Communications: SIF Congress 2020

# Segmented hyperpure germanium detector

W. RANIERO<sup>(1)</sup>(\*), G. MAGGIONI<sup>(1)</sup>(<sup>2)</sup>, S. BERTOLDO<sup>(1)</sup>(<sup>2)</sup>, S. CARTURAN<sup>(1)</sup>(<sup>2)</sup>, F. SGARBOSSA<sup>(1)</sup>(<sup>2)</sup>, C. CARRARO<sup>(1)</sup>(<sup>2)</sup>, E. NAPOLITANI<sup>(1)</sup>(<sup>2)</sup>, D. R. NAPOLI<sup>(1)</sup> and D. DE SALVADOR<sup>(1)</sup>(<sup>2)</sup>

- <sup>(1)</sup> INFN, Laboratori Nazionali di Legnaro Viale dell'Università 2, Legnaro (PD), Italy
- (<sup>2</sup>) Dipartimento di Fisica e Astronomia, Università degli Studi di Padova Via Marzolo 8, Padova, Italy

received 15 January 2021

**Summary.** — The segmentation process of high purity germanium detectors (HPGe) is here described. A photolithography technique is used to obtain a segmented gamma detector. The photolithography is applied on a planar square detector where the junction/contact region is covered with a gold thin film. Small segmented detectors are characterized to verify the quality of the photolithography process. Optical and scanning microscopy measures (SEM) are used to analyse the quality of the segmentation and to improve the photolithography process parameters. Finally, the leakage current of the diode and the passivation resistance between electrodes are studied and discussed.

#### 1. – Introduction

The use of segmented Ge crystals, as in the Advanced GAmma Tracking Array (AGATA) case [1-3], allows to identify single gamma-ray interactions in the volume of the Ge crystals as well as the determination of the deposited energy combining excellent energy resolution with fine position resolution [4]. In AGATA, the entire lateral surfaces of the crystal are electrically divided into 36 segments and a further 1 core contact [5]. One of the most important critical fabrication step is the surface passivation of the detector. The passivation has two different functions: the first is to form a chemical barrier for external agents like humidity, condensable vapour, etc., the second is to form an electrical barrier which isolates contacts/junctions or segments from one another. There are different passivation methods that have been successfully used, such as the use of a layer of  $SiO_2$ , amorphous Ge [6,7], or chemical passivation (methanol, sulfide, hydride termination) [8] onto the detector surface. If there is a charge accumulation on the passivated surface, the electric field in the detector could be distorted and the surface leakage current could increase, thus limiting the collection of charge. This effect generates the so-called *dead layer* that decreases the active volume of the germanium detector and also affects the energy resolution through charge collection fluctuations. Regarding the surface leakage current in the gap between segments, this happens at the edges of the

Creative Commons Attribution 4.0 License (https://creativecommons.org/licenses/by/4.0)

<sup>(\*)</sup> E-mail: walter.raniero@lnl.infn.it



Fig. 1. – Manufacturing steps of a small planar diode. (a) shallow Sb junction by PLM, (b) gold thin film deposition, (c) contact/junction segmentation.

junction where large voltage gradients must be supported over small distances. Also for this reason, it is very challenging to create a good passivation on this area. A possibility to reduce this surface leakage is to bury the junction edge on the germanium itself, like in the silicon detectors case [9].

## 2. – Experimental

The p-HPGe (100) material (Umicore,  $\leq 1 \ 10^{10} \text{ atoms/cm}^3$  impurity concentration), that is used for the photolithography process, is cut in two different sample sizes. The two selected areas are  $10 \times 10 \text{ mm}^2$  and  $35 \times 35 \text{ mm}^2$ . The sample thickness is 2 mm. These samples are lapped with a ceramic slurry up to  $3\mu$ m particle of alumina and deionized water. Afterwards, the samples are etched with acid solution (3:1) HNO<sub>3</sub>: HF (electronic grade, Carlo Erba) for 5 minutes to remove the mechanical defects introduced by the cutting and manual lapping processes [10]. The raw HPGe material is processed to obtain a  $n^+$  contact by pulsed laser melting (PLM) of antimony [11-13], and a  $p^+$ contact with boron implantation. The PLM process [11, 14] is used as a diffusion [15]and dopant activation process compatible with HPGe [16]. When the contact/junction on the diode is produced, a thin layer of gold  $<100 \,\mathrm{nm}$  is deposited by RF Magnetron sputtering on the  $n^+$  junction. This layer is necessary to perform a segmentation of the  $n^+$  junction, because the gold layer resists acid etching of germanium. The two HPGe diodes (fig. 1) produced with  $10 \times 10 \text{ mm}^2$  have two contacts and a guard ring, the gap between the two segments is  $0.4 \,\mathrm{mm}$  and  $0.2 \,\mathrm{mm}$ , respectively [12]. The third detector presents a size of  $35 \times 35 \,\mathrm{mm^2}$ , six segments and guard ring. In this case, the gap between the segments varies between  $0.1 \,\mathrm{mm}$  and  $0.8 \,\mathrm{mm}$ . The reason of this choice is to study the best performance of gap-passivation, that allows to reduce the cross talk between segments in a future detector  $(10^{-5}$  in AGATA detectors [5]). The gap size and the contact/junction thickness are involved to improve the reduction of the dead layers in the detector. Therefore, there is a higher efficiency and resolution of the gamma ray detector with thin dead layers [6, 9].

The photolithography segmentation process [17] is divided into different steps: 1) a thin layer < 1  $\mu$ m of positive photoresist (Microposit S1813) is deposited on top of the PLM junction (with thin gold layer) by spin coating; 2) a soft bake is applied to evaporate the solvent on the photoresist (130 °C, 1 minute); 3) UV irradiation for 50 s (UVA lamp, 350–400 nm) using an acetate mask; 4) the irradiated photoresist is removed by tetramethylammonium hydroxide (Microposit); 5) a long soft bake (130 °C 15 minutes) is carried out to hardening the photoresist on gold layer; 6) a gold etching (KI/I<sub>2</sub>, MicroChemicals) is performed to remove the gold between the segments; 7) the sample is immersed in a hot acetone bath to remove the photoresist on the gold layer in the contact and on the guard ring; 8) the diode is etched with 3 : 1 HNO<sub>3</sub> : HF acid bath for <10 s to remove the junction diffusion on the gap, while the p<sup>+</sup> boron contact is protected by Kapton tape; 9) finally, the segmented detector is quenched with a methanol passivation (Erbatron, Carlo Erba).



Fig. 2. - (a) Sidewall of resist along the edges and corners of square sample. (b) Residual gold layer along the corner and the edge after gold etching segmentation step. (c) Optimization of photolithography segmentation, two segments with gap 0.4 mm and guard ring. The lateral surface and the trenches are etched with germanium acid bath (3:1). (d) p-HPGe multistrip detector with incremental gap size.

The segmented detectors are characterized with an optical microscope (Optika) and SEM (Tescan Vega 3XMH). It is thus possible to analyze the result of the previously described process and in particular the photolithography resolution. Finally, the detectors are tested in diode configuration with an I-V tracer (Keithley 237) to evaluate the leakage current and the passivation resistance.

## 3. – Results and discussion

The photolithography segmented diodes are analysed with optical microscopy to verify the effectiveness of the production process. The observation highlighted the typical problem of the spin coating photoresist deposition, where there is a material stack along the edges of the sample (fig. 2(a)). Especially in the case of squared samples, the air turbulences at the edges and above all the corners of the substrate causes an accelerated drying of the resist and a sidewall of resist [17, 18].

If there is a photoresist accumulation on the gold layer, it will be more difficult to remove it with a hot acetone rinsing. Unfortunately, the result is that many gold layer areas remain on the segmented surface after the gold etchant process step (fig. 2(b)). This generates a region where the antimony diffusion under the gold layer material will not be removed by the 3:1 germanium etching, thus creating a floating contact when the diode works in detector mode configuration, where only the electrodes and guard ring are connected to FETs. This detector could have a much larger dead layer and therefore also a greater loss of charge, with a resulting loss of resolution. Another aspect is that in these regions it is very complicated to make a good uniform passivation, which causes surface leakage current on the diode. The optimization of the photolithography process parameters (time and rotation speed), especially with regard to the spin coating technique, permitted to obtain very defined and clear segmentation (fig. 2(c)). The used spin coating parameters are: 1)  $0.3 \,\mathrm{ml}$  of positive photoresist; 2) rotation speed (500 rpm for 5 s and 4000 rpm for 40 s with an acceleration  $\leq$ 4000 rpm/s). The quality result of the photolithography segmentation is confirmed also with SEM analysis (fig. 3(a)) [18].

Another detector, a p-HPGe multistrip segmented detector is analized below (fig. 2(d)). The n<sup>+</sup> contact is segmented with 6 electrodes and a guard ring. The segmentation gap is incremental from one electrode to another from 0.1 mm up to 0.8 mm. Thanks to this segmentation, we will study and optimize the gap size between the electrodes. This gap size optimization is a very important development in the gamma tracking array detector field, especially for the production of high resolution and high active volume devices [4]. Finally, the diode  $10 \times 10 \text{ mm}^2$  with 0.4 mm gap is tested also in diode configuration to measure the leakage current. The diode shows very low leakage current at 80 K: about 100 pA at 40 V reverse bias apply (fig. 3(b)). Moreover, the methanol passivation [8] is tested to evaluate its electrical insulation. Figure 3(c) shows



Fig. 3. – (a) SEM image of photolithography segmentation. The gap is 0.4 mm wide, and the contact corners are rounded with 0.5 mm curvature radius. (b) Diode characteristic curve of the  $10 \times 10 \text{ mm}^2$  segmented detector. (c) Electrical resistance of methanol passivation between segments.

a high electrical resistance of methanol passivation  $(>10^{12} \Omega)$  that ensures high electrical insulation between segments.

In this work, we described the photolithography segmentation technique of  $n^+$  contacts on HPGe. The spin coating process is optimized in terms of rotation speed and process time. The first result is that the thin film of resist material is more uniform on the gold surface layer, thus permitting to have a defined segmentation after gold etchant step. SEM and optical images confirmed the good quality of the geometry segmentation [19]. The surface is very spatially detailed, and no gold layer remained on the germanium surface, except that of the mask segmentation designed.

Finally, the detector is tested in diode configuration showing low leakage current and a high electrical insulation between segments. These results are a very good starting point that can give a strong impact to perform a future p-type segmented gamma detector.

\* \* \*

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 654002.

### REFERENCES

- [1] BAZZACCO D. et al., Nucl. Phys. A, 746 (2004) 248c.
- [2] www.agata.org.
- [3] AKKOYUN S. et al., Nucl. Instrum. Methods Phys. Res. A, 668 (2012) 26.
- [4] REITER P., Nucl. Instrum. Methods Phys. Res. B, 463 (2020) 221.
- [5] WIENS A. et al., Nucl. Instrum. Methods Phys. Res. A, 618 (2010) 223.
- [6] EBERTH J. et al., Prog. Part. Nucl. Phys., 60 (2008) 283.
- [7] LOOKER Q. et al., Nucl. Instrum. Methods Phys. Res., 777 (2015) 138.
- [8] MAGGIONI G. et al., Eur. Phys. J. A, 51 (2015) 141.
- [9] KNOLL G. F., Radiation Detection and Measurements, 2nd edition (J. Wiley & Sons Inc., New York) 1989.
- [10] CARTURAN S. et al., Mater. Chem. Phys., 161 (2015) 116-122.
- [11] CARRARO C. et al., Appl. Surf. Sci., 509 (2020) 145229.
- [12] MAGGIONI G. et al., Eur. Phys. J. A, 54 (2018) 34.
- [13] SGARBOSSA F. et al., Appl. Surf. Sci., 496 (2019) 143713.
- [14] HUERT K. et al., Mater. Sci. Semicond. Process., 62 (2017) 92.
- [15] DE SALVADOR D. et al., MDPI Proceedings DyProSo, 26(1) (2019) 39.
- [16] BOLDRINI V. et al., J. Phys. D, 52 (2019) 035104.
- [17] www.microchemicals.com.
- [18] TYONA M. D. et al., Adv. Mater. Res., 2 (2013) 195.
- [19] RANIERO W. et al., INFN-LNL Annual Report 259 (2020) ISSN 1828-8561.