Size Dependence of Delamination of High-carbon Steel Wire

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(Received on August 2, 2000; accepted in final form on October 30, 2000)

In order to find out major causes to produce the wire-size dependence of delamination, comparative experiments were performed with high-carbon steel wires. A large-size wire and a small-size wire were patented, drawn, and blued under almost the same conditions and the resultant wires were compared quantitatively. Consequently, the experiments showed that even when the wire-making processes were controlled equivalently between the two different sizes, the delamination of the large-size wire tended to be less reduced than the small-size wire. An analysis with torsion tests revealed that the observed size dependence was not substantially associated with applied shear stress in torsion, while the yield shear stress had a significant effect on delamination occurrence. Close observation with SEM showed that significantly large microvoids form in the large-size delaminated wires, but not in the small-size non-delaminated wires. Microvoids were found to be nucleated preferentially at the interface between a fragmented cementite particle and relatively thick ferrite. The difference in the stress intensity factor between the different size wires seems to be one of the main causes bringing about the size dependence. Another finding that the as-patented large-size wire had a larger volume of proeutectoid ferrite than the as-patented small-size wire suggests the strong likelihood of proeutectoid ferrite being associated with void formation.

KEY WORDS: high-carbon steel wire; delamination; size dependence; fracture mechanics; microvoid; ferrite.

1. Introduction
A high-carbon steel wire, strengthened by high-strain cold working, is one of the strongest materials among the commercially available steels. In recent years, a demand for the ultra-high strength wire has intensified, because an increase in strength is expected to contribute to energy-saving in the future. In order to upscale the strength, one of the main obstacles that must be solved is the occurrence of delamination, which is often seen when the tensile strength becomes significantly large with a high degree of deformation. Delamination is a type of fracture, characterized by a longitudinal split and a serrate drop in a torque–strain curve in torsion.

To reveal factors influencing the delamination, studies have been conducted thus far by many researchers enabling the phenomenological aspects of delamination to be revealed to some extent.\cite{1-7} For example, the delamination is known to be associated with as-patented microstructure,\cite{3,5} texture,\cite{2,3} residual stress,\cite{9} and drawing conditions.\cite{3-7} While the previous studies have undoubtedly heightened our understanding of the delamination, we have not yet reached a full understanding of the whole mechanism of delamination. In particular, our understanding of the fracture aspects of delamination occurrence (e.g., crack formation and stress intensity factor) is limited.\cite{8,9}

The unsolved mechanism of delamination occurrence is also closely related to another controversial problem; that is, the dependence of delamination occurrence on the initial wire size prior to drawing. The problem of the wire size dependence is that a large-size wire is more prone to be delaminated than a small-size wire even at the same reduction of drawing. Several studies have thus far reported many possibilities as origins of the size dependence. They include, for example, the effects of residual stress,\cite{10,11} strain aging, and as-patented microstructure.\cite{1,12} Qualitatively they may be more or less associated with the size dependence; however, little quantitative research has been done to prioritize those influential factors and consequently provide a quantitative explanation of the causes of the size dependence.

Thus, the main objective of this study is to carry out a quantitative experiment to identify the most pronouncing effect that governs the size dependence of delamination. The ultimate goal of this study is also to clarify the delamination mechanism from the fracture mechanics standpoint. To achieve that goal, experiments were performed where a large-size wire and a small-size wire were compared as quantitatively as possible under nearly the same conditions. Analysis with torsion tests and SEM observation revealed that the size dependence is closely correlated with microvoid formation during drawing and torsion.

2. Fracture Mechanics of Delamination
Delamination occurs in a brittle manner during torsion tests where intense torsional stresses are applied to the wire. Since the delamination can be recognized as a frac-
tured stress (principle tensile stress), the other perpendicular to the axis. There are a resultant force for crack propagation to lead to delamination should be the shear stress parallel to the elongated fiber axis. Typically, delamination occurs subsequently after yielding, at which the fiber axis is not substantially tilted or rotated due to the limited amount of torsional deformation. Thus, it is reasonable to assume that the crack propagation leading to delamination is primarily affected by the maximum yield shear stress. This study therefore aims at investigating how stress concentration can be represented by the stress intensity factor, $K_a$, which is a function of latent crack length ($a$) and applied stress ($\sigma$) in such a way as

$$K = \sigma a^{\frac{1}{2}}$$

where $\alpha$ represents a coefficient. Fracture mechanics tells us that when the stress intensity factor $K$ exceeds the so-called fracture toughness $K_c$—a critical material-dependent parameter—the crack propagates spontaneously toward a fracture. This is represented as

$$K_c < K$$

As Lefever et al. suggested, the most influential driving force for crack propagation to lead to delamination should be the shear stress parallel to the elongated fiber axis. Typical delamination occurs subsequently after yielding, at which the fiber axis is not substantially tilted or rotated due to the limited amount of torsional deformation. Thus, it is reasonable to assume that the crack propagation leading to delamination is primarily affected by the maximum yield shear stress. This study therefore aims at investigating how stress concentration can be represented by the stress intensity factor $K$ expressed as Eq. (1) varies with the initial wire diameter.

3. Experimental

In order to determine the major causes of the size dependence of delamination, a large-size wire and a small-size wire were processed and analyzed under equivalent conditions. The compositions of both wires were the same: 0.82% C, 0.23% Si, 0.71% Mn, 0.02% Cr, 0.01% P, 0.005% S (mass%). The initial diameters of the steel rods prior to drawing were 11.5 mm and 4.22 mm for the large-size wire and for the small-size wire, respectively. Both were initially patented with a lead bath furnace. To see the effect of patented microstructure and mechanical properties on delamination occurrence, the lead bath temperature was altered between 520 and 650°C, resulting in significant variations in the tensile strength of the as-patented wires. The austenitizing temperature was kept at 900°C for most of the experiments except for some of the patentings with the large-size rods: 800, 1000, and 1150°C were additionally employed for the large-size wires.

The patented wires were then drawn through dry drawing machines. A draw bench and a dry single block machine were used for the large-size wire and the small-size wire, respectively. To keep the drawing conditions as equivalent as possible, pass schedule and die angle were set to be identical between the two drawing processes. The same lubricating material was also employed for both processes. To prevent an excess amount of strain aging in the course of drawing, the temperature of the wire during and after each path was consistently maintained under 120°C in both cases. Part of the as-drawn wires were blued at 380°C for maximum 5 min for the following two reasons: 1) to induce delamination intentionally by introducing strain aging, and 2) to lessen the effect of the residual stresses in the as-drawn wires on the delamination occurrence.

Finally, the processed wires were analyzed in terms of their mechanical properties with tension tests and torsion tests. Microstructural natures of the wires were investigated by means of SEM/FE-SEM and X-ray measurements.

4. Experimental Results

4.1. Tensile Properties

The tensile properties of both as-drawn wires were compared at the same reduction of 86.5% ($e=2.0$) as shown in Fig. 3. The as-patented strengths were confirmed to increase with the decrease in the lead bath temperature employed for the patenting. There are almost linear relationships between the tensile strengths of the as-patented wires and those of the as-drawn wires for both sizes. Those two lines are somewhat distant from each other, but the difference is not significant when compared to the torsional properties that are discussed below at Sec. 4.2. The reduction of area and elongation of the as-drawn wires were also plotted against the tensile strength in Fig. 3(b). It shows that the reduction of area is of the same level between the two different sizes. As for the elongation, the large-size wire had a more pronounced elongation than the small-size one.

These above results indicate that macroscopically the tensile properties depend on the wire size to a less degree than the torsional properties described below. This consequently suggests that, in uni-axial tension, deformation behaviors (e.g., uniform elongation and necking) and the fracture mechanism (i.e., void formation, void coalescence, and cup-and-corn fracture) are less size-dependent than in torsion.
4.2. Torsional Properties

The torsional properties of both drawn wires of a 86.5% reduction were then evaluated before and after bluing under the same conditions. The numbers of torsions are shown in Fig. 4 for both large and small size wires. Apparently, there was no occurrence of delamination with the small-size wires under any conditions, but some of the large-size wires patented at relatively high temperatures exhibited delaminations when bluing treatment of 5 min was applied. It is of interest to point out that even in this study the size dependence of delamination was present at a relatively low reduction of 86.5%. Even with a further increase in reduction, the small-size wire did not exhibit delamination until the reduction reached 94.4% ($\varepsilon=2.9$) as shown in Fig. 5. Another interesting finding was that the as-blued large-size wires, austenitized at 800 and 1,150°C in patenting, exhibited delaminations, as well.

4.3. Shear Yield Stress

In this study, the torsional shear yield stress, closely associated with the stress intensity factor for delamination, was evaluated for each of the as-drawn wires and as-blued wires. The maximum shear stress at the surface around yielding ($\tau_{0.2}$) can be derived from a torque–angle curve, schematically drawn in Fig. 6, through the following equation\(^1\)

![Fig. 3. Tensile properties of as-drawn wires of 86.5% reduction plotted against those of as-patented wires with varied wire size and patenting conditions: (a) tensile strength, and (b) reduction of area and elongation.](image1)

![Fig. 4. Torsional numbers of drawn wires of 86.5% reduction: (a) for large-size wire with a variation in lead bath temperature, (b) for large-size wire with a variation in austenitizing temperature, and (c) for small-size wire with a variation in lead bath temperature.](image2)

![Fig. 5. Torsional numbers and $\tau_{0.2}$ for small-size wires with patenting temperature varied between 550°C and 650°C; as a function of total reduction in drawing.](image3)
where \( a \) denotes the wire radius and the points, \( B, C, \) and \( D, \) are given in the figure.

**Figure 7** illustrates the empirically obtained \( \tau_{0.2} \) with a variation in the corresponding tensile strength. Characteristic features of the graph are that \( \tau_{0.2} \) is almost proportional to the tensile strength in either case and that a bluing treatment of 5 min significantly enhances the shear stress in every case. Our TEM observation revealed that the bluing treatment \((380 \degree C/H11003 \times 5 \text{ min})\) did not change the macroscopic morphology of the as-drawn pearlite structures \( (e.g., \text{ interfacial morphology of elongated cementite lamellae}) \). Thus, the delamination induced by the bluing was considered to occur due to the increased \( \tau_{0.2} \). Yet, it should be noted that a higher \( \tau_{0.2} \) did not necessarily lead to the delamination occurrence for the large-size wire: \( \tau_{0.2} \) for the delaminated wire \((\text{patented at } 620 \degree C)\) is lower than that of the non-delaminated one \((\text{patented at } 460 \degree C)\). There is a slight difference in \( \tau_{0.2} \) between the two different sized wires, but the difference is not significant.

An increase of \( \tau_{0.2} \) in the small-size wires with increasing drawing reduction can be seen in **Fig. 5**. Obviously, \( \tau_{0.2} \) at a high reduction of 94.4% was larger than any of those seen in **Fig. 7**. As mentioned above, the high value of \( \tau_{0.2} \) did not result in delamination for the small-size wires of a 94.4% reduction. This conclusively indicates that \( \tau_{0.2}\), which has a significant influence on the delamination occurrence, is not necessarily critical to bringing about the size dependence.

In the above evaluation of \( \tau_{0.2} \), the rigidity of the wires was assumed to be homogeneous across the radius. In reality, however, the rigidity may not be uniform across the radius. To assure the applicability of the above analysis, the distributions of Vickers hardness in both wires of a 86.5% reduction were evaluated. Part of the results is shown in **Fig. 8**. It can be seen that the presence of the bluing affected the levels of the hardness; however, the distributions of the hardness did not differ significantly between the large-size wires and the small-size ones, regardless of the presence of the bluing. Assuming that the rigidity is proportional to the hardness, maximum shear yield stresses were calculated with the data in **Fig. 7** and **Fig. 8**. Consequently, the calculated maximum shear yield stresses turned out not to differ over 2% from the obtained values of \( \tau_{0.2} \). This means that the above argument on \( \tau_{0.2} \) is still applicable. Furthermore, the calculation showed that, due to decarburization near the surface, maximum shear yield stresses resulted around 100 \( \mu m \) and 70 \( \mu m \) away from the surface for the large-size wire and for the small-size wire, respectively. The subsequent investigations, therefore, were carried out assuming the initiation of delamination occurs in the region of maximum shear yield stress \( (i.e., \text{ around a depth of } 70 \text{ or } 100 \mu m) \).

4.4. Texture

It has been reported that texture developed through drawing deformation has a strong influence on the delamination behavior.\(^{2,3,8}\) For example, a cyclic texture formed near the surface is more likely to induce delamination than a fiber texture. In this study, the degree of texture development in drawing and its mode were analyzed for the different size wires by means of X-ray diffraction. In the preparation of the specimens, the as-drawn wires of a reduction of 86.5% were cut and arrayed as shown in **Fig. 9**. In the case of the large-size wire, the surface and central regions were separately measured. The obtained (110) pole figures seen in
Fig. 10 clearly show that both wires exhibit similar preferred orientations. It should be noted that the center region of the large-size wire also possessed the same texture as Fig. 10(a). Despite the difference in the means of preparing specimens, the \{112\}\{110\} textures are almost equivalent. The observation that the mode of texture development did not differ significantly between the two wires seems to reasonably exclude the effects of texture from the factors governing the size dependence.

4.5. Residual Stress

Residual stress in a drawn wire is known to have a strong effect on the occurrence of delamination. In this study, the residual stresses involved in both wires were analyzed by X-ray measurements. Special focus was placed on circumferential residual stress in the wire, the importance of which is discussed later. Part of the results is shown in Fig. 11. The bluing treatment was confirmed to lower the tensile residual stress in the as-drawn wires and consequently made the stress levels almost equivalent between both as-blued wires. This result verifies that the residual stress was not a factor affecting the occurrence of the delamination seen with the as-blued wires. It is interesting to note that a large difference was seen in the residual stress between the large-size as-drawn wire and the small-size as-drawn one. This is also discussed later.

4.6. Void Formation in Drawing and Torsion

The other parameter included in the stress intensity factor other than the applied stress is the length of a crack, which originates from microvoids. A search was made for microvoids formed in the as-drawn wires before and after the torsion tests. By using SEM and FE-SEM analyses, it was found that microvoids were formed in the as-drawn wires that exhibited delamination in torsion. Some of the typical voids observed near the surface are presented in Fig. 12. The torsional deformation was also found to increase the number of voids. As opposed to the delaminated wires, other large or small non-delaminated as-drawn wires did not include a noticeable amount of sizable voids (Fig. 13). A prominent feature of the voids found in the delaminated wires is that they are nucleated at a cementite–ferrite interface. It appears that the voids are more likely to be formed on a fragmented cementite particle surrounded by relatively large proeutectoid ferrite than on an elongated lamellar cementite–ferrite interface. The fragmented cementites, on which voids can be preferentially nucleated, seemed to be observed more frequently in the large-size wires of coarse pearlite (patented at high temperatures) than in the large-size wires of fine pearlite (patented at low temperatures).

4.7. Effect of Undissolved Carbides on Void Formation

A marked effect of undissolved carbides on void formation was observed with the as-drawn wire whose austenitizing temperature at patenting was as low as 800°C. Figure 14 clearly exemplifies globular undissolved carbides in the fiber-wise deformed pearlite and microvoids formed around them. It was also found that the number of voids remarkably increased when the wire was distorted (Fig. 14(b)). The size of the voids ranged up to nearly 2 μm at maximum. Since the number of the detected voids in the wire with undissolved carbides was significantly larger than that of any other wires without noticeable undissolved carbides,
it can be said that undissolved carbides are more likely to induce the formation of voids than any other microstructural features of a high-carbon steel wire.

4.8. Microstructural Aspects of Wire Properties

Significantly large microvoids were detected in the delaminated large-size wires, but not in the non-delaminated small-size wires. The drawing procedures were set to be almost equivalent for all the wires in the study; therefore, a possible cause bringing about the difference in the void formation was considered to be a microstructure change of the as-patented wires caused by the varied patenting conditions.

As-patented microstructures were analyzed from various perspectives. A comparison was made between the two wires which are nearly equivalent in terms of tensile strength and as-blued $\tau_{0.2}$: one being the large-size wire patented at 580°C (delaminated in torsion) and the other being the small-size wire patented at 620°C (non-delaminated). The austenitizing conditions were the same for both wires; therefore, the austenite grain sizes were found to be of the same order. The measured block sizes and averaged interlamellar spacings are presented in Fig. 15. This clearly shows that both patented wires have the almost equivalent block sizes and lamellar spacings across the wire radius.

A remarkable difference was found in the quantity and morphology of the proeutectoid ferrite grain. Figure 16 typifies both as-patented microstructures. The large-size wire has thicker proeutectoid ferrite along the austenite grain boundary than the small-size one. An evaluation with a graphic analysis technique also indicated that the volume of proeutectoid ferrite for the large-size wire can be up to 5 times as much as that for the small-size one (i.e., 0.93% vs. 0.18%). Another difference was found in the variability of

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**Fig. 12.** Formation of voids near surface of large-size as-drawn wires (patented at 620°C and 580°C), which were delaminated in torsion; (a) longitudinal section (before torsion), (a) transverse section (before torsion), and (b) longitudinal section (after torsion) (SEM).

**Fig. 13.** Transverse section of as-drawn wires of 86.5% reduction, which were non-delaminated in torsion: (a) large-size wire patented at 540°C and (b) small-size wire patented at 620°C.

**Fig. 14.** Effect of undissolved carbides on void formation in large-size wire, which was austenitized at 800°C, patented at 540°C, and drawn by 86.5% reduction: (a) before torsion and (b) after torsion.
the interlamellar spacings among neighboring colonies. Their variabilities (or standard deviations) differ by a factor of 2 (i.e., \(V: 0.0189 \mu m\) for the large-size as-patented wire and \(0.0095 \mu m\) for the small-size as-patented one.), despite the slight difference in the averaged lamellar spacings shown in Fig. 15.

5. Discussion

5.1. Fracture-mechanics-aspect of Size Dependence of Delamination

Evidence that the observed microvoids near the surface caused the initiation of the delamination could not be obtained in this study. The observed voids, however, did appear similar to those found in Ibaraki et al.'s research; \(i.e.,\) both voids were nucleated on free ferrite. Since they found that their voids were responsible for the initiation of delamination, the agreement between both studies reasonably suggests that our identified voids also initiated the delaminations.

From the fracture mechanics standpoint, the stress intensity factor is the driving force to propagate a crack towards delamination. If the critical length of a crack leading to delamination during torsion is assumed to be proportional to the size of the microvoids observed in the as-drawn wires, the relationship of \(K\) with the wire size can be schematically plotted in Fig. 17. This schematic diagram suggests that \(K\) of the delaminated large-size wires could be significantly larger than that of the other non-delaminated small-size wires. The difference in \(K\) between the large-size wire and the small-size wire was not investigated in the study; however, \(K\) of the small-size wire is expected to be at least no less than that of the large-size wire. This means that the difference in \(K\), essentially the difference in the void formation, was undoubtedly one of the causes bringing about the size dependence of delamination.

In general terms, \(K\) is known to depend on microstructures including grain size, second-phase morphology, preferred orientation, and so on. As mentioned above, the grain sizes and textures were almost the same between the different size wires. Since a crack leading to delamination propagates along the cleavage plane across the grains, it is considered to be unlikely that the difference in the volume of proeutectoid ferrite could cause \(K\) to change more than an order of magnitude. However, an accurate evaluation of
pressure. In torsion, the compressive stress develops nor- 

K<sub>c</sub>, which would require a rigorous test set-up, is needed to report more conclusive findings.

This study successfully revealed that the microvoids are likely to be initiated by both undissolved carbides and coarse pearlite structure which includes a large volume of proeutectoid ferrite. This finding is in good agreement with other studies. It appears that the deformation mode of ferrite and the morphology of carbides play an important role in the void formation. Yet, the exact mechanism by which a void forms preferentially on the fragmented cementite particle in contact with relatively thick ferrite could not be clarified in this study, as well. A further intensive study is expected on this problem.

5.2. Effect of Residual Stress on Delamination Oc-

currence

As mentioned above, the residual stress is not considered to be primarily responsible for the size dependence. In the absence of bluing treatment, however, the significantly large difference in the residual stress observed in Fig. 11 may contribute to the size dependence of delamination which often can be recognized with as-drawn wires. The degree of the contribution was roughly assessed as follows.

The circumferential tensile residual stress is schematically illustrated in Fig. 18(a). As can be easily predicted from this figure, the tensile residual stress near the surface acts to enlarge crack openings leading to delamination. In fact, the crack propagation is known to be affected by hydrostatic pressure. In torsion, the compressive stress develops normal to the fiber axis as shown in Fig. 18(b). When the angle between the fiber axis and the wire axis is represented as θ at yielding and the uni-axial tensile stress described in the figure is given as σ<sub>ut</sub>, the compressive stress developed at the surface (σ<sub>sn</sub>) can be calculated on a two-dimensional basis

\[ \sigma_{sn} = \frac{1}{2} \sigma_{ut} (1 + \cos 2\theta) + \tau_{0,2} \sin 2\theta \] .............(4)

If τ<sub>0,2</sub> and σ<sub>ut</sub> are respectively set to be 45% and 5% of the tensile strength (σ<sub>ut</sub>) for the standard values and θ at yielding is assumed to be around 70°, σ<sub>sn</sub> will become around 28% of σ<sub>ut</sub>. When σ<sub>ut</sub> ranges from 1500 to 3000 MPa, σ<sub>sn</sub> becomes around 420 to 840 MPa in compression. The maximum tensile residual stresses acting normal to the fiber axis (σ<sub>sn</sub>) that were observed in this study were 520 MPa and 245 MPa for large and small size as-drawn wires, respectively, as shown in Fig. 11. This indicates that in some cases of the large-size wire the net circumferential stress in torsion (σ<sub>sn</sub>) can become tensile, resulting in an increased likelihood of crack propagation towards delamination. This suggests that the residual stress can be responsible for the size dependence in the case of as-drawn wires.

5.3. Possible Factors Affecting Proeutectoid Ferrite 

formation

As mentioned above, the difference in the proeutectoid ferrite formation between the two different size wires may somehow be correlated with the difference in microvoid formation observed with the examined wires. The difference in the morphology of ferrite formation can be explained by the following two reasons.

One possible explanation is the difference in the cooling rate in patenting. The cooling rates measured with embedded thermocouples in the range between 800°C and 650°C were 30°C/s and 150°C/s for a 11.5 mm rod and a 4.22 mm rod, respectively. This large difference has potential to vary the volume of proeutectoid ferrite formed along the proeutectoid austenite grain boundary, as suggested by Ibaraki et al. Another possible reason is the effect of the reduction ratio from as-cast bloom to prior-to-patenting rod. It has been revealed that the solidification structure of as-cast steel such as micro-segregation affects the mechanical properties of the subsequent products. Apparently, the larger the reduction ratio, the more improved the mechanical performance of the final products. It is practical to apply this idea to the high-carbon steel wire. This suggests that the homogeneity of solutes may cause the difference in ferrite formation. Yet, verification of this assumption requires thorough research to be conducted in the future.

6. Conclusions

The two different size wires were comparatively examined to clarify the major causes of the wire-size dependence of delamination. As a result, the following results were obtained:

1. Even when equivalent drawing conditions were provided, there was greater tendency for the large-size wire to be delaminated than the small-size wire.

2. Although yield shear stress in torsion undoubtedly affected the likelihood of delamination occurrence, it did not depend greatly on the wire size, not being primarily responsible for the size dependence.

3. Significantly large microvoids were detected in delaminated wires, but not in non-delaminated wires. The difference in the stress intensity factor between the different size wires seems to be one of the major causes bringing about the size dependence.

4. Residual stresses in a drawn wire, along with other drawing conditions, may contribute to the size dependence of delamination to a minor extent, but their contributions are limited.
The as-patented microstructure, which includes lamellar pearlite, proeutectoid ferrite, and undissolved carbides, seems to have a marked effect on the void formation in drawing and torsion. The coarser the pearlite structure and the larger the proeutectoid ferrite grain, the stronger the likelihood of void formation.

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