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Study of body movement monitoring utilizing nano-composite strain sensors contaning Carbon nanotubes and silicone rubber

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Abstract. Multi-Walled Carbon nanotubes (MWCNT) coupled with Silicone Rubber (SR) can represent applicable strain sensors with accessible materials, which result in good stretchability and great sensitivity. Employing these materials and given the fact that the combination of these two has been addressed in few studies, this study is trying to represent a low-cost, durable and stretchable strain sensor that can perform excellently in a high number of repeated cycles. Great stability was observed during the cyclic test after 2000 cycles. Ultrahigh sensitivity (GF>1227) along with good extensibility (ε>120%) was observed while testing the sensor at different strain rates and the various number of cycles. Further investigation is dedicated to sensor performance in the detection of human body movements. Not only the sensor performance in detecting the small strains like the vibrations on the throat was tested, but also the larger strains as observed in extension/bending of the muscle joints like knee were monitored and recorded. Bearing in mind the applicability and low-cost features, this sensor may become promising in skin-mountable devices to detect the human body motions.

Keywords: multi-walled carbon nanotubes; silicone rubber; stretchablity; strain sensors; piezoresistive sensor; body movement monitoring

1. Introduction

highly sensitive sensors, there is still a vast field of sensors' production with better qualities that needed to be studied. Modified stretchability and higher ranges of sensitivity can be added to using them for human-related applications to make them a really good target for researchers. Based on different studies these nonconductive matrix can be in different formats 1D (Liu *et al.* 2015, He *et al.* 2018, Lu *et al.* 2018, Li *et al.* 2017), 2D (Zhao *et al.* 2017, Pan *et al.* 2014, Hempel *et al.* 2012), and 3D (Zhang *et al.* 2016, Li *et al.* 2016, Larimi *et al.* 2018, Wang *et al.* 2016). There are some other materials that show great sensitivity toward human movements that makes them a good choice for these kinds of sensors and namely Piezoresistive sensors have shown a great promise in sensing human movements (Di *et al.* 2015, Wang *et al.* 2018, Hwang *et al.* 2015) heart rate monitoring, actuators monitoring (Rostami *et al.* 2019, Amini *et al.* 2019) and pulse measurement (Kaisti *et al.* 2019, Ren *et al.* 2019, Li *et al.* 2017). In other usages, we can name soft robotic, as well as real-time structural health monitoring (SHM) (Montazerian *et al.* 2019, Cheng *et al.* 2019, Lu *et al.* 2019, Ghiasi and Ghasemi 2019) and resin curing screening (Fernández-Toribio *et al.* 2016, Shang *et al.* 2017) in the structural parts and composites.

Two major parts are included as key reasons for testing new combinations: the first part is the diversity of materials that could be applied as elastomeric polymers and the second part which is a very vital and diverse field is the conductive fillers. For instance, low-dimensional carbons (carbon blacks (CBs) (Zhan *et al.* 2019, Narongthong *et al.* 2019, Chen *et al.* 2019, Zhai *et al.* 2019), Carbon nanotubes (CNTs) (Zarei *et al.* 2017, Min *et al.* 2019, Fu *et al.* 2019, Wang *et al.* 2018, Hajmohammad *et al.* 2018), carbon fiber (Taherkhani *et al.* 2020, Beylergil *et al.* 2019) and Graphene (Lu *et al.* 2019, Liu *et al.* 2019, Li *et al.* 2020), nanowires (NWs)(Aziz *et al.* 2019, Kozio *et al.* 2019, Jiang *et al.* 2019), nanoparticles (NPs) (Min *et al.* 2019, Gao *et al.* 2019, Yang *et al.* 2019), etc. are mentioned to be applicable as conductive materials. A wide range of stretchable polymers has also shown the capability for using as the matrix element in strain sensors involving silicon rubber (Azizkhani *et al.* 2019a, azizkhani *et al.* 2019b), polydimethylsiloxane (PDMS) (Wang *et al.* 2019a, Chen *et al.* 2020, Wang *et al.* 2019b), polyethylene terephthalate (PET) (Qin *et al.* 2019, Eutionnat-diffo *et al.* 2019), epoxy (Sapra *et al.* 2019, Chu *et al.* 2019, Sanli 2019), Ecoflex (Steck *et al.* 2019, Mai *et al.* 2019, Amjadi *et al.* 2015), thermoplastic polyurethane (TPU) (Christ *et al.* 2018, Ren *et al.* 2019, He *et al.* 2019), etc.

Trends and processes toward smart textures and textiles are mirrors of future smart textures' and textiles' technology

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which mostly elaborate with some other materials such as nonconductive and it is because of this very important property that a conductive layers can be combined with elastic fibers through the comfortable, low-price, scalable (Gong *et al.* 2017, Zheng *et al.* 2018, Ding *et al.* 2017) and tunable approaches such as molding methods (Cao *et al.* 2017), printed technology (Wang *et al.* 2018), and coating methods like dip coating (Cheng *et al.* 2015, Huang *et al.* 2019).

Fu *et al.* (2019). has fabricated a high-performance MWCNT/polydimethylsiloxane based sensor by molding method toward human motions that shows a series of properties including high sensitivity and stretchability.

Christ *et al.* (2018), fabricated TPU/ MWCNT by printed technology that was made by two-step excursion as a facile method for wearable gloves (Yao and Zhu 2014, Yao *et al.* 2018) and capable of measuring finger flexure and potential of many other applications.

A Graphene-based fiber was fabricated by Yine Cheng *et al.* (Cheng *et al.* 2015) that has high sensitivity and is able to detect different formats of deformation including tensile, bending and torsion .

Previous studies of this team were dedicated to investigating the performance of strain sensors with carbon fiber in the form of chopped particles as the base for the conductive part. The first purposed structure of the sensor was the mixed combination of the CCFs with various weight percentages in the silicone rubber substrate (Azizkhani *et al.* 2019a). The second structure was the sandwiched layer of CCFs playing the role of the conductive networks, between a 1-mm upper and lower layers of silicone rubber (Azizkhani, *et al.* 2019b). The mixed structure showed a relatively low gauge factor (about 50) in 25% of strain value. Although in human motion monitoring, especially for movements with a low range of strain value like detecting the vibrations while speaking, the sensor showed a great performance. Comparing to the first combination the sandwiched structure showed a way more better results. Low hysteresis (3% in ε=50%), higher sensitivity ($100<GF<120$) and considerably higher stretchability (~300%) were the advances of the new structure. Furthermore, the applied applications for large and small deformation in human body monitoring were also desirably responded to.

Following, to lessen the time and cost of the strain sensor fabrication, the present study developed a new fabrication method based on using functionalized Multi-wall carbon nanotubes (MWCNT) sandwiched in layers of silicone rubber (SR). The low-cost, sensitive, and stretchable polymeric matrix sensors were coated with MWCNTs through a coating process and great durability and sensitivity were obtained. The performance of the sensor was monitored via the help of inserting it in a two-prob installation in a universal testing machine. Strain detection ability of the proposed sensor has led to its applicability for human movement monitoring. Therefore, the sensor was sewed in the textile of a glove and its properties were investigated in various amounts of strain values.

2. Results and discussion

This part is dedicated to the characteristics of the presented sensor, a summary of the fabrication method and the performance of the sensor in human motion detection. The results are also discussed fully to have a better understanding of electromechanical behavior in various strain values.

2.1 Sensor fabrication

MWCNTs' properties are the one which attracts great attention among other nano-materials owing to their better accessibility. MWCNTs have become crucially important owing to their special properties such as extremely high mechanical strength, electrical, and thermal conductivities. These properties alongside the appropriate (AR) which describes the length/diameter value, small diameter and the light weight can lead to the high-performance and multifunctional MWCNTs–loading polymer composites. On the other hand, as MWCNTs are generally insoluble in common solvents and polymers, they actually tend to have poor aggregation and dispersion in a polymer matrix. This influences the properties of the MWCNTs–loading polymer composites deleteriously. In order to improve the dispersion of MWCNTs in the polymer matrix, different methods including ultra-sonication treatment and surface modification (e.g., functionalization of MWCNT) have been used. The uniform dispersion of MWNTs in the polymer matrix can be obtained via the help of the functionalization of nanotubes. Due to the hydrophobic feature of MWCNTs, they gather so easily. Hence, the interaction among the MWCNTs and organic materials is partially problematic. According to the previous studies, there are various methods to distribute the CNTs in a polymeric substrate. Functionalization is an effective approach to establish better interactions and lower aggregation on the resin substrate. On the one hand, this leads to a more stable dispersion and on the other hand better transfer of electrical current can be obtained. The survey has utilized functionalizing MWCNTs that the procedure is explained in the experimental section.

Withstanding the extreme temperatures, being more eco-friendly than plastic and low reactivity with chemicals are the most important features of the SR which make it desirable for applications like making a strain sensor. While polymeric materials like PDMS, Ecoflex are widely in use in sensor fabrications, SR is still in a range of polymers which is highly recommended and getting great attention. In comparison with PDMS in applications including human motion monitoring, SR in a specific range of strain values displays better stretchability. Thus, this makes it worthy of a wide range of applications, most importantly in the human-machine interface. As it was observed in the (Montazerian *et al*. 2019) the comparing experiment between spandex (SpX)/SR and Spandex/PDMS indicates this claim that regardless of the great potential of PDMS, the silicone rubber was suggested for the strain sensors fabrication because of its great stretchability. Hereby, the SR was chosen in this study for the polymeric base.

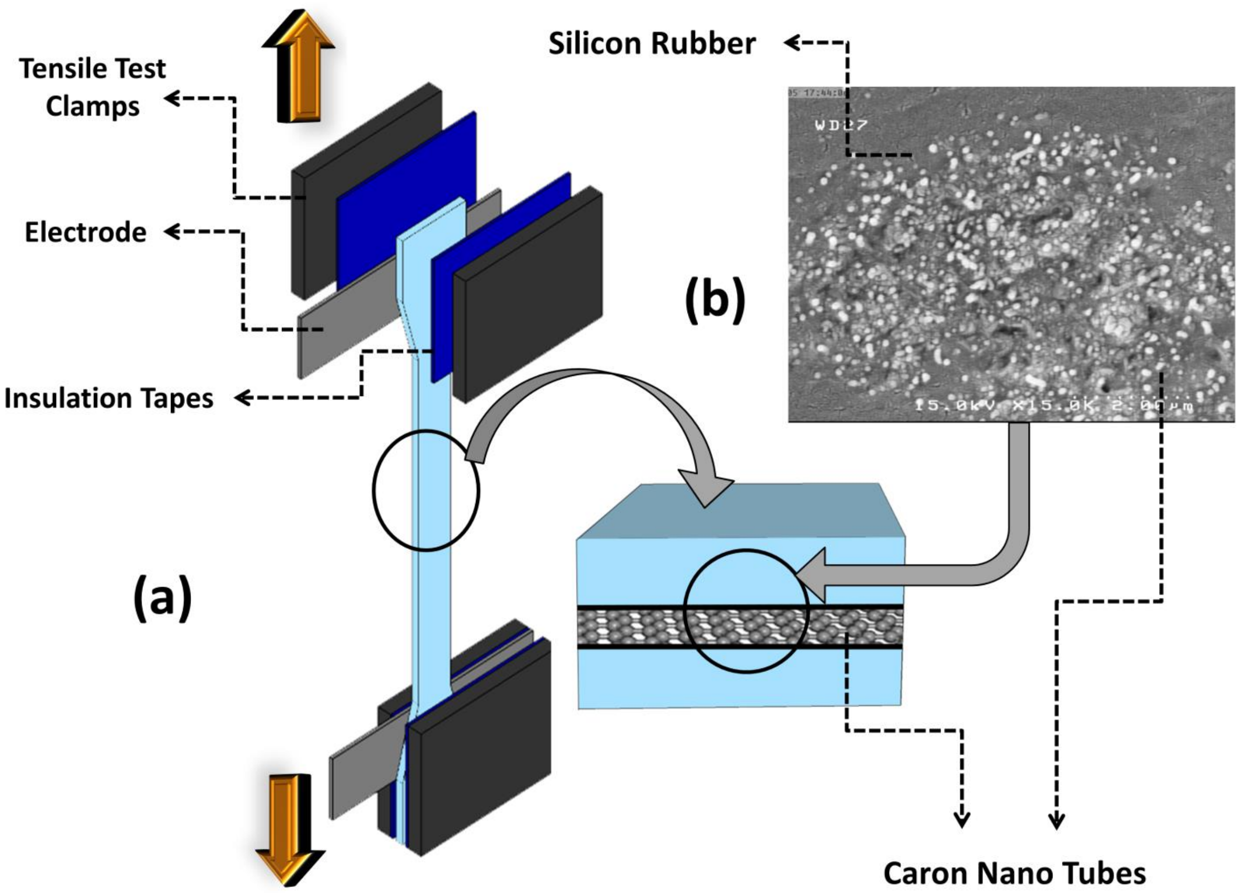


Fig. 1 Schematic view of the CNT/Silicon Rubber strain sensor installation in the Universal tensile testing fixtures while recording the performance by attaching the probes to two electrodes

To achieve the goal of modified sensor fabrication two methods have been applied:

The first procedure included pouring MWCNTs between two SR layers which resulted in the dysfunction of the sensor in the cyclic loadings. That was due to the lack of cohesion between MWCNTs and SR after each strain test (e.g., load and unload). Unless the low durability of the sensor, it could senses the strain values up to 40% of stretching. To overcome this phenomenon, the coating has been used.

In the latter modificative method (Fig. 1(a)), the nanotubes are mixed with other liquids such as water, chloric acid, etc. and the resulting solution is uniformly formed. The solution has been poured in a provided canal-shaped part (0.5 mm*0.5 mm) and then was heated to 100 ℃ in a vacuumed atmosphere for three times. After these three steps, the conductivity was completed and the thin uniform nano-layer laid on the outer part of the sensor. Further, the second silicon layer was laid on nanotubes and the two carbon fiber tows were attached in order to play the role of the connection to the multimeter.

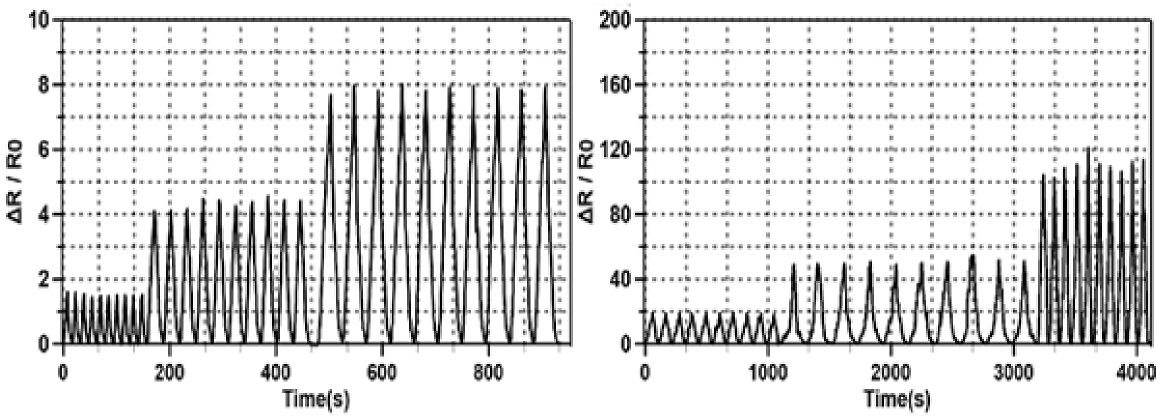
The sandwich-structured cross-sectional SEM was shown in 60% stretchability in Fig. 1(b). As the image demonstrates the carbon nanotubes' the stretch/release process investigation, in spite of slight agglomeration in some parts it resulted in proper nano and conductive path distribution and conductivity on SR.

From ε=0% to ε=120% stretches shows the progressive evolution of MWCNTs configuration between two SR layers. The first image shows 0% of stretch that apparently considered as high conductivity and existence of conductive paths. As the strain goes high the resistance gets higher, for example in the second image that is the illustration of 120% stretch the conductive paths are less than the first image. In 180% that is illustrated in the third scheme, the conductivity has been fully lost. It should be noted that in the range of stretch between 120 to 180% the signals existed but they were not stable enough.

The analysis of the certainly applied deformations to obtain an electrical response. The resistance values by using a two-probe configuration.

There are some parameters that qualify a sensor. The first one can be obtained through the following equation GF of the sensors was obtained

$$\mathrm{GF} = \frac{(\Delta R / R_0)}{\varepsilon}, \quad (1)$$



(a) 10%, 20% and 30% (b) 40%, 50% and 60%

Fig. 2 Resistance changes over time at different strain values

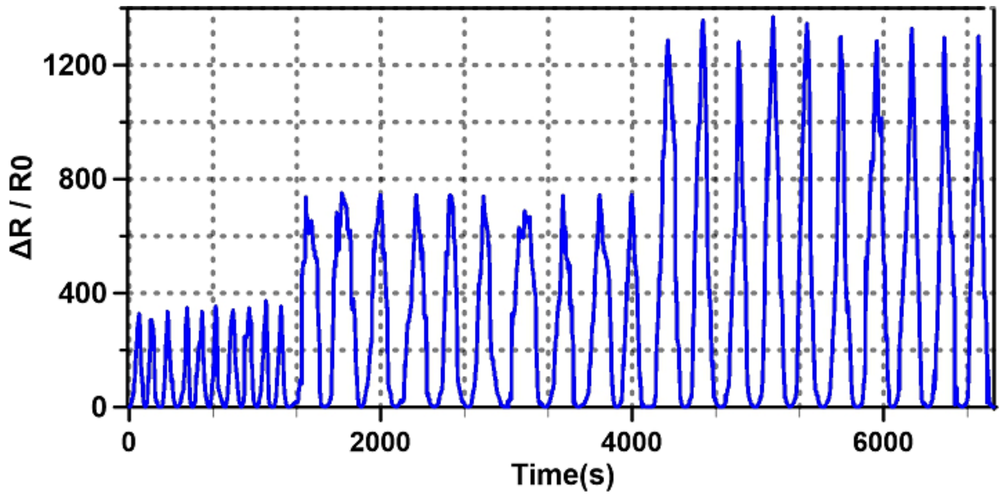


Fig. 3 Resistance changes over time at 80%, 100%, and 120%

The GF, also known as the sensitivity parameter. , is the change between the current resistance and the initial resistance and the initial resistance, respectively. Longitudinal direction strain is shown by e. However, the relationship of relative change in resistance versus strain is close to linearity about 40% strain and experienced nonlinear rise above 40%.

Another important parameter for sensor qualification is the hysteresis behavior of the sensors is also characterized according to the equation:

$\left|A_{Load} - A_{Unload}\right|$ |demonstrates the change in the resistance–strain curve during stretching and releasing cycle and surface area is A_{Load} that is related to stretching. "Electromechanical characterization" is an important way to calculate and discuss the strain with the resistance–strain curves help.

2.2 Results

In the previous study, we have evaluated the effect of the stretchability rate on the sensitivity and hysteresis and as a result, 5 mm/min has been decided to be the rate that is used in this study because of closeness to the actual applications.

Fig. 2 indicates the changes of resistance on stretches between 10% to 60%. The changes have reached from 1.7 to 120. GF in 10% and 40% is 18 and 50 respectively. It can be mentioned that up to this values the linearity of the curve is obvious but from 40% on it is seen to be non-linear.

Fig. 3 indicates the changes of resistance based on time in different strains such as 80% ,100%, and 120%. According to the shape, the resistance is 380,780 and 1368 in 80,100 and 120%. The amount of the changes starts from 0 and for each cycle in any percentage ended up 0 and it shows the stability and the sensitivity of these sensors. According to these three charts, it can be said that the mentioned sensor in 120% stretch has high sensitivity as 1140 which shows the high performance in different industries such as medical sciences and health human detection.

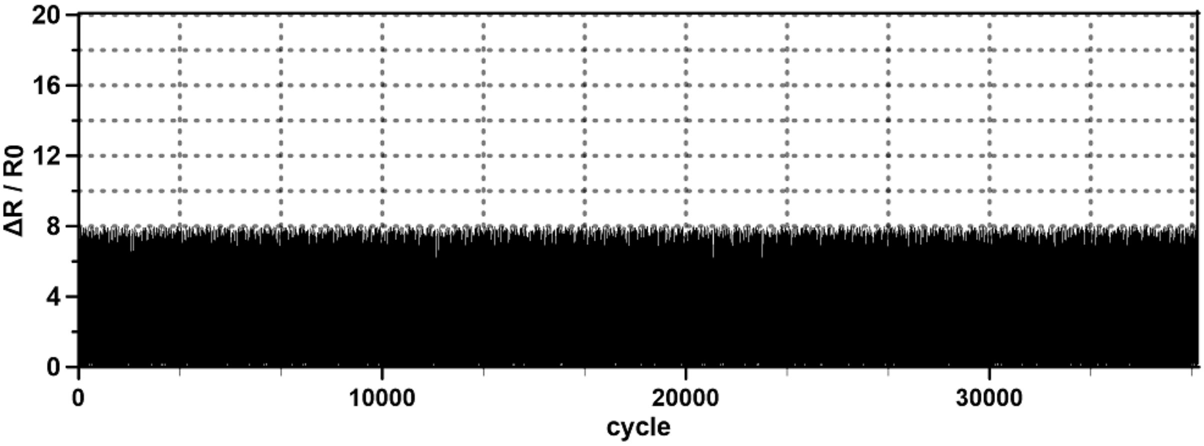
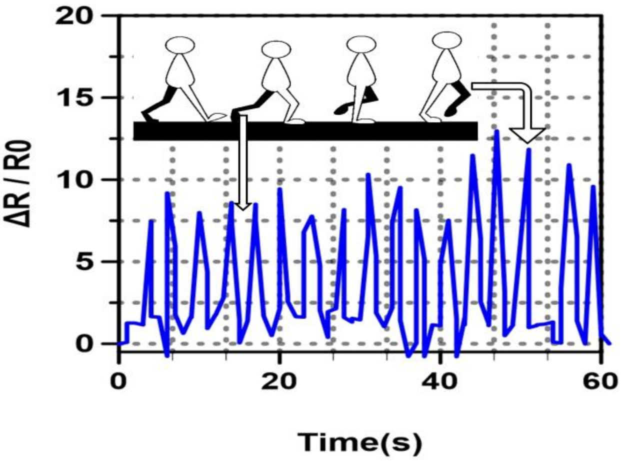
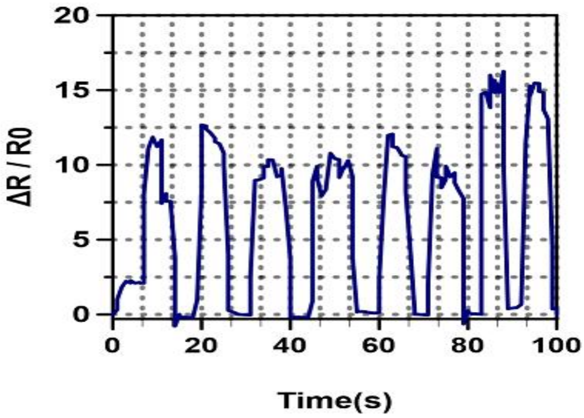


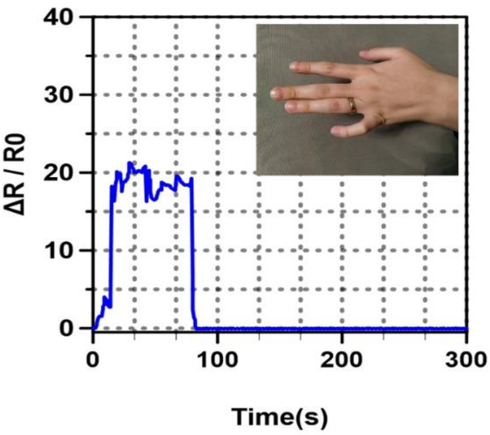
Fig. 4 Time/resistance changes during about 4,000 loading/unloading cycles at 4% strain value



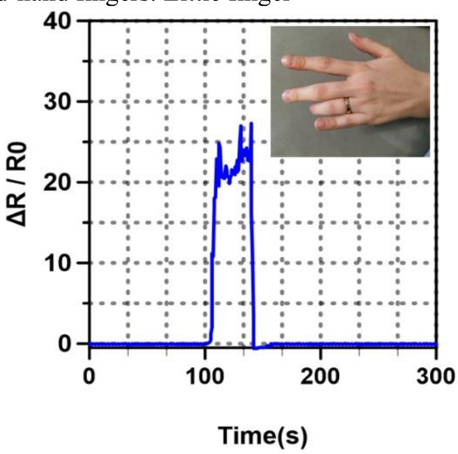
(a) while walking



(b) Time-dependent changes of resistance in knee flexion at 20 ° flexion of the finger. Resistance changes over time in left hand-hand fingers: Little finger

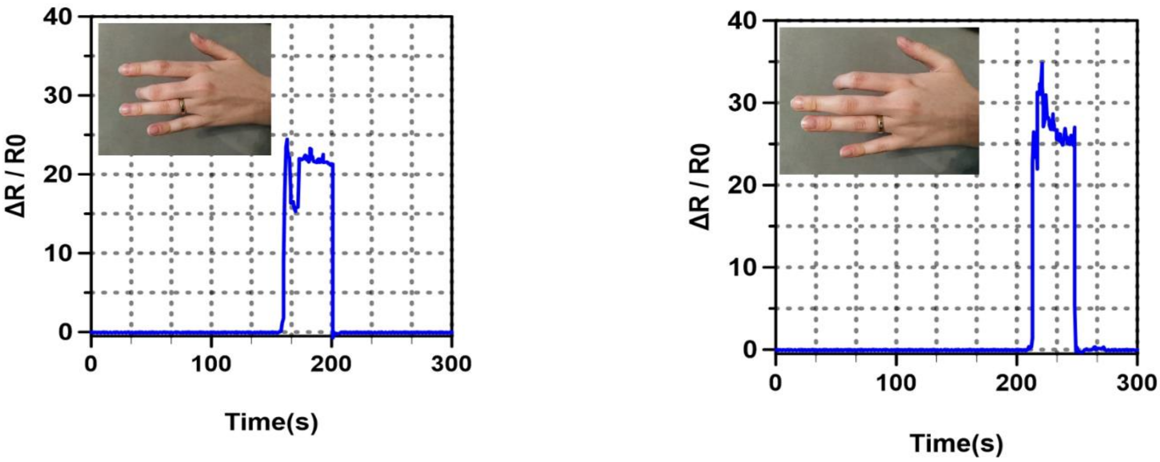


(c) Rng finger



(d) Middle finger

Continued-



(e) Index finger (f) Point Finger

Fig. 5 Resistance changes with time

Finally, for showing the stability of sensors, the change in the resistance of sensors under 4000 cycles of 30% fatigue loading has been used. The maximum and minimum in Fig. 4 were similar that declares the stability and durability of the sensor in each fatigue loading.

2.3 Health human detection

In previous studies, we have analyzed different deformations including large small and minuscule. E.g., knee and wrist joints for large deformation and breathing, etc. For small deformation and minuscule deformation such as heart rate which in all sensor types the durability is an important factor for usage in this field.

In this study, as large deformation, the knee bending /stretching cycle and walking have been analyzed. For small deformations, the finger movement by installing the sensors on a glove and after bending each finger the reaction of the sensor has been monitored. The last analysis was about the minuscule deformation by breathing (chest movement owing to exhale and inhale).

To describe the large deformation it can be said that a sensor was installed on someone's knee and as it's been demonstrated in Fig. 5(a), the person started walking and the sensor performance has been detected. The resistance changes to the initial resistance ratio ($\Delta R / R_0$) were between 7 to 13. In Fig. 5(b) the detection of the same sensor for knee bending/stretching cycle in 20 degrees has been shown in which the changes of the resistance to initial resistance ratio ($\Delta R / R_0$) between 12 to 16.

Small deformation can be detected as it follows:

After installation of the sensor on a glove, in almost 300 seconds, each finger has bent for 50 seconds and got to its initial state. After that other fingers followed the same procedure and got back to their first state. As it is clear according to the image by bending each finger the amount of ($\Delta R / R_0$) has been increased and after getting back to its initial state the amount of ($\Delta R / R_0$) became 0 again. The changes of ($\Delta R / R_0$) have been shown in Figs.5(b)-5(e) between 20 to 30 and it is different according to the bendings. In fact, this subject explains the high sensitivity of the sensor to bending and deformation.

Because of high sensitivity and high stretchability, this sensor has a high ability to distinguishing minuscule deformations. The mentioned sensors have been installed on the chest and in a certain time, the inhale and exhale have been repeated. Considering

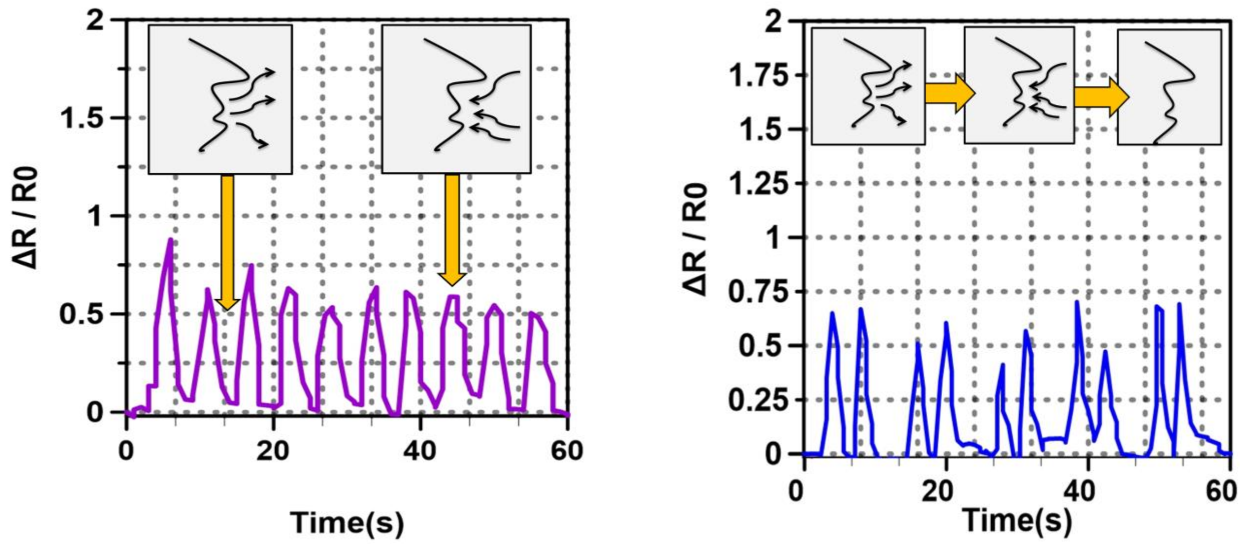
(a) the amount of ($\Delta R / R_0$) gets to its maximum and gets back to 0 as an initial state.

To test the ability to distinguish the deformity in minuscule changes, a different type of breathing has been used. i.e., the breathing was two rapid inhale/exhale and after that, the breath was taken. The mentioned cycle was repeated 5 times. As can be seen in

(b) the amount of ($\Delta R / R_0$) is 0.7 and in inhale, exhale and taking breath times this number is 0 which shows the great sensitivity of the sensor in fast activities.

3. Conclusions

Ultrasensitive strain sensor with a gauge factor greater than 1227 has been presented through this study. A low-cost and easy fabrication method including adding a canal of Multi-Walled Carbon nanotubes (MWCNT) between two layers of silicone rubber (SR) has been used. The connection/disconnection of CNT networks on the polymeric substrate while stretching at different strain values (<120%) is the main mechanism for the repeatability of this sensor. The sensor showed a great result after 2000 cycles as well. Human motion detection was also observed while inserting the sensor on various body part including throat, neck, and fingers. The sensor remarkedly shows great performance. Research into improving the sensor features is already underway while testing new materials for the conductive and also the polymeric base. Considering the new method of fabrication, there is still a



(a) Exhaling and inhaling (b) Exhaling and inhaling with 2-second pauses

Fig. 6 Recording of sensor performance while installation on the neck

Table 1 The characteristic of MWCNTs

purity	>95%
Walls number	3-15
outer diameter	2–6 nm
inner diameter	2–6 nm
length	1-10μm
density	0.15–0.35 g/cm3

broad range of combinations to obtain better and more qualified sensors.

4. Experimental section

4.1 MWCNT/SR sensor fabrication

1. Acid functionalized MWCNTs: The sulfuric acid and nitric acid were purchased from Merck Co, Germany. MWCNTs were purchased from Plasma Chem Co., Germany. The characteristic of MWCNTs is listed in the Table 1.

For forming the carboxylic groups on the micro walls of carbon nanotubes, a solvent including 98% of Sulfuric Acid, 70% Nitric Acid with a 1:3 ratio was applied. In this method, for every 5 grams of carbon nanotubes, 400 ml of the above acidic solution was used. The CNTs were stirred in an acidic solution at 50°C for 20 hours while stirring (Shirkavand Hadavand, Mahdavi Javid, and Gharagozlou 2013).

The following steps were the requirements to have the uniformity, which have been applied to sensor fabrication. In order to neutralize, functionalized MWCNTs were washed with the deionized water until pH: 5.5. Then they were separated with the polyamide filters. In the next step, they were dried in a thermal chamber at 80°C and powdered by milling. To pelletize modified nanotube particles by zirconium pellet mill for 10 min at 300 rpm grinding and carbon nanotubes were obtained as a soft powder.

2. MWCNT/SR strain sensor preparation: The polymer base of this sensor is BISIL 4514 which is the silicone rubber (SR). The thin film of the SR was prepared after the mixing process of the SR with its curing agent with a 25:1 ratio. To remove bubbles, a vacuum chamber was used for 20 minutes. Consequently, 20 grams of the prepared polymer was utilized for each layer of the sensor. Each layer was made via pouring the polymer into the 35 × 5 mm mold which was printed from Polylactic acid (PLA) filament via the help of an FDM 3D-print. Right after the pouring the first layer, in a three-step coating process with a 30-minute time gap for the dehydration, a canal-shaped part (with 35mmm*0.5mm*0.5mm dimension) on the polymeric substrate was filled with MWCNTs. With the aim of monitoring the piezoresistivity of the sensor, the two electrode grips made of carbon fiber have been inserted to both ends of the mold. Respectively, the next layer of SR should be poured after 30 minutes. Finally, mold has been UV cured at 70°C for an hour. The sensor was removed from the mold surface after the curing process.

Measurements: Hitachi Field Emission Scanning Electron Microscopy (FESEM) model s-4160 was used to investigate the microstructural evolution of the conductive paths. The piezoresistivity characteristics of the sensor were observed through the inserting process of the sensor in a two-probe universal testing machine with a 5 mm/min rate cross-head loading called Zwick/Roell D-89079 Ulm. Fully insulation has been made for all the surfaces that could have any slight chance of connection with the sensor or the probes. Finally, the sensitivity of strain sensors was recorded by Victor 86C digital multimeter with computer connection ability. The two probes of the multimeter were connected to the ends of the sensor. The data related to the sensor performance in various types of loading/unloading tests were recorded in the Hand DMM Data software. Gauge factor (GF) defined as the ratio of the relative resistance change divided to the applied strain was calculated by the recorded data. The voltages that have been applied for all the experiments was 1 V.

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