Accepted Manuscript

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PII: S0263-2241(19)30242-8
DOI: https://doi.org/10.1016/j.measurement.2019.03.034
Reference: MEASUR 6469

To appear in: *Measurement*

Received Date: 2 December 2018
Revised Date: 8 March 2019
Accepted Date: 13 March 2019

Please cite this article as: B. Chen, R. Yang, J. Dong, S. Wang, J. Wang, Y. Huan, Development of In-situ SEM Torsion Tester for Microscale Materials, *Measurement* (2019), doi: https://doi.org/10.1016/j.measurement.2019.03.034

Development of In-situ SEM Torsion Tester for Microscale Materials

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Abstract

An in-situ SEM (scanning electron microscope) micro-torsion tester with high resolution is developed based on electromagnetism. The torque is controlled and measured using a coil-magnet component. The torsion angle is measured by a non-contact transducer based on Hall-effect. The calibration results show that the torque capacity of this tester is 1.6×10^{-4} Nm with resolution of 1×10^{-9} Nm. In-situ torsion test of MG (metallic glass) wire ($Pd_{40}Cu_{30}Ni_{10}P_{20}$) with a diameter of 111 μm and a length of 3 mm is performed using this tester in SEM. A linear torque-torsion angle curve is obtained and the calculated shear modulus of the sample is 24.7 GPa. Meanwhile, the deformation process of the sample is simultaneously observed using SEM. It is the first time that the measurement of shear stress-strain and high resolution morphology observation are carried out simultaneously.

Key words: in-situ; torsion test; micro-scale; metallic glass wire

1. Introduction

In-situ SEM test techniques, which combine quantitative testing and high resolution imaging, extend the capability of SEM and open a new gate to the micro/nano-world. In the past three decades, there have been a lot of reports on the in-situ test techniques of SEM, such as tension/compression [1-3], micro/nano-indentation [4,5] and bending tests [6–9]. However, little was focused on the torsion test of micro-wires in SEM. Recently, Jiang et al [10] twisted MG wires in SEM with the aid of a micro-robot. However, only the deformation process is captured. The key information of mechanical properties, namely the shear stress-strain curve, is not obtained because the testing system has no capability of torque measurement. From the above, it can be concluded that the in-situ test techniques with axial load, such as tension/compression, bending and indentation, are relatively mature. Some commercial instruments are available, for example, Hysitron PI85 [11] and Gatan Microtest200 [12]. However, the in-situ torsion test technique for micro-scale samples is relatively backward. The main reason is due to the challenges in these experiments, such as the torque measurement, the horizontal working mode and the size miniaturization of the instrument.

As samples' size decreases to micro-scale, the torque sharply drops to10^{-4} Nm or even less, which is far beyond the ability of the commercial torque sensors. As an alternative, more delicate components, such as glass fibers or tungsten wires are adapted as torque sensors [13-15]. However, vertical working mode has to be used in this kind of instrument in order to fit the slender sensors, which makes the integration with SEM impossible. In addition, AFM (Atomic Force Microscope) tips [16] and MEMS (Micro-Electro-Mechanical System) probes [17] have also been used to measure tiny torques. However, the size of the device can't be miniaturized further to fit into SEM because the low-level integration of hardware.

Thus, an alternative approach has to be found. In 2015, a micro-torsion tester [18], used outside of SEM, was developed based on electromagnetism technique by our team, in which a coil-magnet component was employed for actuator and torque measurement. In addition, the reliability of this technique has been verified by the torsional pendulum experiment [19]. Based on the fore-mentioned work, we developed this technique, which can significantly shrink the device's size, thus improving the level of integration. Moreover, the coil-magnet component has the potential of being horizontally arranged. All these characteristics indicate this technique is suitable for integrating with SEM.

Therefore, an in-situ SEM micro-torsion tester, with horizontal working mode and small size, is developed based on electromagnetism in this work. Subsequently, MG wire ($Pd_{40}Cu_{30}Ni_{10}P_{20}$) with a diameter of 111 μm and a length of 3 mm was twisted using this tester in SEM. It is the first time that the measurement of shear stress-strain and high resolution morphology observation are carried out simultaneously, which can benefit the study of mechanical behavior of micro-scale materials.

2. In-situ SEM micro-torsion tester

2.1 Design principle

The in-situ SEM micro-torsion tester, shown in figure 1(a), is self-developed based on electromagnetism, in which a coil-magnet component is used for actuator and torque measurement. The structure of the tester is illustrated in figure 1(b). The angle sensor and one grip are attached to the coil by framework. Another grip is installed on the baseplate. The sample is fixed between the two grips. The coil is supported by two bearings, which are specially designed in axial direction with about 0.5 mm motion allowance to avoid additional axial force on sample. In order to satisfy the work conditions in SEM, the coil-magnet component is placed horizontally in this tester, which increases the gripping difficulties of delicate samples. In addition, the structure is designed specially in order to avoid the influence of magnetism on SEM electron beam. Specifically, there is a sleeve, made of high-permeability materials, that around the inner magnet to reduce the leakage magnetic flux. The framework, between the grip and coil-magnet component, is designed to be very long, that the inner magnet is far away from the electron beam.

The principle of the coil-magnet component is shown in figure 1(c). When the current passes through the coil, the raw torque T_{me}, generated by Ampere force, can be easily measured as following:

$$T_{me} = Fd = nBILd = \alpha I \tag{1}$$

Where F is the Ampere force produced by the coil, n is the number of coil turns, B is the magnetic flux density at the coil area, I is the current that passes through the coil, L is the length of wire in the magnetic field region and d is the width of the coil frame. It is obvious that the raw torque T_{me} is in direct proportion to the current I in a uniform magnetic field. The sensitivity coefficient α is a constant depending on the structure of the coil-magnet component, which can be determined by the calibration work. According to I, the raw torque T_{me} can be easily measured with high resolution.

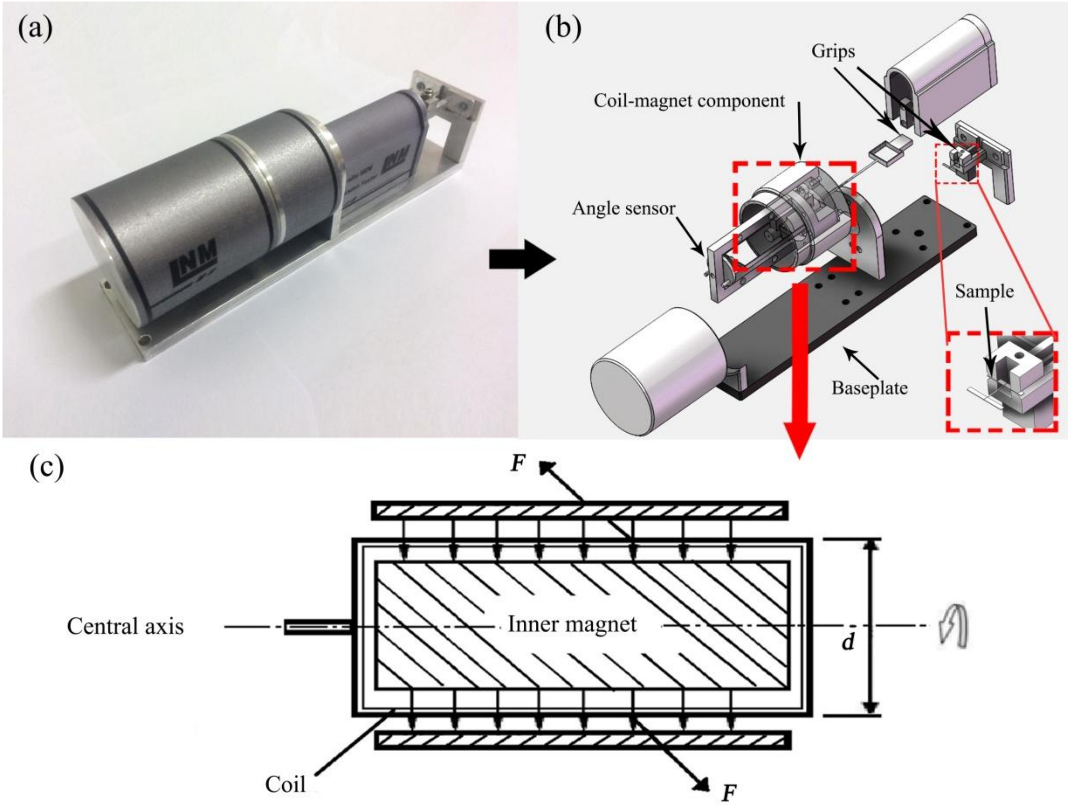

Figure 1. Illustration of the in-situ SEM micro-torsion tester. (a) the picture of the tester (b) The structure of the tester. (c) the principle of coil-magnet component.

In order to reduce the influence of the torque measurement, a non-contact angle transducer based on Hall-effect is applied to measure the torsion angle of the coil θ_{me}. To cooperate with SEM, a mini size framework is specially designed. It allows the electron gun of SEM to be as close to the sample as needed, and far away from the coil-magnet component to reduce the influence of magnetism.

2.2 Mechanical model

Although the raw torque can be easily obtained, the true torque on sample is not absolutely equal to it because of some technical compromises. Firstly, the coil must be supported to rotate in the magnetic field, which introduces the friction torque f of the bearings. Secondly, the coil must be connected to the power, which introduces the stiffness of the lead wire K_l. Thirdly, the center of torsion mass can't align to the torsion axis, which introduces the eccentric torque $T_d(\theta_{me})$. Furthermore, it should be noted that the eccentric torque $T_d(\theta_{me})$ is a location-dependent variable. In order to determine all these influences, the mechanical model of the tester is illustrated in figure 2. The true torque on sample is:

$$T = S \times \theta \quad (2)$$

Where S is the stiffness of sample, θ is the true torsion angle of the sample. Considering the deformation of the framework, the true torsion angle of the sample θ is:

$$\theta = \theta_{me} - \frac{T}{K} \quad (3)$$

Where K is the stiffness of the framework. Thus, T is calculated by the following formula:

$$T = T_{me} - f - T_d(\theta_{me}) - K_l \times \theta_{me} \quad (4)$$

All above parameters, including α, f, $T_d(\theta_{me})$, K_l, K, can be obtained by the following calibration work.

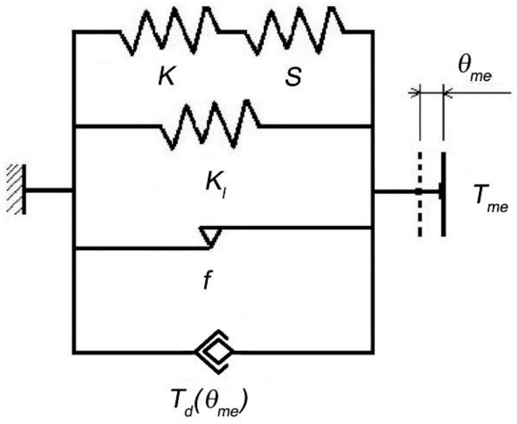

Figure 2. The mechanical model of the in-situ SEM micro-torsion tester. Where K is the stiffness of the framework, S is the stiffness of sample, K_l is the stiffness of the lead wire, f is the friction torque of the bearings, θ_{me} is the torsion angle measured by the non-contact angle transducer, and $T_d(\theta_{me})$ is the eccentric torque. T_{me} is the torque generated by the coil.

2.3 Calibration

The relationship between the current and the raw torque is calibrated using an electronic balance with resolution of 0.1 mg. A slender bar was perpendicularly fixed on the central axis. A probe at the end of the bar is driven by the coil to press the balance pan, shown in figure 3. The change of the balance reading can be transformed into the press force F_p, which can be further transformed into the raw torque.

The relationship between the current and the raw torque can be obtained when the length of force arm is taken into account. In this calibration work, the length of force arm is 34.62 mm measured by Vernier caliper. The result is shown in figure 4. A good linear relationship between T_{me} and I is observed, and the constant α is 4.13×10^{-7} Nm/mA

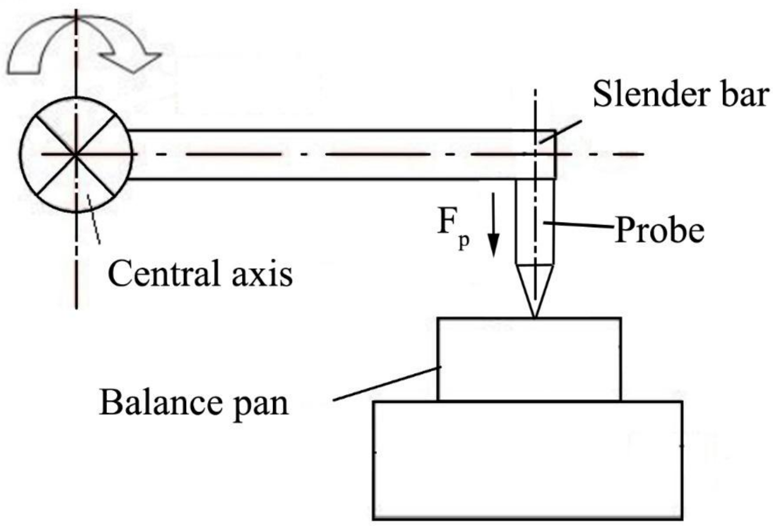

Figure 3. Schematic diagram of the torque calibration.

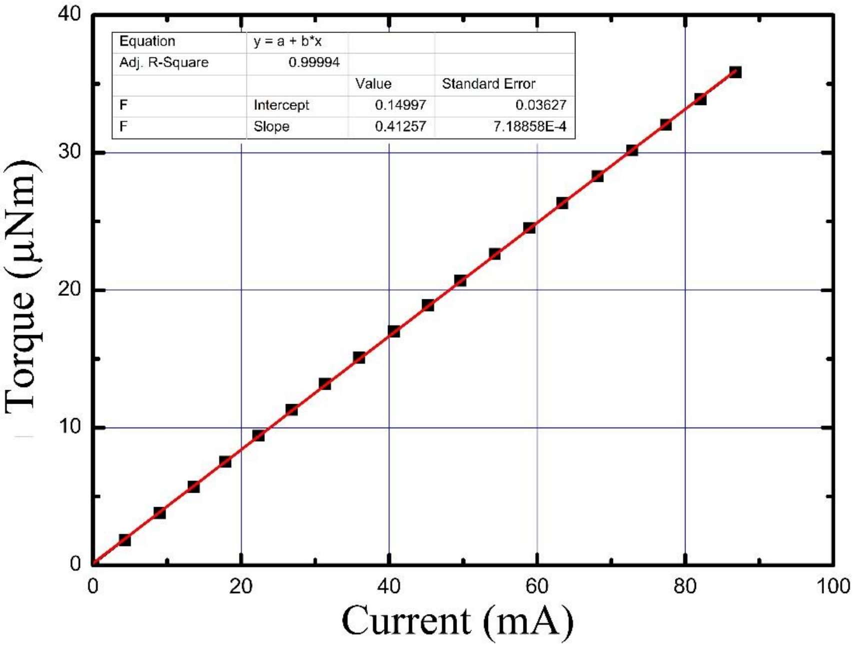

Figure 4. The calibration results of the relationship between the current and the raw torque.

The stiffness of framework K can be calibrated if the grips of champing module are fixed together. The result shows that K is 8×10^{-4} Nm/deg, which is much larger than the sample's (10^{-5}~10^{-7} Nm/deg).

In order to calibrate the friction torque f, the tester is operated without any external load. At the start, the coil will remain stationary for a moment until the current increases to a specific value. The corresponding torque is just f. The result shows that f is 1.3×10^{-7} Nm.

The calibration of the eccentric torque $T_d(\theta_{me})$ is complicated because $T_d(\theta_{me})$ is a variable along with the torsion angle. In addition, the influence of the lead wire is a variable along with the torsion angle too. In order to simplify this calibration work, the total effects $\alpha * I_d(\theta_{me})$ of the eccentric torque and the lead wire are calibrated together. The relationship between $I_d(\theta_{me})$ and θ_{me} is recorded while the tester is operated without any external load. The curve of $\alpha * I_d(\theta_{me})$ and θ_{me} is shown in figure 5. The following polynomial function is used to fit this curve, which is:

$$T_d(\theta_{me}) + K_l \times \theta_{me} = \alpha * I_d(\theta_{me}) = \sum_{i=0}^{n} k_i {\theta_{me}}^i \quad (5)$$

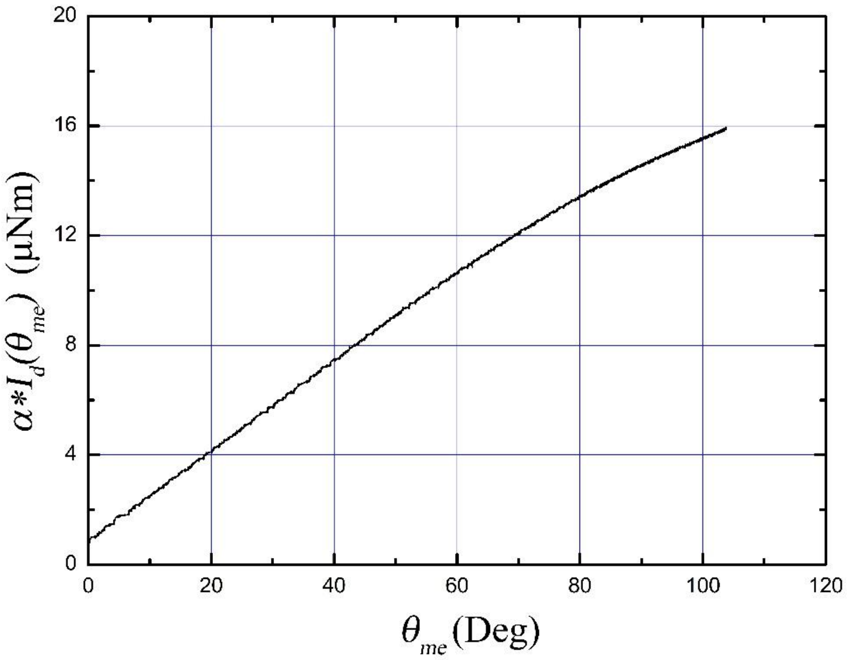

Figure 5. The calibration curve of $\alpha * I_d(\theta_{me})$ and θ_{me}.

The torque capacity of this tester is 1.6×10^{-4} Nm and the torsion angle range is 100° in order to consider the safety of circuit and angle allowance. According to the actual data, collected by data acquisition card, the resolution of torque is 1×10^{-9} Nm and the resolution of torsion angle is 0.001°. The main technical parameters are listed in Table 1.

Table 1. Main technical parameters of the in-situ SEM micro-torsion tester

	Content	**Value**
Torque	Capacity	1.6×10^{-4} Nm
	Resolution	1×10^{-9} Nm
Torsion angle	Range	100°
	Resolution	0.001°

The formula for transforming the torque to the shear stress and the angle to the shear strain are following:

$$\tau = \frac{16T}{\pi D^3} \tag{6}$$

$$\gamma = \frac{\theta D}{2l} \tag{7}$$

Where τ is the shear stress at sample's surface, D is the diameter of the sample, γ is the shear strain at sample's surface and l is the gauge length of the sample.

3. Application on MG wire

In order to inspect the effectiveness of the self-developed tester, the torsion test of MG wire ($Pd_{40}Cu_{30}Ni_{10}P_{20}$) with a diameter of 111 μm and a length of 3 mm was performed in SEM. The in-situ SEM micro-torsion tester, with the sample having been fixed between the two grips, is installed in

SEM (JEOL JSM-IT300), as shown in figure 6(a) and (b). In order to get clear SEM pictures, load is step by step applied on the sample.

The torque-torsion angle curve is measured by the in-situ SEM micro-torsion tester. A linear relationship between the torque and torsion angle is obtained, shown in figure 7(a). Then, the relationship of shear stress-strain at sample's surface can be obtained, shown in figure 7(b). The tested shear modulus is 24.7 GPa. Meanwhile, the deformation process of the sample is observed using SEM. Different deformation states of the sample are shown in figure 7(c)-(g), respectively corresponding to "c" - "g" in figure 7(a) and (b). From the two obvious markers "A" and "B" on the sample's surface, we can find that mark "B" is gradually moving down relative to mark "A" in figure 7(c)-(g), which indicates the twisting progress.

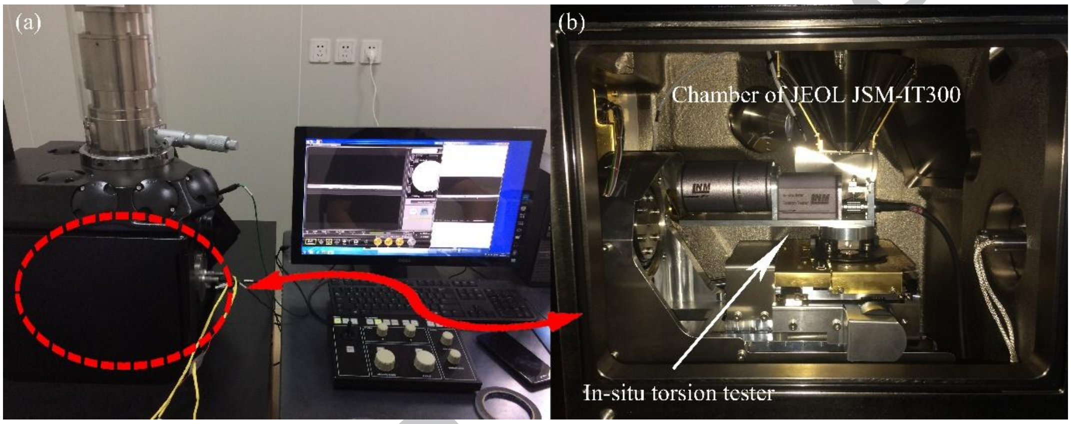

Figure 6. Setup of the in-situ SEM micro-torsion test. (a) Overall view. (b) Close-up inside SEM.

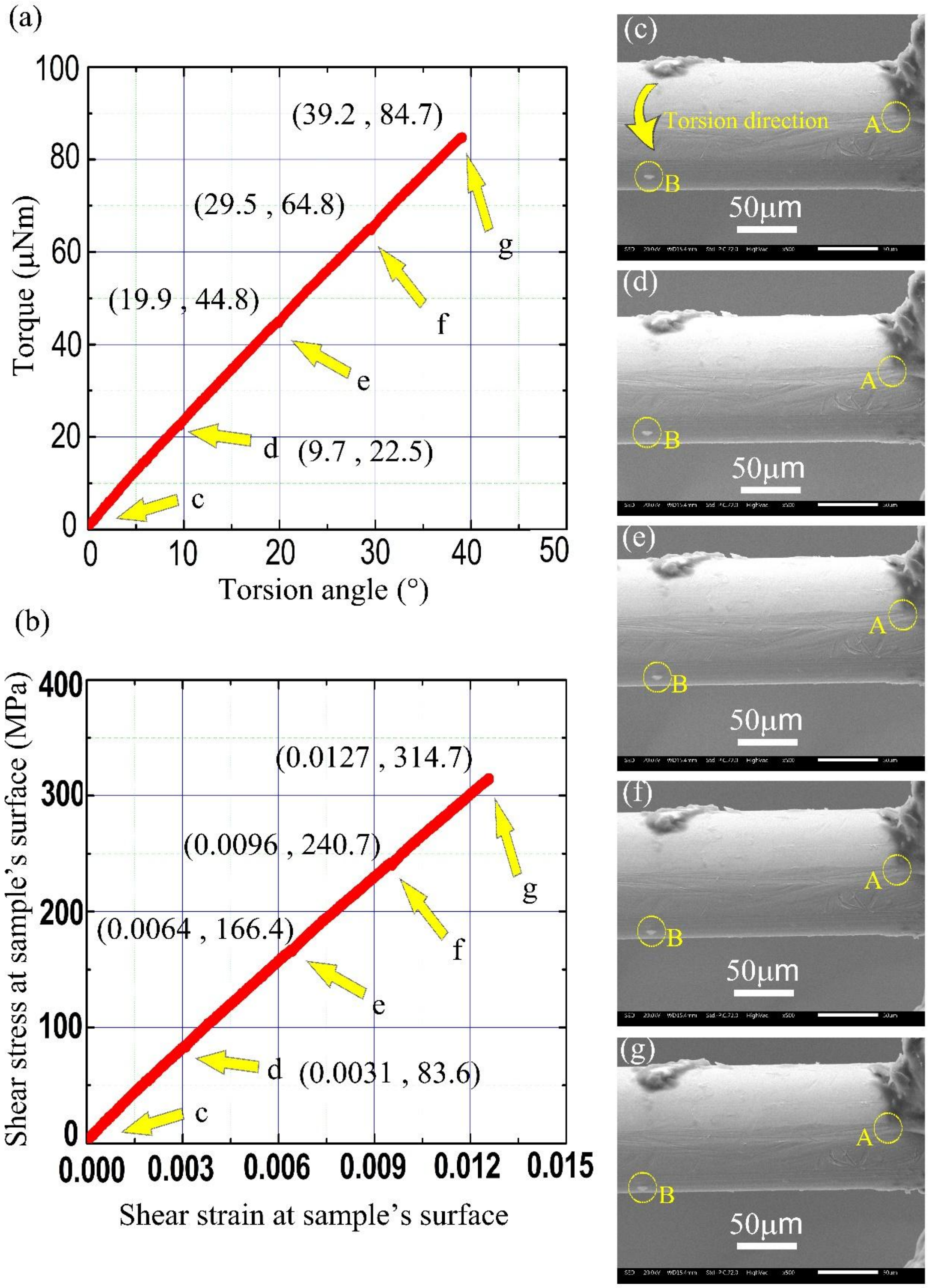

Figure 7. Results of the in-situ SEM micro-torsion test of MG wire. (a) Torque-torsion angle curve. (b) Relationship between shear stress and shear strain at sample's surface. (c)-(g) The deformation process of the sample, corresponding to "c" to "g" in (a) and (b).

4. Summary

An in-situ SEM micro-torsion tester with function of stress-strain measurement was developed based on electromagnetism. The torque capacity is 1.6×10^{-4} Nm with resolution of 1×10^{-9} Nm and the torsion angle range is 100° with resolution of 0.001°.

In-situ torsion test of MG wire ($Pd_{40}Cu_{30}Ni_{10}P_{20}$) with a diameter of 111 µm and a length of 3 mm is performed using this tester in SEM. A linear torque-torsion angle curve is obtained and the calculated shear modulus of the sample is 24.7 GPa, which is close to the former literature value [18]. Meanwhile, the deformation process of the sample is observed using SEM. It is the first time that the measurement of shear stress-strain and high resolution morphology observation are carried out simultaneously. In future work, we are planning to do more experiments to precisely investigate the influence factors, such as axial force, eccentric torsion and so on, in order to deepen our understanding of this technique and expand its applications.

Acknowledgements

This work was supported by the National Natural Science Foundation of China (Grant Nos. 11372323, 11772336), CAS Key Technology Talent Program and the Project of Function Development on Scientific Instruments of Chinese Academy of Sciences.

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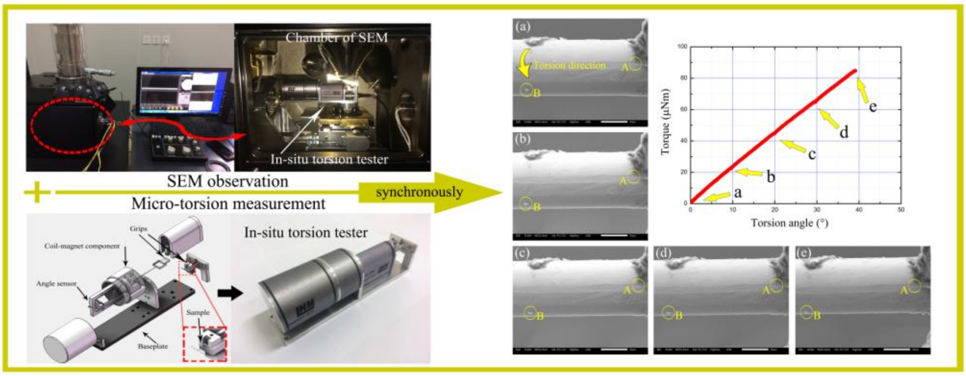
Chamber of SEM
In-situ torsion tester
SEM observation
Micro-torsion measurement
synchronously
In-situ torsion tester
Grips
Coil-magnet component
Angle sensor
Sample
Baseplate
(a)
Torsion direction
A
B
(b)
(c)
(d)
(e)
Torque (μNm)
Torsion angle (°)
a
b
c
d
e

Highlights

1. The in-situ SEM torsion test technique for microscale materials was developed.
2. In-situ SEM torsion test technique is a new way for studying microscale materials.
3. It's the first time that measuring $\tau - \gamma$ curve and SEM observing simultaneously.