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HIGH-PURITY GERMANIUM CHARGED-PARTICLE DETECTORS: A LBL-IUCF UPDATE

R.H. Pehl, P.N. Luke, and D.L. Friesel

July 1985

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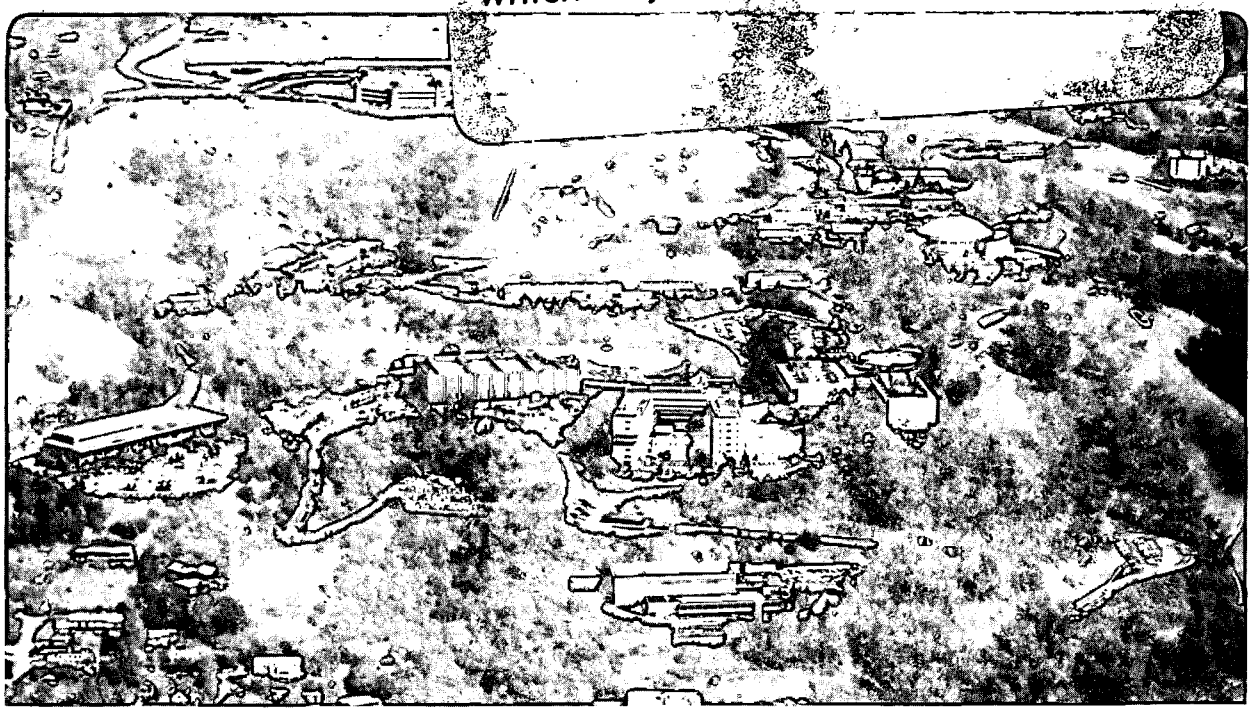

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July 12, 1985

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I. Introduction

The collaborative program between LBL and IUCF which successfully developed high-purity germanium charged-particle detector systems to the point where their use at IUCF could be considered routine was reported in our first paper [1]. During their continued successful use in the intermediate-energy charged-particle and neutron environment at IUCF, additional observations on the characteristics of these detectors have been made. Some of these observations have provided more definitive answers to questions raised in Paper 1 while other observations have provided further insight into detector properties not previously seen or, at least, not previously recognized. The present paper is written in a style that assumes the reader has read Paper 1 or will read Paper 1 in conjunction with the present paper.

We have now used 17 different germanium detectors in various combinations in over 70 experiments since 1976. A list of these detectors and their use at IUCF is provided in Table 1.

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Table I.

IUCF Germanium Detector List

Detector No.	Ge Type	Thickness (mm)	Impurity Concent. ($X10^{10}$ cm^{-3})	Depl. Bias (-V)	Delta (V)	Total hrs Beam Time	No. Expts. Used	No. Thermal Cycles	Total hrs Anneal	Li Layer Depth (mm)	Beginning Service Date month/year
					TRANSMISSION DETECTORS						
[a]541-12.8	n	1.17	6.5	40	35	92	3	12	108	NA	3/79
501- 9.3	n	~ 2.0	4.4	100	150	200	2	5	158	NA	4/82
501- 9.6	n	~ 2.0	4.4	100	400	336	6	9	271	NA	4/82
551-11.8	n	5.18	7.5	1100	200	200	6	12	300	NA	4/80
475-10.7	n	9.07	3.3	1700	100	1276	23	66	1617	NA	8/77
477- 6.1	n	9.52	2.0	1000	350	504	8	14	469	NA	4/80
501- 6.7	n	10.77	2.7	1800	200	1514	30	94	2248	NA	8/77
474- 5.8	n	~12.0	1.6	1600	200	294	3	10	406	NA	7/82
550-10.0	n	~13.0	2.4	2200	500	634	13	22	973	NA	10/82
[a]550- 8.6	n	~13.5	0.7	700	200	464	8	21	395	NA	11/79
517- 9.7	n	~15.0	1.2	1500	200	1580	34	87	2032	NA	8/77
[a]514-11.5	n	~15.0	1.5	2000	500	48	1	6	100	NA	3/79
					STOPPING DETECTORS						
172- 3.1	p	10.6	1.0	350	2000	609	10	23	538+	1.44[c]	11/76
514- 7.0	p	~15.21	1.9	1200	2000	2003	36	111	3022	3.30[b]	3/79
514- 8.6	p	14.94	1.1	800	2000	2067	39	92	2002	3.10[b]	3/79
525- 8.6	p	~12.0	1.1	1000	2000	50	1	1	84	0.93	1/83
601- 6.3	n	~20.0	0.8	1700	1500	50	1	3	61	0.63	7/82

[a]These detectors are currently on the inactive list (see text).

[b]The effective dead layer thickness varies somewhat with operating bias, as shown in Fig. 2

[c]This dead layer thickness measured after 318+ hours of annealing.

II. Philosophy of Germanium Detector Use at IUCF

In most key aspects, the philosophy of using germanium charged-particle detector telescopes at IUCF has evolved into something very different from what has been discussed in previous charged-particle detector telescope papers [2-4].

Obtaining the best energy resolution that germanium detectors can provide has never been of major interest at IUCF because actual resolution is dominated by experimental kinematics and beam-energy spread. The statement made in Paper 1: "An actual working experiment in which the resolution contribution of a germanium charged-particle detector was a significant part of the total measured resolution has yet to be made", remains true today. Even if the degradation in resolution caused by the experimental geometry and beam-energy spread could be made negligible, matching the amplifier gains for the individual detectors in a typical intermediate-energy charged-particle detector telescope to sufficient accuracy to take full advantage of the basic resolution of the germanium detectors is extremely difficult. As a consequence, high-resolution magnetic spectrographs are used for those experiments requiring optimum energy resolution. The resolution advantage of magnetic spectrographs is further enhanced by beam dispersion matching techniques that cancel the effect of beam emittance and kinematic broadening in the reaction plane at the magnetic spectrograph focal plane [5,6]. This technique has been used with the QDDM magnetic spectrograph at IUCF for several years to improve the energy resolution at the focal plane by about 35%, making an overall energy resolution of 0.036% routinely available. Similar beam dispersion matching techniques have also been achieved with a single silicon surface barrier detector; in this

case, the reduction in the effect of kinematic energy spreading was about a factor of three [7]. Although resolution comparable to that obtained with the magnetic spectrograph is theoretically possible for germanium detectors [8], in practice, it is very difficult to achieve with a multi-detector telescope in a typical nuclear-reaction scattering experiment.

Germanium detector telescopes are presently used for "survey" type experiments and for those experiments where more importance is placed on observing a broader energy spectrum. Because of this emphasis, the advantages of stacking detectors together (i.e., a detector telescope) compared to using one large detector of equivalent total thickness largely disappear and, in some respects, actually become disadvantages. Consequently, we now strongly prefer thicker, and thus fewer, detectors in the stack for:

i. More stopping power per detector

ii. Simplication of electronics

iii. Reduction of gain-matching problems

iv. Reduction of the number of transmission detectors required as these remain difficult to fabricate

v. Nuclear reaction losses in broad energy-range applications are usually not of concern.

As listed in Table 1, a 20 mm thick stopping detector (606-6.1) has been placed in service at IUCF. Although this detector has not yet been used extensively, we do not anticipate the additional thickness will cause problems. If the 20 mm thick stopping detector proves successful, we plan on testing a 25 mm thick stopping detector. However, we do not think a stopping detector thicker than 25 mm is viable. By contrast, the thickness limit on a transmission detector is about 18 mm; the reason for this difference will be discussed in the next section.

When the LBL-IUCF germanium detector program began, little emphasis was placed on detector area since the area of routine planar detectors made at LBL at that time far exceeded the area desired at IUCF. All the detectors listed in Table 1 have a diameter of about 36 mm while the diameter of the opening in the detector mounts (Figure 6, Paper 1) is 25 mm. These detectors could still be used if the diameter of the opening in the detector mounts were increased to about 30 mm. Since the diameter of the typical collimating aperture placed in front of the detector telescope is 9 mm, only a small fraction of the presently available detector volume is actually being used even when multiple scattering is taken into account. Consequently, experiments requiring considerably greater solid angles could be done using the currently available detectors. Nevertheless, interest in germanium detectors having a larger area than those presently in use is increasing. Larger diameter, up to 60 mm, high-purity germanium crystals of sufficient quality for reasonably routine detector fabrication are now believed to be available. We plan on fabricating and testing a 55 mm diameter detector in the near future.

III. Optimizing the Impurity Concentration in Germanium Transmission Detectors

Take note: Do not fabricate a germanium transmission detector from material that is "too" pure. Use germanium with a net electrical impurity concentration $|N_D - N_A|$ of no less than 1.5×10^{10} donors/cm^3. In fact, it is desirable to have the largest possible net donor concentration consistent with maintaining a reasonable value for the depletion bias. This is one of the two important new practical points contained in this paper. The reason for this very strong and clear conclusion is as follows:

Because the boron ion implanted p^+ contact will withstand a far higher electric field than will the phosphorus ion implanted n^+ contact, it is highly desirable to form the junction at the p^+ contact; consequently, transmission detectors are made from n-type germanium. The difference between the depletion bias and the maximum operating bias, the "Delta" of the detector, is relatively constant for a given detector. The maximum operating bias of the detector is defined as that bias where the leakage current rises to above 3 nA. Unfortunately, the "Delta" of detectors having a phosphorus ion implanted n^+ contact is rarely more than 200 V. During the course of an experiment the depletion bias of these detectors decreases significantly because radiation damage creates p-type defects in germanium causing the net electrical impurity concentration $|N_D - N_A|$ to diminish. These decreases in depletion bias are observable at particle fluences significantly less than those required to cause energy resolution degradation in the charged-particle spectrum.

As the depletion bias decreases, the leakage current will become excessively high if one attempts to operate these transmission detectors at a "Delta" in excess of what the phosphorus ion implanted n^+ contact will allow.

Although the usefulness of these detectors can be greatly extended by monitoring the leakage current during the course of an experiment and decreasing the detector bias appropriately, this compensatory maneuver clearly has its limits. When the electric field throughout the detector becomes too low the charge collection is degraded; this degradation is further aggravated by the increasing number of radiation-damage induced charge trapping centers. These p-type defects thus cause double trouble. For a given particle fluence the depletion bias decreases as a linear function of the initial impurity concentration.

An example of this point will now be presented. Detector 550-8.6, the second detector in a three-element germanium detector telescope used in a relatively high count rate proton scattering experiment began to show an excessively high leakage current, while the first and third detectors (475-10.7 and 514-7.0) performed normally throughout the experiment. Tests conducted on these detectors following the experiment showed that all had been severely radiation damaged, but all annealed to their original condition. The difference in performance arose because detector 550-8.6 was fabricated from germanium having an impurity concentration of 7×10^9 donors/cm^3 while detector 475-10.7 was fabricated from germanium having an impurity concentration of 3.3×10^{10} donors/cm^3. The number of acceptors created in the detectors during the experiment was determined from the depletion bias changes which occurred. The depletion bias of detector 475-10.7 decreased from its damage free value of 1700 V to 1250 V, which corresponds to a change from 3.3×10^{10} donors/cm^3 to 2.4×10^{10} donors/cm^3. Thus, 9×10^9 acceptors/cm^3 were added to this detector during the experiment. The depletion bias of detector 514-7.0 fabricated from p-type germanium, increased from 1600 V to 2300 V, corresponding to an increase of 8×10^9 acceptors/cm^3 during the experiment.

Note that the increase in acceptor concentration in these two detectors is larger than the initial donor concentration in detector 550-8.6. Consequently, radiation damage caused the germanium in this latter detector to change from n-type to p-type which in turn caused a higher electric field at the phosphorus ion implanted n^+ contact which caused an excessively high leakage current.

An unexpected, and as yet unexplained, observation in the performance of detector 550-8.6 was made during this same experiment. When the detector was damaged to the point where the leakage current at the depletion bias was excessively high without beam on the scattering target, the leakage current was actually reduced by the introduction of charged particles into the detector. With beam present, the leakage current decreased to an acceptable value which allowed the detector to be used throughout the run. This "reverse" leakage current phenomenon is precisely the opposite of that observed when this detector was not severely radiation damaged. This effect has also been observed in detector 517-9.7, which also has a relatively low impurity concentration.

The impurity concentration of germanium used to fabricate a stopping detector, i.e., a detector having a lithium-diffused n^+ contact instead of a phosphorus ion implanted n^+ contact, is far less critical. Most stopping detectors are fabricated from p-type germanium although there is no fundamental reason for this and, as discussed in section V, there may be strong arguments in favor of using n-type germanium. The "Delta" of a good stopping detector is sufficiently large that the depletion bias change caused by radiation damage during the course of an experiment rarely will cause a problem. No problem of this nature has ever ocurred at IUCF.

IV. Increase in the Thickness of the Lithium-Diffused n^+ Contact Caused by Annealing

As discussed in Paper 1, a major concern about detectors with lithium-diffused n^+ contacts has been the increase in thickness of this dead layer caused by annealing following radiation damage. The dead layer thickness on detectors 514-7.0 and 514-8.6 is plotted in Fig. 1 as a function of annealing time at temperatures which varied between 90° and 150°. After about 750 hours for detector 514-7.0 and about 1000 hours for detector 514-8.6 essentially all the annealing took place at 140°C or higher. Although we lack sufficient data to determine the curve precisely, there is little doubt the increase in the dead layer growth rate that occurred in each detector around 1000 hours was caused by the increase in our annealing temperature. The thickness of the dead layer on detector 514-7.0 now appears to be increasing at a slower rate than before, probably because the source of lithium has been largely expended. Whether differences in annealing temperatures can fully explain the differences in dead layer growth rates for these two detectors as discussed in Paper 1 remains uncertain. To answer this question clearly a more controlled experiment is required. However, the important practical conclusion to be drawn from our data is that, when obtaining a stopping detector for use in an environment that will require repeated annealing, one must allow for a significant dead layer. For example, although detectors 514-7.0 and 514-8.6 have a physical thickness of 15 mm, their effective thickness is now only 12 mm.

Note that the accuracy of the dead layer measurement as determined by measuring the ratio of the 40 keV to the 100 keV photon intensities from a

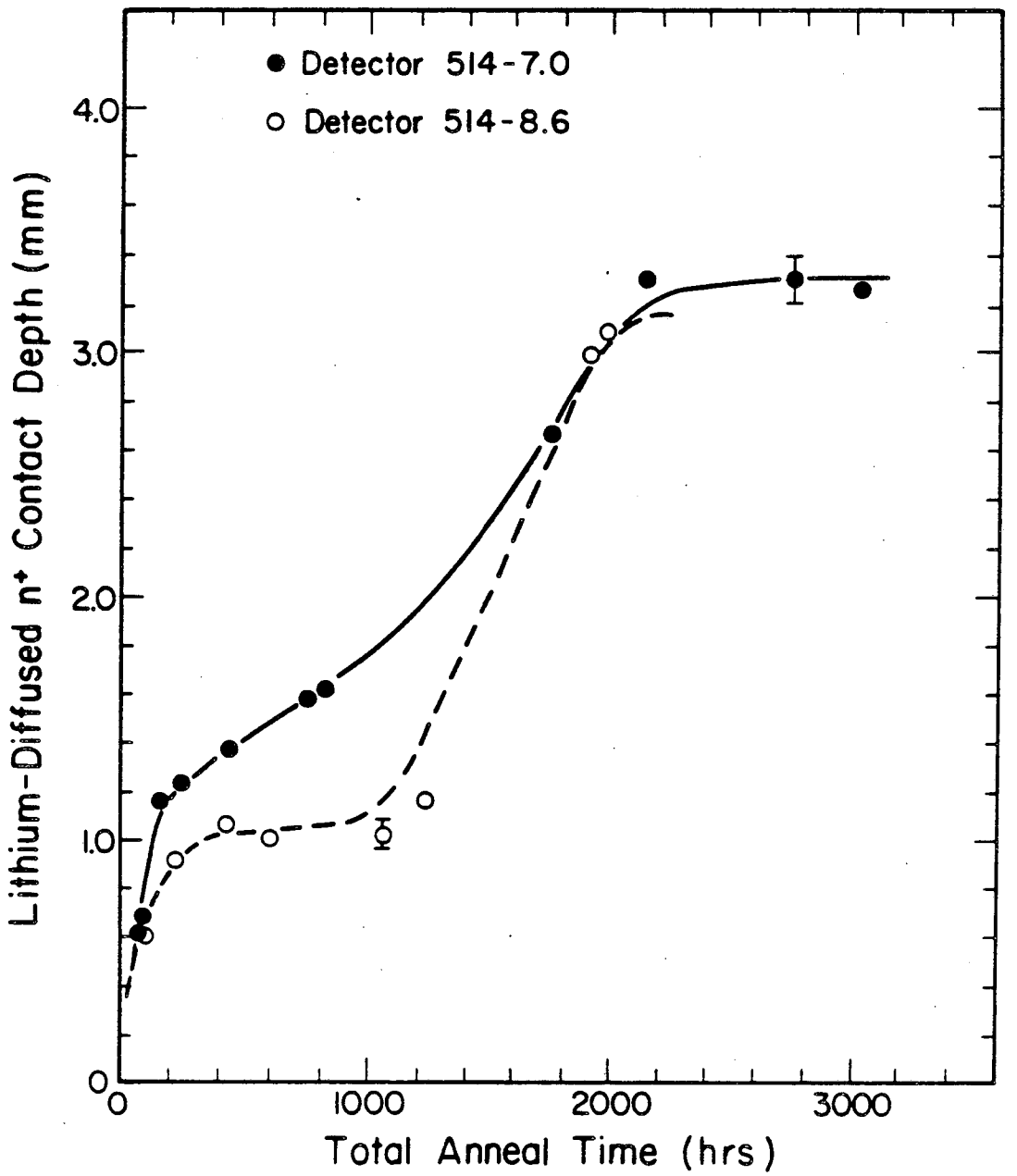

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Fig. 1 Plot of the depth of the lithium-diffused n+ contact as a function of anneal time for detectors 514-7.0 and 514-8.6.

^{153}Gd source decreases rapidly with increasing thickness of the lithium-diffused layer because of the severe attenuation of the 40 keV x ray. With this technique, accurate measurements are possible only up to a dead layer of about 2.5 mm. For measuring dead layers thicker than 2.5 mm, we compare the count rate when photons are incident on the essentially windowless boron-implanted face of the detector to when photons are incident on the lithium-diffused layer. Both the 100 keV gamma rays from ^{153}Gd and collimated 59.54 keV gamma rays from ^{241}Am have been employed although the collimated 59.54 keV gamma rays provide a considerably more sensitive measurement. As demonstrated in Fig. 2, the effective dead layer thickness of the lithium-diffused contact, as measured with collimated 59.54 keV gamma rays, decreases significantly as the bias is increased. This occurs because the concentration of lithium near the end of the diffusion tail is sufficiently low to allow a substantial thickness of this tail to be depleted. This lithium-diffusion tail also diminishes the precision of a C-V measurement for determining the bias at which the germanium not containing lithium is depleted. However, careful observation of the C-V curve in Fig. 2 reveals a change in slope at about 1200 V; the depletion bias determined by measuring both the ratio of 40 keV to 100 keV photon intensities and the count rate of collimated 59.54 keV photons through the boron-implanted contact was also about 1200 V.

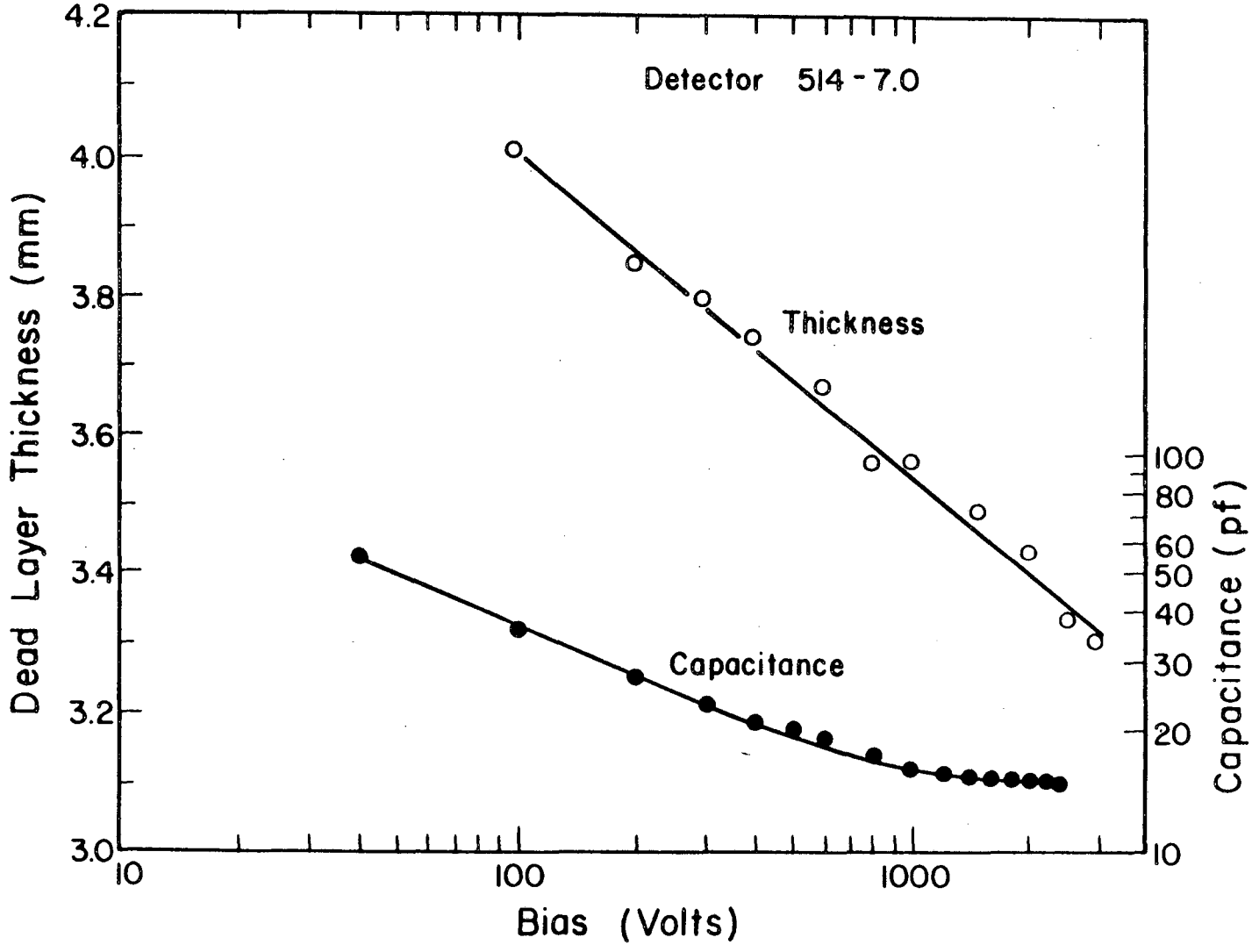

Fig. 2 Plot of the effective dead layer thickness of the lithium-diffused n+ contact as a function of bias for detector 514-7.0. The capacitance of this detector as a function of bias is also shown.

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V. Radiation Damage and Annealing

The ramifications of radiation damage and subsequent annealing have continued to play a central role in the use of germanium detectors at IUCF. The radiation damage discussed in Paper 1 was caused almost entirely by charged particles; a germanium three-element telescope (consisting of detectors 475-10.7, 501-6.7 and 514-7.0) has since been severely damaged by fast neutrons. Unfortunately, the neutron fluence and energy distribution were unknown. Two apparent differences between the effects of neutron damage and charged-particle damage were observed. First, the measured or observed ^{60}Co gamma-ray resolution of all the detectors deteriorated before there was a significant change in detector depletion bias. This was precisely the opposite of what had been observed for charged-particle radiation damage. Detector 475-10.7 had a depletion bias decrease of only 300 V during the course of the experiment, yet the ^{60}Co gamma-ray resolution deteriorated significantly. The explanation for this apparent difference between neutron and charged-particle damage is very simple and has been experimentally verified. Since the diameter of the typical collimating aperture in front of the 36 mm diameter detectors was 9 mm, only a very small portion (less than 10%) of the detector volume was damaged by charged particles. However, the ^{60}Co gamma rays sampled the entire detector volume almost uniformly and, consequently, the "good" ^{60}Co counts were coming from the undamaged portion of the detector. On the other hand, when the damage was caused by neutrons, the entire detector volume suffered damage, leaving no undamaged portion to provide the charge collection necessary to produce "good" ^{60}Co counts.

The second and more significant difference was that the annealing time required following fast neutron damage of detectors made from both n-type and p-type germanium was about three times the annealing time required when these detectors had been damaged by charged particles. This result is consistent with previous observations [9].

We now present the second of the two important new practical points contained in this paper. Detector 514-7.0 fabricated from p-type germanium required about three times the annealing time the two detectors (475-10.7 and 501-6.7) fabricated from n-type germanium required. Although data on the annealing times required following the charged-particle damage of detectors made from n-type germanium relative to detectors made from p-type germanium had existed at IUCF prior to the experiment that caused neutron damage, this annealing time difference had not been previously recognized. These results are summarized in Figs. 3 and 4 which show the annealing history following both neutron and charged-particle damage. The depletion bias was determined by measuring the ratio of the 40 keV to the 100 keV photon intensities from a ^{153}Gd source.

We are now preparing some experiments that should more quantitatively determine this important annealing time difference, but the observations reported here indicate that it is very desirable to <u>use only detectors made from n-type germanium in a high-radiation environment</u>. As a routine consequence of doing an experiment, the 500 hours (~ 21 days) of annealing required for detector 514-7.0 (Fig. 3) is excessive. However, the 150 hours required to anneal the detectors made from n-type germanium is not unreasonable. Another reason for avoiding unnecessarily long annealing times is the additional increase in the thickness of the lithium-diffused n^+ contact that occurs during the additional annealing time.

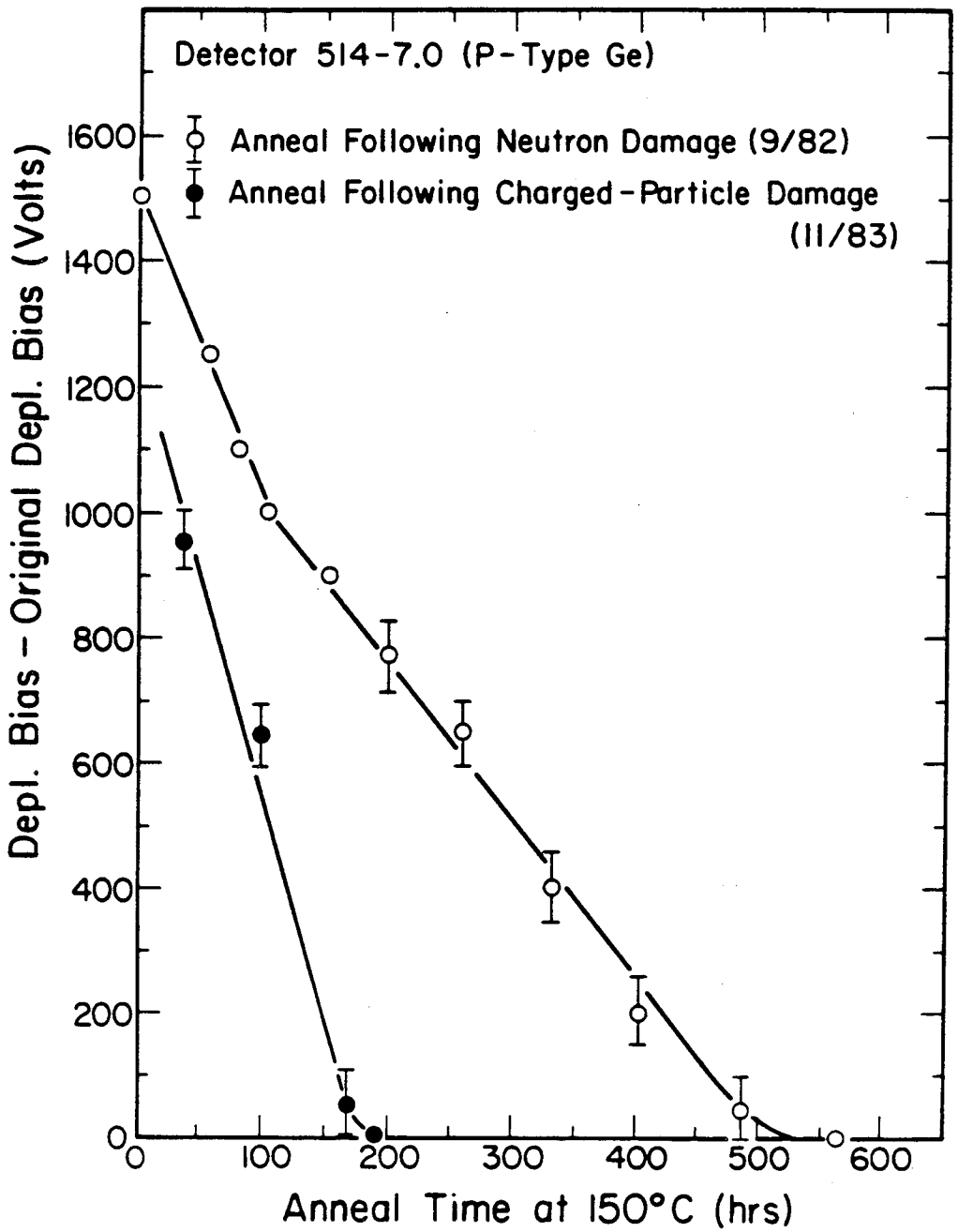

Fig. 3 Plot of the difference between the original (pre-radiation damage) depletion bias and the depletion bias as a function of anneal time for detector 514-7.0 fabricated from p-type germanium.

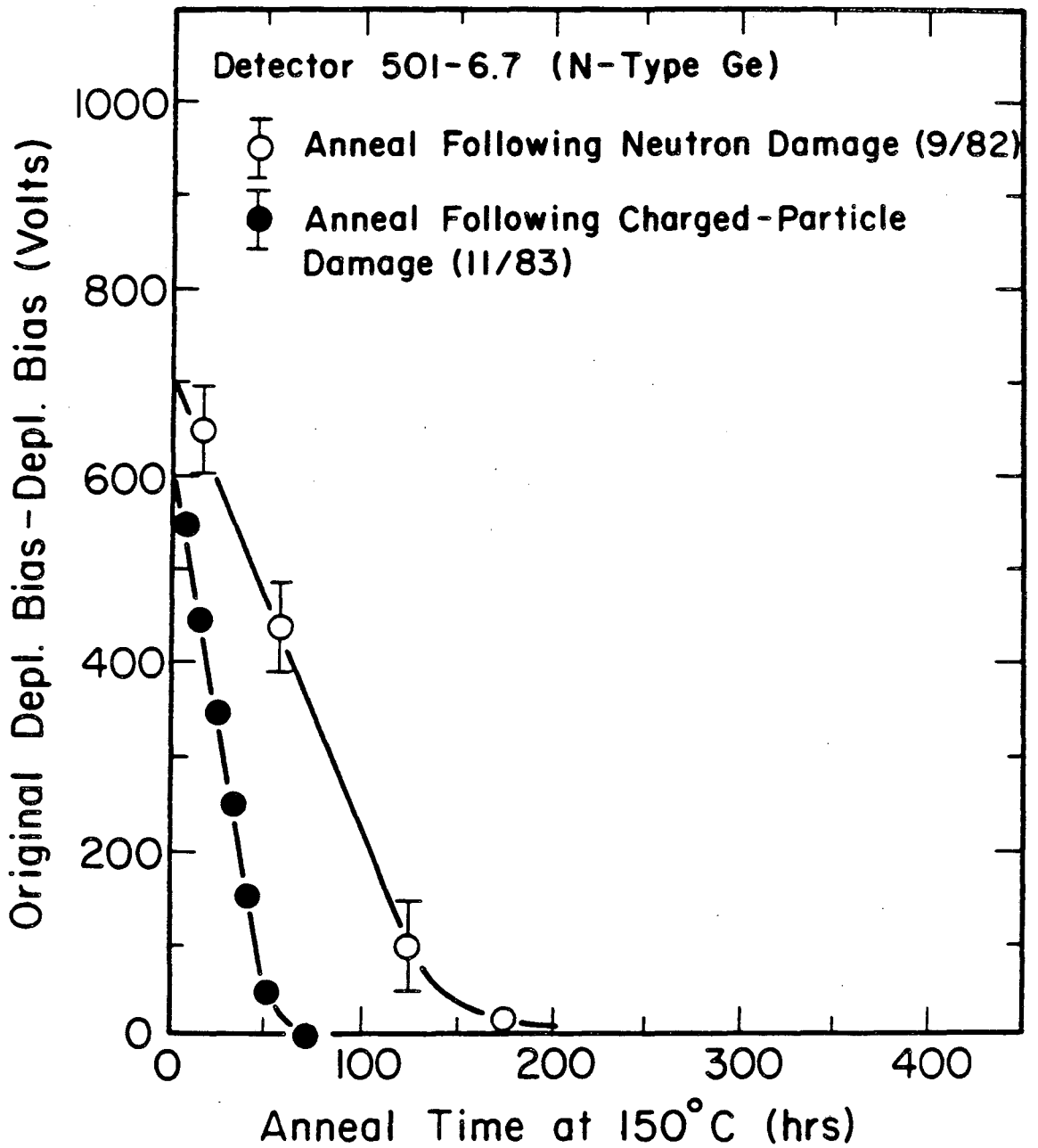

Fig. 4 Plot of the difference between the original (pre-radiation damage) depletion bias and the depletion bias as a function of anneal time for detector 501-6.7 fabricated from n-type germanium.

The discovery that detectors fabricated from n-type germanium require significantly shorter annealing times than detectors fabricated from p-type germanium has important implications for a much larger fraction of the germanium detector world than the miniscule fraction which charged-particle detectors occupy. Germanium coaxial detectors used for measuring gamma rays totally dominate this world. If a coaxial detector is to be used in an environment where radiation damage is likely, the detector of choice has the reverse-electrode configuration [10,11]. To establish a high electric field at the periphery, reverse-electrode coaxial detectors are usually fabricated from n-type germanium. However, some reverse electrode coaxial detectors contain an appreciable amount of very high-purity p-type germanium. Consequently, the n-type vs p-type annealing difference casts serious doubt on the conclusions reached concerning thermal cycling very lightly neutron-damaged reverse-electrode coaxial detectors [12], unless one is certain the detectors were fabricated from almost entirely n-type germanium. Furthermore, the conclusion that the much shorter annealing time required for restoration of a badly neutron-damaged reverse-electrode coaxial detector (compared with a conventional-electrode coaxial detector exposed to the same neutron fluence) can be completely attributed to the relative sensitivities of the respective electrode configurations to hole trapping [13] must be modified to include the n-type vs p-type annealing difference observed with planar detectors.

VI. Conclusion

In Paper 1 we concluded that germanium detector telescopes would operate reliably for several days in most intermediate-energy nuclear research applications. The experience since then has proved the correctness of that statement many times over. Table 1 shows emphatically that these detectors can be used and reused indefinitely in spite of radiation damage sustained during experiments because they can be successfully annealed. Several of these detectors have gone through over forty radiation-damage and anneal cycles. Today all but three of these detectors have essentially the same operating properties as when received from LBL and only one of the three detectors on the currently inactive list is considered retired. Even this lone 1.2 mm thick detector (541-12.8) could be revived in a slightly smaller diameter form by removing the outer 1 mm wide lithium-diffused ring that has diffused through to the p^+ contact causing an electrical short across the detector, but since it is a relatively cheap device, the effort to put this detector back into service at IUCF will probably not be made. Except for the increase in thickness of the lithium-diffused n^+ contact on stopping detectors, the annealing process has not as yet had any adverse effects on the properties or usability of the detectors.

Although the goal of the LBL-IUCF collaboration was only to develop germanium charged-particle detector telescopes, this program has provided important insight on the properties of germanium detectors in general—insight that is having widespread influence on the much larger germanium gamma-ray detector world. In fact, we can say that the LBL-IUCF collaboration now has a second, unofficial, goal of studying detector physics with an emphasis on radiation-damage effects.

VII. Acknowledgements

We would like to thank the many people who have contributed to the continued development of the germanium detector telescope system. At LBL, F. Goulding has provided continuing advice, interest and support; E. Haller and W. Hansen provided advice and assistance in advancing detector technology; D. Landis and N. Madden provided consultation on electronics; D. Malone provided mechanical design assistance. At IUCF, we would like to thank K. Komisarcik for his efforts in maintaining the germanium detector systems and for his assistance in performing many of the measurements quoted here. H. Kraner, T. Raudorf and R. Trammell have contributed many discussions on radiation damage which have been extremely helpful. We also thank Paula Pehl for her editorial assistance.

Appendix

Transmission Detector Technology Update

Transmission detectors usually require relatively thin dead layers on both the entrance and exit surfaces; these surfaces normally also provide the electrical contacts. Thin p^+ contacts which can consistently sustain high electric fields with very low leakage current can be produced by boron ion implantation. Thin n^+ contacts can be formed by phosphorus ion implantation, although the resultant contacts are of much lower quality (sustain lower electric field, higher leakage current) compared to boron implanted contacts. Most of the transmission detectors presently at IUCF were fabricated using the process described previously [14,15]. However, later improvements in this process were used in fabricating the more recent detectors. For completeness, the full updated process is described below.

After a germanium crystal slice of appropriate impurity concentration, thickness and diameter is selected and cut, the two opposing faces are lapped and then chemically polished using 7:2:1 (HNO_3:HF:Red fuming HNO_3) etch to remove all the mechanical damage. Immediately before phosphorus ion implantation, the germanium is given a brief etch in 7:2:1, quenched and rinsed in distilled deionized water (DDW), followed by an ~10 min soak in 1% HF to remove the oxide layer on the surface. The germanium is then rinsed with DDW and blown dry with nitrogen gas. Phosphorus ion implantation is carried out with the germanium cooled to liquid nitrogen temperature. The implant dose is 6×10^{14} ions/cm^2; the ion energy is 25 keV. (The previously reported dose of 1×10^{15} ions/cm^2 was actually 6×10^{14} cm^2 because of an error in the

calibration of the ion implanter.) The beam current is maintained at or below ~1 μA to reduce sample heating. The implant angle is ~8° off the crystal axis to avoid channeling. After implantation, but before thermal annealing, the implanted surface is carefully masked using picein wax and the rest of the germanium is given a brief spray-etch with 7:2:1 followed by a rinse with DDW. This step removes surface contaminants created during implantation which may diffuse into the germanium while the contact is being annealed and cause charge trapping in the detector. After the picein is removed, the germanium is annealed in an argon atmosphere as follows: 150°C for 40-60 hours; from 150° to 330°C in 3 hours; 330°C for 30 minutes; slow cool down to room temperature. Next, the implanted contact is masked again with picein and the p^+ contact is formed on the opposite surface by boron ion implantation (1×10^{14} ions/cm^2, 25 keV, room temperature); pre-implant treatment is the same as that for the phosphorus contact. Following implantation, the boron contact is metalized with palladium by e-gun evaporation. Before the detector can be used, the side surface is etched in 7:2:1 for ~30 sec, quenched and rinsed in methanol, then blown dry with nitrogen. The contacts are protected by etch-resistant tape during this step. Hydrogenated amorphous germanium is then sputtered over the side surface for passivation [16]. This has proven to be a very durable surface passivation which has allowed these telescope detectors to be used over a period of many years with numerous vacuum-to-room air and thermal cycles without significant changes in detector characteristics.

The essential difference between this and the previously published fabrication process is that a lithium-diffused ring surrounding the phosphorus contact is no longer used. It was found that, by careful handling to avoid

mechanical damage or under-etching of the edge of the phosphorus contact during processing, a lithium ring is not necessary. This simplifies fabrication and greatly facilitates the reprocessing of transmission detectors. Furthermore, transmission detectors can be safely annealed to repair radiation damage for an indefinitely long period of time or at higher temperatures without the risk of extended diffusion of the lithium ring which could "short-out" a thin detector (see section VI) or cause a decrease in the effective area of the detector. The absence of a lithium ring also means that thinner transmission detectors are more feasible. Another difference from the previously published process is that the phosphorus-implanted contacts are left unmetalized because evaporation of metal over the contacts apparently degrades their performance. The resultant increases in infrared induced leakage current and contact resistance do not affect the performance of these detectors in this application.

The maximum electric field that the phosporus-implanted contact can sustain before the onset of an unacceptably high leakage current is still, in most cases, limited to several hundred V/cm. While improvements in this area could alleviate some of the constraints imposed on the operation of these detectors as discussed in this paper, no proven process has yet been developed that can consistently produce n-type blocking contacts with the ability to sustain a substantially higher electric field.

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