# Novel Ultraviolet and Ionizing Radiation Detectors Made From TiO<sub>2</sub> Wide-Bandgap Semiconductor

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Abstract—We describe the fabrication and characterization of a novel ultraviolet (UV) and ionizing radiation detector using a polycrystalline  $TiO_2$  wide-bandgap semiconductor as the active material. The detector geometry we have developed and tested is a polycrystalline  $TiO_2$  thin film with planar electrode contacts for signal pickup. Several prototypes were fabricated using two single-step techniques. We present the dc characterization of these devices and the signal response to the UV light and low-energy proton beam irradiation. The detector prototypes show excellent dc characteristics and fast ac signal response at bias voltage as low as 50 V.



Sensors Council

Index Terms—Ionizing radiation sensors, semiconductors, ultraviolet (UV) sensors, wide bandgap.

## I. INTRODUCTION

U V LIGHT detection is an important application in various industrial domains spanning optoelectronics, automotive, and astronomy instrumentation. In particle and nuclear physics, the detection of ionizing radiation emitted by fundamental processes is of prime importance. Silicon detectors with doped p and n regions and a depletion zone active area have traditionally been used in these domains [1]. However, harsh temperature conditions and protection from radiation damage are strong motivations to investigate alternative materials. Wide-bandgap semiconductors have gained interest in this field due to their low intrinsic noise and (often) high radia-

Manuscript received 11 November 2022; accepted 14 November 2022. Date of publication 29 November 2022; date of current version 29 December 2022. The work of Pradeep Sarin was supported in part by the Department of Science and Technology, India, under Grant SR/MF/PS-02/2014. The work of Kantimay Das Gupta was supported in part by the Bhabha Atomic Research Centre under Grant RD/0118/BARC-010-001. The associate editor coordinating the review of this article and approving it for publication was Dr. Richard T. Kouzes. (*Corresponding author: Pankaj Chetry.*)

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Digital Object Identifier 10.1109/JSEN.2022.3224809

tion tolerance [2]. The large bandgap significantly suppresses thermal noise generated by intrinsic carriers, giving these devices a simple structure that can be fabricated in one or two lithographic steps compared to the multistep lithography required for doping traditional silicon-based detectors. Diamond [3] and GaN [4] have been intensely studied as alternative semiconductors. The high cost and variability of the properties of chemically grown single-crystal diamonds from different producers have been a major obstacle in its widespread adoption in particle physics. GaN has found wider use for ultraviolet (UV) detection.

In this article, we present results from the fabrication and characterization of a UV and ionizing radiation detector with a novel active material  $TiO_2$ .

Section II gives details of our fabrication process. Section III provides data on the morphological characterization of the fabricated detector. Section IV provides the electrical performance characteristics of our detector prototype. The detectors show a very small dark current  $\sim$ nA and an excellent signal-to-noise ratio with a fast response at  $\mu$ s time scale.

#### **II. FABRICATION**

 $TiO_2$  thin films can be prepared by different techniques such as sol-gel [5], dip coating [6], spray pyrolysis [7], sputtering [8], chemical vapor deposition (CVD) [9], pulsed laser deposition (PLD) [10], thermal oxidation [11], and so on. We have prepared thin-film detector prototypes using the sol-gel and thermal oxidation methods. All samples prepared

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Fig. 1. Preparation flowchart of a TiO<sub>2</sub> film using the sol-gel method.

by the sol-gel and thermal oxidation methods are labeled SG# and TO#, respectively, where # is the sample number.

Fig. 1 shows our process flowchart for the sol-gel fabrication. Plain microscope glass slides are used as the substrate. The substrate is precleaned with a detergent, followed by ultrasonic immersion cleaning in a mix of acetone, isopropyl alcohol (IPA), deionized (DI) water, and dried with nitrogen flow. We have used titanium (Ti) isopropoxide (TTIP) as the TiO<sub>2</sub> precursor suspended in absolute ethanol and acetic acid. 1.80 mL of TTIP is added into 9.80 mL of ethanol that acts as the solvent. While the solution is being stirred, 0.3 mL of acetic acid is added into the solution. The obtained mixture is then stirred at 600 rpm for 6 h at room temperature to obtain the sol-gel. A few drops of sol-gel are put on the cleaned substrate with a syringe and spin-coated into a thin film at 2500 rpm for 45 s. The film is dried at 100 °C in air for 10 min. Adding a few drops and spin-coating is repeated multiple times to increase the film thickness. After every coating, the film is dried at 100 °C for 10 min. Finally, the TiO<sub>2</sub> thin film is annealed at 400 °C for 2 h in the open air. We prepared three samples SG1, SG2, and SG3 varying the number of spin coatings to 2, 3, and 4, respectively. The thickness and quality of the prepared film increase with the number of spin coatings [12].

For the thermal evaporation process, a semipolished quartz substrate was used, since it can tolerate the higher temperature required for rutile phase formation. The substrate is precleaned with a detergent, followed by ultrasonic immersion cleaning in a mix of acetone, IPA, and DI water. It is dried with nitrogen flow and heated for 2 min at 125 °C to evaporate any remaining residue. The substrate is loaded in a thermal evaporator (Smart Coat 3.0 thermal evaporator) for the deposition of Ti. Alfa Aesar Ti wire (99.99% purity) is used for thermal evaporation and the deposition is done under vacuum ( $<10^{-7}$  torr). The deposition thickness is continuously monitored with a separate quartz crystal monitor. The cavity volume of tungsten boats used in our thermal evaporator allows us to deposit a maximum of 100 nm Ti in one process. The Ti-coated sample is loaded into a tubular annealing furnace. The rise in temperature in the furnace is set at 13 °C/min up to 800 °C and then maintained at 800 °C for 10 h. A continuous flow of O<sub>2</sub> at 50 SCCM is maintained throughout the process. The deposition and annealing steps are repeated



Fig. 2. Preparation of  $\text{TiO}_2$  by thermal evaporation of Ti followed by annealing.

to increase the thickness of the film. We have prepared several samples TO1–TO8 following the above process in a class 10000 clean room. For TO6, the deposition–oxidation cycle of Fig. 2 was repeated four times (100 nm each) but the last cycle was aborted at  $\approx$ 36 nm due to the tungsten boat cracking. Hence, in TO6, the base Ti film thickness is  $\approx$  336 nm on a (1 × 10 × 10 mm) quartz substrate. For TO2, TO6, and TO8, the film thickness is 54, 336, and 136 nm, respectively, corresponding to one, four, and two deposition cycles. Most of the results reported below are for sample TO6.

#### **III. MORPHOLOGICAL CHARACTERIZATION**

The structural characteristics of the prepared samples were analyzed using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The optical properties were determined using a UV–VIS spectrometer.

#### A. X-Ray Diffraction

RIGAKU Smart Lab X-ray diffractometer, equipped with a 9-kW Cu rotating anode was used for high-resolution XRD of both SG and TO samples. The crystalline structure is studied in  $2\theta$  range from 10° to 80° using Cu- $K_{\alpha}$  source (1.541 Å).

The measured XRD spectra for SG# sol-gel samples with a different number of spin coatings (2,3,4) are shown in Fig. 3(a). XRD peaks of the SG# films correspond to A(101), A(004), A(200), A(105), and A(211) planes, respectively. From the peaks, it is clear that the polycrystalline phase of the film is anatase and well matched with standard JCPDS 021-1272 data. The dominant plane is (101) for all the samples.

The XRD spectra of TO# are shown in Fig. 3(b). XRD peaks of the TO# films correspond to R(110), R(101), R(111), R(211), R(220), R(002), R(301), and R(112) planes, respectively (JCPDS 021-1276). The dominant plane is R(101). The bottom XRD data trace in Fig. 3(b) (inset) is for TO2 before annealing at 800 °C in O<sub>2</sub>—it serves as a useful benchmark check of the amorphous Ti as deposited on the amorphous quartz substrate. The average grain size of the TiO<sub>2</sub> films calculated from the XRD peaks using the Scherrer formula [13] is in the range of 30–40 nm for the SG# samples and 20–25 nm for the TO# samples.

The continuum background seen in Fig. 3 is from the amorphous quartz substrate.

In the Rietveld refinement data of Fig. 3 done with Profex software [14], no residual peaks other than  $TiO_2$  are found for both SG# and TO# samples (the residuals are plotted in the bottom panels of Fig. 3). This indicates that  $TiO_2$  is the only phase present in both samples. We, therefore, conclude



Fig. 3. XRD data and Rietveld refinement analysis identifying polycrystalline phases of TiO<sub>2</sub>. (a) XRD data of SG# and Rietveld refinement of SG2. (b) XRD data of TO# and Rietveld refinement of TO6.

that with our simple fabrication process in each case, we can achieve complete penetration and oxidation of Ti for both SG# and TO# samples. The Rietveld refinement further performs a least-squares fit of the relative heights of the XRD peaks and gives the relative proportions of the different crystalline phases in the sample. From this analysis, we conclude that the films are primarily anatase for TO# and rutile for TO#.

The EDX test results for the samples are consistent with a 1:2 Ti:O stoichiometric ratio, but the quantitative interpretation of EDX is made difficult by the additional signal from the thick underlying  $SiO_2$  substrate.

#### B. Scanning Electron Microscopy

SEM studies were performed on a JEOL JSM-7600F instrument for the SG samples and on a Gemini GSM300 for the TO samples.

SEM images of the SG# samples in Fig. 4 indicate that the films are deposited in large clusters. The cross section SEM indicates film thicknesses of 0.6  $\pm$  0.2  $\mu$ m (SG1), 1.5  $\pm$  0.25  $\mu$ m (SG2), and 2.2  $\pm$  0.3  $\mu$ m (SG3). The thickness of the film increases with the number of coatings, and so does



Fig. 4. SEM images of samples SG1, SG2, and SG3 (feature sizes are highlighted as shown in the upper row of figures, whose calibration scale is different from the lower row).



Fig. 5. Topview SEM images of TO5. The base Ti film thickness for TO5 was 336 nm—the same as that of TO6 used for subsequent electrical characterization.

the variation of the thickness, as is expected from the sol-gel process.

The SEM images of TO5 shown in Fig. 5 indicate high uniformity. No cracks in the films are observed in the SEM images. The average surface roughness measured by AFM is very low  $\approx 2.46$  nm. Multiple depositions and O<sub>2</sub> annealing cycles do not introduce any discontinuity in the film. Several nuclear formations are observed with a maximum diameter  $\approx 1 \ \mu$ m.

From the XRD data and SEM images, we infer the polycrystalline phase of the prepared films.

## C. UV–Vis Absorption Spectroscopy

The UV–Vis absorption spectrum for the prepared samples was measured on a PerkinElmer Lambda 950 UV–Vis spectrometer.

The absorbance spectra of the SG# samples (see Fig. 6, inset) indicate absorption is maximum in the UV (300–340 nm) region for all three samples. The Tauc plot Fig. 6 is used to infer the optical bandgap of the material using indirect transitions from the edge of valence to conduction bands [16], [17]. The ordinate ( $\alpha h\nu$ , where  $\alpha$  is the absorption coefficient) has an exponent 0.5 since the material is disordered (polycrystalline) as shown by XRD. From the extrapolated intercept



Fig. 6. UV–Vis absorption spectra and Tauc plots for optical bandgap determination of (a) SG# and SG2 and (b) TO# and TO6. Note the subgap peaks at 416, 490, and 640 nm which most likely arise from interface defect levels (discussed in the text).

of the Tauc plot, we infer the optical bandgap of SG2 to be 3.15 eV.

The UV–Vis absorption spectrum of TO# (see Fig. 6(b), inset) is also maximum in the range (300–340 nm) and shows a corresponding optical bandgap of 2.45 eV.

The thermal oxidation process seems to result in a bandgap about 0.5 eV lower than the expected value of the rutile bandgap, while the XRD data clearly shows the formation of TiO<sub>2</sub> with no unreacted Ti. This could be a consequence of the formation of interband defect energy levels below the conduction band in the TO# samples. In the sol-gel process used for SG#, all the  $TiO_2$  nanoparticles are subjected to annealing at the end at a temperature that is much higher than the "drying" temperature of the layers during successive spin-coating steps. By contrast, in the thermal oxidation process used for TO#, we begin with pure Ti deposition in a vacuum, that is, in the absence of oxygen bonds. The oxygen incorporation is done layer by layer through the high-temperature annealing in O<sub>2</sub> as described above. In this process, the interface of the two layers may be a source of a large number of defects and dislocations leading to the formation of defect energy levels. Tauc's calculation assumes transitions between valence



Fig. 7. Devices prepared for electrical characterization. Upper row: photographs of SG2 (1.2  $\mu m$  TiO<sub>2</sub> on 2-mm glass) and TO6 (336-nm TiO<sub>2</sub> on 1.5-mm quartz). Lower row: device schematic with electrical contacts (not to scale).

and conduction band edges. In the presence of intermediate energy levels, the fit to  $(\alpha h\nu)^{0.5}$  is distorted by the subband absorption peaks resulting in a lower calculated bandgap for TO6 [17]. The arrows highlighting the absorption band edges in Fig. 6 (insets) corroborate this hypothesis: the primary absorption band edge is nearly the same in SG2 and TO6 (387 versus 399 nm), but TO6 has several subsequent subbandgap absorption peaks that distort the linear fit reducing the intercept with the x-axis in Fig. 6(b).

Summarizing the morphology, it is clear that we have been able to fabricate polycrystalline thin films of pure widebandgap TiO<sub>2</sub>, consistent with results reported earlier for sol-gel fabrication [18]. Our novel single-step thermal oxidation technique produces similar quality polycrystalline films of TiO<sub>2</sub>.

SG2 and TO6 show the best morphology, so most of the electrical tests reported in Section IV were performed on these samples.

#### IV. ELECTRICAL CHARACTERIZATION

Ti/Au (20/80 nm) metal contacts were deposited using a simple shadow mask on the top surface of the prepared samples to ensure good adhesion and a low-resistance Ohmic contact [19]. Hence, each device under test (DUT) is a metal-semiconductor-metal (MSM) device as shown in Fig. 7. Charge transport occurs sideways in the thin TiO<sub>2</sub> layer. The electric field driving the charge transport is strongest in the gap between the electrodes.

All electrical characterizations in the lab were done in a vacuum chamber probe station at  $\sim 7 \times 10^{-5}$  torr as shown schematically in Fig. 8.

## A. DC Electrical Characteristics

The dc electrical characteristics were measured under high vacuum using a Keithley 6487 picoammeter/voltage source. As shown in Fig. 7, SG2 has a single gap of  $\approx 0.5$  mm between



Fig. 8. Photograph (above) and schematic (below) of the experimental setup in a vacuum probe chamber for electrical characterization of samples (not to scale). As noted in the text, a Keithley 6487 picoammeter was used for dc *I*-*V* characterization. A self-made LF411 transimpedance amplifier (TIA) with an independent bias supply was used for dynamic ac signal characterization. All signal cables used are triax/coax shielded to avoid noise pickup.

the metal electrodes. TO6 has interdigitated electrodes with a semiconductor gap of  $\approx 1.3$  mm between the electrical contacts, giving a larger effective active area.

The *I–V* characteristics of our DUTs under dark and UV illuminated conditions are shown in Fig. 9. The devices exhibit Ohmic contacts across the metal–semiconductor junctions with a dark current <0.5 nA up to a bias voltage of ±100 V. From the dark current and sample dimensions, we infer sheet resistance  $R_s = 2.1 \times 10^{13} \Omega$  (SG2),  $R_s = 5 \times 10^{12} \Omega$  (TO6) and bulk resistivity  $R_b = 2.1 \times 10^9 \Omega \cdot \text{cm}$  (SG2),  $R_b = 1.5 \times 10^8 \Omega \cdot \text{cm}$  (TO6), consistent with values reported for TiO<sub>2</sub> films grown by other methods [20]. The higher bulk and sheet resistance of SG2 compared to TO6 is also consistent with its larger measured bandgap.

The substrate is UV transparent in all cases, so the active area of the sample is illuminated from below by a UV LED through a small collimation hole in a metal aperture. The UV LED Vishay VLMU3510-365-130 has peak emission



Fig. 9. -V characteristics measured in dark and UV illuminated conditions.

wavelength  $\lambda = 367$  nm (3.37 eV), that is, larger than the optical bandgap of the material.

I-V characteristics of the DUT's under UV illumination of 200 mW radiant power from the LED are also included in Fig. 9. The dc current increases to a few  $\mu$ A in the same bias voltage range under UV illumination. Error bars in the I-V characteristics include statistical and systematic components. The statistical error is estimated from the standard deviation of a few current measurements at a voltage set point. A systematic error is caused by charge trapping at grain boundaries and the electro-migration of defects under high-bias fields. The latter effect manifests itself as an offset between the I-V characteristic in the upward versus downward sweep of the bias voltage.

# B. AC Signal Characterization

We have characterized the charge collection using illumination by pulsed UV light. We also measured charge collection with an ionizing beam-induced current technique.

1) Response to UV Photons: The vacuum probe station setup shown in Fig. 8 is also used for ac characterization. In this case, the UV LED mounted under the sample is switched using a 50-kHz 50% duty cycle square current pulse, triggered from a function generator (Tektronix AFG2021). The sample is biased from an independent high-voltage power supply (Stanford Research PS325). The photocurrent is recorded with an oscilloscope (Tektronix MDO4054-3) at 500 MS/s through a homemade LF411-based charge amplifier. The gain of the amplifier was independently calibrated to be 186 mV/pC at 50 kHz. Fig. 10(a) and (b) shows the response of SG2 and TO6 to pulsed UV illumination at 50 kHz. The bias voltage applied in each case is 50 V. The trigger voltage shows the status of the UV LED. The photocurrent is clearly correlated with the light incidence. We observe a photoresponse at the time scale of 20  $\mu$ s. The magnitude of the photocurrent in TO6 is larger than that in SG2 due to a combination of more uniform film quality and the larger active area.

The pulse response shown in Fig. 10 is fit to an exponential curve with the time constant characteristic of the charge



Fig. 10. Sample response to UV LED in vacuum setup of Fig. 8. Note that the rising edge of the trigger pulse corresponds to the UV-LED turning ON. (a) SG2 response as a function of time to UV LED. (b) TO6 response as a function of time to UV LED.



Fig. 11. Effect of bias voltage on the signal amplitude.

amplifier to obtain the photoresponse in pC as a function of bias voltage as shown in Fig. 11. The photoresponse shows a slight increase at higher bias, with an appreciable value even at zero bias. We surmise this photoresponse at zero bias is due to the same reasons as discussed for the dc



Fig. 12. (Above) SG# mounted on the ceramic sample holder and (below) vacuum chamber used for proton beam irradiation. The proton beam enters from the left.

I–V characteristics earlier: residual charge trapped at grain boundaries in the detector and some as yet undetermined long recombination lifetime of the photogenerated charge carriers. Summarizing the UV photoresponse, we observe the responsivity of our TiO<sub>2</sub> devices to be  $5.0 \pm 0.4$  mA/W (SG2) and  $4.8 \pm 0.3$  mA/W (TO6) at bias voltage 30–70 V at fast (10- $\mu$ s) time scale. These values compare favorably with typical values reported earlier for GaN/AlGaN MSM UV photodetectors [21]. Our fabrication technique has the advantage of being a simple one-step process scalable to largearea detectors.

2) Signal Acquisition in Low-Energy Proton Beam: Typically ionization energy three times larger than the semiconductor's bandgap is required to produce an electron-hole pair [22]. Since the active area is extremely thin (~1.5  $\mu$ m for SG2 and ~336 nm for TO#), the energy deposited by single  $\alpha$  or  $\beta$  particles in such a layer is insufficient to produce an appreciable charge. Simulations [23] indicate that low-energy protons deposit significant energy (~70 keV/ $\mu$ m in TiO<sub>2</sub> for 1-MeV proton).

We have tested our samples in the direct beam path of a proton beam tandem accelerator at the Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, India. We have used a proton beam of energy 1 MeV at a beam current 300 nA



Fig. 13. SG2 response to proton beam fluence. (a) Time-domain response of SG2 in the proton beam scan. The period marked A–B corresponds to maximum beam overlap with the  $TiO_2$  active area as shown in the inset above. (b) Signal amplitude as a function of applied bias voltage (polynomial fit to guide the eye).

for our tests. The proton beam is a continuous dc scanning beam. Fig. 12 shows the experimental setup used for beam irradiation studies. The ceramic sample holder has slots to load four samples, but sample SG2 was primarily used for these beam irradiation tests due to geometrical constraints of holder movement and beam alignment. The chamber is evacuated to  $\sim 10^{-7}$  torr for the beam irradiation studies. Bond wires are kept short, and coaxial cables are used throughout the signal chain to minimize noise pickup.

The results of our proton irradiation measurements are shown in Fig. 13(a). The detector is capacitively coupled through a bias-T and the signal is large enough to be readout directly into a  $50 - \Omega$  terminated DSO. The time-domain response of the detector can be understood based on the beam dynamics. The beam spot size is 10 mm in diameter. The

beam scans a target area of  $30 \times 30$  mm at a frequency of 1.015 kHz at an angle of 45°. The beam scanning cycle repeats every 40 ms. Thus, the beam spot crosses the active area of the detector every 0.985 ms. The modulation observed in the signal correlates with the 1.015-kHz beam scanning frequency across the SG2 active area. Due to the diagonal scan, the maximum flux of protons incident on the TiO<sub>2</sub> active area is between the points highlighted as A and B in the inset of Fig. 13(a). Correspondingly, the signal rises to a maximum stable value between the time markers A and B in Fig. 13(a). Conversely, the signal falls to a minimum every 40 ms at the end of the beam scanning cycle when the beam spot is far away from the active area.

The bias voltage in the device is varied from 0 to 100 V. Average signal amplitude in the maximum beam flux incidence period A–B as a function of the bias voltage is plotted in Fig. 13(b). The signal amplitude increases with bias voltage and reaches a maximum value by 50 V bias, consistent with our benchtop tests with UV irradiation shown earlier in Fig. 11.

Unlike the collimated UV light source for photoresponse, the proton beam is incident over the entire TiO<sub>2</sub> including areas under the metal electrodes. Therefore, proton energy loss and electron-hole pair creation occurs in the whole semiconductor. Considering the effect of a bias field that drops off as  $\sim 1/x$  with x = 0 being the midpoint of the gap between the electrodes, we consider the effective area to extend up to  $x = \pm 1.5$  mm as shown in Fig. 13(a). In this case, we infer a charge collection efficiency of  $69\% \pm 5\%$ , comparable to similar measurements for pCVD diamond detectors [24] and GaN PIN alpha radiation detectors [25].

# V. SUMMARY

We have developed a novel type of UV and ionizing radiation detector using a wide-bandgap semiconductor TiO<sub>2</sub> whose use in this domain has not been explored as far as we know. Our fabrication methods are simple, cheap, and scalable. It opens up a possibility for large area coverage with semiconductor-based detectors. Our MSM detector geometry has a submicrometer thickness active area, so the material budget for placing such detectors in a detector environment is only limited by the thickness of the inert substrate. We have performed dc and ac signal characterization in a well-shielded setup at a high vacuum to preclude the possibility of noise pickup and stray parallel conductance paths through air, which is always a concern for such high resistivity films. The detector response to pulsed UV irradiation in vacuum at the time scale of tens of  $\mu$ s has been tested and found to be consistent with proton beam induced current at low bias voltage up to 50 V. Further studies need to be done on the detailed charge transport within the active area to determine electron/hole mobility. Other fabrication methods including PLD can be explored for creating single-crystal detectors, albeit with the size restrictions inherent in those techniques. We have shown that even in polycrystalline form,  $TiO_2$  is a promising material as a UV and solid-state ionizing radiation detector.

#### ACKNOWLEDGMENT

The authors thank IIT Bombay, Mumbai, India, for the use of the fabrication and characterization equipment mentioned in this article. In particular, they thank Prof. Raghava Varma for his support. They thank all the support staff and operators at the Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, India, for their timely help in accelerator operations to obtain beam fluence results.

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