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# A Flexible Tactile Sensor With Good Consistency

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**ABSTRACT** In recent years, tactile sensors have experienced tremendous progress. Among them, piezoresistive tactile sensors using nanocomposites exhibit good flexibility and sensitivity. Due to their low cost and good performance, piezoresistive tactile sensors have great potential for large-scale commercialization. However, the drawbacks are equally obvious, i.e., poor consistency and low-sensing range. The fundamental reason is that conductive fillers have poor decentralization in polymer and lacks an effective structural design. To solve these problems, we compared six common organic solvents in the solution blending method to prepare the multi-walled carbon nanotubes/polydimethylsiloxane (MWCNT/PDMS) composite. The optimal organic solvent by comparing the dispersion performance of MWCNT and the stability of MWCNT/PDMS is presented. Moreover, the well-consistent tactile sensors were proposed and fabricated using the micro-electromechanical systems technologies. The sensitivity reached 6.25% KPa<sup>-1</sup>, and the reproducibility test of the same sensor has been performed. The result proves that the proposed schemes for nano-materials preparation and processing have greatly improved the consistency of the piezoresistive tactile sensors.

**INDEX TERMS** Humanoid robots, tactile sensors, microelectromechanical systems, nanocomposites.

### I. INTRODUCTION

Thanks to the excellent electrical, thermal and mechanical properties, carbon nanotubes (CNTs) have been widely used in the fields of materials, biomedicine, and micro-nano devices since the discovery of CNTs in 1991 by Iijima [1]. The incorporation of CNTs into polymers to prepare nanocomposites has received extensive attention in recent years. This kind of nanocomposites has good piezoresistive effect under the premise of preserving the flexibility and extensibility of the polymer, and the related research has become an important research direction of electronic skin. Compared to the common nanofillers, such as nano-silver particles [2] and carbon black (CB) [3], CNTs have an ultra-high aspect ratio (typically above 1000: 1), making the CNTs/polymer material maintain high conductivity at a lower doping amount, which greatly improves the mechanical properties of composite materials. Polydimethylsiloxane (PDMS) is an inert, non-toxic polymer organosilicon compound and widely used in the micro-channel structure design of microelectromechanical systems (MEMS) chips because of its good elasticity and biocompatibility [4], [5].

The current issues of piezoresistive tactile sensors using CNT/PDMS composites are lack of consistency among each sensing elements and lack of reproducibility [6]. To solve these, it is necessary to fabricate easy-rebound structure and improve the dispersion uniformity of CNTs in PDMS. If the dispersion is not good enough, the conductivity between different sensors could differ greatly, resulting in poor consistency. Without a good easy-rebound structure, the sensor cannot maintain good consistency over long periods of use.

The biggest challenge in the preparation of CNTs/PDMS composites is to overcome the phenomenon of aggregation to distribute CNTs evenly in PDMS against van der Waals forces in CNTs. In recent years, the common preparation methods include melt blending method [7], polymerization method [8] and solution blending method [9]. Among them, the solution blending method has a good application prospect because of its simple operation and has good dispersion effect. The key of this method is the selection of organic solvent to make CNTs have good dispersion effect and dissolve in PDMS. In literatures, the organic solvents used consist of toluene [10], ethanol [11], chloro-

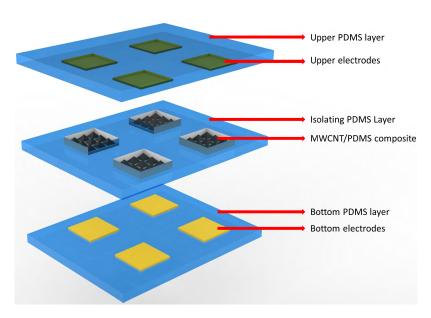


FIGURE 1. Diagram of the tactile sensor structure.

form [12], n-hexane [13], tetrahydrofuran (THF) [14] and dimethylformamide (DMF) [15].

The designs on the easy-rebound structure mainly involves pyramids [10], [16], [17], V-shape grooves [18], and Gaussian random distribution surface profile [19]. The structure was designed not only to increase the response time, but also augment the sensitivity of the sensor. The feature size and density of the structure are also the key parameters that need to be optimized.

In this paper, the dispersion performance of MWCNT and the stability of MWCNT/PDMS in the six organic solvents were compared, and a novel process to distribute CNTs was proposed. Based on the prepared MWCNT/PDMS, a tactile sensor with good consistency was further fabricated based on MEMS technique. The structure diagram is shown in FIGURE 1. The sensor test experiments demonstrate that the proposed preparation process of MWCNT/PDMS and the structure help improve consistency.

# II. EXPERIMENT

## A. PREPARATION OF MWCNT DISPERSION

10 mg MWCNT and 40 mL each of organic solvents, including toluene, ethanol, chloroform, n-hexane, THF, and DMF were separately added into six beakers. The six mixtures were agitated in ultrasound for 1 hour to obtain MWCNT dispersion.

## **B. PREPARATION OF MWCNT/PDMS COMPOSITES**

500 mg PDMS was added to 20 mL chloroform solution and stirred magnetically for 10 min. The PDMS/chloroform mixture was poured into the MWCNT/chloroform dispersion and then sonicated to disperse for 1 hour at 60°C. The evaporation of the solution while sonication can reduce the phenomenon of the composite material adhering to the wall of the beaker compared to the solution evaporation alone. After ultrasonic

dispersion is completed, the mixture is heated in a constant temperature water bath (60°C). The temperature is close to the boiling point of chloroform, so it can accelerate the evaporation of chloroform, and help to maintain uniform dispersion of MWCNTs in PDMS. Heating lasts until the chloroform is completely evaporated. The complete evaporation can be established by measuring the mass of MWCNTs and PDMS left in the beaker. The curing agent was added in a ratio of 1:10 to the base and stirred manually for 5 min. The beaker was placed in a vacuum drying oven and degassed for 15 min. The MWCNT/PDMS composites were then prepared by placing them in a 100°C drying oven for 1 hour.

#### C. FABRICATION OF SILICON TEMPLATE

Si wafer with 100 nm thick SiN layer was used as the substrate. The pyramid base SiN layer was patterned by

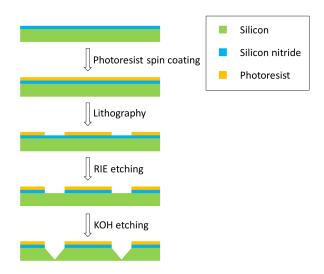


FIGURE 2. Fabrication process of silicon template.



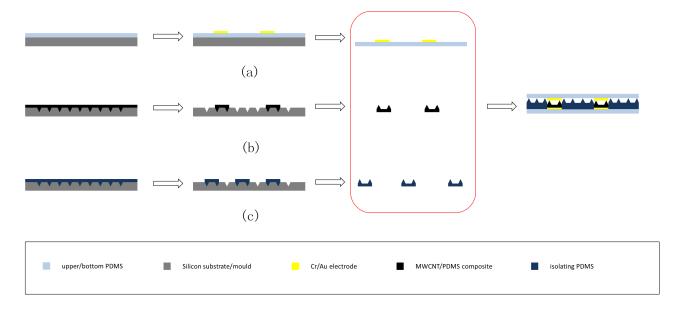


FIGURE 3. Fabrication process of the piezoresistive tactile sensor.

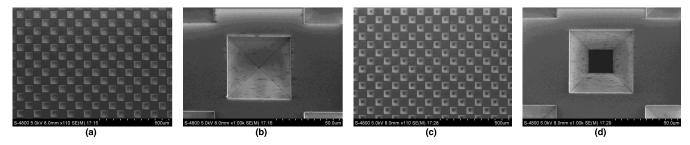


FIGURE 4. SEM image of patterned MWCNT/PDMS composites. (a-b) pyramids; (c-d) truncated pyramids.

photolithography and etched by reactive ion etching (RIE). The pyramid structure was then wet-etched with potassium hydroxide (KOH) and the truncated pyramid structure was obtained by controlling the etching time (FIGURE 2).

#### D. FABRICATION OF TACTILE SENSOR

The fabrication process flow is illustrated in FIGURE 3. The fabrication procedure is divided into three processes. Each process was completed individually.

In the first process, a 200  $\mu m$  thick PDMS was spin coated on a 5 inches silicon wafer substrate and thermally cured at 100 °C in the hot drying cabinet for 30 min. A 50 nm thick Cr layer and a 200 nm Au layer were sputtered on the PDMS film as the electrode layer with a stainless steel shadow mask. The PDMS was then peeled off from the silicon substrate (FIGURE 3a).

In the second process, the prepared CNT/PDMS composite was spin-coated on the prepared silicon mould and thermally cured at 80 °C for 45 min. Then  $4 \times 4$  mm<sup>2</sup> sensing elements were cut out individually and peeled off from the mould (FIGURE 3b). FIGURE 4 shows the scanning electron microscopy (SEM) images of patterned MWCNT/PDMS composites.

Similarly, in the third process, a 170- $\mu$ m-thick PDMS film was spin-coated on the mould as an intermediate isolating layer. A 1  $\mu$ m thick parylene-C was deposited on the mould in between Si and PDMS to avoid the PDMS agglutination on Si. After thermal curing at 100 °C for 30 min, the intermediate isolating PDMS layer was patterned by knife-cutting with a hard mask (FIGURE 3c). Then, the intermediate isolating PDMS layer was stacked in between the top and bottom layers and the composite sensing elements were filled in indentations of isolating layer.

Finally, the upper PDMS layer with metals was attached on intermediate PDMS layer using PDMS gel and the device was thermally hard cured (FIGURE 3d). The image of the fabricated flexible tactile sensor is shown in FIGURE 5a. The sensor is flexible to be attached on cylinder, and is able to twist, and stretch, as shown in FIGURE 5b-d.

#### **III. RESULTS**

#### A. ORGANIC SOLVENT SELECTION

FIGURE 6.a and b are photographs of the six MWCNT after sonication dispersions for 5 min and 30 min. The MWCNT in toluene and n-hexane appeared precipitation soon after sonication, indicating that the dispersion of MWCNT in these

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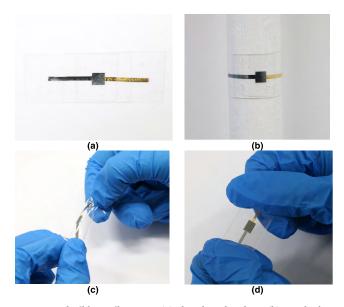


FIGURE 5. Flexible tactile sensor. (a) placed on the plane; (b) attached on the cylinder; (c) twisted; (d) stretched.

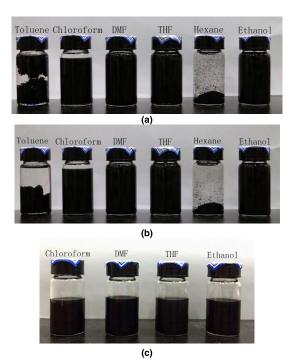


FIGURE 6. The dispersion effect of MWCNT dispersed in different solvents after a period of time. (a) 5 min after sonication. (b) 30 min after sonication. (c) Two weeks after sonication.

two solutions was poor and these two solvents are not suitable to use in this application. Chloroform, DMF, THF and ethanol had good dispersion effect on MWCNT, and their solution could keep steady for longer than two weeks.

FIGURE 7 shows the transmission electron microscopy (TEM) images of these four dispersions. It is seen that the DMF and THF have the best dispersion effect on MWCNTs. There are only minor micron size agglomerations. The ethanol performs worst among the four solvents, where phenomenon of serious agglomeration exhibits.

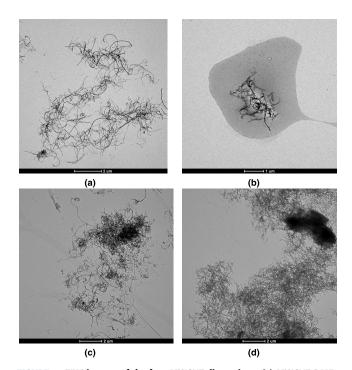


FIGURE 7. TEM images of the four MWCNT dispersions. (a) MWCNT-DMF. (b) MWCNT-THF. (c) MWCNT-Chloroform. (d) MWCNT-Ethanol.

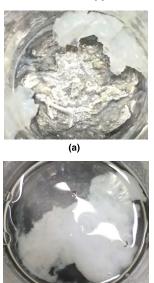


FIGURE 8. The phenomena of adding DMF and ethanol into PDMS matrix.
(a) The substance that PDMS reacts with DMF. (b) PDMS is insoluble in ethanol

(b)

**TABLE 1.** Performance comparison with different solvents.

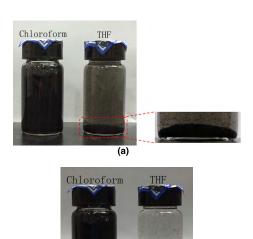
Common organic solvent	Sensitivity	Consistency
Hexane	4.5% KPa <sup>-1</sup>	59.21%
THF	5.8% KPa <sup>-1</sup>	4.46%
chloroform	6.25% KPa <sup>-1</sup>	0.54%
Chlorotothi	0.23% Kra	0.5476

In the experiment of mixture of PDMS and solvents, DMF reacts with PDMS to produce white colloidal material, and PDMS is insoluble in ethanol, either (FIGURE 8). Then,



TABLE 2. Sensitivity comparison with up to date results.

Author	Sensing Principle	Miniaturization Technique	Force/Pressure Sensitivity	Range of Force+(N)/Pressure*(kPa)
J A Dobrzynska et al. [20]	Capacitive	MEMS on Polymer	2.4%/kPa (nf,0-10kPa) 0.066%/kPa (nf,10-140kPa) 0.028%/kPa (shf)	140*
Charalambides et al. [21]	Capacitive	MEMS on Si	190mN(nf) 50mN(shf)	8+(nf) 2+(shf)
Kentaro Noda et al. [22]	Piezoresistive	MEMS on Si	0.17%/kPa	-1.8 - 1.8*
Soonjae Pyo et al. [23]	Piezoresistive	MEMS on Polymer	6.67%/N(nf) 86.7%/N(shf)	2+/163*
Francesco Maita et al.[24]	Piezoelectric	MEMS on Polymer	430mV/N	2+
Eric Fujiwara et al. [25]	Optical	-	0.08N	0.5+
S. Wattanasarn et al. [26]	Magnetic	MEMS on Polymer	0.68 mV/N	2.5+
Our method	Piezoresistive	MEMS on Polymer	6.25%/kPa(0-10kPa) 1.23%/kPa(10-22kPa)	22*



**FIGURE 9.** The dispersion effect of MWCNT / PDMS mixture in chloroform and THF. (a) 1 hour after sonication. (b) 10 hours after sonication.

we compared the stability of MWCNT/PDMS composites in chloroform and THF. The MWCNT dispersion of THF and chloroform were separately mixed with PDMS solution, and left standing for one hour after sonication. It can be seen that the dispersion effect of MWCNT/PDMS mixture in THF solution is poor, and the precipitation phenomenon is obvious, as shown in FIGURE 9a. Since it usually takes 5-6 hours (50 mL of solvent, 60-70°C) to evaporate the solvent in the solution blending method, it is not appropriate to use THF. The mixture in chloroform can maintain a uniform dispersion state for at least 10 hours, as shown in FIGURE 9b. Therefore, chloroform is the optimal solution among the six test solvents. The SEM image of the cross section is shown in FIGURE 10. It's obviously observed that MWCNTs are dispersed evenly in PDMS.

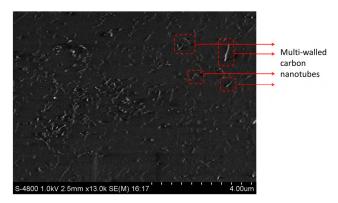


FIGURE 10. SEM image of the cross section of MWCNT/PDMS.

We used Hexane, THF and chloroform as common organic solvents to prepare tactile sensors, and tested their sensitivity and consistency to illustrate the effect of different dispersion levels of MWCNT/PDMS on sensor performance. We tested 10 sensors for each organic solvent, the sensitivity was average, and the difference in consistency was the two with the largest difference. The results were presented in TABLE 1, it could be seen that the sensor prepared by chloroform is superior to the other two in terms of sensitivity and consistency, especially consistency. It is further proved that the dispersion of MWCNT/PDMS is the main factor affecting the performance of the sensor.

# B. ELECTRICAL CHARACTERISTICS OF MWCNT/PDMS COMPOSITE

After evaporation of the chloroform, the prepared MWCNT/PDMS composites were cured into  $25 \times 1.5 \times 1.5 \text{ mm}^3$  strip shape using 3-dimensional (3D) printing mold. We tested the current-voltage (I-V) curve using electrochemical analyzer. The result is shown in FIGURE 11.a, the composites exhibit stable resistance in the range of -15V to 15V. The

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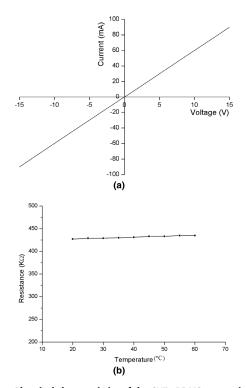


FIGURE 11. Electrical characteristics of the CNTs-PDMS composite.
(a) Current-voltage curve of the CNTs-PDMS composite. (b) Resistance vs. Temperature curve.

resistance of the composite sample was further measured within 20-60 °C in the stability test. The results show that the resistance value fluctuates with 2% in FIGURE 11.b. The prepared MWCNT/PDMS composites exhibit excellent electrical properties and could be used as sensitive materials for piezoresistive tactile sensors.

#### C. CHARACTERISTICS OF TACTILE SENSOR

The parameters such as the length of the pyramid base, the density of the micro-structure, and shape of the pyramid base were explored to find the best performing structural design. Experiment results are shown in FIGURE 12. Sparse pyramid structures are more sensitive than densely arranged pyramid structures, as shown in FIGURE 12. a. This conclusion is consistent with previous work [16]. The pyramid structure has a higher sensitivity in the lower pressure measurement range, while the tactile sensors with truncated pyramid structure could obtain a wider measurement range, as shown in FIGURE 12. a, when the applied pressure is greater than 15 kPa, the resistance of sensor with whole pyramid structure would not change anymore. In addition, the pyramid structure with 20  $\mu$ m feature size narrows the measurement range of the tactile sensor, as shown in FIGURE 12. b due to its small deformation range.

Take the sensor with  $50\mu m$  sparse truncated structure as an example, the sensitivity could reach 6.25%/kPa in the 0 to 100kPa pressure range and 1.23%/kPa in the 10 to  $22\,kPa$  range. The comparison with some up to date results is shown

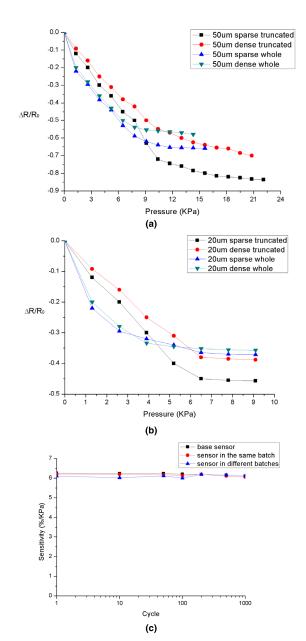


FIGURE 12. Characterization of tactile sensors. (a) Test results of tactile sensors with 50  $\mu$ m feature size. (b) Test results of tactile sensors with 20  $\mu$ m feature size. (c) Sensitivity curve with increasing number of test cycle.

in TABLE 2. It can be seen that the sensitivity of the sensor fabricated by our method reaches state-of-the-art level.

The repeatability of the device (50  $\mu$ m sparsely arranged truncated pyramid structure) was tested by periodically applying a 0.1 N normal force on the device surface by a transmission stage with high-precision force gauge (MARK-10 SH-2). The result is shown in FIGURE 12c black line. The sensor showed excellent consistency in 1000 rounds of testing, with a sensitivity variation of 2.4%. We further tested the resistance of the sensors produced in the same batch and produced with the same parameters in different batch. The initial resistance variation was within 1.2%, and



TABLE 3. Consistency comparison with up to date results.

Author	Sensing Principle	Force/Pressure Sensitivity	Sensitivity Consistency
Shichao Yue et al. [27]	Piezoresistive	0.21 mV/mN·V	200%
Xiang Li et al. [28]	Piezoresistive	−9.95 kPa <sup>−1</sup>	48.44%
		58.3%/N(x-axis)	
Guanhao Liang et al. [17]	Capacitive	57.4%/N(y-axis)	$11.8\% \pm 6.4\%$
		67.2%/N(z-axis)	
Our method	Piezoresistive	6.25%/kPa(0-10kPa)	0.54%
	Flezoresistive	1.23%/kPa(10-22kPa)	0.34%

the sensitivity change was less than 0.54%. Then we test the 1000-rounds sensitivities of these two sensors, as shown in FIGURE 12c red and blue lines. Consistency among different sensors withstood long-term tests with a sensitivity change of less than 3.3%. The consistency comparison with some up to date results is shown in TABLE 3. It can be seen that the consistency of the sensor fabricated by our proposed method is much higher than current results. The methods and processing techniques we proposed to produce composites and sensors show excellent consistency and could be used for reference in large-scale industrial production.

#### IV. CONCLUSION

In this work, we address the main problems, i.e. poor consistency of the nanocomposite based piezoresistive tactile sensor. Optimal solution to prepare the most dispersible MWCNT/PDMS composites was proposed and proved by comparing the six common organic solvents in the solution blending method to prepare MWCNT/PDMS composite. It is demonstrated that chloroform is the best choice of organic solvents by comparing the dispersion performance of MWCNT and the stability of MWCNT/PDMS. Moreover, the MEMS processing technology was utilized to achieve the resilient microstructures to improve the sensitivity and sensing range The effect of different structural parameters on the performance of the sensor was explored. The reproducibility test of the same sensor and the test results of different sensors in the same batch and different batches have proved that the proposed preparation materials and processing schemes have greatly improved the consistency of piezoresistive tactile sensors.

In the future work, dense tactile sensor array will be proposed to achieve tactile sensing over the body. Meanwhile, scanning circuit and tactile signal processing algorithm will be studied.

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