



ELSEVIER

Comparison of vertical gradient freeze bulk GaAs and custom grown vertical zone melt bulk GaAs as radiation spectrometers

D.S. McGregor^{a,*}, H.C. Chui^b, J.E. Flatley^c, R.L. Henry^e, P.E.R. Nordquist^e, R.W. Olsen^a,
M. Pocha^d, C.L. Wang^{1,c}

^aSandia National Laboratories, MS-9671, Livermore, California 94550, USA

^bSandia National Laboratories, MS-0603, Albuquerque, New Mexico 87185, USA

^cDetectronix, Livermore, California 94550, USA

^dLawrence Livermore National Laboratory, Livermore, California 94550, USA

^eNaval Research Laboratory, Washington, DC 20375-5000, USA

Abstract

Custom grown vertical zone melt (VZM) ingots of GaAs have been zone refined and zone leveled. The crystallinity, impurity concentrations, defect concentrations, and electrical properties of the ingots have been studied. Radiation detectors fabricated from the ingots have been compared to radiation detectors fabricated from commercially available vertical gradient freeze (VGF) material. Preliminary results suggest that electrical homogeneity may be a major factor in determining the performance of GaAs gamma ray detectors. Deep level EL2 concentrations were reduced in the VZM material, however gamma ray detectors fabricated from the material exhibited inferior resolution. Commercial semi-insulating bulk VGF GaAs material demonstrated much better resolution than the VZM detectors, giving best energy resolutions of 7.8% FWHM for 122 keV gamma rays and 5% FWHM for 356 keV gamma rays.

1. Introduction

Gallium arsenide (GaAs) is a wide band gap semiconductor that has been investigated as a room temperature radiation detector since the early 1960s [1]. Thin detectors fabricated from high purity liquid phase epitaxial GaAs have shown good gamma ray energy resolution at room temperature [2,3]. Epitaxial detectors are generally only a few hundred microns thick, which restricts their use to charged particle and low energy gamma ray spectroscopy. The obvious solution for increasing gamma ray interaction efficiency is to use larger crystals, or rather, bulk GaAs crystals. Semi-insulating (SI) bulk GaAs detectors have demonstrated acceptably good alpha particle resolution at room temperature [4,5], but have not performed well as gamma ray spectrometers.

SI GaAs ingots are produced through a careful balance between native deep donor defects (EL2) and residual shallow dopants. Generally, EL2 levels are near $10^{16}/\text{cm}^3$ with residual shallow acceptor levels near or below $10^{15}/\text{cm}^3$. It is suggested that EL2 donors may produce space charge in a reverse biased diode, which may be a contributing factor to the truncated electric fields observed in

SI bulk GaAs detectors [4,6–8]. Its overall reduction in concentration could allow for wider active regions at lower voltages than presently required. Impurity levels must also be reduced to ensure that the material remains semi-insulating.

The vertical zone melt (VZM) growth technique allows for zone refinement (ZR) and zone leveling (ZL) of the GaAs ingot before its removal from the crucible [9,10]. Zone refinement offers the prospect of reducing the residual contaminant levels, whereas Ga zone leveling can reduce the overall EL2 concentration. Reported are results from detectors fabricated from zone refined and zone leveled VZM SI bulk GaAs, along with comparisons to detectors fabricated from commercially available vertical gradient freeze (VGF) SI bulk GaAs.

2. Fabrication of Schottky barrier detectors

Samples from VZM ingots 6-57-H, 6-65-H, and 6-90-H were sliced into 1.2 mm thick wafers with a 250 μm thick I.D. saw [11]. One side of the wafers were lapped with 3 μm alumina grit until 350 μm were removed. Preliminary polishing was performed on all samples (VZM and VGF material) with 0.3 μm alumina suspended in a sodium

*Corresponding author.

¹Dr. Ching L. Wang passed away on July 15, 1994.

hypochlorite solution. Final chemical polishing was performed with 0.25% bromine/methanol solution.

The polished side of the wafers were coated with PECVD Si_3N_4 at 250°C. The wafers were patterned with photoresist and the nitride was reactive ion etched (RIE) to the bare GaAs surface. Si n-type implants were performed, followed by an anneal in forming gas at 800°C for 15 s. The wafers were repatterned and Pd/Ge layers were deposited over the implanted areas to form non-spiking n-type ohmic contacts [12]. The wafers were annealed in a tube furnace under N_2 for 30 min at 250°C. The wafers were again patterned and Ti/Au layers were deposited directly over the annealed Pd/Ge contact.

The wafers were mounted ohmic contact down and 400 μm were lapped and polished from the opposite side as previously described. PECVD nitride was deposited over the freshly polished wafer and patterned for liftoff. The nitride was etched to the bare GaAs using RIE. The surface was treated with a light chemical etch, following by vacuum deposition of a thin Ti/Au Schottky contact. The detectors were cleaved from the wafers and bonded into BN collars.

3. Fabrication of AlGaAs heterostructure barrier detectors

MOCVD was used to grow a 1 μm thick p-type AlGaAs layer on a commercial semi-insulating (SI) VGF GaAs wafer. The AlGaAs layer was coated with PECVD nitride and the wafer was mounted AlGaAs side down. The opposite side of the wafers were polished with 0.3 μm alumina grit suspended in sodium hypochlorite solution, followed by chemical polishing with 0.25% bromine/methanol solution. Si n-type implants were performed on the polished surface and annealed at 800°C for 15 s.

Photoresist was used to protect the nitride covering the p-type contact locations. The nitride and AlGaAs layers were reactive ion etched around the contact areas to the bare GaAs surface, leaving behind mesa AlGaAs structures. The p-type AlGaAs contact areas were patterned and the protecting nitride was etched to the bare AlGaAs surface. Au/Be/Au p-type ohmic contacts were vacuum deposited, followed by an anneal at 410°C. Pd/Ge layers were vacuum deposited over the Si implanted areas to form non-spiking n-type ohmic contacts. The contacts were annealed as described in the previous section. Ti/Au layers were subsequently deposited over the Pd/Ge contact. The detectors were cleaved from the wafers and bonded into BN collars.

4. I - V measurements

Reverse leakage currents were measured from the bulk GaAs detectors, and diodes with different contact areas and

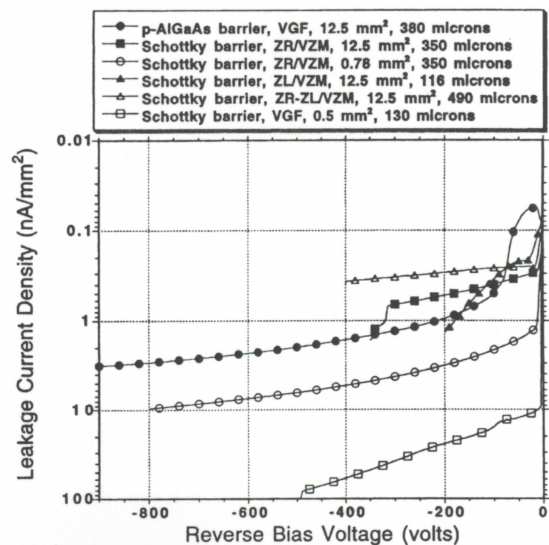


Fig. 1. Characteristic reverse bias I - V curves for several GaAs diodes of different thicknesses. Shown are the reverse bias leakage current densities measured at room temperature (20°C). The diodes are listed by barrier type, material type, contact area, and total thickness.

thickness were investigated. Most of the diodes demonstrated robust characteristics, withstanding over 1 to 2 V per micron of thickness. The p-AlGaAs barrier detector has what appears to be higher leakage current than many of the other detectors, yet from Fig. 1, it has a comparable or lower leakage current density. It appears that the p-AlGaAs structure provides a low leakage barrier. The p-AlGaAs barrier diode was biased to 1000 V with a maximum leakage current density of only 3.5 nA/mm^2 . Most of the Schottky diodes fabricated from the VZM material demonstrated low leakage current densities as well. The I - V curves (and pulser measurements) indicated that shot noise was not a significant factor in determining the resolution for most of the detectors tested. One small area Schottky barrier detector fabricated from VGF material had a much higher leakage current density than average (shown in Fig. 1), however the small detector had surprisingly better resolution than the other detectors.

5. Radiation measurements

Schottky barrier detectors fabricated from the commercial VGF material demonstrated considerably better gamma ray resolution than the detectors fabricated from the zone refined and/or zone leveled VZM material. Poor resolution was observed from most of the detectors fabricated from the VZM material, regardless of the growth process. A 116 μm thick Schottky barrier detector (area = 12.5 mm^2) fabricated from zone leveled VZM material

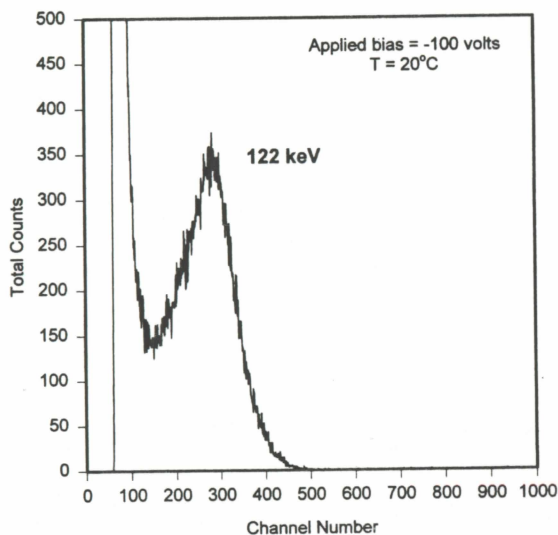


Fig. 2. Room temperature (20°C) ^{57}Co gamma ray spectrum taken with a Schottky barrier detector fabricated from ZL VZM GaAs material. The detector was 116 μm thick, had a contact area of 12.5 mm^2 , and was biased at 100 V. The energy resolution for 122 keV gamma rays is 49% FWHM.

demonstrated a typical resolution of 49% FWHM for 122 keV gamma rays (Fig. 2). Charge collection efficiency (CCE) for the ZR, ZL, and ZR/ZL GaAs detectors was 25% or lower, which is considerably lower than generally reported in the literature for GaAs detectors of similar thicknesses [1].

Fig. 3 shows spectra taken with a 380 μm thick p-AlGaAs barrier detector (area = 12.5 mm^2) fabricated

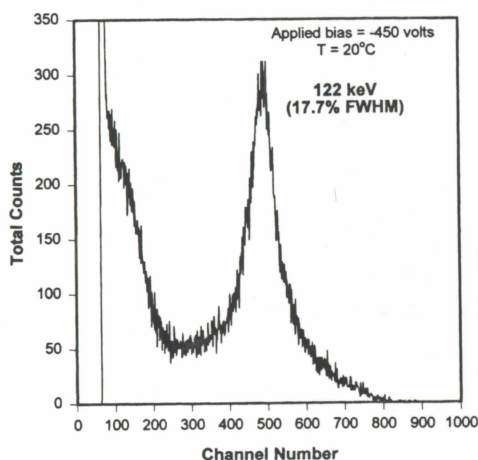


Fig. 3. Room temperature (20°C) gamma ray spectra taken with a 380 μm thick p-AlGaAs heterostructure barrier detector fabricated from commercial VGF bulk GaAs. Shown is a ^{57}Co spectrum with energy resolution of 17.7% FWHM for 122 keV gamma rays. The contact area of the device was 12.5 mm^2 , and the bias voltage during operation ranged between 400 and 450 V.

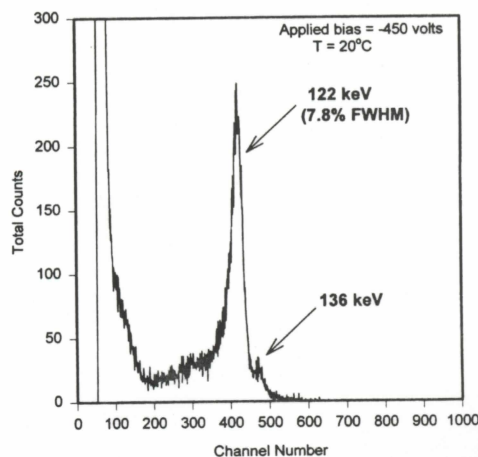


Fig. 4. Room temperature (20°C) ^{57}Co gamma ray spectrum taken with a 130 μm thick Schottky barrier detector fabricated from commercial VGF bulk GaAs. The energy resolution is 7.8% FWHM, and both the 122 keV and the 136 keV gamma ray peaks are clearly visible. The contact area of the device was 0.48 mm^2 , and the bias voltage during operation was 450 V.

from commercial VGF SI bulk GaAs. The detector demonstrated room temperature energy resolution for ^{57}Co 122 keV gamma rays of 17.7% FWHM. Although the detector is over three times thicker than the ZL VZM detector in Fig. 2, it has much better resolution and charge collection efficiency. The spectrum deviates from typical low energy tailing, demonstrating instead a flared base in both the high and low energy directions. Gamma rays were detected from ^{133}Ba at room temperature, in which the 356 keV peak was observed.

A small area (0.48 mm^2) Schottky barrier detector fabricated from commercial VGF SI bulk GaAs demonstrated room temperature energy resolution for ^{57}Co 122 keV gamma rays of 7.8% FWHM (Fig. 4). Both the 122 keV gamma ray peak and the 136 keV gamma ray peak are clearly visible. Additionally, the same detector yielded good room temperature energy resolution for ^{133}Ba gamma rays, demonstrating a resolution of 5% FWHM for 356 keV gamma rays. The small detector had a much higher leakage current and leakage current density than most of the other detectors tested, yet demonstrated significantly better resolution. The capacitance of the detector is only a bit less than the other detectors tested, hence the improved resolution is not thought to be related to reduced capacitance effects.

6. Conclusion

Radiation detectors fabricated from custom grown ingots of VZM SI bulk GaAs have been investigated and compared to detectors fabricated from commercial VGF SI

bulk GaAs. The leakage currents observed from detectors fabricated from both VZM and VGF material were comparably low, indicating similar device parameters. However, the detectors fabricated from the VGF material routinely outperformed the detectors fabricated from the VZM material. Good energy resolution for ^{133}Ba 356 keV gamma rays (5% FWHM) was observed from a small area VGF detector, although the detector operated with a relatively high leakage current density near 60 nA/mm^2 .

Since the VGF material produced better detectors than the VZM material, some material differences should be noted. The VZM material had noticeably lower EL2 concentrations than the VGF material, in some cases being near $10^{15}/\text{cm}^3$. The VZM material did remain semi-insulating, indicating that the average residual impurity concentrations were low enough to maintain compensation. However, measurements indicate large variations and inhomogeneities in electrical characteristics across some of the VZM samples [11], which can understandably degrade the detector performance. It is possible that non-uniform electrical characteristics may be reduced through post growth annealing and will be considered in future work.

Additionally, X-ray diffraction crystal rocking curves of the material tend to show slightly broader peaks for the VZM material than the VGF material, which may be due to problems with the crystal mosaic or even residuals effects from the wafer polishing procedure. Smaller detectors appear to perform much better than larger detectors for both VGF and ZL/ZR VZM material. Should significant nonuniformities in the electrical characteristics be present, one would expect smaller area detectors to outperform larger area detectors. Production of VZM bulk GaAs with improved electrical homogeneity after the zone refinement or zone leveling process may help resolve the issue.

Acknowledgments

The vertical zone melt GaAs ingots were grown at the Naval Research Laboratory in Washington, DC. The GaAs detectors were fabricated at the Compound Semiconductor Research Laboratory at Sandia National Laboratories in

Albuquerque, New Mexico. The authors wish to express their gratitude to Melissa Cavaliere, Richard Corless, Jo Ann Escobedo, Beth Fuchs, Pat Glarborg, Sean Kilcoyne, Geraldine Lopez, Fil Martinez, Johnny Moya, Thomas Plut, Dennis Rieger, Sally Samora, Denise Tibbetts, and John Van Scyoc for their technical assistance with the reported work. We also express our appreciation for the assistance provided by Dr. Arnold Howard, Dr. Stan Kravitz, Dr. Randy Shul, and Dr. John Zolper.

References

- [1] D.S. McGregor and J.E. Kammeraad, in: *Semiconductors for Room Temperature Nuclear Detector Applications*, eds T.E. Schlesinger and R.B. James, *Semiconductors and Semimetals*, 43 (Academic Press, San Diego, 1995) p. 383.
- [2] J.E. Eberhardt, R.D. Ryan and A.J. Tavendale, *Nucl. Instr. and Meth.* 94 (1971) 463.
- [3] D. Alexiev and K.S.A. Butcher, *Nucl. Instr. and Meth. A* 317 (1992) 111.
- [4] D.S. McGregor, G.F. Knoll, Y. Eisen and R. Brake, *IEEE Trans. Nucl. Sci.* NS-39 (1992) 1226.
- [5] M. Alietti, C. Canali, A. Castaldini, A. Cavallini, A. Cetronio, C. Chioggi, S. D'Auria, C. del Papa, C. Lanzieri, F. Nava and P. Vanni, *Nucl. Instr. and Meth. A* 362 (1995) 344.
- [6] K. Berwick, M.R. Brozel, C.M. Buttar, M. Cowperthwaite and Y. Hou, *Inst. Phys. Conf. Proc.* 135 (1993) 305.
- [7] D.S. McGregor, R.A. Rojas, G.F. Knoll, F.L. Terry, Jr., J. East and Y. Eisen, *J. Appl. Phys.* 75 (1994) 7910. (Errata) D.S. McGregor, R.A. Rojas, G.F. Knoll, F.L. Terry, Jr., J. East and Y. Eisen, *J. Appl. Phys.* 77 (1995) 1331.
- [8] T. Kubicki, K. Lübelmeyer, J. Ortmanns, D. Pandoulas, O. Syben, M. Toporowsky and W.J. Xiao, *Nucl. Instr. and Meth. A* 345 (1994) 468.
- [9] E.M. Swiggard, *J. Cryst. Growth* 94 (1989) 556.
- [10] R.L. Henry, P.E.R. Nordquist, R.J. Gorman and S.B. Qadri, *J. Cryst. Growth* 109 (1991) 228.
- [11] D.S. McGregor et al., these Proceedings (9th Int. Workshop on Room Temperature Semiconductor X- and γ -Ray Detectors, Associated Electronics and Applications, Grenoble, France, 1995) *Nucl. Instr. and Meth. A* 380 (1996).
- [12] M.L. Lovejoy, A.J. Howard, K.R. Zavadil, D.J. Rieger, R.J. Shul and P.A. Barnes, *J. Vac. Sci. A* 13 (1995) 758.