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Microstructure Control and Strengthening of High-carbon Steel Wires

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Abstract

Nippon Steel Corporation has developed various wire rods for high strength steel wires in order to satisfy the requirements of strengthening of steel cord, steel wires for bridge cables and PC strand. The nano order microstructure control technique which is the key to the strengthening of high carbon steel wires, and the current state of high strength steel wires were outlined. It is important to clarify the microstructural change of steels wires due to heavy deformation and the effect of cementite decomposition on delamination occurrence for the purpose of achieving the further strengthening of high carbon steel wires. Recent results of such a basic research field were presented as well.

1. Introduction

High-carbon steel wires produced through drawing of high-carbon pearlitic steel wires have the highest strength of all mass-produced steel materials. The high-carbon steel wires are an important industrial material used for a wide variety of applications such as steel cords for reinforcing automobile tires, galvanized wires for suspension bridges and PC wires. The strength of high-carbon steel wires has been enhanced remarkably over the last years; it is one of the most advanced steel materials in terms of pursuit of the ultimate strength of steel material. However, many aspects related to the properties of high-carbon steel wires still remain unclear. Such aspects include the microstructural change resulting from wiredrawing, significant work hardening properties characteristic of pearlitic steels and the mechanism of the occurrence of delamination, one of the toughest obstacles to strengthening of the product.

In addition, reports on some of the latest studies have disclosed new facts such as the following[1-7]: cementite (hereinafter referred to as θ) decomposes as a result of wiredrawing in a greater extent than hitherto believed, and under certain drawing strain conditions, C exists in ferrite (hereinafter referred to as α) in a quantity far exceeding the limit of its solid solution; and θ decomposes virtually completely in a high-carbon hypereutectoid steel wire that has undergone heavy wiredrawing. Since the mechanical properties of α change depending on the concentration of C, as those of martensite do, one can expect the decomposition of θ to affect the work hardening properties, delamination properties and other properties of the product.

The object of this paper is to introduce the results of the latest fundamental research envisaging further strengthening of high-carbon steel wires such as tire steel cords, galvanized suspension-bridge wires and PC wires. Described herein are the current state of nanometer-level technologies to control the microstructure and strengthen product steel wire, the microstructural change of the product caused by heavy wiredrawing and the relationship between the decomposition of θ and the occurrence of delamination.

2. Experimental Procedures

Chemical composition ranges of specimens used in the present study were as follows: C from 0.78 to 1.02%, Si from 0.20 to 1.50%, and Cr from 0 to 0.5%. The patenting consisted of austenitizing at 950˚C followed by pearlitic transformation in a lead bath at 580˚C.

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Patented wires were drawn into large-diameter wires by dry-drawing and into small-diameter wires by continuous wet-drawing. The mechanical properties of steel wires were tested using a tensile tester and a twisting tester. The decomposition behavior of θ was investigated by analysis of C in α using an atom probe field ion microscope. The microstructures of the high-carbon steel wires were observed using a field emission type transmission electron microscope (TEM).

3. Results and Discussion

3.1 Strength of patented wires and work hardening properties

Fig. 1[8] shows the influences of C and Cr contents on the strength after a patenting treatment. The strength of a patented wire increases as C content increases. With addition of Cr, strength increases further with the same C content. C and Cr strengthen steel material, respectively, by increasing the volume fraction of high-strength θ and refining lamella spacing. The lamella spacing of a Cr-containing hypereutectoid steel is controlled to 60 nm at an as-patented stage, and as a result, the strength of the steel is as high as approximately 1,500 MPa[9].

Fig. 2[8] shows the influences of C and Cr contents on the rate of work hardening (true strain ε = 3.89). The graph shows that the rate of work hardening increases with C and Cr contents, and the influence especially of Cr, which has an effect of refining lamella spacing, is significant. Among the measures to strengthen a high-carbon steel wire, i.e., increasing the strength of an as-patented wire and the rate of work hardening and wiredrawing strain, the former two are preferred for suppressing the occurrence of delamination, which will be explained later. Refining lamella spacing increases both the strength of an as-patented wire and the rate of work hardening, and for this reason, the development of wire rods used for steel cord is focused mainly on Cr-containing hypereutectoid steels.

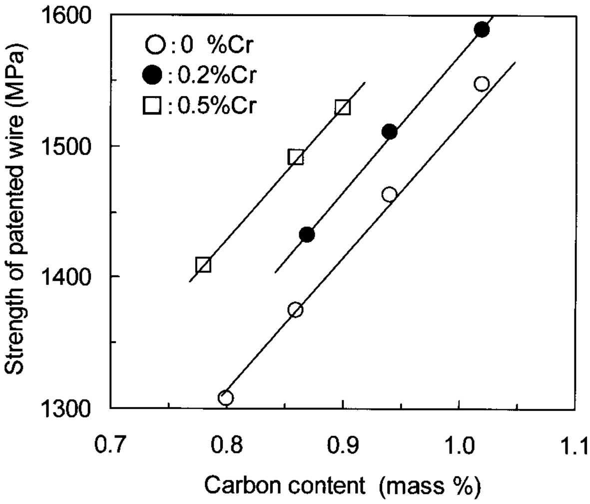

Fig. 1 Effect of C and Cr content on patented wire strength

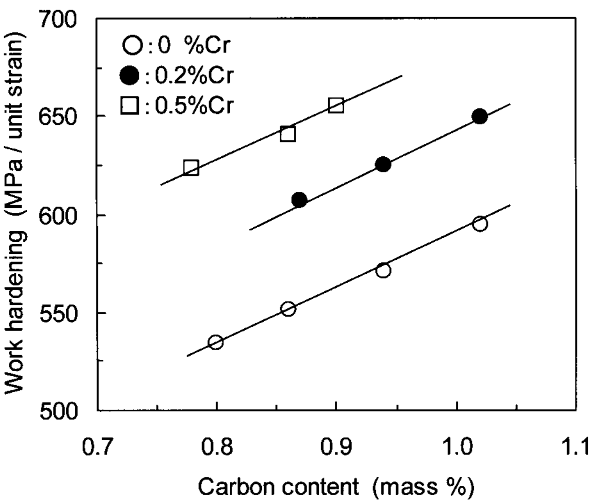

Fig. 2 Effect of C and Cr content on amount of work hardening

3.2 Suppression of softening during hot-dip galvanizing and bluing

The galvanized wires for suspension bridges and PC wires soften during hot-dip galvanizing and bluing processes after drawing, and it is important in the pursuit of further strengthening of the products to inhibit the softening. The softening occurs as a result of the partial division and spheroidizing of heavily deformed θ, since this causes lamella spacing that have been refined to tens of nanometers during the wiredrawing to coarsen[1]. **Fig. 3**[10] shows the influences of the strength of as-drawn wires before galvanizing and the addition of Si and Cr on the loss of strength during hot-dip galvanizing (450˚C). It is clear that the more a specific steel grade is strengthened by applying a higher area reduction ratio at drawing, the greater the loss of strength during galvanizing becomes. It is also clear that the effects of alloying elements are significant: the loss of strength is smaller in high-Si steel wires, and is smaller yet in steels containing Si and Cr.

As seen in **Fig. 4**[11], whereas Si has only a low solubility in θ and segregates at the interfaces between θ and α, Cr enriches in θ. Because the spheroidizing rate of θ in a high-Si steel is determined by the diffusion of Si at the θ-α interfaces, it is considered that, as the content of Si increases, the spheroidizing of θ becomes slower and the loss of strength decreases[1]. On the other hand, since the diffusion rate of Cr is lower than that of Si, the rate of the Ostwald ripening of θ is controlled by the diffusion of Cr, and as a consequence, the spheroidizing rate of θ decreases as the content of Cr increases. This means that the fine lamella structure of a steel wire containing Si or Cr is maintained through hot-dip galvanizing, and the loss of strength is decreased as a result. In addition, since both Si and Cr have the effect of strengthening a patented wire through solid solu-

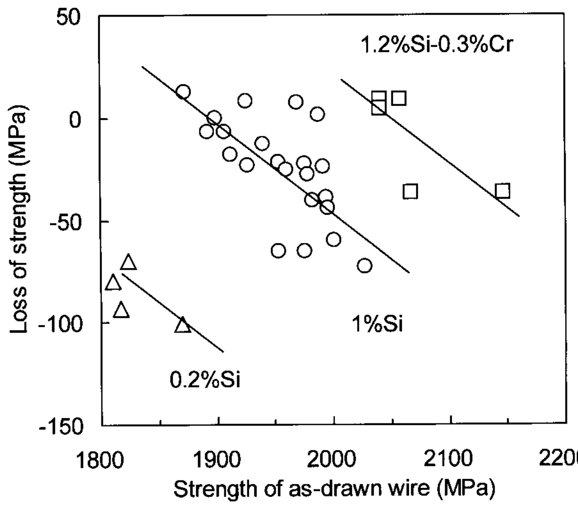

Fig. 3 Effects of as-drawn wire strength and alloying elements on loss of strength during galvanizing

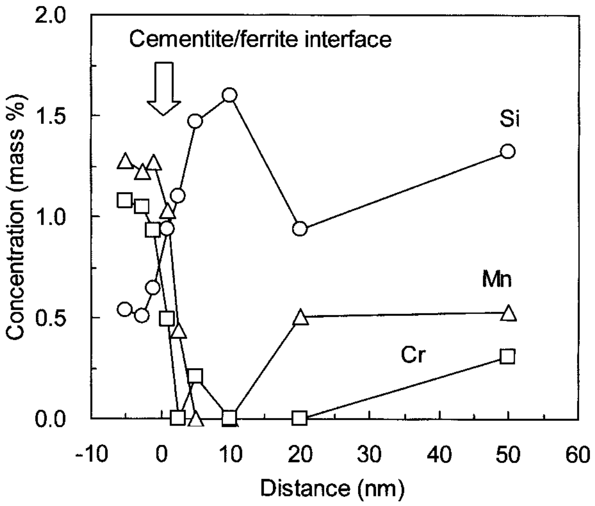

Fig. 4 Distribution of alloying elements across ferrite-cementite interface

tion strengthening and refining of lamella spacing, respectively, they are effective in inhibiting the occurrence of delamination as well.

3.3 Structural change by wiredrawing

The θ and α in the colonies after a patenting treatment are in a single crystal state. However, according to a recent report, the θ breaks into crystals that are nanometers in size during wiredrawing or depending on conditions, turn into an amorphous state[12]. Besides, although it had been generally believed that cell structures formed and dislocation density was high in α of a high-carbon steel wire[13], a paper reported a possibility of decrease in dislocation density, as in steel materials having undergone a mechanical milling treatment, on grounds that the change in contrast in α caused by dislocations was decreased under TEM observation[14].

Fig. 5 shows a lattice image of a heavily drawn Cr-containing hypereutectoid steel wire (ε = 4.2). One can see here that lamella spacing has been refined to somewhere around 10 nm, and the thickness of θ has been reduced to several nanometers through plastic deformation. Macroscopically, θ of the specimen has not been divided, and the α-θ lamella structure is maintained. This is closely related to the fact that heavy drawing is applicable to pearlitic steels and high strength can be realized with steel wires by that process. In addition, as seen from the diffraction pattern, a steel wire having a true strain of 4.2 maintains its crystal structure of θ, although it has been broken into fine grains.

Fig. 6 shows a TEM image of the microstructure of a high-carbon steel wire focusing on the structural change inside α grains. One can see periodical contrast change in the direction of lamella in α grains. The intervals between contrasts were measured to be approximately 10 nm, nearly equal to the lamella spacing. Fig. 6 also shows selected-area electron diffraction patterns of α at areas approximately 2 nm in diameter each. The crystal orientation of the α grain is the same at all the positions, indicating that the α grain did not turn into a multi-crystal state in spite of heavy drawing. The fact that the diffraction spots are slightly different in areas showing different contrasts seems to indicate that the change in contrast results from cell structures that have slightly different crystal orientations. Assuming that the size of a cell is 10 nm, there are 10^{16} cells per m^2, and even if each cell is formed of one dislocation, dislocation density amounts to $10^{16}/m^2$.

Mizoguchi et al.[15] measured the misorientation between adjacent α grains of a pearlitic steel having undergone heavy deformation by rolling and reported that the misorientation was approximately 10° at the largest, and that when the lamella spacing was 20 nm, the density of geometrically necessary dislocations (GN dislocations) required for alleviating the misorientation was on the order of $10^{16}/m^2$. **Fig. 7** shows the relationship between the change of lamella

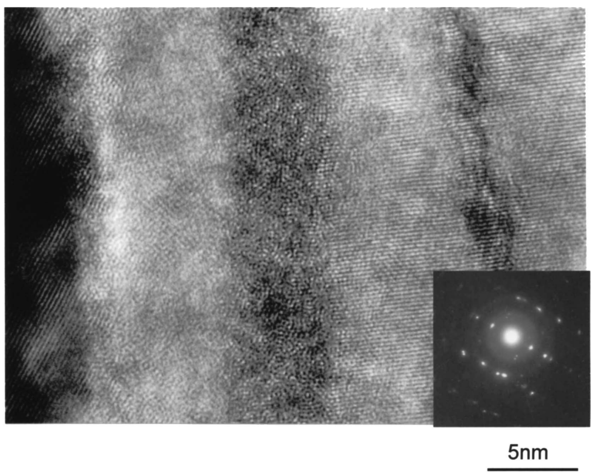

Fig. 5 Lattice image of high carbon steel wire (ε = 4.2)

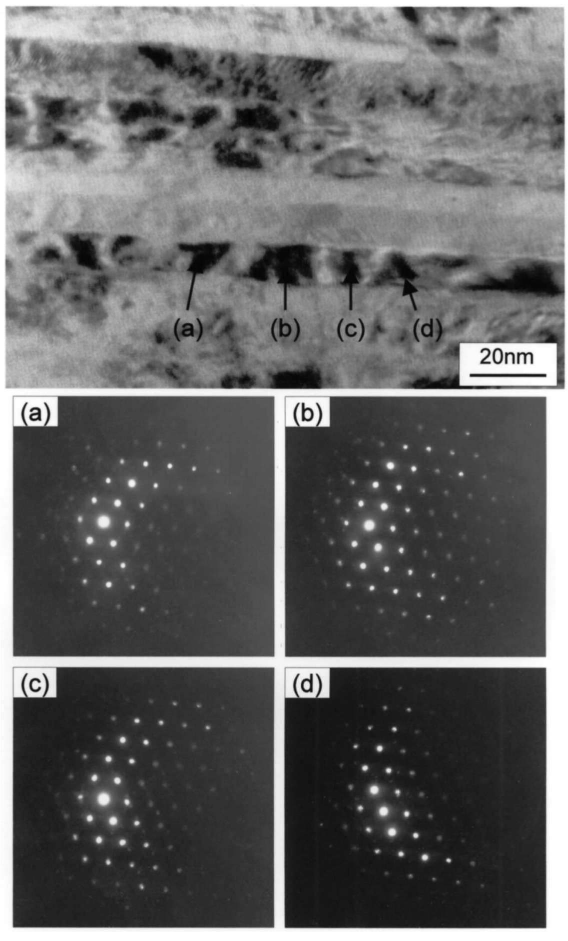

Fig. 6 TEM micrograph and selected area electron diffraction patterns of high carbon steel wire (ε = 4.2)

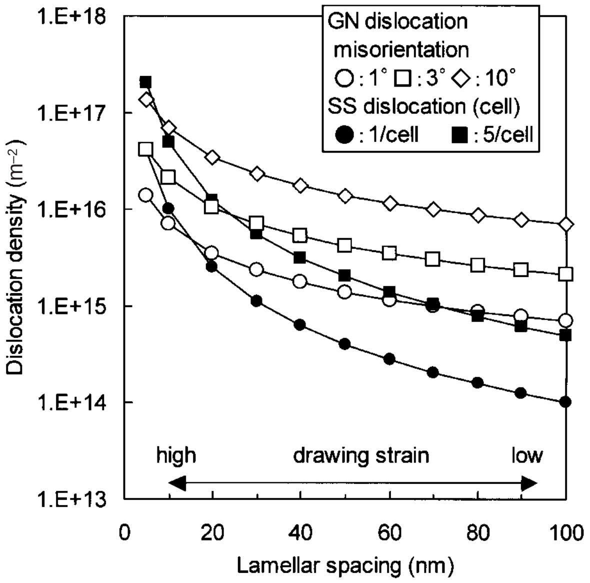

Fig. 7 Relationship between lamellar spacing and dislocation density

spacing resulting from wiredrawing and dislocation density. Here, the cell size was assumed to be equal to the lamella spacing. In a high-carbon steel wire the lamella spacing of which is reduced to 10 nm through heavy drawing, the density of the GN dislocations at α-θ interfaces and cell boundaries in total is on the order of $5 \times 10^{16}/m^2$, even by an underestimation on an assumption that the misorientation between adjacent α grains and between cells is 3˚. Since cells are formed also of statistically stored dislocations (SS dislocations) besides the GN dislocations, the dislocation density of a heavily drawn high-carbon steel wire is presumed to be on the order of as high as $10^{17}/m^2$ in consideration of the SS dislocations.

3.4 Behavior and mechanism of cementite decomposition

Fig. 8[7] shows the change in C concentration in α under different values of drawing strain; the specimens are dry-drawn SWRS82B and wet-drawn SWRS92A. One can see here that the decomposition of θ begins at a comparatively early stage of wiredrawing, and the C concentration in α increases beyond the limit of its solid solution. The C concentration increases as the drawing strain increases, and it exceeds 1 at% in some portions of a steel wire having undergone a high drawing strain. Comparing the specimens under the condition of the same drawing strain, it is clear that the C concentration of dry-drawn SWRS82B was higher than that of wet-drawn SWRS92A, indicating that the decomposition of θ occurred more easily in S82B during wiredrawing. Further, the fact that the C concentration fluctuated significantly at different areas of measurement even under the same drawing strain indicates that the occurrence of the decomposition of θ was not homogeneous. The C concentration tended to be high in regions where lamella spacing was small, as Hong et al. had reported[4].

Fig. 9 shows a three-dimensional mapping of C atoms in a heavily drawn pearlitic structure obtained using a three-dimensional atom probe (3D-AP). C atoms are distributed not only at α-θ interfaces but also inside α grains, and their distribution is inhomogeneous. Since a high-carbon steel wire is presumed to undergo static and dynamic strain aging because of heat generation of wiredrawing[16-18], for clarifying the strengthening mechanism of a high-carbon steel wire it is important to investigate the relationship between the θ decomposition and strain aging.

Fig. 10[19] shows the variations of yield strength and C concentration in α for SWRS82B specimens aged at different temperatures. The age hardening hits a peak at an aging temperature of 250˚C, and the strength falls to below that of an as-drawn wire when the aging temperature exceeds 400˚C. One can see in the graph that the C concentration in α after wiredrawing is already as high as approximately 0.5 at% and through an aging treatment at 250˚C, at which temperature α significant age hardening is achieved, the C concentration increases to above 1 at% as a result of the decomposition of

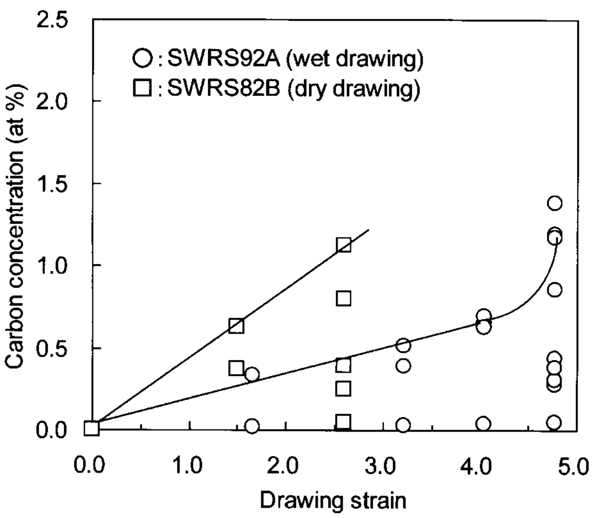

Fig. 8 Variation of carbon concentration in ferrite with drawing strain

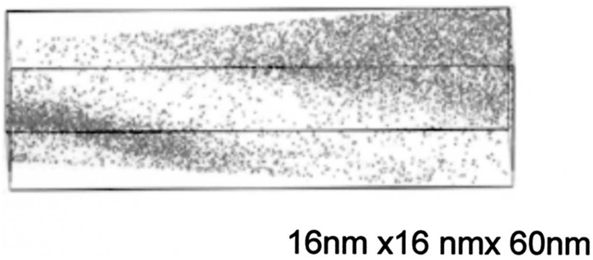

Fig. 9 Three-dimensional mapping of carbon atom

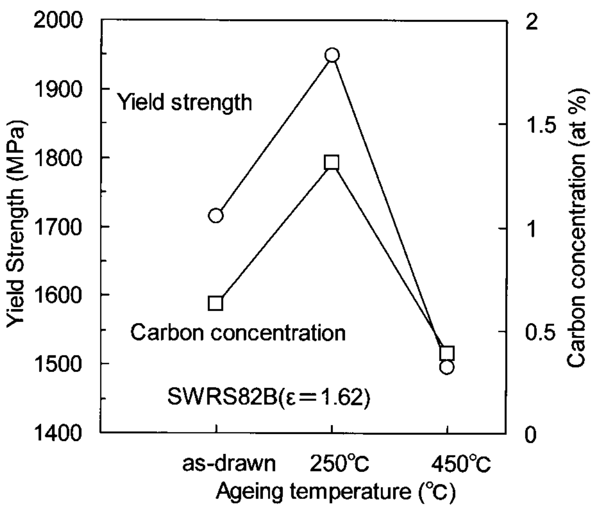

Fig. 10 Variations of yield strength and C concentration in ferrite with aging temperature

θ. This result indicates that the amount of θ decomposition changes depending on the temperature rise of a steel wire owing to the heat generation during wiredrawing. It also indicates that because yield strength increases as the C concentration in α increases, the strain age hardening of a high-carbon steel wire results from locking of dislocation by C that is released as a result of the θ decomposition, as reported by Yamada[16]. On the other hand, the C concentration of a steel wire aged at 450˚C falls below that of an as-drawn wire. In view of the fact that the recovery of dislocations in α, partial recrystallization of α and the spheroidizing of θ take place during aging at this temperature range[1], the above decrease in C concentration suggests that dislocation density also has an influence on the amount of θ decomposition.

While the decomposition of θ was discussed from the viewpoint of elastic interaction energy between dislocations and C atoms[1, 5, 18], a mechanism has been proposed by which wiredrawing causes the refinement of θ and the formation of slip steps in θ, leading to an increase in free surface energy, and as a result, θ decomposes by the Gibbs-Thomson effect[18, 19]. With respect to the state of C in α, which closely relates to the mechanism of θ decomposition, whereas the viewpoint of elastic interaction energy assumes the segregation of C at dislocations, the Gibbs-Thomson effect proposal assumes the supersaturation of solute C, and with respect to the strengthening mechanism by C, the former assumes dislocation locking strengthening, and the latter solid solution strengthening. Besides the above two, other proposals have been made that C segregates at α-θ interfaces[20], and C precipitates in α in the form of fine carbides[21]. For clarifying the θ decomposition mechanism in a high-carbon steel wire, it is necessary to discuss the relationship between the sate of C and the mechanical properties of the material at the same time. In consideration of the report of Daitoh et al.[20] to the effect that the lattice constants of α in a steel wire do not change through heavy drawing, the super-saturation of solute C by several at% seems little probable.

In addition, as shown in Fig. 8, it seems difficult to explain the fact that the amount of θ decomposition is different in dry and wet drawing even with the same drawing strain on account only of the Gibbs-Thomson effect. With regard to the proposal of C segregating at α-θ interfaces, while past results of C analysis by 3D-AP have clarified[4], as shown in Fig. 9, that C atoms exist inside α grains, the C segregation only at α-θ interfaces has not been confirmed. On the other hand, the authors could not confirm the precipitation of fine carbides inside α grains by a high-resolution TEM observation of as-drawn and aged wires. Therefore, it seems appropriate to think that, as has been proposed, most of the C atoms released through the θ decomposition segregate to dislocations.

The strength of a high-carbon steel wire is known to change significantly depending on wiredrawing conditions such as temperature, speed and area reduction ratio per pass. The strength change is considered due to the static and dynamic strain aging caused by heat generation during wiredrawing[3]. Since the dislocation locking strengthening by the C from the θ decomposition is a main factor of the strain age hardening, the dislocation locking strengthening by C is thought to play an important role in the remarkable work hardening properties of a high-carbon steel wire.

Assuming that the C from the θ decomposition segregates at dislocations, the mechanism of θ decomposition in a high-carbon steel wire is as follows. According to Kalish et al.[22], C atoms near a dislocation segregate to the dislocation because of the strong elastic interaction energy (0.48 eV) between a dislocation and a C atom, and consequently the concentration of solute C in α near the dislocation decreases. On the other hand, since the local equilibrium of C between α and θ has to be maintained, it is necessary for θ to decompose and supply C atoms to α, in order to maintain the local equilibrium. In order for this model to be valid, it is necessary for C atoms to diffuse to dislocations within the very short time of wiredrawing and for the C-trapping capacity of dislocations to be sufficiently large. Die temperature measurements[18] and FEM analyses[18, 23] have made it clear that the heat generation in common wiredrawing is large: the surface temperature of a steel wire rises to 300 to 500˚C or so. C atoms can easily diffuse over a distance of tens of nanometers within a very short time of approximately 0.1 s in the above temperature range[19].

In addition, because the dislocation density of a heavily drawn high-carbon steel wire is estimated to be on the order of $10^{17}/m^2$ as shown in Fig. 7, the C-trapping capacity of dislocations is presumed to be sufficiently large. From the above, the θ decomposition in a high-carbon steel wire is considered to result from the strong elastic interaction energy between dislocations and C atoms. The conditions for θ decomposition to occur are a high density of dislocations serving as C-trapping sites and the wiredrawing-induced heating that accelerates the diffusion rate of C atoms.

3.5 Influence of cementite decomposition on ductility of high-carbon steel wire

The ductility of a high-carbon steel wire is evaluated usually in terms of the number of twists and fracture shape at a twist testing. A steel wire having a high ductility is twisted homogeneously over its whole length, exhibits a high number of twists, and fails finally in the direction in right angles to the length. With a steel wire having a low ductility as a result of strengthening, longitudinal cracking called delamination occurs in the direction of drawing at a very early stage of twisting deformation. The delamination is one of the most significant factors that hinder the strengthening of high-carbon steel wires. Although various models were proposed regarding the mechanism of delamination[24-28], none of them succeeded in explaining the effect of wire diameter on the occurrence of delamination. In the meantime, θ decomposes during wiredrawing and C atoms lock dislocations, as explained earlier. The θ decomposition is presumed to have influence not only on the strength but also on the twisting properties of a steel wire. In view of this, the authors examined the relationship between θ decomposition and delamination.

The authors made it clear in a separate paper that, when the maximum C concentration in α exceeded 1 at%, delamination occurred with any of the dry-drawn large-diameter and wet-drawn small-diameter wires shown in Fig. 8 and the steel wire having undergone an aging treatment shown in Fig. 10[29]. Delamination occurs to a high-carbon steel wire more easily when θ decomposition advances. Therefore, the behavior of θ decomposition is suspected to play an important role in the occurrence of delamination. In addition, the effect of wire diameter on the occurrence of delamination and the fact that delamination occurs with low-temperature aging but not with high-temperature aging can be explained in a consistent manner using the increase of C concentration in α resulting from θ decomposition.

Yamada, Fujita et al.[16-18] studied the influence of the strain aging of a high-carbon steel wire resulting from wiredrawing on its mechanical properties in detail, and reported that the strain aging caused by the C resulting from θ decomposition during wiredrawing was the cause of loss of ductility of a high-carbon steel wire, which is the same as the result of the present study. However, they did not clarify the mechanisms behind the reported results. Since the θ decomposition in a high-carbon steel wire is presumed to occur inhomogeneously in microscopic and macroscopic scale, as described earlier, the authors focused attention on the inhomogeneity of θ decomposition, and studied its relationship with the occurrence of delamination. The C atoms released as a result of θ decomposition segregate to dislocations and lock them. The tensile or twisting strength of a high-carbon steel wire is considered to be affected not only by lamella spacing and dislocation density but also by the dislocation locking strengthening by C, and it follows that inhomogeneous θ decomposition means an increase in the microscopic non-uniformity of strength.

What is more, the increase in the fluctuation range of lamella spacing resulting from an increase in drawing strain[30] also increases the microscopic non-uniformity of strength. The strength of a high-carbon steel wire is not homogeneous: an increase in the drawing strain and the aging during wiredrawing or thereafter are considered to increase the non-uniformity of strength. The authors presume that, at a twisting testing, twisting deformation does not propagate easily in a steel wire having a large strength non-uniformity and tends to accumulate locally, and as a consequence, the deformation concentrates at portions where strength is lower, namely where θ decomposed less, leading to cracking. The fact that delamination occurs when the maximum C concentration in α exceeded 1 at% with any of the large- and small diameter wires and one having undergone an aging treatment indicates that delamination occurs when the non-uniformity of strength exceeds a certain limit. However, the quantitative study of this mechanism is a future subject.

3.6 Strengthening of high-carbon steel wire

The strength of steel cords for reinforcing automobile tires was initially 2,800 MPa, but steel cords having strength of a 4,000 MPa class have come to be used[31], contributing to the weight reduction of tires. Cr-containing hypereutectoid steels have been developed for the application on account of the ease of refining lamella spacing, which is effective both in strengthening patented wires and increasing the rate of work hardening[9]. On the other hand, although the strength of galvanized wires for suspension bridges was 1,500 to 1,600 MPa for more than 50 years, steel wires of 1,800-MPa class strength were developed[10] for application to the world longest Akashi Strait Bridge, contributing to reducing the costs and period of the construction. Steel wires having strength of a 2,000 MPa class for suspension bridges have been brought close to actual application[32, 33]. High-Si and high-Si-Cr steels effective in inhibiting the softening during hot-dip galvanizing and strengthening patented wires have been developed for high-strength galvanized suspension-bridges wires[32]. In addition, in the field of PC wires, hypereutectoid steels containing Si and Cr have been developed for 2,300 MPa class PC strands, aiming at realizing a strength one step above the conventional 1,860 MPa class[32].

The requirements for higher strength of high-carbon steel wires typically such as steel tire cords are increasing. It is indispensable for properly responding to the requirements to establish countermeasures against delamination, which is one of the toughest obstacles to strengthening of steel wires. As was stated earlier, delamination occurs principally because of the decomposition of θ during wiredrawing; heat generation of wiredrawing is considered to accelerate the θ decomposition. Since heat generation becomes more conspicuous as steel wire strength increases, the technology to control the decomposition of θ is expected to become the key to further strengthening of high-carbon steel wires. In this respect, close linkage between the development of steels and wire drawing technology will become increasingly important.

4. Conclusions

The strengthening of tire steel cords has advanced through the development of technologies to realize ultra-fine lamella spacing. In the strengthening of galvanized wires for suspension bridges and PC wires, on the other hand, microstructural control to prevent the destruction of fine lamella structure during hot-dip galvanizing and bluing treatment have played an important role in strengthening the product, besides the technologies to refine lamella spacing. It is important for further strengthening high-carbon steel wires to establish technologies to control the decomposition of cementite, which has come to be refined as small as nanometers in size.

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