Application of functionalised MXene-carbon nanoparticle-polymer composites in resistive hydrostatic pressure sensors

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Abstract

The synthesised and functionalised materials were characterised using TEM, SEM, Raman and P-XRD. The characterisation techniques confirmed that a successful functionalisation of Ti_3AlC_2 , the synthesised and functional groups on the carbon nanoparticles. The TEM and SEM were also used to study the surface morphology of the materials. The pressure sensor was fabricated by depositing the polymer composites on the IDE and different types of pressure sensors were prepared by varying the sensing materials in the composites. Generally, the fabricated sensors, based on the two polymers, Ppy and PVP polymer, have shown a linear relationship between the applied pressure and the relative resistance response in their respective ranges. The best sensitive sensors were f-MC/Ppy bases sensor 0.00232 kPa^{-1} in a range from 126 to 168 kPa, in a very wide range, 68–168 kPa, the sensitivity of the f-MC/CNPs based sensor was 0.00017 kPa^{-1} . The sensors recoveries and responses time were also studied and all the sensors based on Ppy polymer responded very fast as compare to PVP based sensors, they responded just in one second and the recovery time was between 3 and 5 s. The PVP based sensors found slower to respond and recover as compare to the Ppy based sensors.

Keywords Hydrostatic pressure sensor · MXene · Carbon nanoparticle · Polymer composites · Recovery and response time

1 Introduction

Pressure sensors have been intensively investigated over the years and used in several areas such as to monitor and control the pressure of a confined space [1–3], bio-medical applications [4, 5], and robotic arms [6, 7]. They are also used to monitor flow [2] and level of liquid inside reservoirs [3]. Among many areas of application, one area that has attracted many researchers for the use of pressure sensors is robotics arm [8]. Consequently, these are mechatronic machines, which are designed to perform repetitive and dangerous tasks.

There has been an evolution in the sensor technology where various pressure sensors with different working principles have emerged and extensively studied such as

piezoelectric, capacitive, resistive [9]. These three prevalent pressures sensing mechanisms have been applied in different applications, with their limitations, for example, the piezoelectric sensing technology is incapable of measuring static and slow variations upon pressure compressions [10, 11], Capacitive touchscreens are prone to surface moisture or other screen contaminants which affect the performance of the sensor [12].

However, piezoelectric sensing technology has been the most preferable mechanism of the three. The sensing materials have a piezoelectric effect and produce an electric charge when pressure is applied [13]. Interestingly, they have been used in a wide sphere, with pressure measurements exceeding 10^8 kPa has been achieved. In capacitive sensors, however, in the sensing mechanism, two parallel-plates is

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involved where the other one is a diaphragm (sensing element), and upon the structural or deflection changes on the active material; the capacitance changes as a function of the applied pressure [14]. This technology is popular in consumer electronics applications, it is currently the leading market in the commercial sector than other pressure-sensitive technologies [15]. The advantages are they possess high spatial resolution than their counterpart (resistive touchscreens), with low activation requirements and rigidity. Consequently, these attributes make them the most used and mark their dominance in touchscreen technology for smartphones, tablets and trackpads [16].

The resistive sensing mechanism has been widely utilized these days mainly because of the signal processing and collecting is easy as compare to other types of mechanisms, the sensor architecture is not complicated as others, and easy fabrication methods [17]. Resistive pressure sensors typically respond to an applied pressure by either decrease or increase in the contact resistance [18].

Recently, a new family of 2D nanomaterials, MXene discovered in 2011 [19] and has been explored due to its remarkable properties. The material has ultrahigh volumetric capacitances of up to 900 F cm^{-3} for energy storage [20, 21], except gradual weight loss in TG curve observed to about 500 K, otherwise the material was fairly thermally stable [22] and the composite if the MXene has demonstrate excellent mechanical stability with out losing much of its electrical conductivity [23, 24]. MXenes have diverse applications, which makes them potential carriers of electron charge, electronic material, optical devices, sensors and energy storage devices. This is mostly driven by high metallic conductivity and charge carrier mobility as well as ease of functionalization. Their electrical properties make them exhibit two states alternating between metallic and semiconductor, upon functionalization [24–26]. MXene is a family of early transition metal carbide and or carbonitrides, and it is made up of 3D precursor MAX phase powder [27], where M is transition metal, A is mainly from group 13 and 14 of the periodic table, and X represents carbon or nitrogen [28].

In this study, we report on the application of functionalised Ti_3AlC_2 -carbon nanoparticle-polymer composites in hydrostatic pressure sensors, and we investigated the response and recovery time of the fabricated sensors.

2 Experimental

All chemicals, except the carbon nanoparticles, were obtained from Sigma–Aldrich and used without further purification. Titanium aluminium carbide was purchased from China (Luoyang Tongrun Info Technology CO., LTD.), and the sample was characterized using XRD to confirm the purity before use.

2.1 Synthesis of carbon nanoparticles

CNPs were prepared from the combustion of the candle [29] purchased at a local supermarket, Johannesburg, South Africa, and yielded black carbon soot, collected and used after purification with acetone using a centrifuge for several times.

2.2 Synthesis of polypyrrole (ppy)

The Polypyrrole was synthesised by a chemical polymerization method following a procedure reported elsewhere [30]. A 1 M Pyrrole solution was prepared in deionized water and then the oxidizing agent, FeCl_3 , the calculated mole ratio of 1:2.4 (monomer: oxidant), slowly added under constant stirring for 30 min in an ice bath. Then the reaction mixture was then allowed to stir for 4 h. Then after the mixture was kept unagitated for 24 h so that Ppy powder settled down. The Polypyrrole powder was filtered out under vacuum and washed with deionized water several times to remove any impurities present. Then finally the Polypyrrole was dried for 2 days at room temperature.

2.3 Functionalization of Ti_3AlC_2

The synthesis of $\text{Ti}_3\text{C}_2\text{T}_x$ was performed by > 40% of HF, then 1.0 g of Ti_3AlC_2 was added gradually to 10 mL HF in Teflon beaker and the mixture was stirring in a fume hood for 24 h at room temperature [19]. Then 50 mL of deionized water was added to the reaction mixture. The mixture was then washed with deionised water, ethanol until the pH of supernatant reached 6.5 with great care of safety using a centrifuge at 6000 rpm. The greyish-like sediment was dried at room temperature for 3 days. The product was ground into a fine powder with a mortar and pestle and was kept in the sample vial until further analysis and application in the study.

2.4 Polymer nanocomposite preparation

The fabrication and electrical measurement methods of all the pressure sensors were followed by early reports [31]. Three mass loadings of f-MC/CNPs/Ppy or PVP nanocomposites with 0.5:1:3, 1:1:3 and 1.5:1:3 mass ratios in 10 mL in dichloromethane (DCM), as a solvent, were prepared along with any two combinations of the two sensing materials. The mixtures were sonicated for 10 min and then allow to stir for 48 h. The prepared polymer nanocomposites (total of 35 μL) were dropped cast onto interdigitated (IDE) gold (Au) electrodes (composed of 0.1 mm width and 0.1 mm gap between metal lines of 18 pairs of 7.9 mm length) using 10–100 μL micropipette. The depositions were done

sequentially (in three portions, 10 μL + 10 μL + 15 μL) for a period of 45 min in between the drops to allow the nanocomposites to form a thin film and dried under vacuum for 48 h before pressure applications.

2.5 Electrical characterization

The electrical characterization of the sensors was conducted by using ISO465TECH LCR meter (LCR 831, 20 Hz–200 kHz) at 500 mV amplitude of AC-signal and at 25 kHz frequency was set to as an input signal and R (resistance) was measured as the output signals. All electrical measurements were performed under at room temperature in all cases. The pressure dependence response measurements were made by placing the sensor in a cylindrical tube in which the pressure was varied manually with the aid of a piston following the relationship $h_i p_i = h_f p_f$ (h = the height, p = the pressure, and i and f denote initial and final, respectively). The pressure in the cylindrical tube was varied by displacing the piston up and down to the desired position by applying an external force. (see Ref. [31] for details of the pressure control experimental setup).

3 Results and discussion

3.1 Characterisation of the synthesis materials

3.1.1 Powder X-ray diffraction analysis

The HF etched Ti_3AlC_2 was characterised with using XRD technique. Figure 1a, b, show the diffraction patterns of the pristine Ti_3AlC_2 and functionalised- Ti_3AlC_2 (f-MC). It is clearly visible that the characteristic peaks of Ti_3AlC_2 become weaker and broader after functionalisation with HF, this indicates a loss of crystallinity [32, 33]. For

instance, the (002) diffraction peak of pristine Ti_3AlC_2 at 9.8° which corresponds to the basal planes for the 2D however after etching the $\text{Ti}_3\text{C}_2\text{X}$ layers became broad and shifted to lower angles, 7.2° this indicates that an increase of c lattice parameter due to the expansion of Ti_3C_2 layers along [002] [19, 34] and evidence for larger c spacing [21, 35]. This is maybe due to the intercalation of water [20]. Furthermore, the strongest peak at 39.1° which corresponds to the (104) peak of Ti_3AlC_2 diminished after etching with HF, this is an evidence of the removal of Al from the Ti_3AlC_2 structure was successful [33].

The synthesised CNPs were also characterised using P-XRD (see Fig. 2a). The powder XRD pattern shows two distinctive and prominent diffraction peaks of high and low intensity at $2\theta = 24.5^\circ$ and 43.7° . The identified peak at $2\theta = 24.5^\circ$ indeed shows that CNPs are mostly amorphous materials, however, they also exhibit traits of the graphitic layer, which is consistent with the TEM images obtained and reveal layer-like connectivity with more in-depth analysis, (see Fig. 3a, b). Hence, the peak $2\theta = 43.7^\circ$ confirms the graphitic nature of the CNPs. Similar observations were reported [36, 37].

Figure 2b shows the P-XRD diffraction pattern the synthesised polypyrrole, a broad diffraction peak suggests that the material prepared using ferric chloride (Fe_2Cl_3) is amorphous in nature and it has been reported that the nature and the broadness of the diffraction peak are due to the order in which the chains are connected [38].

3.2 Transmission electron microscopy (TEM) analysis

The image analysis was done using TEM microscopy. Figure 3 shows the CNPs are agglomerated, with an average diameter determined to be 30–40 nm (see Fig. 3c) The presence of graphitic characteristics of the CNPs is

Fig. 1 Powder XRD pattern of **a** pristine Ti_3AlC_2 and **b** exfoliated Ti_3AlC_2 (f-MC) using HF at room temperature

Fig. 2 Powder XRD pattern of **a** candle-soot derived CNPs, **b** polypyrrole

Fig. 3 TEM image of CNPs **a** at low and **b** high magnifications; **c** the particle size distribution

consistent with the result from Powder X-ray diffractions, similar results were reported [39]. Moreover, agglomeration of nanoparticles between adjacent nanoparticles because of van der Waals interaction.

3.3 Scanning electron microscopy (SEM) analysis

The surface morphological properties of carbon nanoparticles shown in Fig. 4a. it shows that the surface is rough, and it has been reported rough surface does improve the electro-conductivity [40, 41], which is essential in the

application for pressure sensors. Figure 4b, shows the surface morphology of polypyrrole, it is clearly visible that the non-uniform surface of the sample from the SEM image. This may arise from the chains in the ring-structure with the adjacent carbon complimenting each other by carbon-carbon interaction [42]. Furthermore, the SEM-EDX spectrum shown (Fig. 5) the elemental composition of the CNPs samples comprises mostly of carbon (91%) and oxygen (9%).

Scanning Electron Microscopy images of metal carbide are shown in Fig. 6a, b. Figure 6a shows the SEM image

Fig. 4 Show SEM micrographs of **a** CNPs and **b** Ppy

Fig. 5 Show SEM-EDX of the as-synthesized CNPs

of Ti_3AlC_2 of different morphology (rough surface) and grain-like shape and Fig. 6b shows a typical SEM image of multi-layered $\text{Ti}_3\text{C}_2\text{X}$ (f-MC), after exfoliation with aqueous HF (48%) for 24 h. The prolonged reaction time might have resulted in the stacking of the functionalised Ti_3AlC_2 layers, which corresponds with the XRD pattern shown in Fig. 1b. The SEM-EDX of both the pristine and exfoliated metal carbide Fig. 6c, d, respectively indicate that exfoliation was achieved, and it is quite evident from the drop in the percentage of Al from 11.5 to 0.8%, for pristine and exfoliated, respectively, this supports the finding in P-XRD (Fig. 1).

3.4 Raman spectroscopy analysis

Figure 7 shows the Raman shift of the pristine and functionalised Al_3AlC_2 . Two broad and dominate peaks between 1000 and 1700 cm^{-1} emerged after functionalisation. Those two Raman peaks are characteristic for the D- and G-modes of graphitic carbon and are D-band is attributed to the amorphous carbon or deformation vibrations of a hexagonal ring, whereas G-band is assigned to the stacking of the graphite hexagon network plane

[43]. Those two peaks appeared in very weak intensity or absent (see Fig. 7a). The increase in intensities of D- and G bands after etching in the Raman spectra indicate proof of disordered carbon formation [44–46] which is consistent with the P-XRD and SEM-EDX results.

Raman spectrum of CNPs was investigated as well, Fig. 7b shows the spectrum of the candle-soot derived carbon nanoparticles with the D band at 1340 cm^{-1} and G band at 1585 cm^{-1} and the relative ratio intensities were at 0.85. Hence, the value of the ratio (I_D/I_G) is relatively close to 1, implying the distorted graphitic character of the carbon material that is more sp^3 hybridized carbon than sp^2 hybridized carbon. This result is consistent with the SEM-EDX findings (see Fig. 5) that indeed the distortion might be due to the oxygen functional groups.

3.5 Pressure sensor performance

The pressure response measurements for all devices, relative resistance ($\Delta R/R$) versus time were obtained by applying a down or an upward force to the pump enclosed in a cylindrical glass, to create positive air pressure or vacuum inside the system. The applied forces either upward or downward were repeated with different compression factors from low pressure 65 (vacuum) to higher pressure 168 kPa. Moreover, the applied air pressure was held for 4 s (contact time) and released back to the atmospheric pressure and allowed for baseline recovery time about 60 s (stability time).

Three mass loadings of f-MC/CNPs/Ppy or PVP nanocomposites with 0.5:1:3, 1:1:3 and 1.5:1:3 mass ratios were prepared. After the fabricated hydrostatic pressure sensors, the sensors were exposed to the pulses of pressure (65–168 kPa), only the sensors with 1.5:1:3 mass ratio of f-MC/CNPs/Ppy or PVP responded with high

Fig. 6 SEM images of **a** P-MC and **b** f-MC after reacted with HF at room temperature for 24 h, Energy Dispersive X-ray Spectroscopy (EDX) for **c** P-MC and **d** f-MC

Fig. 7 Raman spectra of **a** (1) pristine MC and (2) f-MC; **b** synthesised CNPs

signal-to-noise (S/N) ratio, however, the remaining two composites, unfortunately, gave us a low signal-to-noise ratio for both polymer composites.

After identifying the responsive sensor compositions, the 1.5:1:3 mass ratio, then we prepared additional sensors by combining any two of the sensing materials, f-MC/CNPs 1.5:1 mass ratio, f-MC/Ppy 1:2 mass ratio and CNPs/Ppy

1:3 mass ratio and their electrical response were investigated. A similar procedure was followed for the PVP based composites.

All the response results of the pressure sensors were shown in Fig. 8a–h. All the fabricated pressure sensors were responded well in different response region. Out of all the prepared sensors, only f-MC/CNPs based sensors did

Fig. 8 The relative resistance response variation during applied pressure pulses for **a** f-MC/CNPs/Ppy, **c** f-MC/CNPs, **e** CNPs/Ppy and **g** f-MC/Ppy; the relative electrical resistance as a function of applied pressure for **b** f-MC/CNPs/Ppy, **d** f-MC/CNPs, **f** CNPs/Ppy and **h** f-MC/Ppy

Fig. 9 The relative resistance response variation during applied pressure pulses for **a** f-MC/CNPs/PVP, **c** CNPs/PVP; the relative electrical resistance as a function of applied pressure for **b** f-MC/CNPs/PVP, **d** CNPs/PVP

show a linear relationship between the applied pressure (from 65 to 168 kPa) and relative response in resistance (See Fig. 8b, c), however, f-MC/CNPs/Ppy did show a linear relationship between applied pressure (only in positive applied pressure) and the relative response (see Fig. 8a, b). Similarly, in the case of the f-MC/Ppy, however, although a nonlinear relationship between the applied pressure and relative response was observed in positive pressure range (108–168 kPa) but in a narrow range, from 108 to 126 kPa, displayed a linear relationship between the applied pressure and relative response (See Fig. 8e, f, inset). Generally, the two sensors, f-MC/Ppy and CNP/Ppy had shown little or nonresponsive under low-pressure (65–89 kPa) range (see Fig. 8e, h).

The second set of sensors were PVP polymer-based sensors with the same mass ratios and experimental conditions as Ppy based sensors. Accordingly, f-MC/CNPs/PVP, f-MC/PVP and CNPs/PVP based sensors were prepared. The relative response of the three sensors was investigated, except the f-MC/PVP based sensor which displayed low signal-to-noise ratio (not shown here). Generally, both sensors responded over a wide range, from 65 to 168 kPa, however, both displayed a linear relationship between the applied pressure and response in narrow ranges, f-MC/

CNPs/PVP from 89 to 151 kPa, while CNPs/PVP from 72 to 126 kPa (see Fig. 9b, d, inset).

3.6 Sensitivity measurements

The sensitivity of the fabricated sensors shown in Table 1. The sensitivity was calculated the tangent of the curve applied pressure against the relative change in response. Accordingly, the sensitivity of the sensor to the applied pressure, for the f-MC/CNPs based sensor was $1.7 \times 10^{-4} \text{ kPa}^{-1}$

Table 1 Coefficients of a_R (sensitivity) for fabricated pressure sensors

Composites	Resistive sensitivity (a_R) (kPa^{-1})	Pressure Range (kPa)
f-MC/CNPs/Ppy	0.00017	108–168
f-MC/Ppy	0.00232	126–168
CNPs/Ppy	0.00004	108–136
f-MC/CNPs	0.00017	65–168
f-MC/CNPs/PVP	0.0012	89–151
CNPs/PVP	0.0011	71–126
f-MC/PVP	None	

in the range of 65–168 kPa, however, the sensitivity in a narrow range from 108 to 168 kPa for the same sensor was $3.3 \times 10^{-5} \text{ kPa}^{-1}$ see inset Fig. 7d. The f-MC/CNPs/Ppy based sensor gave us $1.7 \times 10^{-4} \text{ kPa}^{-1}$ and finally, the sensitivity for the CNPs/Ppy based sensor was $4 \times 10^{-4} \text{ kPa}^{-1}$ to the applied pressure in the range of 108–168 kPa. Interestingly, sensor-based on f-MC/Ppy showed excellent sensitivity in a very narrow range, 126–168 kPa, $2.32 \times 10^{-3} \text{ kPa}^{-1}$, which is 10 times more sensitive than f-MC/CNPs/Ppy and CNPs/Ppy based sensors and 100 times more sensitive than f-MC/CNPs based sensor. The comparative sensitivity of the three sensors in a narrow range (108–168 kPa), the f-MC/CNPs/Ppy and CNPs/Ppy based sensors are more sensitive than the f-MC/CNPs based sensor. However, the f-MC/CNPs based sensor has shown better sensitivity in a wide range of the applied pressure, in both under vacuum and positive, while the f-MC/CNPs/Ppy and CNPs/Ppy based sensors shown only better sensitivity under positive applied pressure from 108 to 168 kPa. The sensitivity of both PVP based sensors showed fairly similar which was close to $1.1 \times 10^{-3} \text{ kPa}^{-1}$ in their respective range. Generally, our sensors are best performed at higher pressure as compared to the reported pressure sensors which are flexible and sensitive at low pressure (See Table 2).

$\alpha_R \equiv \frac{\partial(\Delta R/R_0)}{\partial p}$ is corresponds to the specific range of pressure variations.

3.7 Response and recovery time

The response and recovery time for the pressure sensors were measured for all sensors at 127 kPa applied pressure (see Table 3). The response time was considered as the time needed to achieve 90% of the maximum response under the applied pressure, whereas the recovery time as the time required to recover 90% of the maximum response after the applied pressure released (see Fig. 10). All the sensors based on Ppy polymer responded very fast, just in one second and the recovery time was between 3 and 5 s. The fastest responded sensors were f-MC/CNPs/Ppy and CNPs/Ppy which was just one second to reach 90% of the maximum

Table 3 Response and Recovery time for the fabricated pressure sensors

Composites	Response time (s)	Recovery time (s)
f-MC/CNPs/Ppy	1.0	5.0
CNPs/Ppy	1.0	4.0
f-MC/CNPs	1.5	3.0
f-MC/CNPs/PVP	3.0	4.0
CNPs/PVP	2.0	5.0
f-MC/PVP	No response	No response

Fig. 10 Response and recovery time of the f-MC/CNPs/Ppy under positive pressure application at 127 kPa

response and the fastest to recover was f-MC/CNPs it took just 3 s to recover the 90% of the maximum responded. The slowest to recover was the f-MC/CNPs/Ppy based sensor which took about 5 s. However, sensors based on PVP polymer were the slowest to respond and recover, f-MC/CNPs/PVP based sensor took 3 s to reach 90% of the maximum response and 4 s to recover 90% of the response, the CNPs/PVP based sensors shown quicker response, just 3 s to reach 90% of the maximum response and took 5 s to recover, which is the slowest sensor to recover.

Table 2 A summary of the reported performance as compare to our fabricated sensors

Active materials	Perception mechanism	Flexibility	Sensitivity (kPa^{-1})	Minimum detection reported	Maximum detection reported (kPa)	References
ZnO nanorod—CNTs/PDMS	Piezoelectricity	Yes	—	3.5 kPa	31.5	[13]
PDMS microstructure OFET	Capacitance	Yes	0.55	3 Pa	20	[14]
CNTs/PDMS interlocked micro-dome	Piezoresistivity	Yes	15.1	0.2 Pa	59	[18]
f-MC/Ppy	Resistive	No	0.0023	89 kPa	168	This work
f-MC/CNPs/PVP	Resistive	No	0.0012	89 kPa	168	This work

4 Conclusion

After fictionalised the Ti_3AlC_2 and form a polymer composite with carbon nanoparticles, different types of pressure sensors were prepared by varying the sensing materials in the composites. Generally, the fabricated sensors, based on Ppy and PVP polymer, have shown a linear relationship in their respective ranges. The results indicated that all sensors responded differently in different ranges. The advantage of this finding is by simply changing the composition of the sensing materials, it possible to change the sensitivity as well as response region which might have excellent advantages during the practical application, for a specific range of applied pressure. In terms of the recovery and response time, all the sensors based on Ppy polymer responded faster as compare to PVP based sensors, they responded just in one second and the recovery time was between 3 and 5 s. The PVP based sensors found slower to respond and recover as compare to the Ppy based sensors.

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Compliance with ethical standards

Conflict of interest The authors declare no competing financial interest.

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