Materials Research Express

PAPER

Flexural characterization of carbon nanotube (CNT) yarn neural electrodes

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Keywords: neural electrode, carbon nanotube yarn, flexural limit, flexural rigidity, three-point bending test

Abstract

Chronic recording and stimulation in central and peripheral nerves require both high mechanical and biological compatibility. The biocompatible CNT yarn has shown promising mechanical advantage as a novel neural electrode. In this study, we quantitatively characterized the mechanical properties of CNT yarn electrodes from the perspective of the flexural limit and the flexural rigidity. The traditional test configuration was remedied for ultrafine samples with efficient elimination of the bias caused by the manual operation. As compared to the traditional Platinum-Iridium (Pt-Ir) wire electrode, CNT yarns with the similar diameter had a much more promising flexural-limit property, and thus can overcome the severe electrode failure in chronic neural recording or stimulation.

1. Introduction

Numerous kinds of implantable electrodes have shown profound performance in recording and stimulation of neuronal populations along the pathway in the central or peripheral nervous system. The neural electrodes utilized for diagnosing and treatment of the neurodegenerative diseases (such as the Parkinson, blindness, deafness, etc) are typically made of Pt-Ir [1], silicon [2], and polyimide materials [3–5]. However, following a promising start over decades ago, there are still some obstacles for widespread applications of fine neural electrodes. It is mainly due to the deterioration and even loss of neural signals from these implants. A key reason here is the poor understanding of failure modes of current implanted microelectrodes.

It was reported that the mechanical mismatch between the implanted electrodes and neural tissues would significantly shorten the implantation lifetime [6], and thus greatly deteriorate the widespread and chronic clinical application. The displacement caused by this mismatch would lead to natural inflammation process and advance the fibrotic encapsulation that can finally make the electrode fail [7–16].

As compared with currently-used metal wires or polymer-based electrodes, carbon nanotube (CNT) materials have attracted great attention due to their superior mechanical, electrical and electrochemical properties [17]. Among them, the CNT yarn composed of highly aligned CNTs can retain the properties of single tubes [18–23] and has shown great advantages in developing novel implantable electrodes. The CNT yarns can be extremely long, and possess superior electrochemical performance owing to the huge surface area [24]. Many works have been done to realize a stable CNT-based coating to traditional electrode materials to enhance the performance [25, 26]. The CNT-based neural interface is considered to be an important progress [27]. In addition, McCallum et al have demonstrated the feasibility of the intrafascicular chronic recording within the peripheral nerves by using 10 μm-diameter CNT yarn electrodes in rats [28]. To further develop CNT-yarn-based implantable electrodes, delicate and systematic identification of the flexural properties is still of great
necessity toward the fundamental guide for chronic implantation. Our hypothesis is that CNT yarn electrodes possess satisfied flexural characteristics especially in comparison with traditional materials such as fine Pt-Ir wires. Some other work shares similar insight [29].

In this work, we quantitatively characterize the flexural properties of CNT yarns by making a mechanical assessment on both the flexural limit and flexural rigidity through novel testing approaches. The flexural properties are compared between CNT yarns and fine Pt-Ir wires. This work would guide the optimal design of micro-implants for better mechanical matching with neural tissues.

2. Materials and methods

2.1. Materials

The fabrication process illustrated in figure 1 was applied to fabricate the CNT yarns by using on a forest-based spinning method. In this method, a vertically aligned CNT array was first grown by a sustained chemical vapor deposition [20], and then the CNT sheet was drawn and twisted with the infiltration added at the same time, as reported in [30]. Thus, the densely-aligned spiral CNT yarns were achieved. The yarn was clearly revealed in the scanning electron microscopy (SEM) photograph shown in figure 1. The fiber diameter was controlled to be 15 and 30 μm by adjusting the width of the CNT sheet drawn-out from the CNT forest [30], and the twist angle was the optimal one of ≈20°.

The mechanical properties including the flexural strength and the flexural rigidity of CNT yarns were evaluated and compared with those of the Pt-Ir wires. The available CNT yarns were 30 ± 5 μm (mean ± standard deviation) in diameter, and were adopted for both flexural-property testing. An additional 15 μm-diameter yarn was also used for flexural rigidity testing. For comparisons, 30 μm-diameter Pt-Ir wires were selected, which was equivalent to the commonly-used wires as neural implants [23].

2.2. Test of the flexural limit

The displacement between tissue and neural electrodes after implantation would lead to the bending of the electrodes in vivo. Some electrodes might be quite weak against bending, and fail easily inside organisms. Moreover, McCallum et al performed the electrode implantation by winding up an ultrafine CNT yarn around a sharp needle to guide the implantation of CNT yarn electrodes while some other researchers shared similar demands [28, 31]. This winding-up process is also a bending process in mechanics.

In materials science and mechanics, the flexural limit is closely related to the flexural strength which represents the highest stress experienced inside the material at its moment of yield. The test is traditionally performed on a beam made of the tested material using three-point bending method [32]. To accomplish the flexural strength of ultrafine CNT yarns, the micron-scale failure of the fiber must be considerably monitored. However, it is practically hard to observe the micro fracture, and then the flexural strength is difficult to be determined using this traditional test method. Furthermore, great changes of the Young’s modulus would be highly possible to accompany the irreversible damage of the fiber. And thus a more practical and simple test method is put forward to evaluate the flexural limit by correlating the Young’s modulus with the curvature radius.

2.2.1. Experimental setup

To accurately achieve the flexural strength of CNT yarns and Pt-Ir wires, two groups of specimens were prepared, namely the wound and non-wound groups. As shown in figure 2, for the wound group, winding procedures were exerted on the yarns (or wires) with different-diameter plastic rods and fine tungsten needles as the cores for winding. The diameters of these cores lie within the range of 100 μm to 1 mm measured by
microscope photographing. Each yarn was densely and uniformly wrapped around a certain core by four turns in a gentle manner to avoid being stretched, and then was untied softly after about 1 min. For each specimen, the winding and untying procedures were repeated among different cores with varied sizes. Finally, the adjustment of the radius of curvature was determined by the sum of radii including the winding core and the tested specimen.

Following the preparation of the two groups of specimens, conventional tensile tests were performed by using a universal mechanical testing apparatus, at a stretching rate of 1 mm min$^{-1}$ and a gauge length of 10 mm. The flexural limit is defined as a critical radius of curvature where 80% of the pristine Young’s modulus can be remained after the winding.

2.2.2. Quantification: minimum curvature radius.

Bending a fiber to a limit state leads to a mechanical failure. Although this failure may be inconspicuous in outside appearance, it can severely shorten the electrode lifetime in application. This failure is usually related to two factors including the diameter and the maximum strain of the fiber. Theoretically, the failure in bending is similar to that in stretching, and we provide a simple yet effective physical model to explain the bending failure, as well as a formula to calculate the value of the flexural limit.

Consider a tiny part of the fiber with a diameter of $d$ is bent to an average radius of curvature of $\rho$. If the fiber is not stretched, the middle remains the original length. Meanwhile, the outer layer closer to the center of curvature is compressed, and the outer layer farther is stretched with an identical radius of curvature. Thus, the following expression can be reached:

$$\frac{(1 - x)l_0}{\rho} = \frac{(1 + x)l_0}{\rho + d}$$

where $x$ denotes the strain in mechanics, $\rho$ the curvature radius, $d$ the diameter and $l_0$ the length of center layer, which is also the original length of the part of the fiber. To keep the material from the bending failure, $x$ should be less than the maximum strain $x_{\text{max}}$. Then we obtain the inequation as below:

$$\rho > \frac{1 - x_{\text{max}}}{2x_{\text{max}}} \cdot d$$

The in equation (2) indicates that the curvature radius should be larger than a boundary value to avoid the fracture of the outmost layer of the material in bending. Under critical conditions, $\rho_{\text{min}} = \frac{1 - x_{\text{max}}}{2x_{\text{max}}} \cdot d$. The $\rho_{\text{min}}$ is defined as the flexural limit. Clearly it is a function of both the maximum strain and the diameter. Besides, it is specifically noted that a critical condition, which is the fracture of the outmost layer, is used here.
2.3. Test of the flexural rigidity

The flexural rigidity under bending is the other key flexural property. There are already some traditional ASTM (American Society for Testing and Materials) methods concerning the evaluation of the bending properties of plastics. However, the ASTM methods designed for beams do not fit with the ultrafine fibers including the CNT yarns due to limited test precision. Kha et al [7] and Naito et al [33] indicated that the flexural rigidity was calculated by mathematical transformation from the Young’s modulus. However, these testing methods are just validated with the assumption that the tested material can be modelled as a traditional elastomer, and the moment of force required can be completely determined by its elastic modulus and the curvature. In this case, the flexural rigidity is a multiple of Young’s modulus and the second moment of transection area. Of great difference, for nanomaterial assemblies, the second moment of transection area cannot be easily calculated due to the non-uniform and unknown mass distribution in the transverse direction. Therefore, an optimized three-point bending test was put forward to realize a more accurate quantization of the flexural rigidity. First, bending behaviors of ultrafine fibers like CNT yarns were obtained under different tensile forces; and then the extrapolation is applied to eliminate the loading effect, and finally the real flexural rigidity is presented.

2.3.1. Experimental setup

The flexural rigidity of ultrafine CNT yarns and Pt-Ir wires were tested by using a modified three-point bending method based on a universal test machine (T150, Keysight Technologies, Inc., Santa Rosa, USA). This machine is equipped with a sensitive force sensor with a fine resolution of 50 nN. The three-point bending test was illustrated in figure 3, where a fiber was placed on two supports spanned by a distance L of 28 mm, and stretched by a pair of weights m. By applying a bending load F at the middle, a bending angle \( \theta \) and a resistance force f are generated. As depicted, a yarn or wire was horizontally positioned on the parallel supporting bars with the third bar centered above, which was directly connected to the sensitive force sensor. The yarn or wire was loaded symmetrically using a pairs of copper clips whose mass m was confirmed by an electronic balance. During the period of mechanical testing, the two side bars were lifted up, and therefore the middle bar would press down the specimen, leading to the bending process. The resolution of the displacement \( X \) was less than 0.1 \( \mu \)m. The bending force F at the deflection point shown in figure 3(b) was recorded for each tested yarn or wire.

2.3.2. Quantification principles

Figure 3(b) shows the schematic of the three-point bending, where a yarn is stretched to be straight at a pair of weights m, which induce a tension force T = mg inside the yarn. The two circles on the left and right sides are the fixed bars to support the fiber, and the middle movable one is connected to the force sensor. Under a quasi-
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As the result of the determination of exural rigidities, the relationships of the loads and the deflection were plotted in figure 3(c) and (d) for CNT yarns and Pt-Ir wires, respectively. As the load increases, the slope in the

Figure 4. Experimental results of curvature effects on the Young’s modulus: (a) Typical stress-strain curves of the Pt-Ir wires after being wound with different curvature radius. For each radius, at least 3 tensile tests have been performed. (b) Effect of the curvature radius on the Young’s modulus for Pt-Ir wires. The core radius slides from 115 μm to 502 μm. The red line represents the non-wound electrode. The critical curvature radius from the linear fitting curve is 588 μm according to a failure threshold where 80% of the pristine modulus can be retained; (c) The winding process has negligible influence on the Young’s modulus for the CNT yarns. The red dashed lines represent the measured Young’s moduli before winding.

static deflection process, there is a balance between the forces F, f and T, where F is the applied bending load and f is the counterforce generated by the yarn, \( F = f + 2T\sin \theta \).

Determination of the flexural rigidity is closely related to \( f(\theta) \). We assume that \( f(\theta) = b + k\theta \). Further a very simple force balance equation is reached:

\[
F(\theta) = f(\theta) + 2mg\theta
\]

Therefore, by decreasing the \( n \) value, we can obtain a quantitative description of the ability to against the bending, \( f_{lm} \). Here we defined the flexural rigidity as the first order coefficient \( k \) of \( f(\theta) \). The \( k \) is a meaningful factor, which is related to the bending property of a specific fiber, therefore we defined this factor as the flexural rigidity. In this test, we changed \( T \) by altering loads (copper clips with different weights). Then the \( F - \theta \) relation vs. loads can reveal \( k \), which can be obtained by extrapolating and get the intercept. When \( m \) is in a range to induce elastic and slight elongation, the influence of load variations on \( f \) can be negligible. By considering the fiber diameter or film thickness/width, the softness is further described by dividing \( f \) by the cross-sectional area or the linear mass density.

3. Results

3.1. The flexural limit

Since the flexural limit is reflected by the change of Young’s modulus, it is necessary to find out the dependence of the modulus on the curvature radius, for both the Pt-Ir wires and CNT yarns. With increasing the curvature radius, there was a clear decrease in Young’s modulus for the Pt-Ir wires, as shown in the stress-strain curves, see figure 4(a). A linear fitting was adopted to analyze the decrease in Young’s modulus (figure 4(b)). For example, at a curvature radius of 501 μm, the modulus was very close to the pristine value of 107.2 GPa, while it decreased to be \( \approx 20 \) GPa at a small radius of 115 μm, by nearly one order of magnitude. Further, the tested maximum strain \( \epsilon_{max} \) in equation (2) is 2.5% for Pt-Ir wires; then the flexural limit of a 0–30 μm Pt-Ir wires is theoretically estimated to be 585 μm. It indicates that if the curvature radius of a Pt-Ir wire is smaller than 585 μm, there would be a high risk of the mechanical failure. This theoretical value is rather close to the experimental results. As can be seen in figure 4(b), when the Young’s modulus decreases to 80% of its tested normal value (107.2 GPa), which is seen as a threshold here, the corresponding diameter is 588 μm (using the linear fitting result in figure 4(b)).

On the contrary, figure 4(c) showed that the tensile modulus of CNT yarns changed within a very limited range, and even returned to the pristine value at a very small curvature radius of 50 μm. This means that the flexural limit could be even smaller than 50 μm for CNT yarn electrodes, which is greatly promising than that for Pt-Ir wires. In addition, the Young’s modulus of CNT yarns (~10 GPa) is much lower than that of the Pt-Ir wires with similar diameters, and held a superior mechanical biocompatibility with neural tissue with a Young’s modulus of \( 1 - 3 \times 10^{-3} \) GPa [34].

3.2. The flexural rigidity

As the result of the determination of flexural rigidities, the relationships of the loads and the deflection were plotted in figure 3(c) and (d) for CNT yarns and Pt-Ir wires, respectively. As the load increases, the slope in the
is given in equation \[ k = \frac{dF(\theta)}{dx} \] where \( F(\theta) \) is given in equation (3). Here \( x \) denotes the deflection, and \( x = \frac{1}{2} \theta L \). \( L \) is the span length. Then this rigidity slope can be expressed as \( \frac{dF}{dx} = \frac{1}{2} + \frac{k}{T} \) where \( T \) is changeable by varying the clip load. The relationships of the slope in figure 3(c) and the exerting load were plotted in figure 3(e) for both CNT yarns and Pt-Ir wires. The parameter \( k \) is the flexural rigidity which can be obtained from the intercepts listed in table 1.

Note that \( k \) depends on the specimen diameter, which means that it does not characterize a particular material. To be specific, it characterizes a single wire/fiber. To compare the flexural rigidity of two materials given \( k \), the two specimens should have close diameters. As shown in table 1, the parameter \( k \) for the CNT yarn electrode is about four times that of Pt-Ir wire, which indicated that Pt-Ir is much inclined to be bended and less rigid than CNT yarns in terms of the three-point testing method in figure 2.

### 4. Discussion

Mechanical matching between neural electrodes and biological tissues is the key for solid neural recording and stimulation to diagnose and treat some neurological disorders. A CNT yarn is composed of millions of nm-scale CNTs and possesses a continuous structure with a macroscopic dimension [35]. McCallum et al [28] denoted that chronic recording from glosopharyngeal and vagus nerves were achieved by using CNT yarns with the diameter of 10 to 20 \( \mu \text{m} \). To objectively evaluate the flexural characterization of the CNT yarn neural electrodes, the flexural limit and the flexural rigidity were objectively tested based on the optimized testing structure.

In addition to the implantation requirement of bending the Pt-Ir wires or yarns, after the implantation of neural electrodes, there would still be unavoidably some displacement between the electrodes and the biological tissue, which would lead to another bending of the electrodes such as Pt-Ir wires and CNT yarn. As a result of bending, the curvature radius of the wires or yarns would have some variations. In the flexural-limit test, the Young’s modulus of Pt-Ir wires changed linearly with the variation of the curvature radius. This means that the Young’s modulus of Pt-Ir wires would be greatly different from the normal value, which reflects its deviation of the wires from the elastic state, and then the deterioration of the wires leading to the failure of chronic implantation.

On the other hand, the Young’s modulus of the CNT yarns remained close to the normal value with the curvature radius changing. This is because the analysis of the flexural limit is based on an assumption of an ideal elastomer. And only some traditional materials can be modeled (or approximated) by elastomers, e.g. Pt-Ir alloy, silicon, and most of metals. The structure of CNT yarns, however, is significantly different from that of Pt-Ir wires. The CNT yarns are a kind of assembly of CNTs, and the force combining CNTs is Van der Waals force. There exist slides between layers during bending, and therefore the macro strain of the outermost layer of the yarn does not mean the real strain of each CNT in this layer [36]. The entanglement and crosslinks between CNTs in their assembly materials also bring them high viscoelasticity, like the characteristics of rubber [37]. Consequently, in this point of view, CNT yarns possess a promising flexural limit which helps endure strong winding or bending.

The flexural rigidity obtained in this work is not a modulus and it is dependent on the specimen diameter. It is similar to the stiffness studied in [38]. Compared to stiffness which is usually obtained through calculation, the flexural rigidity is a more straightforward metric in experiments. The reason we did not use the modulus is that the CNT yarn electrode is an assembly of CNTs, and is not uniform transversely. While, when the diameters of two specimens are similar, the proposed modulus can still be utilized to distinguish two different materials.

Although the Pt-Ir wire electrode is much inclined to be bended by comparison with the CNT yarn electrode with the same size, it could be much easier to reach its flexural limit during the bending state which happens all the time in vivo. The implanting process and the movement of tissues can easily make the deformation exceed the bending limit of the Pt-Ir alloy. It is also identified by the bending experiment with different curvature radius. Thus the implant life has been found to be commonly less than two weeks [28]. On the contrary, the

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**Table 1. Tested flexural rigidity.**

| Material     | Specimen diameter (\( \mu \text{m} \)) | Intercept (N/m) | \( k \) (mN) |
|--------------|----------------------------------------|----------------|--------------|
| 1 CNT yarns  | 15 ± 2                                 | 0.06           | 0.84         |
| 2 CNT yarns  | 30 ± 5                                 | 0.128          | 1.796        |
| 3 Pt-Ir      | 30 ± 1                                 | 0.034          | 0.476        |
superior flexural limit of CNT yarn electrodes could allow a much longer implant lifetime, close to three months as reported by McCallum et al. [28]. This could be the key to observe a stable magnitude of about 20 kΩ at 1 kHz for the electrochemical impedance within 10 weeks [28]. Clearly, the CNT yarn has shown nice performance in chronic implantation and a potentially longer lifetime toward the next-generation implant electrode.

The purpose of this present work was to present a novel testing method to quantify the flexural properties of fine CNT yarns, and made comparisons with those of Pt-Ir wires. These tests were carried out outside the body. Although no direct testing of flexibility were conducted in vivo, our previous work [28] showed that Pt-Ir wires no longer functioned inside the body, which could be due to its poor flexural limit. In our future work, we will further evaluate the biomechanics about peripheral nerves with CNT yarn electrodes implanted for chronic implantation.

5. Conclusion

The mechanical characteristics of neural electrodes play a significant role for chronic implantation. A novel CNT yarn electrode was presented in our former work, and CNT yarn electrodes preliminarily showed promising chronic implantation in comparison with Pt-Ir wires. In this work, flexural properties of carbon nanotube yarn neural electrodes were characterized in detail, and were compared with those of the Pt-Ir wires. The CNT yarn electrodes showed more comparable Young’s modulus with the neural tissue yet higher flexural rigidity than the Pt-Ir wires. Besides, the CNT yarn electrodes embody great flexural limit, which is the key for chronic implantation, and the chronic lifetime could be achieved. In the future work, we aim to fabricate the CNT yarn with extremely small Young’s modulus which maintains a stable value with the changing curvature radius.

Acknowledgements

This research is supported by the National Natural Science Foundation of China (81671801), the Innovation Studio Fund from School of Biomedical Engineering at Shanghai jiao Tong University, and the Medical-Engineering Cross Project of Shanghai jiao Tong University (YG2017MS53).

Conflict of interest statement

The authors declared that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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