Fabrication and electrical characterization of Organic Field-Effect Transistor based on CSA doped PANi-Ta_2O_5 nanocomposite

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Abstract—***Top-contact, bottom-gate organic field-effect transistors (OFETs) based on Polyaniline (PANi)-Tantalum Pentoxide (Ta_2O_5) nanocomposite doped with Camphor Sulphonic Acid (CSA) as the active semiconductor layer and Poly Methyl Methacrylate (PMMA) as the gate dielectric were investigated. Gold was thermally evaporated for the top source and drain contacts of 80-90 nm thickness with a conducting channel of 1 mm length and 1cm width. A relatively good charge carrier mobility of 0.12 cm^2/V-s was achieved. This may be ascribed to the highly crystalline nature of the nanocomposite, the diminished contact resistance due to the long channel and the symbiosis developed between the organic semiconductor and the polymer dielectric. The smaller source-to-drain current and high saturation drain voltage may be accounted for the long channel effect. The device exhibited a threshold voltage of -12.89 V, a moderate current on/off ratio of ~10^3 and a subthreshold swing of 9.3 V/dec. The agglomerated globular morphology of the PANi nanocomposite and the high carrier mobility can immensely contribute towards using the OFET device for room-temperature based application, particularly in the gas sensing field.***

I. Introduction

Organic electronic devices such as the organic field-effect transistors (OFETs) have emerged as the revolutionary technology towards the advent of new generation low cost, flexible electronics. OFETs based on conducting polymers (CPs) and their nanocomposites have established as a promising constituent in the field of large-area electronic displays, gas sensors [1], integrated optoelectronic devices, wearable devices [2], electronic skin and nose [3,4], implantable medical circuits [5], environmental monitoring devices [6] etc. This has motivated enormously the research fraternity to develop new materials based on organic semiconductors (OSCs), both CPs and small molecules of immense potential to develop the active layer in the design of the OFETs. The performance of OFETs has shown dramatic improvements over the years in terms of charge carrier mobility, stability, low operating voltage and working temperature. Past works reported of pentacene based OFETs that achieved high carrier mobility from 0.3 up to 0.9 cm^2/ V-s [7]. Y.Y. Lin et al. [8] used double layers of pentacene in the OFET to obtain high carrier mobility of 1.5 cm^2/ V-s, current on/off ratio greater than 10^8, threshold voltage almost zero and a sub-threshold swing as low as 1.6 V/dec. L.N. Ismail et al. [9] demonstrated poly (3-hexylthiophene-2,5-diyl) (P3HT) based p-type OFETs with poly methyl methacrylate (PMMA) and titanium dioxide (TiO_2) nanocomposite as the dielectric material that showed carrier mobility 2.01 cm^2/V-s and threshold voltage -3V.

K. Kim and co-workers [10] fabricated single crystal rubrene based top-contact, p-type OFETs with spin coated poly (4-hydroxystyrene) as the gate dielectric and achieved carrier mobility of 1.12 cm^2/V-s, threshold voltage of -0.3 V, current on/off ratio of 10^2 and 1.6 nA of leakage current. They showed that the device exhibited improved performance over the OFETs devised with the ceramic dielectric silicon nitride (Si_3N_4) in bottom contact configuration.

Y. Li et al. [11] fabricated CSA-PANi based OFET with the electrolyte poly(ethyleneimine) (PEI) inserted between the semiconductor layer and the gate dielectric polyvinyl pyrrollidone (PVP) and achieved carrier mobility of 2.48 12 cm^2/V-s. They found that without the PEI, the device exhibited resistor behavior.

Over the several decades, conducting polymers (CPs) have witnessed growing importance owing to their intrinsic potentials such as structural diversity, tailor-made electrical/electronic properties, easy synthesis process and environmental stability [12]. These fascinating properties have made the CPs standout in the crowd of the inorganic competitors. In 1977, H. Shirakawa, A. MacDiarmid and A. Heeger made the revolutionary breakthrough into the discovery of polyacetylene. Since then, various important CPs such as polypyrrole (PPy), polyaniline (PANi), polythiophene (PT), poly(3,4-ethylenedioxythiophene) (PEDOT), and poly(p-phenylene vinylene) (PPV) have been investigated incessantly [13].

Conducting polymers (CPs) are polymers with conjugated backbone chain with alternating single and double (π) bonds along the chain. These π-conjugated systems render the CPs unique optical, electrochemical, and electrical/electronic (conductivity) properties. To achieve enhanced conductivity, the CPs are doped either through redox mechanism or protonation.

As stated above, the reasons behind the numerous research attentions on CPs today are their inherent properties of easy synthesis process, good mechanical and environmental stability and more importantly the ability to tailor their molecular structure and morphology as per the need [14]. CPs such as polyacetylene, polythiophene (PTh), polypyrrole (Ppy), polyaniline (PANi), poly(3,4- ethylenedioxythiophene (PEDOT) have been successfully used in various types of sensors such as chemiresistive thin-film gas sensors, OFETs, surface acoustic wave (SAW), optrode and amperometric sensors [15].

The category of nanocomposite made of CPs and inorganic material develops altogether a new set of properties that depends not only on their individual original properties but also on their interfacial characteristics. The material properties of such hybrid materials can be fine-tuned at the molecular level to achieve the desired result [16]. S. Huh et al. and B. Chae et al. [17,18] devised an innovative photo-patternable OFETs based on poly (3-hexylthiophene), gold nanoparticles and photoreactive cinnamate group. X. Chen et al. [19] reported a flexible low-voltage operated OFET based on cross-linked PVP blended with novel ceramic material calcium titanate nanoparticles as the gate dielectric. Several groups reported gas sensors based on such nanocomposites with promising results [20,21].

Among the CPs, PANi is one of the most commonly used material in OFETs due to its simple chemical structure, good environmental stability, easy synthesis process, interesting redox property, and relatively high conductivity [22]. In several past works, PANi has been used successfully in conjunction with nanoparticles of metal oxides as the active layer to achieve breakthrough particularly in the field of gas sensing [23-26].

The research of the gate dielectrics has found as importance as that of the OSCs to achieve high-performance OFETs. The desirable requirements of the gate dielectrics to be used in OFETs are high capacitance (for high drain current while operating at a lower voltage), solution processibility and compatibility with flexible substrates [27]. Inorganic dielectrics possess high dielectric constant but they offer a rough surface and it is difficult to cast them on a large surface. An organic dielectric makes a smooth surface and helps in the proper growth of the organic semiconductor surface [28]. A polymeric dielectric can operate at a low processing temperature, can lower the leakage current and the operating voltage [29,30], and facilitate a trap-free semiconductor/dielectric interface enhancing the charge carrier mobility [31].

PMMA is a synthetic resist obtained from the polymerization of methyl methacrylate used in the high-resolution process of nanolithography that uses electron beam, UV or X-ray radiation. It has good thermal and mechanical stability, and a high resistivity greater than 10^{15} ohm-cm. PMMA has a permittivity of 2.6 at 1 MHz and a dielectric constant of 3.9 at 60 Hz. These make PMMA similar to silicon dioxide and hence it becomes suitable to be used as dielectric. Also, it is easier to deposit PMMA using spin-coating method and baked at low temperature [32].

J. Puigdollers [32] and coworkers devised a pentacene based OFET with PMMA as the gate dielectric and obtained carrier mobility of 0.01 cm^2/ V-s and threshold voltage of -15 V. These features are attributed to improved morphology of pentacene influenced by the underlying gate dielectric.

K. Amer et al. [33] demonstrated an effective humidity sensor based on PANi doped with dodecyl benzene sulphonic acid (DBSA) using PMMA as the gate dielectric.

A. Maliakal et al. [34] reported charge carrier mobility of 0.2 cm^2/ V-s approx. for a pentacene based OFET incorporating high-K TiO_2/polystyrene nanostructure. Wang et al. [35] reported a flexible OFET based on silk fibroin as the gate dielectric to achieve high carrier mobility of 23.2 cm^2/ V-s and a low operating voltage of -3 V.

An OFET is consisted of three electrodes viz., source, drain and gate, a semiconductor layer as the active layer and an insulator layer as the gate dielectric that lies between the semiconductor layer and the gate (Fig. 1). A voltage is applied at the gate terminal to control the current flow between the source and the drain.

The most common variety of OFETs is the p-type OFETs because of the relatively high hole mobility of the OSC they employ and the ease of synthesis process of most of the p-type OSCs [36,37]. A negative voltage greater in magnitude than the threshold voltage of the OSC is applied between the gate and the source which causes a p-type channel to form at the OSC-gate dielectric interface. A negative drain-to-source voltage is also applied to cause the holes to flow from source to drain. The magnitude of the drain-to-source voltage is increased until the "pinch-off" condition takes place. At this point, the p-type channel converges close to one side and the drain current reaches its saturation. The magnitude of the saturation current depends on the gate-to-source voltage [38].

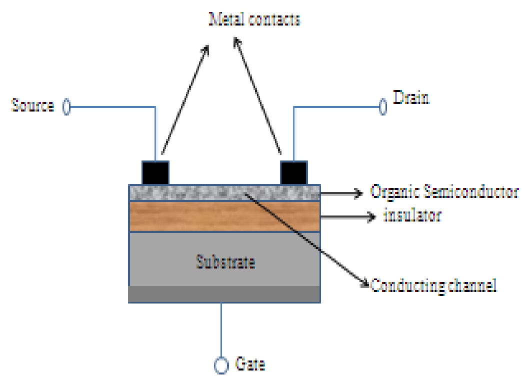

Fig.1. Device configuration of top-contact bottom-gate OFET.

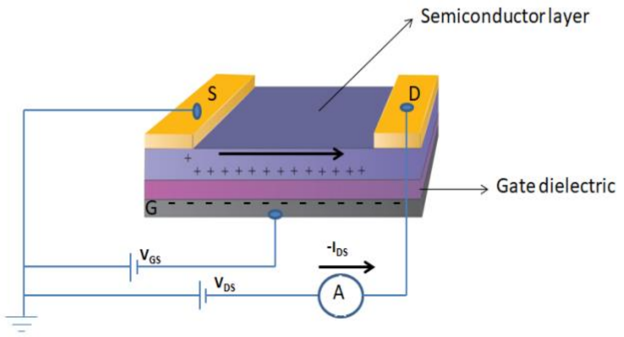

Fig.2. Device operation of p-type OFET with top contacts and bottom gate (both V_{DS} and V_{GS} are negative).

The schematic of the OFET operation for p-type conduction (hole conduction) is shown in Fig. 2. The desirable performance parameters of an OFET are high charge carrier mobility (μ) and current on/off ratio (I_{ON}/I_{OFF}), and low threshold voltage (V_{TH}) and sub-threshold swing (SS).

Previously, our group synthesized and studied the surface morphology and spectroscopic properties of PANi-Ta_2O_5 (50wt%) nanocomposite doped with CSA in 20wt%, 30wt% and 40wt% respectively [39]. The nanocomposite with CSA40wt% was found to exhibit good sensing behavior towards environmentally toxic gas NO_2.

In the current work, we fabricated a top-contact, bottom-gate OFET device based on PANi-Ta_2O_5 nanocomposite doped with CSA in 40wt% using PMMA as the gate dielectric. We, then, investigated the electrical characteristics of the OFET.

The novelty of our present work is based on using the acid doped PANi based organic-inorganic hybrid material which was used as the active layer of the OFET. Material characterization of the PANiTa_2O_5-CSA [39] revealed its potential in the gas sensing field.

The device under study was fabricated using a facile and cost effective fabrication process. It was possible to perform solution deposition of both the organic semiconductor and the polymer dielectric due to their good solubility. The deposition technique is a determining factor of OFET performance.

A relatively good performance of the device under study was achieved at room temperature. We found limited but promising works in the literature on OFETs based on CP based nanocomposite comprising of the polymer and inorganic counterparts [40-42]. Our future work is motivated in implementing the proposed device for gas sensing application.

II. Experimental

2.1. Material Preparation

The synthesis process and preparation of PANi-Ta_2O_5(50wt%) doped with CSA(40wt%) was reported in our previous work [39]. This nanocomposite was used as the active layer of the OFET device.

2.2 Material Characterization

The CSA doped PANi-Ta_2O_5 nanocomposite was analyzed through i) Scanning Electron Microscopy (using ZEISS SIGMA variable pressure field emission scanning electron microscope (FESEM), ii) Fourier Transform Infrared (FTIR) spectroscopy using a Nicolet 6700 spectrometer over the wave number range of 4000-500 cm^{-1} and iii) powder XRD analysis of the samples were recorded using X-ray diffraction (XRD, Brukers AXS, Germany/D8 Focus) with Cu-Kα radiation, beam wavelength=1.54Å operated at 40KVA/40m within the 2θ range of 5^0-85^0.

2.3. Fabrication process

ITO coated glass substrate (0.5 cm × 0.5 cm) was used for making the OFET device and the bottom gate electrode. Before initiating the fabrication process, the substrate was cleaned thoroughly using RCA1 followed by RCA2 and then dried at room temperature.

PMMA was obtained from free radical polymerization of methyl methacrylate and benzoyle peroxide as the initiator at room temperature. 10 mg of PMMA was diluted in 100 ml of anisole and the solution was spun on the ITO coated glass substrate at 800 rpm for 80 seconds. The PMMA layer was then dried in a conventional oven at 50ºC for 1 hour. The thickness of the PMMA layer was measured with Spectroscopic Ellipsometer (Make : Semilab) and found to be ~500 nm.

PANi-Ta_2O_5-CSA nanocomposite was synthesized and produced in the form of fine powder. A good dispersion was obtained by dispersing the nanocomposite in NMP (N-methylpyrolidine) in 10wt% and made it to stir on a magnetic stirrer for 4 hours. The nanocomposite was then deposited on the PMMA layer using spin-coating technique at 1000 rpm for 80 seconds. The deposited layer was then dried in an oven at 50ºC for 1 hour.

Gold was used to make the top source and drain contacts. All the metal contacts, each with thickness 80-90 nm, were deposited using thermal evaporation with a hard mask in a physical thermal vacuum coating system (BC-300, Hind High Vacuum) maintaining the pressure at 2×10^{-5} Torr. The schematic of the OFET with channel length (L) 1 mm and channel width (W) 1 cm was shown in Fig.3.

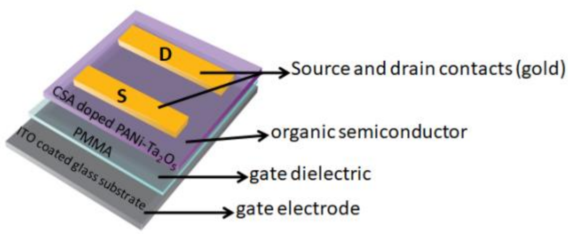

Fig. 3. Schematic of the proposed CSA doped PANi-Ta_2O_5 OFET device.

2.4 Electrical Characterization

The electrical characterization of the OFET device was done under ambient condition at 25^0C room temperature and 55% relative humidity using Keithly 2450 Sourcemeter and Keithly 6517 B electrometer/high-resistance meter. For the output characteristics, the gate was biased with a constant voltage (V_{GS}) and a voltage was applied between the drain and the source (V_{DS}), with the later being grounded. V_{DS} was swept through 0 V to -50V, while the corresponding value of I_{DS} was recorded. For each output plot, V_{GS} was kept fixed at 0 V though -50 V in steps of -10 V.

The quality of the OFET is mainly determined by the values of charge carrier mobility (μ), threshold voltage (V_{TH}), current on/off ratio and subthreshold swing (SS). These parameters were measured subsequently.

III. Results and discussion

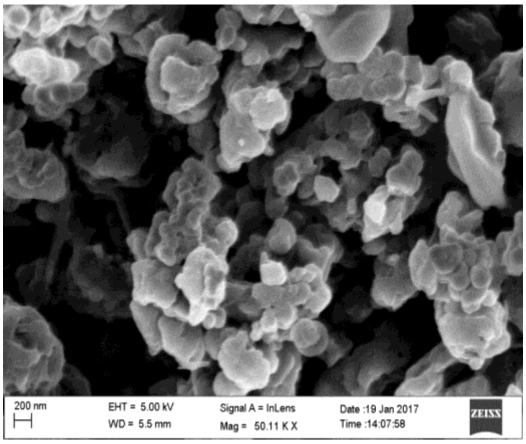

Fig. 4. FESEM image of PANi-Ta_2O_5-CSA nanocomposite [39].

Fig. 4 shows the FESEM micrograph of PANi-Ta_2O_5-CSA nanocomposite which revealed highly aggregated globular structures of ±200 nm diameter. Studies [43] reveal that these PANi nanostructures can possess high conductivity and large surface area and become suitable for gas sensing applications.

Fig.5. shows the FTIR spectrum of PANi-Ta_2O_5-CSA nanocomposite. FTIR studies are known to be useful to identify the important chemical bonds and functional groups of a given material. In the spectra every wavelength of light absorbed characterizes a specific chemical bond.

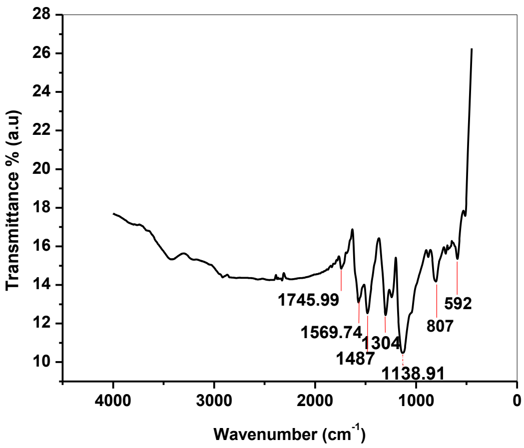

Fig. 5. FTIR spectrum of PANi-Ta_2O_5-CSA nanocomposite [39].

As seen in Fig.5, the peaks at 807 cm^{-1} and 1138.91 cm^{-1} may be responsible for the stretching vibration modes of O=Ta=O bonds. The peak at 1304 cm^{-1} belongs to PANi-Ta_2O_5 molecule. The addition of CSA into PANi-Ta_2O_5 molecule resulted in the formation of peaks at 592 cm^{-1}, 1487 cm^{-1}, 1569.74 cm^{-1} and 1745.99 cm^{-1} and they are ascribed to =S=O bond for CSA.

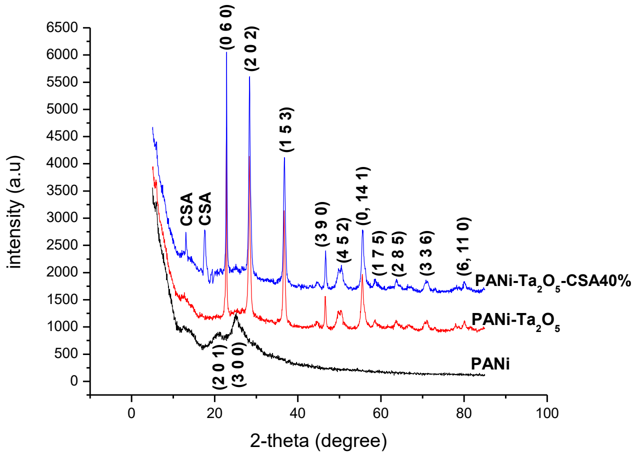

Fig. 6. XRD graphs of PANi, PANi-Ta_2O_5 and PANi-Ta_2O_5-CSA nanocomposite [39].

As seen in XRD graphs of Fig.6, pure PANi possessed two distinct peaks at $2\theta=20.68^0$ and at $2\theta=25.24^0$. This suggests the formation of PANi nanostructure that exists in semi-crystalline form. The XRD pattern of PANi-Ta_2O_5 confirms the presence of Ta_2O_5 in the nanocomposite. It is confirmed from the XRD graph that Ta_2O_5 retained its own crystal structure even being dispersed in the PANi matrix.

The XRD pattern of PANi-Ta_2O_5-CSA40wt% revealed two prominent peaks at $2\theta=13.04^0$ and $2\theta=17.58^0$ which are responsible for the presence of CSA molecule in the nanocomposite. Some other peaks of PANi-Ta_2O_5-CSA40% attained greater intensity with respect to those of PANi-Ta_2O_5. This confirms PANi-Ta_2O_5-CSA40% has greater crystallinity over PANi-Ta_2O_5 [39].

Fig. 6 depicts the output and transfer characteristics of CSA doped PANi-Ta_2O_5 OFET device.

The output characteristics shown in fig.6.a depict a high operating voltage which might have resulted due to the low-k PMMA dielectric [27]. The drain-to-source current, I_{DS} displays quadratic dependence on V_{GS} in saturation due to the long channel effect. Also, the saturation drain current at V_{GS}= -50 V seemed not so pronounced.

The key parameters of the OFET, viz. the charge carrier mobility (μ), threshold voltage (V_{TH}) and current on/off ratio were evaluated as follows.

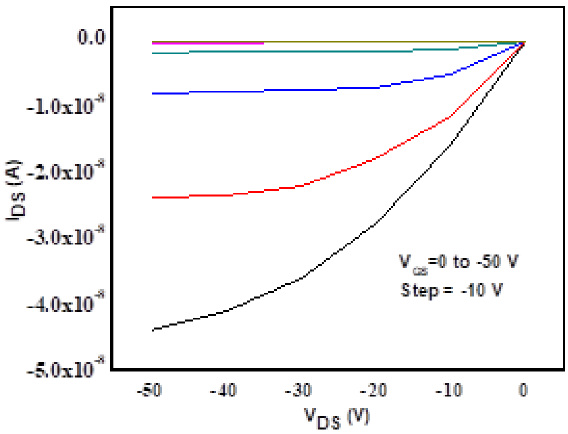

Fig.7.a. Output characteristics of CSA doped PANi-Ta_2O_5 based OFET.

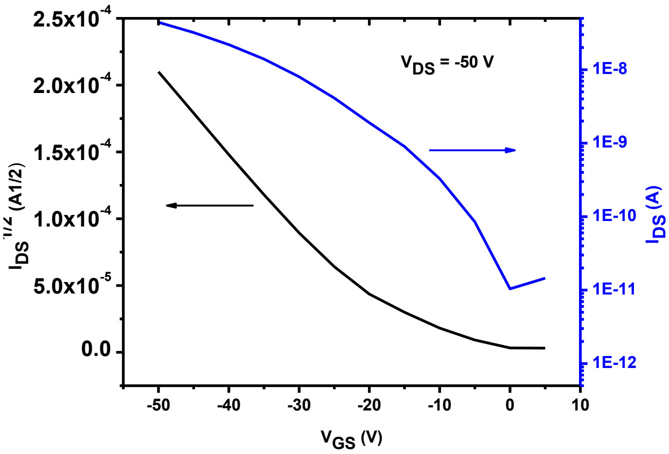

Fig.7.b. Transfer characteristics of CSA doped PANi-Ta_2O_5 based OFET.

The field-effect mobility, μ was estimated from the transfer curve in saturated regime shown in fig.6.b and the following equation:

$$I_{DS} = \frac{W}{L}\mu C_i \frac{1}{2}(V_G - V_{TH})^2 \text{ ----- (1)}, \quad V_{DS} > (V_{GS}-V_{TH})$$

where W and L are the channel width and length and C_i is the capacitance of the gate dielectric per unit area. Here, the values of the these parameters are W=1 cm, L=1 mm and C_i=6.195 × 10^{-9} F/cm^2.

In the saturation regime, mobility was extracted from the $\sqrt{I_{DS}}$ versus V_{GS} plot by solving the following equation:

$$\mu_{sat} = \frac{2L}{WC_i}\left(\frac{\partial\sqrt{I_{DS}}}{\partial V_{GS}}\right)^2 \text{ --------------- (2)}$$

The threshold voltage was obtained by extrapolating the transfer curve.

The device yielded mobility of 0.12 cm^2/V-s and threshold voltage -12.89 V.

Current on/off ratio was calculated from the transfer curve again by taking I_{ON} as I_{DS} at maximum values of V_{DS} and V_{GS}. I_{OFF} is the minimum of I_{DS} at maximum value of V_{DS}. Thus, the current on/off ratio was found as ~10^3.

The sub-threshold swing, SS, was obtained from logarithmic plot of I_{DS} versus V_{GS} plot (Fig. 6.b) using equation 3. This was found to be 9.3 V/dec.

$$SS = \left(\frac{\partial \log|I_{DS}|}{\partial V_{GS}}\right)^{-1} \text{ --------------- (3)}$$

The value of SS should be as low as possible because for low power applications it is crucial to have a small ∂V_{GS} that can turn the device from fully "off" to fully "on" state [44].

Electrical parameters of the CSA doped PANi-Ta_2O_5 OFET are summarized in table I.

TABLE – I

(Summary of device parameters of PANi-Ta_2O_5-CSA based OFET)

Charge Carrier Mobility (μ)	**Threshold Voltage (V_{TH})**	**Current ON/OFF ratio (I_{ON}/I_{OFF})**	**Subthreshold swing (SS)**
0.12 cm^2/V-s	-12.89 V	~10^3	9.3 V/dec.

Y.R. Su et al. [44] demonstrated low-voltage operated OFETs based on copper phthalocyanine (CuPc) utilizing solution processed high-k dielectric Al_2O_y/TiO_x (ATO) and obtained values of μ, V_{TH}, current on/off ratio and SS as 0.15 cm^2/V-s, −1.1 V, 5×10^3 and 232 mV/dec. respectively. The same group also devised the CuPc based OFET using low-k dielectric SiO_2 and attained less satisfactory results. The obtained μ and V_{TH} are 2.8×10^{-3} cm^2/V-s and −6.0 V respectively. The device exhibited moderate current on/off ratio on the order of 10^3 and a sub-threshold swing (SS) of 5.9 V/dec.

J. Tardy et al. [45] devised pentacene based OFETs using high-k HfO_2 as the dielectric deposited using sol-gel process. They attained a high mobility of 0.12 cm^2/V-s with considerable device stability.

Large values of V_{TH} and SS might be due to the existence of localized hole trapping states near the highest occupied molecular orbital and electron trapping states near the lowest unoccupied molecular orbital. This phenomenon generally happens in OFETs [46].

The relatively high charge carrier mobility of our OFET device might have resulted from the decrease in source and drain contact resistance due to the long conducting channel which leads to a weaker influence of the channel resistance [47]. Also, the highly crystalline nature of the organic semiconductor might have been a contributing factor [48]. Charge carriers are more dynamic in crystalline regions in conducting polymers due to the existence of highly ordered and stronger π-π stacking [49].

We studied the atomic force microscopy (AFM- Model No.

NTEGRA Vita from NT-MDT) imaging of the proposed device to obtain insights on the surface morphology and roughness features.

Fig. 8 shows the topographic image of the area between the source and drain contact areas of the PANi-Ta_2O_5-CSA layer deposited on the PMMA dielectric layer. The AFM image clearly shows the well defined morphology of the organic upper layer with granular microstructure. The surface of the PANi-Ta_2O_5-CSA layer was found smooth with a root mean square (rms) roughness of 0.764 nm.

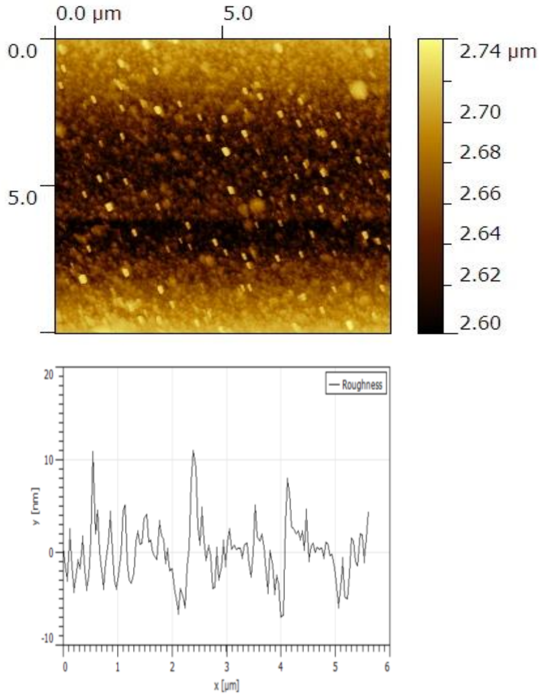

Fig. 8. AFM surface morphology of the CSA-PANi-Ta2O5 layer deposited on the PMMA layer (area between the source and the drain contacts) with rms roughness 0.764 nm.

It can be stated that the PMMA structure beneath the semiconductor layer might have favored the growth of the relatively smooth organic layer surface. There is high possibility of enhanced crystallinity of the CSA-PANi-Ta_2O_5 layer grown on PMMA. These factors are attributed for the improved carrier mobility of the device.

High carrier mobility results into high conductivity which in turn speeds up the charge transport in the active layer. This can facilitate an improved gas sensing feature of the semiconductor layer [50].

Although the polymeric dielectrics like PMMA have comparatively smaller value of dielectric constant, k (and hence smaller capacitance) compared to inorganic dielectrics (but higher than SiO_2), they can be easily solution processed and are compatible with flexible substrates. Thus, they can offer high-quality smooth surface helping in the proper growth of the semiconductor layer [28]. A polymeric dielectric can operate at a low processing temperature, can reduce the leakage current [30] and facilitate a trap-free semiconductor/dielectric interface enhancing the charge carrier mobility [31].

The solubility of PMMA rendered a good synergy between the former and the organic semiconductor and, helped in a favorable growth of the active layer [51]. This facilitates an improved interface leading to supposedly low leakage current and moderately high current on/off ratio [28]. Also, PMMA being hydrophobic in nature hinders the migration of contaminants into semiconductor-dielectric interface, thus reducing the no. of charge trapping sites [52].

However, our proposed device depicted a high operating voltage and relatively high threshold voltage. PMMA possesses a low dielectric constant and hence low capacitance which can lead to high operating voltage and high threshold voltage [27]. These issues can be solved by considering the advantages of high-k oxides for low voltage and low-k polymer for high quality surface with careful blending of the two materials [53-55].

IV. Conclusion

In this study, we demonstrated OFETs based on CSA doped PANi-Ta_2O_5 nanocomposite as the active layer and PMMA as the gate dielectric. The surface morphology of the CSA doped PANi-Ta_2O_5 nanocomposite revealed highly aggregated globular structures. The AFM results revealed a relatively smooth semiconductor layer with roughness (rms) 0.764 nm. The device exhibited a good charge carrier mobility of 0.12 cm^2/V-s and moderately high current on/off ratio of the order of 10^3, but large operating voltage, threshold voltage and subthreshold swing. The role of PMMA as the gate dielectric and the highly crystalline nature of the CSA doped PANi-Ta_2O_5, as revealed by the XRD graphs, contributed in obtaining a relatively high charge carrier mobility of the device.

The fabrication process of the device found cost–effective and straightforward with ease of material processibility. Thanks to the high solubility of the organic semiconductor and the polymer dielectric used which enabled solution-processed fine deposition of both the layers.

It can be concluded that the use of CSA doped PANi-Ta_2O_5 nanocomposite as the active layer and PMMA as the gate dielectric leads to a viable means to develop room-temperature operable OFETs for future use, particularly in the area of gas sensing. There is further scope to improve the device performances in terms of low operating voltage, small threshold voltage and subthreshold swing by working upon a shorter channel length, hybrid dielectric, and tailoring the semiconductor/dielectric interface.

V. Acknowledgement

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