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High Performance Two Dimensional Few Layered Copper Doped MoS_2 Nanosheets Based Flexible Piezoelectric Nanogenerator

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Flexible piezoelectric nanogenerators based on two dimensional (2D) nanosheets are pivotal due to their ultrathin thickness, good mechanical strength and super flexibility with high efficiency. In present work, single crystalline copper (Cu) doped 2D MoS_2 nanosheets was grown via low temperature hydrothermal technique. X-ray diffraction (XRD), High resolution- transmission electron microscope (HRTEM) confirmed the formation of the 2H phase of the Cu-doped MoS_2 nanosheets with thickness and length in range of the 5-7 nm and 300-500 nm, respectively. Pristine and Cu-doped 2D MoS_2 nanosheets based flexible nanogenerator were fabricated and piezoelectric output voltage was measured. Flexible Cu-doped MoS_2 nanosheet: PDMS based flexible nanogenerator exhibited output voltage of 15 V even under low pressure, where as pristine MoS_2 nanosheet based nanogenerator exbited low output voltage of 9 V under same force. Dielectric properties of pristine and Cu- doped MoS_2 nanosheets were also investigated and high dielectric constant of about 6750 was obtained. The mechanism of enhanced output voltage was discussed in terms of the large polarisation and high dielectric constant from Cu-doped MoS_2 nanosheets.

Keywords: MoS_2; 2D Materials; Piezoelectric nanogenerator; Nanosheets

1 Introduction

Recently, two dimensional materials (2D) materials have emerged and gained the attention of scientific community because of their versatile and unique combination of extraordinary properties such as high mechanical strength, catalytic behaviour, piezoelectric, magnetic and semiconducting properties[1-3]. Among various well known 2D materials such as graphene, borophene, transition metal oxides, transition metal dichalcogenides (TMDCs), transition metal carbides and black phosphorous have gained a lot of interest due to their unique properties and extensive applications[4-8]. Specially, TMDCs materials exhibits rich physics with wide range of electrical properties, direct/indirect band gap, non centrosymmetric properties and atomic scale thickness which can be used for numerous advanced applications such as optoelectronics, spintronics, low-power electronics and flexible electronics, super capacitor, biosensors, DNA sequencing and body implantable devices[9-11]. Particularly, molybdenum disulfide (MoS_2) is one of the most studied and typical TMDCs due to its robustness, easy synthesis process, and its exceptional combination of properties. Monolayer MoS_2 exhibits dual semiconductor and piezoelectric properties with direct bandgap of 1.8 eV which makes them a highly suitable 2D materials for piezoelectric nanogenerator applications for scavenging mechanical energy from the living environments. Recently, piezoelectric nanogenerator becomes one of the popular technologies for effective harvesting of ambient mechanical energy for powering nanoscale electronic device and self-powered nano systems. Till date, various piezoelectric nanogenerator based on ZnO nanostructures including nanowires, nanorods, nanodisc, nanospring, lead based $Pb(Zr_{0.52}Ti_{0.48})O_3$ (PZT), barium titanate ($BaTiO_3$), PVDF nanostructures have been reported. However, low output performance of the piezoelectric nanogenerator is still serious issue to power the electronic devices and to improve the output voltage from piezoelectric nanogenerator device is still challengeable and desirable. Recently, 2D materials pristine MoS_2, borophene and WS_2 based piezoelectric nanogenerators have been also reported with improved performance[12-16]. Specially, in case of the pristine MoS_2 nanosheets, It was reported that odd

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number of layers of MoS_2 produces oscillating piezoelectric output voltage and current while the even number of layers of MoS_2 does not produces any piezoelectric output due to the centrosymmetric nature and output performance can be further improved by controlling the charge carriers and sulphur defects[17-19]. In pristine MoS_2 nanosheets, significant amount of inert basal plane, the active sites, sulphur and crystalline defects are usually present, which limit their piezoelectric properties and device performance. To improve the properties of the MoS_2 nanosheets, substitution, doping or intercalation of atoms in the layered materials have been reported as one of the most simple and effective techniques. Among various strategies, doping with Cu is an effective method to tune the properties of the 2D MoS_2 nanosheets as energy of adsorption of Cu atom on MoS_2 surface is about 1.3 eV, which signifies the doping of Cu atoms into 2D MoS_2 through substitution of S atoms. Such approach may improve piezoelectricity and thus performance of the MoS_2 nanosheets based piezoelectric nanogenerator device[20-21]. Recently, Pak *et al.* have reported Cu doped MoS_2 nanosheets based FET devices and they reported Cu doping can improve the output characteristics of FET[22]. Thermoelectric performance of Cu doped MoS_2 nanosheets was also studied by Joseph and their team[23]. Basu *et al.* have reported the effect of Cu-doping on MoS_2 nanosheets for hydrogen evolution reaction[24]. However, till date Cu doped MoS_2 nanosheets based flexible piezoelectric nanogenerators device with improved output performance have been not yet reported. Moreover, to synthesis the 2D MoS_2 nanosheets various method have been reported such as CVD, e-beam, physical vapour deposition and hydrothermal method. Among various routes, hydrothermal method is cost effective, simple and low temperature process and with controlling the temperature, time and ratio of the precursors, various morphologies and phase can also be achieved. In present work, hydrothermally synthesised Cu doped 2D MoS_2 nanosheets and fabrication of the flexible piezoelectric nanogenerators are reported for the first time. The as-synthesised sample was characterised by using XRD and HRTEM techniques. Flexible piezoelectric nanogenerator based on Cu-doped MoS_2-polydimethylsiloxane (PDMS) on flexible ITO/PET substrate was fabricated and performance of the device was measured under vertical pressure. High output voltage from Cu-doped MoS_2 nanosheets based piezoelectric nanogenerator was obtained. Dielectric properties were also studied and dielectric constant of Cu doped MoS_2 nanosheets was found to be 6750 and dielectric loss was about1000.

2 Experimental Section

2.1 Synthesis of copper oxide nanoparticles

Azadirachta indica (Neem leaves) were collected and dried for 4 days and then leaves were grinded into powder form. This neem powder is then transferred into the ball mill jar. The powder was ball milled for 12 hrs at 300 rpm condition using high energy planetary ball milling equipment. The resultant ball milled Neems leaves powder and $CuCl_2.2H_2O$ were taken in equal weight and added in 100ml water and then kept for magnetic stirrer at 90 °C until the mixture turns from bluish green colour to dark brown-black colour. The obtained solution is then transferred to quartz crucibles and kept in microprocessor controlled furnace at 400 °C for 10 minutes. Finally, black coloured copper oxide nanoparticles is collected. The obtained CuO nanoparticles was used as doping materials in MoS_2 nanosheets.

2.2 Synthesis of Cu doped MoS_2 nanosheets

The Cu doped MoS_2 nanosheets was prepared in aqueous solution by using Molybdenum trioxide (MoO_3), thiourea (CH_4N_2S) and CuO nanoparticles as precursor materials. In the synthesis process 0.1g MoO_3, 0.26 g thiourea and 0.003 g of CuO (0.3 mmol) were taken in 80 mL distilled water.The solution was kept under magnetic stirring for 1 hour at 200 rpm. The resultant mixture was then transferred to 100 mL Teflon lined stainless steel autoclave. The autoclave was then kept in furnace at 220 °C for 24 h. After, cooling down to room temperature naturally the black powder was obtained. The product was centrifuged, washed by ethanol and distilled water to remove the impurities, adsorbed ion on the surface of nano-material. The final product was then dried at 60 °C overnight.

2.3 Fabrication of flexible Cu doped MoS_2nanosheets: PDMS based nanogenerator device

Flexible Cu-doped MoS_2 nanosheets based piezoelectric nanogenerator device was fabricated by mixing Cu-doped MoS_2 nanosheet with polydimethylsiloxane(PDMS) polymer, which is well known, elastomer with excellent optical, electrical and mechanical properties with non-piezoelectric properties, in volume ratio of the 30:70 %. The mixture was then spin coated on the cleaned indium–tin oxide coated polyethylene terephthalate (ITO/PET)substrate for 60 seconds under 1500 rpm. A separate conducting Cu electrode was used as the top electrode and mechanically integrated with nanocomposite film. It is worth to mentioned that no electrical poling was done before measurement of the output voltage from the device. The detailed synthesis process of sample and device fabrication is shown in schematic diagram of the Fig. 1.

3 Measurements

X-ray diffraction (Rigaku Mini Flex II) with Cu Kα radiation (λ = 1.5401 Å) was used to investigate the crystal structure of pristine and Cu-doped MoS_2 nanosheets. Ball milling was performed using the Retsch, Germany high energy Planetary Ball Mill PM 400. Spin coating was performed using the spin coating systems using the Apex Instruments (Kolkata, India). High-Resolution Transmission Electron Microscopy (HR-TEM) experiments were carried out under an accelerating voltage of 200 kV JEOL-JEM-F200 (at CSIR-AMPRI, Bhopal). Dielectric constant and tangent loss of nanosheets were measured at room temperature using an Agilent E4980A LCR, USA metre in frequency range of 1kHz–2MHz. The Piezoelectric output voltage was measured using mixed domain oscilloscope (Tektronix-MDO3052, USA) and current was measured using Keithley electrometer (6517B).The vertical force on the nanogenerator device was applied through the function generator interfaced with dynamic shaker (K2007E01, USA).

4 Results and Discussion

Figure 2(a) shows the X-Ray diffraction (XRD) pattern of pristine copper oxide nanoparticles grown by green synthesis route using the Azadirachta indica. The formation of pristine copper oxide nanoparticle was confirmed by the X-ray diffraction pattern (Fig. 2(a)). The XRD pattern shows several sharp peaks corresponding to planes (110), (111), (202), (020), (113) and (004) and well matched with JCPDS card No. 892531. The average crystallite of CuO nanoparticles was obtained by Debye Scherrer formula which is shown below:

$$D = \frac{k\lambda}{\beta \cos\theta} \quad \ldots(1)$$

The average crystallite size as calculated from Debye Scherrer formula was estimated to be 24.4 nm[25]. XRD pattern of the few layered Cu-doped MoS_2 nanosheets was also recorded and pattern is shown in the Fig. 2(b). XRD pattern of Cu doped MoS_2 nanosheets exhibited different planes (002),

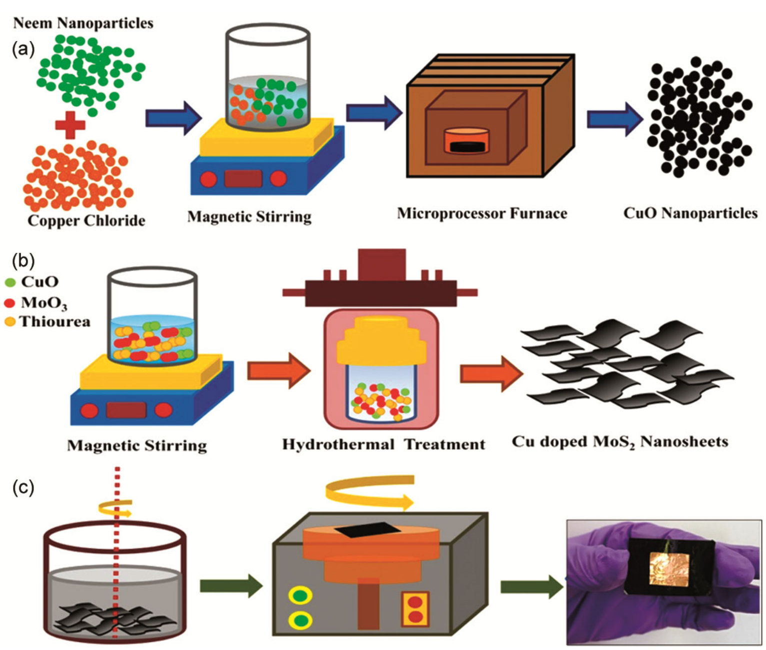

Fig. 1 — (a) Schematic diagram of the synthesis process of copper oxide nanoparticles and (b) Schematic presentation of synthesis process of copper doped MoS_2 nanosheets and (c) fabrication process of flexible piezoelectric nanogenerator based on the Cu-doped MoS_2 nanosheet: PDMS on ITO/PET substrate.

(100), (103), (105), (110) and (201), which corresponds to well known pristine semiconducting and non-centrosymmetric of 2H phase MoS_2 nanosheets (JCPDS :65-1951). The average crystallite size of Cu doped MoS_2 nanosheets was found to be 10 nm along (100) plane[26].

The morphology of the hydrothermal grown Cu doped MoS_2 nanosheets was recorded by HR-TEM technique. Fig. 3 shows the HRTEM images of Cu doped MoS_2 nanosheets at low and high resolution. Fig. 3(a) shows the formation of the aggregated nanosheets like structure of Cu-doped MoS_2 nanosheets at low resolution. At high resolution, as depicted in Fig. 3(b), a layered structure with high lateral area is depicted. Fig. 3(c) shows the 3-5 layers of the nanosheet of Cu doped MoS_2 nanosheets. The HRTEM images exhibits the uniform stacking of layered structure of Cu doped MoS_2 nanosheets. The lateral area and thickness of the single Cu doped MoS_2 nanosheets estimated to be approximately 500 nm and 0.68 nm respectively[27-28].

To fabricate the flexible piezoelectric nanogenerator of as grown 2D Cu-doped MoS_2 nanosheets, non-piezoelectric PDMS is used as polymer matrix. A mixture of MoS_2: PDMS solution was spin coated on ITO/PET substrate. The developed flexible Cu-doped MoS_2 nanosheet based piezoelectric nanogenerator is shown in the Fig. 1(c). The output performance of the device is measured by applying controlled vertical mechanical force through computer controlled dynamic shaker. The piezoelectric output voltage generated from 2D Cu-doped MoS_2 nanosheets based flexible piezoelectric nanogenerator was presented in Fig. 4(a). The Cu-doped MoS_2 nanosheets based nanogenerator device exhibited high output voltage of magnitude 15 V under vertical pressure of the 1kgf. It is worth to mentioned that, no electrical poling was applied on the device to orient the electric dipoles in one direction.The output voltage was very high as compared to the previous reported ZnO, graphene, borophene and other similar 2D materials based nanogenerator under low compressive strain. For example, Mishra *et al*., have reported enhanced piezoelectric output of 285 mV from ZnO nanosheets based piezoelectric nanogenerator[29]. PVDF

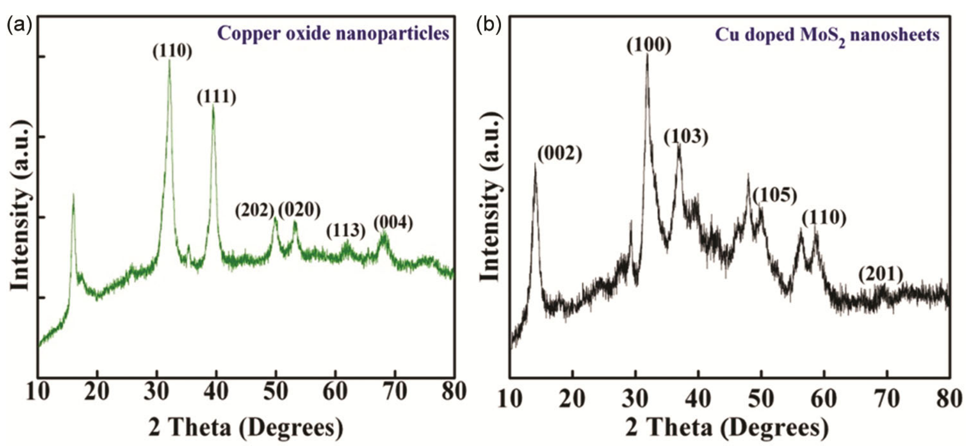

Fig. 2 — X-ray diffraction pattern of (a) CuO nanoparticles and (b) 2D few layered Cu doped MoS_2 nanosheets.

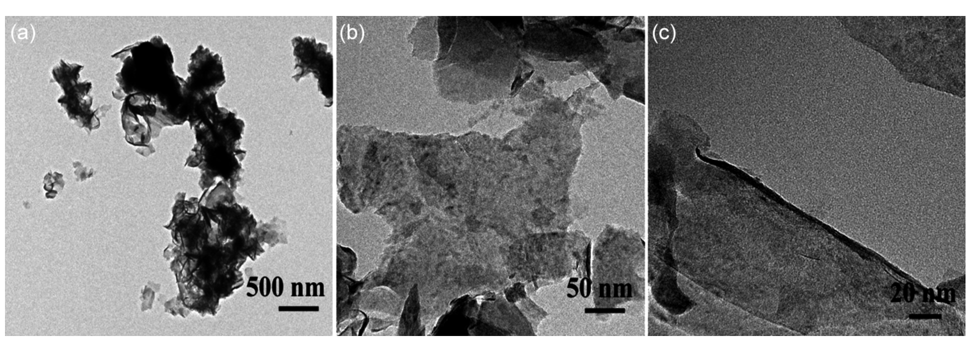

Fig. 3 — HR-TEM images of Cu doped MoS_2 nanosheets (a) Low resolution TEM image of the as grown Cu-doped MoS_2 nanosheets (b,c) HighresolutionTEM image of the Cu-doped MoS_2 nanosheets, confirming the formation of the nanosheet like morphology.

encapasulated reduced graphene oxide nanosheets was also used to fabricate the piezoelectric nanogenerator and an output voltage of 1.915 V was reported by Anand and their team[30]. Monolayer $MoSe_2$ synthesised from CVD method was used to fabricate the piezoelectric nanogenerator that generated output voltage of 60 mV at 0.6% strain[31]. Song and his team have fabricated monolayer WS_2 based piezoelectric nanogenerator with an output voltage of 2.26 V[32]. A novel piezoelectric device based on borophene nanosheets fabricated by our team with an output voltage of 8 V[33].

For comparison, we have also constructed a separate device based on the pristine MoS_2 nanosheets-PDMS, fabricated under the same condition and the piezoelectric output voltage is also obtained under the same force of 1kgf. The pristine MoS_2 nanosheets based nanogenerator generated an output voltage of 9V (Fig. 4(b)) which is low as compared to the output voltage generated from 2D Cu doped MoS_2 nanosheets based flexible piezoelectric nanogenerator under same vertical pressure. It was proposed that Cu-doping in MoS_2 nanosheets improved the piezoelectric and dielectric polarization though passivation of the crystalline defects in MoS_2 nanosheet such as sulphur defects, which are usually act as free charge carrier and screen the piezoelectric charges under mechanical deformation.

The working mechanism of Cu-doped MoS_2 nanosheets based flexible piezoelectric nanogenerator can be explained by formation of electron dipoles and thereby generation of piezoelectric potential under mechanical strain. Working mechanism of the Cu-doped MoS_2 nanosheets based flexible piezoelectric nanogenerator is shown in the Fig. 5. When no pressure is applied, no output voltage is generated from Cu-doped MoS_2 nanosheets based nanogenerator, as no piezoelectricity produced net zero dipole moment (Fig. 5(a)). Under vertical mechanical strain, electric dipoles are developed due

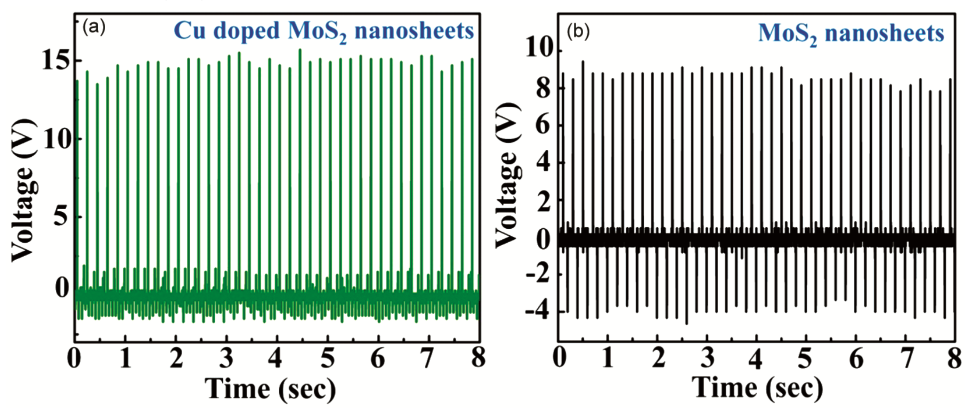

Fig. — 4. Piezoelectric output voltage generated from (a) Cu doped MoS_2 nanosheets based piezoelectric nanogenerator and (b) piezoelectric output voltage generated from pristine MoS_2 nanosheets based piezoelectric nanogenerator.

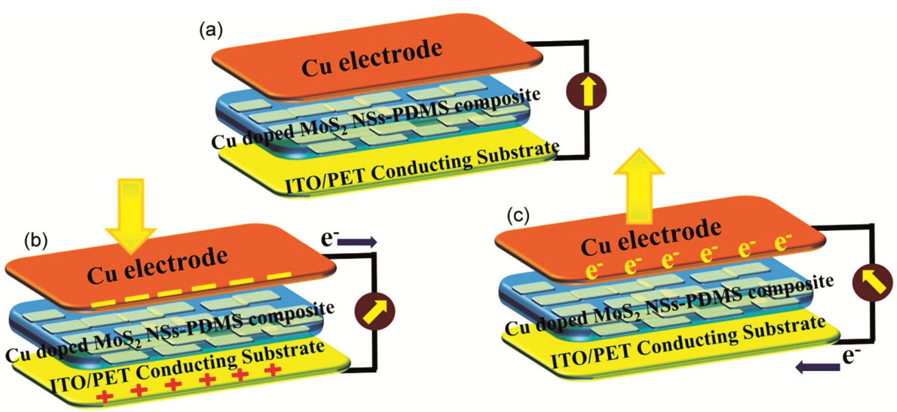

Fig. — 5. Working mechanism of Cu doped MoS_2 nanosheets based piezoelectric flexible nanogenerator (a) When no external vertical force is applied on Cu doped MoS_2 nanosheets based nanogenerator, no output voltage was obtained; (b) When vertical mechanical force is applied on the nanogenerator, piezoelectric potential is developed and electric signal is obtained; (c) When the vertical applied mechanical force is removed from the flexible Cu doped MoS_2 nanosheets piezoelectric nanogenerator, piezoelectric potential dismissed and electron transported back to the top electrode and electric signal in opposite direction is obtained.

to the piezoelectric effect negative and positive charges are developed towards top (Cu electrode) and bottom electrode (ITO/PET) thereby net current flow from top electrode to the bottom electrode and output electric signal is obtained in forward direction(Fig. 5(b)). When vertically applied mechanical force is removed, as shown in Fig. 5(c), all the piezoelectric charges developed gets diminished and as a result current starts to flow in negative direction from bottom electrode to top electrode and therefore the electric signal in reverse direction output is obtained from device. Under continuous application and release of mechanical force, AC type signal is generated from the Cu-doped MoS_2 nanosheets based nanogenerator[34].

The piezoelectric performance of the Cu-doped MoS_2 nanosheets based nanogenerator is also depends on the dielectric property of the 2D materials. Dielectric properties of the Cu doped MoS_2 nanosheets were investigated in the frequency range of 1 kHz -2 MHz. For the dielectric measurements of the Cu doped MoS_2 nanosheets, a circular pellet was made and conducting silver paste was applied on its both sides. Dielectric constant of the Cu-doped MoS_2 nanosheet sample was calculated by using the formula:

$$C = \frac{\varepsilon_0 \varepsilon_r A}{d} \quad \ldots(2)$$

Where ε_0 and ε_r is the permittivity of free space and relative permittivity, respectively, C is the capacitance, A is the area of electrodes and d is the thickness of the testing sample. The dielectric constant of pristine CuO nanoparticle and 2D Cu doped MoS_2 nanosheets are shown in Fig. 6. The dielectric constant and dielectric loss of Cu doped MoS_2 nanosheets were found to be about 6750 and 1000, respectively at low frequency of 1 kHz. The dielectric constant and dissipation factor decreases with increase in the high frequency and reached to value of 1883 and 2.74 at 2 MHz, respectively.

Dielectric constant of pristine CuO nanoparticles was also calculated and found to be 225 at 1KHz and 77 at high frequency of 2MHz. The dielectric loss of CuO nanoparticles was found to be 2.0 at low frequency and 0.009 at high frequency. In both the cases, the dielectric constant and dielectric loss are decreased with the increase of the applied frequency. At the higher frequency side, there is rapid variation in the electric field due to which electric dipoles does not cope-up with this rapid variation of electric field and thus dielectric constant decreased with frequency. Moreover, larger value of dielectric constant of Cu doped MoS_2nanosheets can be attributed to the formation of large number of nanodipoles per unit volume as a result polarisation per unit volume

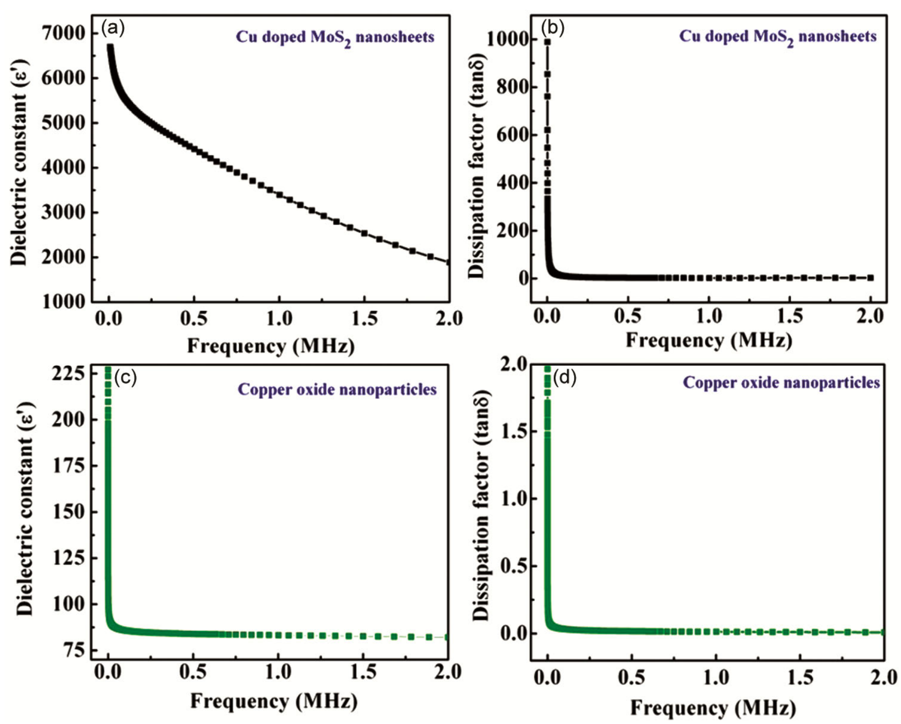

Fig. 6 — Variation of the (a) dielectric constant (b) dielectric loss with frequency for Cu doped MoS_2 nanosheets at room temperature (c) Variation of the (c) dielectric constant (d) dielectric loss for pristine copper oxide nanoparticles.

increases and thus the increase in dielectric constant value[34-35]. It was also reported that high dielectric constant leads to high piezoelectric charge coefficient of the 2D materials and thus improved output performance from Cu-doped MoS_2 nanosheet is obtained. Such high output voltage from flexible nanogenerator is highly beneficial to develop self-powered nano-system for various next-generation device application.

5 Conclusion

To summary, highly crystalline few layered piezoelectric 2D Cu doped MoS_2 nanosheets were grown by hydrothermal method under low temperature condition. XRD analysis confirmed the formation of 2H phase and crystalline nature of the Cu doped MoS_2 nanosheets. HRTEM confirmed the nanosheets like morphology with 3-5 layers growth of MoS_2 nanosheets. Piezoelectric nanogenerator based on Cu doped MoS_2 nanosheets exhibited high output voltage of 15 V under low vertical pressure of the 1kgf, which was higher than voltage obtained from pristine MoS_2 nanosheet nanogenerator. High dielectric constant of 6750 was obtained from Cu-doped MoS_2 nanosheets. Our results demonstrated a novel approach to synthesis the to high crystalline Cu-doped MoS_2 nanosheets and also an effective method to develop fully flexible high performance Cu-doped 2D MoS_2 nanosheet based nanogenerator for harvesting mechanical energy.

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Conflict of interest

The authors declare no competing financial interest.

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