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Measurement of residual stress by using focused ion beam and digital image correlation method in thin-sized wires used for steel cords

Y S Yang[1,2], J G Bae[3], C G Park[1,2]
[1]Department of Materials Science and Engineering, POSTECH, Pohang, KOREA
[2]National Center for Nanomaterials Technology, POSTECH, Pohang, KOREA
[3]KISWIRE R&D center, Pohang, KOREA

cgpark@postech.ac.kr

Abstract. Residual stress in the axial direction of the steel wires has been measured by using a method based on the combination of the focused ion beam (FIB) milling and digital image correlation software. That is, the residual stress was calculated from the measured displacement field before and after the introduction of a slot along the steel wires. The displacement was obtained by the digital correlation analysis of high-resolution scanning electron micrographs, while the slot was introduced by FIB milling with low energy beam. The fitting of the experimental results to an analytical model with the independent Young's modulus determined allows us to find the residual stress. The complete experimental procedures are described and its feasibilities are also evaluated for the thin-sized steel wires.

1. Introduction

High carbon steel wires are usually twisted together to the steel cords used for pneumatic tires. Since the steel wires are fabricated through repeated drawing and heat treatment, they show various lamellar structures of cementite and ferrite [1]. The important mechanical properties required for steel wires are outstanding fatigue resistance, which depends on lamellar spacing and cementite thickness. Among them, residual stress is one of the major factors to influence the fatigue resistance.

X-ray diffraction (XRD) and stress relaxation method have been tried to measure the residual stress of the steel wires in the past [2]. The measurement of residual stress for these steel wires is, however, not a simple task due to their cylindrical shape and small dimension of 180 μm. It is, therefore, worthy to find the suitable method in evaluating the influence of residual stress on mechanical properties of the steel wires.

In the present study, the focused ion beam (FIB) equipment combined with strain mapping software (DIC) for measurement of the residual stress in thin-sized wires is proposed. The detailed general slot-milling procedure and data analysis to obtain the best results with high accuracy are described.

2. Experimental procedures

The specimens investigated in this study are the hyper-eutectoid steels with the composition of Fe-1.02C-0.2Cr-0.25Si-0.24Mn-0.005S-0.009S (wt.%). The steel wires were fabricated by following procedures: 1st drawing (5.5 to 3.05 mmϕ), 2nd drawing (3.05 to 1.41 mmϕ), lead patenting and final drawing (1.41 to 0.18 mmϕ). After final drawing, the thin-sized steel wires were stress-relived by normal practice involving exposure at temperatures range from 100 to 500 °C for 60 s and then cooled down to room temperature in air to change the value of the residual stress.

To measure the residual stress perpendicular to wire axis, we used the dual beam FIB (SMI 3050) machine to introduce the slot into the wires. The displacement field generated by slot milling into steel wire was measured by using VIC-2D software commercially available from Correlated Solutions Inc, which had a resolution with 1/100th in a pixel.

3. Results & discussion

3.1. Slot-milling procedures to form a displacement

The first step is to introduce a slot on the plane of interest perpendicular to wire axis, as shown in figure 1. When a narrow slot having precise width, depth and length is introduced into the specimen, the stress can be formed due to the creation of two traction-free surfaces. However, if it is not satisfied with this condition, any change of displacement (u_x) generated is not formed. At first, we have exposed the steel wires to the low energy ion beam (I = 40 pA@5 kV), which can make the contour in clear manner. After making the reference line to observe the area of interest, the first SEM image is captured at the magnification of 5.3 K, corresponding to image dimensions of 50 x 50 μm (figure 2 (a)). This image capturing is performed by using SEM rather than FIB to decrease any possible ion beam damage at the slot. Then, a slot is introduced into the region of interest under high energy ion beam (I = 100 pA@30kV). A second SEM image is taken from the same area (figure 2 (b)). In order to obtain a precise measurement of the depth, a small FIB cut is made normal to the slot and SEM image (figure 2 (c)). To reduce FIB ion beam damage during cutting, a compound C gas is used to protect materials.

Figure 3 describes the contour maps showing the displacement field perpendicular to the slot, which was obtained after correlation of the two images. Since the perpendicular displacement shows an oval shape at the middle of the slot, we know that the tensile residual stress exists in the steel wires.

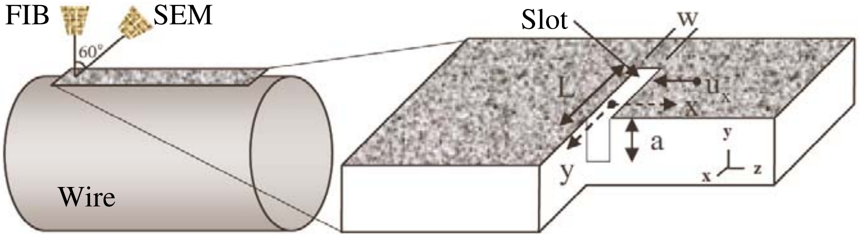

Figure 1. Schematic diagram of the slot introduction by focused ion beam milling into the steel wire

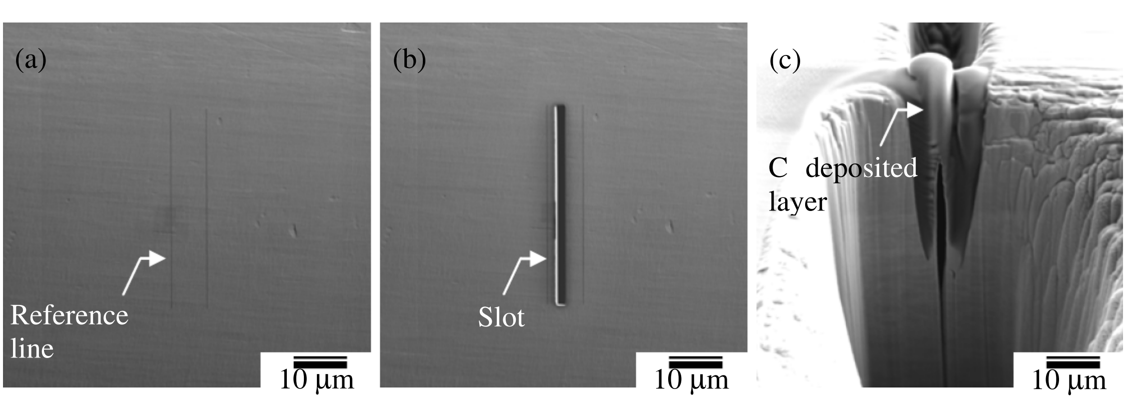

Figure 2. SEM images of the surface plane of the steel wire; (a) before milling, (b) after milling and (c) after milling for measuring depth

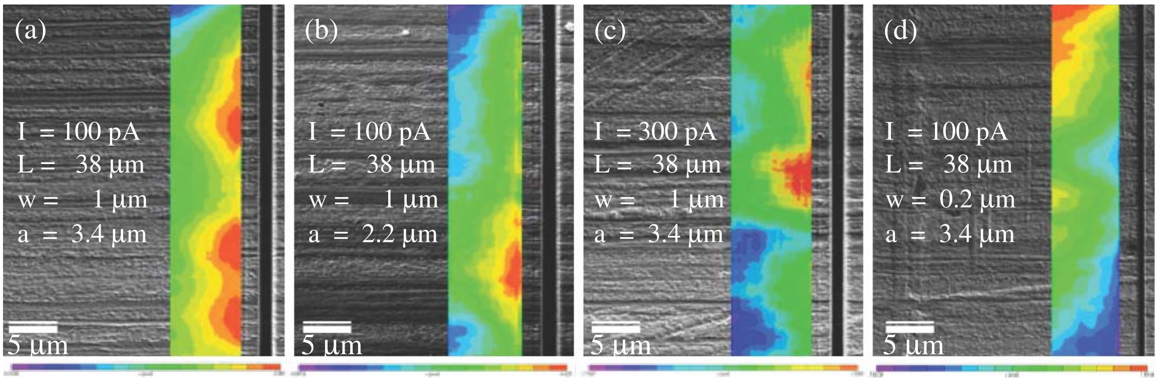

Figure 3. Contour maps showing the displacement fields along the slot depending on the slot dimensions and FIB beam conditions.

The shape of displacement contour, shown in figure 3, is strongly influenced by the FIB beam condition and slot dimension. That is, as the FIB beam current and slot dimension increased, the data points forming the contour are scattered, and the displacement contour is shifted to the upper side. In this study, we have determined that the optimum beam condition for the good contour is about 100 pA. The optimum slot dimensions were also determined to 3.4 µm in depth (a) and 1.0 µm in width (w), which we can provide perfect rectangular-type slot (aspect ratio (w/a) = 0.29). In addition, several authors have proposed the minimum slot length forming the displacement is about ten times lager than the depth of the slot [3,4]. Thus, we have set the slot length (L) to the 38.0 µm (L/a = 11.17) in the present case.

3.2. Data analysis of displacementt

Based on two SEM images, the displacement fields can be obtained by using 'VIC-2D' software. To evaluate the displacement (u_x), the maximum realistic number of image points was used. In this study, subset size and step was maintained in the conditions of 61 pixels and 1, respectively. To determine the precise value of the displacement, we have selected the real displacement field occurred at the centre region of steel wires. The data closest to the slot have been excluded because of ion beam discoloration and the limited availability of feature. When the modulus of the steel wires and depth of the slot are known, the numerical results can be used to determine the virtual displacement (U_x) at each measurement locations (x/a) under virtual stress (σ_{VR}). To calculate the virtual displacement (U_x), we referred to the equation proposed by Kang et al [4] and simplified this formula to suit the steel wire, as follows

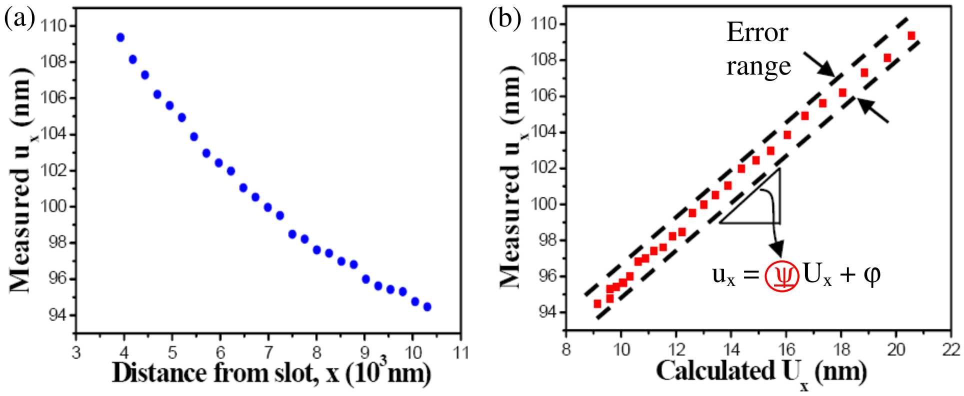

Figure 4. (a) Displacement along the x direction (wire axis) and (b) the residual stress obtained from the steel wires in the figure 3 (a)

Table 1. Summary of residual stress and deviation value obtained from the steel wires

Steel wire	Equation ($u_x = \psi\ U_x + \varphi$)	Deviation in slope (%)	Residual stress (MPa)
As-drawn	$u_x = 1.284\ U_x + 82.99$	1.9	1284
Blued at 100 °C	$u_x = 0.839\ U_x + 4.833$	1.4	839
200 °C	$u_x = 0.673\ U_x + 13.25$	1.2	673
300 °C	$u_x = 0.613\ U_x + 14.17$	3.1	613
400 °C	$u_x = 0.425\ U_x + 53.79$	1.5	425
500 °C	$u_x = 0.257\ U_x + 24.62$	2.1	257

$$U_x = 2 \times 1.1215 E^{\circ^{-1}} \sigma_{VR} \int_0^h f(\theta) dh \tag{1}$$

$$f(\theta) = \cos\theta[1 + 2(1-\nu)^{-1}\sin^2\theta][1.12 + 0.18\sec h(\tan\theta)]$$

where θ = arctan (x/a), ν for Poisson ratio (0.3 for carbon steel), $E^\circ = E/(1-\nu^2)$ for Young's modulus in plain strain (210 GPa for carbon steel) and σ_{VR} for its virtual residual stress (-1 GPa). After completing measurement of virtual displacement, this value is, thus, plotted against the distance from the slot edge and measured displacement. Figure 4 (a) shows the displacement profile along the x direction (wire axis) derived from analytical expression (1). The value of higher displacement takes place at the edge of the slot and the displacement decreases at the long distance from slot, since stress relaxation formed due to creation of traction-free surface occurred actively near slot. In this graph, we identified the displacements were well formed by the slot milling without no scattering points. Figure 4 (b) exhibits the residual stress from comparison between measured u_x and calculated U_x. That is, the slope and the deviation of the data points in graph means the residual stress in the axial direction of steel wire, respectively. In this experiment, we have obtained the exact value with high accuracy.

The results of residual stress and reliable level in the measurement of the steel wires depending on annealing temperature are summarized in table 1. In all steel wires tested in the present study, the value of deviation in the slope was less than 3.1 %. In case of steel wire without annealing, the residual stress is about 1284 MPa. As annealing temperature increased from 100 to 500 °C, the residual stress is rapidly decreased. That is, the residual stress decreased rapidly down to 257 MPa due to local stress relief in steel wires.

4. Conclusions

A novel technique for stress measurement on thin-sized steel wires based on the combined focused ion beam (FIB) and high resolution strain mapping program (DIC) has been presented. It allows local stress measurement by means of digital image correlation of nano-scale deformation fields. The present method has been successfully applied to high carbon steel wires. From the experimental results, we found that FIB-DIC method has a high accuracy level of below 3.1% in all of the steel wires. Because the FIB imaging system has been well proved to work regardless of materials, especially, this technique is expected to be extended to measure the residual stress of the nano-structured materials.

Acknowledgments

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