Fabrication and characterization of Screen-printed Graphite and Nickel Based Thick Film Resistive Strain Sensor.

K. SAUJANYA
Department of Instrument Technology
AUCE(A), Andhra University
Visakhaptnam, Andhra Pradesh
INDIA

B. POORNAIAH
Department of ECE
L. B. Reddy College of Engineering
Mylavaram, Krishna District, AP
INDIA

A. KAMALA KUMARI
Department of Instrument Technology
AUCE(A), Andhra University
Visakhaptnam, Andhra Pradesh
INDIA

Y. SRINIVASA RAO
Department of Instrument Technology
AUCE(A), Andhra University
Visakhaptnam, Andhra Pradesh
INDIA

Abstract: - Piezo resistive properties of thick film resistors are shown by a variety of nanomaterials, in which graphite and nickel are used to study the piezo resistive response in this paper. The present work proposes to fabricate stain sensor on substrates like PVC, and transparent plastic sheet. Screen printing method is used for patterning of sensor on the substrates with two different inks namely piezo resistive ink made of graphite and nickel powder and conductive ink made of silver. Change in resistance of the fabricated sensor is noted for the changes in force applied on the sensor and corresponding gauge factor is observed to be around 10.5 and 11 for PVC and OHP respectively. The screen-printed strain gauge performance is investigated and presented in this paper. This study of mechanical test results demonstrate that the sensor can be used for micro strain detection in various applications.

Key-Words: - Piezo resistive, gauge factor, flexible sensor, screen printing, strain sensor, thick film resistor.

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1. Introduction

Flexible strain sensors have promising applications in the field of healthcare, wearable electronics, automotive, human motion detection, robotics etc. after the discovery of nanomaterials [1]–[5]. High sensitivity, flexibility, good stretchability and durability are some of the essential properties that are considered while fabricating any sensor.

Many nanomaterials and nanocomposites such as CNTs, Graphene, graphene oxide, carbon black, carbon nanofibers, carbon Ni composite, are investigated to observe piezo resistive, piezo capacitive and piezoelectric effects [6]–[13]. Among them Nickel and graphite are also found to be strain sensitive material and they are used with other materials to enhance the piezo resistive nature of the overall compound [9], [14]–[16]. The effect of nickel particles in the composite enhances the piezo resistive effect of the compound [16]–[18] and the property is enhanced when there is change in temperature[19], [20]. In this paper, a composite of nickel nanoparticles and graphite is used as sensing material on a flexible substrate and the change in resistance for mechanical deformation is investigated and studied.

Depending on the applications and range of production of sensors, the materials and fabrication technique will be chosen. Inkjet printing[21], screen printing [1], [22], 3D printing [34], laser induction and gravure printing , soft lithography, drop casting, spray coating, spin coating, direct dry transfer etc.

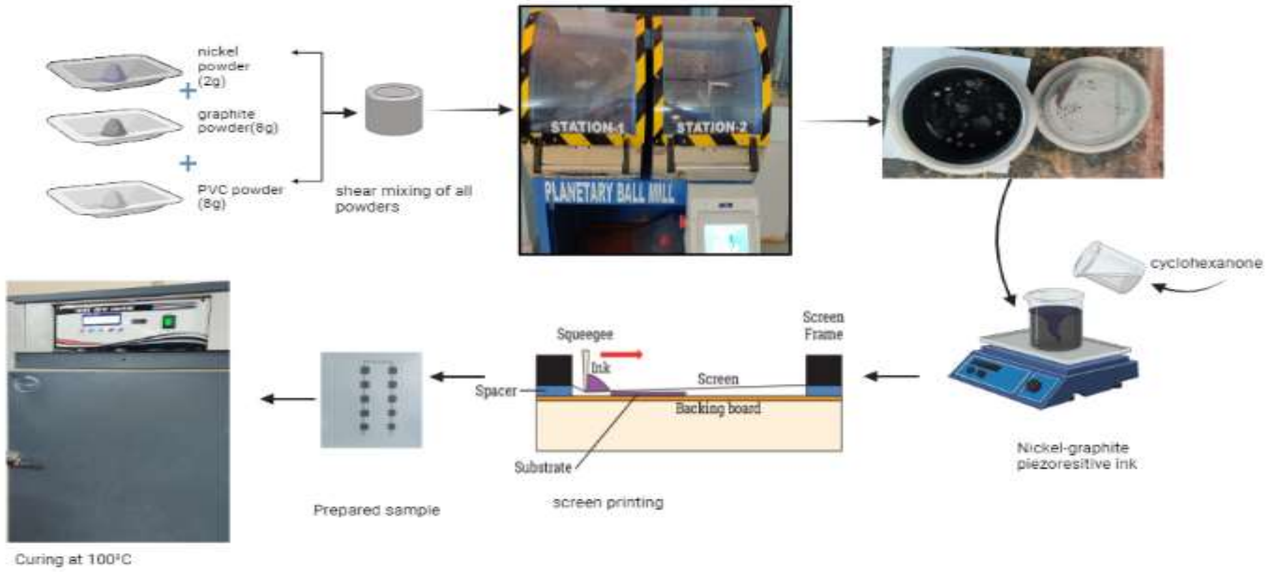

Fig. 1: Schematic diagram illustrating the fabrication of strain sensor

[23], [24] are some technologies for printed electronics on substrates. The advances in screen printing technologies provide many excellent advantages like cost effectiveness, throughput, ease in fabrication, and many more[1]. In the present work screen printing technique is used for printing of strain sensors on flexible substrates. The screens are made on A4 size with 200 mesh detailed at 45 lines per inch. Screen printing method provides the researcher an ease in fabrication of sensor and the advantages of this method encouraged us to use the method in this work.

When a stress is applied on the strain sensor electrical parameter changes which is due to the deformation in material. The mechanical deformation can be applied on the sensor by stretching, pressing, bending, tapping methods. [25][26], [27] has used a Tensile test machine experimental setup for mechanical analysis of the sensor. [22] Reported strain sensing of a silver carbon composite screen printed strain gauge on TPU by applying different loads on the sensor to observe the resistance changes. [28] Studied the sensing behaviour of flexible conductive PDMS/NCG nickel coated graphite composite fabricated by natural sedimentation method. The author reported resistance of the sensors were increased and then decreased when the sensor was under twisting, stretching and bending mode. [26]–[28] used bending property for applying mechanical strain to the sensor which were used mostly in human motion detection and soft robotics. Another method to give strain to the sensor is stretching, in which force is applied in the opposite direction on the sensor. This can be achieved on a Universal Testing Machine that is reported by [29] for styrene based ternary composite elastomer for supercapacitor integrated strain sensor system, [30]–

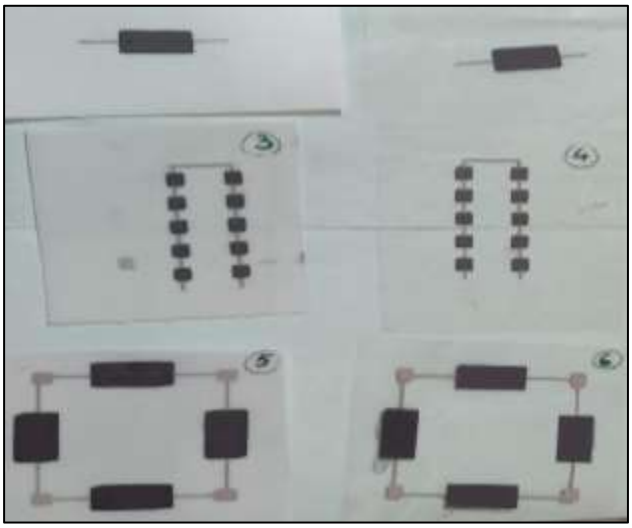

Fig. 2: Screen printed samples, left are PVC sheet and right are OHP sheet

[32] reported similar testing method for testing the strain sensing of the composite materials. In this investigation, the electromechanical behaviour of the prepared sensor is being stretched using UTM and relative change in resistance is observed.

In this work, a strain sensing resistor is fabricated using the screen-printing method on a flexible substrate that has nickel and graphite as sensing material and silver as conductive material. The strain response characteristics and stretchability of the sensor is studied.

2. Experimental Section

2.1 Materials:

Nickel nanoparticle powder, graphite powder and silver paste were purchased from SILTECH CORPORATION INC, India. PVC (poly vinyl chloride) powder is purchased from local vendor, High purity solvent Cyclohexanone (00087) purchased from Loba Chemie pvt. Ltd., India. Silver conducting paste was utilized to fabricate conductive contacts at the ends of the sensor. Acetone is purchased for the cleaning of equipment and substrates from sigma Aldrich.

Substrates used: PVC Sheet and transparent plastic sheet are used as flexible substrates in this work.

All the materials and chemicals were used as received without further purification.

2.2 Sensor Fabrication:

The schematic diagram of fabrication of strain sensors is shown in Fig 1. Firstly, two screens are prepared (one for conductive ink and other for nickel graphite ink) with different patterns for the printing process. Piezo resistive ink is prepared by mixing 8g of graphite powder and 2g of nickel Nano powder in planetary ball mill for 60 minutes. 2g of PVC powder is added to the mixture and ball milling is done for 30 min using 10 balls at 1000 rpm. 20 ml of cyclohexanone is mixed to the powder to form a paste for screen printing. Conductors are screen printed on the flexible substrates using Silver conductive paste to evaluate the performance of the sensor. Samples are cured at 100^0C for 2 hours. Then piezo resistive ink is printed to form a resistor in different patterns as shown in Fig. 2. Curing at 100^0C for 4 hours.

The screen-printed thick film resistors with 20mm length and 10mm width (single resistor), 5mm x 5mm (series resistors) and bridge formation using the first pattern on PVC sheets and transparent plastic sheets are shown in Fig. 2. The nickel and graphite powder have a particle size of 325 mesh that is suitable for prepared screens for the printing process. The thickness of the resistors on the substrates is found to be 45µm using a thick film

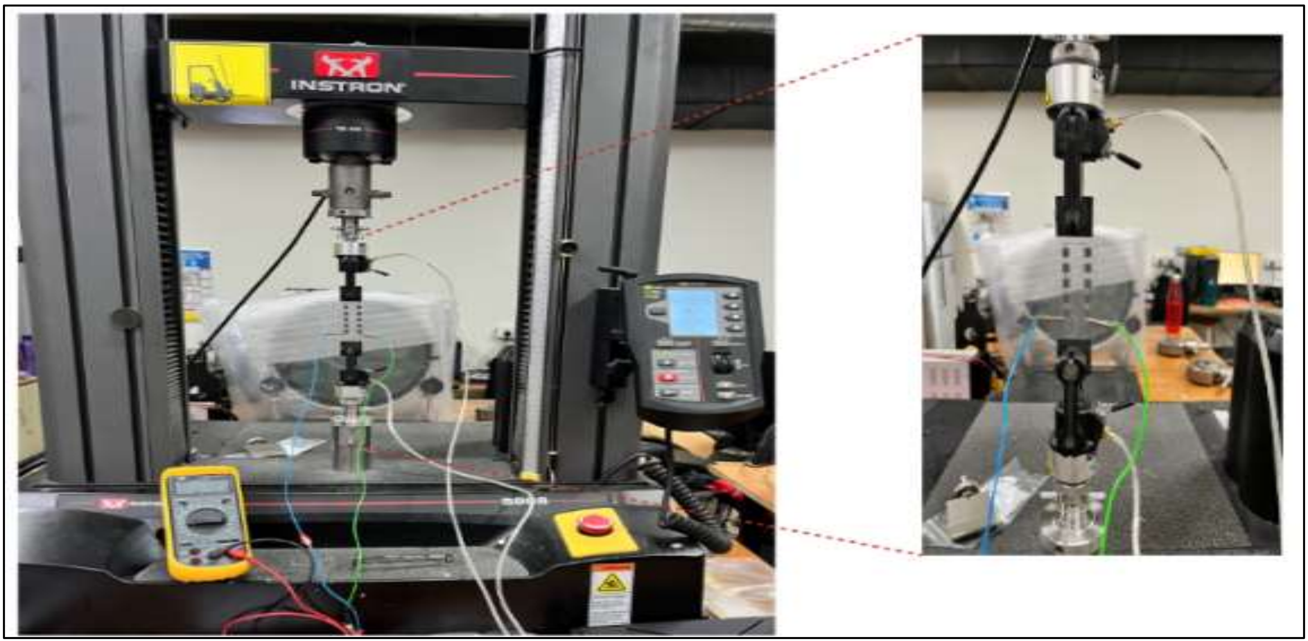

Fig 3: Tensile testing of the sample on Universal Testing Machine Instron 5966 and a digital multimeter is used to measure electrical resistance.

thickness measuring gauge.

2.3 Characterization

Morphological and structural studies of the fabricated sensor were characterized by SEM (ZEISS ULTRA55, INDIA). The Rigaku Smart Lab XRD is used for chemical composition and crystalline size of the material. FTIR (Fourier Transform Infrared) spectra were recorded on Perkin Elmer. Electromechanical studies of the sensor are done by performing tensile testing on a Universal Testing machine (INSTRON5966) and electrical resistance for applied load is noted using a digital multimeter as shown in Fig 3.

3. Results and Discussions

Characterization by XRD (X-ray Diffraction) was carried out for the prepared sensor to observe the structural analysis and crystallinity. The corresponding spectra is shown in Fig 4 where the large peak at 25.8 degrees and shorter peak at 44.38 degrees represents the presence of Graphite and Nickel respectively. Individual XRD patterns of nickel powder and graphite powder to study the strain sensing behaviour are reported and crystallinity is found to be 99.57% and 82% respectively [13].

Fig 5 shows the SEM images of the Nickel graphite composite screen-printed sensors on PVC substrate before and after mechanical force applied on the sensor. The images show that the low viscosity composite is roughly and uniformly deposited on the substrate which is due to curing of the sensors

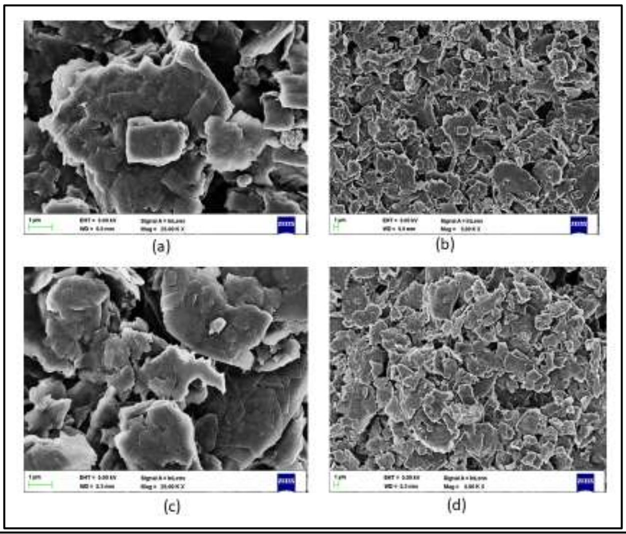

Fig 5: The SEM images of Screen-printed strain sensors on PVC film: (a) and (b) before stretching; (c) and (d) after stretching

after printing. A representative high magnification view of Fig 5 (a) and (c) are shown in (b) and (d). When mechanical strain is applied on the sensors the conductive nature of the material reduces resulting resistive behaviour. Fig 5 (c) and (d) shows the morphology of the PVC strain sensor after strain is applied on the sensor.

The electromechanical response of the strain sensor printed on a flexible substrate by tensile test is done on a Universal Test Machine Instron 5966. The strain sensing measurement setup is shown in Fig 6.

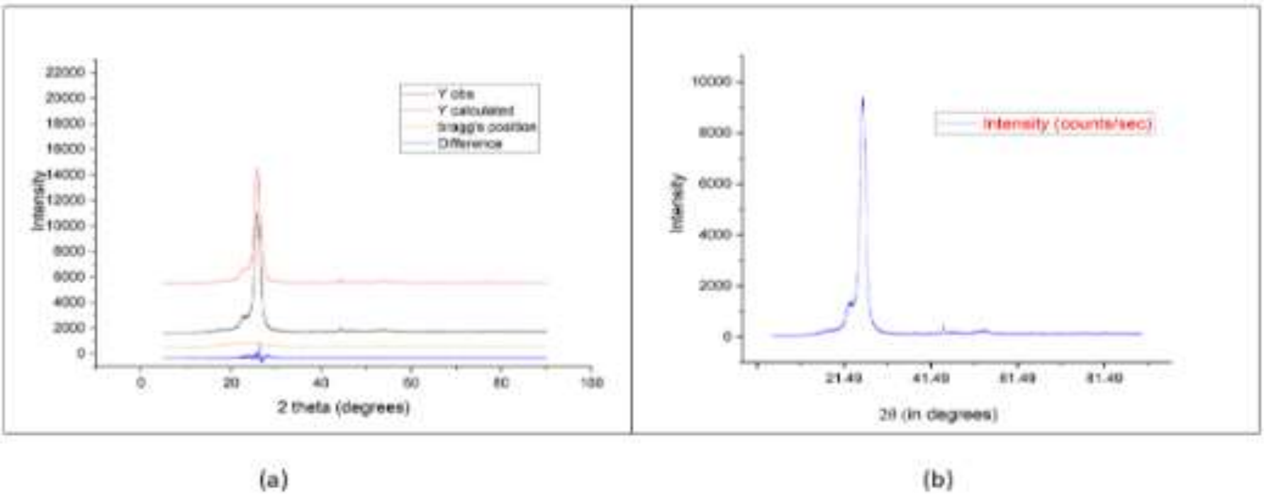

Fig 4: XRD spectra of prepared piezo resistive screen-printing ink

The prepared sensors were stretched with a constant velocity of 0.5 mm/min on the UTM and the resistance of the sensor was noted before stretching. The applied force on the sensor was converted to

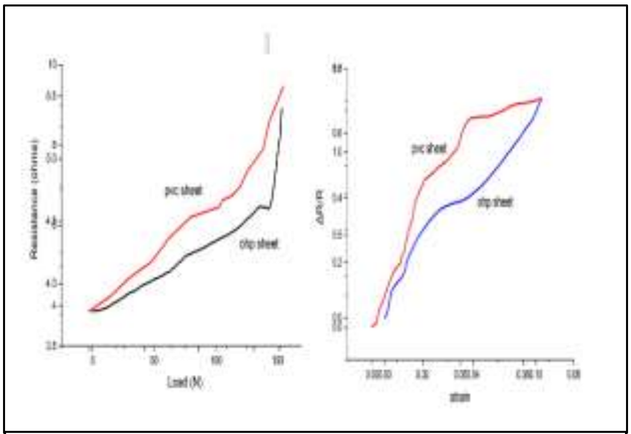

Fig 6: The change in electrical resistance (ohms) on application of load ranging from 0N to 150N (left); the electrical resistance as a function of strain for screen printed sensor on different substrates (right)

change in the original resistance which shows the piezoresistive behaviour of the sensor. The resistance is measured using a digital multimeter. The response of strain vs change in resistance and load vs resistance of the strain sensor on two different flexible substrates are shown in Fig 6.

The Gauge factor of the sensor can be calculated using following equations

$$Gauge\ factor = \frac{(\Delta R/R)}{\epsilon} \quad (1)$$

Where, $Strain, \epsilon = \frac{\Delta L}{L}$, (2)

ΔR is change in resistance,

R is original resistance of the sensor,

ΔL is extension or change in length for applied load,

L is the original length of the sensor.

4. Conclusion

In this paper, the screen-printed strain sensor using a Nickel-graphite composite is demonstrated for measuring low to high strain by observing the relative change in resistance of the sensor. Gauge factor for PVC sheet is found to be 10.5 and for OHP sheet is 11. The results of the electromechanical tests of printed strain sensor indicates the strain sensing capability of the prepared Ni-graphite composite material and this sensor can be further be used in health monitoring [35], electronic applications, gauges, medical prosthetics, nanosensors, wearable electronics etc.

The studies of the fabricated sensor are so promising that the sensor can further undergo temperature sensitivity, durability and thickness analyses. The fabricated sensor is providing GF near to 11 that is comparable to previous studies [33] which depends on the type of substrate used for fabrication. The future work can be done to improve the sensitivity and bendability of the sensor using other flexible substrates like Polydimethylsiloxane (PDMS), Polyimide, Fabrics etc. that can be used in flexible and wearable electronic applications.

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References

[1] N. Zavanelli and W. H. Yeo, “Advances in Screen Printing of Conductive Nanomaterials for Stretchable Electronics,” *ACS Omega*, vol. 6, no. 14, pp. 9" Review Review in preparation and application of nickel-coated graphite composite powder." 05 May. 2021, https://www.sciencedirect.com/science/article/pii/S0925838820343784. *344*–9351, 2021, doi: 10.1021/acsomega.1c00638.

[2] V. J. Babu *et al.*, “Intelligent Nanomaterials for Wearable and Stretchable Strain Sensor Applications: The Science behind Diverse Mechanisms, Fabrication Methods, and Real-Time Healthcare,” *Polymers (Basel).*, vol. 14, no. 11, 2022, doi: 10.3390/polym14112219.

[3] W. Liu *et al.*, "Stable Wearable Strain Sensors on Textiles by Direct Laser Writing of Graphene," *ACS Appl. Nano Mater.*, vol. 3, no. 1, pp. 283–293, 2020, doi: 10.1021/acsanm.9b01937.

[4] L. E. Aygun *et al.*, "Large-area resistive strain sensing sheet for structural health monitoring," *Sensors (Switzerland)*, vol. 20, no. 5, pp. 1–15, 2020, doi: 10.3390/s20051386.

[5] Z. Liu *et al.*, *Functionalized Fiber-Based Strain Sensors: Pathway to Next-Generation Wearable Electronics*, vol. 14, no. 1. 2022.

[6] O. Kanoun, A. Bouhamed, R. Ramalingame, J. R. Bautista-Quijano, D. Rajendran, and A. Al-Hamry, "Review on Conductive Polymer/CNTs Nanocomposites Based Flexible and Stretchable Strain and Pressure Sensors," *Sensors*, vol. 21, no. 2, pp. 1–29, 2021, doi: 10.3390/s21020341.

[7] T. Yan, Z. Wang, and Z. J. Pan, "Flexible strain sensors fabricated using carbon-based nanomaterials: A review," *Curr. Opin. Solid State Mater. Sci.*, vol. 22, no. 6, pp. 213–228, 2018, doi: 10.1016/j.cossms.2018.11.001.

[8] X. Zhang *et al.*, "Flexible and high-performance piezoresistive strain sensors based on carbon nanoparticles@polyurethane sponges," *Compos. Sci. Technol.*, vol. 200, no. September, p. 108437, 2020, doi: 10.1016/j.compscitech.2020.108437.

[9] D. D. L. Chung and X. Xi, "Piezopermittivity for capacitance-based strain/stress sensing," *Sensors Actuators A Phys.*, vol. 332, p. 113028, 2021, doi: 10.1016/j.sna.2021.113028.

[10] H. Liu *et al.*, "Flexible, Degradable, and Cost-Effective Strain Sensor Fabricated by a Scalable Papermaking Procedure," *ACS Sustain. Chem. Eng.*, vol. 6, no. 11, pp. 15749–15755, 2018, doi: 10.1021/acssuschemeng.8b04298.

[11] D. Zymelka, T. Yamashita, X. Sun, and T. Kobayashi, "Printed strain sensors based on an intermittent conductive pattern filled with resistive ink droplets," *Sensors (Switzerland)*, vol. 20, no. 15, pp. 1–14, 2020, doi: 10.3390/s20154181.

[12] P. Walter *et al.*, "CNT/Graphite/SBS Conductive Fibers for Strain Sensing in Wearable Telerehabilitation Devices," *Sensors*, vol. 22, no. 3, 2022, doi: 10.3390/s22030800.

[13] S. J. Lee, I. You, S. Kim, H. O. Shin, and D. Y. Yoo, "Self-sensing capacity of ultra-high-performance fiber-reinforced concrete containing conductive powders in tension," *Cem. Concr. Compos.*, vol. 125, no. November 2021, p. 104331, 2022, doi: 10.1016/j.cemconcomp.2021.104331.

[14] Y. Bai, F. Qin, and Y. Lu, "Lightweight Ni/CNT decorated melamine sponge with sensitive strain sensing performance for ultrahigh electromagnetic absorption in both GHz and THz bands," *Chem. Eng. J.*, vol. 429, no. June 2021, p. 132393, 2022, doi: 10.1016/j.cej.2021.132393.

[15] R. Zhang *et al.*, "Facile one-step preparation of laminated PDMS based flexible strain sensors with high conductivity and sensitivity via filler sedimentation," *Compos. Sci. Technol.*, vol. 186, no. July 2019, pp. 1–7, 2020, doi: 10.1016/j.compscitech.2019.107933.

[16] J. W. Um, S. Y. Kim, B. H. Lee, J. B. Park, and S. Jeong, "Direct writing of graphite thin film by laser-assisted chemical vapor deposition," *Carbon N. Y.*, vol. 169, pp. 163–171, 2020, doi: 10.1016/j.carbon.2020.07.035.

[17] X. Xi and D. D. L. Chung, "Effect of nickel coating on the stress-dependent electric permittivity, piezoelectricity and piezoresistivity of carbon fiber, with relevance to stress self-sensing," *Carbon N. Y.*, vol. 145, pp. 401–410, 2019, doi: 10.1016/j.carbon.2019.01.034.

[18] Š. Meškinis *et al.*, "Giant Negative Piezoresistive Effect in Diamond-like Carbon and Diamond-like Carbon-Based Nickel Nanocomposite Films Deposited by Reactive Magnetron Sputtering of Ni Target," *ACS Appl. Mater. Interfaces*, vol. 10, no. 18, pp. 15778–15785, 2018, doi: 10.1021/acsami.7b17439.

[19] C. Karapepas, D. Nestler, D. Wett, and G. Wagner, "Annealing effects of high sensitive thin strain gauges consisting of nickel carbon nanocomposites," *J. Reinf. Plast. Compos.*, vol. 37, no. 22, pp. 1378–1384, 2018, doi: 10.1177/0731684418796309.

[20] C. Karapepas, D. Nestler, and G. Wagner, "Influence of sputtering temperature and layer thickness on the electrical performance of thin film strain sensors consisting of nickel-carbon composite," *Key Eng. Mater.*, vol. 809 KEM, pp. 413–418, 2019, doi: 10.4028/www.scientific.net/KEM.809.413.

[21] T. K. Kang, "Inkjet printing of highly sensitive, transparent, flexible linear piezoresistive strain sensors," *Coatings*, vol. 11, no. 1, pp. 1–8, 2021, doi: 10.3390/coatings11010051.

[22] A. K. Bose *et al.*, "Screen Printed Silver/Carbon Composite Strain Gauge on a TPU Platform for Wearable Applications," *FLEPS 2020 - IEEE Int. Conf. Flex. Printable Sensors Syst.*, 2020, doi: 10.1109/FLEPS49123.2020.9239547.

[23] N. Ivanova, V. Gugleva, M. Dobreva, I. Pehlivanov, S. Stefanov, and V. Andonova, "We are IntechOpen , the world ' s leading publisher of Open Access books Built by scientists , for scientists TOP 1 %," *Intech*, vol. i, no. tourism, p. 13, 2016.

[24] M. J. Yee *et al.*, "Carbon nanomaterials based films for strain sensing application—A review," *Nano-Structures and Nano-Objects*, vol. 18, p. 100312, 2019, doi: 10.1016/j.nanoso.2019.100312.

[25] D. Zymelka, T. Yamashita, X. Sun, and T. Kobayashi, "Large-scale printed strain sensors based on carbon ink incorporated into an intermittent conductive silver pattern," *Jpn. J. Appl. Phys.*, vol. 60, no. SB, 2021, doi: 10.35848/1347-4065/abd6db.

[26] A. Nag *et al.*, "A Transparent Strain Sensor Based on PDMS-Embedded Conductive Fabric for Wearable Sensing Applications," *IEEE Access*, vol. 6, pp. 71020–71027, 2018, doi: 10.1109/ACCESS.2018.2881463.

[27] S. Zhao, P. Zheng, Q. Liu, L. Niu, H. Cong, and A. Wan, "Highly stretchable strain sensor with tunable sensitivity via polydopamine template-assisted dual-mode cooperative conductive network for human motion detection," *Mater. Des.*, vol. 206, p. 109780, 2021, doi: 10.1016/j.matdes.2021.109780.

[28] S. Li *et al.*, "The tunable sensing behaviors of flexible conductive PDMS/NCG composites via regulation of filler size prepared by a facile sedimentation method," *Compos. Sci. Technol.*, vol. 216, no. June, 2021, doi: 10.1016/j.compscitech.2021.109037.

[29] D. Park, Y. K. Park, S. Selvam, and J. H. Yim, "Styrene-based ternary composite elastomers functionalized with graphene oxide-polypyrrole under iron(III)-alkyl benzenesulfonate oxidants for supercapacitor integrated strain sensor system," *J. Energy Storage*, vol. 51, no. March, p. 104543, 2022, doi: 10.1016/j.est.2022.104543.

[30] S. Nuthalapati *et al.*, "Highly sensitive, scalable reduced graphene oxide with palladium nano-composite as strain sensor," *Nanotechnology*, vol. 31, no. 3, 2020, doi: 10.1088/1361-6528/ab4855.

[31] P. Zhang *et al.*, "Flexible piezoresistive sensor with the microarray structure

based on self-assembly of multi-walled carbon nanotubes," *Sensors (Switzerland)*, vol. 19, no. 22, 2019, doi: 10.3390/s19224985.

[32] Z. Tian, Y. Li, S. Li, S. Vute, and J. Ji, "Influence of particle morphology and concentration on the piezoresistivity of cement-based sensors with magneto-aligned nickel fillers," *Meas. J. Int. Meas. Confed.*, vol. 187, no. October 2021, p. 110194, 2022, doi: 10.1016/j.measurement.2021.110194.

[33] Daniel Zymelka et al., "Large-scale printed strain sensors based on carbon ink incorporated into an intermittent conductive silver pattern" 2021 Jpn. J. Appl. Phys. 60 SBBM01, DOI 10.35848/1347-4065/abd6db

[34] A. Arivarasi, R. Anand Kumar, "3D Printing of Copper Filament for Layered Fabrication" Volume 7, 2016, WSEAS TRANSACTIONS on ELECTRONICS. E-ISSN: 2415-1513

[35] M. J. Burke, C. Molloy, H. Fossan, "Low-Power Measurement of Contact Impedance in Dry Electrocardiography" Volume 7, 2016, WSEAS TRANSACTIONS on ELECTRONICS. E-ISSN: 2415-1513

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