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RADIATION EFFECTS ON SILICON

Fourth Quarterly Progress Report Covering the Period
September 25, 1965, through December 31, 1965

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1. INTRODUCTION

This fourth quarterly progress report on Contract NAS7-289, "Radiation Effects in Silicon Solar Cells," covers the period September 25, 1965, through December 31, 1965. On July 21, 1965, a Final Report[1] was written covering the work performed under this contract from June 1, 1964, through May 31, 1965. That Final Report should be considered an Annual Summary Report, and the work discussed in the present progress report is a continuation of the same contract.

During this period the experimental setup to study microwave conductivity was modified to obtain improved low-temperature operation of the sample holder and better accuracy in conductivity measurements. Modifications have also been made in the cavity to be used for the microwave resonance studies. Preliminary experiments have begun to determine the indirect production of the divacancy in p-type silicon.

[1] V. A. J. van Lint and D. P. Snowden, "Radiation Effects on Silicon - Final Report," Contract NAS7-289, General Atomic Report GA-6556, July 1965.

2. MICROWAVE CONDUCTIVITY

Work during this quarter has concentrated on improvement of the experimental setup in order to obtain improved low-temperature operation of the sample holder and also better accuracy in conductivity measurements.

2.1 SAMPLE HOLDER

The sample holder has been redesigned in order to obtain better thermal contact with the coolant at liquid helium temperatures. The stainless steel sheath which surrounded the waveguide (see Fig. 5, Ref. 1) has been eliminated. A vacuum-tight seal is made at the bottom end of the waveguide by soldering the sample to a 0.002 in. thick copper sheet which is then soldered to the waveguide after the sample is inserted into it. The waveguide is evacuated through an 0.008 in. wide slot cut in the broad face of the guide just below the upper flange and pressure window. The styrofoam plug is provided with a diagonal hole through it to allow evacuation of the lower part of the waveguide.

Temperature sensors are attached to the bottom of the copper sheet; the copper-constantan thermocouple is soldered to the sheet and the carbon resistor is glued with a low-temperature varnish (General Electric 7031). In order to effect annealing studies, a nichrome heater is wound around the lower part of the waveguide and insulated from it. With this arrangement, temperatures between 4° and $400^{\circ}K$ are easily obtainable. Temperatures above those of the liquid coolants (either liquid helium or liquid nitrogen) are obtained after expelling the coolant by adding small pulses of room-temperature gas to the top of the working space in the dewar which contains the sample holder. This working space is enclosed with a top plate through which passes the waveguide, electrical feedthrus and fill and vent lines. The gas enclosed in the dewar acts as a thermal reservoir which provides adequate thermal stability for several minutes between such stepwise temperature increases.

2.2 MICROWAVE CIRCUITRY

As mentioned in Reference 1, small leakage signals through the magic tee could combine in random phase and produce serious errors in the measured conductivity. We have carefully investigated the nature of such signals and have found that, in addition to leakage through the magic tee, small reflections off of various other components in the microwave bridge can also create standing waves which mix with the desired signal and give erroneous results.

The seriousness of small spurious reflections on quantitative measurement of conductivity by the microwave-reflection technique can be easily seen. The voltage standing-wave ratio (VSWR) of various microwave components typically ranges from 1.02 to 1.15, with the lower values corresponding to simple components, such as bends, and the higher values to components, such as isolators and directional couplers. The lower value of 1.02 corresponds to a reflection coefficient of $\Gamma = 0.01$. Such a reflection mixing in arbitrary phase with a signal of amplitude 1 can yield an uncertainty in the measured microwave voltage of 2 percent. At values of sample conductivity which yield high reflectivity, large errors are possible because of the flatness of the curve relating conductivity to reflected voltage. Using the curves of Fig. 7 of Ref. 1 we can see that at $-\log A = 0.1$ a 2 percent error in the reflected voltage can yield an error as high as 30 percent in the conductivity. At lower values of reflectivity (i.e., at higher values of $-\log A$), since the curves are steeper, the errors in conductivity are reduced. Clearly, spurious reflections, especially at high values of sample reflectivity, must be reduced to a very low value in order to obtain acceptable errors in conductivity measurements.

We have measured the VSWR of all microwave components available in our laboratory to use in our bridge and have selected those with the smallest spurious reflections. In addition, in redesigning the bridge circuitry, we have adopted the philosophy of using the minimum number of microwave components. To this end, the directional coupler in the sample arm which served to measure the incident signal has been eliminated. In its place a waveguide switch (Hewlett-Packard X930A) has been substituted. The incident power is now determined by measuring the signal reflected from the switch in the short-circuit position. In addition, to further reduce unnecessary components, the magic tee is positioned directly above the sample chamber with no bends between it and the sample.

The microwave circuit now employed is shown in Fig. 1. The VSWR of the various important components of the bridge are as follows:

Matched load: 1.006

Magic tee: between 1.09 and 1.12 for the various arms

Input isolator: 1.09

Output isolator: 1.07

Waveguide switch (open): 1.02.

The cross coupling of the magic tee between the E-plane and H-plane arms is -45 dB.

Since the spurious reflected signals and the cross coupling could result in errors larger than desirable, tuners were constructed and included in the microwave circuit as shown in Fig. 1. These tuners consist of two screws (provided with lock nuts) which penetrate the center of the broad face of a length of waveguide and are separated by one-fourth of a guide wavelength at the operating frequency. By adjusting the penetration of these two screws, and possibly by moving this tuner forward or backward one-quarter of a wavelength with respect to the reflection being tuned out, it is possible to cancel any spurious reflected signal.

The tuning procedure is as follows. The crystal arm (consisting of tuner No. 1, the isolator, the transmission cavity and the crystal detector) is attached to a slotted line, and tuner No. 1 is adjusted to minimize the VSWR looking into the arm. The VSWR obtained was 1.007. Next, with the crystal arm in place and a matched load terminating the sample chamber, tuner No. 2 in the reference arm is adjusted to minimize the signal in the crystal arm. After adjusting tuner No. 2, the coupling to the signal arm was down more than 70 dB. Next, with a sliding short terminating the sample chamber, tuner No. 3 is adjusted to minimize the variation of the microwave signal in the crystal arm as the phase of the reflection in the sample arm is varied. The function of this tuner is to cancel components of the incident signal which are derived from the signal reflected from the short (or from the sample during an actual measurement), which are reflected a second time from the various components. These spurious components of the incident signal will, of course, depend on the magnitude of the signal reflected from the sample, and therefore

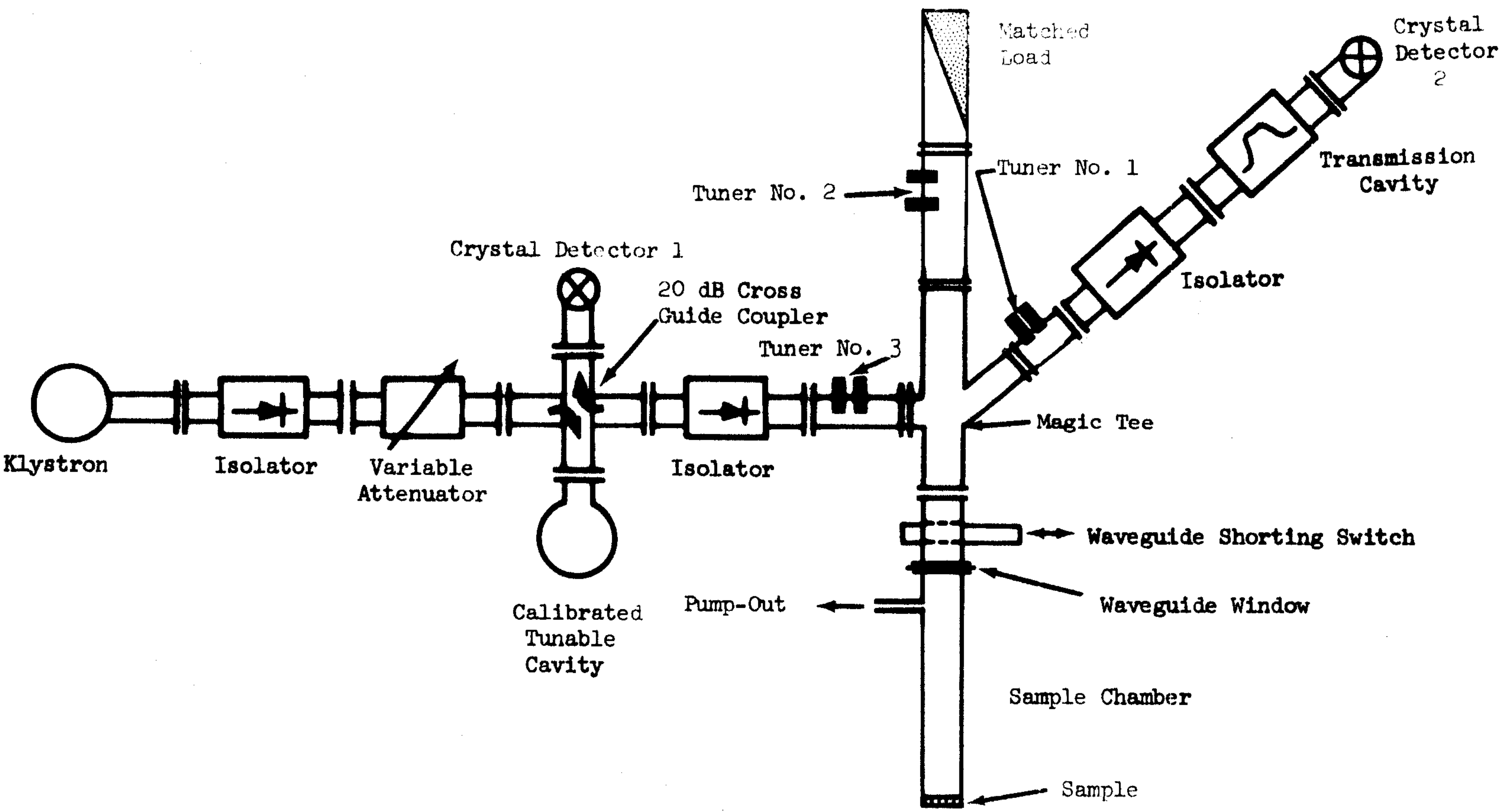

Fig. 1--Microwave circuit for conductivity measurements

this last tuning can be expected to be perfect at only one level of the reflected signal. Since, as discussed previously, the maximum error in conductivity occurs when the reflectivity of the sample is a maximum, we make this tuning with 100 percent reflection from the sample arm. Variation of the microwave voltage in the crystal arm with variation of the sliding short was not detectable after tuner No. 3 was adjusted, and therefore was less than 0.2 percent. With a sliding mismatch which attenuated the reflected signal by 9 dB, this variation was 4.6 percent. The maximum uncertainty in conductivity from spurious reflections has therefore been reduced by this tuning procedure to below 4 percent for values of $-\log A > 1$, an acceptable value.

2.3 EXPERIMENTS PLANNED

The first experiment to be performed will be an investigation of the change of lifetime on irradiation and annealing of ultrapure silicon. Irradiations will be performed at liquid helium temperature, and the change of lifetime due to annealing will be studied up to 400°K. A sample of n-type silicon with a room-temperature conductivity of $\sim 10^{-4}$ $(\text{ohm-cm})^{-1}$ and a carrier lifetime of 7 msec has been prepared for mounting in the microwave sample chamber and will be investigated first. We also have a boule of ultrapure p-type silicon which will be studied.

3. MICROWAVE RESONANCE EXPERIMENTS

The main emphasis in the microwave resonance studies has been in preparing for future experiments, in particular the $80^{\circ}K$ irradiations, the transfer of samples at $80^{\circ}K$, and the subsequent step annealing to room temperature. This involved a modification of the present cavity in addition to the incorporation of a heater for the annealing experiments. These experiments will be to measure the indirect production of the divacancy in p-type silicon and the vacancy-phosphorus complex in n-type silicon. In the report written on work done previously under this contract[1] the energy dependence of the introduction rates at $300^{\circ}K$ was reported and will be used for comparison with the $80^{\circ}K$ irradiations and subsequent $300^{\circ}K$ anneals.

Two pulled 0.1 ohm-cm B-doped samples have been irradiated at $80^{\circ}K$ with 2×10^{17} 30 MeV electrons/cm^2. The number of divacancies formed will be ascertained initially without allowing the sample to exceed $80^{\circ}K$ in temperature. Then annealing experiments to $300^{\circ}K$ will be performed to find in what temperature regions additional divacancies form, presumably reflecting the temperatures at which the vacancy moves in p-type silicon. This experiment is now under way.

In addition, a major goal of the program is to correlate the lifetime and resonance results. Material similar to that used in the lifetime experiments on n-type silicon, i.e., 10 ohm-cm P-doped, has been irradiated to flux levels between 10^{15} and 10^{17} electrons/cm^2. No resonances have been observed. Resonances were observed in more heavily doped material, but none can be correlated as yet with the recombination center.

The recombination center in n-type silicon (to be called the X-center) is at an energy of more than 0.40 eV from the conduction band. Its introduction rate is large (~ 1 cm^{-1}) and its charge states are -1 and 0. As pointed out above, a difference in the resonance and recombination studies has been that the experiments are performed on dissimilar material. In 10 ohm-cm P-doped silicon there are about 3×10^{14} P/cm^3, and, with our sample sizes, this means about 10^{14} P donors. The X-center may remove an electron from the conduction band, but even if all the 10^{14} electrons populated the X-centers and were observable with resonance techniques, they would be difficult to observe in our experiments because 10^{14} spins can be seen only under the most ideal conditions. One way to circumvent this is to use larger samples, essentially building samples which act as one wall of the cavity and can increase the signal by a factor of 5 or 10. This will be tried in the near future.

There are other questions which arise due to the properties of the X-center, and these will be discussed on the basis that the original donor concentration is not a significant parameter.

With the Fermi level above the A-center, the X-center responsible for the recombination is charged negatively. It is introduced at ~ 5 times the rate of the A-center, but only the A center is seen under these conditions in the resonance experiments. It cannot be said that it is not present in sufficient quantities, unless the introduction rate changes with flux, for instance at 10^{14} electron/cm^2, becoming much lower (say 2 orders of magnitude). However, there seems to be no good reason for the introduction rate to change when the density of radiation-induced centers is small and when the Fermi level is far above 0.40 eV.

Another possibility is that the g-value of the center may be quite different from 2. This is possible, but up to now all centers in silicon are near $g = 2$, and it is very difficult to understand why the spin-orbit coupling, which determines deviation of g-value from 2, should be very different for the X-center.

The electron-spin lattice relaxation time may be different. Relaxation times are long for all centers in silicon so far studied, and possibly this center has not been seen because of unusual passage conditions. However, the X-center is apparently present in high concentration and many samples have been studied under many conditions of passage. Therefore, it is surprising that it has not been seen. One other possibility is that this center has a very broad line width which has not been noticed. Most of our measurements have concentrated on looking for centers with a width of a few gauss, but even using field modulation amplitudes of only a gauss or two, we probably would have seen a center present in high concentration even if it were 100 gauss wide.

Thus at this time there is a question as to why the resonance experiments and the recombination experiments do not show a positive correlation.