Indium Oxynitride (InNO) Radiation Sensors Calibration

Marina Sparvoli, R. K. Onmori, F. O. Jorge, and Mario Alexandre Gazziro

Abstract-Aerosol effects evaluation on Earth's radiation budget demands knowledge of its concentration, spatial, and temporal distribution. Therefore, long-term measurements of aerosol properties in different regions of Earth are very important. In this paper, indium nitride (InN)- and indium oxynitride (InNO)-based photodiodes calibration was realized. A pyranometer was used in this paper. Calibrated InN and InNO Schottky photodiodes will be used for aerosol detection. According to the calibration tests, it was found that the most suitable sensors for the continuation of an aerosol statistical study in the atmosphere are those made with the InNO deposited with oxygen 10% and 20%; their correlation coefficient values are almost one. Besides, their measures do not reach the saturation point. In future, we intend to use these devices in a quantitative study of aerosols present in the center of a large city, to get a sense of human interference in the atmosphere composition and its consequences.

Index Terms—Indium oxynitride, indium nitride, optoelectronic and photonic sensors, photo detectors, thin film sensors.

I. INTRODUCTION

FOR solar technologies application, it is very important to know the local solar radiation data. Besides the global (or total) irradiance values, direct and diffuse components are often also necessary. In practice direct radiation can be measured by pyrheliometers or diffuse component can be determined from a pyranometer [1], [2].

For this purpose, monitoring systems of atmospheric aerosol effect have been implemented in different sites around Earth. In general, these systems use the sun photometry technique to estimate the aerosol optical depth. But the accurate retrievals of this technique estimate are necessary to characterize the aerosol effect.

Using indium nitride (InN) and indium oxynitride (InNO) material is possible to build a Schottky photodiode. This photodiode can detect and measure the solar radiations. A pyranometer was used in this study to make the calibration of these Schottky photodiodes and will be use for aerosol detection. InNO is a new class of materials with optical,

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Digital Object Identifier 10.1109/JSEN.2017.2670080

TABLE I Deposition Parameters

Target	99,999% Indium, 4 in. diameter
Substrate	Silicon p-type
Substrate distance	6 cm
Base pressure	1.33 Pa
RF Power	250 W
O2 gas concentrations	0%, 10%, 20%, 50%, 80%, 85%.
Deposition time	1 hour

mechanical and electrical properties potentially interesting for industrial applications. Numerous properties of the InNO, such as the refractive index and the photoelectric effect intensity, vary according to the proportion of oxygen and nitrogen contained in the formed film. InNO is transparent, it has a wide band gap (around 3 eV) and nearest indium oxide (3.4 eV) [3], [4]. Comparing with photoconductor and metal semiconductor metal (MSM) photodiode, a Schottky photodiode has many advantages in aspects of high quantum efficiency, high response speed, low dark current, high UV/visible contrast, and possible zero-bias operation. This is an original application for material; in literature there are examples as filters or gas sensors [5], [6].

II. EXPERIMENTAL

To built the photodiode, initially, a cleaning process $(4H_2SO_4 + 1H_2O_2)$ followed by HF dipping (2%), was performed in Si p-type substrate (75 mm). In Table I is showed deposition parameters of InN and InNO thin films. Deposition processes were performed in a home build magnetron sputtering system, using a four-inch pure In target (99.999 %), nitrogen (99.995 %) and oxygen (99.998 %) as plasma precursors. Pressure was kept constant in 1.33 Pa and RF power (13.56 MHz) was constant 250 W. Six different oxygen gas concentrations were used in deposition processes.

In photodiode fabrication, aluminum circle contacts were deposited at wafer front side by evaporation in order to obtain 300 nm thick layer. At backside of wafer, a 500 nm thickness aluminum film was evaporated.

A Software named "traçador de curvas II" written in Labview and an analog/digital (A/D) board NI6008USB from National Instruments were used to measure the photodiode. Sensor feed voltage can be adjusted between 0 and 5 V and program can adjust the sample rate in milliseconds. The results are given through a screen and the sensor response, the intensity of solar radiation, is measured by the electrical current across the sensor.

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Manuscript received December 9, 2016; revised February 10, 2017; accepted February 11, 2017. Date of publication February 16, 2017; date of current version March 22, 2017. This work was supported by Fapesp and CNPq. The associate editor coordinating the review of this paper and approving it for publication was Dr. Carlos R. Zamarreno. (*Corresponding author: Marina Sparvoli.*)

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Fig. 1. Instruments for data acquisition.



Fig. 2. Schematic of experiment setup.

NI6008USB from National Instruments realized data acquisition from sensors (figure 1). It has the ability to analyze three sensors simultaneously. The result is a current (from the sensor) through a circuit formed by the 10 kOhm resistor in series and it is used to convert the current into a voltage that is read by the analogical input board, as the diagram of Figure 2.

A Pyranometer model CMPII Thies clima is connected to the Datalogger model DLx-MET to make data acquisition. With the acquired data through the pyranometers is possible to make the comparison with data from the sensors.

The equipment data are obtained in W/m^2 and it is possible to make a calibration curve to obtain the intensity of radiation flux as a function of time. This procedure is made simultaneously with the acquisition of data from sensors (curve of voltage versus time).

Results are acquired simultaneously. Using these data obtained by pyranometer and InNO sensors or InN sensor is possible to obtain a "W/m² in function of Volts" graph (pyranometer data in function of data sensors) and the sensor can be calibrated (in W/m²).

Linear and angular coefficient generate a calibration equation for each sensor. They can be used as a standard for calibration of other sensors manufactured with the same conditions. It is possible to obtain the chi-square test value of the calibration curves to confirm the results relationship.

TABLE II Sensors Characteristics

Sample	Thickness	Area (cm ²)
	(nm) [3]	
Pure InN	825.2	3.0
$10\% O_2$	222.5	8.8
$20\% O_2$	220.0	9.6
$50\% O_2$	226.9	9.6
$80\% O_2$	93.7	9.6
85% O2	117.0	9.1



Fig. 3. InNO sensors measurements: in top InNO 10% O_2 , in middle InNO 20% O_2 and in botton InNO 85% O_2 . Voltage corresponds to radiation flux intensity as a function of time.



Fig. 4. Pyranometer measurement during sunset. This result is similar to behavior of InNO sensors in same period (Figure 3).

III. RESULTS

Six sensors were built and used. In table II, characteristics of these sensors were showed.

At first study, ambient radiation characteristics curves were obtained in function of time. Measurements were performed by photodiodes and a pyranometer, which will serve for the calibration of manufactured InN and InNO devices.

In figure 3, the voltage versus time for oxynitride indium sensors deposited with 10%, 20% and 85% oxygen is showed. It was observed that deposited sensors with low oxygen percentage had more sensibility and response to ambient radiation spectra.

In figure 4, in graphic obtained through a pyramometer, it was obtained the radiation in function of time.

Pyranometer measurement was performed simultaneously with measurements of manufactured InNO sensors.



Fig. 5. InNO photodiode deposited with 0% O₂ calibration.



Fig. 6. InNO photodiode deposited with 10% O₂ calibration.

It was concluded that curve behavior of sensors is similar to pyranometer behavior. Because this fact, it was possible to perform a calibration of the data sensors (V) according to data from pyranometer (W/m^2) .

The calibration was made for InNO photodiodes deposited with oxygen (devices deposited with 0%, 10%, 20%, 50% and 80% O₂). The results are shown in the graphics below (figure 5 to figure 9).

Sensors deposited with 0%, 10% and 20% oxygen were measured at a temperature of 23 °C, humidity 50\%, on a cloudy day, in afternoon period.

In pure indium nitride case, many points are dispersed and far from line set by linear fit. Moreover, there are points with high standard deviation (31% of measured value). This behaviour results in a lower correlation coefficient if compared with the other samples.

The points obtained for the devices fabricated with InNO deposited with 10% oxygen are in close proximity of the straight line. Moreover, the standard deviation values of the points are low, below 2% of the experimental values. This fact results in a correlation coefficient of 0.99.

Correlation coefficient for indium oxynitride deposited with 20% oxygen sample is also close to value 1. The measured



Fig. 7. InNO photodiode deposited with 20% O₂ calibration.



Fig. 8. InNO photodiode deposited with 50% O₂ calibration.

points are aligned and have a small standard deviation below 3%.

Sensors with 50% and 80% oxygen were measured at a temperature of 27 °C, humidity 54% and on a sunny day with few clouds in morning.

The InNO photodiode deposited with 50% oxygen showed a correlation coefficient (0,93) lower than the deposited devices with 10% and 20% oxygen. Measured points oscillated more around obtained Linear Fit line.

In figure 9 (InNO sensor deposited with 80% oxygen) is observed high values for standard deviation in the measured data. This indicates that there are noises, radiation oscillates in a wide range and the sensor does not have the appropriate response. Response is very low in volts, even at high intensities of solar radiation.

In this calibration, it is evident that the sensor does not have optimized sensibility if compared with others. This sensor is the less indicated for use in statistic study.

From the Linear Fit line obtained for each sample, equations were extracted for calculation of theoretical points for all sensors. The calibration equations are presented in table III.

Based on the calibration theoretical equations, it was obtained a graphic with the results for each device, in order to make a comparison.



Fig. 9. InNO photodiode deposited with 80% O2 calibration.

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Sample	Correlation coefficient	Calibration equation
Pure InN	0.93	y=-329.80 (7.39) + 82.63 (1.77)x
$10\% O_2$	0.99	y=-152.28 (9.66) + 207.89 (7.57)x
$20\% O_2$	0.99	y=-43.01 (5.37) + 202.99 (6.96)x
$50\% O_2$	0.93	y=28.50 (1.7) + 803.5 (20.09)x
80% O2	0.67	v = -13043(22846) + 246704(121757)x

TABLE III CALIBRATION EQUATIONS FOR SENSORS





Fig. 10. Graphic with the results for each device based on the calibration theoretical equations.

Angular coefficient was calculated and results were showed in Table IV. The slope of each line provides an indication of which sensor is the most appropriate to implement aerosols measures.



Fig. 11. Indium oxynitride and indium nitride sensors Spectral Response.



Fig. 12. Indium oxynitride and indium nitride sensors quantum efficiency.

The calibration theoretical straight lines for the photodiodes deposited with 10% and 20% oxygen exhibit similar behavior (Figure 10). They are the most indicated for the study of aerosols in the atmosphere of a particular region.

The most sensitive sensor is manufactured from pure InN; however, experimental curve saturates at higher radiation fluxes. It is probably the response of this sensor is more effective because the indium nitride respond to infrared radiation, producing an increase in vibration of phonons, which interferes with the measured voltage.

For InNO photodiodes deposited with 50% and 80% oxygen, the theoretical straight lines have an abrupt increasing, indicating an ineffective response (in Volts) to the radiation intensity.

As result of sensors elements calibration it is possible to determine its applicability in radiometers, which could be used to measure the radiation in different sites of a city. With this application, it is possible to calculate the aerosol dispersion and to verify the pollution scale in the region.

It was observed that there is a variation of measured radiation during test period, depending on the variation in brightness due to presence of clouds.

An analysis of spectral response was performed for each sensor (Figure 11) in laboratory. There is a higher current intensity in the near infrared region. As a function of spectral response and area can be calculated quantum efficiency (Figure 12).

The quantum efficiency describes the response of the sensor to different wavelengths of the electromagnetic spectrum.

In the case of measurements of quantum efficiency, the greater response is seen for the wavelength of 1050 nm in the near infrared region for InNO sensors. Devices fabricated in this work have a higher sensitivity between 950 and 1050 nm. InNO thin film with 20% oxygen sensor has higher response.

A comparison made between all sensors leads to the conclusion that the best sensor is manufactured with InNO containing 20% oxygen. Its quantum efficiency spectrum shows that it presents the most efficient results for response to different wavelengths. If a comparison is made, the InNO sensors deposited with 10% and 20% oxygen have better performance than the sensor deposited with 80% oxygen. One possible explanation may be given as a function of materials band gap. InNO deposited with large amount of oxygen present wide band gap [3], being closer to an insulator than a semiconductor. Band gap interferes with sensor sensitivity. From this parameter depends on which range of electromagnetic spectrum the sensor will be able to detect. If it is desired that the sensor only detects the visible range, value of band gap of the material should be lower than a sensor that has capability to detect radiation in the ultraviolet range. InNO deposited with 80% oxygen has a 3.04 eV band gap. InNO deposited with 10% and 20% have 2.47 eV and 2.65 eV, respectively [3].

IV. CONCLUSION

According to the calibration tests, it was found that the most suitable sensors for continuation of a statistical study of aerosol in the atmosphere are those made with InNO deposited with 10% and 20% oxygen.

These devices are innovative because they are manufactured based on new materials. InNO does not yet have many applications. There are few works with results describing its electrical, structural and optical characteristics. For example, in Nukeaw et al. patent, an application of indium oxynitride as a filter for different wavelengths is described [7].

Circuit used for sensors operation is simple, so it is possible to reduce costs to implement the sensors for data acquisition.

In future, we intend to use these devices in a quantitative study of aerosols present in the center of a large city, to get a sense of human interference in the composition of the atmosphere and its consequences.

ACKNOWLEDGMENT

The authors would like to thank the Laboratório de Sistemas Integráveis da Universidade de São Paulo for measurements, to Mr. Nelson Ordonez for technical support.

REFERENCES

- P. Bajons, U. Wernhart, and H. Zeiler, "A sensor element for direct radiation measurement," *Solar Energy*, vol. 63, no. 2, pp. 125–134, 1998.
- [2] B. Plesz, Á. Földváry, and E. Bándy, "Low cost solar irradiation sensor and its thermal behaviour," *Microelectron. J.*, vol. 42, no. 4, pp. 594–600, 2011.
- [3] M. Sparvoli, R. D. Mansano, L. S. Zambom, and J. F. D. Chubaci, "Optical and electrical properties of sputtered InNO thin films," *Phys. Status Solidi C*, vol. 9, no. 6, pp. 1384–1387, 2012.
- [4] J. T-Thienprasert, J. Nukeaw, and A. Sungthong, "Local structure of indium oxynitride from X-ray absorption spectroscopy," *Appl. Phys. Lett.*, vol. 93, no. 5, p. 051903, 2008.
- [5] H. Steffes *et al.*, "New In_xO_yN_z films for the application in NO₂ sensors," Sens. Actuators B, Chem., vol. 77, nos.1–2, pp. 352–358, 2001.
- [6] A. Sungthong, S. Porntheeraphat, A. Poyai, and J. Nukeaw, "An extreme change in structural and optical properties of indium oxynitride deposited by reactive gas-timing RF magnetron sputtering," *Appl. Surf. Sci.*, vol. 254, no. 23, pp. 7950–7954, 2008.
- [7] J. Nukeaw, S. Porntheeraphat, and A. Sungthong, "Nanocrystal indium oxynitride thin film optical filter," U.S. Patent 20090015907 A1, Jan. 15, 2009.

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