects in ferrite and cementite. For ferrite, these are dislocation subboundaries. It can be assumed that the carbon concentration in ferrite increases insignificantly compared to the equilibrium one (as in hypoeutectoid steels [3,4]); the main proportion of carbon is not in solid solution, but in the form of small carbide precipitations and lattice defects.

Dislocations inside the cementite plate belong to planes, the trace of which in the cementite plate is a projection of the slip planes of dislocations in the ferrite plate. An accelerated dissolution of cementite was found near planar defects, which were present in the structure of cement plates of undeformed lamellar perlite. According to [26], such defects could appear during the eutectoid decomposition of austenite. Such dissolution leads to the formation of characteristic layering in the structure of a partially dissolved cementite plate.

According to [26], one of the mechanisms of carbide particle shape change is the formation of clusters or walls of dislocations crossing the carbide particle, which leads to an accelerated outflow of carbon near these defects into the ferrite matrix. The activation energy of carbon diffusion near dislocations is 2.5 times lower than in the volume of cementite. The formation of coil pile-ups of dislocations near ferrite/cementite interphase boundaries is another possible mechanism. In this case, the removal of carbon from the region with an increased density of dislocations leads to an effective displacement of interphase boundary inside the carbide particle.

In different ferritic plates, the presence of dislocations of opposite signs in the sub-boundaries may be the cause for alternation of diffraction contrast (contours of the excision) observed in TEM images during the transition from one ferritic plate to another. Similar places of formation of extinction contours and the causes of their occurrence for hypoeutectoid steels are analyzed in [3,4]. The presence of bend extinction contours in the TEM images indicates the bending-torsion of crystal lattice of the foil (formation of internal stress fields).

Plates of ferrite (Figure 5) and cementite (Figure 6) of pearlite colonies are fragmented, i.e. they are divided into regions with a low-angle disorientation. Fragmentation did not fracture the pearlite colonies and did not create chaos in the structure – the initial structure of the pearlite colonies was preserved. Inside the fragments, a well-developed network or cellular dislocation substructure is observed. The average scalar density of dislocations inside the fragments is $2.9 \cdot 10^{10} \text{ cm}^{-2}$, which is

even less than in unfragmented pearlite. This is in good agreement with the literature data, namely, the fragmentation of the material leads to a decrease in the scalar density of dislocations in the body of the formed fragments [36]. It is explained by the energy decrease in the dislocation subsystem during the formation of a fragmented substructure [37]. At the nodes of dislocation networks or cells, there are fine ($\sim 5 \times 15$ nm) cementite particles. Their volume fraction is small and amounts only to 0.16%. The analysis of microelectron-diffraction patterns makes it possible to estimate the azimuthal component of the angle of complete disorientation of fragments of cementite and ferrite plates by the blurring of reflections. It was found that the fragments of cementite are disoriented at angles (3.0-3.5) degrees. Disorientation of ferrite plate fragments is (2.5-3) degrees. It can be assumed that fragmentation of ferrite plates and cementite plates is another mechanism for relaxation of elastic stresses that occur during differentiated welding and subsequent long-term operation. For hypoeutectoid rails of the Paris metro with comparable values of the passed tonnage, the formation of fragmented structure was observed at a depth of ~ 4 mm from the rolling surface [38,39]. The resulting three-dimensional gradient of the microstructure may be the cause of microcracks.

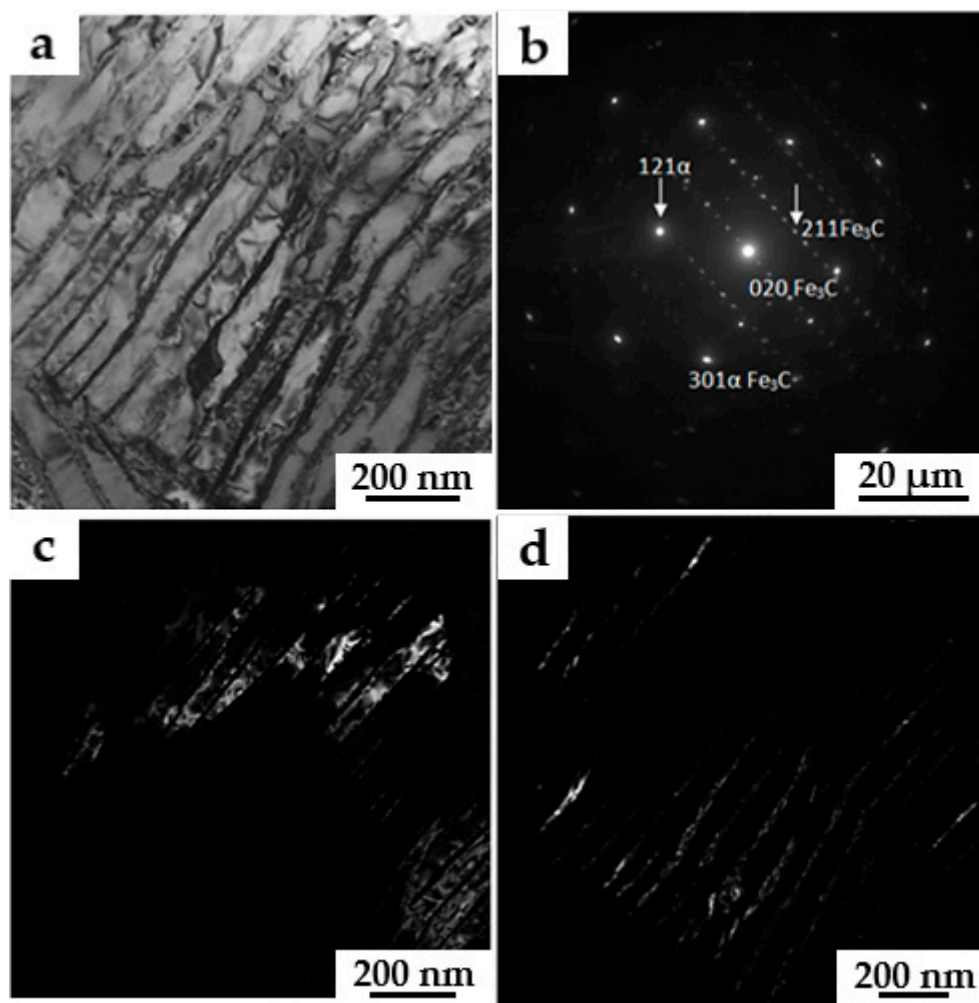


Figure 5. TEM image of the fragmented structure of lamellar pearlite colonies; a – bright field; b – microelectron diffraction pattern; c – dark field obtained in 121 α Fe reflection; d – dark field obtained in 211 Fe_3C reflection.

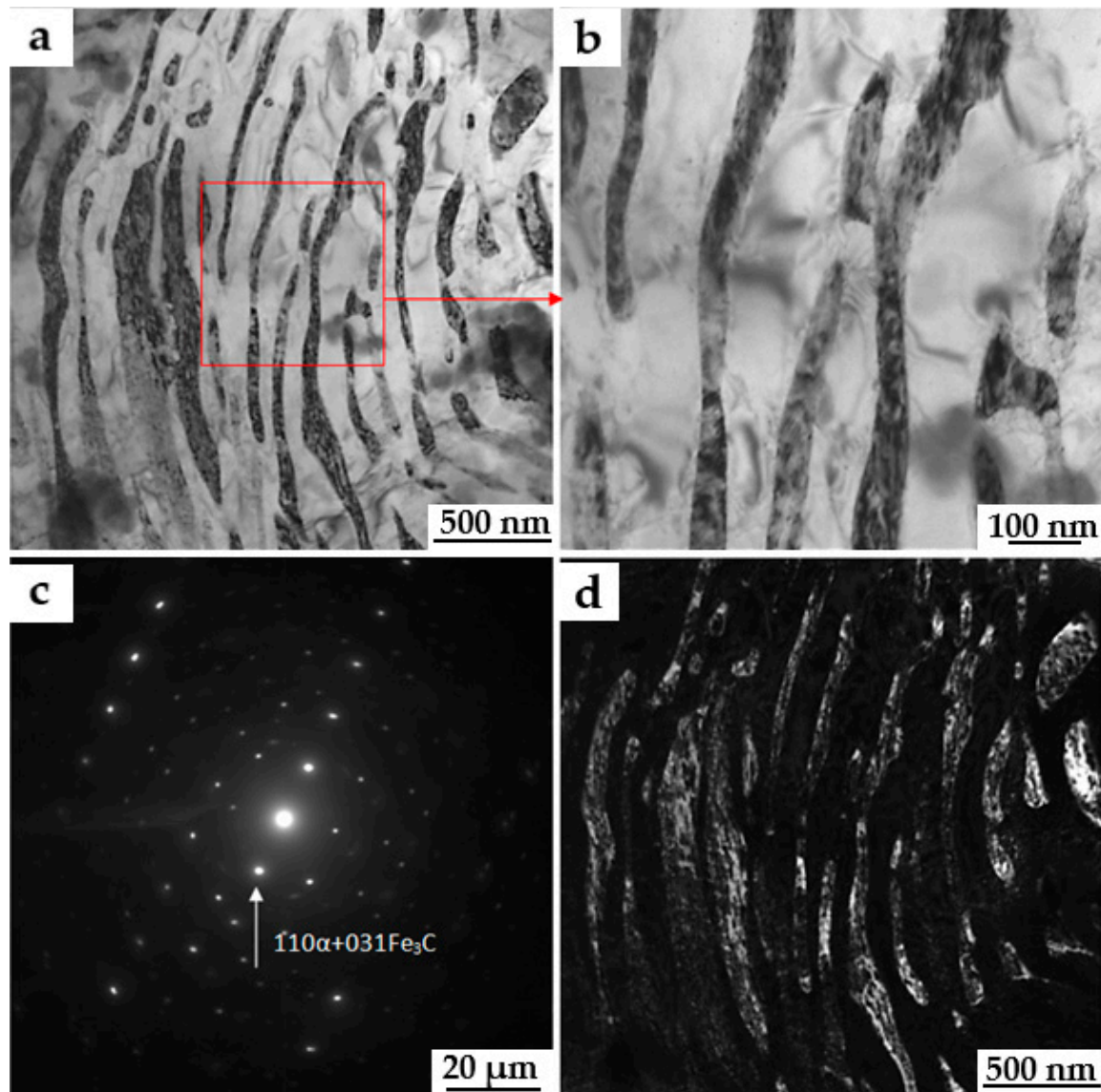


Figure 6. TEM image of fragmented structure of cementite plates; a, b – bright fields; c – microelectron diffraction pattern; (d) dark field obtained in $211\text{Fe}_3\text{C} + 110\ \alpha\text{Fe}$ reflection.

As already noted, thermomechanical processing of hypereutectoid steel and subsequent long-term operation lead to the release of nanoscale cementite particles (5-10 nm) in the volume of ferrite plates of pearlite colony (Figure 7). It can be assumed that this is tertiary cementite formed as a result of the decomposition of a supersaturated ferrite-based solid solution during the cooling of steel from the temperature of the pearlite transformation and subsequent long-term operation.

It was suggested in [26] that facilitating the outflow of carbon through defects can lead to the formation of such ferritic interlayers in cementite near such defective planes. The new interfacial surfaces, in turn, can be places of separation of nanoscale carbides. Nanoscale cementite particles are distributed unequally in ferritic plates, their density is greater in the locations of ferritic subgrains, near interfacial boundaries and other defects. In the dark field images, the diffraction contrast is heterogeneous in the thickness of the cementite plates due to the decoration of the interfacial boundaries with carbides.

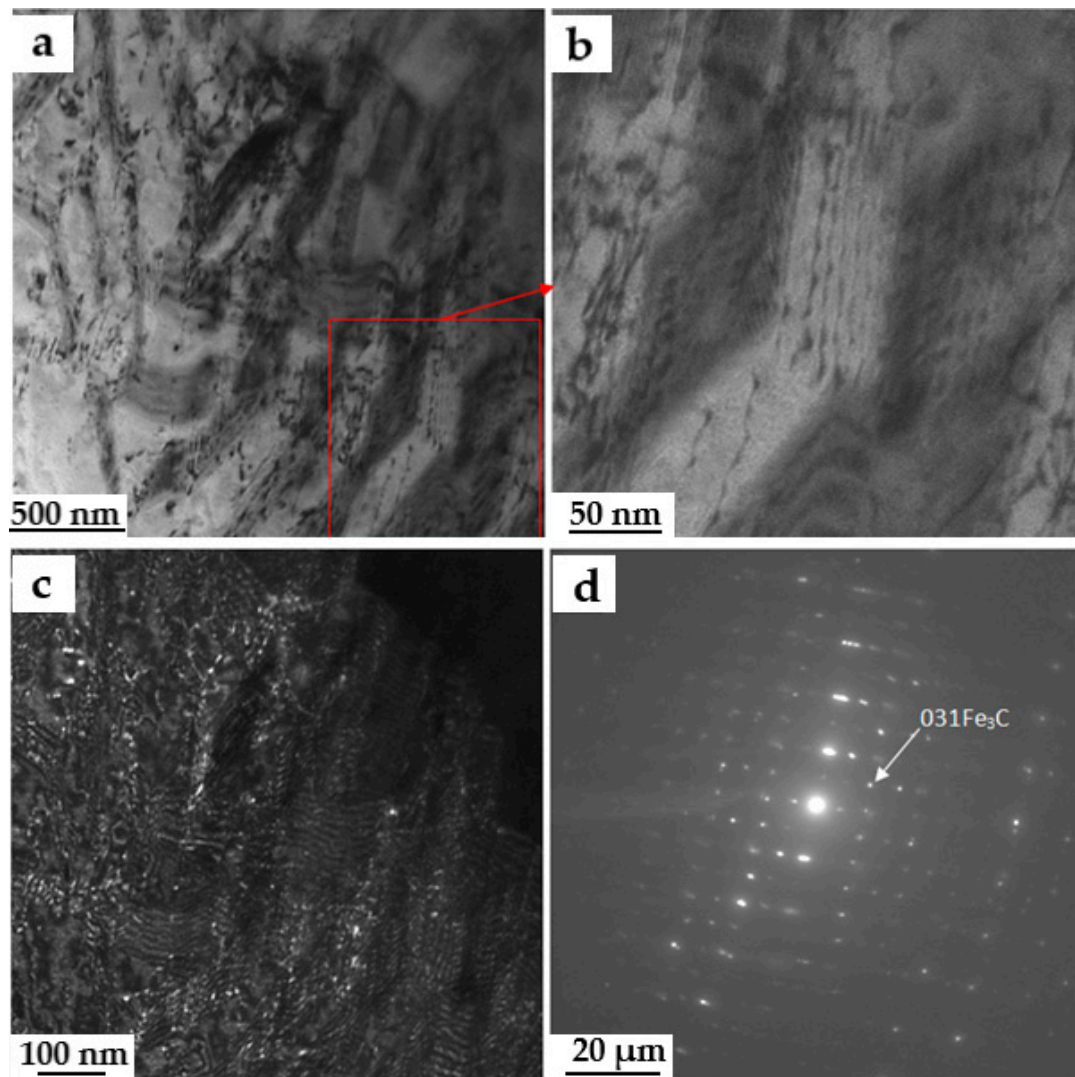


Figure 7. TEM image of nanosized cementite particles located in ferrite plates; a, b – bright fields; c – dark field obtained in 031Fe₃C reflection; d – microelectron diffraction pattern.

4. Conclusions

Long-term deformation action in the rails initiate complex processes that contribute to the degradation of structure and properties, and the removal of rails from operation. Obtaining information in this area is determined by the fundamental nature of the problem and its practical significance. Transmission electron diffraction microscopy methods were used to analyze the structure and phase composition of the rail steel of hypereutectoid composition. The research showed that at a depth of 10 mm from the rolling surface along the central axis, the structure of DT400IK steel after long-term operation is represented by three morphological components: lamellar, fractured and fragmented pearlite. It was found that the ferrite and cementite plates are fragmented, i.e. divided into regions with small-angle disorientation. Dislocation substructure is present in the volume of ferrite plates and cementite plates. The dislocation substructure of ferrite plates is represented by chaotically distributed dislocations or dislocation networks. Dislocations are arranged in parallel rows in cementite plates. It is suggested that the revealed defective substructure is a consequence of stress relaxation formed in steel during thermomechanical treatment and operation. It is established that the steel cooling from the temperature of pearlite transformation and subsequent long-term operation are accompanied by the decomposition of a ferrite-based solid solution followed by the formation of nanoscale particles of tertiary cementite. The formed nanosized particles of cementite are located unevenly in ferrite plates, most of them are concentrated at the locations of ferrite subgrains and interfacial boundaries. The possible mechanisms of deformation transformation of lamellar pearlite

are analyzed: the destruction of cementite plates by moving dislocations and the pulling out of carbon from the lattice of the carbide phase. The role of “ α -phase – cementite” interphase boundaries in the dissolution of cementite plates is noted. The features and mechanisms of mass transfer of carbon atoms over vacancies, interstitial sites, dislocation tubes, grain boundaries and fragments are considered. A comparison with experimental data for rails made of pre-eutectoid steels is carried out and the mechanisms of transformation of the cementite substructure are discussed. The results obtained can be used to analyze and correct the modes of thermomechanical strengthening of rails made of hypereutectoid steel, monitor the flaw detection of rails, justify the timing of scheduled works to check the condition of rails, and develop non-destructive testing methods.

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Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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