Tunable force sensor based on carbon nanotube fiber for fine mechanical and acoustic technologies

Maria A Zhilyaeva1, Oyedamola A Asiyanbola1, Maksim V Lomakin2, Dima M Mironov3, Boris S Voloskov4, Bjørn Mikladal5, Dzmitry O Tsetserukou3, Fedor S Fedorov1, Anna I Vershinina2, Sergey D Shandakov7 and Albert G Nasibulin1,6

1 Laboratory of Nanomaterials, Center for Photonic Science and Engineering, Skolkovo Institute of Science and Technology, Nobel St., 3, Moscow 121205, Russia
2 Laboratory of Carbon Nanomaterials, Kemerovo State University, Krasnaya str. 6, Kemerovo 650000, Russia
3 Robotics Research Center, Skolkovo Institute of Science and Technology, Nobel St., 3, Moscow 121205, Russia
4 Center for Design, Manufacturing and Materials, Skolkovo Institute of Science and Technology, 121205, Bolshoy Boulevard 30, bl. 1, Moscow, Russia
5 Canatu ltd, Tiilenlyöjänkuja 9 A, FI-01720 Vantaa, Finland
6 Department of Applied Physics, Aalto University, 15100, FI-00076 Aalto, Espoo, Finland

E-mail: a.nasibulin@skoltech.ru

Received 30 April 2022, revised 15 August 2022
Accepted for publication 18 August 2022
Published 8 September 2022

Abstract

Design of new smart prosthetics or robotic grippers gives a major impetus to low-cost manufacturing and rapid prototyping of force sensing devices. In this paper, we examine piezoresistive force sensors based on carbon nanotube fibers fabricated by a novel wet pulling technique. The developed sensor is characterized by an adjustable force range coupled with high sensitivity to enable the detection of a wide range of forces and displacements limited by the experimental setup only. We have demonstrated the applicability of the developed unit in tactile sensing, displacement sensing, and nanophone vibration monitoring system and evaluated its force sensing characteristics, i.e. displacement/force input and resistance/mechanical response. In the experiments it measures 0–115 N force range within 2.5 mm displacement. Moreover, the sensor demonstrates good linearity, low hysteresis, and stability when tested over 10 000 cycles. The developed sensor suits multiple applications in the field of soft and transparent sensors, nanophones, actuators, and other robotics devices for both regular and extreme environments, e.g. deep underwater and radioactive environment.

Supplementary material for this article is available online

Keywords: single-walled carbon nanotubes, fiber, tunable sensor, force sensor, transparent sensor, nanophone

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

1. Introduction

Progress in new technologies and application areas related to smart prosthetics, remote control, and robot grippers stimulate the design of new force sensing devices [1–4]. The target characteristics of these devices are adjustable and wide...
sensing ranges to be important for applications in lifting variable loads, the rehabilitation of patients, vital signs monitoring, body movements monitoring and many others [1, 5–7]. Recent studies in this field are aimed at miniaturization of the sensing devices and utilization of stretchable substrates, including design of sensors with architectures that would even ensure compliance matching to human tissue [8, 9].

There are several approaches to realize force sensor, e.g. using piezoresistive materials or mechanical architectures. Most of conventional force sensors, like strain gauges, employ piezoresistive materials, i.e., composites, dual physically cross-linked hydrogels, and others [7, 10–14]. Piezoresistive force sensors based on carbon nanomaterials enable the achievement of a wide force operation range offering stable, uniform and highly reliable sensor response. However, because the bearing capacity of a force sensor depends primarily on the force sensor modulus [7, 13, 14] use of such new or existing materials does not allow to achieve significant changes in sensor performance [7, 15, 16].

Alternatively, the force sensing devices with high modulus are made using mechanical architectures including diaphragms, springs, or cantilevers. Still, such devices do not meet flexibility criteria needed for delicate applications in birobotics for tissue identification [11, 17, 18].

Some new approaches to design force sensor for soft robotics systems employ wavy circuits and soft microfluidics with conductive liquids [7, 9, 19, 20]. Microfluidics systems are limited to conductive liquids such as electrolytic solutions or liquid-phase metal alloys, e.g., a gallium-indium alloy [9]. Fabrication of either wavy circuitry or microfluidic channels still requires lithography or other delicate technologies.

Overall, the fabrication process of the force sensors is still fraught with laborious material processing that demands rather costly equipment [4] Moreover, conventional manufacturing methods yield brittle units which can typically accommodate a strain of less than 5% for optimum performance [7, 21].

One of the most promising materials, single-walled carbon nanotube (CNT) fibers, possess all necessary properties to create superb sensors: good electrical conductivity, high mechanical strength and flexibility, supreme chemical stability, tiny dimensions, and facile fabrication pathway. CNT fibers have a higher range of conductive and electrical sensitivity for strain and force-sensitive devices because of their higher self-sensing capabilities [22, 23]. A recently developed novel CNT fiber manufacturing technique, called wet pulling (WP) [24], enables the simplified production of a tactile sensor with an adjustable force range. Still, the sensor architecture requires components of particular flexural rigidity to adjust its modulus [1]. Usually it is achieved by utilizing springs or plates of different stiffness, layer jamming or even fluids dome [7, 25, 26].

In this paper, we propose an adjustable piezoresistive force sensor based on a WP single-walled carbon nanotube (SWCNT) fibers with flexural rigidity attained by mounting the sensor with stiffener plates of different thickness. Thus, one may vary the elastic properties of the sensor assembly and therefore provide an adjustable force sensing range up to 104 N with high cyclic stability (>10 000 cycles) during dynamic loading conditions, good linearity, low hysteresis characteristics. Finally, the sensors have been applied for vibration monitoring system or as a sensitive part of special sound recording system, which we denote further as a nanophone.

2. Materials and methods

2.1. Sensing materials

To prepare sensors we employed a fiber made of SWCNT films produced by an aerosol CVD method [27–29]. The film is composed of a randomly oriented high-quality tubes with a diameter of about 2 nm. Usually, tubes are collected in bundles whose length varies from 20 to 40 μm. The SWCNT film is characterized by low sheet resistance of about 84 Ω [21, 29, 30]. In all our experiments, the fibers were prepared from a 5 mm width and 10 mm gauge length strip of a SWCNT film with a thickness of 93 nm. The films were soaked in ethanol (ethanol 95%, Bryntsalov-A company) and, under pulling action, rolled into fibers as illustrated in figure 1(a) and described in details elsewhere [24].

2.2. Force sensor fabrication

The force sensor consists of three major elements: the base, the sensitive part, and the stiffener (figures 1(d) and (e)). We designed two similar assemblies: magnet assembly (figure 1(d)) and screw assembly (figure 1(e)). The magnet assembly is designed with two pairs of magnets for both fixation and flexibility in assembling and disassembling the sensor. These magnets enable clamping to the base, which also has an embedded magnet at corresponding locations. This architecture is preferable in the need of quick and numerous changes of the stiffeners. Still, the fixation of the top of the sensor has a significant effect on the buckling behavior during the 3-point bending test. The screw assembly enables a solid fastening of all sensor elements and is useful when the force range is defined and without significant sudden variation, since changing the stiffener gets time-consuming.

As shown in figure 1(b), the fiber was placed on a polydimethyilsiloxane (PDMS) (SYLGARD®, silicone elastomer base 184 and curing agent 184) substrate before ends of the fibers were attached to thin copper wires using CircuitWorks® conductive epoxy, Product #CW2400, which has the resistivity <10−5 Ohm-m. Then, it was covered with PDMS to encapsulate the fiber; it forms the sensitive part (Supplementary data A). The PDMS encapsulation allows for stress to be evenly distributed along the fiber’s length and mechanically protects the fiber.

The stiffeners were made from thermoplastic polyurethane (TPU), polylactide (PLA), and Flex (very flexible polymer) (REC-3D company, Russia) plastics ( Supplementary data B).

The base was cast out of the PDMS, using 3D printed molds (figure 1(c)) (more details in Supplementary data C). Light feedback LED with wires was encapsulated in the base during the manufacturing process (figure 1(f)).
The void in the base gives a space when the bending is induced under force applied.

2.3. Experimental setup

An impinger, 1 cm in diameter, was used to simulate a finger tap in all mechanical experiments: the three-point bending, fatigue, and step load tests. An electromechanical testing system Instron® ElectroPlus E3000 was used for displacement-controlled experiments. During all tests, there was a maximum deflection of 2.5 mm, applied loads with the 50 kN load cell and 3 mm min⁻¹ crosshead speed.

We measured the resistance and corresponding resistance changes of the fiber in two-electrode configuration, using Digital Multimeter Keithley 2000 (Tektronix company©). The relative change of the resistance of fiber sensing element (sensitivity) was calculated as \( \frac{\Delta R}{R_0} = \frac{R - R_0}{R_0} \), where \( R_0 \) and \( R \) are resistances without and under applied load, respectively.

Figure 1. Design and fabrication of the sensor. (a) Schematic illustration of preparation of fiber using the wet pulling technique. (b) Fiber encapsulation: top layer with the sensitive fiber encapsulated in PDMS. (c) Molding stages for the base of the sensor. Sensor assembly with two different architectures: (d) demoldable with magnets and (e) with screws. (f) Semitransparent sensor with light feedback in operation.

Figure 2. Acoustic experiment scheme: (a) arrangement of the encapsulated fiber and the smartphone’s speaker. (b) Schematic setup of the experiment. (c) Image of the sensor placed on the smartphone speaker.
The relative changes of the resistance of WP SWCNT fibers under cyclic load to elongation of 2 and 10% are described in Supplementary data F.

2.4. Acoustic test

Sound sensing capabilities of the SWCNT fibers were demonstrated by recording a fragment of the Georges Bizet — ‘Les Toreadors’ from Carmen Suite No. 1 (more details in Supplementary data G, H, J). The experimental scheme is depicted in figure 2. The sensor covers the smartphone speaker and PDMS touches the phone case. The input vibrational notes imposed an air pressure difference on the PDMS layer to serve as a membrane, which encapsulates the sensing fiber element. The membrane deformations, in turn, cause a change in the resistance of the encapsulated sensor-fiber measured during the experiments. The electrical circuit used in this experiment is described in Supplementary data H.

All recordings are monophonic. From the obtained data, we generated spectrograms using a windowed Fourier transform.
To construct spectrograms and waveforms, a Python™ script (Librosa and Matplotlib libraries) was utilized.

3. Results

3.1. Mechanical properties of force sensors

The application of mechanical stress to the sensor is accompanied by a displacement of individual CNTs with respect to each other. Accordingly, it is expressed in changes in the fiber resistance that remain the ones of the ideal piezoresistive sensing material. The length of the CNT fiber increases accordingly to the Poisson effect just as shearing and compression stresses are induced in the fiber. Hence, a total resistance of the sensor increases due to CNT displacements along the fiber axis during the bending test.

The adjustable flexural rigidity of the sensor is attained by mounting the sensor with stiffener plates of different thicknesses, thereby varying the elastic properties of the sensor assembly. The stiffener plates considered in our work offer variable stiffness (or sensor modulus) as a promising solution to outweigh the contradiction and tradeoffs between maintaining compliance with materials’ physical properties and maximizing the force sensing capacity. To determine the operating characteristics of the finger and force sensor under impinging pressure, it is necessary to choose the appropriate stiffener plates. Figure 3(a) shows the deflection (mm) starting with the sensor without a stiffener, followed by the sensor with the selected five stiffeners. Changing the stiffeners adapts the force range to the required one; figure 3(b) linear fit ($R^2 = 0.99959$) of the stiffness versus the force. The load-deflection ranges and characteristic curve for different stiffener materials are depicted in figure 3(c), the stiffener increases the modulus of the sensor; ‘no stiffener’ corresponds to the inherent stiffness of the sensor. We utilized TPU (infill density of 80%) & PLA (90% and 80%) 3D printed stiffener plates with different thicknesses. A softer sensor easily deforms under the load and achieves the maximum deflection rapidly; the stiffer sensor bends a little under the load, because of the greater stiffness coefficient. Thus, taking into account the maximum sensor deflection of the exact application, one can choose the optimum stiffener, with the appropriate force range and the greater resolution. Figure 3(d) gives relative deflection variation of the sensor load response evaluated based on different stiffeners at the same loading force of 10 N. This thereby validates that increasing modulus of the sensor allows for adjustable force sensing.

3.2. Electromechanical properties of the force sensor

Following the evaluation of mechanical properties, we tested corresponding electromechanical characteristics. The relative change in resistance of sensor regarding the repeated deflection of 2.5 mm for different stiffeners as a function of the applied force is presented in figure 4. Individual stiffeners have different slopes and corresponding detection limits.
3.3. Fatigue and step load tests. Sensor training

Fatigue test shows poor stability without training the sensors and drifts of similar delta values as the resistance changes due to a load input (figure 5(a)). However, the training of the sensor helps to mitigate such unstable performance. The training procedure consists in applying the load to stretch the sensor for 10%, then unload, and finally applying the planned force for several times. Figure 5(b) shows the output resistance plot during the fiber training used to stabilize the sensor. After the 6th cycle the fiber possesses the stable response. Subsequent fatigue test results are shown in figure 5(c). The results of relative change of the resistance of WP SWCNT fibers under cyclic load to elongation of 2% and 10% are presented on figures F1 and F2 (see Supplementary data F). The curves presented as a dependence of the relative resistance on elongation also demonstrate the stabilization of the fiber response after a small number of cycles of preloading both at low (2%) and at ‘working’ (10%) stretching, comparable to the stretching of the fiber in the sensor. A more stable response with minimal drift shows that the training is effective to achieve excellent sensor performance. During our experiments, the same sensors were used for fatigue tests for several times and possess good stability for more than 10 000 cycles in total. The major effect of training stems from irreversible small deformations in the fiber to disappear. In this way, the true elastic deformation is achieved within the trained displacement.

Figures 5(d)–(f) show the sensor response to the step load. Figure 5(d) shows the recorded displacement input. Zero deflection is the position at which the impinger touches the sensor. Figure 5(e) presents the step load directed to the sensor. The correspondence of these two plots shows the stability and repeatability of the sensor structure and proves the linear behavior. The sensor resistance output is depicted in figure 5(f). It shows mild inertia peaks originated from the testing machine (circles in figures 5(d)–(f)).

3.4. Acoustic experiment

We have evaluated the performance of the encapsulated WP SWCNT fiber in an acoustic experiment; configuration is shown in figure 2. The recorded resistance changes closely replicate the frequency domain and corresponding notes amplitude when compared to the commercially available microphone after minimal filtering. We called the device a nanophone, because its core sensing elements are CNTs. The audio recordings obtained for music from the sensors and microphone (with and without filtering) are given in Supplementary data J. Supplementary data G and H describe the procedures of filtering, the nanophone manufacturing details, and divider impact.

Figure 6 shows the ‘Les Toreadors’ waveform and a spectrograph of the electrical signal (resistance changes) from the sensor (nanophone) and the standard microphone. The background noise obtained is due to the diaphragmatic vibration sensing architecture employed (see figure 6(a)). After the filtering procedure, the signal resembles the one of
the microphone, which can be clearly seen in figures 6(b) and (c). Such proof-of-the-concept test shows that the sensor can be used for acoustic sensing; proper filtering circuit would demonstrate a more robust opportunity for the nanophone implementation. The nanophone recordings quality is fine enough for average machine music recognition systems (we used Shazam player, Apple Inc.) to easily define the composition (see https://youtu.be/QUPqaKBd5Y).

We also tested the sensor for vibration monitoring (see https://youtu.be/d_QndEFklfk). It showed a perfect response to table tap and incorporated light feedback corresponded to the amplitude of the vibration impulse.

It should be noted that the presented sensor can be utilized to measure the force load in a wide range and significantly shorter response time, when compared to other sensors. Table 1 highlights the most advanced results, which overview can be found in [31].

4. Conclusions

This work demonstrates a great potential of wet pulled SWCNT fibers in the design of an adjustable force sensor. While annexing piezo-resistive properties of the wet pulled SWCNT fibers under strain, we can measure the force applied and the deflection of the structure by measuring fibers resistance. The force range can be tuned by changing the modulus with the stiffener plates. The selection of different stiffener materials (thin plate stiffeners), load limit ranges can be increased from 1.24 to 104 N. We benchmarked our force sensor with a safe deflection limit of 2.5 mm to allow for reliability and longer service life (>10,000 cycles). Fiber PDMS encapsulation opens the opportunity for broad applications, e.g. underwater, bio-compatible, light feedback implementation, and increases the size of the sensor for comfortable usage.

In addition, we demonstrate the quick response of the sensor, by recording the music and testing it as a vibration monitor. Good merits, while simple production, give the sensor a great potential for the rapid prototyping of sensitive flexible fiber-based devices for soft robotics to be employed as touch, displacement, and force sensors.

Acknowledgments

This work was supported by the Council on grants of the Russian Federation (project no. FZSR-2020-0007 in the framework of the state assignment no. 075-03-2020-097/1). Russian Science Foundation (Project Number 22-13-00436) is acknowledged for the synthesis of SWCNTs.

Data availability statement

All data that support the findings of this study are included within the article and supplementary file.

ORCID iDs

Maria A Zhilyaeva @ https://orcid.org/0000-0001-5511-0221
Fedor S Fedorov @ https://orcid.org/0000-0002-2283-0086
Albert G Nasibulin @ https://orcid.org/0000-0002-1684-3948

References

[1] Cheng M, Zhu G, Zhang F, Lai Tang W, Jianping S, Quan Yang J and Ya Zha L 2020 A review of flexible force sensors for human health monitoring J. Adv. Res. 26 53–68
[2] Templeman J O, Sheli B B and Sun T 2020 Multi-axis force sensors: a state-of-the-art review Sensors Actuators A 304 111772
[3] Zhang B, Xie Y, Zhou J, Wang K and Zhang Z 2020 State-of-the-art robotic grippers, grasping and control strategies, as well as their applications in agricultural robots: a review Comput. Electron. Agric. 177 105694
[4] Ilami M, Bagheri H, Ahmed R, Skowronek E O and Marvi H 2020 Materials, actuators, and sensors for soft bioinspired robots Adv. Mater. 33 2003139
[5] Sorrentino I et al 2020 A novel sensorised insole for sensing feet pressure distributions Sensors 20 747
[6] Choi W H, Kim S, Lee D and Shin D 2019 Soft, Multi-DoF, variable stiffness mechanism using layer jamming for wearable robots IEEE Robot. Autom. Lett. 4 2539–46
[7] Cao P-J et al 2020 A stretchable capacitive strain sensor having adjustable elastic modulus capability for wide-range force detection Adv. Eng. Mater. 22 1901239
[8] Iida F and Laschi C 2011 Soft robotics: challenges and perspectives Proc. Comput. Sci. 99–102
[9] Majidi C 2014 Soft robotics: a perspective—current trends and prospects for the future Soft Robot. 1 5–11
[10] Chi C, Sun X, Xue N, Li T and Liu C 2018 Recent progress in technologies for tactile sensors Sensors 18 948
[11] Xiao Z G and Menon C 2019 A review of force myography research and development Sensors 19 4557

Table 1. The performance results of force sensors demonstrating the most advanced results in the field.

| System                     | Force range | Response time, ms | References |
|----------------------------|-------------|-------------------|------------|
| CNTs/PDMS                 | 6 kPa (96 N)| 6.8               | [31]       |
| Patterned CNTs on a 3D polymer substrate | 5 N | <100 | [32] |
| SWCNTs/PDMS               | 100 N       | 0.1\*             | Our work   |

* Based on acoustic results.
Nanotechnology 33 (2022) 485501

[12] Nie B, Yao T, Zhang Y, Liu J and Chen X 2018 A droplet-based passive force sensor for remote tactile sensing applications Appl. Phys. Lett. 112 031904
[13] Pan S, Liu Z, Wang M, Jiang Y, Luo Y, Wan C, Qi D, Wang C, Ge X and Chen X 2019 Mechanocombinatorially screening sensitivity of stretchable strain sensors Adv. Mater. 31 1903130
[14] Duan J, Liang X, Guo J, Zhu K and Zhang L 2016 Ultra-stretchable and force-sensitive hydrogels reinforced with chitosan microspheres embedded in polymer networks Adv. Mater. 28 8037–44
[15] Zhu Y, Li J, Cai H, Wu Y, Ding H, Pan N and Wang X 2018 Highly sensitive and skin-like pressure sensor based on asymmetric double-layered structures of reduced graphite oxide Sensors Actuators B 255 1262–7
[16] Souri H and Bhattacharyya D 2018 Wearable strain sensors based on electrically conductive natural fiber yarns Mater. Des. 154 217–27
[17] Cutkosky M R, Howe R D and Provancher W R 2008 Force and tactile sensors Springer Handb. Robot. (Berlin, Heidelberg: Springer) pp 455–76
[18] Li J, Liu H, Althoefer K and Seneviratne L D 2012 A stiffness diagnosis probe based on force and vision sensing for soft tissue Proc. Annu. Int. Conf. IEEE Eng. Med. Biol. Soc. EMBS pp 944–7
[19] Rogers J A, Someya T and Huang Y 2010 Materials and mechanics for stretchable electronics Science 327 1603–7
[20] Cheng S and Wu Z 2012 Microfluidic electronics Lab Chip 12 2782–91
[21] Gilshteyn E P, Romanov S A, Kopylova D S, Savostyanov G V, Anisimov A S, Glukhova O E and Nasibulin A G 2019 Mechanically tunable single-walled carbon nanotube films as a universal material for transparent and stretchable electronics ACS Appl. Mater. Interfaces 11 27327–34
[22] Xi X and Chung D D L 2019 Capacitance-based self-sensing of flaws and stress in carbon-carbon composites, with reports of the electric permittivity, piezoelectricity and piezo-resistivity Carbon 146 447–61
[23] Chung D D L 2019 A review of multifunctional polymer-matrix structural composites Composite B 160 644–60
[24] Zhilyaeva M A, Shulga E V, Shandakov S D, Sergeichev I V, Gilshteyn E P, Anisimov A S and Nasibulin A G 2019 A novel straightforward wet pulling technique to fabricate carbon nanotube fibers Carbon 150 69–75
[25] Zhang X, Kow J, Jones D, de Boer G, Ghanbari A, Serjouei A, Culmer P and Alazmani A 2021 Adjustable compliance soft sensor via an elastically inflatable fluidic dome Sensors 21 1–15
[26] Zeng X, Hurd C, Su H J, Song S and Wang J 2020 A parallel-guided compliant mechanism with variable stiffness based on layer jamming Mech. Mach. Theory 148 103791
[27] Nasibulin A G, Moisala A, Brown D P, Jiang H and Kauppinen E I 2005 A novel aerosol method for single walled carbon nanotube synthesis Chem. Phys. Lett. 402 227–32
[28] Kaskela A et al 2010 Aerosol-synthesized SWCNT networks with tunable conductivity and transparency by a dry transfer technique Nano Lett. 10 4349–55
[29] Nasibulin A G et al 2011 Multifunctional free-standing single-walled carbon nanotube films ACS Nano 5 3214–21
[30] Moisala A, Nasibulin A G, Brown D P, Jiang H, Khriachtchev L and Kauppinen E I 2006 Single-walled carbon nanotube synthesis using ferrocene and iron pentacarbonyl in a laminar flow reactor Chem. Eng. Sci. 61 4393–402
[31] Sun X et al 2019 Flexible tactile electronic skin sensor with 3D force detection based on porous CNTs/PDMS nanocomposites Nano-Micro Lett. 11 57
[32] Hu C F, Su W S and Fang W 2011 Development of patterned carbon nanotubes on a 3D polymer substrate for the flexible tactile sensor application J. Micromech. Microeng. 21 115012