INTRODUCTION

With the successful isolation of graphene in the year 2004 (Novoselov et al., 2004), 2D materials have gained significant attention for research owing to their novel properties, enabling a broad applications ranging over semiconductors, (Cao, Kang, Sarkar, Liu, & Banerjee, 2015) flexible/printed nanotechnology (Kim, Choi, Lee, Kim, & Hong, 2015), optoelectronics, (Mak & Shan, 2016) sensors (Selamneni, Barya, Deshpande, & Sahatiya, 2019), health care (Jayakumar, Surendranath, & PV, 2018; Veeralingam, Sahatiya, & Badhulika, 2019) and energy storage. (Anasori, Lukatskaya, & Gogotsi, 2017) One of the greatest challenges has been its zero bandgap limiting its use in applications requiring suitable bandgaps. Transition metal dichalcogenides (TMDs) with their sizeable bandgap do overcome this drawback but their low mobility of less than ~200 cm²/V·s hinders their applications in high-frequency electronics. (Liu et al., 2014; Splendiani et al., 2010; Wang, Kalantar-Zadeh, Kis, Coleman, & Strano, 2012) Hence, BP is being researched as a viable alternative with its high electron mobility and a sizeable direct bandgap.

Black phosphorous possesses high carrier mobility of ~1,000 cm²/V·s and a direct bandgap that can be modulated (0.3–1.5 eV) based on the number of layers stacked on top of each other (Liu et al., 2014; Xu, Shi, Shi, Zhang, & Zhang, 2019). It has a puckered

Abstract

This report demonstrates synthesis of black phosphorus (BP) from red phosphorus using sonochemistry and the direct growth of highly air-stable BP on cellulose paper using hydrothermal method with PDMS as passivation and its utilization in human motion monitoring system. PDMS not only hinders the oxidation of BP but also provides flexibility to the device, thereby allowing the use of paper-based device as strain sensor. Fabricated device exhibits gauge factor of 6.1, which is comparable with the devices fabricated using sophisticated cleanroom techniques, and device is tested for 2,000 bending cycles revealing excellent reliability and repeatability. Device displayed excellent hysteresis suggesting that tunnelling effect is responsible for the current change of the device upon the external strain. Mathematical model is formulated to describe tunnelling conduction which matched the experimental results. Further, device exhibits excellent air stability for 98 days wherein negligible change in resistance and strain sensing performance is observed. Fabricated device is integrated onto different parts of the human body for its real-time application in human motion monitoring. Successful demonstration of the direct growth of highly air-stable BP on flexible and biodegradable cellulose paper opens up new avenues of research in optoelectronics, health care, security etc.

KEYWORDS

Black phosphorous, human motion monitoring, hydrothermal, papertronics, sonochemistry
honeycomb lattice which can be visualized as rows of orthogonally coupled hinges along the zigzag direction. This makes the structure of BP soft, yet, mechanically strong that can withstand large strains (Jiang & Park, 2014). The P atoms are sp (Kim et al., 2015) hybridized, and phosphorene layers are held together by weak van der Waals forces. Lack of dangling bonds on the surface of BP makes it ideal in withstanding large localized deformations without breaking (Papageorgiou et al., 2004). It has been demonstrated that the application of ~1.2 GPa can close the bandgap which can raise the conductance by one order of magnitude (Jiang & Park, 2014). The above results have hinted that BP can be utilized as a potential candidate for the development of piezoresistive flexible strain sensors.

There have been several reports of utilization of BP in RAM applications, field-effect transistors, sensors and energy devices (Castellanos-Gomez, 2015; Du, Liu, Deng, & Ye, 2014; Gui, Jin, Wang, & Li, 2018; Guo, Zhu, Song, Wang, & Yan, 2019; Hao et al., 2016; Koenig, Doganov, Schmidt, Castro Neto, & Özyilmaz, 2014; Lee et al., 2016; Li et al., 2014; Lin, Li, Qian, & Lau, 2019; Zhou et al., 2017). Despite the advancements in its applications, the direct growth of BP on flexible substrates still remains a challenge. The issue is intensified by the ability of BP to oxidize when exposed to air (Du et al., 2014). BP has been synthesized using various methods which include chemical and mechanical exfoliation and chemical vapour deposition (CVD), which are not only low throughput processes but also require a lot of post-processing for the full development of the device. (Wu, Hui, & Hui, 2018) These methods cannot be used for the synthesis of the BP on flexible substrates due to the temperature and pressure requirements are higher than the critical values of the flexible substrates. Hence, there is a need to develop a process for the facile fabrication method for the direct growth of BP on flexible substrates. However, BP is unstable in ambient conditions and gradually degrades. In dealing with the instability of BP under ambient conditions, different passivation techniques have been developed. Deposition of materials like HfO2, SiO2 and Al2O3 (Sahatiya, Jones, Mattela, & Badhulika, 2019; Wan et al., 2015; Xing et al., 2019) among others on the surface of the BP flakes has successfully isolated them from contact with air. Some of these materials have proven to enhance BP’s electronic properties, while some hinder the same (Wood et al., 2014).

Over last few years, the research on micro-gesture sensor monitoring has been tremendously increased because of their applications in biomedical field smart houses, automobiles, wearable devices, etc (Lorussi, Scilingo, Tesconi, Tognetti, & De Rossi, 2005). Gesture monitoring is the process of recognizing the movements of human’s fingers, arms, hands and head movement, and it significantly eases the human–machine interaction. Among hand gestures, microhand gestures have great potential for many applications like portable, wearable mobile devices (Sang, Shi, & Liu, 2018). Various techniques have been developed for microhand gesture identification, which are related to computer vision, utilize RGB cameras (Chen, Fu, & Huang, 2003), infrared cameras (Suarez & Murphy, 2012), etc. However, the reliability of this sensor is an issue in night-time or under direct sunlight because of the variance of the light conditions. Hence, there is a need to develop a gesture sensor which is reliable, cost-effective and biodegradable. In this report, we demonstrate the direct growth of BP on cellulose paper using sonochemistry and hydrothermal method which is flexible, cost-effective and biodegradable. The obtained device was later passivated using layers of cured PDMS which not only assists in increasing the flexibility(Sahatiya & Badhulika, 2017) of the device but also provides excellent air stability. The gauge factor measured for the fabricated device was 6.1, and 2000 bending cycles revealed excellent reliability, and it is still comparable to the some of the reported strain sensors. (Huang, Pascal, Kim, & Goddard, 2011) Further, negligible change in the resistance of the device and also the strain sensing performance was observed for 98 days suggesting a highly air-stable nature of the fabricated device. As per the author’s knowledge, this report is the first demonstration of direct growth of highly air-stabilized BP on cellulose paper and its utilization as a strain sensor.

2 | RESULTS AND DISCUSSION

The process of conversion of red phosphorus to BP and its subsequent hydrothermal growth on cellulose paper followed by encapsulation using PDMS is illustrated in Figure 1. Details regarding the synthesis of BP, fabrication and encapsulation of the device can be found in experimental section. Different characterization techniques were employed to identify and confirm the surface composition and attributes of the device. To confirm the growth of BP on cellulose paper and also the oxidation states of BP, X-ray photoelectron spectroscopy (XPS) analysis was performed. As shown in Figure 2a, deconvolution of P 2p peak results in doublet corresponding to 2p1/2 and 2p3/2 which can be attributed to the as-grown BP (Papageorgiou et al., 2004). The broad peak at 133.22 eV suggests the oxidation of BP, thereby forming P-O and P = O bonding states. It should be noted that the BP was taken in pristine form while performing XPS which leads to the oxidation peaks. Crystal structure of the as-synthesized BP was studied using X-ray diffraction (XRD) analysis. As shown in Figure 2b, the 2θ range of the XRD was 10° to 9O°. The peaks at 16° and 22° are due to the cellulose paper substrate. The peak present at 34° is corresponding to (040) plane of BP, match with the Joint Committee on Powder Diffraction Standards (JCPDS) card no: 76–1967.

To study the morphology of the as-grown BP on cellulose paper, field emission scanning electron microscopy (FESEM) analysis was performed. Figure 2c shows the low magnification FESEM image wherein the growth of BP on cellulose paper and the microfiber morphology of the cellulose paper is visible suggesting that the growth of BP does not affect the cellulose paper substrate. Figure 2d shows the high magnification FESEM image wherein a connected structure of nanoparticles is visible.

Figure 3a provides a top and cross-sectional view of the schematic for the enclosure of BP grown on paper with PDMS. Keithley 2,450 SourceMeter was used for the electrical characterization of the fabricated device at ambient conditions. Figure 3b, c shows the
I-V characteristics of the fabricated device under different compressive, tensile strains, respectively, and device exhibited ohmic characteristics. The device displayed an increase in the sensor current with the application of strain. Both compressive (inward bend) and tensile strain (outward bend) record an increase in the sensor current. The degree of strain applied displayed proportional changes in current. Reason for the increment in the current for both strains can be attributed to the tunnelling resistance and ohmic flow between the BP nanosheets. When the compressive strain is applied (bend inwards), the BP nanoparticles overlap with each other, and hence, the tunnelling effect reduces and the ohmic flow dominates. When the tensile strain is applied, the BP nanoparticles move away from each other, and hence, the ohmic flow of electrons cannot happen; therefore, during tensile strain, only tunnelling effect dominates and the current increases. It should also be noted that BP was directly grown on cellulose paper, and because of the porous nature of the cellulose paper, the growth of BP occurs on both side of the cellulose paper. So, compressive strain on one side will give rise to tensile strain on the other side. Under tensile strain, the other part of the cellulose paper still experiences compressive strain, and hence, on the other side of the BP/cellulose paper, ohmic conduction prevails, and because of the interconnected BP nanoparticles, there is a creation of a conductive pathway for the electrons to flow and reach the contact. Figure 3d shows the temporal response of the fabricated device under increasing dynamic strain where an increase in the sensor current was observed upon the increasing strain. Figure 3e shows the sensitivity graph wherein a linear fit between the changes in the resistance with applied compressive strain calculates the gauge factor and was found to be 6.1. Data from three independent devices were recorded, and similar results were obtained suggesting the repeatability of the reported process. Responses of the device to variant strain applications were recorded to test robustness.
Figure 3f shows the temporal response of the fabricated device under compressive strain of ~1.0%, wherein increment in the sensor current was observed. Figure 3g shows the temporal response for 2000 bending cycles wherein the negligible change in the sensor performance was observed suggesting the highly robust nature of the fabricated device.

The primary role of PDMS is to prevent the oxidation of the BP, present on the surface of cellulose paper with air. The enclosure of the device in PDMS also provides a sturdy and noise-free set-up to carry out strain measurements. PDMS plays the additional role of a flexible construct aiding the strain response of the device. The interface between BP and the cellulose paper plays a vital role in exercising strain sensitivity for the device. The firm binding of the BP to the cellulose microfiber substrate accounts for the negative resistance coefficient of the device. The thickness of the PDMS film used for all the strain measurements was 1.5 mm. However, to observe the effect of the PDMS thickness, PDMS of varying thickness (0.5, 1.5 and 3 mm) was used. Interestingly, all the three devices displayed sufficient protection to the BP/cellulose paper device. But when the external strain was applied to a device where the PDMS film thickness is ≤0.5 mm, there were cracks developed in the PDMS film which makes it highly unstable for strain sensing measurements. Figure S1 shows the device performance encapsulated using different PDMS thickness films. On the application of strain, a change in the RC network prevalent among the BP nanoparticles and cellulose paper leads to a change in the observed resistance. The applied strain brings the connected structure of BP nanoparticles closer, leading to a more conductive pathway for the flow of electrons. The conductivity in the lesser strain margins can be attributed to the tunnelling conduction among the BP nanoparticles, which are placed relatively far off compared to when strain is applied. The change in nanoscopic distances among the BP nanoparticles supported by the theory of tunnelling can be used to explain the observed trend in the measured resistive properties. In the initial state, the tunnelling effect seems to prevail over the ohmic flow, while in the bent state both the factors contribute to an increase in the conductivity of the device. Most of the pressure sensors report a rupture or crack in the active material, thereby creating modulation in conductance, but, in such cases, the hysteresis of the device is poor; that is, the device resistance does not return to the initial value which affects the device reliability. In this case, excellent hysteresis was observed as seen in Figure 3h which can be attributed to the absence of rupture of BP nanoparticles on cellulose paper. Figure 3i shows the plot of the measured resistance change $\Delta R/R_0$ to the applied compressive strain. The evident decrease in slope indicates that the change in resistance saturates with applied strain. Few-layer BP on cellulose paper being a moderately conductive material, the device associates its conductivity significantly to the phenomenon of electron tunnelling. With an increase in compressive strain applied, the intermolecular distance of the BP nanoparticles reaches a saturation low. Hence, further increase in strain contributes very little to the change in the intermolecular distance and hence the change in resistance. We propose a mathematical model to explain the experimental results. The tunnelling resistance ($R_{\text{tun}}$) can be calculated as in equation (Zhang, Pan, Zheng, & Yi, 2000).

$$R_{\text{tun}} = \left( \frac{e}{N} \right) \left( \frac{8 \pi h d}{3 \beta a^2 e^2} \right) \exp \left( \frac{\beta d}{h} \right)$$

where $N$ is the number of conducting paths, $l$ is the number of particles forming a single conducting path, $a^2$ is the effective cross section, $e$ is the electron charge, $h$ is the Plank constant, $d$ is the smallest distance between conductive particles, $m$ is the electron mass, and $\lambda$ is the potential barrier height between the adjacent BP nanoparticles.
As modelled by Lin et al. (2016) a similar approach was undertaken to model tunnelling conduction. On the application of compressive strain on the device, assuming that the separation reduces linearly from $d_0$ to $d$ and the number of conductive paths increases from $N_0$ to $N$, the following Equation 2–4 have been modelled:

$$d = d_0 (1 - b \epsilon)$$  \hspace{1cm} (2)

$$N = N_0 \exp (A \epsilon + B)$$  \hspace{1cm} (3)

$$\frac{\Delta R}{R_0} = \frac{R_0 - R}{R_0} = 1 - \left( \frac{N_0 d_0}{N d} \right) \exp [\beta (d - d_0)]$$  \hspace{1cm} (4)

where $N_0$ is the initial number of conducting paths; $b$, $A$ and $B$ are constants; $R_0$ is the initial resistance; and $\Delta R$ is the change in the resistance under applied strain.

The substitution of Equations 2 and 3 into 4 yields the following Equation 5.

$$\frac{\Delta R}{R_0} = 1 - (1 - b \epsilon) \exp (A_1 \epsilon - B)$$  \hspace{1cm} (5)

where $A_1 = -A - \beta d_0 b$. 
Appropriate values of $A_1$, $B$ and $b$ were modelled to fit the experimental data ($A_1 = -2.79$, $B = 0.024$ and $b = 1.415 \times 10^{-5}$) and provide a good fitting, supporting the electron tunnelling theory as shown in Figure 3i. Further, the same model was used for tensile strain applied on the fabricated device and the corresponding graph can be found in the Figure S2. The hydrothermal method ensures uniform growth of BP on both the surfaces of the cellulose paper; hence, the same model accounts for the observed conduction on the application of tensile strain as well.

Inspired by the excellent sensitivity and hysteresis, the fabricated device was integrated onto different parts of the human body and the corresponding motions were monitored. Figure 4a–c shows the integration of fabricated device onto a human hand, wrist and neck, respectively, and their corresponding response towards the respective motions. As seen from Figure 4a–c, the fabricated sensor was able to respond to various human motions which finds potential applications in the personal healthcare monitoring, human–machine interaction, gesture communication, etc. When the fabricated device was integrated onto the wrist of an individual, the bending of the finger produces tensile strain. This tensile strain gets developed in PDMS and then gets transferred to BP, thereby increasing the current. Similarly, the fabricated BP-based strain sensor was tested on wrist and neck of an individual and the respective movement produces different current trends. As the movement of the body part will not be always same, change in the current peak pattern of the results was observed as shown in Figure 4. This suggests that the fabricated device is not only capable of detecting the human movement but also has the capability of identifying which part of the body caused the current change.

2.1 | Air stability studies of BP/paper strain sensor

The major drawback of BP is instability in ambient air condition, which is attributed to unstable bonding structure of BP, where lone-pair electrons reside on phosphorus. The degradation process results in a thin insulating oxide layer that can affect the device performance (Abate et al., 2018). The oxidation process can be significantly delayed by isolating samples from ambient environment. PDMS was used for passivation of the fabricated device and to test air stability, resistance of the device under 0% strain was measured for over 3 months. Figure 5a shows the graph of the measured resistance v/s number of days, wherein the 6%–7% decrement in the resistance was observed, suggesting that the fabricated device has been stable even after 98 days. Further, strain sensing performance of the fabricated sensor after 98 days was tested, wherein the response is as shown in Figure 5b, and negligible change in the sensing performance was observed compared to the day 1 response of the fabricated sensors shown in Figure 5c. The reason for this highly air-stable BP device is its proper passivation with PDMS which was performed immediately after the direct growth of BP on cellulose paper. Figure 5d shows the resistance change of the BP on cellulose paper without PDMS encapsulation, and as seen from the graph, the device resistance falls to $\sim 180$ GΩ in 30 hr further suggesting that PDMS encapsulation was responsible for the device excellent air stability.

There are few reports on studying the effect of strain on BP films. Tao et al. studied the mechanical and electrical anisotropy of few-layer BP and measured Young’s modules and maximum breaking strain in their experiments. (Tao et al., 2015) Quereda et al. explained the controlling of BP bang gap through local-strain engineering (Quereda et al., 2016). Zhang et al. (2017) demonstrated the continuous bandgap modulation of few-layer BP by external mechanical strain applied through flexible substrates and observed a large piezoresistive effect in black phosphorus, which enabled to build an ultrasensitive strain sensor. Li, Zhang, Wu, Cao, and Wu (2018) investigated the impact of strain and underlying mechanism of flexible BP ion sensors. Cai, Wan, Wei, and Qin (2016) studied the strength and stability of single walled BP nanotube under
axial compression. In spite of excellent electronic and mechanical properties of BP, fabrication of BP-based strain sensors on flexible substrates remains to be unexplored. There are no reports on direct growth of BP on flexible substrates. In this report, a facile synthesis procedure was developed for the direct growth of BP on cellulose paper and the fabricated device was encapsulated in PDMS; further, fabricated sensor was utilized for real-time applications in human motion monitoring. The fabrication procedure is scalable and can be used for large area synthesis. The overall cost of each sensor was ~$0.35. The successful demonstration of such low-cost, large area, scalable synthesis and fabrication process for BP-based flexible devices is a great step ahead in wearable electronics and has potential applications in optoelectronics, transistors, security, medical, etc.

3 | CONCLUSION

This report is the first demonstration of direct growth of BP on cellulose paper. PDMS is used for the passivation of the synthesized BP on paper; PDMS not only hinders the oxidation of BP but also provides flexibility to the device, thereby allowing the use of cellulose paper-based device as a strain sensor. The fabricated sensor is utilized for human motion monitoring; the gauge factor was calculated to be 6.1, and measurements for 2000 cycles revealed excellent reliability of the fabricated sensor. Experimental hysteresis characteristics suggest that the sensing mechanism is due to the tunnelling resistance. Further, a mathematical model to explain the experimental results through the phenomenon of tunnelling resistance was developed. The sensor was integrated onto human hand, wrist and neck for its real-time application in motion monitoring. The successful demonstration of the BP on cellulose paper using a low-cost approach opens up new avenues of BP-based flexible electronics research.

4 | EXPERIMENTAL SECTION

4.1 | Growth of Black Phosphorus on Cellulose paper

Commercially available red phosphorus was used for the synthesis of BP. Red phosphorous (30 mg) was dispersed in deionized water (200 ml) using magnetic stirring for 1 hr. The resultant solution was kept for ultrasound for 10 min, and it was repeated 10 times which produced a significant yield of bulk BP at the bottom of the beaker. The supernatant with traces of red phosphorous was discarded, and the bulk BP sediment at the bottom of the vessel was first dried at 90°C for 15 min. The obtained bulk BP powder was then dispersed in dimethylformamide (DMF, 20 ml); later, the well-mixed solution was transferred to Teflon-lined autoclave and cellulose paper of size 3 × 3 cm was kept at bottom of the autoclave; hydrothermal reaction was performed at 140°C for 6 hr. The obtained BP-coated paper was dried at 100°C for 15 min to remove any liquid residues. BP-coated cellulose paper was cut into a 1 × 1 cm dimension, and copper tapes were attached at either side of the sample as contacts to make the device, as illustrated in Figure 1b.

4.2 | Encapsulation of the device in PDMS

Poly(dimethyl)siloxane (PDMS) is a two-part polymer (base elastomer and curing agent). The standard mixing ratio for PDMS is 10:1 (10-parts base elastomer and 1-part curing agent). (Ramalingame, Hu, Gerlach, Kanoun, & Shoe, 2017) This ratio gives the desirable mechanical properties and optimum biocompatibility. Both the base elastomer and the curing agent were mixed, and the container was placed in the vacuum desiccator till all air bubbles were extracted in the mixed solution. The container was then taken out, and PDMS was poured onto the prepared mould (2 × 2 cm) and
TABLE 1  Comparison of the fabricated BP/paper strain sensor with reported sensors

| S. No | Material | Substrate | Fabrication process | Functionality | Gauge factor | Biodegradability | Ref. |
|-------|----------|-----------|---------------------|---------------|-------------|------------------|------|
| 1     | ZnO NW/PSNF | Polystyrene | Electrospinning, Hydrothermal | Strain sensor | 116          | No               | Xiao et al. (2011) |
| 2     | Suspended graphene nanoribbon | Si | Mechanical exfoliation | Strain sensor | 1.9          | No               | Huang, Pascal, Kim, Goddard, and Greer (2011) |
| 3     | Monolayer MoS2 | SiO2/Si | Chemical vapour deposition (CVD) | Strain Sensor | 104 ± 26 | No | Zhu et al. (2019) |
| 4     | WS2 | PET | Atomized spray casting method | Strain Sensor | 14 | No | Qi et al. (2017) |
| 5     | Black Phosphorus | Paper | Sonocchemistry, hydrothermal | Strain Sensor | 6.1 | Yes | This Work |

4.3 | Materials and characterization

Red phosphorus and DMF were purchased from Sigma-Aldrich and were used as received. Field emission scanning electron microscopy (FESEM) analysis of the BP grown on cellulose paper was performed using FEI, Apreo LoVac. X-ray diffraction (Rigaku, ULTIMA-IV) was used to obtain the XRD data. The chemical composition of the active material was further confirmed by X-ray photoelectron spectroscopy (XPS), Thermo Scientific K-Alpha XPS system. Keithley 2,450 SourceMeter was used for electrical measurements.

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Lee, H. U., Park, S. Y., Lee, S. C., Choi, S., Seo, S., Kim, H., ... Lee, J. (2016). Black phosphorus (BP) nanodots for potential biomedical cure in the oven for 20 min at 90°C. Cured PDMS was then slowly and carefully peeled off the mould. (Jiang & Park, 2014) Fabricated device was encompassed in between the layers of cured PDMS. The entire process of PDMS curing and device encapsulation is illustrated in Figure 1b.
