Direct measurement of torsional properties of single fibers

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Abstract

In order to characterize the torsional behavior of micron-scale specimens, a direct technique is established based on the principle of torsion balance. The technique applies twist to the specimen and balances the resulting torque against a torsion wire of known torsional rigidity. The torsional rigidity of the torsion wire is determined by a torsion pendulum. To measure the rotation of the torsion wire, a sensitive angle detector, comprising a thin cross-beam attached between the torsion wire and the fiber specimen and a laser displacement sensor, is developed. The presented technique permits the measurement of torque in single fibers as low as $10^{-9} \text{Nm}$ with a reasonable resolution. Using this technique, torsion tests on micro-diameter copper wires, silver wires and carbon fibers were performed. The longitudinal shear modulus and other torsional properties of these samples, such as yielding shear strength, were obtained.

Keywords: torsion balance, torsion pendulum, micro-diameter, single fiber, shear modulus

(Some figures may appear in colour only in the online journal)

1. Introduction

Quasi-static torsion testing on small specimens has been recognized as an excellent approach to the study of the mechanical behavior of small-scale specimens, from elastic deformation, through yielding, to the strain-hardening regime, whereby strain gradients are naturally generated in plasticity [1–3]. There are two main factors motivating the development of torsion testing techniques for single fibers/wires: the recent interest in size-dependent plasticity at small scales [4] and the requirement for the shear properties of single high-performance fibers in textile manufacture [5] and in the design of high-strength composites [6]. However, compared with tensile techniques used at small scales that have been mature commercially [7, 8], the development of torsion techniques at small scales has been rather slow due to several challenges [1], including difficulties in detecting micro-torque and in handling small specimens. Over the past decades, a series of non-standard techniques for measuring the torsional properties of single fibers/wires have been proposed. Most of them are based upon the principle of torsion balance [9] that can be traced back to the landmark works by Charles Coulomb [10] and Henry Cavendish [11]. In fact, torsion-balance devices are ubiquitous in experimental physics, measurement science and various other fields of engineering [9]. Here, we only focus on the torsion-balance techniques applied in torsional characterization of single fibers, wires and yarns.

In an ideal torsion balance for thin fibers, the specimen is generally fixed between a twisting head at its lower end and a torsion wire of known properties at its upper end. The upper end of the torsion wire can be fixed or be rotated manually. A pointer, or other indicating device, is positioned between the torsion wire and the fiber specimen and a laser displacement sensor, is developed. The presented technique permits the measurement of torque in single fibers as low as $10^{-9} \text{Nm}$ with a reasonable resolution. Using this technique, torsion tests on micro-diameter copper wires, silver wires and carbon fibers were performed. The longitudinal shear modulus and other torsional properties of these samples, such as yielding shear strength, were obtained.

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suspended from a torsion head. Following a similar principle, Huan et al [13] recently used a coil placed in a radial magnetic field to measure the torque of micro-scale specimens. Additionally, an optical lever system has been widely used to measure the rotation of the indicating device [5, 6, 14–16]. For example, Mitchell and Feughelman [14] used a photographic film to record the movement of the light image in an optical-lever system. Postle et al [15] adopted a lamp-and-scale arrangement to measure the torque in a twisted yarn. Skelton [17] used a circular scale to detect the angular rotation of the mirror that was attached between the torsion wire and the specimen. More recently, Behlow et al [6] proposed a design for the measurement of torque using a position sensitive-detector based optical device that tracks a torsion reference. Rather than using an optical lever system, Kawabata [18] adopted a pair of linear differential transformers to detect the rotation of the torsion wire. Mccord and Ellison [19] employed an optical encoder module and a slot incremental code wheel disk to measure the rotation of the torsion wire. The main issue with these two methods is that the moment of inertia attached to the torsion wire seems too large to give an instantaneous measurement of torque. Another problem is the influence of friction on the torque measurement introduced by the jewel bearing [6, 13] or air bearing [19].

On the other hand, in 1994, Fleck et al [3] developed a screw-driven torsion balance to perform torsion tests on polycrystalline copper wires ranging in diameter from 12 to 170 µm to study the strain-gradient effect in plasticity. Since Fleck et al’s pioneering work [3], torsion of thin metallic wires has been recognized as a benchmark experiment in

Figure 1. (a) Schematic diagram of the torsion tester. (b) Diagram of the optical detection system for detecting the rotation angle of the torsion wire.
micromechanics [4]. However, it is hard to distinguish the elastic range from the normalized torque-twist curves in Fleck et al.’s experimental data due to the poor resolution of their apparatus. This drawback has motivated many other researchers to develop a range of new techniques for measuring wire torsion, including the improved fiber-based torsion balance [1, 20, 21], an atomic force microscopy-based torsion balance [20, 22] and a load–unload technique [2, 23, 24]. However, most of techniques mentioned above are unable to test single fibers/wires with diameter of less than 10 µm [20, 22]. Also the calibration methods are all different, making the comparability and traceability of the experimental results worse.

As reviewed by Liu et al [25], another well-known method for measuring the torsional properties of single fibers is the torsion-pendulum technique. Although the torsion-pendulum approach is simple in design and rapid in application, the information it provides is severely limited since the rigidity is only measured for small strains. It is generally unable to provide information related to plasticity, creep or hysteresis [26]. The torsion-balance technique introduced here overcomes many of these limitations, and can be used to measure the torsional yield and plasticity information as well as the shear stress–strain response.

In this paper, a new torsion tester with high resolution (up to $10^{-10}$ N m) and reasonable precision (about 8%) for measuring the torsional properties of individual fibers is established based on the principle of torsion balance. In this tester, a fiber specimen is subjected to a given twist and the resulting torque is measured by a torsion (tungsten) wire of known torsional rigidity. The inserted twist can be controlled accurately so that both the shear modulus and the shear yield strength of specimens can be determined. The sensitivity and reproducibility of the technique is discussed. Finally, the proposed technique is applied to investigate the torsional properties of micro-diameter copper and silver wires and carbon fibers.

2. Experimental details

2.1. Torsion balance setup

Based on the static torsion-balance principle, a torsion tester has been established for measuring the torque-twist curves of an individual fiber, as shown in figure 1(a). It is designed to balance the torque acting on the specimen against the torque of a torsion wire (i.e. a torque meter). In this arrangement, a fiber specimen is fixed between a twisting head at its lower end and a torsion wire (a tungsten wire is used here) of known torsional rigidity at its upper end. The upper end of the torsion wire of length $l$ and of diameter $d$ is glued, using cyanoacrylate glue, onto a thin rigid beam mounted on a 3D translation stage. The lower end of the fiber specimen is attached to a tensioning mass made of one or two washers to give a constant stress of about 2–3 MPa, far below its yield strength, during the measurement. The tension mass is inserted into a slot (i.e. twisting head) to prevent lateral movement while still allowing the mass to slide freely in the
vertical direction, as shown in figure 1(a). The twisting head is mounted on the stepper motor (i.e. turntable) located at the 3D translation stage. A constant rate of twist is applied to the specimen by the stepper motor.

As the specimen is twisted, the applied torque in the fiber specimen is transmitted to the torsion wire through the stiff cross-beam. Twisting of the specimen leads to a rotation of the cross-beam, resulting in a torque in the torsion wire. At equilibrium, the torque in the torsion wire is equal and opposite to that in the specimen, i.e.

$$\theta = \frac{T}{K}$$

where $\theta$ is the rotation of the cross-beam, and $K$ is the torsion constant (torque per unit twist) of the torsion wire. The angular displacement $\theta$ is detected by an optical detection system (see figure 1(b) for details), which is calculated as

$$\theta = \arctan(\delta/L)$$

where $\delta$ is the distance of the laser spot deviated from the initial position, and $L$ is the horizontal distance between the laser spot and the torsion wire centered in the cross-beam. The twist of the specimen is the difference between the torsion angle applied by the stepper motor and $\theta$. Then the relative twist of the two ends of the specimen is

$$\varphi = \psi - \theta$$

where $\psi$ is the twist angle given by the stepper motor. If a suitable torsion wire is used, the value of $\theta/\psi$ can be less than $10^{-4}$. Therefore, one can adopt the twist angle provided by the stepper motor, $\psi$, to denote the twist inserted into the specimen, i.e. $\varphi \approx \psi$.

If the value of $K$ in equation (1) is known, the torque $T$ can be obtained. It is known that the value of $K$ depends on the length, $l$, and the torsional rigidity, $GIp$, of the torsion wire, i.e.

$$K = \frac{Gl_\theta}{l} = \frac{G\pi d^4}{32l}$$

where $G$ is the shear modulus, $d$ is the diameter of the torsion wire and $I_\theta$ is its moment of inertia. The determination of $G$ can be achieved by means of a torsion pendulum (see section 2.3 for details). Finally, according to equations (1)–(3), one can obtain the torque–twist data continuously for the specimen. The sensitivity of the apparatus can be altered by exchanging the torsion wire for one of greater or lesser torsional rigidity. Generally, the torque per unit angular displacement of the torsion wire should be approximately ten times the magnitude of the highest torque applied in the fiber specimen [5].
The design is realized within the previous self-developed torsion testing instrument [1, 21]. The rotation rate provided by the stepper motor ranges from 0.05236 to 0.5236 rad s$^{-1}$. The minimum angular increment of the stepper is 0.0314 rad, but if necessary a servomotor could be substituted to provide an increased resolution or rotation rate. The optical detector for measuring the angular displacement of the torsion wire consists of a laser displacement sensor (Keyence, LK-G80) with a resolution of 0.1 µm and a cross-beam attached between the torsion wire and the specimen. The cross-beam is initially adjusted to be perpendicular to the laser beam emitted from the laser displacement sensor. The laser beam is reflected off the cross-beam and collected by a CCD within the displacement sensor, as illustrated in figure 1(b). The experiment control and data acquisition for the torsion testing is automated through a LabVIEW (National Instruments Corp.) program in a computer, which provides for control of a range of experimental parameters, such as the rotation speed, twist amplitude and cycle number (if cyclic torsion is desired). During testing, the data are displayed in real time.

Figure 4. SEM images of torsion wire and fiber specimens: (a) tungsten wire of diameter 30.53 ± 0.10 µm; (b) copper wire of diameter 49.89 ± 0.27 µm; (c) silver wire of diameter 49.57 ± 0.25 µm; and (d) T300 carbon fiber of diameter 7.04 ± 0.02 µm. The silver wires and carbon fibers used here are the same as those in [26].

Figure 5. Torsional stress–strain curves within the elastic range for copper wires of diameter 18 µm.
2.2. Calibration of the torsion wire

Calibration of the torsion wire is crucial for torsion testing. Several researchers have determined the torsional rigidity of a torsion wire by applying a known torque to the wire [3, 27]. This was achieved by using a dead weight suspended over a pulley arrangement [3, 27]. Such a calibration method has been proved to be unsatisfactory because of the friction in the pulley used to redirect the load. Here, we adopt an improved torsion pendulum based on image processing [26] to determine the shear modulus of the torsion wire accurately, as shown in figure 2(a). We chose tungsten wires ranging in diameter from 15 to 40 µm as the torsion wires. The real diameter of each tungsten wire was determined with a scanning electron microscope (SEM) (see figure 4(a), for example). A length of tungsten wire was suspended at one end, and two washers with a suitable mass were bonded to the other end. The oscillation motion of the suspended mass was recorded with an overhead video camera. An image processing method was developed to calculate the twist angle of the pendulum. The oscillation frequency was then determined by FFT, and is found to be 0.520 Hz, as shown in figure 2(c). The length of the tungsten wire is 64.52 mm. Then, the shear modulus of the tungsten wire is calculated as $G_{\text{tor}} = 121.43 \pm 3.0$ GPa, which is in agreement with the results in literature [25, 26]. Substituting the values of $G$ and other parameters into equation (4), one can obtain the torsional constant of the torsion wire.

2.3. Preparation of the specimens

The fiber specimens were prepared using the following procedures. A single strand of the fiber was isolated with the aid of an optical microscope (Keyence VHX-500FE). In order to

Table 1. Shear moduli for copper and silver wires and carbon fibers.

| Specimen        | Diameter (µm) | Diameter of torsion wire (µm) | $K$ of torsion wire $^d$ (nN m deg$^{-1}$) | Measured $G$ (GPa) | $G$ in refs. (GPa) |
|-----------------|---------------|-------------------------------|------------------------------------------|--------------------|-------------------|
| Copper wires    | 18.19 ± 0.14  | 30.53 ± 0.10                  | 6.3–7.1                                  | 54.11 ± 2.14       | 40–49 [6, 25, 26] |
| Copper wires    | 49.89 ± 0.27  | 40.58 ± 0.20                  | 32.8–58.8                                | 36.84 ± 2.42       | 40–49 [6, 25, 26] |
| Silver wires    | 49.57 ± 0.25  | 40.58 ± 0.20                  | 46.5–55.8                                | 25.48 ± 1.02       | 29.32 ± 0.32 [26] |
| Carbon fibers   | 7.02 ± 0.18   | 15.20 ± 0.10                  | 0.43–0.78                                | 19.88 ± 2.42       | 14–22 [25, 26, 29, 30] |

$^d$ Note that the different values of $K$ for torsion wires with the same diameter are due to the different lengths of torsion wire used.
avoid damage, the two ends of the fiber specimen of length about 15 cm were glued to two V-holders on a paperboard, as shown in figure 3(a). Similarly, a torsion wire of approximate length 5 cm was also bonded on another paperboard in the same way (figure 3(b)). These two paperboards were then placed together in such a way that the specimen and the torsion wire were aligned to each other, as illustrated in figure 3(c). Afterwards, the cross-beam, consisting of an aluminum foil with a thickness of 200 µm, a height of 8 mm and a length of 30 mm, was bonded to a suitable position on the torsion wire and the specimen to fix them together. With the cross-beam being a dividing line, we cut a segment of specimen to the left and a segment of torsion wire to the right (see figure 3(d)), respectively. Finally, a washer with a suitable mass was attached to the end of the specimen, and the torsion wire was glued to a thin rigid beam, as shown in figure 3(e).

We chose three types of fibers/wires for testing, i.e., copper wire (99.999% purity, Henan Youk Electronic Material Co., Ltd, China), silver wire (>99.99% purity, MK Electron (Kunshan) Co., Ltd) and carbon fiber (T300, PAN, Toray Industries, Inc.). The diameters of the specimens and the torsion wires were measured by a SEM (Quanta 3D Dual Beam system FIB-SEM), as shown in figure 4.

3. Evaluation of the technique

To test the sensitivity and reproducibility of the torsion tester, three repeated measurements were performed on 18 µm diameter polycrystalline copper wires in the elastic range, as shown in figure 5. The torsion data are displayed in the form of surface shear stress \( \tau = 2T/\pi a^2 \) versus surface shear strain \( \gamma = \kappa a \). Here, \( T \) is the torque, \( a \) the wire radius and \( \kappa \) the twist per unit length. The experimental curves exhibit a reasonable repeatability. We used the mean curve as a representative response (see figure 5), in which the error bar marked on the mean curve is ±2 MPa. The corresponding shear modulus is calculated as 54.11 ± 2.14 GPa, as listed in table 1. The fluctuations in the curves are mainly due to the misalignment between the specimen and the twisting head, as shown in figure 6. As pointed out by Walter and Kraft [22], it is hard to align the specimen perfectly into the twisting head. Therefore, the cross-beam moves around a circular track during one rotation (see figure 6(a)). Such a motion leads to an alternating signal in the angular displacement measurement of the torsion wire, as well as in the torque measurement, as illustrated in figures 6(b) and (c). One can see that the closer the cross-beam is to the fixed end of the torsion wire, the lower the influence of misalignment on the torque measurement. Therefore, we used torsion wires of length 10–25 mm throughout testing, which was much shorter than the gauge length of the specimens. In addition, the roughness of the cross-beam also influences the torque signal, which turns out to be much smaller than that caused by the misalignment. According to equations (1) and (4), one can see that the measured torque is strongly dependent on the wire diameter. That is, there is a dramatic decrease in torque with decrease in the fiber/wire diameter.

Compared with the previous study [1], there are three noteworthy features in the present technique, although both are based on a similar measurement principle. Firstly, the misalignment was a major problem in the previous design since...
both ends of the torsion wire were fixed and the specimen was connected to the torsion wire through a rigid frame. Here, the specimen is straightforwardly connected to the torsion wire via the cross-beam, which improves the coaxiality between the torsion wire and the specimen. Secondly, the cross-beam, which is symmetrical about the torsion wire and paired with a laser displacement sensor, is used as the angle detector for measuring the instantaneous rotation of the torsion wire as the specimen is twisted. Thirdly, the method for preparing the specimen has been improved. By making these improvements, the error due to the off-axis rotation has been greatly reduced. Therefore, the modified torsion tester design can measure torques as low as $10^{-9}$ N m with a resolution of up to $10^{-10}$ N m, which enables the characterization of single fibers with a diameter of less than 10 $\mu$m. For example, the torsional properties of individual carbon fibers of a diameter 7.02 $\pm$ 0.18 $\mu$m can be studied (see section 4).

4. Results and discussion

All the torsion measurements were performed in a twist-controlled mode and carried out at room temperature. In a twist-controlled mode, the test is conducted in such a way that the twisting rate given by the stepper motor during the test is maintained constant and the torque required is measured. The gauge length of the tested wires was varied between 60 and 100 mm. The rotating speed of the turntable used here was 6.28 rad min$^{-1}$. Due to the difference in the diameter and gauge length of the specimens, the surface shear strain rate of each specimen was not the same, but all were below $3 \times 10^{-5}$ s$^{-1}$.

The shear stress–strain curves for the 50 $\mu$m diameter copper wires and silver wires are shown in figure 7. The curves are scattered but still exhibit reasonable repeatability. We used the mean curve as a representative response. The error bars marked on the mean curve are not the results of calculating deviations but are only used to indicate the upper and lower boundaries of the curve scattering. Note that the large perturbations on the curves are caused by the air current. Both the copper wires and the silver wires exhibit linear elastic behavior followed by non-linear yielding behavior at small strains. The elastic shear moduli for these two types of wires are calculated to be 36.84 $\pm$ 2.42 GPa and 25.48 $\pm$ 1.02 GPa, respectively. The shear yield strengths for these two types of wires are around 40 MPa and 60 MPa, respectively. These values are consistent with those in [25, 26, 28].

To study the capability as well as the resolution of the tester, the torsion tests were also conducted on 7 $\mu$m-diameter carbon fibers by following the same procedure, as shown in figure 8. Measurements were repeated seven times under the same testing condition. One can see that the response of carbon fiber is fully elastic even for a large shear strain at surface as the carbon fiber is brittle in nature, as confirmed by the torsion experiments performed by Sawada and Shindo [29]. The shear modulus of the T300 carbon fibers is measured to be 19.88 $\pm$ 2.42 GPa, which is consistent with the published values [25, 26, 29, 30]. We have also measured the Young’s modulus for the same carbon fibers using a specially designed fiber tensile module [31], which gives $E_{\text{carbon}} = 235.4 \pm 12.4$ GPa. Therefore, the resulting Poisson’s ratio would be $\nu = E/2G - 1 = 4.9$, indicating that the carbon fiber is anisotropic since the isotropic materials must have a Poisson’s ratio of $-1 < \nu < 0.5$. This conclusion is in agreement with that obtained by Tsai and Daniel [32] and Behlow et al [6].

5. Conclusions

A torsion-balance tester has been developed to measure the torsional properties of single micron-diameter fibers/wires. The design of the tester allows one to perform torque–twist measurement on single fibers/wires under a condition of constant tension. As examples, the torsion tester was applied to investigate the torsional properties of thin metallic wires and carbon fibers with diameters from several microns to tens of microns. The experimental results demonstrate that the technique can be used to measure the torque down to the nN m regime with a precision of about 8%. Unavoidably, the misalignment between the torsion wire and the twisting head gives some noise in the measured torque–twist curves, which is analyzed in detail. Many torsional parameters, such as the shear modulus and shear yield strength, can be obtained with reasonable accuracy. For further progress, the proposed technique is expected to be extended to characterize other torsional characteristics, including creep, hysteresis, fatigue, torque relaxation, etc.

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