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Ion-implanted charge collection contacts for high purity silicon detectors operated at 20 mK

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We have developed a technique for fabricating high resolution, ohmic contacts for cryogenic silicon detectors operated at temperatures well below 1 K. In this paper, we give a detailed description of the techniques used to fabricate these boron-implanted contacts, and present characterization data obtained on 24 test samples studied during the design phase of our program. We then describe the fabrication and operation of a 23 g prototype silicon hybrid detector which simultaneously senses both the phonons and ionization produced by a single event, and which incorporates these new contacts into its design. Finally, we present data obtained using a radioactive source of ^{241}Am and this detector operated at 20 mK, and conclude that the contacts are fully sufficient for applications in particle astrophysics as well as in many other areas of physics. © 1995 American Institute of Physics.

I. MOTIVATION AND BACKGROUND

Several groups throughout the world are developing phonon-sensitive cryogenic particle detectors for primary applications in particle astrophysics.¹⁻³ A variety of detector substrates, including insulators (e.g., Al_2O_3 , LiF, and CaF), semiconductors (e.g., Ge and Si), and superconductors (e.g., Nb, In, and V), are being investigated for these purposes. It is believed that arrays of cryogenic detectors composed of different target materials could be used to determine both the amount and nature of the dark matter in the universe.⁴

These novel detectors typically operate by using neutron transmutation doped germanium (NTD Ge) thermistors or thin films of superconducting metal to sense phonons generated by particles elastically scattering off nuclei or electrons in high-purity crystals operated at temperatures well below 1 K. In some designs, the detectors are also sensitive to the ionization produced by a single event. Such cryogenic "hybrid" detectors are being developed by two groups in the Center for Particle Astrophysics Dark Matter Collaboration, one at UC Berkeley and one at Stanford University. Calibration experiments performed at 20 mK with germanium hybrid detectors exposed to radioactive sources of photons and neutrons are described in Ref. 5. Currently, the major limitation of the Ge devices is their rather poor energy resolution in the ionization measurement for events which interact within a few tens of microns of the boron-implanted surfaces. Work is underway to better understand the origin of these effective "dead layers" in the germanium detectors, and it is expected that a more thorough and systematic study of the ionization collection contacts will provide the information necessary to remedy this situation in the future.

In this paper, we describe the design, fabrication, and characterization of ion-implanted charge collection contacts for silicon hybrid detectors operated at 20 mK.⁶ These detectors utilize NTD Ge thermistors for the phonon measurement

and are being developed at UC Berkeley, in parallel with work there on germanium hybrids. Silicon hybrid detectors utilizing superconducting films to make Schottky barrier contacts are also being investigated for these applications by the group of Cabrera at Stanford University,⁷ and a group in England⁸ worked briefly on Si hybrid detector development, but recently abandoned that aspect of their research program.

One particularly attractive feature of silicon hybrid detectors is that, if present, any effective dead layers at the detector surfaces are intrinsically less problematic than those observed in Ge detectors. This is due to the significantly smaller stopping power of silicon ($Z=14$) compared to that of germanium ($Z=32$), which translates into event locations typically beyond that of any dead layer introduced by ion-implanted contacts. However, unlike for Ge detectors, before beginning this research we found no references in the literature describing the fabrication of ohmic charge-collection contacts for silicon detectors operated at these extremely low temperatures. It was the need for ionization contacts appropriate for NTD Ge-based silicon hybrid detectors that originally prompted us to do the work reported in this paper. We note, however, that our results presented here are quite general, and are not limited in application to the fabrication of hybrid detectors operated at 20 mK.

Below, we first discuss various physics constraints on the general design of semiconductor hybrid detectors. We then summarize our work to successfully develop ionization collection contacts for cryogenic silicon detectors. Data from several characterization experiments performed on test samples of silicon are presented. Finally, we provide a detailed description of the technique used to fabricate reliable ohmic contacts for a prototype cryogenic silicon hybrid detector, and show first results from an experiment performed with that detector operated at 20 mK.

II. GENERAL DETECTOR REQUIREMENTS

The ability to simultaneously detect phonons and ionization is crucial for a dark matter or neutrino experiment, where the expected event rates are very small (\sim few events/keV recoil energy/kg of detector/day), because it allows one to discriminate between nuclear recoils, i.e., dark matter signals, and electron recoils, i.e., photon background, by virtue of their differing ionization efficiencies.⁹ In general, semiconductor hybrid detectors applicable to a WIMP dark matter or neutrino experiment should have: (a) an energy threshold ≤ 1 keV and a rms energy resolution ≤ 500 eV for both phonons and ionization, (b) a mass (per detector in array) of at least ~ 100 g, (c) an operating temperature < 0.1 K, so that both the heat capacity and the population of thermal phonons in the detector are negligible, and (d) nearly 100% charge collection efficiency for an applied ionization collection bias ~ 1 V. For typical detector geometries, the latter requirement corresponds to an applied electric field of ~ 1 V/cm, which is 1000 times smaller than that used for more conventional ionization detectors operated at 77 K. Hybrid detectors require this low operating voltage because at higher voltages the population of secondary phonons produced by drifting charges can become comparable to the phonon signal produced directly by the initial scattering interaction, thereby reducing the electronic versus nuclear recoil discrimination capability. This constraint on the operating voltage of the ionization channels implies that detectors must be fabricated using extremely pure substrates, and with processing temperatures as low as possible, so that the total number of impurities (and therefore the trapping of drifting charges) in the substrates is minimal. In most detector designs, the phonon channels are not as sensitive to the total impurity concentration as are the ionization channels, although for hybrid devices which are sensitive to nonthermal and quasidiffusive phonons (rather than just thermal phonons), the presence of structural defects and atomic impurities, including isotopic impurities can become important.¹⁰

The high-purity germanium hybrid devices developed and routinely operated at UC Berkeley typically have $< 10^{11}$ cm^{-3} total impurities ($N_A + N_D$), and this is sufficient for our applications. Unfortunately, it is both difficult and quite expensive to obtain (100), *p*-type, float-zone, single-crystal silicon of comparable purity, although at least three companies (Topsil, Westinghouse, and Wacker) can produce reasonably pure Si ingots ($N_A + N_D < 10^{12}$ cm^{-3}) with relatively few structural impurities. In collaboration with the group of Cabrera at Stanford, several samples of high-purity silicon are being evaluated specifically for their charge collection efficiency as a function of applied electric field at 0.3 K. These studies are performed using superconducting titanium Schottky-barrier contacts which have not been heated above 150 °C. The results from these characterization experiments are being used both to select the best source (vendor) of silicon for our upcoming large scale experiments, and to develop a general model for the somewhat complex physics of ionization collection at subkelvin temperatures.

Finally, although Schottky-barrier contacts work well for these characterization studies, as well as for several other applications (including the simultaneous detection of

phonons and ionization, in some detector designs), such contacts are not appropriate for use with the Berkeley-style hybrid detectors which utilize NTD Ge thermistors for the phonon sensors. In the simplest design, a Schottky-barrier contact made simply from a uniform film of superconducting metal on the entire face of a Berkeley-style detector would likely inhibit the coupling of thermal phonons to the NTD Ge thermistor, and a Schottky barrier fabricated with normal metal would have too large a heat capacity to be useful. Therefore, for the germanium and silicon hybrid detectors currently under development at UC Berkeley, the ionization collection contacts are ohmic, and are fabricated using ion implantation.

III. ION-IMPLANT AND ANNEAL STUDIES

Our specific goals for the ohmic ionization collection contacts developed in this study were to fabricate *p*⁺ contacts with the following properties: (a) a uniform and relatively shallow dopant profile with the peak boron concentration at or near the surface of the detector, (b) not an excessive net concentration of dopant atoms, but above the metal-insulator transition at the detector surface (even after annealing), (c) a reasonably low sheet resistance ~ 50 Ω/\square , and (d) stable and reproducible characteristics when operated at temperatures below 1 K.

For this purpose, we boron implanted and annealed 24 0.5-mm-thick samples taken from three equivalent (100) commercial silicon wafers. As purchased, these three polished wafers were *n*-type, lightly doped with phosphorous, and had resistivities of ≈ 10 $\Omega \text{ cm}$. *n*-type wafers were chosen so that we could most effectively measure the sheet resistance of each boron (*p*-type) implanted sample. The three wafers were first cleaved in half, and then cleaned in solvents (TCA, acetone, and methanol) and soaked in 1% HF for 10 min. The wafer halves, excluding a "control" sample taken from each half, were individually implanted cold (77 K), with the substrates mounted at an angle of 7° relative to the ion beam. Except for the control samples which received no implants, all substrates were implanted with boron at 33 keV. Three different implant doses were used: 3×10^{14} , 1×10^{15} , and 3×10^{15} cm^{-2} , and eight different anneal schedules. The various samples were annealed in a homebuilt furnace at temperatures ranging from 400 to 900 °C (one temperature per sample), and for either 30 or 60 min. In all cases the samples remained in an Ar-purged quartz tube until the temperature dropped to below 50 °C. Most likely, a rapid thermal anneal would have also satisfied our requirement that the silicon processing introduce nominal additional electrical impurities into the Si substrates. However, this technique was not used in our contacts study because it cannot be used to fabricate Ge and Si hybrid detectors, where the substrates are large (~ 10 cm^{-3}) and thick (~ 1 cm), and are susceptible to cracking when thermally stressed.

The 24 processed samples were characterized visually (e.g., to qualitatively evaluate how amorphous the substrate was after being implanted and in some cases also annealed), and the resistivity of each was measured at room temperature, using a four-point probe. A simple two-point measurement of the resistance of each sample was made at 300, 77,

TABLE I. Characterization of 24 test samples prepared in this study on ohmic contacts. The samples were fabricated using three 500- μm -thick, magnetic CZ, 10 Ω cm (P-doped), (100) silicon wafers. All implants were performed at 77 K, with the Si substrate tilted 7° relative to the 33 keV boron ion beam.

Anneal temperature (°C)	Four-point probe (Ω/\square)	Two-point resistance (Ω)			Visual inspection of silicon surface (G=greyish=crystalline) (S=silvery=amorphous)
		300 K	77 K	4 K	
No implant					
Not heated	64, -64	G
500 (30 min)	53, -53	G
600 (30 min)	...	58	8.8×10^6	$>30 \times 10^6$	G
900 (30 min)	58, -58	55	108×10^3	...	G
$3 \times 10^{14} \text{ cm}^{-2} \text{ B}^+$ (implant at 77 K)					
Not heated	57, -57	G
400 (30 min)	361, -372
450 (30 min)	560, -396
500 (30 min)	582, -504	2525	31900	182400	G
600 (30 min)	679, -557	2050	28150	88000	G
900 (30 min)	352, -339	419	634	586	<S
$1 \times 10^{15} \text{ cm}^{-2} \text{ B}^+$ (implant at 77 K)					
Not heated	56, -57	S
400 (30 min)	467, -441
450 (30 min)	317, -306	G/S
450 (60 min)	274, -267	G/S
500 (30 min)	169, -168	281	829	708	G/S
500 (60 min)	148, -143	115	107	129	G
600 (30 min)	160, -157	141	184	210	G
900 (30 min)	178, -177	193	230	197	G
$3 \times 10^{15} \text{ cm}^{-2} \text{ B}^+$ (implant at 77 K)					
Not heated	61, -61	S
450 (60 min)	143, -142	G/S
500 (30 min)	60, -60	56	91	68	G/S
500 (60 min)	57, -55	41	33	36	G
600 (30 min)	58, -58	52	46	50	G
900 (30 min)	84, -83	56	52	51	G

and 4 K using a Fluke 77 multimeter. The results of these various measurements are shown in Table I. As can be seen from Table I, several of the samples evaluated had an acceptably low sheet resistivity at room temperature ($<100 \Omega/\square$). However, for many of these samples, the implanted layer "froze out" and became poorly conducting when cooled to 77 or 4 K. In terms of their low temperature electrical conductivity, the best samples measured were those boron implanted with a dose of $3 \times 10^{15} \text{ cm}^{-2}$ and annealed at $\geq 500^\circ\text{C}$. A subset of the samples listed in Table I were further characterized using the Rutherford backscattering (RBS) technique, as discussed in the next section of this paper.

IV. CHARACTERIZATION OF IMPLANTED AND ANNEALED SAMPLES

Rutherford backscattering spectrometry (RBS), in conjunction with channeling in the $\langle 110 \rangle$ direction using a 1.80 MeV He ion beam, was used to study the damage profiles of the Si samples after boron implantation. Figure 1(a) shows a series of RBS spectra aligned in the $\langle 110 \rangle$ direction from samples implanted with 3×10^{14} , 1×10^{15} , and 3×10^{15} boron ions/cm². Spectra of the annealed samples (500 °C for 60 min) for the 1 and 3×10^{15} /cm² implants are also shown in Fig. 1(a). For the sample implanted with low dose of B

(3×10^{14} /cm²), a damage peak is observed in the RBS spectrum due to the direct scattering of the He ions at the displaced Si atoms. This damage peak reaches the random level for the sample implanted with 1×10^{15} /cm² indicating the formation of an amorphous layer at the sample surface ($\sim 1100 \text{ \AA}$ thick). For even higher implant dose (3×10^{15} /cm²), this amorphous layer becomes thicker ($\sim 1300 \text{ \AA}$).

Figure 1(b) shows the $\langle 110 \rangle$ aligned RBS spectra from the samples implanted with 1 and 3×10^{15} /cm² annealed at 500 °C for 60 min. The $\langle 110 \rangle$ RBS spectrum of an unimplanted Si crystal is also shown for comparison. The peak at 1020 keV arises from backscattering of He ions at the Si surface. The higher Si surface peak in the spectra taken from the implanted and annealed samples indicates that these samples have a slightly thicker random surface layer ($\sim 100 \text{ \AA}$), probably in the form of surface oxide due to the annealing process. Notice that the backscattered yields of these samples below the surface are only slightly higher than that from the unimplanted sample. This reveals that the amorphous layers in these two samples created by the B implants are completely regrown by solid phase epitaxy at 500 °C. There is, however, residual structural damage in these annealed samples as suggested by the slightly higher backscattering yields in the RBS spectra. These residual defects

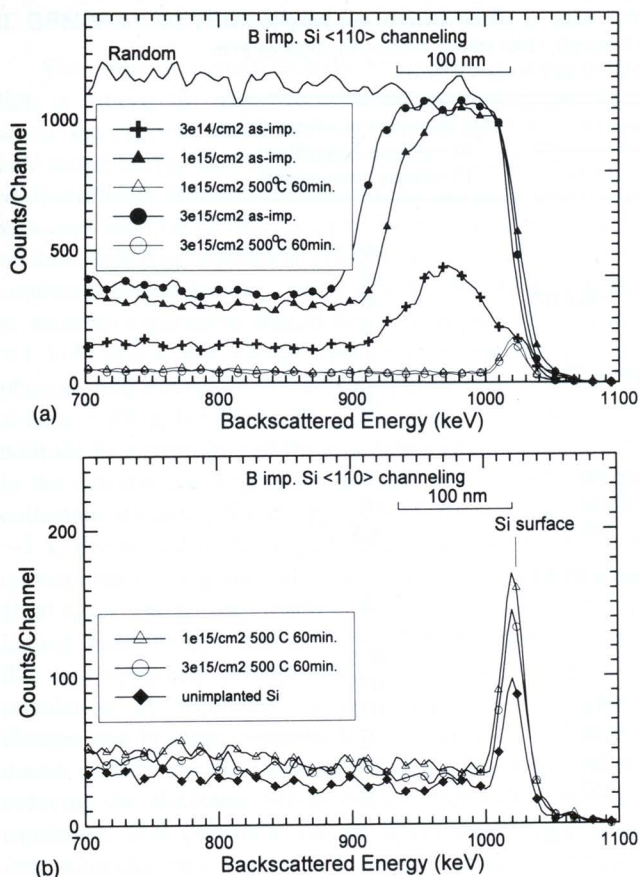


FIG. 1. Superimposed RBS pulse height spectra obtained by scattering 1.80 MeV He ions off three silicon samples boron implanted with $3E14$, $1E15$, and $3E15/cm^2$ boron atoms at 33 keV. (a) RBS spectra for samples "as implanted," and after annealing at 500 °C for 60 min; (b) RBS spectra shown on an expanded vertical scale for the two annealed samples shown in (a). The peaks at 1020 keV result from the backscattering of He ions at the Si surface. The observed plateau in the RBS spectra of the annealed samples at lower energies (i.e., deeper into the Si substrate) is evidence that the amorphous layer produced during the boron implantation was effectively regrown during the annealing process.

are most probably in the form of dislocation loops in the regrown layers.

The RBS results correlate well with the electrical measurements presented in Table I. For ohmic contacts, higher dopant concentration is desired. However, doping with ion implantation introduces structural defects which increases as a function of implant dose. Our RBS and electrical results on the B-implanted Si samples suggest that the sample implanted with 3×10^{15} B/cm² and annealed at 500 °C for 60 min has low contact resistivity due to its high dopant concentration as well as good crystallinity with very low level of structural defects in the implanted and regrown surface layer.

V. FIRST RESULTS FROM A 23 g SILICON HYBRID DETECTOR

The results of the implant and anneal studies described above were used to design ohmic ionization collection contacts for a 23 g prototype Si hybrid detector (Fig. 2), named Silicon-1 (Si-1), which was recently characterized at UC Berkeley. The device is 3.8 cm in diameter and 0.85 cm thick. It has two concentric ionization channels, one of which

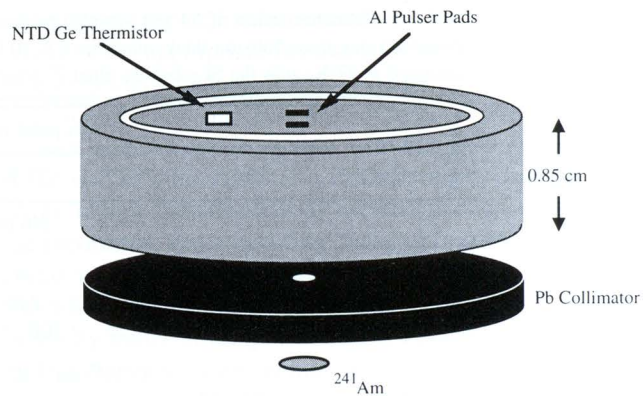


FIG. 2. Schematic diagram of the 23 g silicon-1 hybrid detector and radioactive source/collimator arrangement discussed in the text. The 3.8-cm-diam device has two concentric ionization channels which are separated from each other by a 0.5-mm-wide groove. A NTD Ge thermistor provides the phonon measurement for each event. Aluminum pulser pads deposited on one face of the crystalline substrate are used to calibrate the phonon measurement. In this study, the pads were also used to measure the resistance of the implanted ionization contact at 20 mK.

can be used to tag events occurring within 4 mm of the detector edge. These two independent ionization channels (" Q_{main} " and " Q_{guard} ") are separated from each other on both the top and bottom faces of the detector by 0.5-mm-wide grooves, which were produced by precisely masking and chemically etching the boron implants after the substrate was thermally annealed. The phonon signals are provided by $1.5 \times 1.5 \times 0.3$ mm³ NTD Ge 29 thermistors that are attached to one face of the detector using a minimal amount of conductive epoxy. The detector substrate is (100), float-zone, undoped, high-purity silicon (*p*-type) that was obtained from Topsil. The substrate has a nominal resistivity of 8 kΩ cm and a measured minority carrier lifetime of 1.7 ms, as measured by the photoconductive decay method (ASTM F28-75). The ohmic ionization collection contacts on Si-1 were made by implanting $3e15$ cm⁻² boron ions at 33 keV. During the boron implantations, the Si substrate was cooled to 77 K, and the substrate was mounted at a 7° tilt relative to the axis of the ion beam. The (front and backside) implants were annealed simultaneously at 500 °C for 60 min. A complete description of this detector and a discussion of the results obtained with it operated at 20 mK will be presented in an upcoming paper.⁶

The implanted contacts on Si-1 were evaluated at 20 mK by measuring the electrical resistance of the ion implanted layer on one face of the detector using a four-wire ac resistance bridge at low power. For this measurement, contact to the implanted layer was established by wedge bonding four 0.0007-in.-diameter Al(Si) wires to two 2.5-mm-long \times 0.5-mm-wide-aluminum "pulser" pads evaporated on the top face of the detector. The innermost edges of the Al pads were separated by 2 mm, and the resistance of the boron implanted layer between the two pads was measured to be 19.2 Ω. This is approximately five times lower resistance than is typically measured for Ge hybrid detectors, which also work well but are fabricated using an implant and anneal schedule inappropriate for use with Si detectors. The measured, low resistance of the Si-1 ionization collection contacts was an important

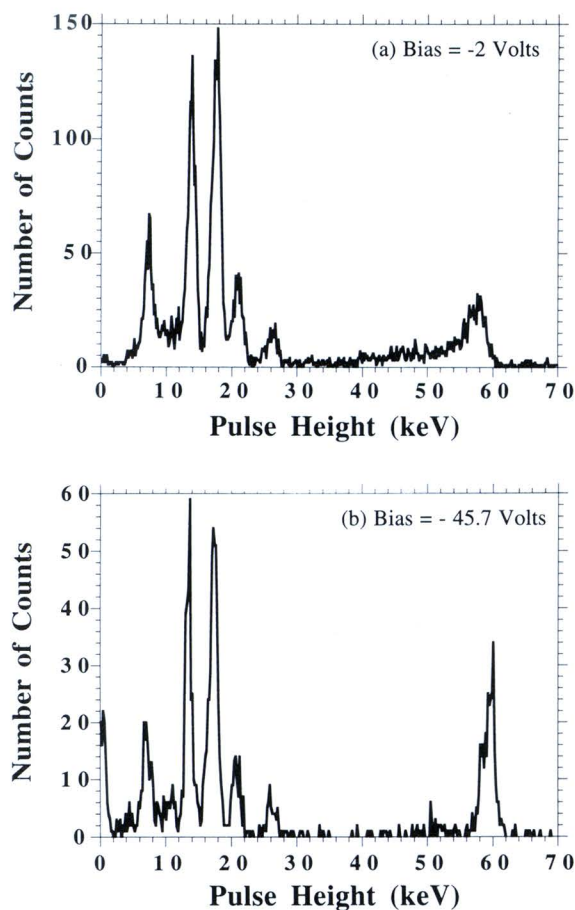


FIG. 3. Ionization pulse height spectra obtained with the 0.85-cm-thick Si-1 detector operated at 20 mK and biased at (a) -2 V; (b) -45.7 V. The peaks shown correspond to photon calibration events of 8, 14, 18, 21, 26, and 60 keV.

result, for it confirmed that these contacts do not freeze out at subkelvin temperatures. In addition, we found that the contacts did not break down electrically at applied voltages up to 45.7 V (applied field of 53.8 V/cm), which was the maximum provided by our power supply.

The Si-1 detector was characterized at 20 mK using a collimated source of ^{241}Am which emits photons at 60, 26, 21, 18, and 14 keV. In addition, the detector was exposed to secondary x rays produced by the fluorescence (by photons from the ^{241}Am source) of Pb (10 and 13 keV x rays) and Cu (8 keV x rays) near the detector. Ionization pulse height spectra obtained with the Si-1 detector operated at 20 mK and with bias voltages of -2 and -45.7 V are shown in Fig. 3. In both spectra, peaks are clearly seen at energies corresponding to photon events of 60, 26, 21, 18, 14, and 8 keV. As measured using the 14 keV photon peak, the rms energy resolution of this detector was observed to be 630 eV for a -2 V bias [Fig. 3(a)] and 520 eV for a -45.7 V bias [Fig. 3(b)]. Spectral structure in the region between the peaks at 8 keV (Cu) and 14 keV (Am) is generally consistent with the expected distribution of *L*-shell fluorescence x rays produced in the Pb collimator, combined with an additional contribution from the low energy tails of the higher energy peaks. The dependence on bias voltage of both the absolute pulse height and the energy resolution (more specifically, the pres-

ence or absence of a low energy “tail”) for each peak in the spectra is closely related to the purity ($N_A + N_D$) of the detector substrate, rather than to the quality of the ionization collection contacts. Bulk trapping effects due to the presence of electrical impurities in detectors operated at subkelvin temperatures has been studied in both Ge¹¹ and Si.^{7,12} As can be also seen in Fig. 3, the dependence on bias voltage of the peak heights and shapes is most pronounced for the 60 keV events. This is primarily because the $1/e$ attenuation length for 60 keV photons in silicon (1.5 cm) is larger than the thickness of the detector substrate (0.85 cm), and therefore the device is well illuminated by these photons at all depths. In contrast, nearly all of the lower energy (Am, Pb, and Cu) events occur within ~ 100 μm (8 keV) to ~ 1 mm (21 keV) of the detector surface. Thus, for the lower energy events, the electrons and holes produced by the initial interaction need to travel a relatively short distance through the crystal before producing (by virtue of their image charges) an ionization signal in the detector. For a large fraction of the 60 keV events, however, the electrons and holes must travel several mm to reach the contacts, and are therefore much more likely to be adversely affected by impurity sites along the way. The effects of this energy loss mechanism become increasingly more pronounced at low drift fields. This is readily seen by comparing the spectra shown in Figs. 3(a) and 3(b): at low applied field (2 V/0.85 cm) there is a noticeable low energy tail on the 60 keV peak, and at relatively high field (45.7 V/0.85 cm) the tail is nearly absent. Another intrinsic difference between the 60 keV photon events and the events produced by the lower energy photons is that Compton scattering accounts for approximately 50% of the primary interaction cross section for 60 keV gamma rays in silicon. Compton scattering accounts for at most a few percent of the interaction cross section for the lower energy x rays. And finally, we note that the near absence of a 60 keV Compton continuum and Compton “edge” in these data is well understood in terms of the source energy and known detector-source geometry.

VI. DISCUSSION

We have successfully designed, fabricated, and characterized ohmic contacts for use as ionization collection electrodes on cryogenic, high-purity silicon detectors. These contacts maintain their low sheet resistance and perform very reliably even after being thermally cycled several times from room temperature down to a typical detector operating temperature of 20 mK. We believe that our results presented above can be readily and easily applied to a range of applications in cryogenic physics and solid state physics, in addition to the further development of cryogenic particle detectors.

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