SEGMENTATION OF THE OUTER CONTACT ON P-TYPE COAXIAL GERMANIUM DETECTORS

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Sponsored by DOE Office of Nuclear Physics

Contract Nos. DE-FG-02-05ER84157¹ and DEAC05-RLO1830²

ABSTRACT

Germanium detector arrays are needed for low-level counting facilities. The practical applications of such user facilities include characterization of low-level radioactive samples. In addition, the same detector arrays can also perform important fundamental physics measurements including the search for rare events like neutrino-less double-beta decay. Coaxial germanium detectors having segmented outer contacts will provide the next level of sensitivity improvement in low background measurements. The segmented outer detector contact allows performance of advanced pulse shape analysis measurements that provide additional background reduction. Currently, n-type (reverse electrode) germanium coaxial detectors are used whenever a segmented coaxial detector is needed because the outer boron (electron barrier) contact is thin and can be segmented. Coaxial detectors fabricated from p-type germanium cost less, have better resolution, and are larger than n-type coaxial detectors. However, it is difficult to reliably segment p-type coaxial detectors because thick (~1 mm) lithium-diffused (hole barrier) contacts are the standard outside contact for p-type coaxial detectors. During this Phase 1 Small Business Innovation Research (SBIR) we have researched the possibility of using amorphous germanium contacts as a thin outer contact of p-type coaxial detectors that can be segmented. We have developed amorphous germanium contacts that provide a very high hole barrier on small planar detectors. These easily segmented amorphous germanium contacts have been demonstrated to withstand several thousand volts/cm electric fields with no measurable leakage current (<1 pA) from charge injection over the hole barrier. We have also demonstrated that the contact can be sputter deposited around and over the curved outside surface of a small p-type coaxial detector. The amorphous contact has shown good rectification properties on the outside of a small p-type coaxial detector. These encouraging results are the first fundamental steps toward demonstrating the viability of the amorphous germanium contacts for much larger segmented p-type coaxial detectors. Large segmented p-type coaxial detectors based on this technology could serve as the gamma-ray spectrometers on instruments such as the Radionuclide Aerosol Sampler/Analyzer (RASA). These detectors will provide a more sensitive, lower background measurement than currently available unsegmented p-type coaxial detectors.

OBJECTIVES

Germanium detector arrays are needed for low-level counting facilities. The practical applications of such user facilities include characterization of low-level radioactive samples. In addition, the same detector arrays can also perform important fundamental physics measurements including the search for rare events like neutrino-less double-beta decay (Miley et al., 1991; Miley, et al., 1990; Majorana Collaboration White Paper, 2003; and Goulding et al., 1984). Coaxial germanium detectors having segmented outer contacts will provide the next level of sensitivity improvement in low background measurements. The segmented outer contact allows performance of advanced pulse-shape analysis measurements. These techniques can be used to discriminate between multiple Compton-scattered gamma-ray events and single-point beta-decay events. Recently, such techniques have been demonstrated with Clover detectors at Los Alamos National Laboratory (LANL) and confirming simulations done at Pacific Northwest National Laboratory (PNNL), Lawrence Berkeley National Laboratory (LBNL), and Oak Ridge National Laboratory (ORNL). Because of their complexity, the segmented coaxial detectors are expensive and available only after relatively long lead times. Improved detector segmentation techniques would be both important and timely. Such technological advances will reduce fabrication costs and improve availability of these detectors for the low-level counting community.

Currently, n-type (reverse electrode) germanium coaxial detectors are used whenever a segmented coaxial detector is needed. To obtain reasonably accurate coaxial detector segmentation, the outer detector contact must be the segmented contact. The most conveniently segmented outer contact is the boron-implanted outer contact of an n-type coaxial detector. The ability to segment the outer boron contact is the reason segmented n-type coaxial detectors are suggested for use in low background gamma-ray measurements. N-type coaxial detectors should be used in environments where radiation damage is a concern and/or a thin outer detector contact is desired. However, segmented p-type (conventional electrode) coaxial detectors would have technical and financial advantages in low background counting experiments.

P-type coaxial detectors are significantly less expensive and have better gamma-ray energy resolution than n-type coaxial detectors. Fundamentally, this is due to the presence of electron-trapping sites found in even the best detector-quality germanium. A small percentage of the electrons arising from gamma-ray interactions in the detector are trapped before reaching the electron-collecting contact. The charge is trapped for a sufficient duration and is not included in the processed signal for that event. The resulting pulse-height deficits cause broadening of gamma-ray peaks. The magnitude of this energy-resolution degradation from electron trapping is strongly dependent on the geometry of the detector. In detectors of coaxial geometry, the charge carriers collected on the inner contact are responsible for inducing most of the total signal from gamma-ray interactions occurring in most of the volume of the detector. In n-type coaxial detectors, electrons are collected on the inner contact. Consequently, the gamma-ray energy resolution of n-type coaxial detectors is degraded by even small amounts of electron trapping. On the other hand, the spectroscopy of p-type detectors of coaxial geometry relies more heavily on the collection of holes on the inner contact. As a result, electron trapping causes much less resolution degradation in a p-type coaxial detector than in an n-type coaxial detector. The decreased sensitivity to electron trapping makes a greater fraction of the germanium crystals viable for fabrication into p-type coaxial detectors having excellent energy resolution. The lesser importance of electron trapping also allows fabrication of larger diameter p-type coaxial detectors. Thus fewer detectors are needed to make an array of a given total volume. In a detector array like Majorana, we estimate the cost savings associated with the use of segmented p-type coaxial detectors, rather than n-type detectors, to be about \$13M. It is important to note that electron trapping is still not thoroughly understood and difficult to control in the growth of detector-quality germanium. Any large-scale low-level counting facility employing segmented coaxial detectors would greatly benefit, both technically and financially, from the use of p-type coaxial detectors.

Currently the segmentation of the outer lithium-diffused n+ contact of a p-type coaxial detector is a nontrivial operation. The outer contact of a p-type coaxial detector is conventionally made using a rather thick (as much as $\sim 1 \text{ mm thick}$) lithium-diffused layer as the hole barrier contact. Thick lithium-diffused contacts are very rugged and reliable but require rather drastic techniques for segmentation. Some techniques involve cutting through the lithium-diffused layer with a saw to segment the contact. Although it can work, such detector fabrication techniques are expensive, time consuming, and mechanically cumbersome. In the event that a saw-cut lithium contact does not successfully function, successive fabrication attempts may prove difficult. Accommodating the saw-cut grooves during the subsequent fabrication attempts may be sufficiently complicated to compel regrinding the crystal to a smaller diameter or even starting over again with a new crystal. In addition, such saw cuts can cause

charge-collection and surface-channel problems in the vicinity of the grooves between the segments. Grooves often result in effectively "dead" germanium near the grooves. The initial saw cuts and electronically "dead" germanium consume valuable isotopically enriched germanium. Some recent data has proven that this is a significant issue. A side-by-side comparison between saw-cut lithium contacts and thin amorphous germanium contacts on planar strip detectors show that charge-collection problems associated with saw-cut lithium can be quite significant (Gros and Lister, 2006).

To make segmented p-type coaxial detectors viable, better outer contacts must be developed to replace saw-cut segmented thick lithium contacts. There are other contact technologies with the potential to provide hole-barrier contacts that are more easily segmented than thick-lithium n+ (hole barrier) contacts. This study seeks to determine the best solution for producing thin-segmented hole barrier contacts on p-type germanium detectors. This will make p-type coaxial detectors viable for large-scale low-level counting arrays. We have started investigating alternative techniques for making segmented hole-barrier contacts in lieu of conventional thick lithium-diffused n+ contacts. Amorphous germanium contacts represent one possible alternative. During this Phase I SBIR we fabricated and tested many small planar test detectors (\sim 2-4 mm thick, \sim 30 mm diameter) having segmented amorphous germanium contacts as the hole-barrier (+ biased) contacts. We focused on making the amorphous germanium hole barrier as high as possible. A larger hole barrier provides better rectification and a higher probability of successful fabrication of large diameter p-type coaxial detectors using the thin amorphous germanium contact over the entire outside area of the detector. Amorphous germanium contact technology naturally lends itself to the simple fabrication of finely segmented germanium detectors (Luke et al., 1992; Hull et al., 2002; and Hull et al., 2003). By making many planar test detectors, we studied the rectification and segmentation of amorphous germanium contacts with a focus on increasing the hole barrier contact. Our detector processing parameters have been tuned to make reliable segmented amorphous germanium contacts having a large hole-injection barrier specifically for large diameter p-type coaxial detectors. We demonstrated the viability of our fabrication techniques by fabricating a small p-type coaxial detector (MJ1) having an amorphous germanium outer contact. The successful rectification of this contact over the large curved outer contact of MJ1 serves as a first step toward a viable manufacturing process for segmented p-type coaxial detectors. Segmented planar germanium detector technology is our specialty. We believe that the best way to approach the fabrication of segmented coaxial detectors is to first understand the fabrication processes for planar detectors. With the fundamental physics and technology well in hand, the technology can be extended to accommodate the nonplanar geometry issues arising in coaxial detector fabrication.

RESEARCH ACCOMPLISHED

The progress made during the Phase I is described here. Extremely important strides were made toward the commercialization of large p-type coaxial detectors having highly segmented outer contacts for low-level counting arrays. The use of p-type germanium detectors will greatly decrease the cost and difficulty associated with the production of segmented coaxial detectors for such arrays. We have made the first steps toward demonstrating the viability of segmented p-type coaxial detectors. This has been done through fabrication and evaluation of many small planar test detectors and a small p-type coaxial detector. We focused on the amorphous germanium contact as the hole barrier contact. We presented some of our early Phase I results at the 2005 Seismic Research Review meeting in Rancho Mirage, CA, in September 2005 (Hull and Pehl, 2005).

We made a number of small planar test germanium detectors with the goal of increasing the hole barrier formed by the amorphous germanium contact. A larger amorphous germanium hole barrier contact recipe should provide the best rectification characteristics possible under the high electric fields present in large p-type coaxial detectors. Four planar test detectors were made and tested to evaluate each fabrication process. The detectors were etched in a mixture of 3:1 HNO₃:HF for approximately 30 s each. The detectors were rinsed with methanol and blown dry with N₂ gas. The detectors were placed into our RF sputtering chamber and a layer of amorphous germanium was sputter deposited onto the test detectors. After that, the detectors were placed in our thermal evaporator and a layer of aluminum was evaporated through a shadow mask to establish the active areas of the segmented amorphous germanium contact. A center and guard-ring are the two segments on our test detectors. The detectors were then placed in a four-detector test cryostat, pumped, cooled, and tested the following day. Figure 1 shows a set of the test detectors in the four-detector test cryostat. The segmentation is created by evaporating aluminum through a shadow mask, establishing the center and guard-ring features.



Figure 1. A four-detector test cryostat contains planar test detectors. The detectors in the picture have segmented amorphous germanium contacts.

We repeated fabrication and testing on both n- and p-type planar test detectors. The amorphous germanium contact was finely tuned to maximize the rectifying hole barrier height. Rectifying charge-injection barriers exponentially decrease the leakage current from thermionic charge injection over the contact barriers. This is apparent when comparing our new amorphous germanium contact barrier with our earlier detector fabrication recipe. Figure 2 shows a plot of leakage current vs. bias voltage for detectors made with our earlier amorphous germanium detector recipe. The data show the standard linear (exponential) voltage dependence that we have observed repeatedly from the contacts on many detectors. The solid curves in the figure are produced by the amorphous-crystalline heterojunction equation for fully depleted devices: $j = j_{\infty} \exp(-\{\phi - [(\epsilon_0 \epsilon_{Ge}/N_F)^{1/2}(V+V_{depl})/d]\}/k_BT)$ (Hull et al., 2005). The critical values used in the expression are the barrier height $\phi = .30$ eV and density of states $N_F = 5 \times 10^{17}$ /eVcm³. The barrier height dictates the temperature dependence and the density of states dictates the slope of the voltage dependence of the leakage current. These leakage currents were rather high due to the relatively low hole barrier height. By repeatedly fabricating and testing many detectors we successfully increased the barrier height enough to eliminate all measurable leakage current thermionically emitted over the hole barrier.



Figure 2. This plot shows the leakage current from a detector fabricated using our previous amorphous germanium contacts. The high leakage current results from relatively low (~.30 eV) hole barriers.

We tuned the amorphous germanium contact to form the very highest hole barrier possible for the sake of eventually making large p-type coaxial detectors. Many sets of planar test detectors were made and tested. We varied the sputter chamber pumpdown time and the percentage of hydrogen in the argon sputter gas in an attempt to fabricate an amorphous germanium contact having a very high hole barrier. Eventually we were routinely able to make planar test detectors that withstood several thousand volts/cm with no measurable leakage current! This is plenty of electric field for a large p-type coaxial detector. Figure 3 shows a plot of leakage current as a function of bias voltage for a detector fabricated using our new amorphous germanium contact recipe for p-type coaxial detectors. These detectors were 2.8 mm thick. There is no measurable leakage current from the center section of the detector at 1,000 V. Leakage currents below 1 pA are difficult for us to measure and are insignificant for the operation of a germanium detector. We assign a value of .01 pA to make the data points appear on the logarithmic plot. The center section of this detector withstood 1,000 V with no measurable leakage current. This corresponds to an electric field of ~1,000 V/.28cm = 3,571 V/cm, plenty to make a large p-type coaxial detector.



Figure 3. The leakage current from a the center and guard ring sections of a planar test detector. The center contact shows no measurable leakage current up to 1,000 V. This detector is only 2.8 mm thick. The guard ring shows a large amount of surface leakage current.

The leakage current measured on the guard ring of the detector is very high. Unfortunately, this is typical of detectors having our new α -Ge contacts. The current measured on the guard ring is "intrinsic surface" leakage current caused by conductive surface channels (Hull et al., 1995). We have carefully tuned the amorphous germanium sputtering process to produce a high hole barrier contact. Unfortunately, the contact wraps around the sides of the planar test detector during the sputtering process. Most likely, this layer forms a fairly strong n-type surface channels on the intrinsic surface. We normally have a guard ring on our planar strip detectors; consequently, surface channels of this magnitude do not affect the performance of the electrodes within the guard ring. In the future, we may treat this surface with an acid etch after the contacts are deposited on the detector to eliminate this strong n-type surface channel. If high surface leakage currents continue to be a problem we also have the capability to make a thin guard-ring on the inside contact of coaxial detectors to eliminate the ill effects of surface leakage current.

A major accomplishment of this work was the establishment of the fact that the amorphous germanium contact can be made to form a very high hole barrier contact. The amorphous-germanium contact can be a candidate for the outer-segmented contact on large diameter p-type coaxial detectors. As far as we can tell, the detector contacts do not contribute to the leakage current of the detector. In fact, the dominant observed leakage current appears to be bulk generation current from generation-recombination sites near the mid-band-gap region in crystal. If these mid-gap sites are responsible for the leakage current, the leakage current should be proportional to $\sim \exp(-(E_{gap}/2)/kT)$, where E_{gap} is the band gap ($E_{gap} = .7 \text{ eV}$) (Grove, 1967). To investigate this, the leakage current from the center and guard-ring sections of the detector were monitored as a function of temperature of the detectors. These data were taken with 200 V on the detector. The leakage current on the center contact is not measurable (< 1 pA) until the detector reaches the 125-130 K region. Above the 125 K region, the leakage current follows the $\sim \exp(-.35eV/kT)$ behavior expected from bulk leakage current due to generation-recombination sites near the mid-gap position. These data are consistent with the results in the seminal work by Pehl, Haller, and Cordi (Pehl et al., 1973).



Figure 4. The leakage current from the center segment of a planar test detector as a function of temperature shows the characteristics of bulk generation current. The detector contacts contribute no measurable charge injection.

If the contacts had hole barriers that were low enough to contribute to the leakage current, we would measure leakage current increases at much lower temperatures. This is why we see the temperature dependent leakage current plotted in Figure 2 in the 80–87 K region from the earlier amorphous germanium contacts. According to our calculations, an amorphous germanium hole barrier height greater than .42 eV is sufficient to prevent the contact from injecting a measurable amount of leakage current. With contact barrier heights greater than .42 eV, the dominant leakage current arises from bulk charge generation. Using capacitance vs. pulser voltage techniques, we have measured the amorphous germanium hole barrier height to be \sim .6 eV. If we had not been able to make an amorphous germanium contact with such a high barrier height, we could not have hoped to make large diameter p-type coaxial detectors with this technology.

Having this detector recipe in hand, we began trying to fabricate MJ1. We obtained a right cylindrical piece of p-type germanium of 4 cm diameter and 2 cm length having a net electrically-active impurity concentration of $\sim 9x10^9$ /cm³ at 77 K. The more impure (seed) end of the crystal was lapped from a sharp 90-degree angle into a smooth curve of approximately 7-mm radius. This end would be the top or closed end of the detector. The other side of the detector was left flat. The detector was polish etched in 3:1 HNO₃:HF for about 2 minutes to eliminate any damage done by the lapping. Figure 5 shows a photograph of the MJ1 detector after etching.



Figure 5. The MJ1 germanium crystal after lapping and etching. The ruler in the foreground is in centimeters. The flat side is face down and the closed end of the detector is face up.

The outer contact was made by sputtering amorphous germanium on the outside of the crystal while it was sitting in the sputter chamber on the intrinsic surface just as pictured in Figure 5. We hoped that the atomic germanium scattering that occurs in the argon during the sputter-deposition process would be sufficient to coat the sides and top of MJ1 evenly in one deposition step. Then the amorphous germanium was covered with evaporated aluminum. A special motorized turntable was built inside our thermal evaporator to rotate the detector during evaporation. This allows the amorphous germanium contact to be coated evenly with aluminum during a single vacuum evaporation. With these physical modifications to the fabrication process, the same amorphous germanium deposition parameters used to make excellent planar test detectors were used to make the small p-type coaxial detector. The amorphous germanium contact forms a rectifying amorphous crystalline heterojunction with the p-type crystalline germanium at the outside diameter of the detector. Positive bias applied to the outer contact depletes the detector from the outside toward the small p+ dot contact in the center of the flat intrinsic surface making a hemispherical or pseudo-coaxial detector. We attempted this process three times with very poor results. The leakage current measured from the flat center contact was several hundred nanoamperes with the application of only 100 Volts, far too high to make a functional detector. Between these three fabrication attempts, we fabricated sets of planar test detectors using the same amorphous germanium deposition parameters to check the integrity of our process. The process appeared to be fine in all cases. We suspected that the dot-like center contact in the middle of the intrinsic surface might be the problem. Generally, such a detector would have some physical indentation into the flat surface. The p+ contact would usually be deposited down in the hole. We used our glass bead blaster to make a 7-mm diameter hole approximately 7-mm deep at the center of the intrinsic surface to accommodate the p+ contact. We etched and fabricated the detector again achieving far better results. After a fourth fabrication attempt, the detector depleted at about 650 V, corresponding to a p-type net electrically active impurity concentration approximately 9×10^9 /cm³. The capacitance and leakage current were measured as a function of voltage to establish the diode performance of the detector. The capacitance-C(V) and leakage current-I(V) curves are shown in Figure 6.



Figure 6. The leakage current-I(V) and capacitance-C(V) curves from MJ1 as a function of bias voltage. The outer amorphous germanium contact is rectifying on the small p-type coaxial detector. The rather high leakage current is due to surface-channel leakage surface. The C(V) curve indicates a very abrupt depletion at ~650 V. The depleted capacitance of the detector was 2.8 pF.

CONCLUSIONS AND RECOMMENDATIONS

Admittedly, the leakage current is somewhat high to call this a tremendously good germanium detector. This leakage current is due to surface-channel leakage currents. The shape of the I(V) curve looks very much like normal surface leakage current we observe with our planar detectors. It can be eliminated with changes in the detector processing, including a simple post-processing surface etch. The important feature to note is the absence of any sharp jumps in the I(V) curve. When an amorphous germanium contact ceases to rectify on a germanium detector, it instantly injects very large currents into the detector. This shows up in the I(V) curve as an abrupt step increase in the leakage current. There are no such features in this I(V) measurement curve. The contact is rectifying. The leakage current here is due to surface channel current. This proves that the amorphous germanium contact does rectify well when sputtered around the curved closed end of a small pseudo-coaxial p-type detector.

The C(V) curve shows a very sharp change in slope at the point of full depletion, \sim 650 V. Once the detector is fully depleted, there is no measurable additional decrease in detector capacitance as is often observed in detectors having thick lithium contacts. The diffuse junction formed by a thick lithium contact causes the depletion point to have a less sharp discontinuity in slope. This is often called a "soft C(V)." The MJ1 detector clearly shows a very "hard C(V)" because both contacts are very thin and well defined. When fully depleted, this detector represents only

2.8 pF of input capacitance. The low capacitance plus the thin (~2,000 angstroms) amorphous germanium contact would make this a nice p-type x-ray detector if the leakage current were lower.

Although the detector did have rather high leakage current, it was still a functional gamma-ray detector. When operated at 500 V with a unipolar peaking time of 6 μ s, the leakage current was low enough to make the gamma ray spectrum shown in Figure 7. Closer analysis of the shapes of the gamma-ray peaks indicate very little asymmetry from poor charge collection. Although the ~3 keV full width at half maximum (FWHM) noise makes the measurement somewhat insensitive to poor charge collection from trapping, the higher energy gamma-ray peaks show no noticeable tailing associated with trapping. The electric field is rather low in the detector at 500 V, so this is a worst-case scenario with respect to charge collection. We believe that this is due to the fact that holes (rather than electrons) are the charge carrier collected on the inner contact. Holes are responsible for most of the signal. Hole trapping generally occurs less than electron trapping in germanium detectors. We would almost certainly see tailing of the 1,332 keV peak if this were an n-type coaxial detector collecting electrons on the inner contact. This observation is in line with the supposition on which this work is based.



Figure 7. An energy spectrum shows the performance of the detector. The noise of the system was FWHM ~3 keV due to noise from intrinsic surface leakage current.

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