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CR-113558

NCL 70-51R

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STUDY OF DOPANTS FOR RADIATION-RESISTANT SILICON

by

R.F. Bass, O.L. Curtis, Jr., and J.R. Srour

FINAL REPORT

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Contract No. 952523

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This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, as sponsored by the National Aeronautics and Space Administration under Contract NAS 7-100

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ABSTRACT

This report describes the results of investigations of the effects of gamma irradiation on the electrical properties (primarily carrier lifetime) of both Al-and Li-doped silicon. The results of initial studies of the radiation sensitivity of Li-doped material were rendered uncertain due to an inhomogeneous distribution of Li throughout the bulk of the silicon samples employed. Subsequently, a procedure was developed involving a diffusion and distribution cycle employing considerably longer times and higher temperatures than previously used. Use of this technique resulted in the preparation of a considerably more homogeneous Li-doped sample. Studies of Al-doped material demonstrate that Czochralski-grown Al-doped Si that undergoes an appreciable resistivity change upon heat treatment ($\lesssim$ a factor of two) apparently is significantly less sensitive to gamma irradiation than comparable B-doped crucible-grown material. It is felt that these findings constitute an important breakthrough that may have significant impact on future solar cell technology.

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1. INTRODUCTION

This report describes the results of investigations of the effects of radiation on the electrical properties (primarily carrier lifetime) of bulk silicon which has been doped with aluminum or lithium. Although considerable practical interest is centered around radiation effects in solar cells, it is important that the processes associated with radiation-induced defects be understood. Studies of bulk material are the logical way to study at least part of these processes, and provide a means for separating the effects that are intrinsic to silicon from those that are associated with the presence of a junction. For example, in the case of a mobile impurity such as lithium, the impurity distribution near the junction may be extremely nonhomogeneous. It is unlikely that the behavior with such a distribution will be understood until an adequate understanding of the homogeneous case is obtained.

Interest in lithium-doped material is extensive, and a great deal of data have been accumulated on the radiation vulnerability of lithium-doped solar cells.[1-5] Great promise has been shown for the potential use of these devices in a space radiation environment. However, the obvious dependence of the observed behavior on the material from which the solar cells have been fabricated, and apparent inconsistencies among cells thought to be identical, point to the necessity of a better understanding of the basic processes of defect creation and subsequent annihilation.

Another material of great interest, and the one upon which the most effort has been currently expended is Al-doped Si. In early studies of the effects of dopant impurities on neutron degradation of Si,[6-8] it appeared possible that Al-doped material was substantially more radiation-resistant than material doped with boron or gallium. Subsequent studies[9] appeared to support this result. Further interest in this phenomenon was promoted by personal communication with various members of the technical community who expressed the belief that they, too, had seen specific instances in which Al-doped material seemed particularly invulnerable to radiation. On the other hand, there was concern that these results were not reproducible. We were particularly concerned about the validity of our data due to the poor quality of the materials we had used. At the time the experiments were performed, the only Al-doped crystals available were grown by the Czochralski technique. These materials contained obviously large concentrations of both trapping and recombination centers. Because of the poor quality of the starting material, we found it necessary to anneal the samples before performing experiments. In the process, we observed something that had been discovered earlier,[10,11] that the equilibrium carrier concentration changed after annealing in the region of 400°C and above. This did not particularly concern us, and it gave a means of obtaining a range of resistivities from a single ingot.

To refine the experiment, some high-quality float zone material was obtained later. Since the lifetime was long and trapping effects small, the

difficulties of earlier experiments were avoided. However, the experimental results were disappointing. The Al-doped material was just as sensitive to radiation as was B-doped silicon. The information to be presented here concerning Al-doped materials deals primarily with an effort to reconcile the experimental data and learn under what conditions Al-doped material is relatively invulnerable to radiation.

The type of space radiation most important to the users of solar cells is usually electrons with energies on the order of 1 MeV. For the purposes of this study we chose instead to use Co^{60} gamma radiation. Such radiation introduces damage homogeneously into large bulk samples so that the effects of nonhomogeneous damage distributions are avoided. The physical nature of the defects introduced must be very similar to that obtained with electron irradiation since the damage mechanism occurs through the interaction of Compton electrons produced by the gamma rays with the silicon lattice. Those electrons having energies below the threshold for displacement are unimportant, and the remaining part of the spectrum is reasonably similar to that of interest. In addition, the relatively low dose rate produced by the gamma source ($\sim 10^5$ R/h) more nearly approximates a space radiation environment than that provided by usual electron accelerators. A further advantage of the gamma source is that it permits a large number of samples to be irradiated simultaneously and thus insures that any observed impurity effects are not influenced by dosimetry measurements.

2. EXPERIMENTAL TECHNIQUES

2.1 Preparation of Lifetime Samples

All the samples employed in these studies were prepared from single crystal ingots which were purchased from commercial suppliers. However, Li-doped samples were prepared in our laboratory by diffusing lithium into sample blanks cut from purchased crystals. The techniques used to produce these samples are described in Section 2.4.

To insure that the diffusion lengths were small compared to the minimum dimension of the samples, relatively large samples were employed in the investigations. This precaution minimized surface effects and thus insured that bulk lifetimes were being observed. Most of the samples were approximately 7mm thick, 7mm wide and 30mm long, but some samples approximately one-half as thick were also used.

The ends of the samples were ultrasonically tinned with solder to provide pressure contacts with the sample holders used for the various electrical measurements. The quality of the contacts was determined by measuring the forward and reverse resistances of each sample at both room temperature and at dry ice temperature. If no rectification was observed at low temperature, a potential profile was obtained for the sample in our initial investigations to determine the resistivity, linear homogeneity, and the

contact resistances. In later studies, conventional four-point probe techniques were used to measure resistivities (especially for Al-doped samples).

2.2 Lifetime Measurements

Carrier lifetimes were measured using the photoconductivity decay technique following injection of excess carriers by a 50 ns pulse of either 100 or 150 kV x-rays. Measurements were performed as a function of temperature in the temperature range from ~208 to ~526 $^{\circ}$K for samples having initial room temperature lifetimes longer than ~15 µs. Samples having shorter lifetimes were measured at 30 $^{\circ}$C only, in most cases. Details of both the technique and the experimental apparatus used for the measurements have been described previously[12,13]. In order to obtain exponential photoconductivity decays it was usually necessary to perform the measurements at carrier injection levels of $\sim 10^{-4}$ or less.

2.3 Sample Irradiation

Samples were irradiated in the Northrop Co^{60} gamma source, which at the time of the experiments, produced a dose rate of 1.36×10^{5} R/h at the sample positions. In a typical experiment, as many as 22 samples were irradiated simultaneously with a fluence of 1.1×10^{16} gammas/cm^{2}. To minimize annealing during the relatively long exposure period required to obtain this dose (~50 hours), the irradiations were performed with the samples at dry

ice temperature (-78°C). The samples were allowed to warm to room temperature for post-irradiation lifetime measurements but were then returned to dry ice storage in the event that more extensive measurements of lifetime as a function of temperature might be desired later.

2.4 Preparation of Li-Doped Samples

All of the Li-doped samples employed in these investigations were prepared by diffusing lithium into sample blanks prepared from commercial crystals. The source of lithium was a 4.2 molar solution of $LiAlH_4$ (lithium aluminum hydride) in ether. Samples were freshly etched in CP-4 etch, immersed in the hydride solution and allowed to dry in air. They were then heated in vacuum to diffuse the lithium. Following the diffusion treatment, the samples were cooled to room temperature and all of the surfaces were lapped to remove excess lithium remaining on or near the surface. The samples were then re-etched and reheated in vacuum to distribute the lithium more uniformly through the samples. Various diffusion and distribution cycles were employed in an attempt to determine the optimum method of distributing the lithium uniformly through the samples.

3. RESULTS

3.1 Lifetime Degradation Studies

In order to compare the relative sensitivity of various materials or devices to radiation, it is convenient to express the sensitivity quantitatively in terms of a lifetime damage constant. For our purposes, we define the damage constant, K, through the relationship

$$\frac{1}{\tau} = \frac{1}{\tau_o} + \frac{\phi}{K} \qquad (1)$$

where τ_o and τ, respectively, are the initial and post-irradiation lifetimes at 30^oC and ϕ is the gamma fluence. If the lifetimes are expressed in microseconds, the damage constant is numerically equivalent to the fluence required to reduce the lifetime of an initially perfect sample ($\tau_o \approx \infty$) to 1 μs, and is thus indicative of the radiation resistance or "hardness" of the material. The constant defined in this manner is the reciprocal of that employed in some device studies, which is proportional to the damage rate.

Lifetime damage constants for a number of samples which were irradiated with a similar fluence of 1.1×10^{16} gammas/cm^2 are listed in Table I. The sample designation employed in the table and used throughout this report denotes the crystal manufacturer, growth method, dopant and the

TABLE I Lifetime Degradation of Silicon Irradiated with 1.1×10^{16} Co^{60} gammas/cm^2 (6.8×10^6 R).

Sample Designation	τ_o µs	τ_ϕ µs	Damage Constant, K (γ's cm^{-2} µs) x 10^{17}
GCB 8.4	16.2	9.8	2.74
GCB 8.2	18.1	7.9	1.51
DCB 11.6	11.0	7.9	3.14
TLB 4.3	433	31.0	3.67
TLB 4.3	411	32.5	3.88
TLB 9.4	433	45.4	5.58
TLB 8.9	433	46.2	5.69
DVB 9.4	193	32.5	4.30
DVB 9.1	153	31.7	4.40
GCAl 6.3	17.3	7.2	1.36
GCAl 15.2	27.1	11.0	2.04
TLAl 4.4	462	12.3	1.39
TLAl 4.4	483	11.5	1.30
TLAl 6.1	115	8.4	0.99
TLAl 6.4	115	8.2	0.97
TLAl 8.9	153	10.8	1.28
TLAl 9.0	164	12.6	1.50
GFAl 4.2	60.6	8.2	1.05
GFAl 6.3	164	9.1	1.06
GFAl 6.3	274	7.9	0.90
GFAl 8.4	77.9	8.4	1.03
GFAl 8.7	77.9	9.4	1.17
GFAl 9.4	310	12.2	1.40
GFAl 10.0	382	11.9	1.35
GFAl 9.9	164	25.2	3.28
GFAl 10.0	170	26.4	3.44
GFAl 10.1	167	25.1	3.25
GFAl 10.2	173	25.1	3.23

TABLE I, continued

Sample Designation	τ_o µs	τ_ϕ µs	Damage Constant, K (γ's cm^{-2} µs) x 10^{17}
TLP 126*	96.7	2.7	0.30
TLP 130	136	2.8	0.31
TLP(Li) 1.0	144	6.5	0.74
TLP(Li) 1.2	164	3.6	0.41
TLP(Li) 2.1	82.2	4.5	0.53
TLP(Li) 3.9	222	4.7	0.52
TLP(Li) 4.6	231	5.8	0.65
TLP(Li) 5.1	188	5.4	0.61
TLP(Li) 6.2	162	7.9	0.92
TLP(Li) 12.1	289	21.4	2.54

* Annealed 12 hours at 400°C before irradiation.

initial room temperature resistivity, respectively. Manufacturers D, G, and T are the Dow Corning Corp., General Electric Co., and Texas Instruments, Inc., respectively. Additional Al-doped crystals were obtained from the Allegheny Electronic Chemicals Co. and Electronic Space Products, Inc. These crystals are not listed in Table I. However, some of these materials were used in later studies and will be denoted by the appropriate manufacturer symbol A or E when they are listed. Growth techniques C, L, V, and F represent the Czochralski (pulled), Lopex, vacuum-float zone and float-zone (argon atmosphere) methods, respectively.

For ease of comparison, samples which were prepared from the same crystal are grouped together in Table I. In some cases only one sample was prepared from a particular crystal due to the poor quality of the material or because the crystal was too small to provide multiple samples.

The various crucible-grown crystals obtained from the General Electric Company were grown using a crystal puller designed for research purposes. The puller had a limited capacity and the crystals were consequently smaller than those obtained from usual commercial sources. Twelve different crystals weighing a total of 550 grams were grown in an attempt to produce $\sim$100 grams of single crystal, Al-doped material having a nominal resistivity in the 10 ohm cm range. Many of these ingots were unsuitable because they contained extremely large resistivity gradients or were

polycrystalline. The large gradients were presumably due to the extremely small segregation coefficient of aluminum in silicon (0.002), and were especially large in the shorter crystals. All these crystals were produced during preliminary experiments performed to determine optimum crystal growth parameters and were not intended for use in this contract. However, a lengthy labor strike at the General Electric facilities prevented further crystal growth attempts during a critical period of the contract and we were forced to employ the material on hand. We also attempted to obtain Al-doped crucible-grown crystals from other manufacturers. These attempts were unsuccessful due to the technical difficulties associated with the production of such crystals and to the reluctance of manufacturers to employ production apparatus for this purpose during the current worldwide shortage of single cyrstal silicon.

An examination of the data in Table I shows that the damage constants of samples prepared from the same crystal are very similar, but the values vary from crystal to crystal without any clear dependence upon the pre-irradiation characteristics. Evidently, the observed differences between damage constants of Al-doped samples and various crystals which are similar in other respects are the result of subtle differences in the growth processes.

With the exception of the four GFAl samples which were prepared from the same $\sim$10 ohm cm crystal, all of the Al-doped samples listed in Table I

appear to be more sensitive to radiation than do the comparable B-doped samples. Recent studies in which some of these materials were irradiated with neutrons revealed that they are also at least as sensitive to neutrons as are comparable B-doped materials.[14] The results shown in Table I agree with those obtained in a previous study of gamma-irradiated silicon in which low resistivity Al-doped crystals appeared to be more sensitive to lifetime degradation than did comparable B-doped crystals.[13] However, in Section 3.5, all these results are placed in proper perspective and the conditions under which Al-doped Si is relatively less sensitive to irradiation are demonstrated.

All of the Li-doped samples listed in Table I were prepared from blanks cut from a P-doped crystal grown by the Lopex process. This crystal was relatively long (> 9 cm), and the resistivity varied from ~100 ohm-cm at one end to ~160 ohm-cm at the other. In most instances, the initial lifetimes of samples cut from the crystal exceeded 100 μs, but the values varied more than the resistivity.

Samples TLP 126 and TLP 130 indicated in the table were used to compare the effects of the lithium diffusion on the properties of this material. As noted in the table, sample TLP 126 was annealed for 12 hours at 400°C before it was measured. This treatment was performed to determine whether

possible effects observed in Li-diffused samples were due partially to the heat treatment employed in the diffusion and distribution cycles. The fact that the damage constants exhibited by these two samples are identical within experimental error indicates that this treatment did not affect the radiation sensitivity of this material.

The Li-diffused samples exhibit a relatively large range of initial resistivities, lifetimes, and damage constants. The resistivity range is partly due to differences in the diffusion and distribution cycles employed in preparing the samples. In general, the higher resistivity samples were produced by using shorter diffusion times or heating at lower temperatures. However, the final resistivity of the samples could not be controlled,and the apparent lithium concentration in samples given the same treatment often varied by more than a factor of three.

Compared to the starting material, the Li-doped material tends to have an even higher initial lifetime, in spite of its lower resistivity. Evidently the introduction of Li drastically lowers the concentration (or effectiveness) of recombination centers. Although there is some scatter, the variation of damage constant with carrier concentration is close to that expected from theory, being roughly constant at higher concentrations and increasing with decreasing number of carriers. However, many of the diffused samples, especially those containing apparently large concentrations of lithium,

exhibited trapping behavior below room temperature which was not observed in undiffused samples. These effects are apparently due to trapping centers associated with defects involving lithium and become effective only below room temperature.

The minimum in the curve of Figure 1 illustrates the effect of these centers on the apparent pre-irradiation lifetime of sample TLP (Li) 1.0 of Table I. In contrast, the pre-irradiation curve of sample TLP (Li) 12.1, shown in Figure 2, does not display the minimum near room temperature which is characteristic of the trapping behavior.

Although there is considerable scatter in the values of the damage constants of the Li-doped samples, the results indicate that they are significantly more radiation-resistant than the higher resistivity starting material or phosphorus-doped samples of comparable resistivity and growth type.[13]

3.2 Isochronal Annealing Studies

Figure 2 shows the temperature dependence of the lifetime of sample TLP (Li) 12.1 before irradiation and after isochronal (30 minutes) anneals at the indicated temperatures. Anneals were performed at eleven different temperatures in the range from 72° to 253°C (1000/T range from 2.9 to 1.9 $^{o}K^{-1}$) corresponding to equal intervals on a reciprocal absolute temperature

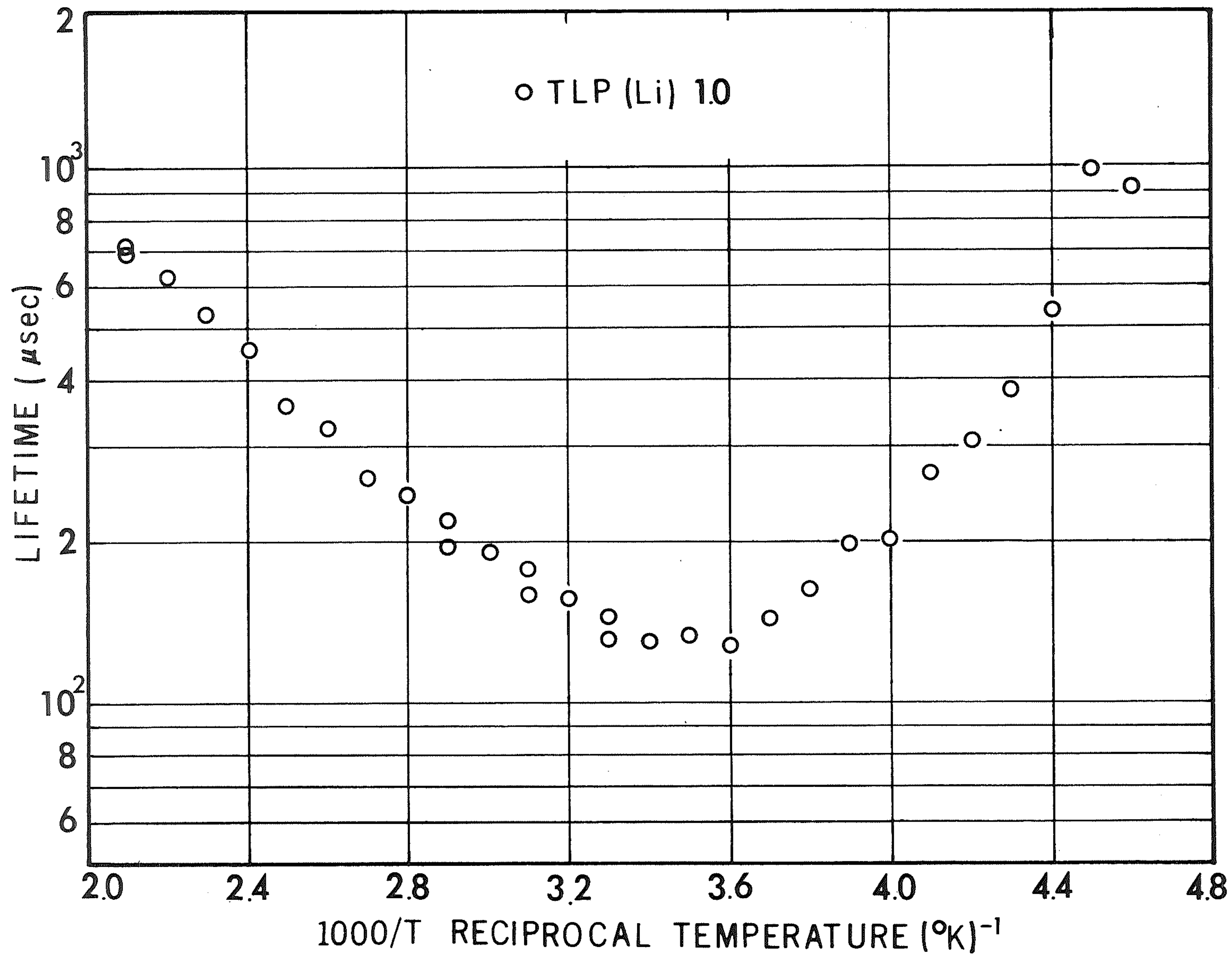

Figure 1. Temperature Dependence of Lifetime of TLP(Li) 1.0.

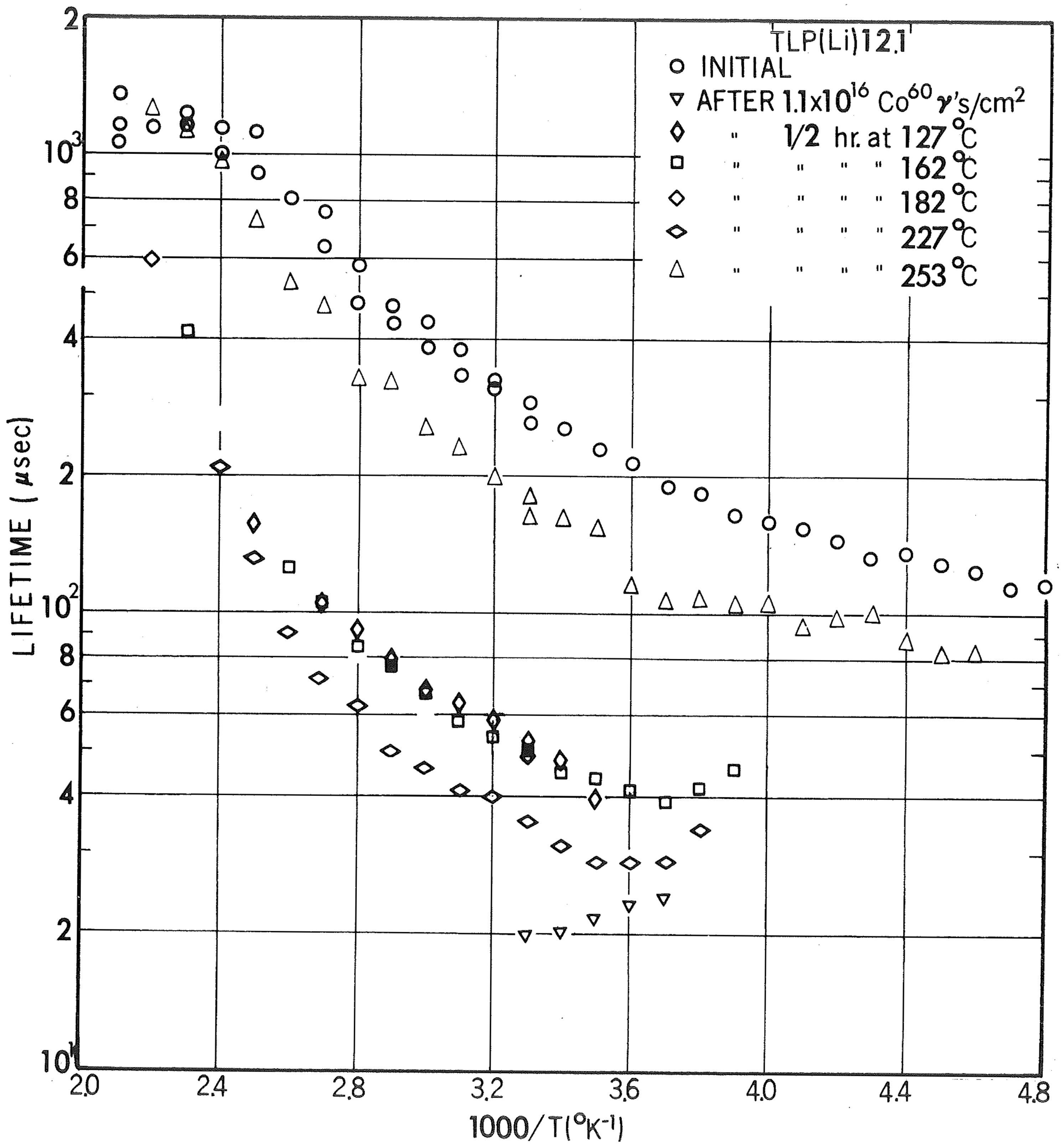

Figure 2. Temperature Dependence of Lifetime of TLP(Li) 12.1 Before and After Gamma Irradiation and After Subsequent Anneals.

scale. Temperature dependence data were not obtained after each anneal and some additional curves have been omitted from the figure for the sake of clarity.

The radiation-induced lifetime τ_ϕ and pre-irradiation lifetime τ_o are expected to add reciprocally, i. e.

$$\frac{1}{\tau} = \frac{1}{\tau_o} + \frac{1}{\tau_\phi} \tag{2}$$

It is often useful to plot the "induced reciprocal lifetime" ($1/\tau_\phi$) as a function of temperature when the slopes of the initial and post-irradiation curves differ. The data of Figure 2 have been replotted in this manner in Figure 3. As will be discussed below, the slope value assigned to the lower temperature linear regions does not correspond to an energy level position.

Figure 4 shows the recovery of the reciprocal lifetime of this sample measured at 30°C following a one-half hour anneal at each of the indicated temperatures. Since the lifetime is expected to vary inversely with the recombination center concentration, the curve represents the fraction of radiation-induced centers remaining after each anneal. The fraction not annealed, f, is defined as

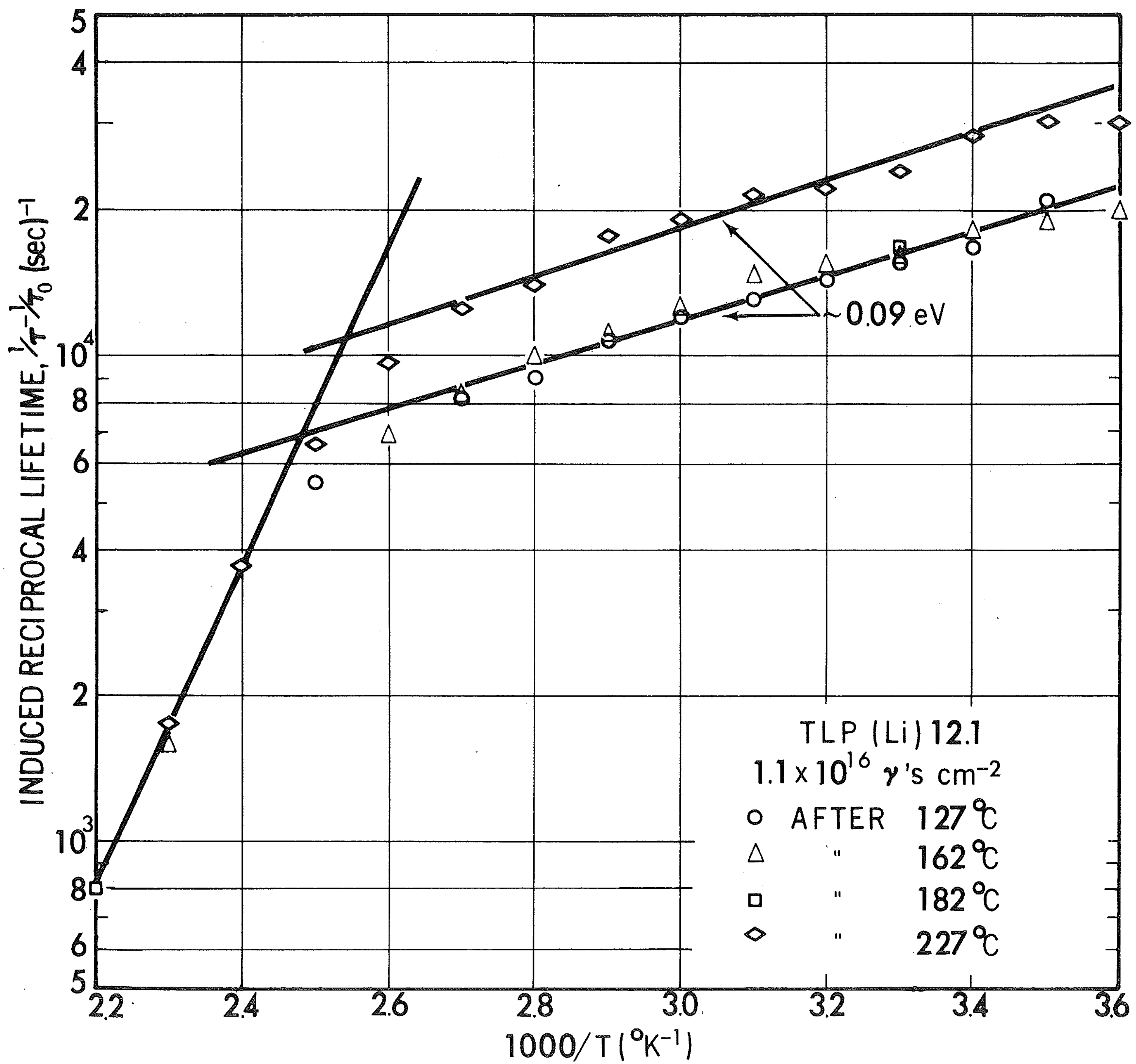

Figure 3. Induced Reciprocal Lifetime vs Reciprocal Temperature of TLP(Li) 12.1 After Gamma Irradiation and Subsequent Anneals.

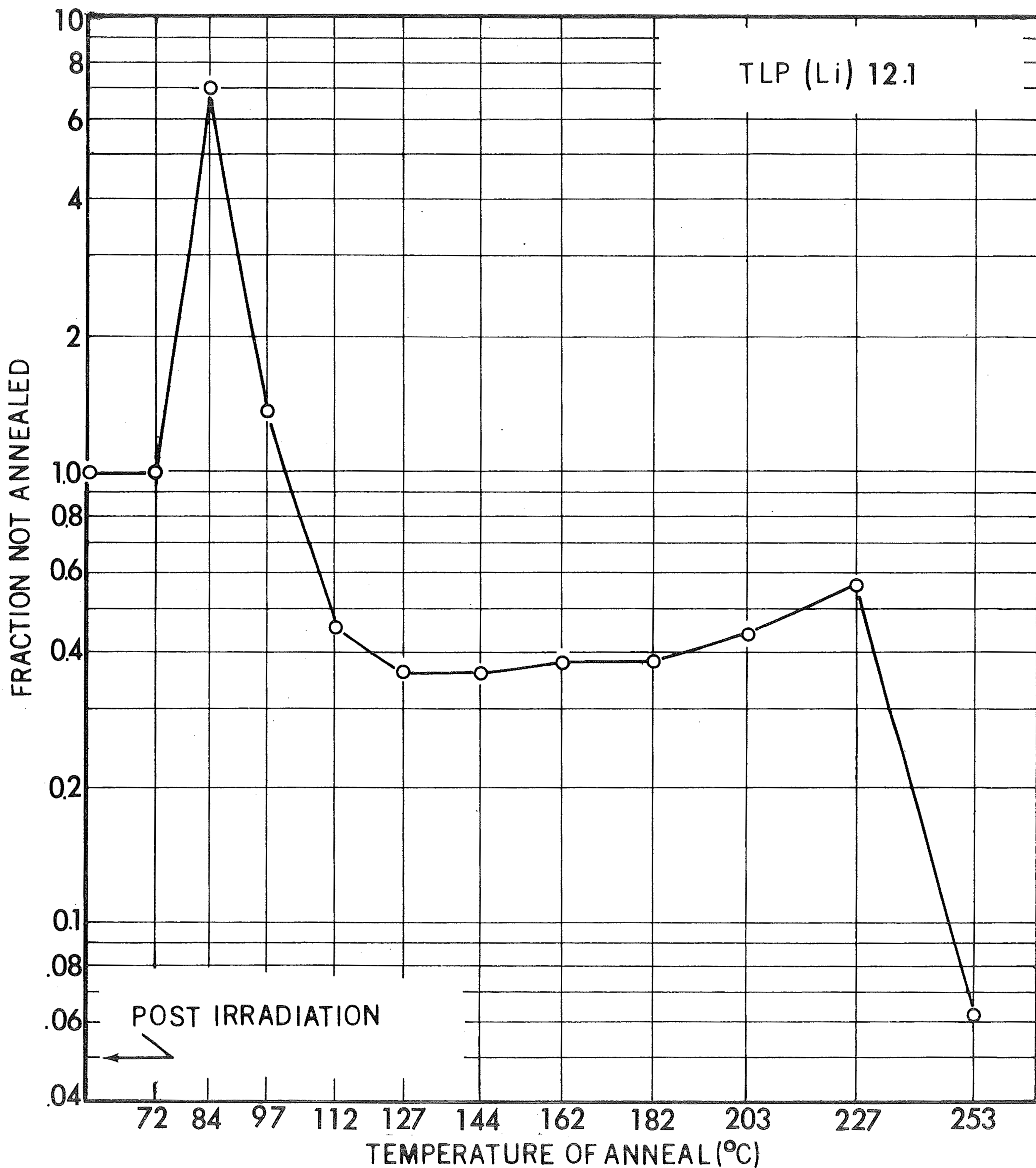

Figure 4. Isochronal Annealing of Reciprocal Lifetime at 30°C of TLP(Li) 12.1 Following Gamma Irradiation.

$$f \equiv \frac{\frac{1}{\tau_T} - \frac{1}{\tau_o}}{\frac{1}{\tau} - \frac{1}{\tau_o}} \quad (3)$$

where τ_o and τ, respectively, are the initial and post-irradiation lifetimes at 30°C and τ_T is the lifetime at 30°C following the anneal at temperature T.

The absence of any significant recovery after the anneal at 72°C and the large reverse annealing stage at 84°C evident in the figure are surprising in view of reported spontaneous recovery of Li-doped solar cells at room temperature following proton and electron irradiation[8, 15-17]. It is interesting to note that the approximately seven-fold decrease in lifetime damage after the anneal at 84°C was accompanied by a five-fold increase in the amount of trapping, indicating the creation of both recombination and trapping centers.

Three additional Li-doped samples were isochronally annealed to determine whether the reverse annealing behavior exhibited by TLP (Li) 12.1 is characteristic of this material. The annealing curves for these samples are shown in Figure 5. Although two of the samples display reverse annealing behavior after annealing at 72°C, the magnitude of the decrease in lifetime is much smaller than that of TLP (Li) 12.1. In contrast, sample TLP (Li) 3.9 does not exhibit reverse annealing behavior. On the contrary, more than 60% of the lifetime damage was removed from this sample after the anneal at

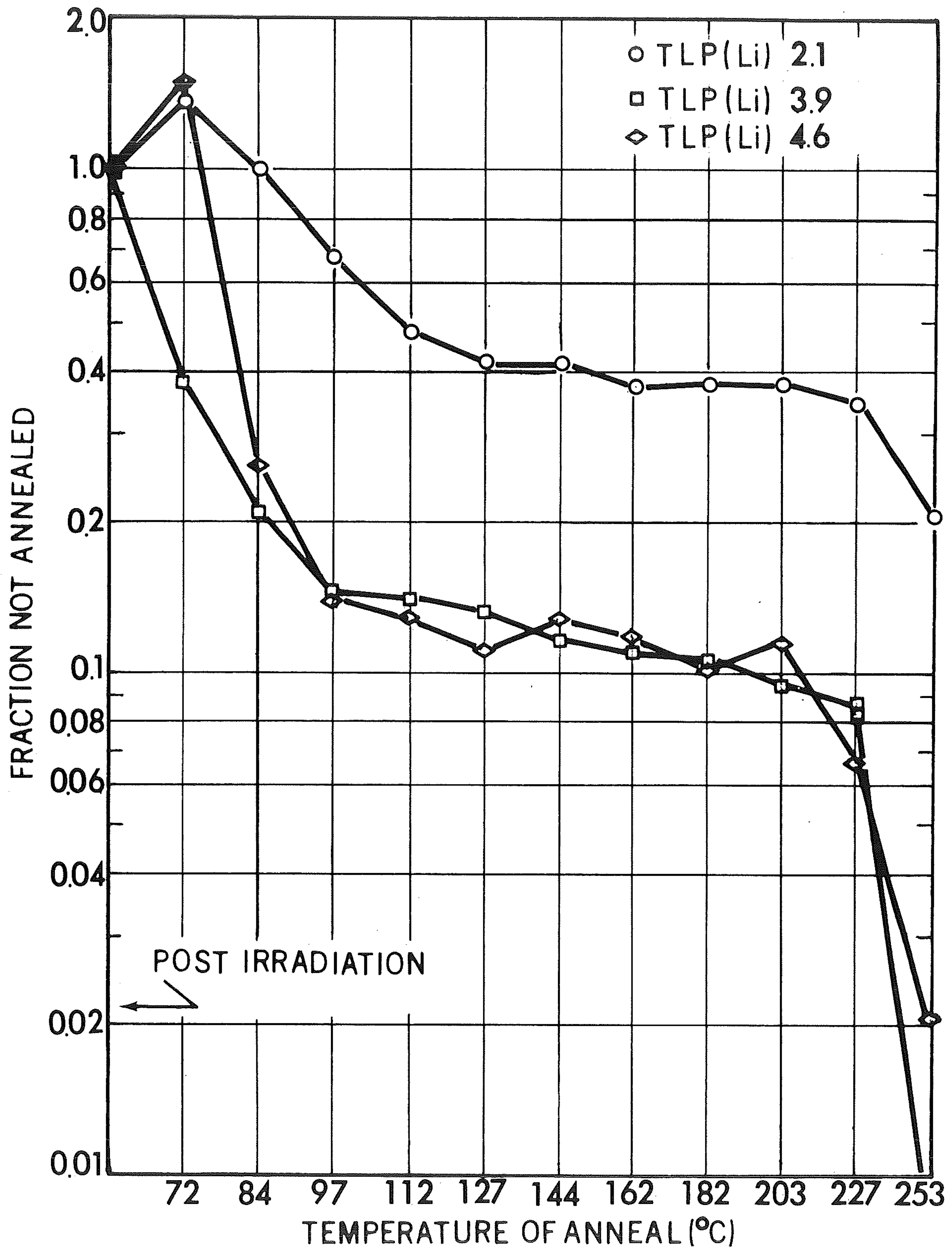

Figure 5. Isochronal Annealing of Reciprocal Lifetime at 30°C of Three Li-Diffused Gamma-irradiated Samples.

72°C and all of the damage was removed at 253°C. The large differences in the isochronal annealing behavior of the samples of Figures 4 and 5 is another indication of variations among samples which was also evident in the temperature dependence of the lifetimes and the induced reciprocal lifetimes of the samples following various anneals.

3.3 Homogeneity of Li-Doped Samples

In specifying sample resistivities, potential profiles of Li-diffused samples were used to give an indication of the dopant distribution throughout the volume of the sample. This procedure was based upon the results of a number of earlier experiments in which lithium was evaporated on two opposite faces of a slab of silicon approximately 7mm thick and potential profile measurements were performed on small samples which were cut from this slab following various heat treatments.

Figure 6 shows the effect of two different heat treatments on the potential profile of a sample which was cut from a slab of ~60 ohm-cm P-doped material into which lithium was diffused for 4 hours at 425°C. Note that the profiles were taken in the transverse direction and are consequently expected to reflect the distribution of lithium as a function of distance from the evaporated surfaces. The results indicate that after the sample was heated for 66 hours at 425°C, two regions of relatively uniform resistivity

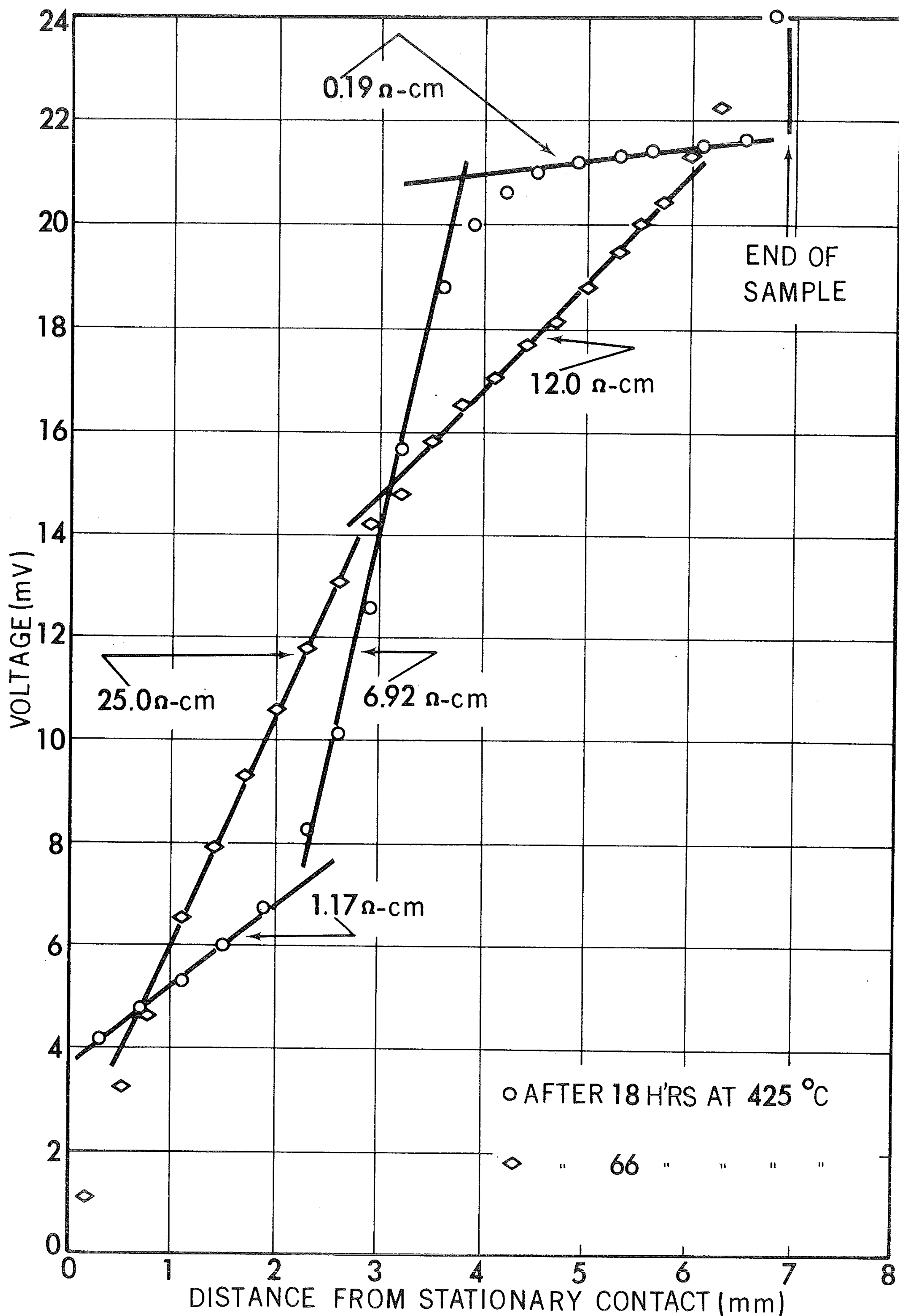

Figure 6. Effect of Different Distribution Heat Treatments on the Potential Profile of Li-diffused Silicon.

were formed. These regions were approximately 3mm thick, which suggests that suitable samples could probably be obtained by cutting ~7mm thick samples lengthwise following similar diffusion and distribution heat treatments. However, when these techniques were applied to normal sample blanks, potential profiles measured along the 30mm length of the samples were non-linear, and the apparent lithium concentration in the samples was much higher than desired.

Other investigators have reported similar results in attempting to prepare samples having significantly smaller dimensions[18]. Results of further experiments in which both evaporated lithium and $LiAlH_4$ sources were used revealed that the longitudinal potential profiles of samples which were diffused for longer than approximately 15 minutes at 400° to 450^oC were non-linear regardless of the time and temperature of subsequent distribution heat treatments. On the other hand, linear profiles were nearly always obtained for samples which were diffused for 5 to 10 minutes at 400^oC and distributed for periods of from 12 to 24 hours at this temperature. A typical profile obtained for such a sample is illustrated in Figure 7. Sample TLP (Li) 12.1 indicated in the figure was diffused for ~8 minutes at 400^oC and distributed for 24 hours at the same temperature. Since diffusions were performed from four sides rather than two, the procedure was expected to provide reasonable transverse homogeneity along with the obviously good

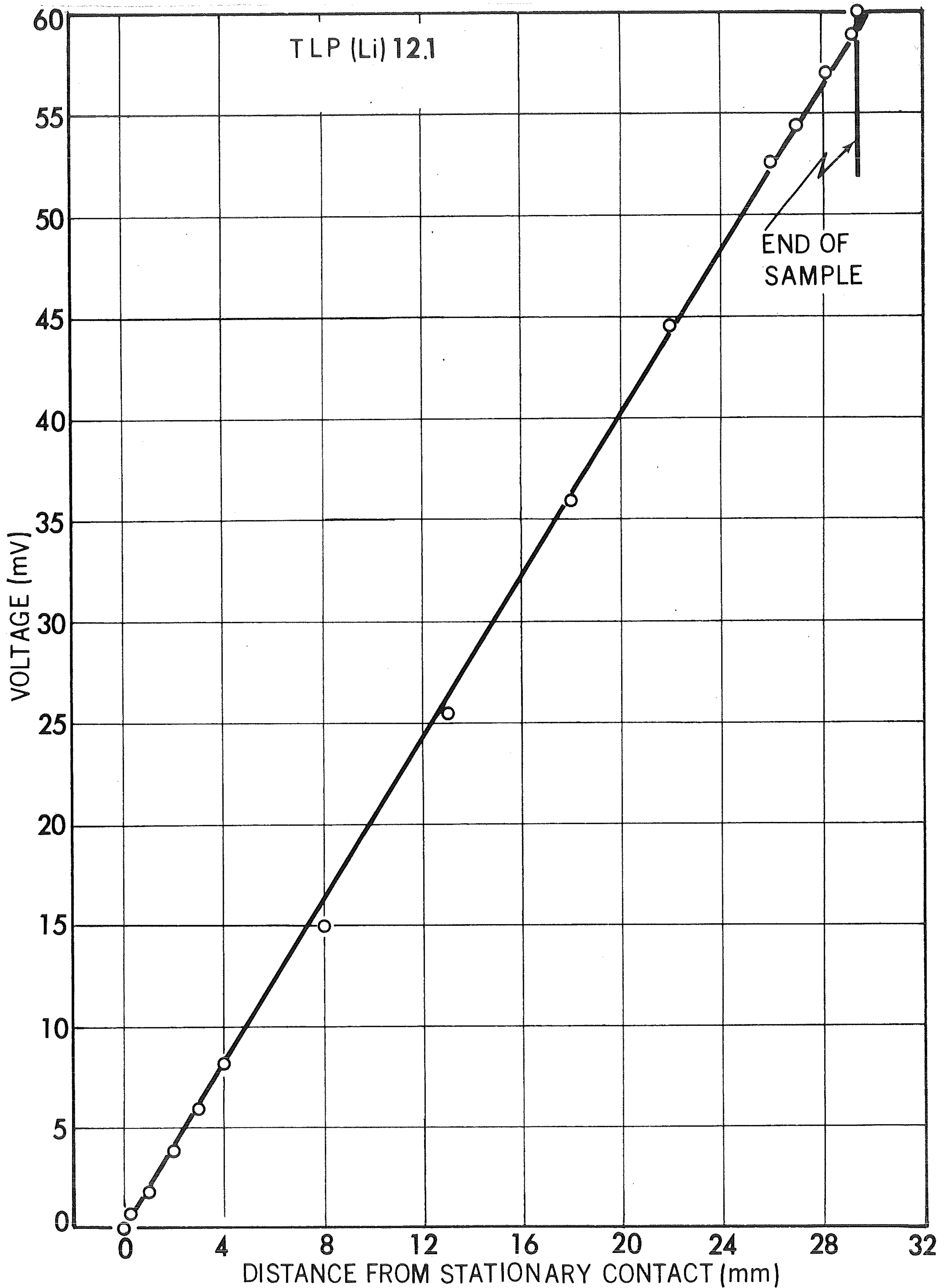

Figure 7. Potential Profile of TLP(Li) 12.1.

longitudinal uniformity. The very small voltage drops near the ends of the sample further indicate that the soldered contacts were excellent.

The sample was later lapped on all four sides to remove the surface layer, and the potential profile measurements were repeated. This procedure was repeated three times until the cross-sectional dimensions of the sample were reduced more than one-half. Results of these measurements are shown in Table II and reveal that the resistivity of the sample increased very rapidly with depth, although the surface appeared to be uniform. The exterior and interior halves of the sample volume had resistivities of ~7.5 and 33 Ω-cm, respectively. Considering the initial doping level, the amount of lithium introduced into the exterior half of the volume was about five times that present in the interior. After the third lapping, the resistivity of the sample was almost the same as that of the undiffused material. This fact indicates that little or no lithium diffused into the central 20% of the sample during the diffusion and distribution heat treatments. Similar studies were performed on other samples which exhibited linear potential profiles after short diffusion and distribution cycles, and the results were similar to those shown in Table II. For instance, one sample with only a 12 hour redistribution at 400°C had a measured resistivity of 4.1 Ω-cm, while the interior one-half showed a value of 18 Ω-cm.

TABLE II. Effect of Surface Removal on the Apparent Resistivity of Sample TLP (Li) 12.1

Sample Status	Resistivity (ohm-cm)	Thickness, t (cm)	Width, w (cm)	% Volume Remaining
After Co^{60} γ-irradiation and isochronal anneals to 253°C	12.1	0.710	0.660	100%
After first lapping	33.5	0.510	0.441	48%
After second lapping	85.4	0.410	0.340	30%
After third lapping	112	0.331	0.277	19%

After it was discovered that the potential profile measurements were not indicative of the transverse sample homogeneity, and that the relatively short diffusion and distribution heat treatments did not introduce lithium into the center of the samples, further experiments were performed in which samples were diffused and distributed for much longer periods and at higher temperatures. Following these treatments the resistivity was measured at various points on the surface using a four-point probe.

Figure 8 shows the results of such measurements on a sample which was diffused for 60 hours and distributed for 69 hours at 450°C. The resistivity indicated in the sample designation (3.6 Ω-cm) was determined from the potential profile which is shown in Figure 9. The results indicate that the sample is nonuniform near the ends but is relatively uniform over the central region. Although the four-point probe measurements were performed on the same surface used for the potential profile measurements, the former results indicate a much less uniform sample with a higher resistivity than the potential profile measurements **show**. Similar results were obtained for other samples which were measured using these techniques. In addition, four-point probe measurements performed at the center of each face of a given sample exhibited a wide range of values.

Potential profile measurements were performed on TLP (Li) 3.6 after it was successively lapped to remove the surface. The effect of surface

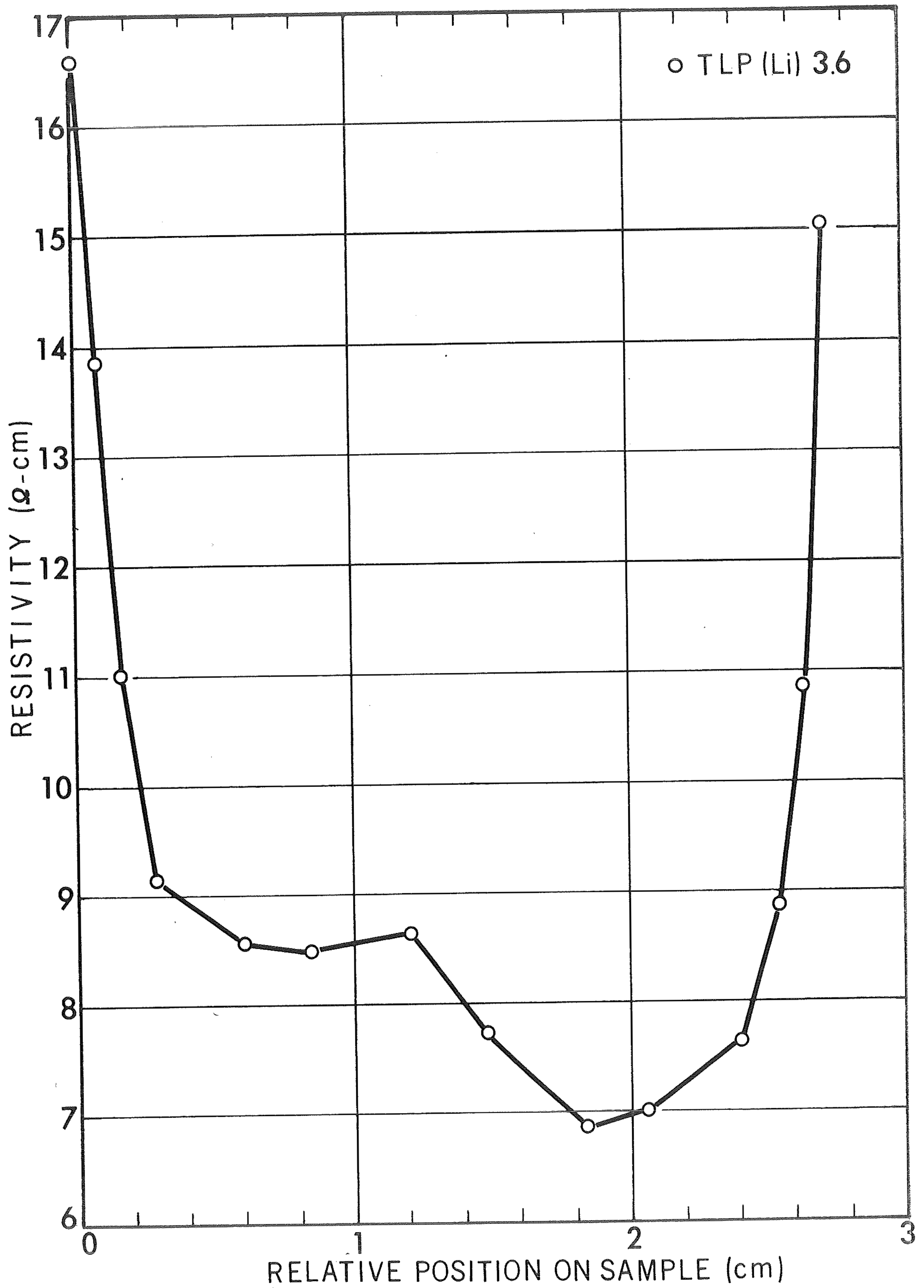

Figure 8. Resistivity as a Function of Position Along One Side of a Li-diffused Sample. (Measurements determined by four-point probe technique).

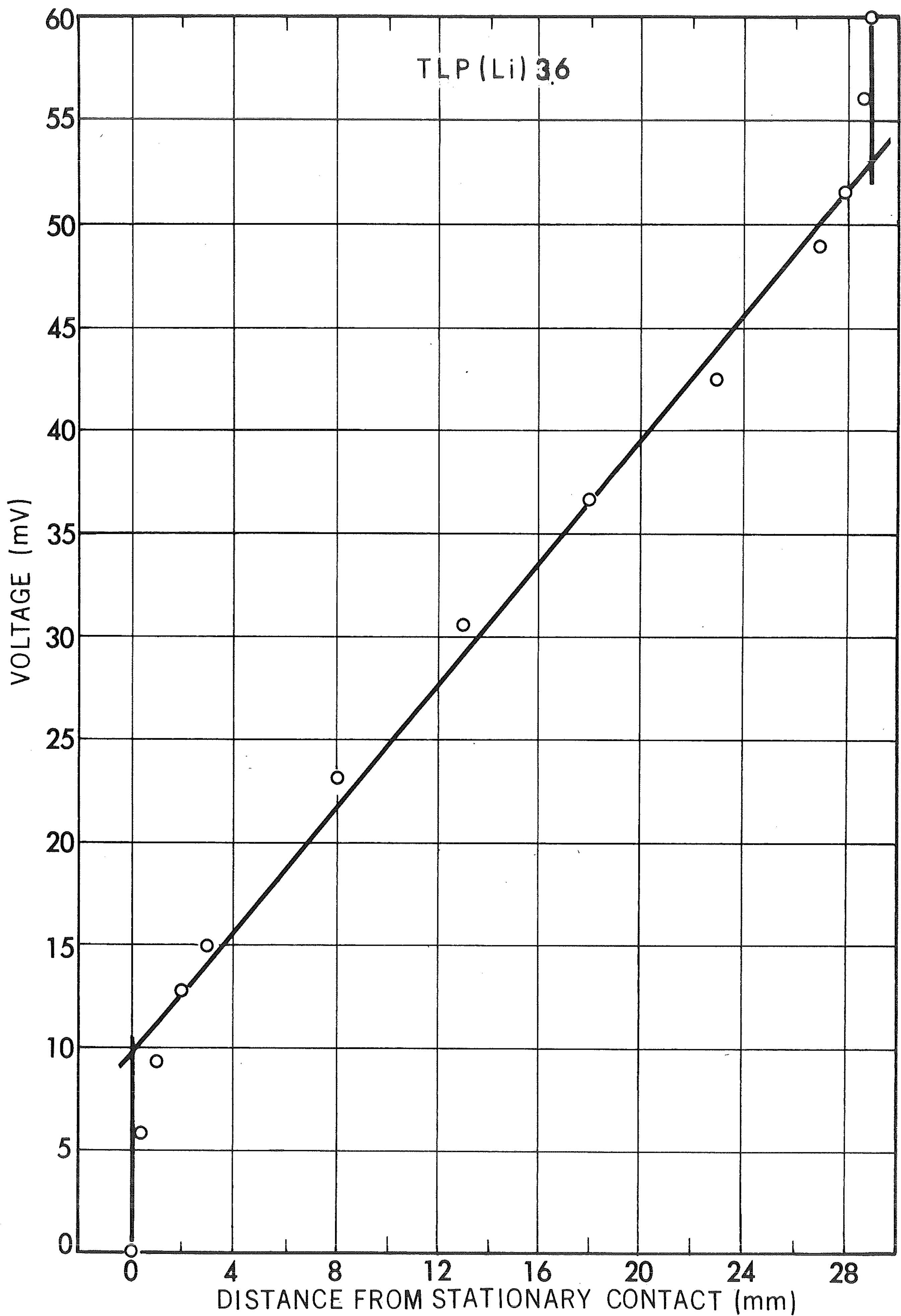

Figure 9. Potential Profile of TLP(Li) 3.6.

removal on the apparent resistivity of the sample is illustrated in Table III. Two points are apparent: 1) The longer diffusion did reach the entire crystal volume, and produced a relatively uniform resistivity. 2) The outer portion of the sample actually attained a higher resistivity than the interior, indicating some surface loss mechanism. This agrees with the data of Figure 9, where the ends of the samples appear to exhibit high resistivity. Evidently some sample treatment intermediate between that given in Tables II and III would be optimum.

TABLE III. Effect of Surface Removal on the Apparent Resistivity of Sample TLP (Li) 3.6

Sample Status	Resistivity (ohm-cm)	Thickness t (cm)	Width w (cm)	% Volume Remaining
After 60 hour diffusion and 69 hour distribution at 450°C (sample shortened by cutting 1mm from each end)	3.3	0.676	0.677	100%
After first lapping	2.1	0.475	0.475	48%
After second lapping	1.9	0.375	0.375	29%
After third lapping	1.9	0.300	0.302	20%

3.4 Recombination Parameters

Comparison of the data of Figure 3 with that of Downing[19] indicates a similarity at the temperatures for which the data overlap. However, our higher temperature data cannot be explained on the simple basis used by Downing. It is clear that a much deeper energy level is responsible for recombination. Although these limited data do not provide an accurate estimate of a position, several worthwhile conclusions can be drawn. First, since the lifetimes associated with two levels add reciprocally, the observed slopes cannot be attributed to activation energies of two energy levels because the steeper slope would occur at the lower temperatures, not at the higher temperatures as is the case here. This fact is readily demonstrated by the reciprocal addition of two lifetime components, one with a shallow slope and one with a steep slope. We conclude that the shallow slope in the lifetime temperature dependence curve cited by Downing and observed at the lower temperatures in the present case does not directly yield a recombination center energy level position.

Further analysis was based on the Hall-Shockley-Read model. The expression employed for minority carrier lifetime is

$$\tau = \frac{p_1}{c_n n N} + \frac{n_1}{c_p n N} + \frac{1}{c_p N}, \qquad (3)$$

where all terms have their usual meaning. The apparent activation energy at lower temperatures, from 0.06[Ref. 19] to 0.09 eV, quite likely corresponds to a variation of hole capture probability with temperature (dominance of the $1/c_p N$ term). At the higher temperatures the dominant term is either

$p_1/c_n nN$ or $n_1/c_p nN$. If the latter is the case, the energy level position can be determined from the intersection of the two lines through the high and low temperature portions of the curve. At that point, $n_1 = n$. This occurs at $1000/T \approx 2.52^{\circ}K^{-1}$, yielding an energy level position at $E_c - E_r \approx 0.40$ eV. Since the capture probability temperature variation observed at lower temperatures and the temperature dependence of the density-of-states function would affect the higher temperature slope, the observed slope should be ~ 0.53 eV. This value is similar to that observed. On the other hand, if the term containing p_1 dominates, the level would be near the center of the gap. Further investigation will be required to provide more definitive information, particularly in view of the lack of homogeneity of the sample as discussed in the previous section.

3.5 Additional Studies on Al-Doped Material

We have seen up to this point that the radiation sensitivity of both pulled and float-zone gamma-irradiated Al-doped Si (not heat treated) does not appear to differ significantly from that of B-doped material. Our previous studies[8] have indicated that heat-treated Czochralski-grown Al-doped Si, which exhibited significant resistivity changes, is less sensitive to neutron irradiation than B-or Ga-doped material. Having eliminated all other major variables in our gamma-irradiation studies of Al-doped material, it remained to examine the radiation sensitivity of heat-treated pulled crystal specimens. Before discussing the experiments, a summary of some pertinent work contained in the literature is given.

3.5.1 Literature Background

Resistivity changes in undoped, heat treated, pulled-crystal silicon were first reported 16 years ago.[20] Such changes were explained qualitatively on the basis of the formation of silicon-oxygen compounds, one of which (SiO_4) acts as a donor below $\sim 500^\circ C$.[21] In studies of the effects of dopants (B, Ga, and Al) on the observed phenomena, it was found that considerably larger carrier concentration changes occurred in doped specimens than was previously noted for undoped material.[22] Additionally, a considerably larger percentage (using the acceptor concentration as a base) of donors was found to form in Al-doped material than in the B or Ga cases, and the donors appeared to be considerably more stable above $500^\circ C$.

A more detailed study of the effects of acceptors and oxygen on heat-treated silicon was performed by Fuller, et al.[11] (referred to as I below). In this study, results for Ga-doped material were found to be quite similar to those obtained for B doping, but, as before, Al-doped material exhibited a somewhat different behavior. Before discussing the differences, some general features of the results in I are summarized. Heating oxygen-containing, acceptor-doped silicon for long periods at the proper temperature (typically in the range of $400\text{-}500^\circ C$) results in the formation of donor sites with accompanying carrier concentration changes. The amount of change depends primarily on four parameters: 1) acceptor concentration; 2) oxygen concentration; 3) heat-treatment temperature; 4) amount of time the specimen is heat-treated at a particular temperature. The reactions that produce the observed carrier concentration changes were not definitely determined in I, but the data supported the postulation of the formation of primarily three distinct compounds: 1) SiO_4 donor sites; 2) neutral oxygen-acceptor sites;

3) acceptor-oxygen sites that act as donors. Considerably larger carrier concentration changes could be obtained in Al-doped material than in comparable B-doped specimens. In fact, particular Al-doped samples were actually converted to n-type; this was not possible in the B case. It is postulated that the reason for this difference may be that acceptor-oxygen donor sites are formed more readily in the Al case than for B doping.

3.5.2 Experimental

We first describe preliminary experiments performed to study resistivity changes in heat-treated specimens. Eleven Al-doped crucible-grown samples which had initial lifetimes shorter than ~ 10 μs and which exhibited excessive trapping were annealed for 8 hours at $460^{\circ}C$ to determine the effect of this treatment on the electrical properties of this material. In an earlier experiment, two similar samples were converted to high resistivity n-type after heating for 24 hours at this temperature. The use of a shorter annealing period was expected to produce more moderate changes in the later samples. Four of the heat treated samples were obtained from four different General Electric crystals which had very short lifetimes. Before they were annealed, the recombination behavior of these samples was dominated by a very slow trapping center with an effective lifetime of approximately 1 second at room temperature. The lifetime of one of the samples was measured as a function of temperature and the results are shown in Figure 10.

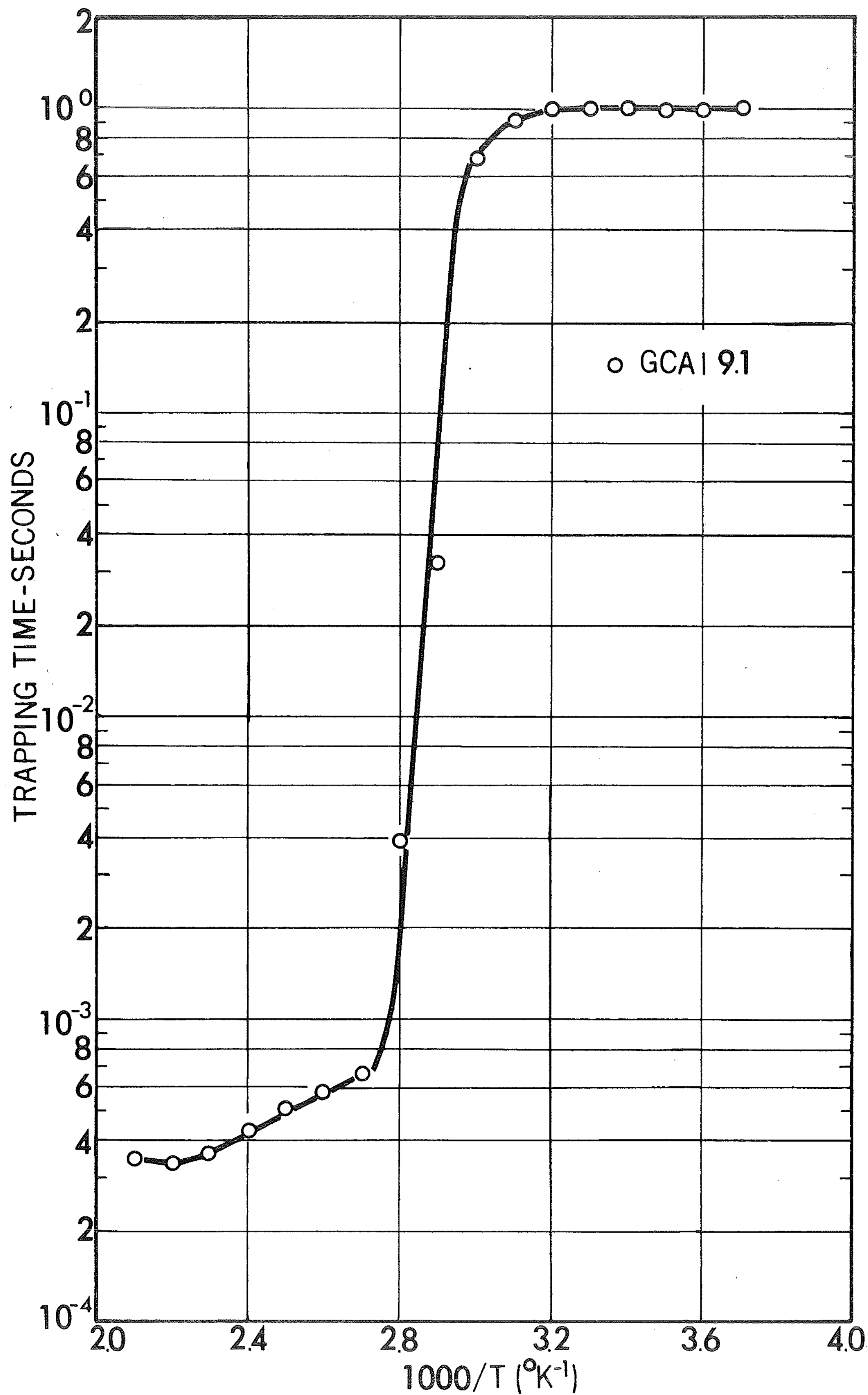

Figure 10. Recombination Behavior of an Al-doped Sample Showing the Effects of Slow Traps.

Observe that in the range from approximately 40°C to 100°C ($2.7 \lesssim 1000/T$ ($^{\circ}K^{-1}$) $\lesssim 3.2$), the apparent lifetime of the sample decreased by more than three orders of magnitude. This behavior and the apparent decrease in lifetime with increasing temperature confirms that the photoconductivity decays were associated with trapping time constants rather than recombination processes prior to annealing.

The presence of these traps rendered the initial lifetimes of most of the eleven samples uncertain. The sample resistivity was also monitored to determine the effects of the 8-hour anneal. The resistivities and lifetimes at room temperature before and after the anneal are shown in Table IV. The initial resistivity values were determined from potential profiles which were performed on each sample before the anneal. Since potential profile measurements are relatively time-consuming, these measurements were not repeated on annealed samples unless the samples exhibited significant lifetime increases after the heat treatment. If no such improvement was observed, the post-anneal resistivity value was calculated from the measured resistance at room temperature. Consequently, the small resistivity changes indicated for some samples may be due to differences in the measurement techniques.

An examination of the data in Table IV shows that the resistivity of most of the samples increased as a result of the anneal. Since soldered contacts were used on the samples, it was necessary to remove them before the anneal and to replace them afterward. The quality of the replaced contacts was determined by measuring the sample resistance in reverse directions

TABLE IV - Effect of Heating for 8 hours at 460°C on the Lifetime and Resistivity of Al-doped Silicon

Sample Designation	Crystal No.	Resistivity - ohm cm		Lifetime - μs*	
		Initial	Final	Initial	Final
GCAl 4.0	CZ 88	4.0	333	(T)	65 (T)
GCAl 4.0	CZ 82	4.0	20	(T)	15 (T)
GCAl 5.8	CZ 87	5.8	~75	(T)	(R)
GCAl 9.1	CZ 84	9.1	~94	(T)	(R)
ACAl 1.8	6170-1	1.8	3.0	<1.0(T)	(T)
ACAl 1.8	6170-1	1.8	1.9	<1.0	(T)
ACAl 3.7	6170-3	3.7	8.6	14.3(T)	11 (T)
ACAl 4.9	6170-3	4.9	5.3	<1.0(T)	1.6
ACAl 5.0	6170-3	5.0	4.9	8.7(T)	8.1 (T)
ECAl 1.5	202	1.5	4.2	<1.0(T)	(T)
ECAl 1.7	202	1.7	177	<1.0(T)	(T)

*Lifetime value uncertain due to rectification (R) or trapping (T).

at both room temperature and dry ice temperature. With the exception of samples GCAl 5.8 and GCAl 9.1, none of the samples rectified after the anneal. Additional measurements on these samples revealed that the rectification occurred at a junction in the material which was not observed before the anneal and did not involve the contacts.

The preliminary experiments described above, which resulted in the data of Table IV, were helpful because they demonstrated particular conditions under which appreciable resistivity changes could be realized and also made us aware of the special problems associated with measuring recombination lifetimes in Al-doped pulled crystal material. In the light of this information, a new experimental approach was utilized in subsequent investigations. The main differences were the following. First, it was attempted to obtain samples with resistivities (after heat-treatment) that were within a factor of ~ 2 of those values commonly used in solar cells. Second, in order to closely monitor resistivity changes at various times during heat treatment with a minimum of effort expended, four point probe techniques were used instead of the usual potential profile method. Several comparisons of the two methods yielded agreement within ~ 10-20% for unannealed Al-doped specimens. Third, multiple pulse techniques were found to be quite useful in separating recombination lifetimes and trapping effects. The method involves using from 5 to 10 x-ray pulses in rapid succession to excite carriers in the samples. The x-ray apparatus used in the measurement of lifetimes is capable of emitting seven 150 keV x-ray pulses per second. Repeated pulsing fills up

long time constant traps, and a saturation condition is reached in which exponential decays due to recombination can be separated from trapping effects.

Samples measuring $\sim$7 x 7 x 30mm were prepared from Al-doped crucible-grown ingots obtained from Texas Instruments, Electronic Space Products, and Allegheny. Comparison samples of B-doped material (obtained from Dow and Knapic) were also prepared. After lapping all surfaces, resistivity measurements were made using a four-point probe. Readings were typically made at three positions on all four sides of the samples for forward and reverse polarities. An average resistivity reading was obtained, based on all readings. The Al-doped specimens were then subjected to long periods of heat-treating. Resistivity measurements were made a number of times during the treatment in order to monitor closely any changes with time. Table V gives the annealing history of each sample and the initial and final resistivity values. Figure 11 shows the resistivity variation with time for sample TCAl 0.2 during heat treatment at 450°C for 315 hours.

Following heat treatment of the Al-doped specimens, room temperature lifetime was measured for all samples and is given in Table V. It is interesting to note that prior to heat treatment the lifetime in Al-doped material is usually too short to be measured. Heat treatment increases the lifetime considerably. All samples were then gamma-irradiated

Sample	Initial ρ Ω-cm	Heat Treatment	Final ρ Ω-cm	τ_o μsec	τ (after 2.2×10^{16} γ's/cm^2) μsec	K (after 2.2×10^{16} γ's/cm^2 (γ's cm^{-2} μs) $\times 10^{17}$	τ (after 4.5×10^{16} γ's/cm^2) μsec	K (after 4.5×10^{16} γ's/cm^2) (γ's cm^{-2} μs) $\times 10^{17}$	τ (after 1.0×10^{17} γ's/cm^2) μsec	K (after 1.0×10^{17} γ's/cm^2) (γ's cm^{-2} μs) $\times 10^{17}$
TCAl 0.252	0.252	315h@450°C	0.431	6.20	5.05	6.05	4.33	6.48	3.17	6.75
TCAl 1.26	1.26	133h@500°C	1.86	36.1	7.94	2.26	6.49	3.57	6.20	7.79
TCAl 2.51	2.51	5h@600°C + 94h@500°C + 56h@600°C	2.68	2.81	2.02	1.60	1.27	1.05	0.75	1.06
ACAl 1.77	1.77	8h@460°C + 64h@450°C[a]	2.13	4.33	2.06	0.87	2.67	3.14	1.39	2.13
ECAl 1.53	1.53	97h@350°C + 8h@460°C	2.94	36.8	18.2	7.99	28.9 (?)	60.7 (?)	11.8	18.1
DCB 2.44	2.44	None	2.44	23.8	6.49	1.98	3.10	1.61	1.67	1.87
DCB 2.53	2.53	None	2.53	47.6	7.07	1.84	3.82	1.87	1.73	1.87
DCB 2.54	2.54	None	2.54	43.3	6.64	1.74	3.97	1.97	1.73	1.87
KCB 0.393	0.393	None	0.393	42.6	4.33	1.07	2.31	1.10	1.30	1.40
KCB 0.372	0.372	None	0.372	57.7	4.33	1.04	2.16	1.01	0.87	0.92

[a] Heated for 64 hours in a molten tin bath

Table V. Sample History and Damage Constants for Gamma-Irradiated Al- and B-doped Crucible-Grown Silicon

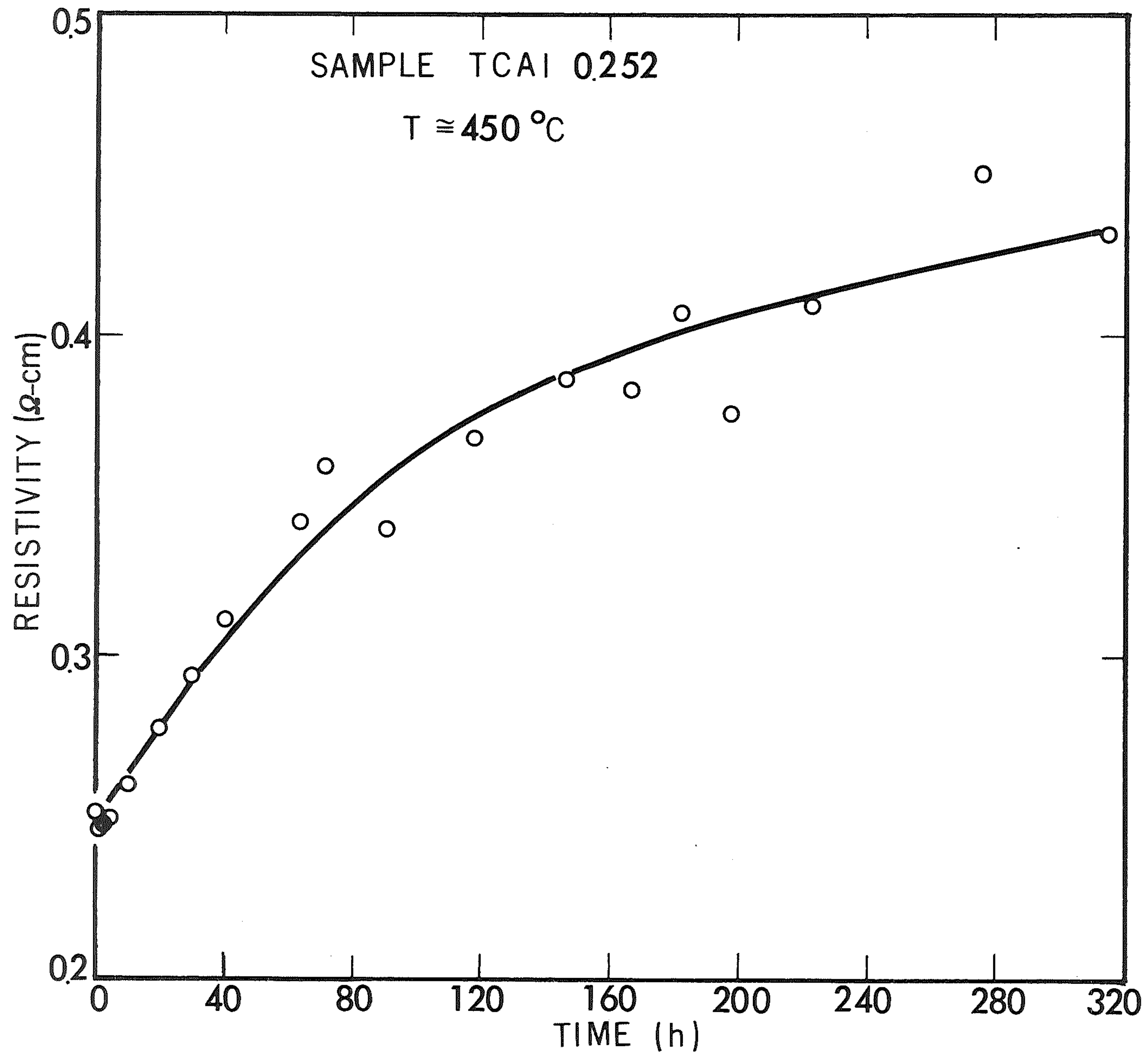

Figure 11. Variation of Resistivity With Time at ~ 450°C for Sample TCAl 0.252

at dry ice temperature to a dose of $\sim 2.2 \times 10^{16}$ photons/cm^2, room temperature lifetime was remeasured after ~ 1 hour at room temperature and damage constants calculated (see Table V). Following this, irradiation was resumed to a total dose of $\sim 4.5 \times 10^{16}$ photons/cm^2. Post-irradiation lifetimes and damage constants following this second irradiation are also given in Table V, as are results following a third irradiation to a total dose of $\sim 1.0 \times 10^{17}$ photons/cm^2.

3.5.3 Results

Before discussing the damage constant results, it should be mentioned that it is difficult to predict how much the resistivity of a particular sample will change at a specific temperature in a given time period unless the oxygen concentration is known. We did not perform studies to determine oxygen content, but instead used a trial-and-error method while employing the results of I as a guide. In any future studies, our experience on a number of samples using the rough technique, coupled with the data of I and accurate oxygen concentration determinations, should enable us to accurately specify the temperature and annealing time necessary to produce desired resistivity changes in Al-doped samples.

The results of Table V indicate an apparent correlation between damage constant and resistivity change; i.e., those samples which experienced the largest percent increase in resistivity upon heat treatment (TCAl 0.252 and ECAl 1.53) exhibited a significant decrease in radiation sensitivity. After all three irradiations, the damage

constant for sample TCAl 0.252 was a factor of ~ 6 greater than that for the low resistivity B-doped samples (KCB 0.393 and 0.372). After 2.2×10^{16} photons/cm^2, sample ECAl 1.53 had a damage constant that was a factor of ~ 4 greater than that for the three B-doped ~ 2 Ω-cm control samples. After 1.0×10^{17} photons/cm^2, the damage constant for this sample was $\sim$an order of magnitude greater than that for the controls. (The anomalously high damage constant for sample ECAl 1.53 after 4.5×10^{16} photons/cm^2 may possibly reflect an error in measurement, especially since the measured lifetime is actually <u>higher</u> than that measured after the lower dose irradiation.) To insure that recombination lifetimes were being measured instead of trapping times for samples TCAl 0.252 and ECAl 1.53, lifetime was measured as a function of temperature slightly above and below room temperature after the 2.2×10^{16} photons/cm^2 irradiation. A decrease in lifetime with decreasing temperature was found for both samples, which supports the conclusion that recombination lifetimes were being observed.

Sample TCAl 1.26, which experienced about a 50% resistivity increase, exhibited a damage constant slightly greater than the control samples after the first irradiation. After the second and third irradiations, K was a factor of ~ 2 and of ~ 4, respectively, greater than in the controls. Sample TCAl 2.51, the resistivity of which did not change very much upon heat-treating, exhibited a damage constant that was slightly less than that of the B-doped ~ 2 Ω-cm specimens after all three irradiations. After the second irradiation, the K value for sample ACAl 1.77 was slightly less than a factor of 2 greater than that for the control samples. After the third irradiation, this factor decreased to near unity. The resistivity change in this sample was $\sim 20\%$.

We see that the results obtained are qualitatively similar to our previous data for neutron-irradiated material.[8] Pulled-crystal Al-doped Si that undergoes an appreciable resistivity change upon heat-treatment ($\lesssim$ a factor of two) appears to be significantly less sensitive to both neutron and gamma irradiation than comparable B-doped crucible-grown material. It is encouraging to note that although more work will be necessary to thoroughly explore the limitations, optimization, and applicability of our findings, all data obtained to the present support the conclusion stated above. Because of this favorable support, we feel that it is reasonable to predict that additional work (which would extend our initial results reported here) may well result in the development of a solar cell which is $\sim$one order of magnitude less sensitive to radiation than currently used devices.

The interesting question is raised as to whether heat-treated B-doped material, which experiences an appreciable resistivity change, would also be more radiation tolerant. The data of I indicate that significant resistivity changes are possible for oxygen-rich B-doped specimens. We have not examined this question in detail as yet, but such a study would seem to merit attention. However, it is indicated in I that donors produced in heat-treated B-doped material disappear above $\sim 800^{\circ}C$, as compared to $\sim 1100^{\circ}C$ in Al-doped material. Hence, heat-treated B-doped material would seem to be less suited to applications requiring high temperature diffusions ($> 800^{\circ}C$).

The question arises as to what mechanism is responsible for the observed decrease in radiation sensitivity for Al-doped material. It is difficult to even speculate at this early stage precisely what interaction(s) are occurring. The effect of the radiation-induced defects on lifetime is being diminished presumably by an interaction between these defects and one or more of the three types of compounds (mentioned above) thought to be formed during heat treatment. The SiO_4 donors can most likely be ruled out because they presumably are present in considerable quantity in heat-treated material which has not experienced a significant resistivity change. Because of the indicated correlation between resistivity change and decreased radiation sensitivity, it is tempting to speculate that the Al-oxygen donor sites, which apparently enhance the change in carrier concentration, are involved in reducing the effectiveness of the radiation-induced defects.

4. CONCLUSIONS AND RECOMMENDATIONS

In this report, we have described investigations of radiation effects on both Al- and Li-doped bulk silicon. Initial studies of Li-doped material indicated that this material is significantly more radiation-resistant than P-doped samples of comparable resistivity and growth type. Isochronal annealing studies revealed the presence of reverse annealing behavior in several samples after annealing at 72°C. In another sample, more than 60% of the damage was removed after the 72°C anneal. These differences led us to critically re-analyze the homogeneity of our Li-doped samples, and it was discovered that Li had not been diffused uniformly throughout the bulk of the specimens. This fact makes the interpretation of our initial radiation-resistance studies on Li-doped material somewhat uncertain. We then developed a procedure involving a diffusion and the distribution cycle employing considerably longer times and higher temperatures than previously used. The homogeneity of a sample prepared by this technique was markedly improved over those initially employed.

Considerably more positive results were obtained in the studies of Al-doped material. It is demonstrated that Czochralski-grown Al-doped Si that undergoes an appreciable resistivity change upon heat treatment ($\lesssim$ a factor of two) appears to be significantly less sensitive to gamma irradiation than B-doped crucible-grown material of comparable resistivity. We feel that these results represent

an important breakthrough which should have a significant impact on future solar cell technology. Because these new findings occurred quite late in our program, our data is somewhat limited in scope and it is evident that additional work will be necessary to more thoroughly explore the limitations, optimization, and applicability of the discovery.

In view of the potential impact of radiation-insensitive Al-doped material on solar cell applications, we recommend that the following research and development program be initiated in the very near future.

(1) Obtain a variety of low resistivity (< 1 Ω-cm) Czochralski-grown Al-doped silicon ingots with a range of oxygen concentrations.

(2) Use infrared absorption measurements to determine oxygen concentrations in the various crystals.

(3) Establish as a final goal the development of radiation insensitive material of a particular resistivity (for example, 1 Ω-cm) that would be appropriate for a solar cell base.

(4) Using the results of the present report and those of reference 11 as guides, heat-treat all specimens in an attempt to increase the

resistivity of each to the desired goal (i. e., 1 Ω-cm). It will be necessary to optimize the temperature, total annealing time, and oxygen concentration to accomplish this task for a specimen with a given initial resistivity.

(5) Irradiate all of the heat-treated specimens that have attained the desired resistivity goal and determine damage constants, using boron-doped samples as controls. Determine what conditions (i. e., starting resistivity, oxygen concentration, temperature and length of heat treatment) optimize the radiation tolerance of Al-doped material.

(6) Conduct long-term gamma irradiations of the optimum material to study dose effects.

(7) Determine what effects the technology used in the preparation of a solar cell will have on the properties of the optimum material. Specify the technological steps to be used in the manufacture of an Al-doped n-on-p solar cell that will insure radiation tolerance.

(8) Prepare and test the radiation sensitivity of Al-doped silicon solar cells.

5. NEW TECHNOLOGY

This report contains findings which we feel comprise new technology. It is demonstrated that heat-treated Czochralski-grown Al-doped silicon that undergoes an appreciable resistivity change ($\lesssim$ a factor of 2) is significantly less sensitive to gamma irradiation than comparable B-doped material. The optimum resistivity change (or, more precisely, carrier concentration change) remains to be determined. As is shown in reference 11, the amount of change depends on the initial carrier concentration, the oxygen concentration and the temperature and length of heat-treatment. We have typically used 450°C for periods of time ranging from hours to hundreds of hours to obtain significant resistivity changes. Following further study of these initial findings, the development of solar cells that are significantly more radiation tolerant than those currently employed may well become a reality.

TITLE:	Decreased Radiation Sensitivity in Heat-Treated Czochralski-Grown Al-Doped Silicon
INNOVATORS:	O. L. Curtis, Jr., and J. R. Srour
PROGRESS REPORT:	Not reported earlier
CURRENT REPORT:	Details of the innovation are presented in Section 3.4 of this report.
DATE:	July 31, 1970

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