Dedicated Wearable Sensitive Strain Sensor, based on Carbon Nanotubes, for Monitoring the Rat Respiration Rate

Tieying Xu, Mohamad Yehya, Abhishek Singh, Thierry Gil, Patrice Bideaux, Jérôme Thireau, Alain Lacampagne, Benoit Charlot, Aida Todri-Sanial

To cite this version:

Tieying Xu, Mohamad Yehya, Abhishek Singh, Thierry Gil, Patrice Bideaux, et al.. Dedicated Wearable Sensitive Strain Sensor, based on Carbon Nanotubes, for Monitoring the Rat Respiration Rate. ECSA 2021 - 8th International Electronic Conference on Sensors and Applications, Nov 2021, Online, France. pp.27-34, 10.3390/ecsa-8-11293 . lirmm-03441142

HAL Id: lirmm-03441142
https://hal-lirmm.ccsd.cnrs.fr/lirmm-03441142
Submitted on 22 Nov 2021

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Distributed under a Creative Commons Attribution 4.0 International License
Dedicated Wearable Sensitive Strain Sensor, based on Carbon Nanotubes, for Monitoring the Rat Respiration Rate †

Tieying Xu 1,*, A. Mohamad Yehya 2,*, Abhishek Singh Dahiya 1, Thierry Gil 1, Patrice Bideaux 2, Jerome Thireau 2, Alain Lacampagne 2, Benoit Charlot 1 and Aida Todri-Sanial 3

Abstract: This paper presents a highly sensitive and novel wearable strain sensor using one-dimensional material for monitoring the respiration rate of an anesthetized rat. The dedicated sensitive sensor, based on carbon nanotubes mixed with poly(3,4-ethylenedioxythiophene) polystyrene sulfonylate, was attached above the rat chest. A Wheatstone bridge electrical circuit, associated with a multifunction portable device, was connected to the strain sensor. The change of the strain sensor’s resistance value, induced by the mechanical deformability during the rat respiration, was detected and transformed into a voltage signal. The respiration information could be thus extracted and analyzed.

Keywords: wearable strain sensor; rat respiration rate monitoring; carbon nanotubes; poly(3,4-ethylenedioxythiophene) polystyrene sulfonylate

1. Introduction

Cardiovascular disease is a group of disorders of the heart and blood vessels, including coronary heart disease, cerebrovascular disease, and rheumatic heart disease, etc. [1] It is one of the most common causes of death in the world. In 2013, cardiovascular disease claimed a total of 17.3 million lives (31% of total deaths), which is a significant increase from 12.3 million in 1990 (25.8% of total deaths). Since the 1970s, death rates from cardiovascular diseases increases in developing countries regardless of age group, while it declines in most developed countries [2].

There is an increasing demand for a reliable heart monitoring system to capture intermittent abnormalities and detect serious heart behaviors, leading to sudden death in extreme cases. In addition to cardiac monitoring, there is a growing need to capture the respiratory function in several contexts such as sleep apnea. For example, “Internet-of-Medical-Things (IoMT)” are now considered to be a good strategy for monitoring the abnormalities of breathing and cardiac rate. A wearable monitoring strain sensor detects the accidents and sends immediately the information to the medical staff [3].

Therefore, there is a need to develop a highly sensitive, durable, and biocompatible strain sensor. In this paper, a new one-dimensional nanomaterial based strain sensor will be introduced for the respiratory rate monitoring, followed by the fabrication process and
the experimental platform. Then, we perform stretch tests on the strain sensor for the verification of the sensor’s durability. Finally, the results of the respiration monitoring of the anesthetized rat will be presented.

2. Theory

Compared to other strain sensors like capacitive, piezoelectric and triboelectric sensor, piezoresistive sensors have gained interest as they provide high sensitivity with simple device design and readout circuits. The working principle of the piezoresistive sensor is based on the strain effect: when the conductor or semiconductor material is mechanically deformed under the action of external force, its electrical properties change accordingly. The sensitivity of the strain sensor could be characterized by the gauge factor (GF) \[4\]:

\[
GF = \frac{\Delta r/r_0}{\Delta l/l_0},
\]

where \(r_0\) represents the initial resistance of the strain sensor, \(\Delta r\) represents the difference between its real-time resistance under stretching \(r\) and the original value \(r_0\) (\(\Delta r = r - r_0\)), \(l_0\) represents the initial length of the strain sensor, \(\Delta l\) represents the change in the length (\(\Delta l = l - l_0\)), \(l\) the real-time length).

In general, metallic materials have a significantly low GF (about 2), indicating a low sensitivity [5]. In addition, the appearance of the microcracking of the metallic thin-films affects its stretch capability. Non-linear electromechanical behavior is the discrete deformation behavior of the conductive networks at different strain regimes. It impacts the long term sensitivity and stretchability of the strain.

One-dimensional material was thus chosen to solve the problem of the metallic piezoresistive strain sensor. In this paper, carbon nanotubes (CNTs) [6] were mixed with poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) [7] to obtain the filler networks more stable to avoid the resistance value shift caused by device aging and the repetitive tests. CNT network formed a thin film less compact than the metallic layer, whereas the conductive polymer PEDOT:PSS filled the space in the network to ensure the tolerance of the stretch.

To encapsulate the wearable strain sensor, instead of the traditional polymers like Polydimethylsiloxane (PDMS), Dragon skin (DS FX-Pro) was considered for its good elasticity, its robustness as well as its biocompatibility.

3. Experimental

The conventional method of the strain sensor fabrication was realized by the deposition of gold evaporation on Polydimethylsiloxane (PDMS), and then covered all patterned surface with another PDMS layer. The obtained GF was very high (GF from \(2.5 \times 10^6\) to \(10 \times 10^6\)), however, two of the major limitations were: (i) the response was linear up to 0.3% strain only followed by the sudden change in the resistance value, and (ii) poor recoverability. The nominal resistance of the device significantly changed after releasing the applied strain. Therefore, the nanocomposite material like carbon nanotubes was chosen to have a GF high enough (from 3 to 6) to sense subtle movements but more robust and show linear change in resistance up to strain of 40%. Moreover, instead of linear shape, such 1D material was encapsulated in the polymer with ‘U’ shape, which was very useful to increase the sensitivity of the strain sensor.

3.1. Fabrication

To fabricate the strain sensor by the unconventional method, here is the process to follow (See Figure 1):
1. In the first step, dragon skin (DS) chip containing macro channels was realized using a standard molding process. The Kapton tape was cut with a blade to define the dimensions of the macro channel.

2. Then, carbon nanotubes (CNTs) suspension (5 or 6 times, approximately 30 µL at a time) was dropped into DS channel. The suspension, approximately 0.1–0.5wt % in isopropanol (IPA), filled the channels of the DS mold through capillary forces. The conductive polymer poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS), which like an elastic sheet, was deposited and mixed with CNTs. It enables CNTs to stay connected to each other although the strain sensor was under stretch force.

3. Finally, copper (Cu) wires were attached using silver epoxy paste. A ‘sandwich’ type structure was realized by pouring another layer of DS, which flows into the CNT network and cross linked with the bottom layer of DS. The top dragon skin layer encapsulated fully and firmly the CNT sensing material, thus improving the robustness of the device.

![Fabrication process of the strain sensor based on carbon nanotubes (CNTs) mixed with poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS).](image)

3.2. Experimental Set-Up

The wearable strain sensor was first tested using a linear stage controlled by E-861 PiezoWalk® NEXACT® Controller (See Figure 2a). Thanks to the monitoring with LabVIEW controlled by Keithley 2400 source meter, the strain sensor was stretched between 0.01 mm/s to 10 mm/s. When there was no stretch force, the strain sensor’s resistance was equal to other three resistances with fixed value (22 kΩ). While the strain sensor was stretching, the resistance value change due to the mechanical deformability was detected by the Wheatstone bridge and the induced current signal was transformed into voltage signal via an operational amplifier (MCP6401UT) with approximately a gain of two (See Figure 2b). This allows the verification of the viability and durability of the CNTs/PEDOT:PSS based strain sensor. The transformed current signal (or output voltage signal) was monitored by an oscilloscope (DSOX2024A-Keysight Technologies) (See Figure 2c) or a multifunction hardware (Analog Discovery 2) and recorded for data processing. Thanks to Analog Discovery 2, the fast Fourier transform was obtained at the same time under the output voltage signal curve (See Figure 2d).
4. Results and Discussion

With the application of a direct current (DC) source signal (1.8 V) to the dedicated Wheatstone bridge circuit, the evolution of the output signal of the amplifier connected by the Wheatstone bridge circuit is shown in Figure 3. When the stretch length of the strain sensor is equal to 0.5 mm, the output voltage signal changed from 0.04 V to 0.14 V. As the applied stretch force was cyclical, the signal curve was also repetitive. There are some peaks, which were observed at the beginning and the end of the square signal. Such “instantaneous stretch-relax” phenomenon might be related to the elasticity of the strain sensor.

After the validation, such CNTs/PEDOT:PSS based strain sensor was reversibly attached above the rat chest for its respiration monitoring during anesthesia. Isoflurane (2.5%) was used to make the rat sleep during all the experiment [8]. The regular respiration curve for 60 seconds is shown in Figure 4a. The output voltage evolution is between 0.6 and 0.7 V. For 1 min, the rat respiration rate during the rest equals to 62 times/min, which corresponds with the theory value [9]. The electrical monitoring result is very reliable and repetitive. The experiments were carried out by using three different rats, and
stable electrical sensing curves were obtained (Figure 4b–d). The rat’s state was also monitored by the cardiogram, which detected the cardiac frequency during the rat anesthesia (See Figure 5.).

![Figure 4](image.png)

**Figure 4.** (a). Voltage signal for the rat respiration rate monitoring during its rest. Reproductive electrical monitoring curves of rat respiration rate (for 3 different anesthetized rats); (b). For first rat; (c). For second rat; (d). For third rat.

![Figure 5](image.png)

**Figure 5.** Cardiogram for the rat respiration rate monitoring during its anesthesia.

One major drawback we found with this type of sensor is the drifting of the nominal resistance that occurs after intensive stretching of the sensor. We experiment the aging of the sensor by measuring its nominal resistance over several weeks and by stretching at different times. We found that the nominal resistance increases slightly with time but mainly after testing experiences, as shown in Figure 6. The mechanism behind this shift in resistance is not well known and we think that permanent changes in the CNTs network can occur with time but also when the sensor is used for large (>10%) elongations. Carbon nanotubes may move within the polymer composite and contacts between CNTs may break during large elongations.
Finally, in order to build the sensor with common shape and functionalities, a filament mold printed by 3D printing was realized to create the repetitive polymer substrate to insert the CNTs.

5. Conclusions

We developed a new wearable strain sensor, based on one dimensional material (CNTs) mixed with conductive polymer (PEDOT:PSS) and report that the sensitivity and the durability of the wearable strain sensor was improved compared to other CNT based strain sensors. The respiration of anesthetized rat was successful monitored using CNTs/PEDOT:PSS based strain sensor.

In addition to many advantages such as low cost fabrication, simple fabrication process, ease of testing as well as the biocompatibility, stretchable strain sensors will play a crucial role to the monitoring patient with cardiovascular disease as well as their breathing and heart rate. Such sensors will enable the new technological drive called “Internet of Medical Things (IoMT)”, which links wearable devices/sensors into a communication network for real-time or periodic patient-doctor communications. The patient information could be immediately recorded and transformed to healthcare service providers and that might reduce the death rate of cardiovascular disease.

Author Contributions: Conceptualization and methodology, T.X. and A.S.D.; validation, T.X., M.Y., T.G. and P.B.; formal analysis, investigation and resources, T.X. and M.Y.; data curation, T.X.; writing—original draft preparation, T.X.; writing—review and editing, M.Y., A.S.D. and A.T.-S.; visualization, J.T., A.L.; supervision, J.T., A.L., B.C. and A.T.-S.; project administration, A.T.S.; funding acquisition, J.T., A.L., B.C. and A.T.-S. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by European Union’s Horizon 2020 research and innovation programme under grant agreement No. 825114.

Institutional Review Board Statement: All investigations conformed to European Parliament Directive 2010/63/EU and were authorized by local and national ethics committee (CEEA-036-LR, N°22699-2019110611232613 v3).

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.
References

1. Anderson, K.M.; Odell, P.M.; Wilson, P.W.F.; Kannel, W.B. Cardiovascular disease risk profiles. *Am. Heart J.* 1991, 121, 293–298.

2. Moran, A.E.; Forouzanfar, M.H.; Roth, G.A.; Mensah, G.A.; Ezzati, M.; Murray, C.J.L.; Naghavi, M. Temporal Trends in Ischemic Heart Disease Mortality in 21 World Regions, 1980 to 2010. *Circulation* 2014, 129, 1483–1492.

3. Dahiya, A.S.; Thireau, J.; Boudaden, J.; Lal, S.; Gulzar, U.; Zhang, Y.; Gil, T.; Azemard, N.; Ramm, P.; Kiessling, T.; et al. Review—Energy Autonomous Wearable Sensors for Smart Healthcare: A Review. *J. Electrochem. Soc.* 2020, 167, 037516.

4. Yamada, T.; Hayamizu, Y.; Yamamoto, Y.; Yomogida, Y.; Izadi-Najafabadi, A.; Futaba, D.N.; Hata, K. A stretchable carbon nanotube strain sensor for human-motion detection. *Nat. Nanotechnol.* 2011, 6, 296–301.

5. Kang, D.; Pikhitsa, P. V.; Choi, Y.W.; Lee, C.; Shin, S.S.; Piao, L.; Park, B.; Suh, K.-Y.; Kim, T.; Choi, M. Ultrasensitive mechanical crack-based sensor inspired by the spider sensory system. *Nature* 2014, 516, 222–226.

6. Tas, M.O.; Baker, M.A.; Masteghin, M.G.; Bentz, J.; Boxshall, K.; Stolojan, V. Highly Stretchable, Directionally Oriented Carbon Nanotube/PDMS Conductive Films with Enhanced Sensitivity as Wearable Strain Sensors. *ACS Appl. Mater. Interfaces* 2019, 11, 39560–39573.

7. Shi, H.; Liu, C.; Jiang, Q.; Xu, J. Effective Approaches to Improve the Electrical Conductivity of PEDOT:PSS: A Review. *Adv. Electron. Mater.* 2015, 1, 1500017.

8. Liu, Z.M.; Schmidt, K.F.; Sicard, K.M.; Duong, T.Q. Imaging oxygen consumption in forepaw somatosensory stimulation in rats under isoflurane anesthesia. *Magn. Reson. Med.* 2004, 52, 277–285.

9. Williams, K.A.; Magnuson, M.; Majeed, W.; LaConte, S.M.; Peltier, S.J.; Hu, X.; Keilholz, S.D. Comparison of α-chloralose, medetomidine and isoflurane anesthesia for functional connectivity mapping in the rat. *Magn. Reson. Imaging* 2010, 28, 995–1003.