Experimental Techniques (2019) 43:1–6
https://doi.org/10.1007/s40799-018-0259-9

Effect of Deformation Induced Microstructure Faults on the Elastic Mechanical Parameters of Micro-Scale Copper

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Received: 11 January 2018 / Accepted: 17 May 2018 / Published online: 24 May 2018

Abstract

The elastic mechanical parameters of initial and pre-twisted copper wires were investigated with the aid of a self-developed micro-torsion tester. It was found that the apparent elastic shear modulus and the apparent Young tensile modulus of the copper wires decrease by 13.3 and 8.4% after pre-twist deformation, respectively. In addition, the indentation modulus also decreases as the deformation is increased. When the deformation is more than 30%, the indentation modulus of pre-twisted copper wires is smaller than that of initial copper wires. Microstructural analyses show that the elastic modulus decreases with the proportion of faults.

Keywords Micro-scale · Apparent elastic shear modulus · Apparent Young tensile modulus · Nanoindentation

Introduction

Micro-scale metallic materials have been widely used for fabricating sensors or energy converters in microelectronic devices [1, 2]. The elastic mechanical parameters of the material are crucial factors determining the performance of the sensors or energy converters made thereof. As is known to all, these microelectronic components are subjected to different deformations during the machining process. However, studies related to the effect of deformation with respect to the elastic parameter have, to date, primarily been focused at the bulk materials level [3–7]. For example, in 1993, Akhmadeev et al. found that the Young's modulus of bulk copper with severe shear deformation was lower than that without deformation. The results showed that the elastic property of crystals was related to structure defects [3]. In 2001, Valiev stated that the Young's moduli of deformed bulk copper, as compared to undeformed, were lower by 10–15%. He believed these changes are associated not only with the significant change of the grain size but also with the specific defect structure of non-equilibrium grain boundaries [4]. In 2017, Chen et al. found that apparent Young's modulus of pure titanium rods decreased after suffering either or both of torsion deformation and tension deformation [5]. Chen et al. argued that the decrement of the apparent Young's modulus depended on the increment of equivalent plastic strain [5]. In contradiction to the above results, in 2013, Wang et al. found that the Young's modulus of copper bars gently increased after the severe plastic deformation by combined tension–torsion, resulting from the saturation of mechanical damage and crack introduced by severe deformation [6]. In addition, research related to the elastic mechanical parameters of micro-scale materials follow deformation are still insufficient.

In this paper, the apparent elastic shear modulus and the apparent Young tensile modulus of the copper wires are investigated using torsion and tension experiments respectively. In addition, the indentation modulus of the copper wires at different positions is measured using nanoindentation tests. Then, in order to determine the main factor causing the change of the elastic mechanical parameters, the microstructure of copper wires is investigated by electron backscattered diffraction (EBSD).

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Experimental Procedure

Specimen Preparation

All tests were performed on commercially cold drawn polycrystalline copper wire (99.99% purity) with a nominal diameter of

200 μm. In this study, there were two types of copper wire: initial copper wires (namely copper wires without pre-twist deformation) and pre-twisted copper wires (namely copper wires with pre-twist deformation). Pre-twist deformation was introduced into some of copper wires for investigating the effect of varying degrees of deformation on modulus. The top end of copper wires for pre-twist deformation was fixed, and the bottom end was attached to a suitable metal bar giving about 8 MPa tensile stress which was far below the yield strength (150 MPa). Then, the copper wires were twisted by a rotating stage with a rotational speed of 4.5 rpm. The Pre-twist test was stopped when the 100% shear strain was produced at the outside surface of the copper wires. Consequently, the pre-twisted copper wire has a deformation increasing linearly along its radial direction and the 100% shear deformation is produced on the outside surface. All tests were carried out at room temperature in this study.

Torsion Tests

Torsion tests were conducted on copper wires using a self-developed micro-torsion tester based on electromagnetism, in which a coil-magnet element is used for actuating and torque measuring [8, 9]. The reliability of this technique has been carefully verified [10]. The torque capacity of this tester is 1.3×10^{-3} N•m with resolution of 4×10^{-8} N•m, and the angle capacity is 115° with resolution of 0.016°. Specimens of the copper wires for the torsion tests each had a nominal gauge length of 5 mm. The exact diameter of each specimen was measured using an optical microscope. To avoid accidental damage, a hexagon paper-window was used to fix the specimen. After two ends of the specimen had been fixed on the tester's grip, the paper-window was cut off and the test started with a strain rate of 10^{-4} s^{-1} at the peripheral surface of the specimen. Generally, at least three individual tests were repeated for each type specimen.

Tensile Tests

Tensile tests were performed on the copper wires using INSTRON ElectroPuls E1000 test instrument to obtain the apparent Young tensile modulus. Similarly, the diameter of each specimen was measured using the optical microscope. To eliminate the effect of frame deformation on the tested value, initial copper wires with two nominal gauge lengths (55 mm and 100 mm) were tested respectively. Then, the apparent Young tensile modulus of initial copper wires was calculated on the basis of differential stiffness method [11]. Then, the true tensile Young's modulus E_t of specimen can be easily calculated by equation (1):

$$E_t = \left(\frac{l_1}{d_1^2} - \frac{l_2}{d_2^2}\right) / \left(\frac{l_1}{d_1^2 E_{e1}} - \frac{l_2}{d_2^2 E_{e2}}\right) \quad (1)$$

Where l_1 and d_1 are length and diameter for specimen with nominal gauge length of 100 mm, l_2 and d_2 are the same parameters for specimen with nominal gauge length of 55 mm. E_{e1} and E_{e2} are the measured tensile Young's modulus of the two different length (l_1 and l_2) specimens respectively. Following the same procedure, the apparent Young tensile modulus of pre-twisted copper wires was obtained. In order to ensure the repeatability, at least three specimens were separately tested for each nominal gauge length.

Indentation Tests

Nanoindentation tests of the copper wires were carried out by using the Agilent Nano Indenter G200 equipped with a Berkovich diamond indenter. The experimental strain rate is 0.05 s^{-1} and the maximum indentation depth is 400 nm. Fifteen points were tested on each specimen, as shown in Fig. 1, and the distance was 20 μm between two indentation points along the diameter direction. The distance between indentation points and the periphery of the specimen is 20 μm at least. Generally, at least three specimens were separately tested for each type copper wire. The data for per indentation location is given as the average of three specimens.

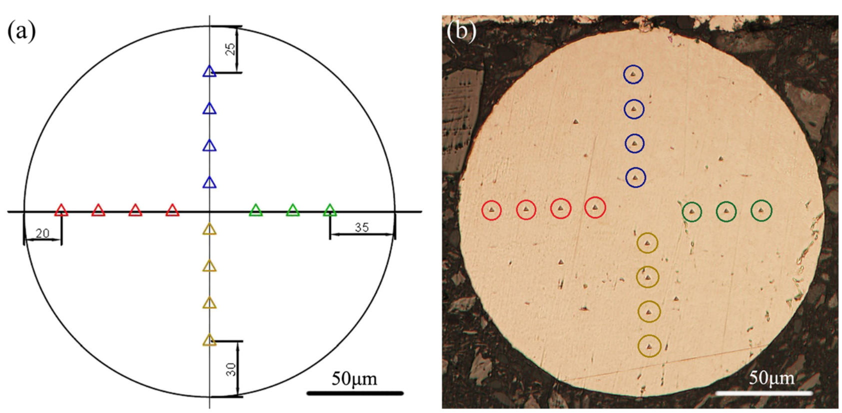

Fig. 1 Indentation location distribution: (**a**) schematic diagram and (**b**) physical picture of optical microscope

Table 1 The testing results of initial and pre-twisted copper wires: G -apparent elastic shear modulus, E_T-Tensile modulus, E_N- Indentation modulus

material parameter		Initial copper wire	Pre-twisted copper wire
Elastic mechanical parameters	G (GPa)	36.9 ± 2.3	32.0 ± 1.0
	E_T (GPa)	98.7 ± 0.6	90.4 ± 0.8
	E_N (GPa)	136.5 ± 1.4	
Microstructure	average grain diameter (μm)	2.5	0.56
	Faults Density (μm μm^{-2})	3.49	12.15

The indentation modulus is determined from indentation load–displacement data obtained during one cycle of loading and unloading. It can be computed from below equations:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1+\nu_i^2}{E_i} \tag{2}$$

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \tag{3}$$

$$A = 24.5h_c^2 + \sum_{i=0}^{7} C_i h_c^{1/2^i} \tag{4}$$

Where E and ν are indentation modulus and Poisson's ratio for the specimen and E_i and ν_i are the same parameters for the indenter. E_r is the composite response modulus. S is the contact stiffness. A is the projected area of the elastic contact. β is a constant. h_c is the contact depth. C_i is constant that describes the imperfections of the tip from ideal geometry. For Berkovich diamond indenter, β =1.034, E_i =1141GPa, ν_i =0.07.

Results and Discussion

Testing results of the initial and the pre-twisted copper wires are listed in Table 1. Stress–strain curves and load–displacement curves are shown in Figs. 2 and 3 respectively. The results show that the elastic mechanical parameters of pre-twisted copper wires are smaller than those of initial copper wires. The apparent elastic shear modulus of the copper wires decreases by about 13.3% after pre-twist deformation. The decrease of the apparent Young tensile modulus is about 8.4%.

In this study, as is shown in Table 1 and Fig. 4, the microstructure of the copper wires has been also investigated. Average diameter represents the grain size, and fault density reflects total length of the grain boundary and subboundary per unit area. The fault density is obtained with the aid of Image-Pro Plus software which can identify the grain automatically by distinguishing the color intensity value of the image. In order to identify the grain accurately, the grain boundaries and subboundaryies are delineated using

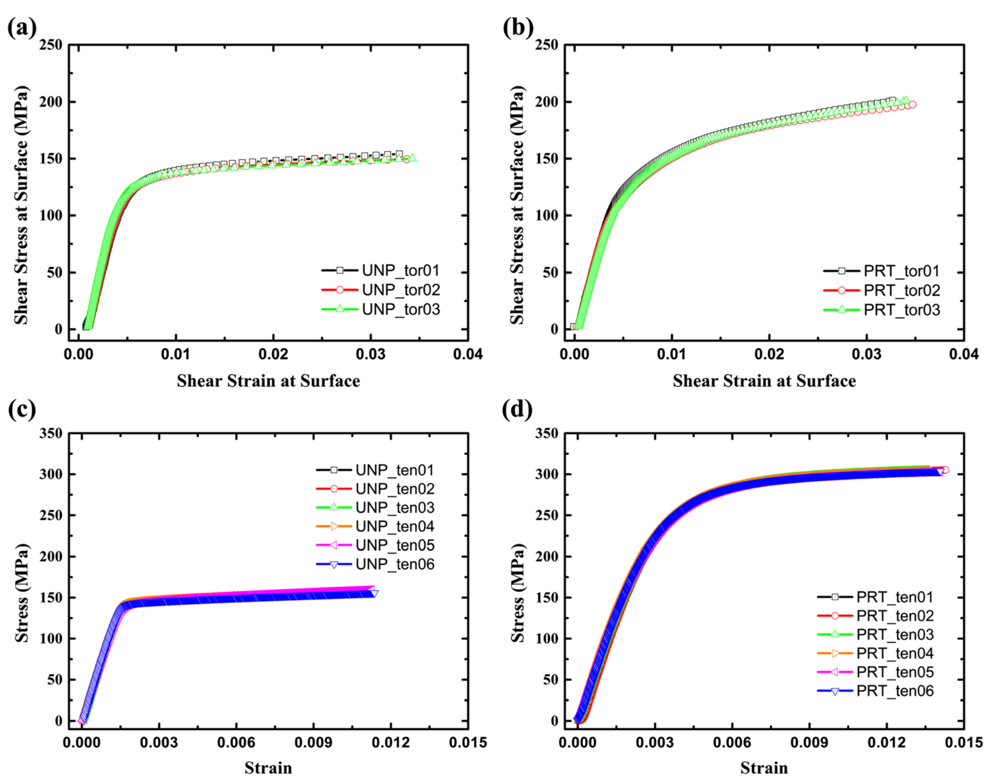

Fig. 2 The stress–strain curves of tests: (**a**) Shear stress–strain curves at surface of initial copper wires; (**b**) Shear stress–strain curves at surface of pre-twisted copper wires; (**c**) Tensile stress–strain curves of initial copper wires; (**d**) Tensile stress–strain curves of pre-twist copper wires. Note: Tor01, tor02 and tor03 represent the specimens with nominal gauge length of 5 mm. Ten01, ten02 and ten03 represent the specimens with nominal gauge length of 100 mm. Ten04, ten05 and ten06 represent the specimens with nominal gauge length of 55 mm

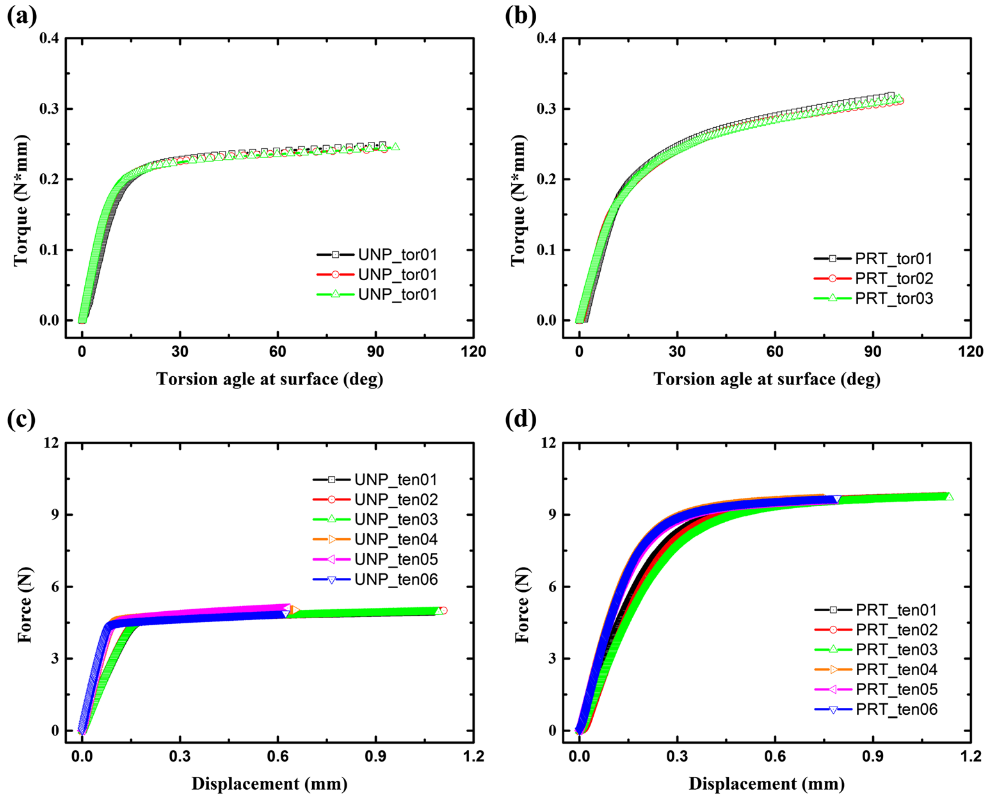

Fig. 3 The torque–torsion angle and load–displacement curves of tests: (**a**) Torque–torsion angle curves of initial copper wires; (**b**) Torque–torsion angle curves of pre-twisted copper wires; (**c**) Load–displacement curves of initial copper wires; (**d**) Load–displacement curves of pre-twisted copper wires

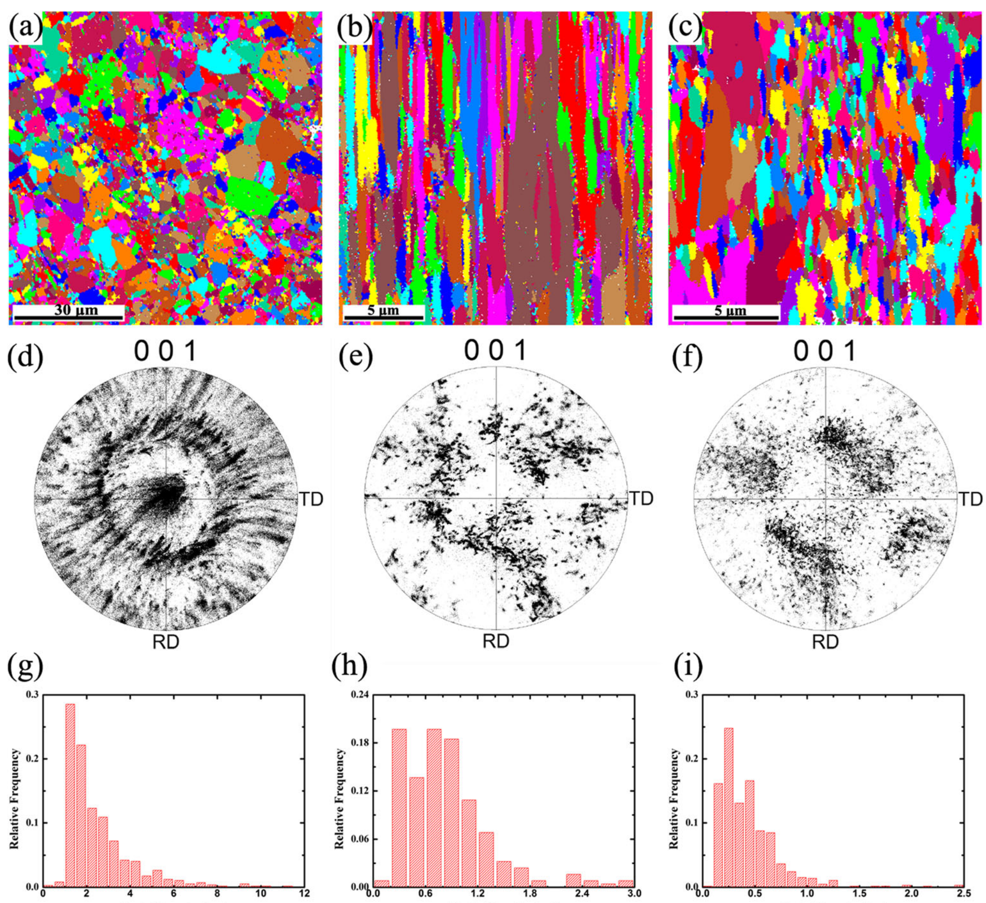

Fig. 4 The microstructure of copper wires: (**a**) and (**d**) the pole figure of initial copper wires; (**b**) and (**e**) the pole figure of pre-twisted copper wires near center; (**c**) and (**f**) the pole figure of pre-twisted copper wires near surface; (**g**) the grain size cartogram of initial copper wires; (**h**) the grain size cartogram of pre-twisted copper wires near center; (**i**) the grain size cartogram of pre-twisted copper wires near surface

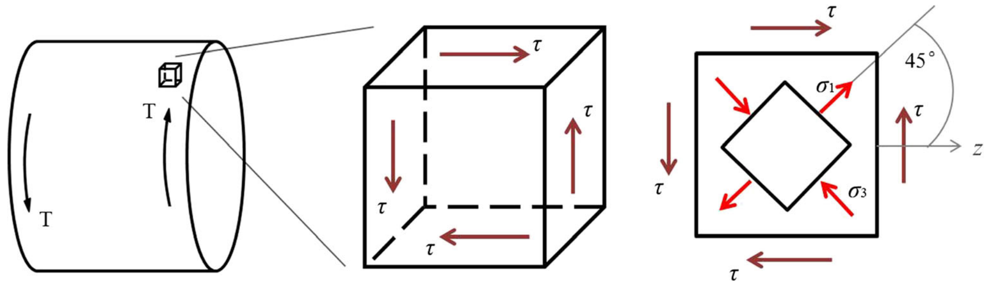

Fig. 5 The stress state analysis of pre-twist deformation

photoshop software firstly. Then, grains are filled with different colors. Finally, total length of the grain boundaries and subboundaries and total area are obtained using Image-Pro Plus software. Results show that the difference between the initial and the pre-twisted copper wires is obvious in the average diameter and the fault density. The average grain size of initial copper wires is 2.5 μm. However, the average grain size of pre-twisted copper wires is 0.56 μm. The fault density of pre-twisted copper wires is three times more than initial copper wires. According to Hall-Petch relationship, it is known that the yield stress increases as the grain size is decreased. As shown in Fig. 2 (c) and (d), despite the diminution of elastic modulus the yield stresses after a pre-twisted are greater that the initial ones. This is because the grains of pre-twisted copper wires are obviously refined.

For the pre-twist deformation, stress state analysis of an arbitrary element taken from the copper wires is given in Fig. 5. The angle between principal stress and the axis z is 45°. The deformation direction of element is consistent with the direction of principal stress. The pre-twisted copper wire has a deformation increasing along its radial direction, with the 100% shear deformation produced on the outside surface. Therefore, strain gradient is produced due to pre-twist. During the pre-twist deformation, slip bands are generated within each equiaxed grain when the strain of the copper wires reaches a certain value. Then, geometrically necessary boundaries (GNBs) are initiated within the grain to adjust lattice bending. The density of the GNBs increases with the increase of the strain gradient. Next, the dislocations begin to form cell walls and then develop into subboundaies. At the meantime with the strain continuously increasing, the grains are elongated in the direction of the principal stress to columnar crystal. Finally, the elongated grains are broken down into fine equiaxed grains with the accumulation of strain [12, 13], as shown in Fig. 6.

There are many fault structures (grain boundary, subboundary and dislocation) within the pre-twisted copper wires. These faults are in the state of non-equilibrium of energy. Lattice distortion is large in the surrounding area of faults. The degree of the lattice distortion is greater in the region of grain boundary and the interatomic bonding capacity is weaker. Elasticity modulus is the result of the coupling between the modulus of normal lattice-zone and the modulus of fault-zone. Therefore, mechanical behaviors of metallic material are largely affected by the faults. More the faults proportion is greater, smallest is the elasticity modulus.

In addition, Fig. 7 presents the relationship between indentation modulus and indentation position. It is noted that measuring principle and mechanical model of the tensile testing and the indentation testing are quite different so that the modulus measured with two methods is not simple equivalent. Generally, the indentation modulus is greater than the tensile modulus [14]. The indentation modulus, reflecting the mechanical properties of materials on partial region, is related to structure and arrangement of atom at the indentation position. As shown in Fig. 7, it can be concluded that the indentation modulus of initial copper wires is independent of the indentation position. The indentation modulus of pre-twisted

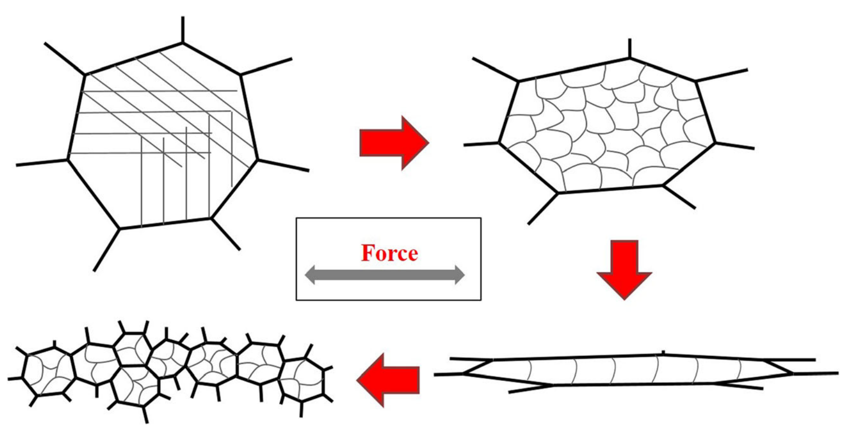

Fig. 6 Appearance of grain subdivision due to plastic deformation

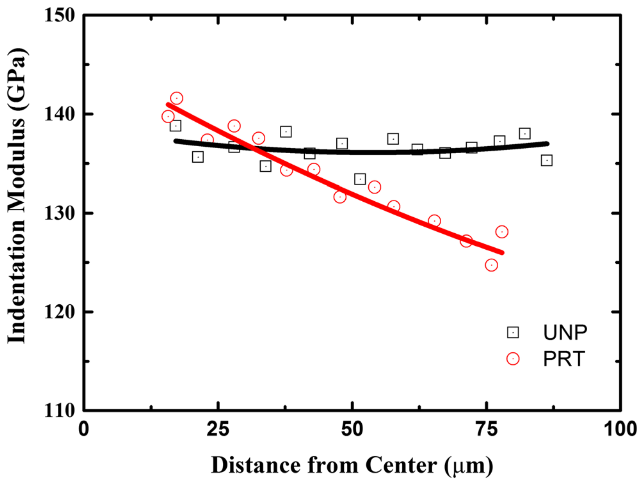

Fig. 7 The variation of the indentation modulus versus different indentation position: UNP and PRT represent the initial and pre-twisted copper wires, respectively. The indentation modulus of pre-twisted copper wires diminishes gradually with the increasing of deformation

copper wires diminishes as the indentation position close to the surface. Furthermore, when the deformation is more than 30%, the indentation modulus of pre-twisted copper wires is gradually smaller than that of initial copper wires.

Conclusions

In this paper, the elastic mechanical parameters of initial and pre-twisted copper wires are tested respectively. The pre-twist deformation has significant influence on the elastic mechanical parameters of copper wires. The apparent elastic shear modulus and the tensile modulus of copper wires are decreased after pre-twist deformation. By analyzing the microstructure of all specimens with the aid of EBSD, it is indicated that the fault density is an important factor. In addition, the indentation modulus decreases with the increase of deformation. When the deformation is more than 30%, the indentation modulus of pre-twisted copper wires is smaller than that of initial copper wires. The conclusion drawn from experimental research and theoretical analysis is that the fault density is the dominant factor causing the decrease of the elastic mechanical parameters of micro-scale metallic material after pre-twist deformation.

Acknowledgments This work is supported by the National Natural Science Foundation of China (Grant Nos. 11372323), and CAS Key Technology Talent Program and the project of function development on scientific instruments of Chinese Academy of Sciences.

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