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A Highly Sensitive, Direct X-Ray Detector Based on a Low-Voltage Organic Field-Effect Transistor

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Title: A highly sensitive, direct X-Rays detector based on a low-voltage Organic Field-Effect Transistor

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Keywords: organic field-effect transistor, X-Rays detectors, low voltage, high sensitivity, flexible electronics.

A novel organic transistor-based sensor for direct X-Rays detection is proposed. The device operates at low voltages (≤ 3 V) and is entirely fabricated on flexible, plastic substrates with techniques that can be easily up-scaled to an industrial size. To the best of our knowledge, flexible, low voltage organic transistors have never been employed as direct ionizing radiation detectors, as two terminal photodetectors are typically considered for this application. We demonstrate that, differently from two-terminal photodetectors, X-Rays detection ability of the proposed sensor can be tuned acting on the transistor polarization conditions. Thanks to such a peculiar feature of our device, outstanding values of sensitivity are observed (up to 1200 $nCGy^{-1}$), much larger than the ones reported for two terminal, direct organic photodetectors. Noteworthy, the reported performances have been obtained using as sensing layer a standard, commercially available organic semiconductor: a complete explanation of the mechanism behind the detection ability is thoroughly discussed. The device functionality is perfectly maintained even after the exposure to high X-Rays doses (160 Gy), thus demonstrating the significant radiation hardness of the detector.

1. Introduction

The detection of ionizing radiation over large areas is becoming crucial in different application fields including energy, national security, biological and nuclear research. Inorganic materials, such as amorphous silicon,[1] amorphous selenium[2] and diamond,[3] have been intensively explored; though well performing, the complex growth and fabrication methods needed for large crystalline inorganic materials often result in significant production costs for a number of applications, in particular when large area production is considered. Moreover, the typical stiffness of such materials is a limiting factor especially where device flexibility is a preferential requirement.

In order to overcome these limitations, sensors employing organic semiconductors as active layer have been recently investigated.[4]-[12] Indeed, fabrication techniques for organic devices, such as inkjet printing, physical and chemical vapor deposition, are generally low cost for large area production. As most organic materials can be processed at low temperature, fabrication over plastic substrates is possible, thus allowing the development of transparent, light-weight and flexible devices. Finally, organic semiconductor materials can be synthesized with specific properties, and this has justified their success in optoelectronic applications.

As regards ionizing radiation detection, two terminal structures such as photodiodes and photoresistors have been so far considered. Two terminal organic photodetectors have been reported mostly used for the indirect detection of ionizing radiation, i.e. integrated with a scintillating material.[4]-[6] More recently, a growing interest has been paid on their employment for direct radiation detection, i.e. the direct conversion of impinging radiation to electrical signal.[7]-[12]

On the contrary, Organic Field-Effect Transistors (OFETs) are still scarcely considered as organic photodetectors for ionizing radiation. Although inorganic thin-film transistors are commonly used in backplane flat panel detectors, the employment of their organic counterparts have been scarcely considered, even if several studies demonstrate that OFETs

can show a significant radiation hardness.[13],[14] This is even more true for direct detectors: as a matter of fact, a few examples of organic equivalent for devices such RADiation sensitive FETs (RADFETs)[15] have so far been reported in literature, in all cases being fabricated over rigid substrates and showing very poor sensitivities.[16],[17]

Among the reasons that may be invoked for justifying the lack of OFET-based ionizing radiation detectors in literature, an important role is played by their relatively high biasing voltages (typically, a few tens of volts), which still represent a serious limitation for actual device portability and results in a significant power consumption. Only one direct detector based on a low voltage OFET has been reported in literature to the best of our knowledge:[17] it has been fabricated on a rigid substrates, and low voltage operation has been obtained using a thin inorganic dielectric layer deposited by radio-frequency sputtering, that is a small area, low throughput technique.

Nevertheless, transistor structures have peculiar advantages that can be efficiently exploited in the field of radiation detection. For instance, transistors are multi-parametric devices, where different elements in the transistor structure, and thus different electrical parameters, can be employed for transducing the sensing event. In addition, peculiar characteristics, such as the possibility of finely tuning the conductivity by acting on the gate voltage and the intrinsic signal amplification ability, are unique among electronic devices, and impossible to implement in two terminal structures like resistors and diodes. Moreover, transistor-based sensors can be easily integrated in electronic systems, such as amplifiers and logic stages, which can provide an easy readout of the signals. It is noteworthy that all these characteristics have, as a matter of fact, determined the success of OFET-based sensors over two terminal devices in many other applications, including biochemical[18]-[21] and physical[22]-[27] sensing.

According to these considerations, the possibility of employing the organic active layer of a transistor structure as the detecting layer, overcoming the problems related to the high

operating voltages of such kind of devices, would represents a huge step forward in the development of large area electronic systems for ionizing radiation detection.

In this work, the first X-Rays direct detector based on a low-voltage OFET structure fabricated on a flexible, plastic substrate is reported. The proposed device structure is fabricated with a highly reliable process that can be entirely performed at room temperature and easily up-scalable to an industrial size. The detection mechanism will be discussed, and basic figures of merit will be reported. In particular, these results will demonstrate how the X-Rays detection performance can be tuned by properly choosing the biasing conditions of the transistor, allowing reaching unprecedented sensitivity values among organic direct detectors.

2. Results and Discussion

2.1. Device Electrical Characteristics

In **Figure 1**, an example of typical output (a) and transfer (b) characteristic curves is reported. Drain-to-source and gate-to-source voltages are in the range of 3 V, thus demonstrating the low voltage operation ability of the transistor. From output characteristic curves, ideal device features such as good current saturation, significant field-effect modulation and negligible contact resistance effect can be noticed. Moreover, transfer characteristics show negligible hysteresis and very low leakage current (in the range of tens of pA). The device average threshold voltage (V_{TH}) is 0.4 V; mobility values up to 0.1 $cm^2V^{-1}s^{-1}$ are obtained. Average and standard deviation of basic transistor parameters evaluated from a set of 40 devices are reported in Table S1 in Supplementary Information. As flexibility is a peculiar feature of the reported devices, mechanical stress test have been carried out: as shown in Figure S1 in the Supplementary Information, the device performances are well maintained even after several bending cycles.

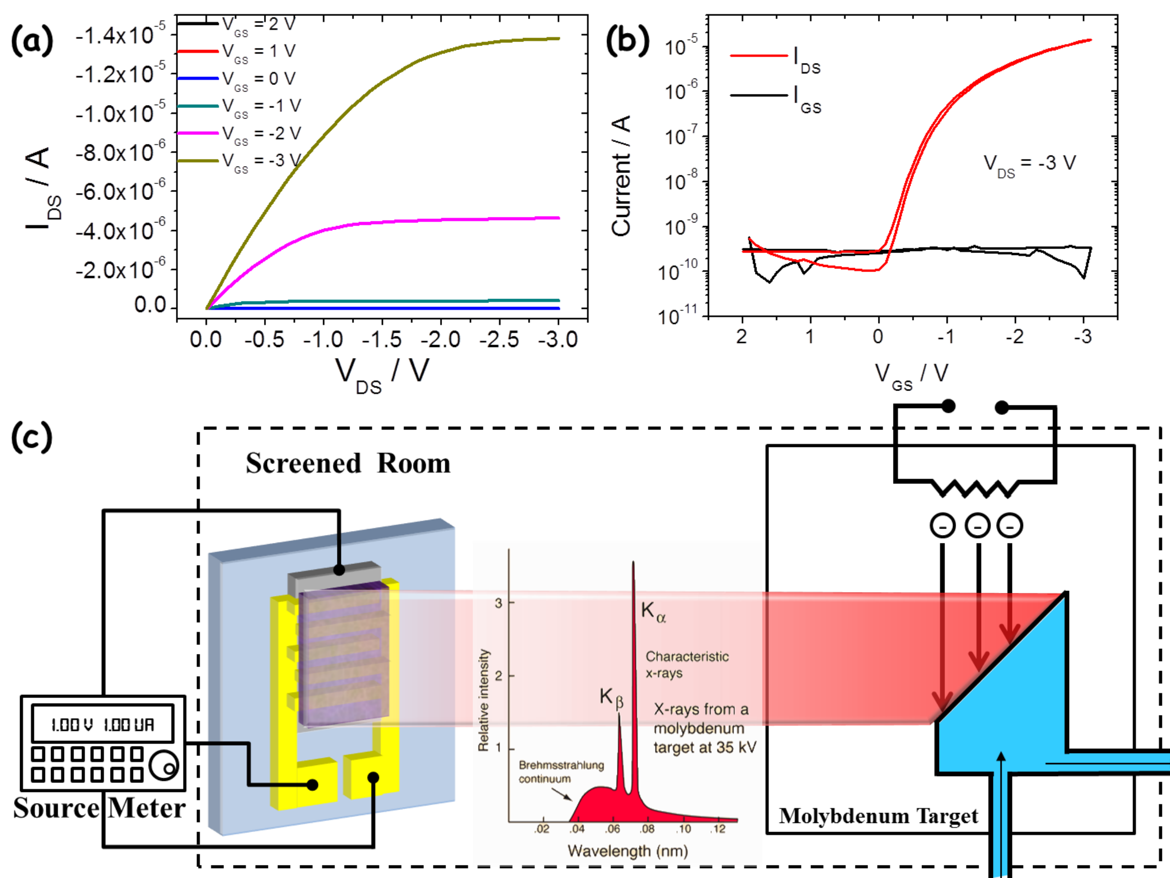

Figure 1. Typical output (a) and transfer (b) characteristic curves of low voltage devices fabricated on plastic substrates. (c) Schematic representation of the measurement setup for characterization under X-Rays.

2.2 Response to X-Rays in different bias conditions

In Figure 1(c), the experimental setup for the device characterization under X-Rays is depicted. The devices have been kept in dark conditions inside a Faraday cage mounted in the leaded glass-screened room of the X-Rays tube. More details are reported in the Experimental section.

In **Figure 2(a)** and **(b)**, real-time responses of the device to different X-Rays dose rates are reported in terms of drain-to-source current (I_{DS}) variation in linear regime (V_{DS} = -0.25 V, V_{GS} = -2 V) and in saturation regime (V_{DS} = -4 V, V_{GS} = -2 V), respectively. In both cases, the device was subjected to three X-Rays consecutive exposures, each one with a duration of 60 s (highlighted by the red-dashed rectangles). The current variation is evaluated as the difference between the actual value of the acquired current, and the dark current, i.e. the current level of

the transistor before the X-Rays exposure. It is evident that, for both operating regimes, the output current increases with the applied dose rate for tens of seconds, and a similarly slow dynamic is observed for relaxation after X-Rays exposure. The device response to X-Rays is well reproducible, with similar amplitude and dynamics for each subsequent exposure. The maximum current variation is generally higher in saturation regime; in linear regime, for the highest dose rate applied (54.8 mGys^{-1}), a minor reduction of current after reaching the saturation was noticed. As recently reported in Basiricò et al. for two-terminal devices,[29] the observed X-rays photocurrent can be ascribed to a photoconductive gain mechanism while the transient behavior can be attributed to stretched exponential recovery, due to the slow relaxation time constant of trapped charge carriers. In Figure 2(c) and (d), the gate-to-source current (I_{GS}), which is commonly employed to describe vertical leakage in the transistor current, is reported for linear and saturation regime, respectively. In both cases, a variation of the leakage current with X-Rays is clearly evident: with respect to I_{DS} variations, leakage current response to X-Rays is faster than 200ms. It is noteworthy that this signal is limited to a few tens of pA, thus being negligible with respect to I_{DS} variations (up to 140 nA for 54.8 mGys^{-1} dose rate exposure). The presence of such a small response of leakage current to X-Rays can be justified in terms of photo-emission at the aluminum gate electrode: indeed, due to the low X-ray absorbance of organic materials, the radiation easily pass through both TIPS pentacene (0.0019%) and Parylene C (0.0071%) layers, thus reaching the aluminum oxide (0.01%) and, finally, the underlying aluminum gate electrode (0.012%). X-Rays absorption by the aluminum induces photo-emission of charge carriers, which contributes to leakage current, thus justifying the observed variations.

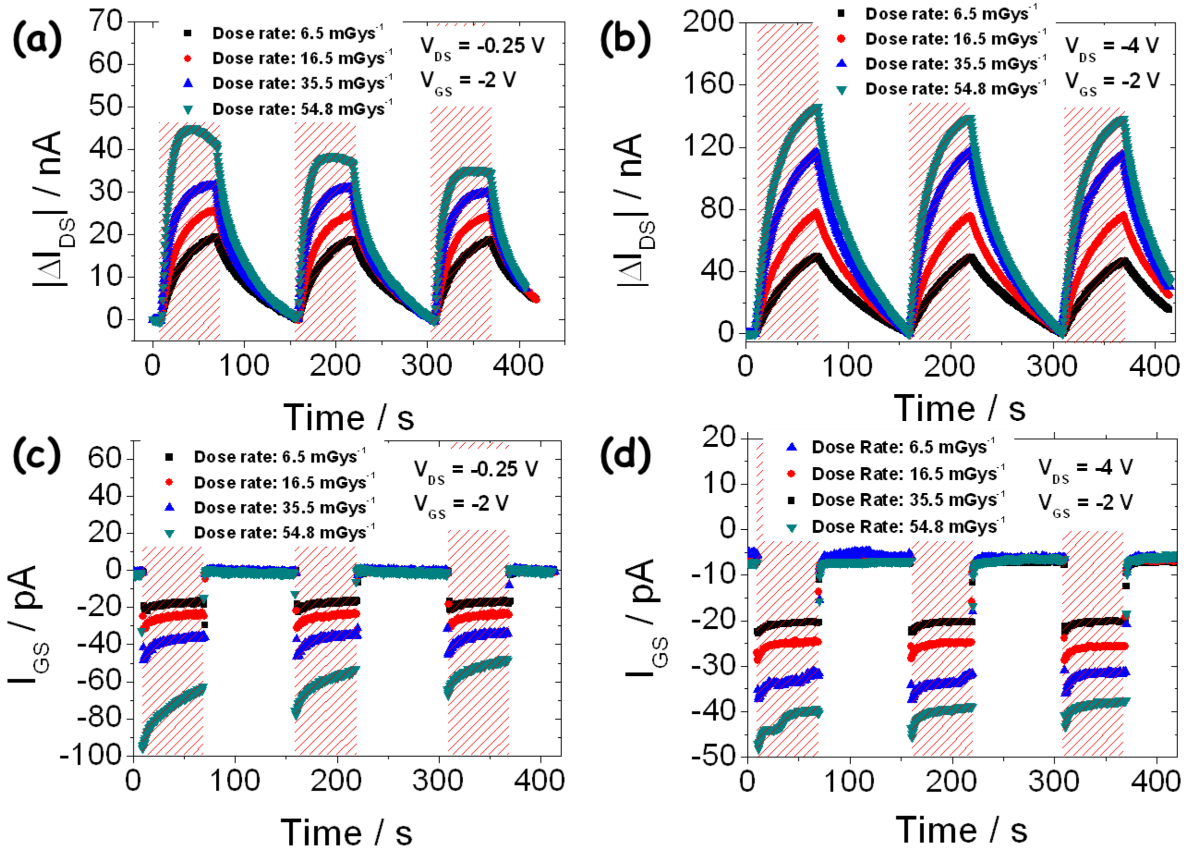

Figure 2. Typical response of the low voltage transistor to different dose rates in linear (a) and saturation (b) regimes, as variation in the drain-to-source current; corresponding variation in the leakage (gate-to-source) current in linear (c) and saturation (d) regime.

Similar experiments have been performed applying different bias voltages to the device, i.e. maintaining the transistor at a constant drain-to-source voltage drop (V_{DS} = -0.25 V in linear regime and V_{DS} = -3 V in saturation regime) but varying the V_{GS} value, in order to to span the device working regime from the OFF state ($V_{GS}<V_{TH}$), to the ON state ($V_{GS}>V_{TH}$). The results are summarized in **Figure 3(a)** and **(b)**, where the X-Rays induced photocurrent signal (I_{DS} variation, $|\Delta I_{DS}|$) is reported as function of the dose rate for different V_{GS} values, in linear and saturation regime respectively. In both cases, $|\Delta I_{DS}|$ under X-Rays is negligible when the device is in its OFF state (V_{GS} =0 V), whatever is the dose rate of the impinging radiation. By setting negative values of V_{GS}, the transistor is progressively switched on and the response to the different dose rates progressively enhances in both regimes. Interestingly, a significant increase of the response in linear regime is obtained for larger over-threshold conditions than

the ones necessary in saturation regime. When the transistor channel is completely formed, i.e. the gate voltage sets the transistor in the ON state, the detector sensitivity, defined as the first derivative of the current amplitude with respect to the dose rate, reaches the higher values. In other words, the sensitivity of the device to X-Rays is a function of the V_{GS} values. Results are reported in Figure 3(c). Summarizing, X-Rays response can be completely inhibited when the device is in under-threshold conditions, while progressively increases by switching it on. Such a feature is absolutely unique among photodetectors, and represents a significant advantage of the proposed structure with respect to two-terminal devices. For V_{GS} = -3 V, a remarkably high sensitivity of almost 1200 $nCGy^{-1}$ has been obtained for devices polarized in saturation regime. Such a value is comparable to or larger than the most up-to-date X-Rays detectors employing hybrid organic/inorganic compounds.[6],[30],[31]

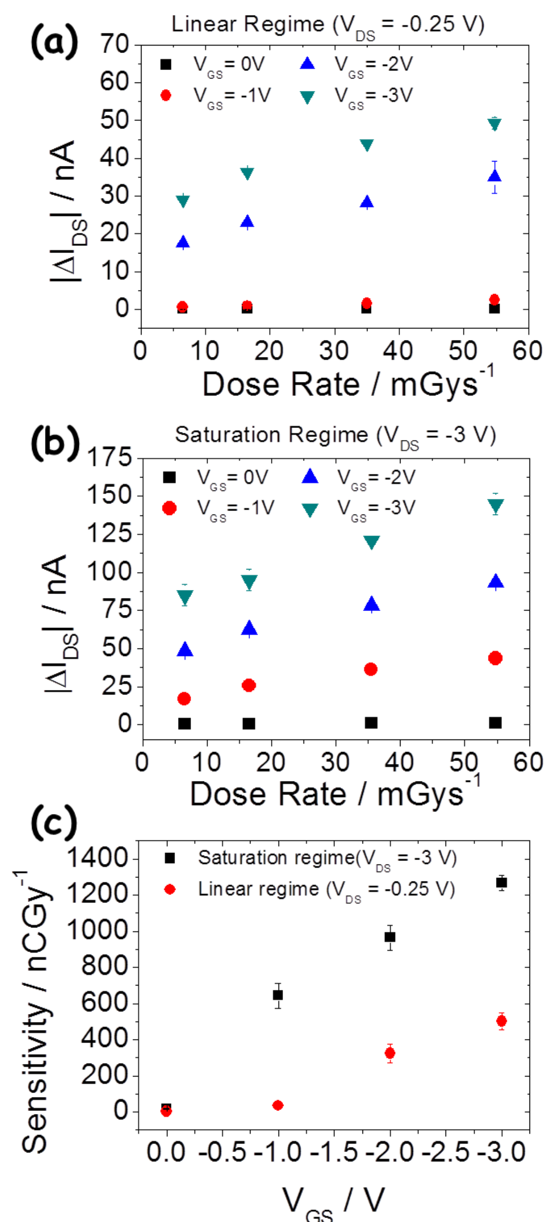

Figure 3. Average transistor response to X-Rays as a function of dose rate in linear (a) and saturation (b) regimes. (c) Sensitivity as a function of the gate-to-source voltage in linear and saturation regime.

As mentioned, an interpretation on the obtained results can be derived according to the model developed in Basiricò et al. for a low voltage photoresistors using TIPS-pentacene as photoconductive layer.[29] In that structure, the reported current variation is not simply ascribed to the charge collection of electron-hole pairs created by the X-Rays absorption of the semiconductor, very low in such low atomic number organic materials. The proposed explanation is that, in the semiconductor layer, photo-generated charge carriers pairs follow a different fate: holes can easily flow in the p-type semiconductor, thus being finally collected

by the electrodes, while electrons remain trapped within the semiconductor. This excess of electrons, which progressively accumulate within the semiconductor, induces a further injection of holes from the electrodes, in order to maintain the charge neutrality in the semiconductor film. Therefore, more than one hole is globally induced in the semiconductor by the interaction with the radiation. This mechanism leads to a photoconductive gain effect[32]-[34] that, acting as a doping of the organic semiconductor, justifies the amplitude and the dynamics of the observed X-ray induced photocurrent. Such a trap-assisted photocurrent enhancement has been previously observed in UV-vis organic photoconductors[35] and attributed to a similar charge accumulation and photoconductive gain mechanism.[36] The observed slow recombination dynamics of X-Rays generated carriers is related to the presence of electron trap levels deep in the bandgap of the organic semiconductor, which remove electrons from the recombination process and resulting thus crucial in the mechanism of the photoconductive gain; such states have been commonly observed in several organic semiconductors.[37]

The photoconductive gain can be evaluated as $G = \tau_r(\rho)\ \tau_t^{-1}$, i.e. as the ratio between the charge carrier lifetime (τ_r) and the charge carrier transit time, τ_t. According to the model reported in Basiricò et al.,[29] the charge carrier lifetime is a function of the charge carrier density in the channel (ρ) according to the equation

$$\tau_r(\rho) = \frac{\alpha}{\gamma}\left[\alpha \ln\left(\frac{\rho_0}{\rho}\right)\right]^{\frac{1-\gamma}{\gamma}} \qquad (1)$$

where α, γ and ρ_0 are material specific constants. The charge carrier transit time $\tau_t = L^2 V^{-1} \mu^{-1}$, being L the channel length, μ the mobility and V the voltage applied. From experimental data, a value of photoconductive gain in saturation region (V_{GS}=-2V, V_{DS}=-4V) of 3×10^5 has been calculated, which is significantly larger than the one obtained for the two-terminal, TIPS-pentacene based device in Basiricò et al..[29] As in the transistor the TIPS-pentacene layer is

integrated in a metal-insulator-semiconductor structure, additional effect related to the gate potential can be invoked for justifying the larger value of the gain here obtained. Indeed, as V_{GS} becomes more negative, the charge density in the transistor channel increases, and this brings to the enhancement of the efficiency of both holes conduction and electron accumulation; moreover, the contact resistance decreases,[38],[39] i.e. holes are more easily injected from the electrodes. Moreover, τ_t decreases with over-threshold conditions, and this further contributes to the enhancement of *G*. This interpretation also explains the lower sensitivity obtained, for a given V_{GS} value, in the linear regime. Indeed, in the linear regime the influence of the injection resistance is larger than in the saturation regime, thus limiting the photoconductive gain enhancement with V_{GS}. The effect of contact resistance also explains the non-linear dependence of sensitivity from V_{GS} in linear regime, being contact resistance itself non-linear with V_{GS}.[40]

2.3 Radiation hardness

The evaluation of significant changes of device performances as a consequence of damages in the materials related to X-Rays exposure is fundamental to define the reliability of the photodetector. To explore this aspect, device characteristics have been evaluated in different stress conditions.

First, the OFET output characteristics have been acquired for a pristine device in dark and during X-Rays exposure. Results are reported in **Figure 4(a)** as normalized transfer characteristic curves. The curves perfectly overlap. These results are consistent with the previously reported model: i) electron trapping in the semiconductor film brings the threshold voltage towards slightly more positive values, even if this mechanism is not so important to cause such a shift effective in the overall curve fluctuations of different measurements (see inset); ii) the X-Rays interaction with the device does not determine significant variations in the charge carrier mobility of the organic layer.

Prolonged exposure to X-Rays has also been taken into account. In Figure 5(b), normalized transfer characteristic curves of a pristine device is compared to the one acquired after a complete characterization under X-Rays (similar to those reported in Figure 2 and Figure 4), in both linear and saturation regime, thus resulting in a total dose of about 160 Gy. After the characterization, a significant shift of the threshold voltage towards more negative values, and a reduction of mobility can be noticed. Interestingly enough, after a relaxation time of 24 hours, an almost complete recover in both parameters was observed, thus demonstrating that no effective permanent damages occur in the materials. This result further demonstrates that charge traps involved in the photoconductive gain effect are those already present in the transistor structure, and are not related to a degradation effect due to prolonged X-Rays exposure. To explain the temporary shift of the transfer characteristics, a reversible stress effect can be invoked. Such a feature has been previously observed in OFETs fabricated onto silicon substrates and using silicon dioxide as gate dielectric.[13] Indeed, as already demonstrated in Cosseddu et al.,[41] the proposed OFET structure shows a memory effect when, by applying a proper voltage pulse, charge carriers tunnel through the Parylene C and are blocked by the aluminum oxide, due to its larger dielectric constant. Here, a prolonged X-Rays exposure results in a similar effect. As a matter of fact, we already observed that X-Rays exposure results in leakage current variations related to charge carriers moving through the insulating layer (see Figure 2(c) and (d)). When a prolonged X-Rays exposure occurs, a part of these charges can be trapped in the Parylene C-aluminum oxide interface, generating the threshold voltage shift. The original threshold voltage value is recovered when charges are released by relaxation of the traps.

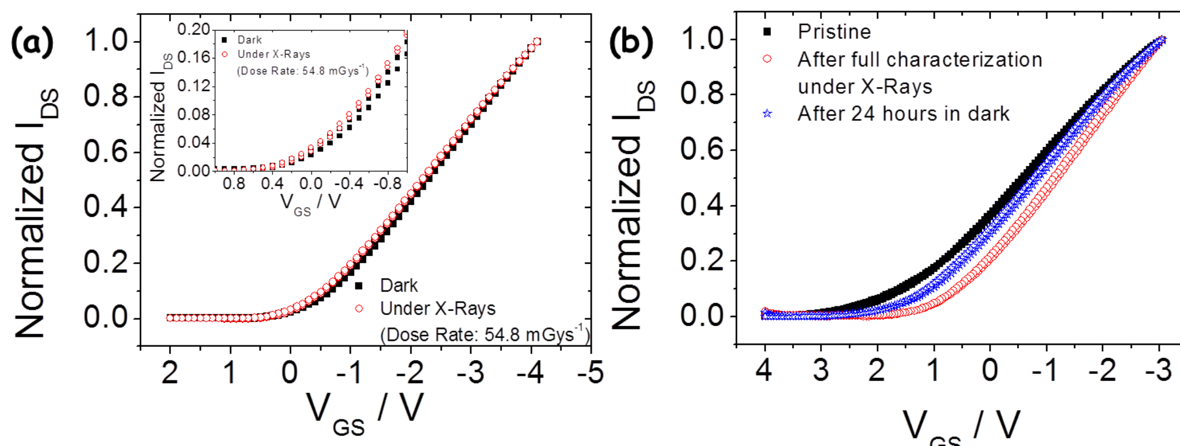

Figure 4. (a) normalized transfer characteristic curves acquired before (black squares) and during (red circles) X-Rays exposure at a dose rate of 54.8 mGys^{-1}; magnification on the threshold region is reported as inset. (b) radiation hardness test showing a normalized transfer characteristic curve of a device in pristine state (black squares), after X-Rays exposure with a total dose of 160 Gy (red dots) and after 24 hours kept in dark (blue stars).

3 Conclusions

In this paper, a low voltage OFET structure is successfully employed as a direct X-Rays detector fabricated on flexible plastic substrate. The reported results demonstrate that X-Rays directly produce significant, reversible variations in the output current of the transistor, depending on the X-Rays dose rate and, interestingly, on the operating point of the device. In particular, by varying the gate-to-source voltage drop, a tuning of the sensitivity can be performed: the response to X-Rays is negligible for devices maintained at an operative bias below the threshold voltage, while a very large sensitivity can be obtained by progressively increasing (in module) V_{GS}. Radiation hardness tests have been also reported, showing that non-relevant modifications are induced in the device response after a prolonged exposure to X-Rays.

The results presented herein represent a step forward in the field of organic photodetectors, as the possibility of employing an organic low voltage transistor for direct X-Rays detection has been demonstrated. Such a device exhibits a peculiar feature, i.e. the tunability of the sensitivity, that is unique among all the organic photodetectors reported in literature so far, also showing performances comparable to the state-of-the-art of two terminal, non organic

photodetectors. In this sense, it is also noteworthy that the reported performance has been obtained using a commercially available organic semiconductor with moderate charge carrier mobility, and, more importantly, without specific modifications that are normally employed to enhance X-Rays absorption. Therefore, as the low voltage operation of the presented transistor structure is independent of the choice of the semiconductor, it is possible to foresee further improvements in the device performance by employing materials engineered for X-Rays absorption. Taking into account the characteristic of the sensor in terms of response time, if we exclude some high frame-per-second medical applications, such as Computed Tomography, where a much faster response is needed, the proposed approach results particularly attractive for X-Rays detectors and imagers for medical diagnostic, industrial processes monitoring, and for security and cultural heritage applications.

4. Experimental Section

Device fabrication. Devices have been entirely fabricated over 175 μm-thick, biaxially oriented polyethylene terephthalate (PET) substrates. The surface of PET has been carefully cleaned by subsequent cycles of acetone, isopropyl alcohol and deionized water, then an aluminum layer (100 nm thick) has been deposited by thermal evaporation, at room temperature and in high vacuum conditions (5×10^{-5} Torr). The gate electrode was patterned by means of standard photolithographic process, using a 1% solution of hydrofluoric acid (HF) in water as etchant. The aluminum surface was then subjected to 1 hour exposure to UV-produced ozone, in order to let an aluminum oxide layer grow on it. By capacitive measurements, an average thickness of the aluminum oxide layer of 6 nm was estimated. The hybrid organic/inorganic dielectric layer, originally proposed in Cosseddu et al.,[28] has been then completed by depositing a layer of 150 nm of Parylene C all over the gate. A final gate capacitance of 18 $nFcm^{-2}$ was evaluated from capacitive measurements for the two-layer dielectric. Source and drain have been patterned by means of standard photolithography from

a unique, 80 nm-thick gold layer, deposited by thermal evaporation at room temperature and in high vacuum conditions (5×10^{-5} Torr). An interdigitated geometry (W/L = 50000 μm/45 μm = 1223) has been chosen. The semiconductor layer is a polycrystalline film of 6,13-Bis(triisopropylsilylethynyl)pentacene (TIPS-pentacene), deposited by drop casting from a solution in toluene (0.5wt%). Drop casting has been carried out onto a hot plate at 60°C, in order to let the solvent dry and enhance crystal growth. The thickness of the polycrystalline film is in the range of 100 nm in average, as extrapolated by Atomic Force Microscopy (AFM) imaging (see **Figure S2** in Supporting Information).

Electrical characterization. Electrical characterization has been performed using a dual channel Keithley 2614B SourceMeter, using a custom made Labview® software. All measurements have been carried out keeping the device in dark in a metal Faraday cage to reduce electrical noise and avoid light-induced photogeneration in the organic semiconductor.

X-Rays irradiation. Characterization under X-Rays has been performed using an X-ray broad spectrum provided by a tube with molybdenum target and accelerating voltage of 35 kV and dose rates in the range 2.5÷60 $mGys^{-1}$.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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