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GACD-6249
(3/31/66) 3

RADIATION EFFECTS ON SILICON

Fifth Quarterly Progress Report Covering the Period
January 1, 1966, through March 31, 1966

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National Aeronautics and Space Administration
Contract NAS7-289
Project 430

April 11, 1966

1. INTRODUCTION

This fifth quarterly progress report on Contract NAS7-289, "Radiation Effects in Silicon Solar Cells," covers the period January 1, 1966 through March 31, 1966.

During this period, the electrical conductivity for the n-type ultra-pure silicon was measured from 4°K to 300°K. Also the transient conductivity was measured from 20°K to 300°K at high injection levels. The annealing characteristics of the divacancy formed at 80°K in p-type Si and the vacancy phosphorus complex formed at 80°K in n-type Si were studied. In addition microwave resonance studies were made to try to identify the recombination center responsible for this lifetime degradation. A portion of the results presented here were obtained under contract DA-49-186-AMC-65(X).

2. MICROWAVE CONDUCTIVITY

Work during this quarter has been concentrated on making the initial microwave measurements of the electrical conductivity of the ultra-pure n-type silicon.

2.1 EXPERIMENTAL MODIFICATION

As described in the previous quarterly report,[1] the microwave bridge is now tuned to reduce to a minimum the effect of small reflections off of the various components in the bridge. To attest to the validity of this tuning, the temperature characteristics of the reflected power of an empty sample holder shorted with a piece of copper were measured over a temperature range from 4.2°K to 300°K. There was no observable change in the reflected energy.

Since the stability of the tuning of the bridge is dependent on the relative movement of the microwave components, the bridge was fastened securely to the experimental bench. A piece of 2-mil Mylar is now used as the waveguide vacuum window replacing the previously used mica window. This window produces no measurable attenuation of the microwave energy.

It was also found necessary to stabilize the frequency of the klystron signal. This stability was obtained by the use of an automatic frequency control (AFC). Looking at a signal from a reference cavity which acts as discriminator, one obtains an amplitude-modulated signal whose phase depends on whether the klystron frequency is above or below the frequency of the reference cavity. This signal is then phase-sensitive detected, thus providing a dc voltage which is fed back to the klystron reflector for stabilization.

Because of the low conductivity of the ultra-pure silicon sample, maximum sensitivity is needed. Consideration of the dependence of -log A on sample thickness shows that maximum microwave absorption (maximum -log A) occurs for samples which are an odd number of quarter-wavelength long and that the greater the number of wavelengths of microwave power in the sample, the greater will be the absorptions. However, because of the rapid variation of a -log A with z (z = sample thickness), it is advisable to choose a value of z slightly different than the quarter-wavelength value, so that uncertainties in the conductivity are not introduced by small uncertainties in z. A sample thickness of 1.326 cm was chosen, slightly greater than 5/4 wavelength. Although the sensitivity for conductivity measurements would be improved by use of even a longer sample, measurements of bulk recombination time would not, due to competition of surface recombination. The magnitude of the surface recombination will be determined by the smallest sample dimension, in this case the narrow dimension of the waveguide: 1.03 cm.

As a preliminary testing of the transient conductivity using the microwave technique, a 500 ohm-cm silicon sample was used. The excess carriers were introduced by a 0.5 rad, 120 nsec, 600 keV pulsed X-ray source.

The lower trace on Fig. 1 shows the decay of the excess conductivity at room temperature. At this temperature, the conductivity decay is basically one recombination process with a time constant of about 300 μsec. Figure 2 shows the decay of the excess conductivity at about 78°K. At this temperature the recombination mechanism is made up of two processes, one with a long time constant of about 9 msec, indicating trapping, and a much faster component.

The transient conductivity data for the 500 ohm-cm n-type silicon was not extensively analyzed, since studies of the ultra-pure silicon were the main goal of the experiment. These measurements show that the previous difficulties in the microwave conductivity experiment have been overcome and that quantitative determination of the temperature dependence of conductivity and transient conductivity is now possible.

2.2 ULTRA-PURE n-TYPE SILICON

The high-purity n-type silicon was cut, lapped, and mounted in the sample chamber. The base of the sample was nickel plated and then tinned with 60-40 solder before it was soldered into the sample chamber.

The measured electrical conductivity versus 1000/T°K is shown in Fig. 3. It is seen that the room temperature conductivity of this ultra-pure silicon is about 10^{-4} $(\text{ohm-cm})^{-1}$. The general characteristics of the shape of the σ versus 1000/T°K are in agreement with previously published data on n-type silicon.[2,3] In the temperature region from 60°K to 300°K,

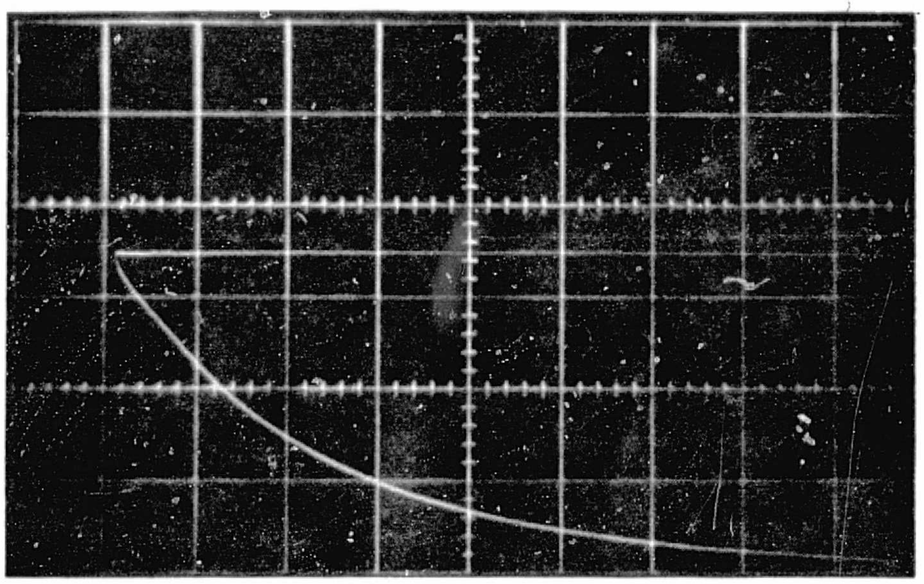

Fig. 1--Microwave signal showing decay of excess conductivity at room temperature

Horizontal: 100 μsec/div
Vertical: 5 mV/div

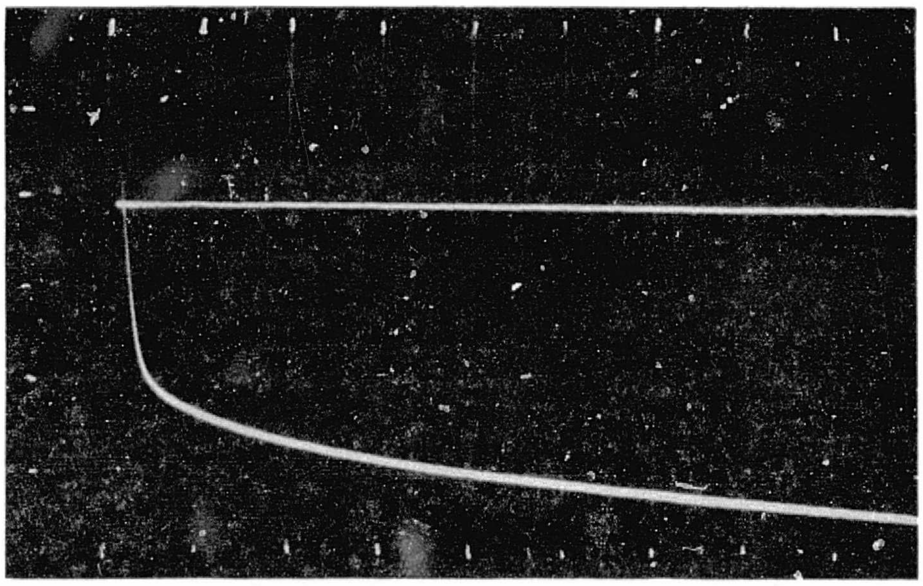

Fig. 2--Microwave signal showing decay of excess conductivity at liquid nitrogen temperature

Horizontal: 1 msec/div
Vertical: 5 mV/div

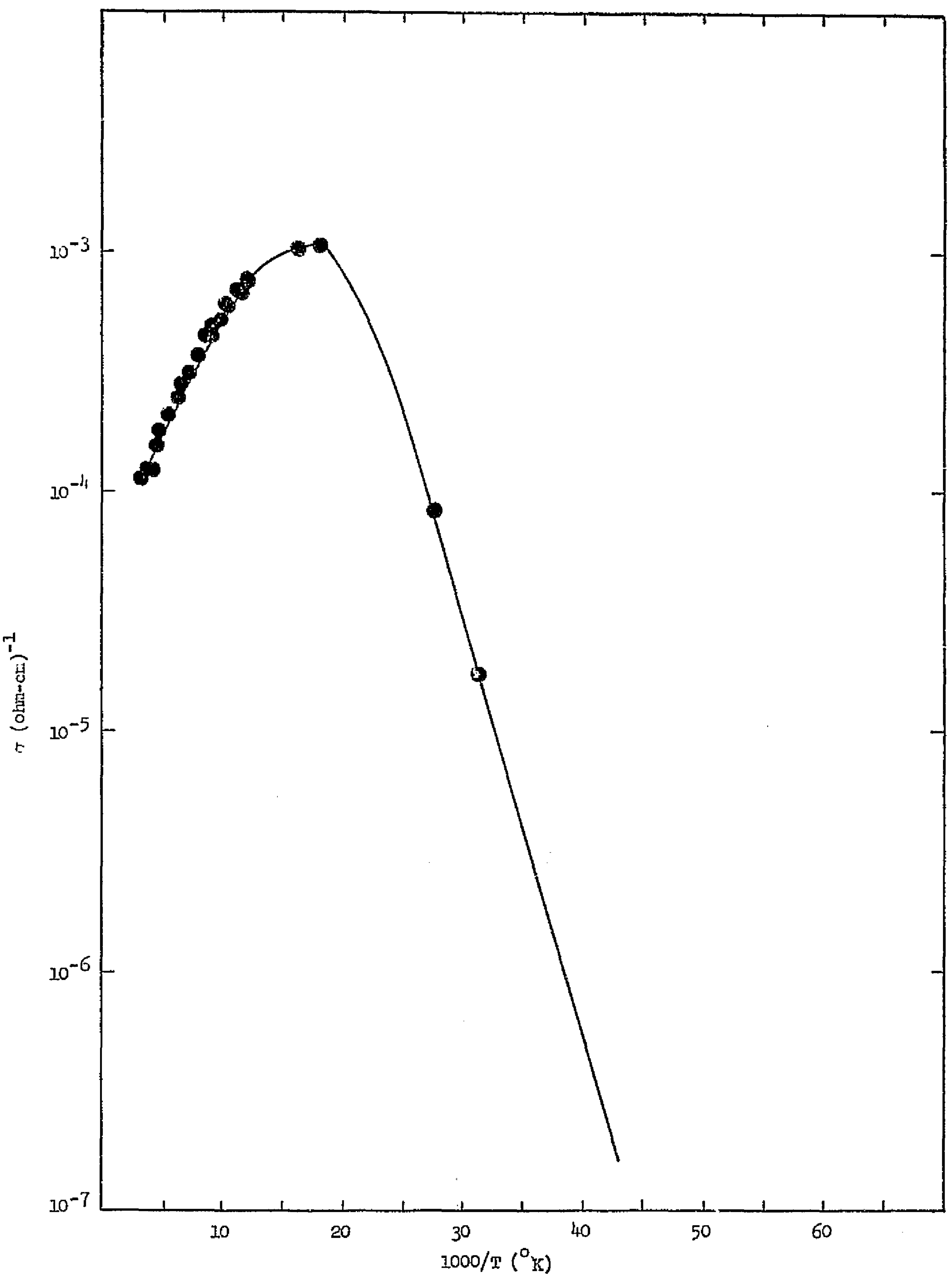

Fig. 3--Electrical conductivity of ultra-pure n-type silicon versus 1000/T°K measured by the microwave reflection technique

the conductivity shows a temperature dependence of $\sigma \sim T^{-1.57}$. This is the expected temperature dependence due to lattice scattering.[3]

The transient conductivity for the ultra-pure n-type silicon sample was measured from 20°K to 300°K and is shown in Fig. 4. This data was taken for carrier injection levels which produced conductivity changes greater than 200%. This data was analyzed by means of a computer program.

For all the data points in Fig. 4, the recombination is first order, i.e., it follows a relationship of the form

$$\frac{\sigma_f - \sigma_t}{\sigma_f \quad \sigma_0} = e^{-t/\tau}$$

where ρ_0 and ρ_f are initial and final conductivities, respectively, and ρ_t is the conductivity at any time t.

A preliminary analysis[4] of this data suggests that the donor impurity level is the recombination center for the excess carriers in the unirradiated sample. The slope of the lifetime versus 1000/T curve shows an activation energy of $\sim$ 0.04 eV. The donor level is located at 0.044 eV below the conduction band.

2.3 FUTURE PLANS

Lifetime measurements using low carrier injection levels are now being completed. The effect of electron damage and thermal annealing on the carrier lifetime will now be investigated.

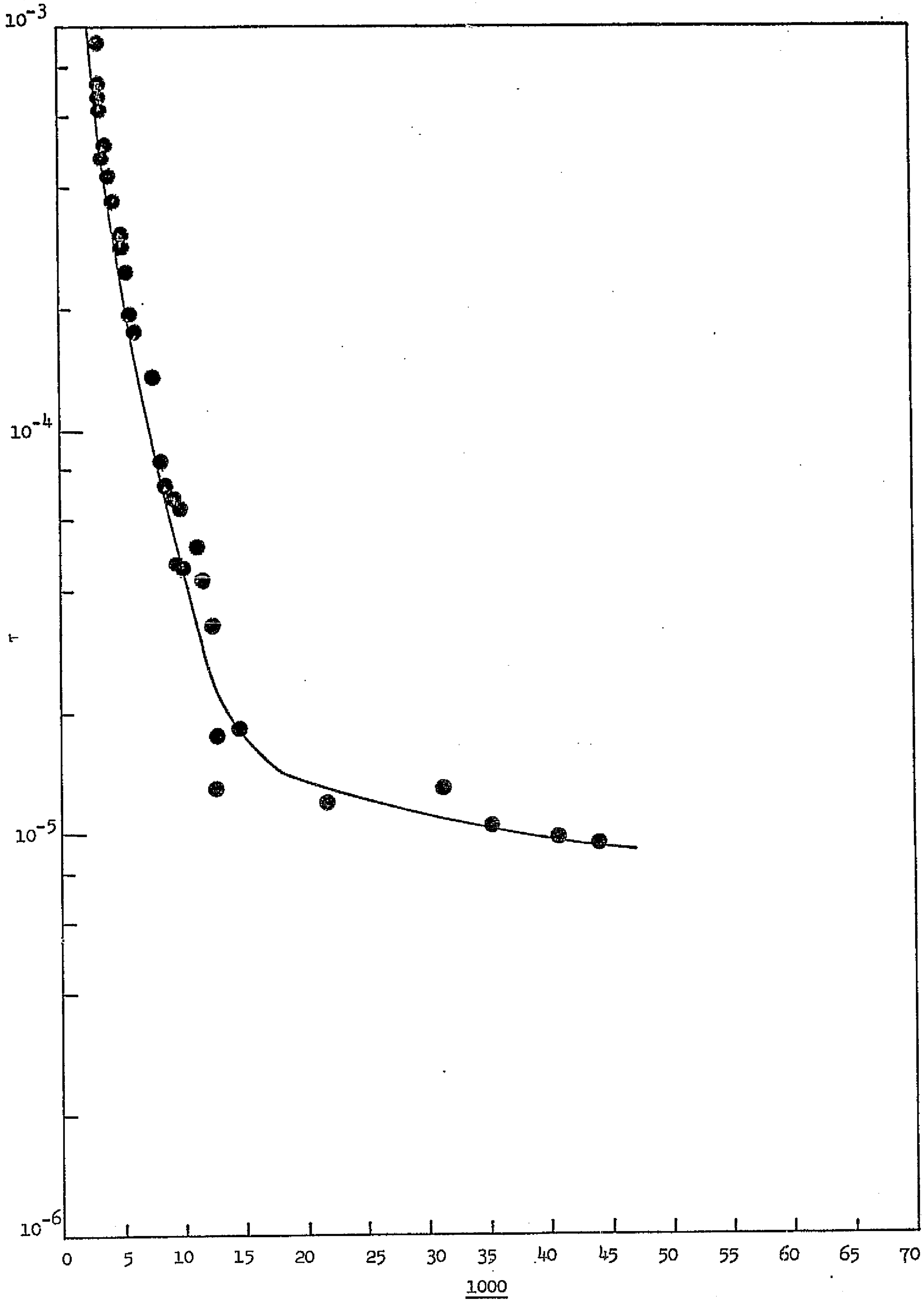

Fig. 4--Transient conductivity of unirradiated ultra-pure n-type silicon as a function of temperature

3. ELECTRON SPIN RESONANCE EXPERIMENTS

A study has been made of the annealing characteristics of the divacancy in p-type silicon and the E center, which is the vacancy phosphorus complex, in n-type silicon. The irradiations were performed at 80°K and the samples then annealed to 400°K. The resonance intensities were compared before and after the anneals. For the divacancy in p-type silicon, it was observed that there is at most a 10% increase in the number of centers after a 300°K anneal. This signifies that there is a very small number of indirectly produced divacancies in the 80 to 400°K temperature range which could mean that vacancy motion occurs below 80°K in B-doped silicon, assuming any mobile vacancies associate with one another to create additional divacancies.

In n-type P-doped silicon, a study of the intensity of the E center between 80°K and 400°K was also made. It has been found by Watkins[5] that the number of E centers increases around 120° and 280°K. In our experiments we have not observed this behavior. Up to 150°K there is at most a very small change in the number of E centers. Above this temperature, the number of E centers observed decreases drastically such that at about 300°K only 10 to 20% of the original number remains. Coincident with this observation, the resonance associated with the divacancy appears. To explain these observations, it would appear that a number of deep-lying levels are formed with highly energetic radiation. The Fermi level is lowered by these levels to a position such that the divacancy is no longer in the correct charge state for observation by spin resonance. The annealing rids the samples of the deep-lying levels and the Fermi level then rises to just above the E-center level. Thus that center is no longer observable with electron spin resonance but the divacancy, whose energy level we assume to be just above the E center level, is now seen. A complete interpretation of this phenomenon is now undergoing intensive study, especially since it is so radically different from that seen after low energy electron radiation.

A sample which covered one wall of the cavity was irradiated to a flux level of 9×10^{17} electrons/cm^2. The material was floating-zone-grown 10 ohm-cm P-doped silicon similar to that used in the lifetime studies.

At least two resonances were observed, but since they overlapped it is difficult to identify them. Also their introduction rate was about 0.01 cm^{-1} which is considerably smaller than that observed for the recombination center, 1 cm^{-1}.

To gain further information on this material, a number of 7 ohm-cm pulled and 10 ohm-cm floating-zone conductivity samples were irradiated at 300°K and then cooled in order to determine the position of the Fermi level after various amounts of flux. It was found that for flux levels between 5 and 12 x 10^{17} electrons/cm^2, the Fermi level was below 0.40 eV from the conduction band. It has also been found from the lifetime results that the recombination center is below 0.4 eV. Additional studies relating these three measurements will be made.

REFERENCES

1. E. G. Wikner and D. P. Snowden, "Radiation Effects on Silicon," Fourth Quarterly Report on Contract NAS7-289, General Atomic Report GA-6249 (12/31/65).

2. W. Crawford Dunlap, Jr., An Introduction to Semiconductors, John Wiley and Sons, New York, 1957, p. 270.

3. C. Kittel, Introduction to Solid State Physics, John Wiley and Sons, New York, 1957, p. 365.

4. O. L. Curtis, Jr., J. Appl. Phys. 30, 1174 (1959).

5. G. D. Watkins, private communication.