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RESTORATION OF LITHIUM DRIFTABILITY IN SOME VACUUM-GROWN GERMANIUM CRYSTALS FOR γ-RAY DETECTORS

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Paper presented at the Twelfth Scintillation and Semiconductor Counter Symposium, Washington, March 1970

ABSTRACT

Germanium crystals used for γ-ray detectors frequently exhibit slow or non-uniform lithium-ion drift. The presence of oxygen is one known cause. However, it has been found in these laboratories that in large ($\sim$ 1 kg) germanium crystals grown by the Czochralski method under vacuum, the presence of a fast diffusing impurity (probably copper) also leads to variable drift.

Normal, uniform drift in these crystals has been restored by using gallium-indium alloy as a getter at $\sim$ 800°C. The method is described in detail and the effects on crystal resistivity, dislocation density and lithium drift rate are presented.

Gamma-ray detectors fabricated from gettered material show high resolution characteristics.

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CONTENTS

(continued)

1. INTRODUCTION

Germanium crystals used in the preparation of lithium-drifted γ-ray detectors frequently have slow or non-uniform lithium-ion drift rates. The presence of oxygen is one factor (Fox 1966, Adda et al. 1968) in the production of slow drift germanium and the presence of crystal lattice vacancies is possibly another (Lopes da Silva 1968, Williams 1969).

In the course of growing large ($\sim$ 1 kg) germanium crystals under vacuum by the Czochralski method in these laboratories, it was found that large radial variations occurred in both resistivity and lithium drift rate. It was suspected that these properties could be accounted for by the existence of a multi-ionized, fast diffusing acceptor impurity (probably copper) emitted from the heat shields which surrounded the crystal during the pulling process. This was confirmed when a simple gettering technique, for selected fast diffusants in germanium, following the principles described by Logan and Schwartz (1955), was successfully applied to samples of these crystals to extract the contaminant and restore normal driftability.

This paper gives results describing the effects of gettering on germanium driftability, resistivity, dislocation density and the performance of Ge(Li) γ-ray detectors made from gettered material.

2. GERMANIUM CRYSTAL PROPERTIES

A series of sixteen crystals were pulled in vacuo ($\sim 10^{-5}$ mm Hg) from a melt contained in a graphite crucible which was indirectly heated (50 Hz mains supply) by an annular furnace element also of graphite. The germanium was supplied by Sylvania Electric Co. and Metallurgie Hoboken.

The crystal orientation was $< 1.1.1 >$. No p-dopant was intentionally added to the melt, since it was found that all crystals grown showed p-type conductivity due to a shallow acceptor impurity. This impurity was probably boron, originating from the reduction of B_2O_3 in the base material by reaction with carbon in the crucible.

In all crystals the resistivity showed a marked radial variation, particularly at the seed end, being typically as much as 35 percent lower at the periphery than at the crystal axis where resistivity was usually >20 ohm-cm. Dislocation density ranged from $\sim$2000 cm^{-2} on small diameter ($\sim$ 3 cm) crystals up to $\sim$ 10,000 cm^{-2} on larger ($\sim$ 5 cm) crystals.

Samples taken from those crystals selected for absence of gross lineage all showed some non-uniformity in lithium driftability, the rate being less at the crystal periphery, as determined by the copper plating technique for exposing junctions on n^+-i-p diodes. Figure 1 shows copper electroplate patterns obtained on two Ge(Li) n^+-i-p diodes prepared from adjacent half-sections of the same crystal slice. The untreated sample shows non-uniform lithium drift while drift in the gettered sample is uniform.

3. THE GETTERING TECHNIQUE

Logan and Schwartz (1955) described experiments in which samples of germanium were doped with copper which was then gettered by a thin surface coating of lead or gold alloyed at 700°C. The distribution coefficient (ratio of concentration of solid to that of liquid) of copper in the ternary alloy system formed with lead and germanium is very small (6.3×10^{-6}) (Thurmond and Logan 1956), and therefore the copper concentration in the bulk germanium is reduced. The gettering action is aided by the rapid diffusion of copper in germanium ($D \approx 2.8 \times 10^{-5}$ cm^2 sec^{-1}) (Fuller et al. 1954). Gold and other metals such as indium have been observed to have even lower distribution coefficients

(more effective getters) (Thurmond and Logan 1956). It should be noted, however, that whereas complete restoration of resistivity after gettering was observed by Logan and Schwartz, lifetimes returned to only ~ 40 percent of the original values (gold as getter), possibly due to some plastic deformation of the gettered sample.

To estimate the time required for satisfactory completion of the gettering of our samples, the fraction of copper remaining at the centre of a semi-infinite slab of germanium (variable thickness) with gettering time t was calculated assuming the above value for diffusion constant of copper in germanium. The results were derived from a theoretical relation (Boltaks 1963) for surfaces which are true sinks rather than practical getters. For example Figure 2 shows that a gettering time of 14 hours should be sufficient to reduce the copper concentration at the centre of a slab 10 mm thick to 10^{-6} of its original value.

A gallium-indium eutectic alloy (16.5 at.% In; eutectic temp. = 15.7 °C; 99.999% pure) was chosen as gettering agent mainly because it is a liquid at ordinary temperatures and can be easily wiped or painted on to the sample surface.

The resistivity profiles of samples were first checked using a four-point probe on a surface lapped with No. 600 silicon carbide grit on glass. The dislocation density was then measured by etch pit count taken following a ten minute etch in a $4HNO_3$:1HF acid mixture at 40 °C. After this the samples were completely coated with Ga-In eutectic and mounted on an alumina platform located in a crucible, also of alumina, which had a loose-fitting lid. The alumina components had previously been acid cleaned, soaked in demineralised water and heated in air for several hours at ~ 1000 °C. The platform and the internal walls of the crucible were also coated with the eutectic. Gettering was carried out in an argon gas flow-silica tube furnace at ~ 800 °C for times typically ranging from 6 to 30 hours for samples of thickness 3 to 14 mm. Gettering temperature was reached in 5 hours and the furnace was cooled to room temperature at its natural time constant of ~ 10 hours.

After gettering, the samples were completely lapped free of any alloy. Because the samples used were of reasonably large volume, up to 10 cm^3, loss of germanium by alloying with gallium and indium was tolerable.

Next, at least a further 500 μm was removed from the surface by lapping. This operation is essential in order to eliminate the alloy sub-layer in which a very high density of dislocations is generated by alloy regrowth. The resistivity and etch pit count measurements were repeated as before.

4. EXPERIMENTAL RESULTS

Measurements of resistivity, dislocation density and lithium drift rates for untreated and gettered samples of germanium taken from three typical vacuum-grown crystals are given below. In addition, the results from a commercial gas-ambient grown crystal (Metallurgie Hoboken) are given for comparison.

The data are summarised in Tables 1 and 2.

4.1 Resistivity and Dislocation Density

Figures 3 and 4 show variations found in the radial resistivity and dislocation (etch pit) density profiles of two vacuum-grown germanium samples, before and after gettering. Uniformity of resistivity is noticeably improved. The lower resistivity of the peripheral regions of the ungettered samples indicates that the source of copper acceptor impurities was external to the crystal during

growth, indiffusion from the surface producing the bell-shaped profile. Untreated samples taken from the 'tail' ends of crystals showed less curvature in the resistivity characteristic, since these regions, being the last to grow, were held for less time at the high temperatures ($\gtrsim 700\,^{\circ}C$) necessary to promote diffusion.

To estimate the densities of both the copper and the residual, shallow-level acceptor impurities present in the material, measurements of conductivity variation with temperature (to $\sim 77\,^{\circ}K$) were made on several small bar samples of both untreated and gettered germanium. These were adjacent samples cut from the same crystal slice to ensure identical conductivities. A pulse technique with a low duty cycle to avoid ohmic heating effects was used to measure conductivity. From the conductivity versus temperature characteristics of the samples (Figure 5) an approximate determination can be made of the net acceptor impurity density from the temperature given at the intersection of the extrapolated intrinsic and extrinsic conductivity regions of the curve, provided the intrinsic carrier concentration and carrier mobilities at this temperature are known. Values of 4.4 and $1.0 \times 10^{13}\ cm^{-3}$ for the untreated and gettered samples were measured (Figure 5). The copper concentration in the germanium was therefore $3.4 \times 10^{13}\ cm^{-3}$, assuming that all the copper was extracted by gettering, that the donor impurity concentration in both samples was negligible, and also that only the lowest of the three possible states of copper (0.04 eV above the valence band) is ionized at the temperature at which the intrinsic and extrinsic contributions to conductivity are equal in these samples.

The radial dislocation density profiles given for two sections in Figures 3 and 4, before and after gettering, demonstrate that no serious crystallographic deformation occurs during the gettering cycle. Although the profile of the sample in Figure 4 indicates that the dislocation density had possibly been lowered in the central regions of the slice, the overall average dislocation density for both samples remained unaltered by gettering.

4.2 Lithium-Ion Drift

The Ge(Li) n^{+}-p diodes were prepared using standard techniques. The lithium diffused contacts were formed by vacuum evaporating lithium onto one face of a slice of germanium heated to $375\,^{\circ}C$. After a ten minute diffusion the samples were cooled to room temperature in ten minutes by a flow of nitrogen gas. Lithium-ion drift rates were measured by clamping the Ge(Li) diodes between two hollow copper plates temperature stabilised by an oil flow from a controlled temperature bath. Temperature stability was $\pm 0.2\,^{\circ}C$ during runs of up to 1000 minutes and was monitored by a copper-constantan thermocouple. The diodes were drifted in a nitrogen gas atmosphere. Diode capacitance was monitored by a modified Q-meter operating at 3.5 MHz (± 1 pF accuracy). With the exception of one sample, drift temperatures close to the boiling point of Freon TMC (b.p. $36.5\,^{\circ}C$) were chosen, since this coolant is used in these laboratories in the drift stage of Ge(Li) detector preparation.

When drift time t is long, the diode depletion layer W obeys the relation:

$$W = (2\mu Vt)^{\frac{1}{2}}, \qquad (1)$$

where μ is the lithium ion mobility and V is the applied bias (Pell 1960); thus the diode capacitance C_d will vary as $t^{-\frac{1}{2}}$. It is therefore possible to measure μ by experiment and, further, using the Einstein relation:

$$\mu = \frac{De}{kT} \quad , \tag{2}$$

to calculate the diffusion constant for lithium in germanium.

Figure 6 shows the variation in diode capacitance with drift time for two Ge(Li) diodes made from gettered, vacuum-grown germanium. The region in which the relation in Equation 1 holds is not entered until after several hours drifting. This is because initially the drift field is not constant across the diode junction, a necessary condition in the derivation of the relation. Sher (1969) reported similar observations made in a series of carefully controlled measurements of lithium drift-rate in germanium. For reference, Figure 6 also shows diode reverse leakage currents as a function of drift time.

The variation in capacitance with drift time from two Ge(Li) diodes of the same physical dimensions, one prepared from untreated and the other from gettered vacuum-grown germanium, is shown in Figure 7. The smaller C_d values from the diode made from gettered germanium indicate an improvement in lithium drift rate by a factor of 2.5. From a comparison of the reverse leakage currents in these two diodes, assuming that the currents were contact diffusion currents only, it is estimated that the minority carrier lifetime in the gettered germanium was reduced (at 37.3°C, the drift temperature) to 55 percent of that in the original ungettered material.

To compare the effects of gettering on drift rate in germanium known to have been grown in a gaseous atmosphere, two samples were selected from a crystal supplied by Metallurgie Hoboken and classed by the suppliers as normal drifting material. The diode capacitance and leakage current versus drift time characteristics are given in Figure 8. The lithium drift rates for the two samples are identical within experimental error. However, in this instance the leakage current of the gettered sample is less than that of the untreated sample by a factor of 7.8 indicating a 60-fold improvement in minority carrier lifetime by gettering (at 37.2°C).

Generally, leakage currents of Ge(Li) diodes from gettered, vacuum-grown germanium were not very different from those of other devices made in these laboratories from Hoboken crystals. For example, two diodes from our gettered material (different crystals) with depletion layers 6 and 9 mm (areas 2.8 and 3.5 cm^2 respectively) both gave saturated current densities of 1.7 mA cm^{-2} (~22°C) after re-diffusion of the lithium contact and 'cleanup'.

Lithium ion drift mobilities were calculated from the linear, $C_d \propto t^{-\frac{1}{2}}$, regions of the experimental curves of Figures 6, 7 and 8 using Equation 1. The dielectric constant of germanium was taken as 16 for determination of drift depths from diode capacitance. The corresponding lithium diffusion constants are plotted in Figure 9 with experimental curves from Fuller and Severiens (1954) and Sher (1969) for comparison. They agree well with the results obtained by Sher.

4.3 Gamma-Ray Detectors

Following an initial shallow drift under controlled conditions for lithium-ion drift mobility measurement, the Ge(Li) diodes were removed, mounted on water-cooled plates and the drift completed at bias voltages up to 1 kV (current limited). The lithium contact was removed by lapping and renewed as before, and the diodes were 'cleaned-up' for 15 hours at approximately room temperature. After a final etch they were further 'cleaned-up' (1–2 hours) in the test cryostat at a temperature which permitted the application of a bias field of 100 V mm^{-1} at a current density of ~1 mA cm^{-2}. The performance of the diodes as γ-ray detectors was checked using an uncooled FET preamplifier.

The spectral responses to ^{137}Cs source γ-rays from two Ge(Li) detectors, one constructed from untreated and the other from gettered germanium of the same crystal, are shown respectively in Figures 10 and 11. The detector made from gettered material (W = 2.5 mm) shows good resolution (2.5 keV FWHM) and no obvious spectral peak tailing due to trapping, in contrast with the performance of the detector made from untreated germanium (3.4 keV FWHM) where there was some tailing. However, since the untreated sample has a variable depletion layer (W = 2.5 – 4.5 mm) due to non-uniform lithium drift, a direct comparison of relative trapping effects in the two devices is not possible.

The spectral response with bias up to 1 kV from a deep depletion layer (W = 9 mm) Ge(Li) detector from gettered material is shown in Figure 12 and some trapping in this detector is evident. Detectors constructed from gettered germanium totalled five, of which four were operated as useful spectrometers.

5. DISCUSSION

The restoration of normal lithium-ion drift in the vacuum grown crystals produced in these laboratories by gettering has confirmed the presence of a fast diffusing acceptor impurity. The impurity is most likely copper but the possibility that other similar fast diffusants such as nickel and iron may be present cannot be definitely excluded. The reduction of lithium drift rate in the untreated crystals is due to the formation of lithium-ion complexes with the impurity (or impurities) which can exist in multiple ionized acceptor states (copper has three levels). Firm identification of the impurity would require Hall measurements of ionization levels or a determination, similar to that described by Armantrout (1969), of the infra-red response of Ge(Li) detectors made from the material.

The contamination experienced at these laboratories is not uncommon in germanium pulled in vacuo since impurity pickup by emission from hot reflectors and crucibles is difficult to avoid, unlike the situation in gaseous filled systems where the collision mean free path of the impurities is very small. However, it is considered that by correct choice of pure reflector materials and elimination of copper base alloys it should be possible to reduce the contamination.

A small degree of copper-like contamination in germanium for γ-ray detectors can evidently be permitted, as seen from the spectral response of the Ge(Li) detectors from non-gettered material (Figure 10). Indeed, a 30 cm^3 coaxial Ge(Li) detector with good resolution has also been made from similar germanium (unlike the situation in planar detectors, radial drift non-uniformity is unimportant in the coaxial structure). The probable reason is that ion-pairing, by screening, reduces the coulombic attraction of carriers by the impurity trap. Reduction of the trapping cross-section for Ni in germanium by lithium ion pairing has been confirmed by Armantrout (1969).

While it should be possible to measure the effects of gettering on the density of minority carrier recombination centres by measuring relative changes in Ge(Li) diode leakage currents under reverse bias as described above, too much reliance should not be placed on the accuracy of these results because unknown, variable surface currents also contribute to the total current measured. However, because leakage currents in diodes from our gettered, vacuum grown material were similar to those from most Hoboken crystals, the general observation can be made that no serious reduction in lifetime occurs.

The very large improvement in carrier lifetime seen in the diode from gettered, gas ambient grown crystal is of special interest. Since this crystal contained little or no copper-like impurity (normal lithium drift rate before and after gettering), the recombination centres may well be lithium-

vacancy pairs known to form in pulled crystals (1969). The gettered sample of this crystal was not examined for trapping as a γ-ray detector but detectors from the untreated material do show serious trapping. Again, because the dislocation density of this crystal is low, a high vacancy content is expected, since dislocations act as sinks for vacancies during crystal growth (Hansen et al. 1969). It may well be that the effect of the heat cycle during gettering has been to promote the diffusion to, and annihilation of, vacancies at the surface of the sample with subsequent improvement in carrier lifetime. If this proposition is correct, then it may be possible to reduce trapping effects usually seen in detectors made from dislocation-free germanium. Dispersion of vacancy clusters by first diffusing copper into the germanium, followed by a gettering cycle, as described by Tweet (1959) may be more effective than a simple getter cycle. It has already been demonstrated in these laboratories that germanium which has been saturated with copper at 850 °C shows normal lithium drift properties after gettering.

Although the gettering method described is not suitable for routine extraction of copper-like impurities in germanium for γ-ray detectors, it is nevertheless a useful diagnostic technique for determining whether the contaminant is of that class. The gettering process may also be applicable to the extraction of copper remanent in hyper-pure germanium grown for deep depletion layer junction detectors.

6. ACKNOWLEDGMENTS

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TABLE 1

RESISTIVITY AND DISLOCATION PROPERTIES OF UNTREATED AND GETTERED GERMANIUM

Sample	Before Gettering		After Gettering	
	Resistivity (ohm-cm)	Dislocation Density (cm^{-2})	Resistivity (ohm-cm)	Dislocation Density (cm^{-2})
10–4A	47.8 (20.5° C)	2720 (± 70)	46.0 (23.2° C)	2720 (± 70)
10–4B2	42.5 (20.8° C)	–	45.3 (23.3° C)	–
10–4C2	42.5 (20.8° C)	–	–	–
11–3	37.5 (22.2° C)	2175 (± 60)	40.5 (24.5° C)	2820 (± 70)
13–6B3–B	42.6 (26.3° C)	7080 (± 90)	48.6 (24.7° C)	9220 (± 120)
Q – 1A	15.2 (21.0° C)	79 (± 13)	–	–
Q – 1B	14.9 (21.0° C)	103 (± 15)	15.7 (24.5° C)	133 (± 20)

Note: The first number or letter identifies the crystal from which the sample was taken. Crystals 10, 11 and 13 are vacuum-grown. Crystal Q is a gas ambient grown crystal.

Standard errors are given for the dislocation density (averaged over the sample surface).

TABLE 2

LITHIUM DRIFT PROPERTIES OF UNTREATED AND GETTERED GERMANIUM

Sample	Treatment	Drift Bias (V)	Drift Temp. (°C)	Lithium Ion Mobility ($cm^2\ V^{-1}\ sec^{-1}$)	Lithium Diffusion Constant ($cm^2\ sec^{-1}$)
10 – 4A	Gettered	100	30.4	$3.7\ (\pm 0.4) \times 10^{-10}$	$9.5\ (\pm 1.1) \times 10^{-12}$
10 – 4B2	Gettered	200	37.4	$6.4\ (\pm 0.9) \times 10^{-10}$	$17.2\ (\pm 2.3) \times 10^{-12}$
10 – 4C2	Untreated	200	37.4	$2.4\ (\pm 0.3) \times 10^{-10}$	$6.5\ (\pm 0.9) \times 10^{-12}$
11 – 3	Gettered	100	37.5	$5.8\ (\pm 0.7) \times 10^{-10}$	$15.7\ (\pm 1.8) \times 10^{-12}$
13 – 6B3–B	Gettered	200	37.7	$7.8\ (\pm 0.7) \times 10^{-10}$	$21.0\ (\pm 1.9) \times 10^{-12}$
Q – 1A	Untreated	100	37.2	$6.3\ (\pm 0.6) \times 10^{-10}$	$16.7\ (\pm 1.7) \times 10^{-12}$
Q – 1B	Gettered	100	37.2	$6.7\ (\pm 0.6) \times 10^{-10}$	$18.1\ (\pm 1.5) \times 10^{-12}$

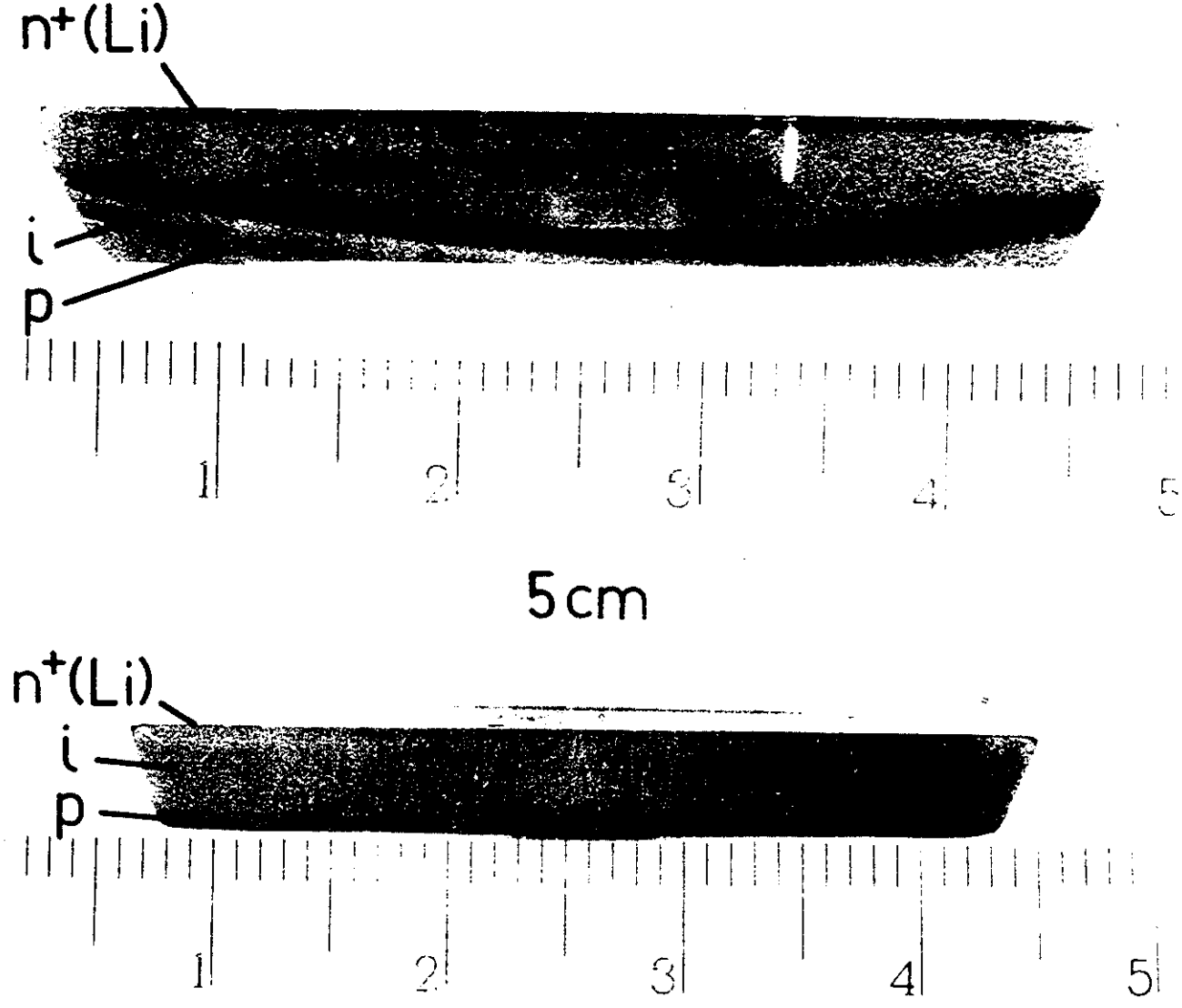

FIGURE 1. COPPER ELECTROPLATE PATTERNS SHOWING THE DRIFT PROFILES IN TWO Ge(Li) n⁺-i-p DIODES (ADJACENT HALF-SECTIONS OF A CIRCULAR SLICE) FROM A VACUUM-GROWN CRYSTAL CONTAMINATED DURING GROWTH

The untreated sample (upper) shows non-uniformity in drift while the gettered sample (lower) is uniform

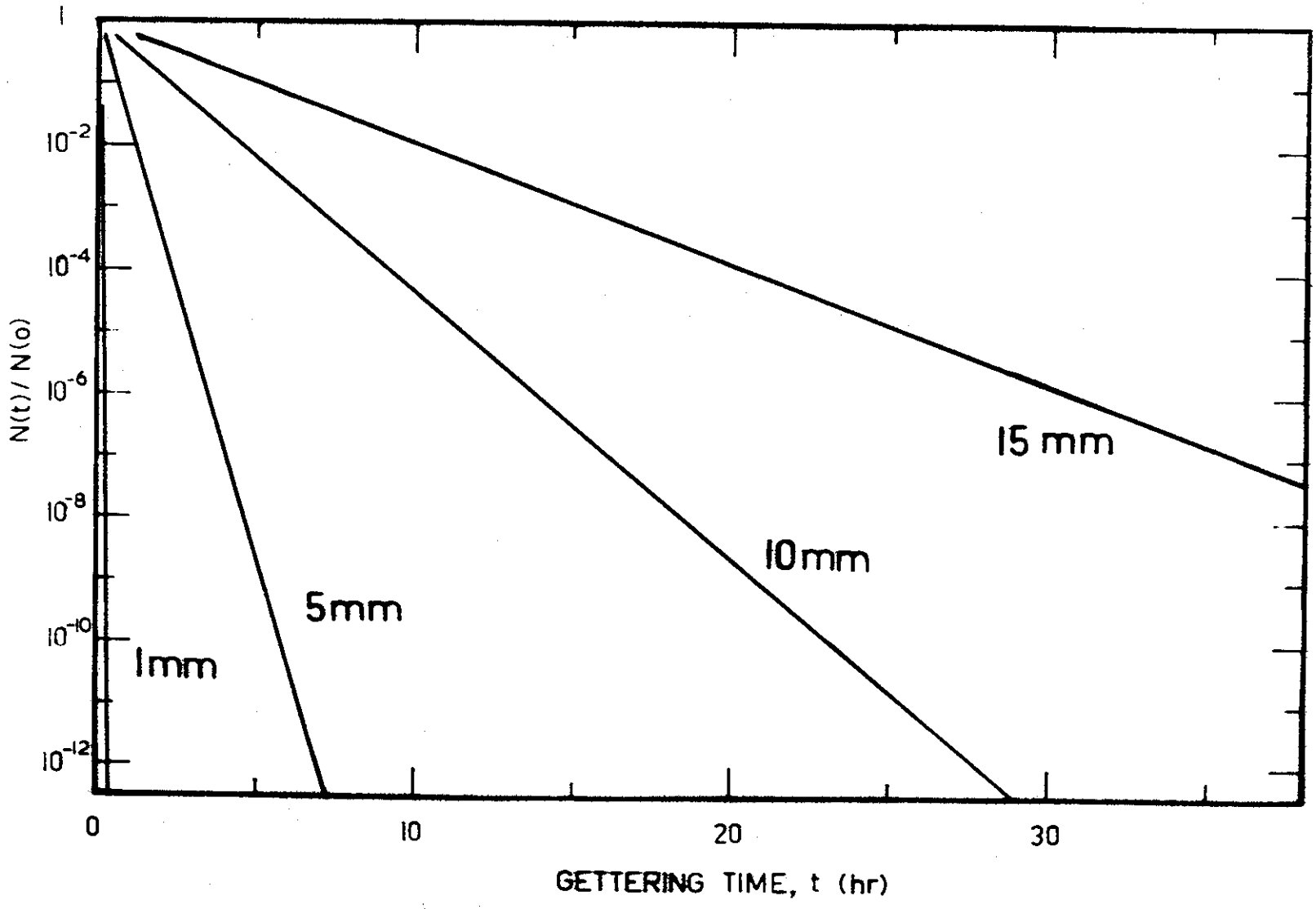

FIGURE 2. THE FRACTION OF COPPER REMAINING, N(t)/N(o), AT THE CENTRE OF A SEMI-INFINITE SLAB OF GERMANIUM OF VARYING THICKNESS WITH GETTERING TIME t

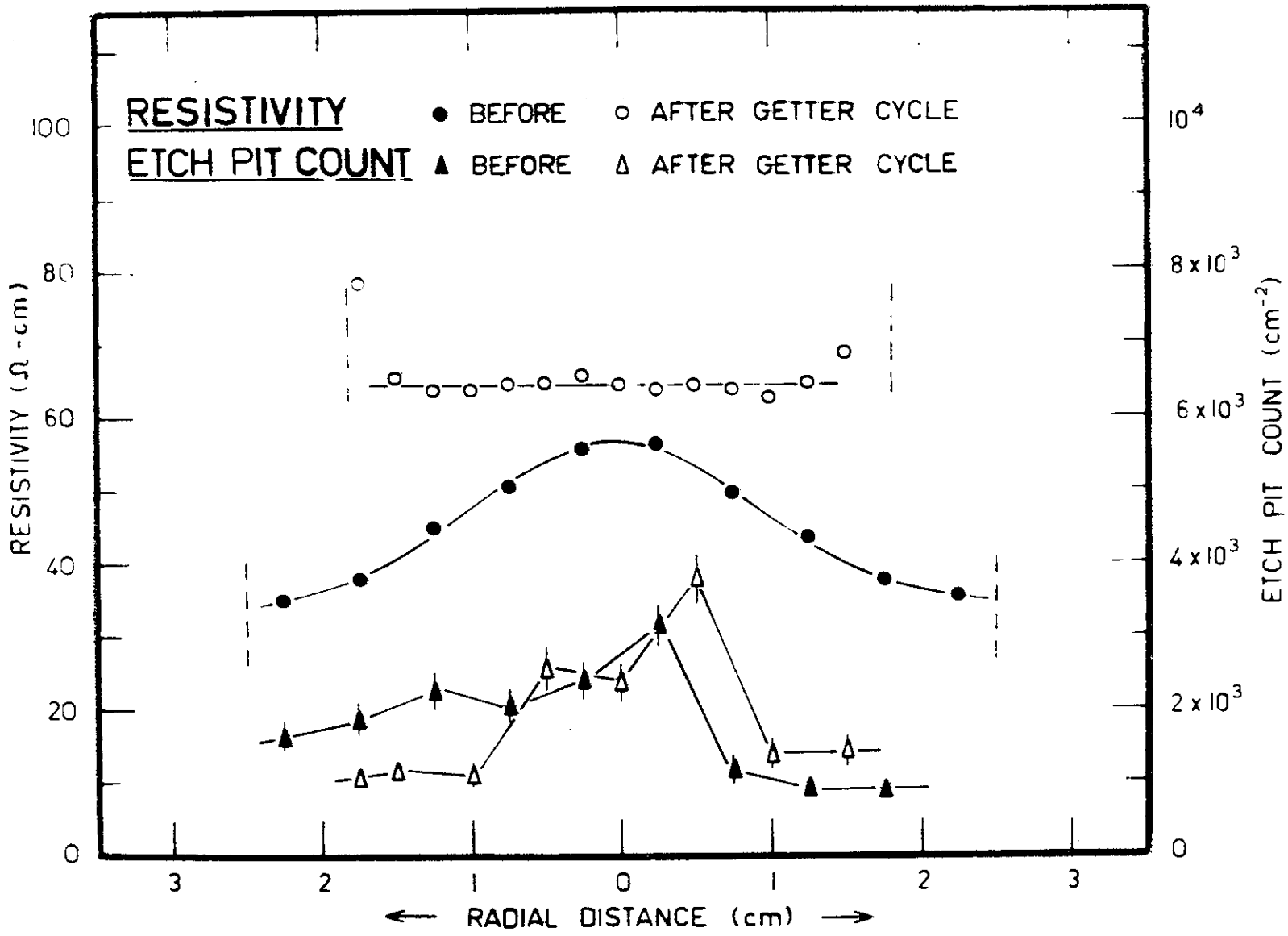

FIGURE 3. RADIAL VARIATIONS IN RESISTIVITY AND DISLOCATION (ETCH PIT COUNT) DENSITY FROM SAMPLE 13-4B

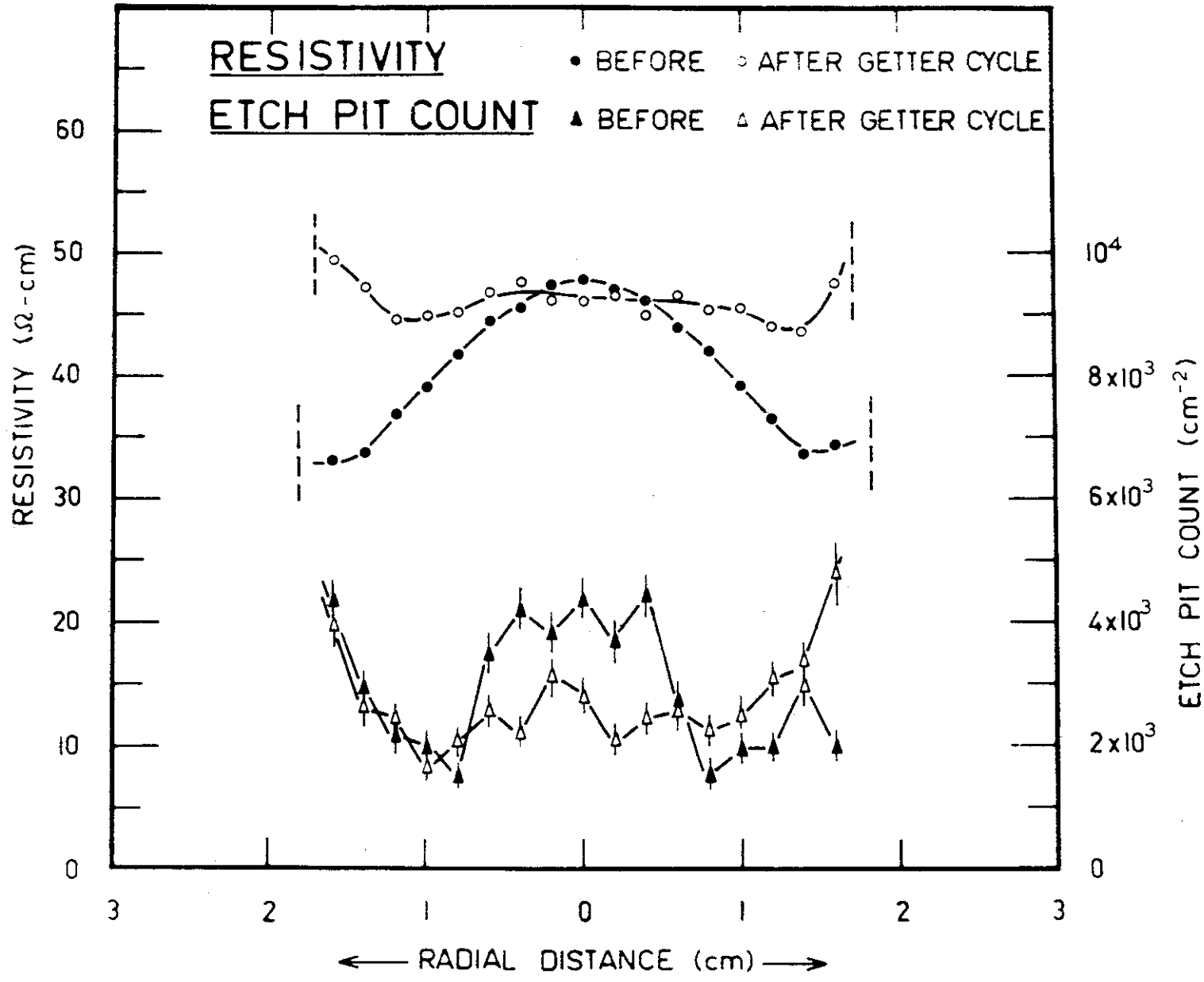

FIGURE 4. RADIAL VARIATIONS IN RESISTIVITY AND DISLOCATION (ETCH PIT COUNT) DENSITY FROM SAMPLE 10-4A

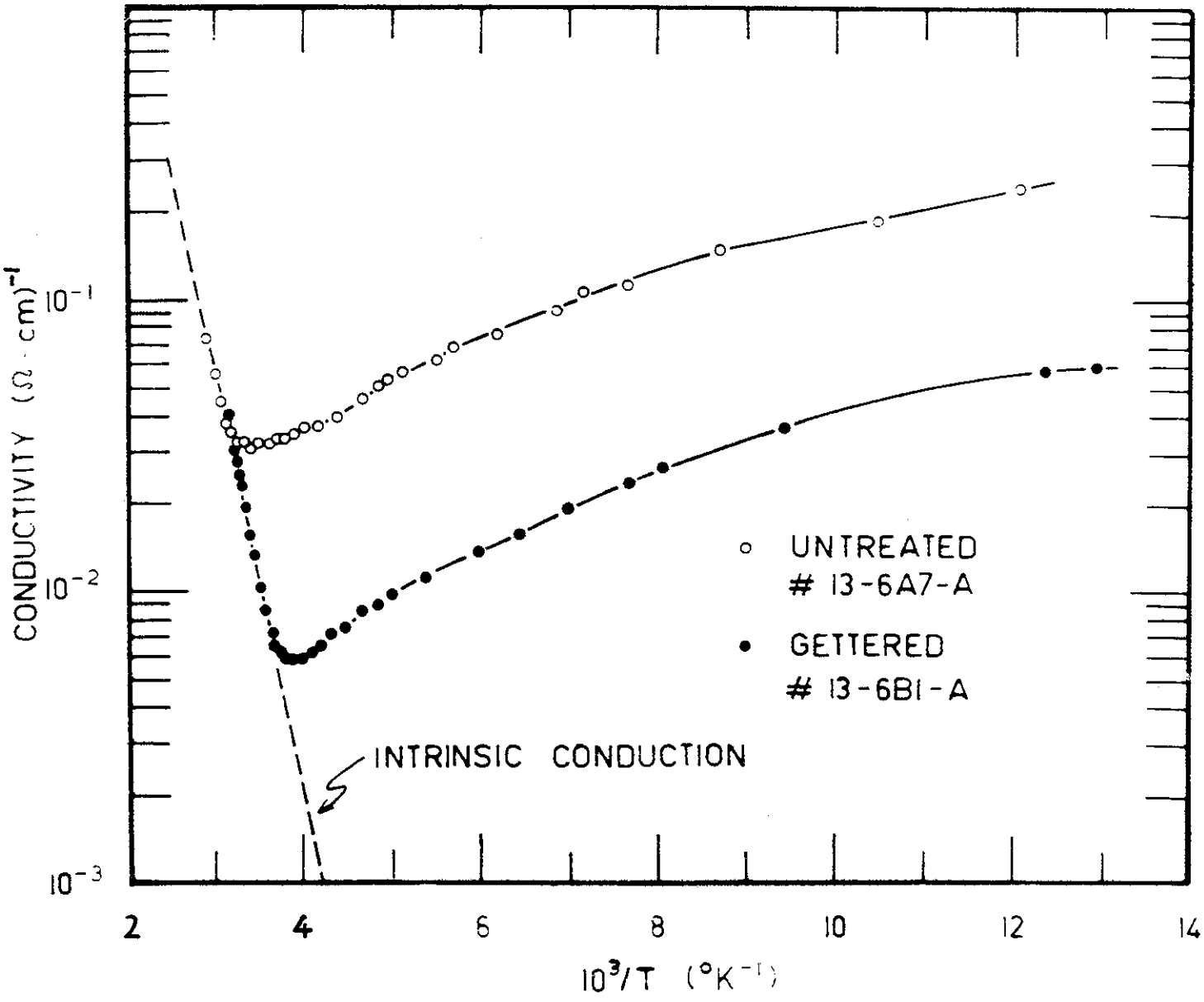

FIGURE 5. TEMPERATURE VARIATION OF CONDUCTIVITY IN UNTREATED AND GETTERED SAMPLES OF VACUUM-GROWN GERMANIUM

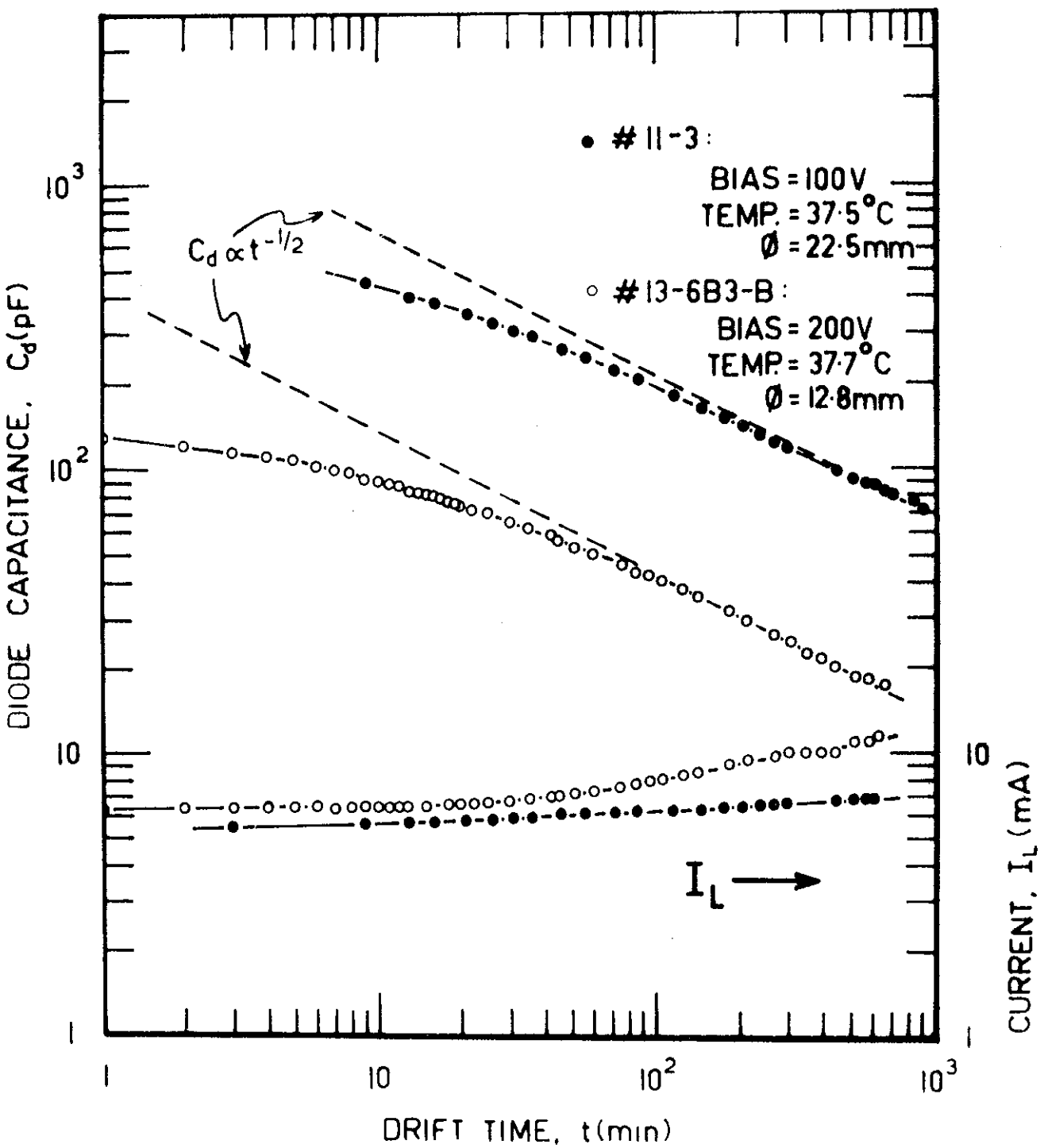

FIGURE 6. VARIATION IN DIODE CAPACITANCE AND LEAKAGE CURRENT WITH DRIFT TIME IN TWO Ge(Li) DIODES PREPARED FROM GETTERED VACUUM-GROWN GERMANIUM

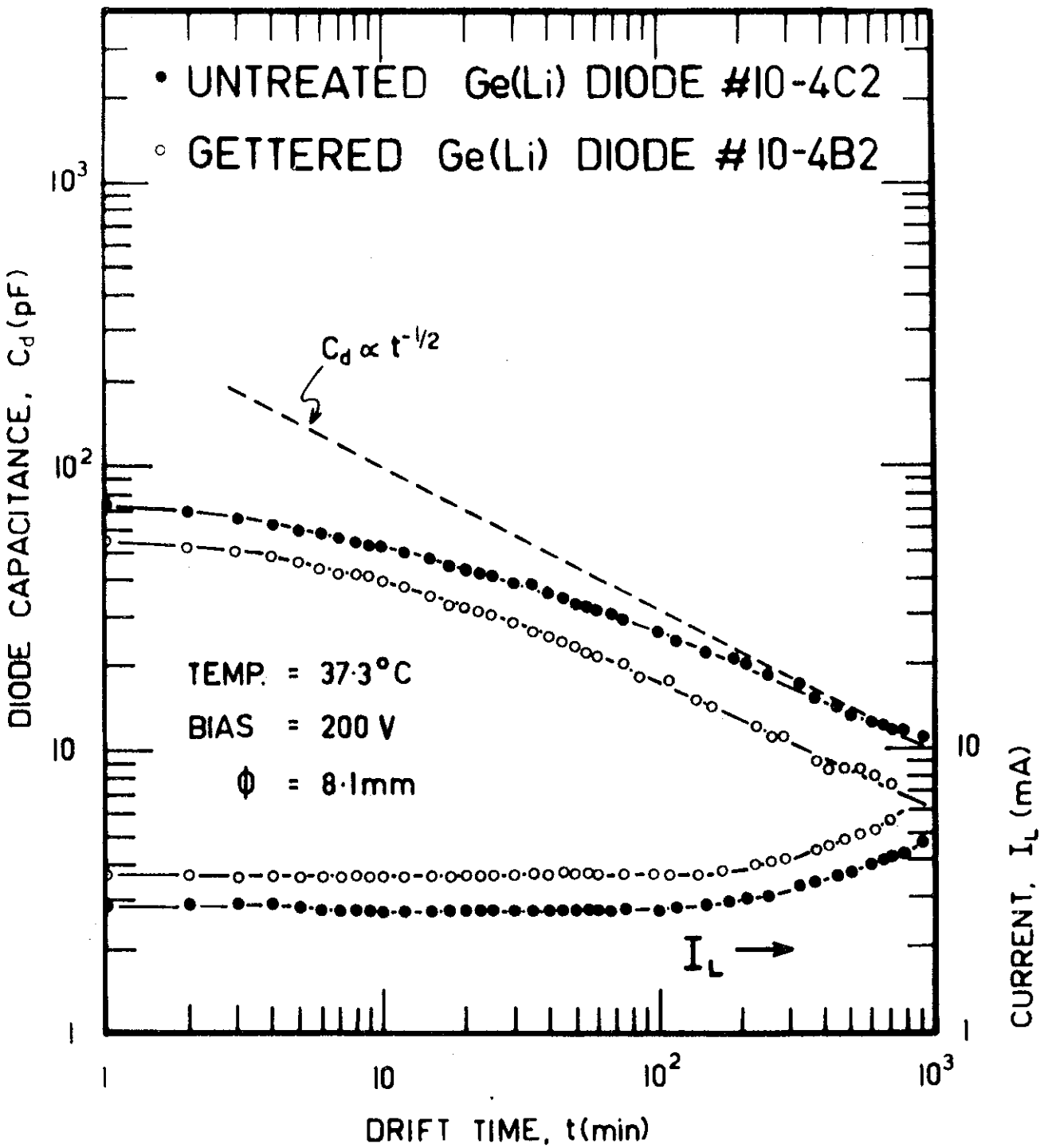

FIGURE 7. VARIATION IN DIODE CAPACITANCE AND LEAKAGE CURRENT WITH DRIFT TIME IN TWO Ge(Li) DIODES (10-4C2 AND 10-4B2)

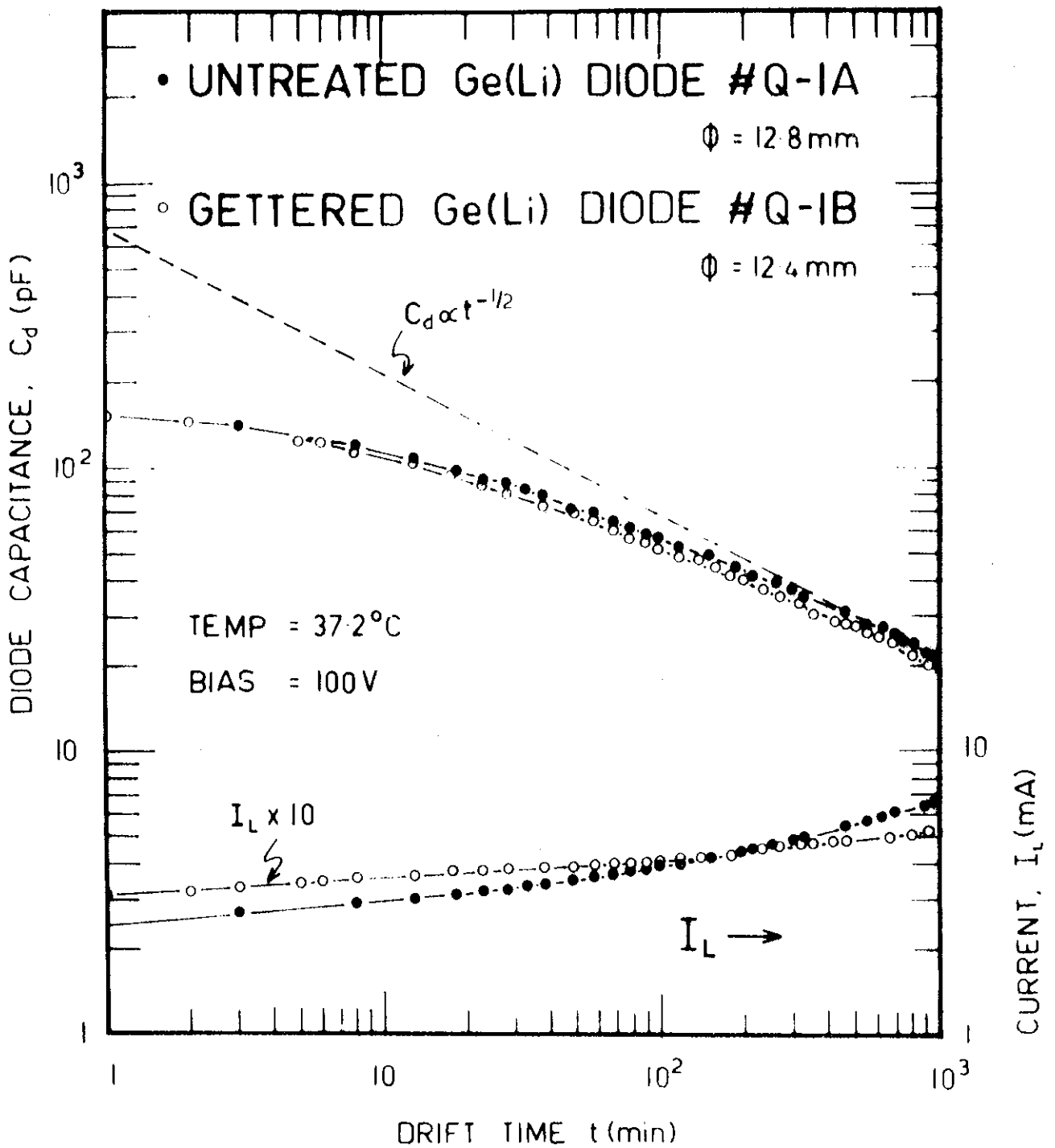

FIGURE 8. VARIATION IN DIODE CAPACITANCE AND LEAKAGE CURRENT WITH DRIFT TIME IN TWO Ge(Li) DIODES (Q-1A AND Q-1B)

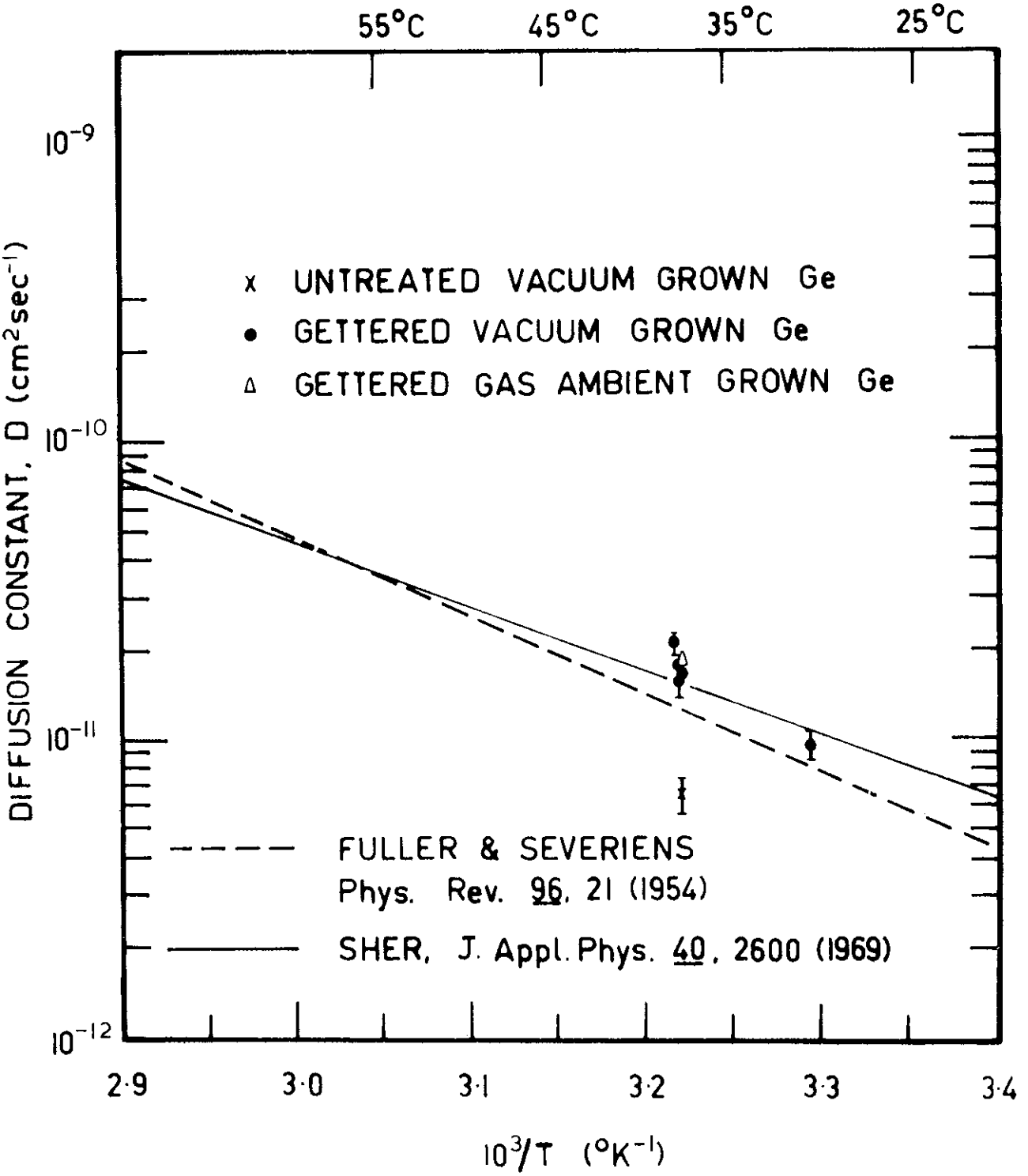

FIGURE 9. EXPERIMENTAL VALUES OF THE DIFFUSION CONSTANT FOR LITHIUM FROM ION-DRIFT MOBILITY IN UNTREATED AND GETTERED GERMANIUM

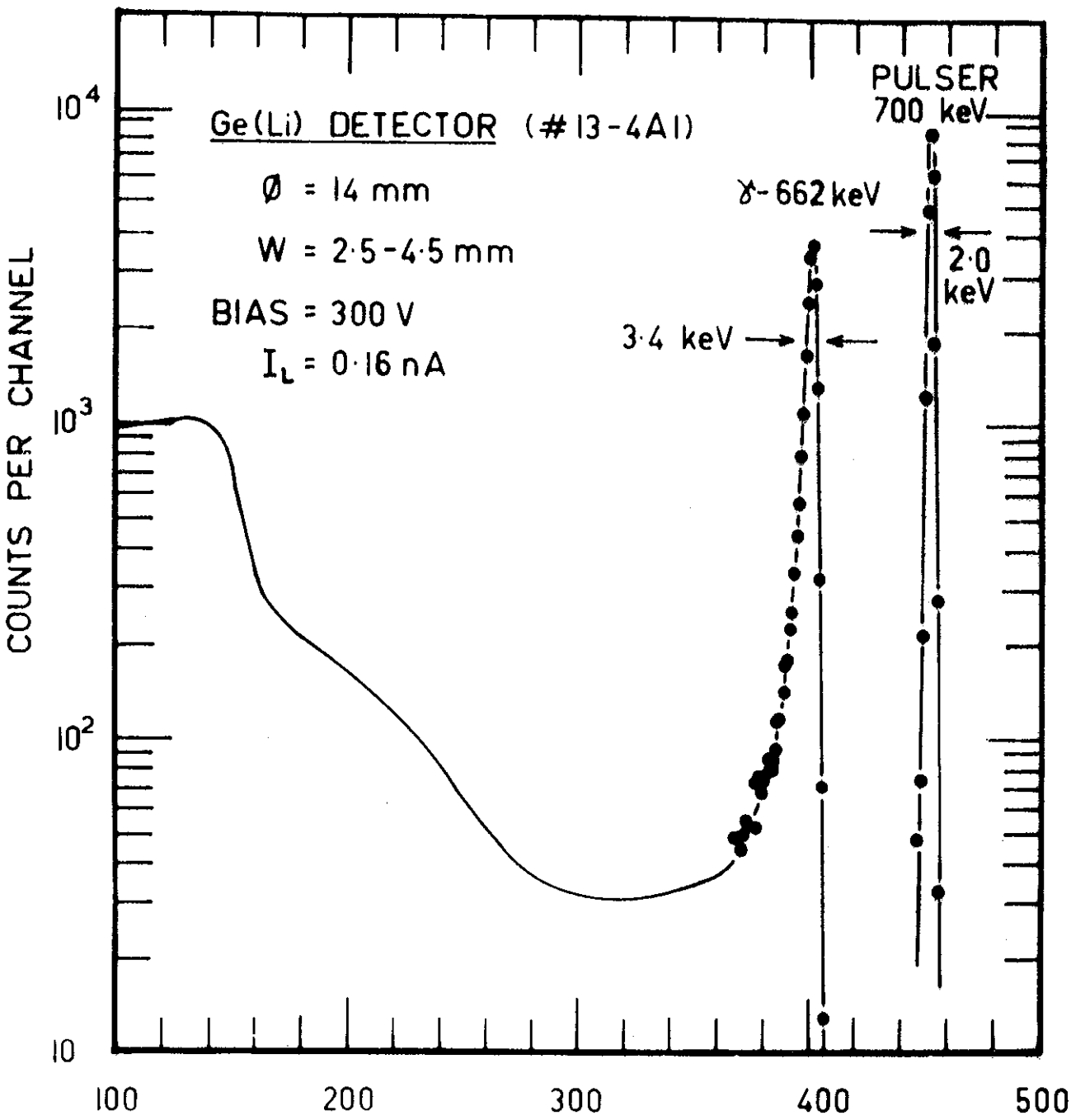

FIGURE 10. RESPONSE TO ^{137}Cs SOURCE δ-RAYS OF A Ge(Li) DETECTOR PREPARED FROM UNTREATED VACUUM-GROWN GERMANIUM

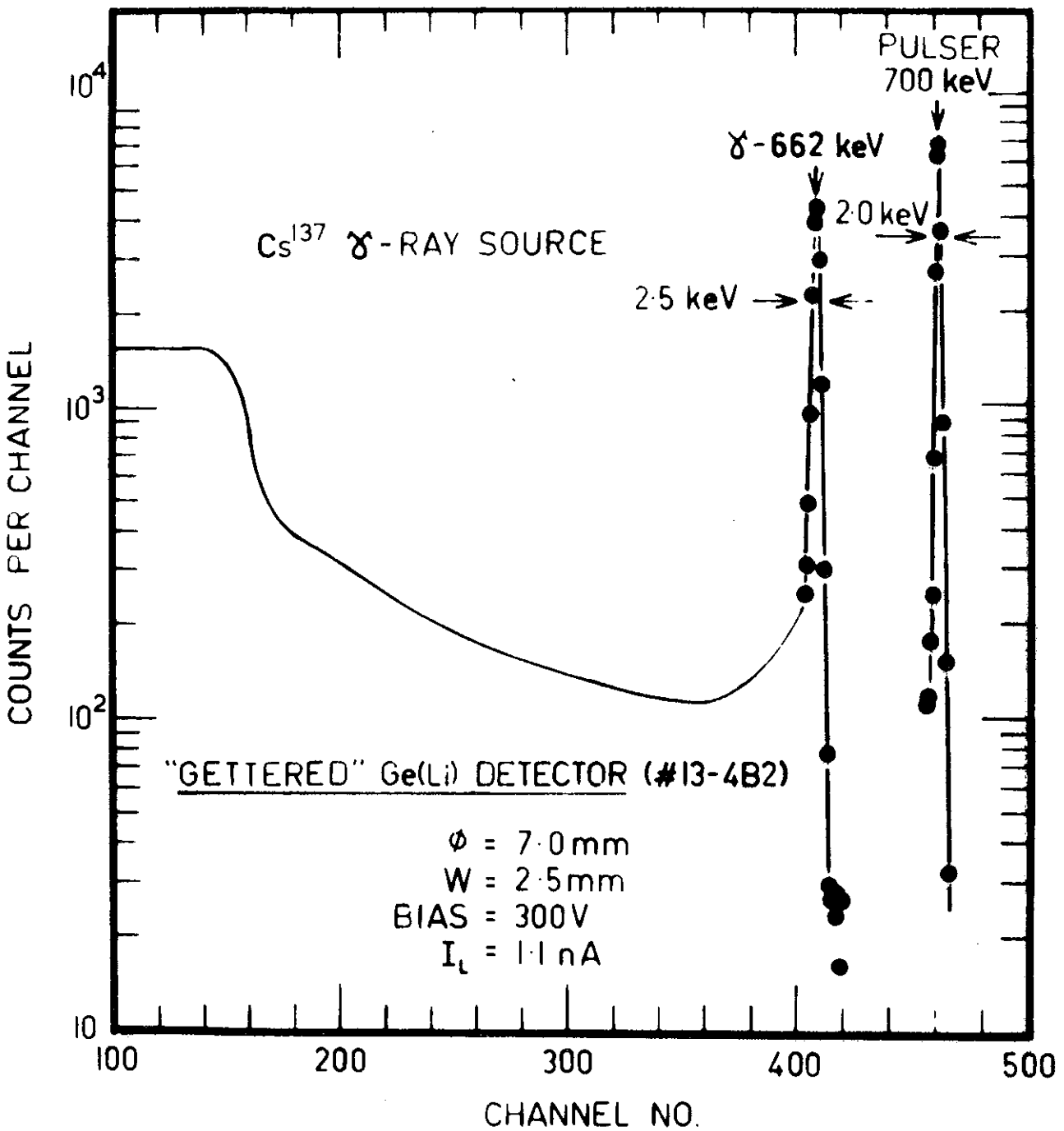

FIGURE 11. RESPONSE TO ^{137}Cs SOURCE γ-RAYS OF A Ge(Li) DETECTOR PREPARED FROM GETTERED VACUUM-GROWN GERMANIUM

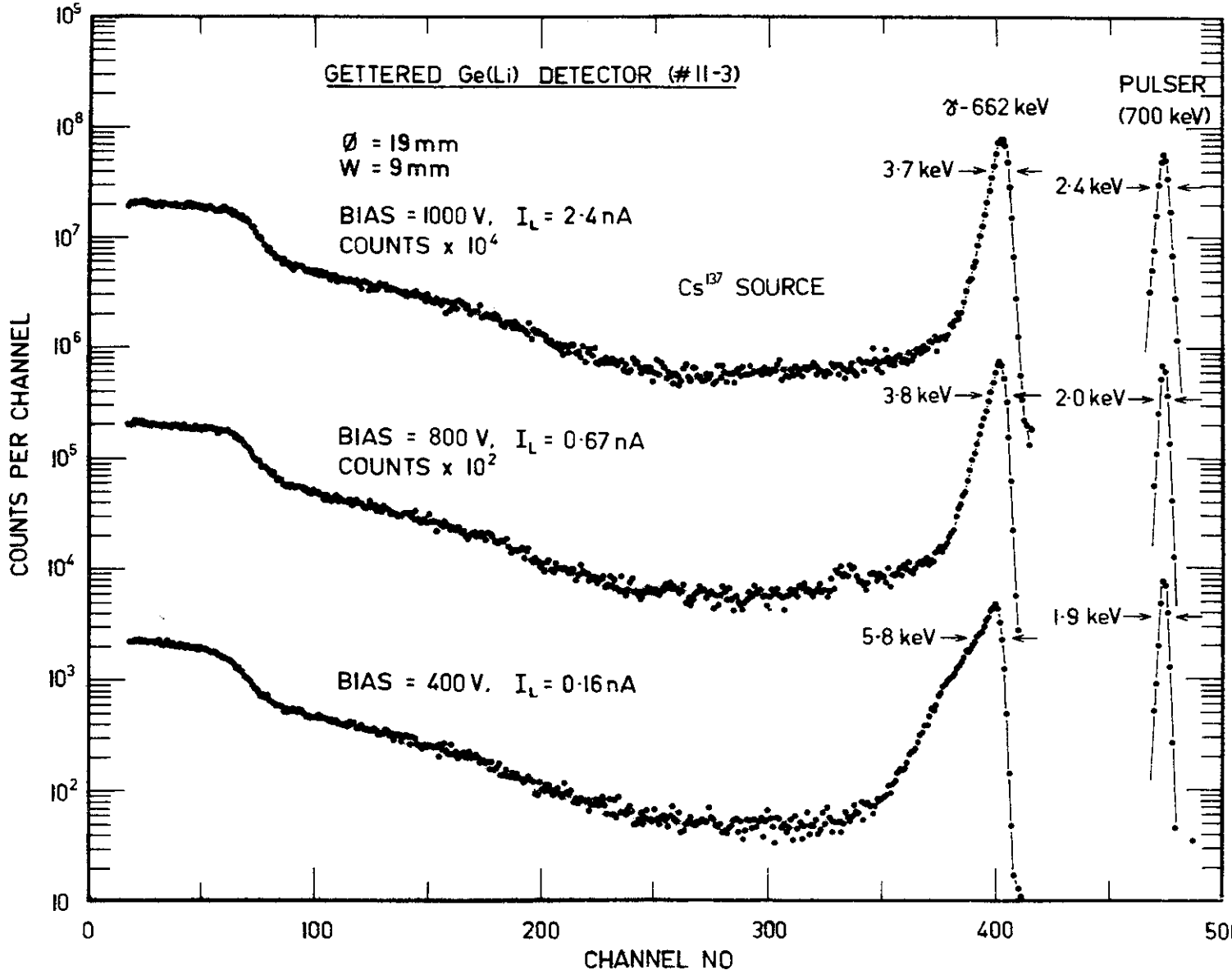

FIGURE 12. RESPONSE TO ^{137}Cs SOURCE γ-RAYS (WITH BIAS UP TO 1 kV) OF A Ge(Li) DETECTOR PREPARED FROM GETTERED VACUUM-GROWN GERMANIUM