Atom probe study on microstructure change in severely deformed pearlitic steels: application to rail surfaces and drawn wires

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Abstract. Pearlitic steel is used as the material for high tensile steel wires, rails and wheels due to its high work hardening and wear resistance. These properties arise from a layered structure comprising deformable lamellar ferrite and hard lamellar cementite. This paper reviews the microstructural change in heavily drawn pearlitic steels wires and worn surfaces of pearlitic rails using atom probe tomography analysis. The cementite decomposition mechanism was elucidated for heavily drawn pearlitic steel wires. For pearlitic rail steels, atomic scale characterization of worn surfaces and of the white etching layer (WEL) were performed, and a mechanism for the formation of the WEL was proposed. The differences and similarities in microstructure and in the state of the cementite in these severely deformed pearlitic steels are discussed.

1. Introduction
Pearlitic steel exhibits high work hardening ability due to its layered structure comprising lamellar ferrite and lamellar cementite, wherein the former is a deformable soft phase and the latter is an undeformable hard phase. The microstructure of pearlitic steel is composed of pearlitic blocks (nodules) with the same crystallographic direction. Each block is composed of pearlitic colonies with the same orientation of pearlitic lamellae [1]. When pearlitic steel is drawn, these colonies rotate to be nearly parallel to the drawing direction and then the interlamellar spacing of pearlitic lamellae decreases. The increase in hardness and strength of pearlitic steels with reduction of the interlamellar spacing of pearlitic lamellae, and the yield strength of pearlitic steels has been explained by Hall–Petch relationship [2,3] or by an Orowan model [4], wherein the grain size corresponds to the interlamellar spacing.

High strength pearlitic steel wires are produced by heavily drawing high carbon pearlitic steels. Nowadays, steel wires with tensile strengths of greater than 4000 MPa have been commercialized by drawing hypereutectoid steel wire rods, and wires of tensile strength more than 6000 MPa can be produced experimentally [5]. High tensile strength wires are widely used for cable wires of bridges, wire ropes, saw wires, and steel-cords for tires. Developments of still higher tensile strength wires are required for weight reduction of automotive tires, which leads to a reduction of carbon dioxide emissions. However, the lamellar cementite forming the pearlitic structure is decomposed partially or fully by heavy drawing, and the mechanical properties of the wires are affected by the cementite decomposition [6,7]. The mechanism of the cementite decomposition has been discussed without a common consensus being reached [8].
On the other hand, pearlitic steels are also used for rails and wheels of trains, where high wear resistance is required [3,9]. It is reported that pearlitic steel has better wear resistance than steels with other microstructures, such as martensite, bainite, and ferrite containing spherical cementite [9]. However, the exact reason for the high wear resistance has not been sufficiently understood. The change in the microstructure and cementite state by wear needs therefore to be more thoroughly investigated. Furthermore, after use for a long time pearlitic rails often show a “white etching layer” (WEL) at the contact surface [10]. The WEL causes defects in the rail surface but the formation mechanism of the WEL has not been clarified.

We have investigated these traditional issues of pearlitic steel products by atomic scale characterization techniques using atom probe tomography (APT). In this paper we describe three research topics regarding the change in microstructure and cementite state of severely deformed pearlitic steel products [11–13].

2. Cementite decomposition of heavily drawn pearlitic steel wires [11]

2.1. Cementite decomposition

Cementite decomposition in cold-worked steels was first detected by Mössbauer spectroscopy in the 1970s [14]. Cementite decomposition in heavily drawn wires has been directly observed using transmission electron microscopy (TEM) and atom probe field ion microscopy (AP-FIM) since the second half of the 1990s [15–19]. It was reported based on APT analysis that most cementite was decomposed in drawn wires with true strains of 3.5–4.5 [16,17] and that the lamellar cementite was fully decomposed in wires heavily drawn to a true strain of 5.1 [18]. In contrast, Nam et al. reported using Mössbauer measurements that at most 50% cementite was decomposed in drawn wires with true strains of more than 3, and the ratio tended to saturate even as the true strain was increased, because of carbon saturation at dislocations located near the cementite/ferrite interfaces [20].

The proposed mechanisms for cementite decomposition can be classified into three hypothesis models. The first is the strong attractive interaction between carbon atoms and the high density of dislocations introduced by heavy deformation [6,19,20]. This interaction energy is larger than the binding energy between carbon and iron atoms in cementite (~0.8 eV/atom compared to ~0.5 eV/atom), and thus carbon atoms can move to the dislocations at temperatures where diffusion of carbon atoms is enabled. The second is the Gibbs–Thomson effect [15,17]. Due to the fragmentation of cementite by heavy deformation, the free energy of the interface of cementite increases and the local solubility of carbon increases, which leads to the decomposition of cementite. The third is the so-called “carbon drag effect.” In this model, moving dislocations during deformation carry the trapped carbon atoms because of the large attractive interaction between carbon and dislocations [8,21]. The dislocations generated from the interface of lamellar cementite move to the lamellar ferrite with trapped carbon atoms, and carbon atoms remain in the ferrite matrix by pairwise annihilation of the dislocations, and consequently highly supersaturated solute carbon is formed.

One of the reasons for the difficulty in reaching a consensus on which of these mechanism are most dominant is that the characterized wires in previous studies in general do not have the same drawing and aging conditions. In particular, heavily drawn wires were significantly influenced by aging at room temperature (RT) after drawing, and in most of the previous reports, not much attention has been paid to this unintentional aging. We have investigated the mechanism of cementite decomposition and the sites of carbon atoms after decomposition using sample wires where such unintentional aging can be avoided.

2.2. Preparation of sample wires

The material used in this study is composed of hypereutectoid steel with composition Fe-0.92C-0.48Mn-0.22Si mass% (Fe-4.13C-0.47Mn-0.42Si at.%). A wire rod with a diameter of 5.5 mm was cold-drawn with a powder lubricant to a diameter of 2.0 mm and heated to 950°C, followed by patenting in a lead bath at 580°C. The patented wire was further cold-drawn to 0.2 mm in a liquid lubricant to a true strain of \( \varepsilon = 4.61 \), wherein true strain \( \varepsilon \) is defined as \( \ln \left( \frac{A_i}{A_f} \right) \), where \( A_i \) and \( A_f \) are the initial and final cross-sectional areas, respectively. The speed of the final drawing was fixed at 30
m/min, which is significantly lower than that used in the commercial production process. The as-drawn wires were immediately cooled with dry-ice or immersed in liquid nitrogen, and afterwards were kept in a freezer below −30°C to prevent the wire from aging at RT. The aim of the lower-speed drawing and sample freezing is to distinguish between the heavy deformation effect during drawing and thermal effects after drawing. The as-drawn wires \((\varepsilon = 4.61)\) were also intentionally aged for 30 min at each temperature from 100°C to 400°C in 50°C intervals.

Figure 1 shows the change in yield and tensile strengths of sample wires as a function of aging temperature. The tensile strength of the as-drawn wire was about 4480 MPa. The tensile strength increased with low temperature aging and showed a maximum strength after aging at 100–150°C. The peak aging region is represented by the shaded area in Figure 1. For aging above the temperature showing the maximum strength, the tensile strength decreased strongly with increasing aging temperature. The maximum increment in tensile strength due to aging of the as-drawn wire was estimated to be approximately 150 MPa. In carbon steels, such a strengthening through low temperature aging is caused by dislocation locking by carbon atoms (so-called Cottrell atmosphere formation) [22], and the lowering of the strength after the peak aging is caused by recovery and/or recrystallization [23]. We discuss whether a similar interpretation is suitable for heavily drawn steel wires in terms of the change in the microstructure and carbon state. For this detailed characterization using TEM, FIM, and APT was performed in the sample wires indicated by arrows in figure 1.

2.3. TEM observations

Figure 2 shows example microstructures of sample wires \((\varepsilon = 4.61)\) with and without aging in the longitudinal section by TEM bright-field observation. Cementite lamellae and ferrite lamellae in heavily drawn pearlitic wires are preferentially aligned along the drawing direction, namely, the wire axis. The drawn pearlitic lamellar structure was not uniform in interlamellar spacing, and the thickness of the ferrite lamellae was in the range of 5–20 nm. Large internal strains and dislocations were observed in the lamellar ferrite. Distinct lamellar cementite was not observed using TEM. The lamellar ferrite exhibited a strong <110> texture along the wire axis, but each lamella showed different contrast, which indicates that each ferrite lamella has a different crystallographic direction around the axis. Differences in microstructure were unobservable in wires aged at temperatures below 200°C. In contrast, a recovered microstructure was observed in the wire aged at 350°C, in which the morphology of the ferrite lamellae changed to a slightly rounded shape.

2.4. APT and FIM analyses

Needle-shaped specimens for APT analysis were fabricated directly from the sample wires using a standard two-stage electro-polishing technique [24]. In the preparation method, the tip position of the needle specimen always corresponds to the center region of the wire. Consequently, APT analysis is conducted in the center region of the wire along the probing direction parallel to the drawing direction (wire axis). Figure 3 shows APT analysis results of the sample wires (a) before aging, and after aging at (b) 150°C, (c) 200°C, and (d) 350°C, wherein the representative 3D carbon maps and concentration profiles are shown. In the as-drawn wire (before aging), a high carbon concentration (>20 at.%) and a low silicon concentration in the lamellar cementite remain, and thus the lamellar ferrite maintains a
low carbon concentration. The results indicate that cementite decomposition hardly took place regardless of the heavy drawing ($\varepsilon = 4.61$).

![Figure 2](image-url)  
**Figure 2.** TEM bright-field images of longitudinal sections of the sample wires with (a) unaged, (b) 150°C aging, (c) 200°C aging and (d) 350°C aging [11].

In the wire aged at 150°C, which had the maximum tensile strength (peak aging), the carbon state significantly differs from that of the as-drawn wire. Carbon atoms are distributed almost homogeneously, with about 4–5 at.% in the analyzed volume, which concentration is almost coincident with the average carbon content in the wire. A similar special carbon distribution was reported using APT in a heavily drawn pearlitic wire of $\varepsilon = 5.1$ by Hono et al., where they suggested complete decomposition of cementite was due to the heavy deformation [18]. Such a uniform distribution of carbon atoms was observed in most of the analyzed volumes of the aged wire. The interlamellar spacing of the wire was estimated to be 5–20 nm, and thus it is considered that the analyzed volumes mostly include the region of the prior lamellar cementite and its interface. Therefore, the local carbon concentration was almost the same throughout the lamellar ferrite, the lamellar cementite, and their interface.

![Figure 3](image-url)  
**Figure 3.** 3D carbon maps and concentration profiles of the sample wires (a) unaged, and after aging at (b) 150°C, (c) 200°C, and (d) 350°C [11].

In the wire aged at 200°C, carbon atoms are enriched in a wide band region with a much lower carbon concentration than the 25 at.% stoichiometric concentration of cementite. Thus, the enriched...
region is considered to be a prior lamellar cementite or lamellar ferrite boundaries with dislocations. The carbon concentration in the ferrite matrix except the enriched region is much lower than that of the 150°C aged wire. In the wire aged at 350°C, which had tensile strength much lower than that of the as-drawn wire, small features with a high carbon concentration appeared along ferrite boundaries (i.e. prior lamellar cementite) and the carbon concentration at these features is more than 20 at.%, whereas the silicon concentration is low within these features and is slightly higher near their edges. These results indicate that small spherical cementite or carbide was generated along such boundaries, wherein silicon atoms were discharged by the partitioning. In contrast, the carbon concentration in ferrite was much lower than that of the wires aged at lower temperatures.

Figure 4 shows FIM images of the sample wires without and with aging (compare with figure 3). The FIM images cover a much larger region than the APT analysis region, wherein bright and dark areas correspond to ferrite and cementite lamellae, respectively [24]. The FIM images significantly changed with aging temperature, which supports the APT results. In particular, it was surprising that both the APT carbon map and the FIM image changed by low temperature aging at 150°C for 30 min.

**Figure 4.** FIM images showing the change in the carbon state of the sample wires (a) unaged, and after aging at (b) 150°C, (c) 200°C, and (d) 350°C [11].

2.5. Mechanism of cementite decomposition

The carbon distribution in drawn pearlitic lamellae in the wire changed significantly, depending on the aging temperature, namely, the degree of aging. In our experiment, under the low-speed drawing and sample freezing after drawing conditions, the cementite decomposition hardly took place just after drawing. However, through low temperature aging at 150°C, the lamellar cementite was significantly decomposed and an almost homogeneous carbon distribution with high concentration corresponding to the average carbon content was formed. The results definitely indicate that the cementite decomposition took place during aging after drawing and not during drawing. Thus, the carbon drag effect wherein the moving dislocations carry carbon atoms from cementite to ferrite lamellae during drawing is not the main mechanism of the cementite decomposition. The fact that carbon atoms moved during aging after drawing suggests the possibility that it is either the large dislocation-carbon interaction energy effect or the Gibbs–Thomson effect, as both these effects can be limited by the diffusion of carbon atoms.

Furthermore, by increasing the aging temperature, the carbon concentration in ferrite decreased and carbon enrichment at ferrite boundaries (prior cementite lamellae) took place. This phenomenon is unexpected because the movement of carbon atoms changed to the opposite direction between 150°C and 200°C. In order to understand the phenomenon, temperature dependent factors associated with the cementite decomposition are taken into consideration. Based on the Gibbs–Thomson effect [25], it is calculated that fragmented hemi-cylindrical cementite with a radius of 0.5 nm showed a carbon content of about 0.41 at.% at 150°C. This value is one order of magnitude lower than the observed carbon concentration of 4–5 at.% in lamellar ferrite (figure 4b). If a high concentration of defects is introduced in the cementite phase, the solubility of carbon is predicted to increase more because the free energy of cementite is effectively increased to a higher level. Such a solid solution state is unstable, and thus the precipitation of carbon clusters and/or carbides should be expected. However, such clusters were not observed in either the APT and TEM investigations. Therefore, the cementite decomposition cannot be explained by the Gibbs–Thomson effect alone. The main mechanism of
cementite decomposition is therefore presumed to be the interaction between carbon atoms and lattice defects introduced during heavy drawing.

2.6. Origin of the uniform distribution of carbon atoms

The experimental observations reveal that not only heavy drawing, but also aging, plays an important role in the cementite decomposition. Two factors are required for the decomposition of cementite: the first is diffusion of carbon atoms over a distance substantially greater than the interlamellar spacing, and the second is a large number density of more stable sites rather than the binding energy between carbon and iron in cementite [19,20]. The dislocations introduced by heavy drawing, as well as interfaces between lamellar cementite and ferrite, have been proposed as such stable sites for carbon atoms.

The 3D carbon maps of the wire aged at 150°C showed almost an homogeneous carbon distribution of 4–5 at.% throughout the lamellar ferrite, lamellar cementite, and their interfaces. The high spatial resolution of the atom probe technique can sufficiently discriminate carbon atomic positions when they are located at dislocations and/or at dislocation walls. The diffraction analysis with TEM showed that there were dislocation walls at the interfaces of the lamellar ferrite and at the small angle boundaries across the lamellar ferrite [19]. The homogeneous carbon distribution observed in the 150°C aging wire cannot be explained therefore by carbon enrichment at dislocation walls in ferrite and/or at interfaces within the lamellar ferrite.

It has been reported that plastic deformation introduces excess vacancies in steel [26,27]. Vacancies of the order of $10^{-4}$ per bcc lattice site at a strain of about 20% were estimated from thermal desorption spectroscopy of tritium, and the concentration of vacancies was found to increase with decreasing temperature and by increasing the given amount of strain [27]. In addition, it has been reported that the vacancies in steel have a large attractive interaction with carbon atoms and that the interaction energy (~0.85eV/atom) is sufficiently larger than the binding energy between carbon and iron in cementite [28]. The change in the carbon state in the experiment is therefore explained using the presence of a high number density of excess vacancies. Carbon migration over a sufficient distance is difficult at RT although a high number density of vacancies was introduced into the drawing wire. In the aging at 150°C, the carbon atoms are able to migrate and become trapped at the vacancies distributed homogeneously in the matrix ferrite. Therefore, the cementite decomposition proceeded significantly at 150°C aging. Carbon enrichment at the ferrite boundaries (locations of prior lamellar cementite) was observed in the wire aged at 200°C, and this temperature is almost coincident with the decomposition region of the carbon-vacancy complex [28,29]. The complexes were decomposed and the dissolved carbon atoms segregated at the boundaries, as shown in figure 3. X-ray diffraction measurements have already confirmed that the lattice parameter of the ferrite phase was not affected by the cementite decomposition [18], which supports our proposed model that carbon atoms segregate at the lattice defects such as vacancies, dislocations, and boundaries in ferrite. In the aging at higher temperatures, the carbon atoms segregate at the boundaries and precipitate as small spherical cementite along the boundaries (figure 3).

3. Rolling contact wear surface of pearlitic rail steel [12]

3.1. Wear resistance

The rails in modern railway systems are subjected to serious wear and rail corrugation from fast trains with massive axle loads that cause structural defects and modulation of the rail surfaces. In order to prolong rail life, rails that have high wear resistance must be developed. Increasing the hardness of rail materials has been studied as a means of improving wear resistance because the mechanism of rail wear is considered to be adhesive wear [30]. In addition, increasing the hardness of the contact surface through work hardening has improved wear resistance [3,9]. Pearlitic steel has high potential for the hardening of the contact surfaces due to its high work hardenability. To understand the mechanism, nanoscale characterizations of the worn surfaces have been performed.
3.2. Specimens produced by rolling-sliding wear tests

A hyper-eutectoid pearlitic steel with a chemical composition of Fe-1.0C-0.70Mn-0.40Si-0.25Cr mass% was used in this study. The steel was austenitized by annealing at 1100 °C for 5 min and was cooled to 600 °C at a cooling rate of 30 °C/s. The pearlite transformation was completed during isothermal holding at 600 °C for 5 min, and then the steel was quenched with nitrogen gas. The steel was fully pearlitic with interlamellar spacing of about 100 nm and a Vickers hardness of about 400 HV. Using cylindrical test specimens extracted from the sample steel, a two-cylinder rolling-sliding wear test was conducted on a Nisihara wear test machine capable of simulating the wear of the rail and wheel system [9,31]. The test conditions to simulate the extreme wearing conditions of the railhead side on curved tracks were as follows: maximum Hertzian contact pressure of 640 MPa (load of 666 N), pearlitic wheel specimen hardness of 380 HV, rotational speed of 800 rpm, and slip ratio of 20% (wheel rotation speed of 640 rpm). The rolling contact surface was cooled with compressed air (0.1 Nm³/min) to prevent temperature increases and to remove debris. The weight loss was measured to evaluate the wear property of the test specimens. The rate of weight loss was almost constant after more than $1 \times 10^5$ rolling cycles, i.e., a steady state was nearly achieved. Thus, test specimens with more than $1 \times 10^5$ cycles of wear were used in this study.

Figure 5 shows the hardness depth profiles estimated using either on a polished surface or on the contact surface [12]. The hardness significantly increases as the contact surface is approached from the depth range of 20–30 µm and reaches a value of more than 800 HV just beneath the surface. It should be noted however that the experimental data vary with the inhomogeneity of the microstructure.

3.3. Microstructure of contact surface layer

Figure 6 shows TEM bright-field images of a longitudinal section from the surface layer of a specimen after $1 \times 10^5$ wear cycles. The arrows indicate the contact surface. The low-magnification image (figure 6a) of the entire area shows that pearlitic lamellae were plastically deformed and are increasingly refined with decreasing distance from the contact surface. The thinned lamellae were elongated in the direction opposite to the rolling direction, which indicates that the microstructure was formed by metal flow of the surface material, based on the tangential force caused by sliding friction between the rail and wheel specimens [3,9]. The high-magnification image just beneath the contact surface (figure 6b) shows that the surface region contains very fine lamellae parallel to the surface. The interlamellar spacing on average reached nearly 10 nm at the surface. Different domains having slightly coarse lamellae were also observed in the image. These domains most likely arose from pearlite colonies with an initial lamellar structure of a different orientation. Moreover, the high-magnification image at 4 µm-depth below the surface (figure 6c) shows that deformed pearlitic lamellae of about 50 nm interlamellar spacing extend obliquely upward and that the lamellar ferrite contains dislocations and cell boundaries.

Figures 7a and 7b show TEM bright-field images and diffraction patterns of a longitudinal section and a cross section, respectively, of the surface layer in a specimen after $7 \times 10^5$ cycles of wear. The diffraction patterns were taken from a selected area of about 500 nm diameter. The longitudinal section image shows clear fine lamellar structures parallel to the contact surface, as seen also in figure 6. In contrast the cross section image reveals shows a more broken up morphology rather than lamellar
structures. The discontinuous contrast in the images of the elongated ferrite lamellae suggest that subgrain boundaries and/or large strains are present within each lamellae. The two diffraction patterns suggest that the deformed ferrite lamellae have strong [110] crystallographic texture along the elongating direction, with random rotation around the [110] axis. Such a texture was also observed in heavily drawn pearlitic wire [11,15,16]. Figure 7c shows a schematic of the grain structure just beneath the contact surface.

![Figure 6. TEM bright-field images of a longitudinal section of the rolling-sliding wear surface layer; (a) entire area (0–15 μm depth), (b) contact surface region, and (c) 4 μm-depth region [12].](image)

![Figure 7. TEM bright-field images and diffraction patterns from (a) cross section and (b) longitudinal section of the rolling-sliding wear surface layer. (c) Schematic of the rolling contact surface [12].](image)

The bright-field and dark-field TEM observations were not able to reveal the presence of cementite lamellae just beneath the contact surface. Therefore, the carbon distribution of the surface layer was investigated by APT. For this a needle tip was produced from the topmost surface in the direction normal to the contact surface by a FIB lift-out method. Figure 8a shows a FIM image of the region just beneath the contact surface in the specimen with $1 \times 10^5$ cycles of wear. The image mostly consists of regions of alternating bright and dark concentric rings, which suggests that ferritic lamellae pile up in the needle direction. However, the bright rings are divided by extra dark bands corresponding to boundaries, which indicate that the thinned ferritic lamellae are partially refined, as suggested by the TEM observations. Figure 8b shows the APT analysis results at the 100–200 nm depth region. The
arrow indicates the direction of the contact surface. The carbon map shows that carbon-enriched regions forming plate-like structures appear at intervals of 5–20 nm, which correspond to thinned cementite lamellae deformed by the rolling-sliding wear test. The concentration profile of carbon atoms in a box selected nearly perpendicular to these regions shows peak concentrations of carbon are in the range of 15–25 at.%. The high carbon concentrations near the stoichiometry of 25 at.% for the carbon-enriched regions indicate that the lamellar cementite is slightly decomposed but most lamellar cementite is retained as thinned lamellae. In addition, there is an enrichment of chromium and manganese atoms in these regions, whereas silicon atoms are denuded in the same regions. Such a distribution of alloying elements is caused by partitioning during the pearlite transformation. Based on these results it can be concluded therefore, that a mixing of alloying elements with the iron matrix and/or a sufficient diffusion of alloying elements did not occur during the rolling-sliding wear testing.

3.4. Hardening mechanism of the contact surface
In rolling-sliding wear, the microstructure of the contact surface is formed by the rate balance between the removal of surface material and the deformation of core material. Thus, a worn contact surface in a steady state was characterized in this study. The contact surface after the wear test was twice as hard as initially, which leads to better wear resistance in pearlitic steels. TEM observation showed that interlamellar spacing was reduced as the contact surface was approached, and it reached about 10 nm just beneath the surface. APT showed that the lamellar cementite in the deformed pearlitic structure mostly existed as thinned lamellae. Therefore, the increase in hardness in the surface layer can be explained basically by refinement of pearlitic lamellae. It has been reported that the hardness of pearlitic steels obeys the Hall–Petch rule written by

\[
H_v = 150 + 2.15 S_t^{-1/2},
\]

where \(S_t\) (mm) is the interlamellar spacing [3]. For spacing of about 10 nm, the hardness of the contact surface is estimated to be 830 HV, which is nearly equal to the result of the Vickers hardness tests (figure 5). Furthermore, the TEM and FIM results suggested that the thinned ferrite lamellae just beneath the contact surface were refined by the boundaries. The refinement of the thinned lamellae and the presence of high-density dislocations should further increase surface hardness [32]. However, the exact hardness of the surface layer within 400 nm, corresponding to the topmost surface, is not measured in this study because the depth of indenters normally exceeds 1 \(\mu\)m. The surface hardness within 400 nm is predicted to be much higher than 800 HV [33].

In conclusion, a plastically deformed fine pearlitic structure with interlamellar spacing of about 10 nm was formed just beneath the contact surface by the rolling-sliding wear test. The reduction of the spacing considerably increased the hardness of the contact surface. However, lamellar cementite was sufficiently retained as thinned lamellae because of low temperature deformation.

Figure 8. (a) FIM image of the region just beneath the contact surface. The tip direction is perpendicular to the contact surface, (b) 3D elemental maps and carbon concentration depth profile of the region just beneath the contact surface [12].
4. Origin and formation mechanism of WEL in rail track surface [13]

4.1. WEL in rail track surface
The WEL is one of the defects caused in the surface of rail tracks [10,34–39]. It is a very hard thin layer and is considered to be a source of microcrack formation. Much researches has been conducted on the origin and the formation mechanism of WEL in the past decades. Newcomb et al. [34] first pointed out that although the WEL has a martensitic microstructure, it is formed by cementite decomposition without austenization due to repeated shear fatigue. Österle et al. [37] and Stadlbauer et al. [39] reported that martensite is formed after austenization due to a temperature rise caused by frictional heating at the rail-to-wheel contact areas. In contrast, Baumann et al. [35,36] reported that the WEL is composed of nano-ferrite crystals or is in the process of nano-ferrite crystallization caused by repeated severe plastic deformation. There still remain various questions and uncertainties therefore regarding the origin and formation mechanism of the WEL. In this study, we investigated the origin and formation mechanism of the WEL in terms of the distribution of alloying elements in the WEL using APT analysis. We also discuss the difference in the microstructure and the cementite state between worn surfaces and in the WEL.

4.2. Sample rail
For this study a sample rail having a WEL on its surface was extracted from a high speed rail track. The material was JIS60k pearlitic steel rail, made in 1984, and the accumulated passing tonnage was estimated to be about 150 million tons. The chemical composition of the rail was Fe-0.70C-0.93Mn-0.23Si-0.03Cr mass% (Fe-3.37C-0.46Si-0.95Mn-0.03Cr at.%). Figure 9a shows an image of the sample rail. The WEL can be visually recognized as the band of a slightly different contrast elongated in the track direction. This band, of about 5 mm in width, was located near the center of the rail head, at which location the rail and the wheel make contact. Figure 9b shows an optical micrograph of the surface of a longitudinal section perpendicular to the rail track direction after nital etching. The white featureless contrast region in the pearlite matrix corresponds to the WEL stripe, with thickness and width in the range of 20–30 µm and 300–500 µm, respectively.

Figure 9c shows micro-Vickers hardness test results for the WEL and pearlite matrix regions of the rail sample, wherein the dashed line represents the hardness of the quenched martensite of the same material. The transverse section parallel to the track direction after nital etching was used in the test. In
the figure the numerical number near each mark represents the hardness (HV) in that region. The hardness of the pearlite matrix is approximately 300 HV and it slightly increases close to the interface between the WEL and the matrix. The hardness discontinuously increases at the interface and reaches around 1000 HV. The value is higher than the hardness (840 HV) of the quenched martensite. It should be noted that the hardness within the WEL is dependent on the depth position [40].

4.3. TEM observation
Figure 10 shows cross-sectional TEM bright-field images of the surface region containing both the WEL and the matrix. A low-magnification image (figure 10a) shows the entire area containing the WEL, the matrix, and their interface region. The arrowheads indicate the approximate interface position. The microstructure of the WEL region is completely different from that of the matrix pearlite. Figures 10b and 10c show high-magnification images and selected area diffraction patterns of the matrix and WEL regions, respectively, from the locations indicated in figure 10a. The microstructure of the matrix shows a slightly deformed pearlitic structure with an interlamellar spacing of about 200 nm, and with high dislocation density in the ferrite lamellae. In contrast, the WEL region consisted of fine grains of a few hundred nanometers with very high dislocation density. The diffraction pattern, using an aperture diameter of 500 nm, shows discrete diffraction spots, not indicative of nano-ferrite crystals, and no lamellar cementite was observed in the region. Such a microstructure is very similar to lath martensite although the grain size is smaller than in a normal martensitic structure.

4.4. APT analysis
Figure 11a shows the results of APT analysis of a volume at the topmost surface of the rail sample but outside the WEL region. The analysis position was about 1 mm away from the WEL band and 5 μm beneath the surface. The arrow represents the surface direction. The 3D elemental maps indicate definite pearlite features with lamellar cementite of 25 at.% carbon, suggesting that decomposition of lamellar cementite does not occur. In the cementite lamella, manganese is enriched and silicon is depleted due to the partitioning of these alloying elements [41]. The cementite lamella seen here is about 12 nm in thickness, which is comparable to those in the initial pearlite. This suggested that lamella thinning due to heavy deformation did not proceed to any considerable extent regardless of the topmost surface.

Figure 11b shows the results of APT analysis of a volume at the topmost surface (depth 2 μm) inside the WEL. From the carbon map it is seems that cementite lamellae were thinned by heavy deformation. However, the peaks of carbon concentration are in the range of 5–10 at.%, which is much lower than the cementite composition 25 at.%. A manganese-enriched and silicon-depleted zone is
observed in the lower part of the box, which corresponds to a prior cementite lamella, although no cementite exists in this region. Therefore, the carbon-enriched layers in the carbon map are not deformed cementite lamellae but carbon segregation to boundaries and/or defects. In the WEL, the lamellar cementite is completely decomposed and carbon atoms are redistributed throughout the ferrite lamellae in solid solution and by segregation to defects. The manganese-enriched and silicon-depleted zone is slightly diffuse but its thickness indicates that lamella thinning does not significantly proceed even in the WEL. Fine carbide precipitates were also observed at a different location of the same depth region inside the WEL.

Figure 11c shows the APT analysis result for the lower region inside a WEL (depth 25 µm), just above the interface between the WEL and the matrix, at which location a maximum hardness was found. The solute carbon concentration in the bottom region is higher than that in the topmost surface regions. The manganese-enriched and silicon-depleted zone is also much more distinct than seen in the topmost surface regions, which indicates that the diffusion length of manganese and silicon atoms (substitutional atoms) in the bottom region was smaller than in the topmost surface region after the decomposition of the lamellar cementite. The APT analysis result of the matrix region just beneath the interface (depth of 30 µm) showed the presence of deformed pearlite with high dislocation density as observed with TEM. The elemental maps indicated typical pearlitic features with the cementite lamellae having the carbon composition of around 25 at.%

![Elemental Maps and Concentration Profiles](image)

Figure 11. 3D elemental maps and concentration profiles of (a) the topmost surface of the matrix region outside the WEL (depth 5 µm), (b) the topmost surface inside the WEL (depth 2 µm) and (c) the lower region inside the WEL near the interface (depth 20 µm) [13].

4.5. Origin and formation mechanism of WEL

It was found that the cementite lamellae in the WEL decomposed completely and carbon atoms distributed throughout the lamellae in the forms of solid solution, segregation and precipitation. However, manganese-enriched and silicon-depleted zones corresponding to prior cementite lamellae were also observed, and the manganese-enriched zone at the bottom of the WEL was much more distinct than those at the topmost surface regions. It was found that lamella thinning did not considerably proceed at the surface around the WEL, which implies that the surface region did not undergo heavy deformation in the sample rail. These results suggest that the change of microstructure is caused not by severe plastic deformation but probably by phase transformation due to frictional
heating. Therefore, the temperature change of the rail surface regions was investigated by thermal distribution calculations of frictional heating at the rail-to-wheel contact areas using the finite element method [42].

For the model the main calculation parameters an axle load of 10000 kgf \((9.8 \times 10^4 \text{ N})\) per wheel, a contact length and width both of 10 mm, and a train speed of 30 m/sec \((108 \text{ km/hour})\). The dynamic friction coefficient was taken to be 0.4, a value one order higher than the rolling contact friction coefficient [43]. Figure 12 shows the temperature profiles calculated at different depth positions below the contact surface as a function of the time from the contact start. The temperature at each depth position increases rapidly at the contact start point and reaches a peak value at the contact end point. The temperature falls exponentially with the time after contact ceases, mainly due to thermal conduction as the rail functions as an efficient heat sink. The highest achieved temperature increased with decreasing distance from the contact surface. The rate of temperature rise was estimated to be \(4 \times 10^6 \degree \text{C/s}\), which is significantly higher compared with normal heating methods. The time at which high temperatures were maintained was estimated to be approximately 0.5 ms, more or less corresponding to the duration of wheel-rail contact.

Temperatures at the depth positions of 2 \(\mu\text{m}\) and 20 \(\mu\text{m}\) increased considerably above the \(A_1\) point, i.e., the thermal equilibrium austenization temperature \((727\degree \text{C in Fe-C alloy})\), and the temperature at the depth position of 60 \(\mu\text{m}\) almost reached the \(A_1\) point. The actual austenization temperature is considered to be higher than the \(A_1\) point. The result suggests that the austenization border region, namely, the interface between the WEL and the matrix, appears clearly at depth positions of less than 60 \(\mu\text{m}\). In the calculations, however, the achieved temperature strongly depends on the parameters used. Therefore, it is difficult to quantitatively determine the temperature rise only from calculations because the calculation parameters are not well known. Evidence of the achieved temperatures based on the experiment results was therefore sought to enable a meaningful and quantitative discussion of the temperature rise.

For this we focused on the concentration profile of the manganese-enriched zone. The higher the temperature achieved and the longer the time after the decomposition of lamellar cementite, the more diffusive the concentration profile appears to be, due to the long diffusion length of manganese atoms. Diffusive concentration profiles of the manganese-enriched zone were not observed in the deformed pearlitic structure in which cementite lamellae remained (figure 11a). Thus, the actual achieved temperature can be estimated by analyzing the degree of change in the concentration profile of the manganese-enriched zone based on the diffusion length in austenite.

![Figure 12. Temperature profiles of various depth positions obtained by frictional heating calculation at rail-to-wheel contact areas [13].](image-url)

Figure 13a shows a schematic explaining the change in atomic concentration profile caused by diffusion, wherein the initial concentration profile is assumed to be a rectangle with the height \(C_0\) and width \(2h\) of the enriched zone [44]. The number of each line represents the ratio of the diffusion length to the zone width \((Dt)^{1/2}/h\), where \(D\) and \(t\) are the diffusion coefficient and time, respectively. As time passes, the rectangle changes to a diffusive shape. Figure 13b shows the actual concentration profile of the manganese-enriched zone, perpendicular to the prior cementite lamella in the topmost surface region within the WEL (figure 11b). With the assumption that \(C_0\) is 3.6 at.\% and \(2h\) is 12 nm from the
analysis result of the pearlite matrix, the value of \((Dt)^{0.5}/h\) is estimated to be nearly 3/4. The value indicates that the diffusion length \(2(Dt)^{0.5}\) is approximately 9 nm at the topmost surface. From the calculations presented in figure 12, the maximum achieved temperature is estimated to be about 1300°C. Table 1 summarizes the achieved temperatures estimated by the shape analysis of the manganese-enriched zones observed at different depth positions. The achieved temperatures are in the range of 1300–1400°C at the topmost surface (depth of 2 µm) and lower than 1150°C at the bottom position (depth of 25 µm) inside the WEL. The results indicate that the temperature of the WEL region can increased significantly above the austenization temperature and that the achieved temperature increases more closer to the contact surface. The achieved temperatures estimated from our experiments support therefore the temperature rise profiles predicted by the frictional heating calculation.

| Region | Depth position (µm) | Mn peak concentration (at.%) | Shape | \((Dt)^{0.5}/h\) | \(2(Dt)^{0.5}\) | Estimated achieved temperature (°C) |
|--------|---------------------|-----------------------------|-------|-------------------|----------------|-----------------------------|
| WEL 2  | 2.7 ± 0.16          | Diffused                    | ~3/4  | ~9                | ~1300          |
| WEL 2  | 2.2 ± 0.14          | Diffused                    | ~1    | ~12               | ~1400          |
| WEL 25 | 3.6 ± 0.18          | Slightly diffused           | <1/4  | <3                | <1150          |
| Pearlite | 3.6 ± 0.18       | Not diffused                |       |                   |                |

Speich et al. [45] reported from experiments on rapid heating using laser radiation that the growth rate of austenite into pearlite was nearly 479 nm/ms at 829 °C in pearlitic steel of 0.96 mass% carbon and that the rate increased with increase in temperature. Furthermore, Ganeev [46] reported that the irradiation of a pulse laser with a pulse width of 6 ns and frequency of 20 Hz hardened a surface region of 3–8 µm in depth in pearlitic steel of 0.6 mass% carbon, which suggested that the nucleation of austenite occurred within the pulse width of 6 ns. These reports indicate that the nucleation and growth of austenite in pearlitic steel are sufficiently fast to complete the austenite transformation in the period during which a high temperature is maintained by frictional heating.

Based on the above we conclude that the WEL was generated by a martensitic transformation after rapid austenization, resulting from a temperature rise due to the contact frictional heating. It is known that the rail contact surfaces undergo heavy deformation and are subjected to hydrostatic pressure due to the passage of trains in addition to heating [47]. In our proposed model of the WEL formation, heavy deformation due to the passage of trains was not taken into consideration. Such a heavily deformed pearlite seems to transform more easily to austenite kinetically than undeformed pearlite because (1) the density of cementite interfaces that can act as nucleation sites of austenite grains increases and (2) the austenite temperature decreases further with an increase in the carbon
concentration in ferrite. However, significant thinning of the pearlitic lamellae was not observed in the WEL region in our sample. This fact indicates that the transformation to austenite could take place in the surface region without requiring heavy deformation of pearlite.

Figure 14 shows a schematic explaining the change in hardness on the cross-sectional surface of the rail according to our proposed model. First, the passage of trains produces plastic deformation in the rail surface region due to the load from the trains, and consequently, the hardness of the surface region slightly increases due to work hardening. In rare case that the special condition of rail-to-wheel contact is satisfied, large frictional heating locally increases the surface temperature above the austenization temperature. The WEL is then generated in the contact surface region wherein martensite transformation occurs due to rapid cooling. The hardness of the WEL region increases uniformly just after the WEL formation. Then, the further passage of trains causes the surface region to warm up due to rolling contact friction and plastic deformation, which leads to a tempering of the WEL martensite in the surface region. The hardness of the surface region is thus lowered, depending on the degree of tempering. The final hardness profile observed in the sample rail is explained well by the model.

Figure 14. Schematic explaining the hardness change of the rail surface region due to WEL formation and tempering [13].

5. Microstructure change in pearlitic steels.
Very thin pearlitic lamellae of 5–20 nm in interlamellar spacing were observed both in the heavily drawn pearlitic steel wires and in the topmost wear surface of pearlitic rails. The reduction of the interlamellar spacing results in the high work hardening of pearlitic steels, which leads to high strength and high hardness, resulting in high wear resistance. The microstructures of the drawn wires and the topmost wear surface of the pearlitic steels were very similar, wherein the deformed ferritic lamellae have a strong [110] crystallographic texture along the elongation direction, with a random rotation around the [110] axis, and where most of the lamellar cementite retained in the form of thin layers when aging is prevented by keeping the material at a low temperature. Cementite decomposition proceeded during aging after drawing in the heavily drawn pearlitic steel wires. These results suggested that cementite decomposition results in a strong attractive interaction between carbon atoms and defects in steels. Therefore, the following two factors are required to enable cementite decomposition: (i) diffusion of carbon atoms over a distance substantially greater than the interlamellar spacing, and (ii) a large number density of more stable sites with lower energy compared to the binding energy between carbon and iron in cementite.

On the other hand, the WEL seen in rail-track surfaces was completely different from the microstructure of the deformed pearlitic steels. The experimental observations and analysis suggest that the WEL is generated by a martensitic transformation after rapid austenization, resulting from a temperature rise due to the contact frictional heating. The final microstructure and hardness characteristics of the WEL can be explained by additional tempering effects due to the further passage of trains after formation of the WEL. The likely temperature rise, predicted by finite element calculations, was actually confirmed by analysis of the distribution of manganese in prior lamella cementite, based on a diffusion model. It is considered that WELs are generated under special contact conditions when a sufficiently high load transfer takes place locally and/or the contact area becomes
extremely small during the passage of a high speed trains. Such a condition may also be satisfied when the brakes of a train traveling at a high speed are applied.

6. Summary
In this paper we have reviewed a number of APT investigations of industrially important pearlitic steel products. The change in microstructure and state of cementite in the pearlitic steels significantly influences the properties in each case. An understanding of the mechanisms responsible for the differences in microstructure, and their change during operation, as well as their properties, is expected to bring improvements not only of pearlitic steel products in current use, but also in materials design for achieving high strengthening.

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