International Conference on Radiation Effects and Semiconductors, Albany, New York

Aug. 24-27, 1970

COO-1198-744

MASTER

Conf-700824-1

Electron Irradiation Effects in Silicon at Liquid Helium Temperatures Using AC Hopping Conductivity*

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ABSTRACT

Changes in ac hopping conductivity with electron irradiation have been measured in n-type silicon, p-type silicon, and high purity silicon. Irradiations were carried out at both 4.8 and 1.6 degrees kelvin, with measurements made at reference temperatures of 4.2 and 1.3 degrees kelvin, respectively. In the p-type crystals, the changes in ac hopping conductivity depended strongly on the concentration of chemical acceptors, indicating a concentration dependence on impurities in the defect production rate. The production rates at 1.6°K were generally very similar to those for a 4.8°K irradiation. No significant thermal annealing stages below room temperature were observed in any of the crystals. A small amount of "reverse annealing," i.e. an increase in ac hopping conductivity was observed below 30°K. No radiation annealing effects due to a beam of either .500 Mev or .350 Mev electrons were observed, either at 4.5°K or at 1.45°K.

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*This work was supported in part by the U. S. Atomic Energy Commission under Contract AT(11-1)-1198.

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INTRODUCTION

The measurement of ac hopping conductivity requires compensating minority impurities (or defects) in a crystal. Conduction takes place by hopping of electrons from occupied to unoccupied localized donor states (for an n-type crystal). The model is that of an electron tunneling from an occupied donor site to an unoccupied site in the presence of the coulomb fields arising from compensating acceptors and ionized donors. M. Pollak[1/] and co-workers found experimentally that the ac hopping conductivity (σ_{ac}) is approximately given by:

$$\sigma_{ac} = K\,\omega^{0.8}\,N_{min}^{\;0.85}$$

where K is a constant, ω is the frequency of the ac signal, and N_{min} is the concentration of compensating minority impurities. At the low temperature end (below ~ 20° K for silicon), the conductivity is roughly proportional to the minority impurity concentration and is almost independent of the majority impurity concentration.

EXPERIMENTAL

The ac hopping conductivity was determined by measuring the dielectric loss (or dissipation factor) of the samples using a General Radio model 1615-A capacitance bridge with a PAR model HR8 lock-in amplifier used as a null detector. The bridge is capable of 6-figure resolution in capacitance and 4-figure resolution in dissipation factor. Measurements were made at frequencies from 0.1 KHZ to 100 KHZ. Samples were typically 1.0×10^{-2} cm to 1.5×10^{-2} cm thick and had an area from 1.0 to 1.5 cm^2. Sample electrodes were 1/4 inch diameter of either evaporated gold or silver. The temperatures of the samples during irradiation ranged from 1.6° K to 5.2° K and were measured with an Allen-Bradley carbon resistance thermometer mounted next to the samples. Below 4.2° K the carbon resistor was calibrated against the vapor pressure above the liquid helium bath. Beam currents ranged from 2.0×10^{-8} to 1.0×10^{-7} $amps/cm^2$.

To decide whether the changes observed are to be associated with changes in surface conductivity, four samples with electrodes placed a centimeter apart on the bombarded surface were measured. Two intrinsic, one n-type, and one p-type specimen were used. With various energies from 0.300 to 2.0 Mev and a total fluence up to 1.4×10^{16} e^-/cm^2, changes in the surface conductivity were too small to measure. This puts an upper limit on surface counductance of 12.5×10^{-15} $(ohm\text{-}cm)^{-1}$ per square of surface area. Samples typically had only 5 to 10 squares of surface area between front and back electrodes.

RESULTS

The changes in σ_{ac} with electron fluence for p-type, aluminum doped silicon are illustrated in Figures 1 and 2 and for p-type, boron doped silicon in Figure 3. The changes are fairly linear with fluence, at least up to 5.0×10^{15} e^-/cm^2. The notable thing about the p-type samples is that the changes in σ_{ac} for a given electron fluence depend quite strongly on the concentration of impurities in the crystal. The changes in n-type, phosphorous doped silicon are shown in Figure 4. The magnitudes of σ_{ac} in the n-type sample were much smaller than those typically obtained in p-type crystals of similar impurity concentration. In high purity silicon, small initial changes in σ_{ac} were observed, which quickly saturated with electron flux as shown in Figure 5. After irradiation, samples typically showed a frequency dependence of the form $\sigma_{ac} \sim \omega^s$, where s ranged in value from 0.81 to 0.95. This compares with the value of $s = 0.79$ observed by Pollak and Geballe[1/] for crystals compensated with chemical impurities.

Since Watkins'[2/] data suggests that interstitials are mobile even at 4.2°K, irradiations were also performed holding the sample temperature well below 2.0° K to see if there would be any significant difference in production rates between a 1.6° K irradiation and a 5.0° K irradiation. The results for the various crystals are shown in Figures 6-8. The changes in σ_{ac} for a 1.6° K irradiation are similar to those observed in a 5.0° K irradiation. It should also be noted that for an irradiation made at 1.6° K and measurements made at 1.3° K, p-type crystals doped with either aluminum or boron of similar concentration yield similar values of σ_{ac} for a given electron fluence. This indicates that the large changes in σ_{ac} observed in a sample doped with 1.0×10^{17} boron/cm^3 are due to the concentration dependence and not due to the type of impurity. Measurements were also taken as the samples were allowed to slowly warm up from 1.3° K to 4.2° K. A rise in σ_{ac} from 1.3 to 4.2° K is expected because the tunneling rate should be temperature dependent. Measurements were then again taken at 1.3° K to see if any changes ("annealing") took place between 1.3° K and 4.2° K (see Figure 9). No significant changes were observed in the p-type specimens but there was a small change in the n-type crystal. Radiation annealing effects due to a .500 Mev electron beam following damage introduced by a 1.500 Mev beam were not observed in any of the samples, neither at 5.8°K, 5.0°K, or at 1.45°K. No annealing stages is isochronal (ten minute) anneals up to room temperature following irradiation were observed in any of the samples. A small increase in σ_{ac} on warming to 20 - 30°K was usually observed.

DISCUSSION

In p-type silicon, one expects the interstitial to be positively charged and the vacancy to be neutral when the Fermi level is pinned to the acceptor level at these low temperatures. Therefore, the interstitials (silicon or group three impurity) will act as compensating minority impurities in a p-type lattice and one expects σ_{ac} to be proportional to their concentration. Furthermore the vacancies should not contribute to the hopping conductivity in this situation.

In n-type silicon, one expects the interstitial to be neutral (or positive), whereas the vacancy probably has a double negative charge. In this case the vacancies will be acting as the minority impurity and one expects σ_{ac} to be proportional to their concentration but independent of the interstitual concentration.

We have estimated the production rate of defects from the changes observed in σ_{ac}, assuming singly charged defects, and the results are summarized in Table I, for an electron energy of 1.50 Mev. These values are only rough estimates, good to ± 50%.

TABLE I

Crystal Type	Dopant	Growth Method	Resistivity (ohm-cm)	Dopant Concentration (cm^{-3})	Defect Production Rate (cm^{-1})
P	Aluminum	Float Zone	4.0	3.3×10^{15}	0.03
P	Aluminum	Pulled	0.40	5.0×10^{16}	1.0
P	Boron	Float Zone	0.60	3.0×10^{16}	1.0
P	Boron	Float Zone	0.27	1.0×10^{17}	10.0
N	Phosphorous	Float Zone	0.54	1.0×10^{16}	0.07

Note that for a crystal doped with $1.0 \times 10^{17}/cm^3$ boron, the production rate is 10.0 cm^{-1}, assuming singly charged defects, or 5.0 cm^{-1}, assuming doubly charged defects. The value of 5.0 cm^{-1} is very close to the theoretically calculated production rate.

In summary the major results are that: (1) there is no significant thermal annealing below room temperature; (2) there is no radiation annealing observed at liquid helium temperatures; (3) the production rates at 1.6°K are essentially the same as at 5.0°K (within a factor of two); (4) the production rates seem to depend on impurity concentration in p-type crystals. These results serve to confirm the suggestion of Watkins that the interstitial is mobile at liquid helium temperatures and is either trapped at impurities or interchanges with them in a p-type lattice, creating impurity interstitials which migrate only above room temperature.

The measurement technique employed is also capable of monitoring, for the first time, the small changes produced in non-degenerate n-type silicon during a liquid helium temperature irradiation. The data suggests that during the irradiation most of the interstitial-vacancy pairs recombine in n-type silicon. A few interstitials escape to be trapped at impurities, thus accounting for the small changes observed.

REFERENCES

1. M. Pollak and T. H. Geballe, Phys. Rev. 122, 1742 (1961), also Phys. Rev. 133, A564 (1964).
2. G. D. Watkins, in Radiation Damage in Semiconductors (Dunod Cie., Paris, 1965), p. 104.

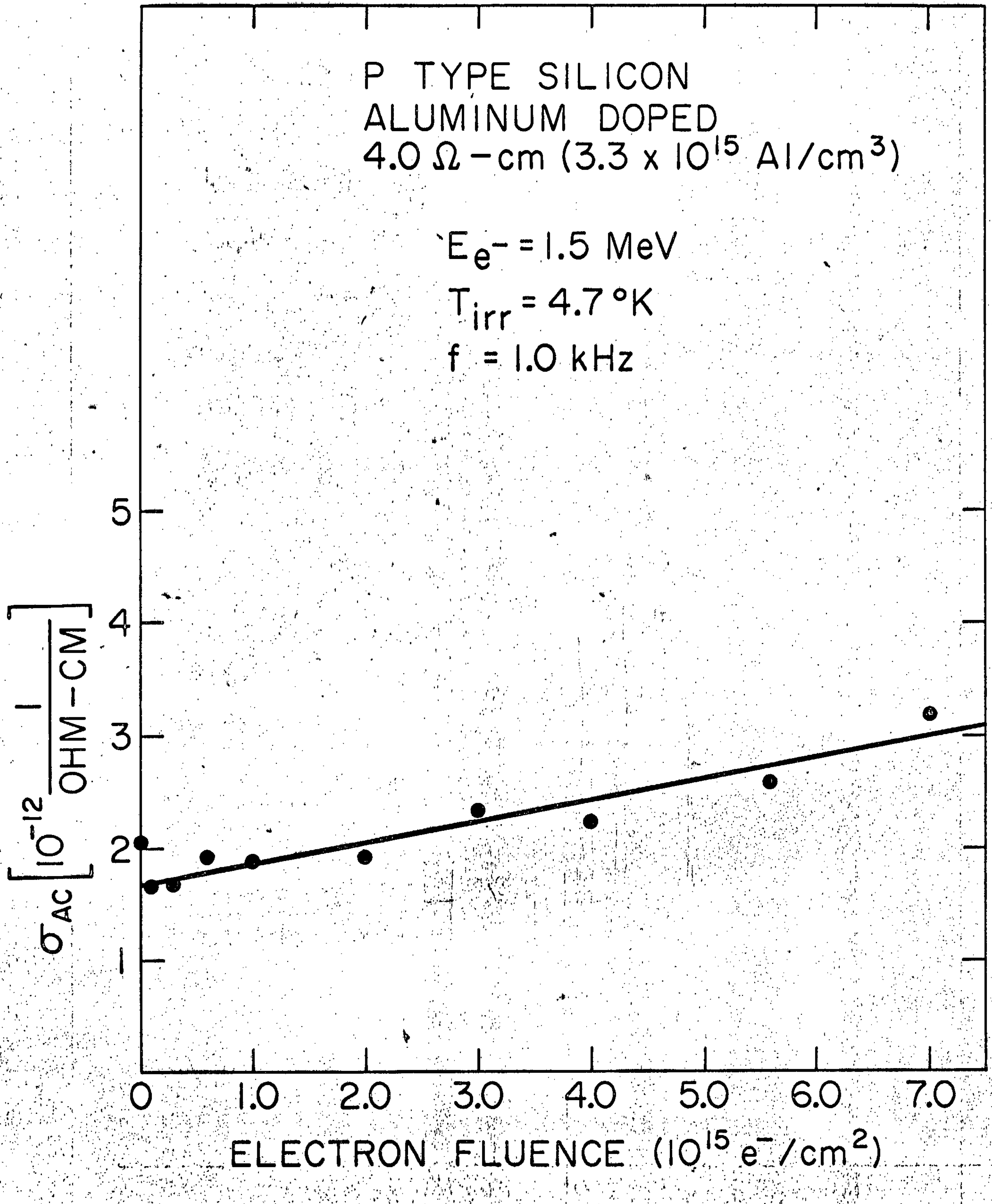
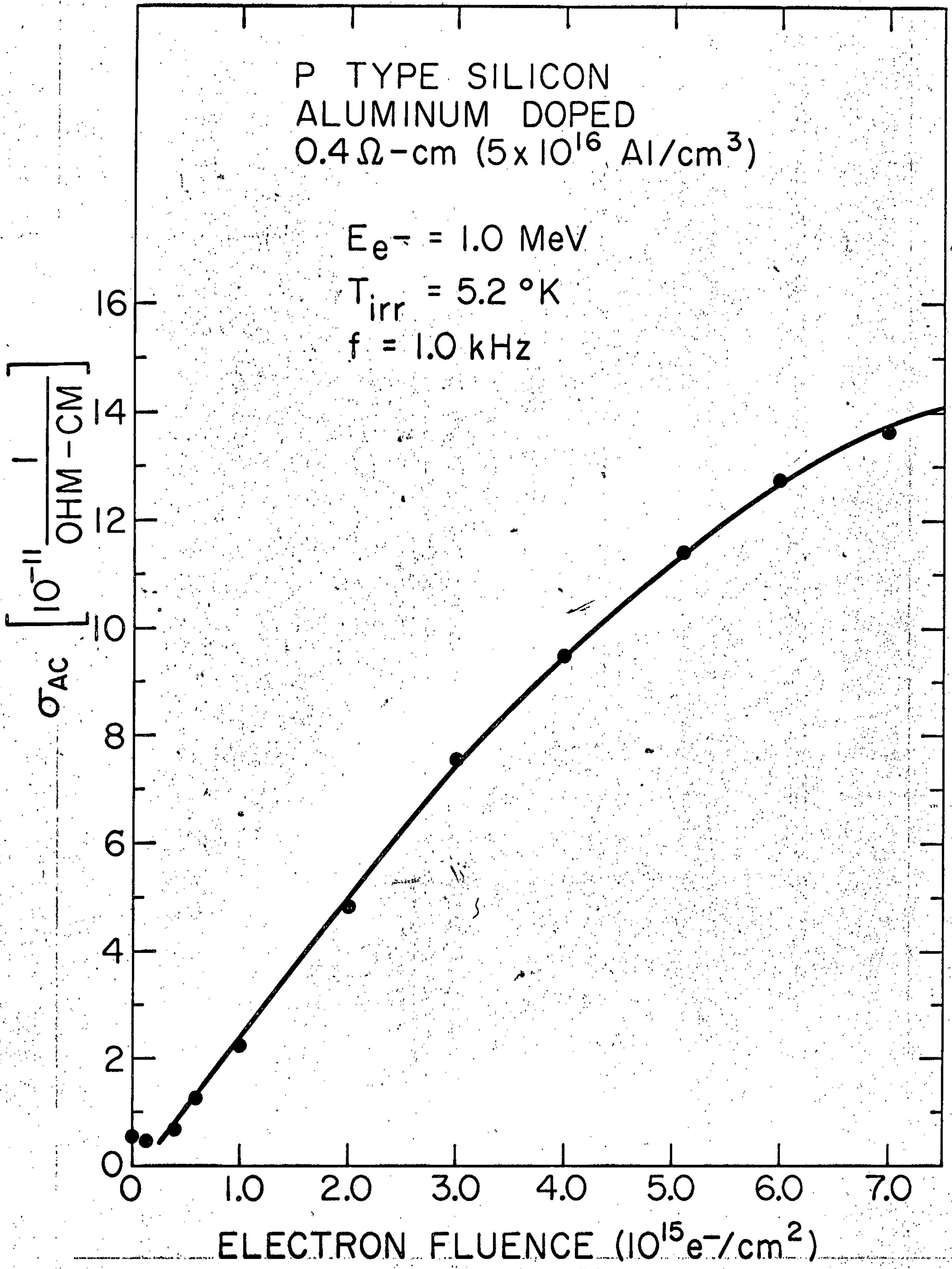

P TYPE SILICON
ALUMINUM DOPED
4.0 Ω – cm (3.3 x 10^15 Al/cm^3)
E_e- = 1.5 MeV
T_irr = 4.7 °K
f = 1.0 kHz
σ_AC [10^-12 1/OHM-CM]
1
2
3
4
5
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
ELECTRON FLUENCE (10^15 e^-/cm^2)

Fig. 1



P TYPE SILICON
ALUMINUM DOPED
0.4 Ω-cm (5 x 10^16 Al/cm^3)
E_e- = 1.0 MeV
T_irr = 5.2 °K
f = 1.0 kHz
σ_AC [10^-11 1/OHM-CM]
0
2
4
6
8
10
12
14
16
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
ELECTRON FLUENCE (10^15 e-/cm^2)

Fig. 2

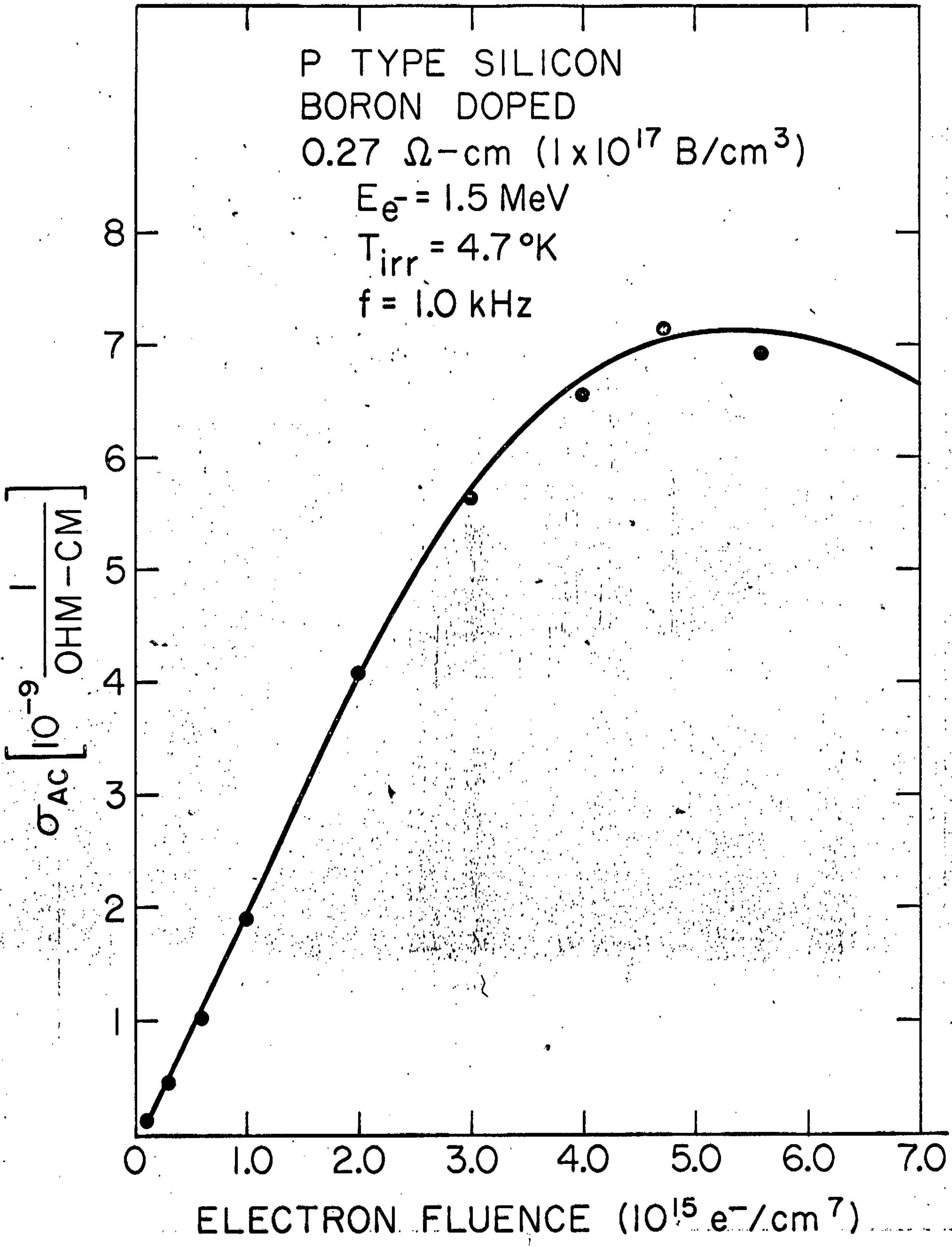

P TYPE SILICON
BORON DOPED
0.27 Ω-cm (1x10^{17} B/cm^{3})
E_{e^-} = 1.5 MeV
T_{irr} = 4.7 °K
f = 1.0 kHz
$\sigma_{AC}\left[10^{-9}\frac{1}{\text{OHM-CM}}\right]$
1
2
3
4
5
6
7
8
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
ELECTRON FLUENCE (10^{15} e^{-}/cm^{7})

Fig. 3

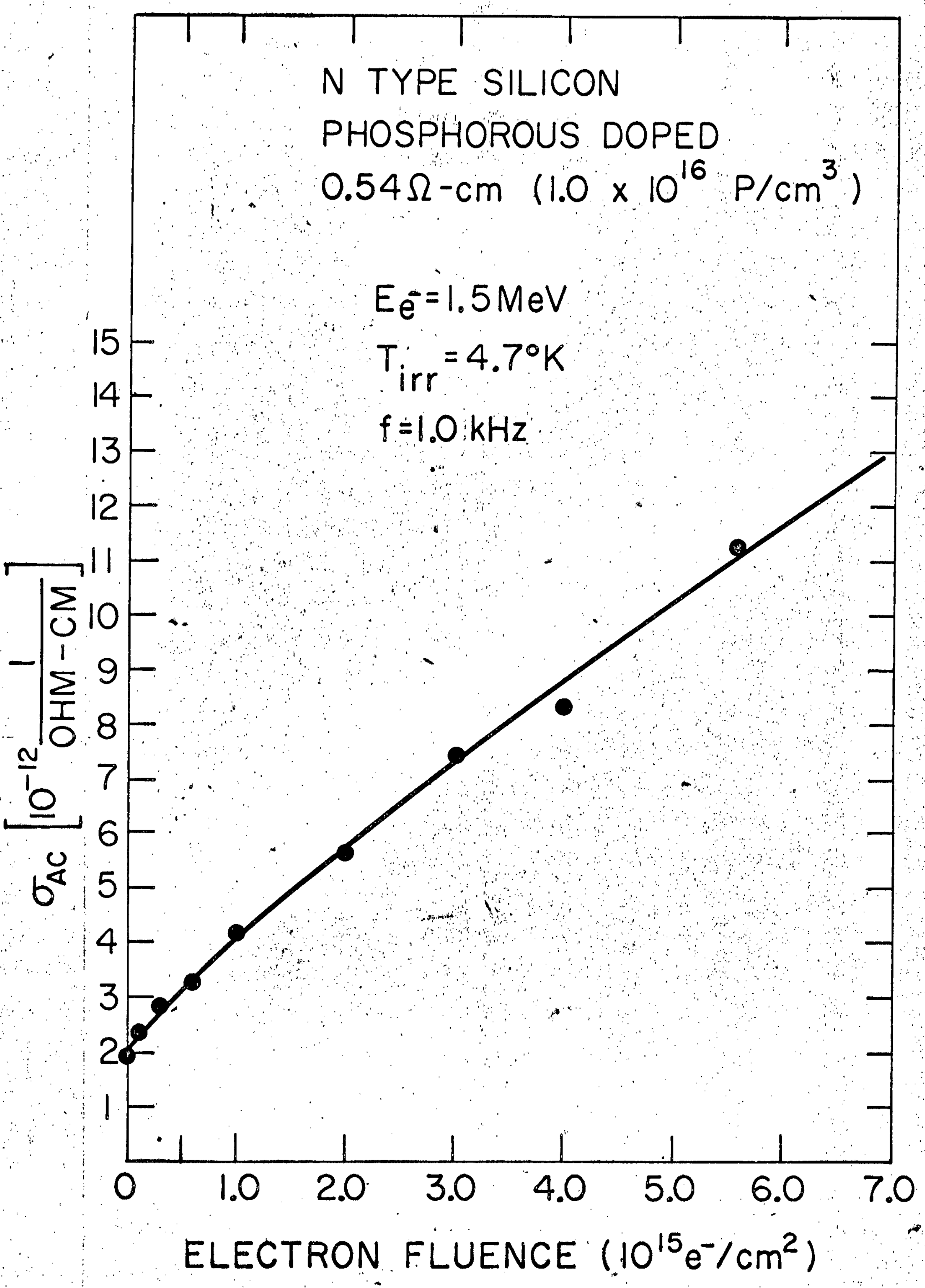

N TYPE SILICON
PHOSPHOROUS DOPED
0.54Ω-cm $(1.0 \times 10^{16}\ P/cm^3)$
$E_{e^-} = 1.5$ MeV
$T_{irr} = 4.7°K$
f = 1.0 kHz
$\sigma_{AC}\ \left[10^{-12}\ \frac{1}{OHM-CM}\right]$
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
ELECTRON FLUENCE $(10^{15} e^-/cm^2)$

Fig. 4

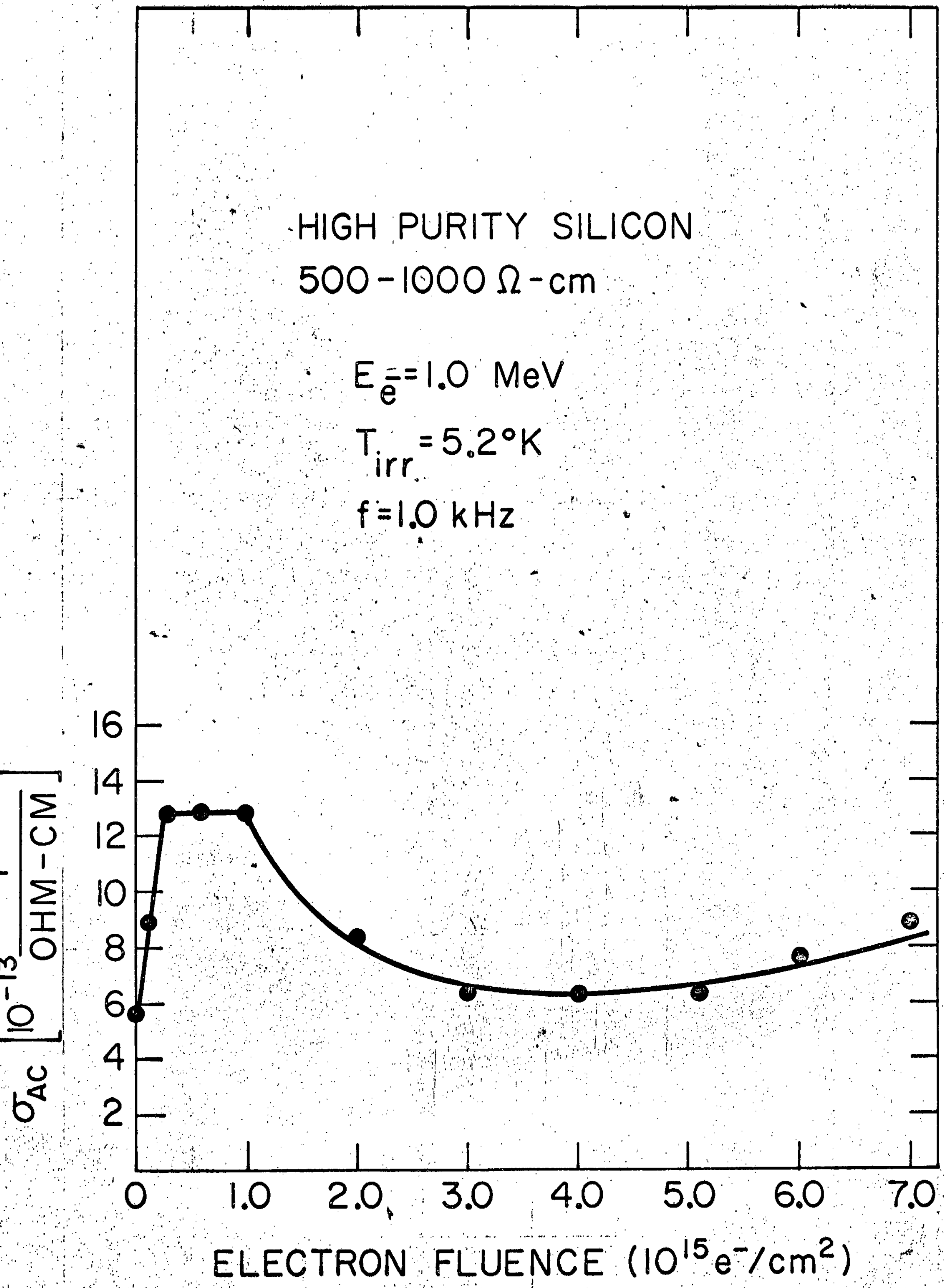
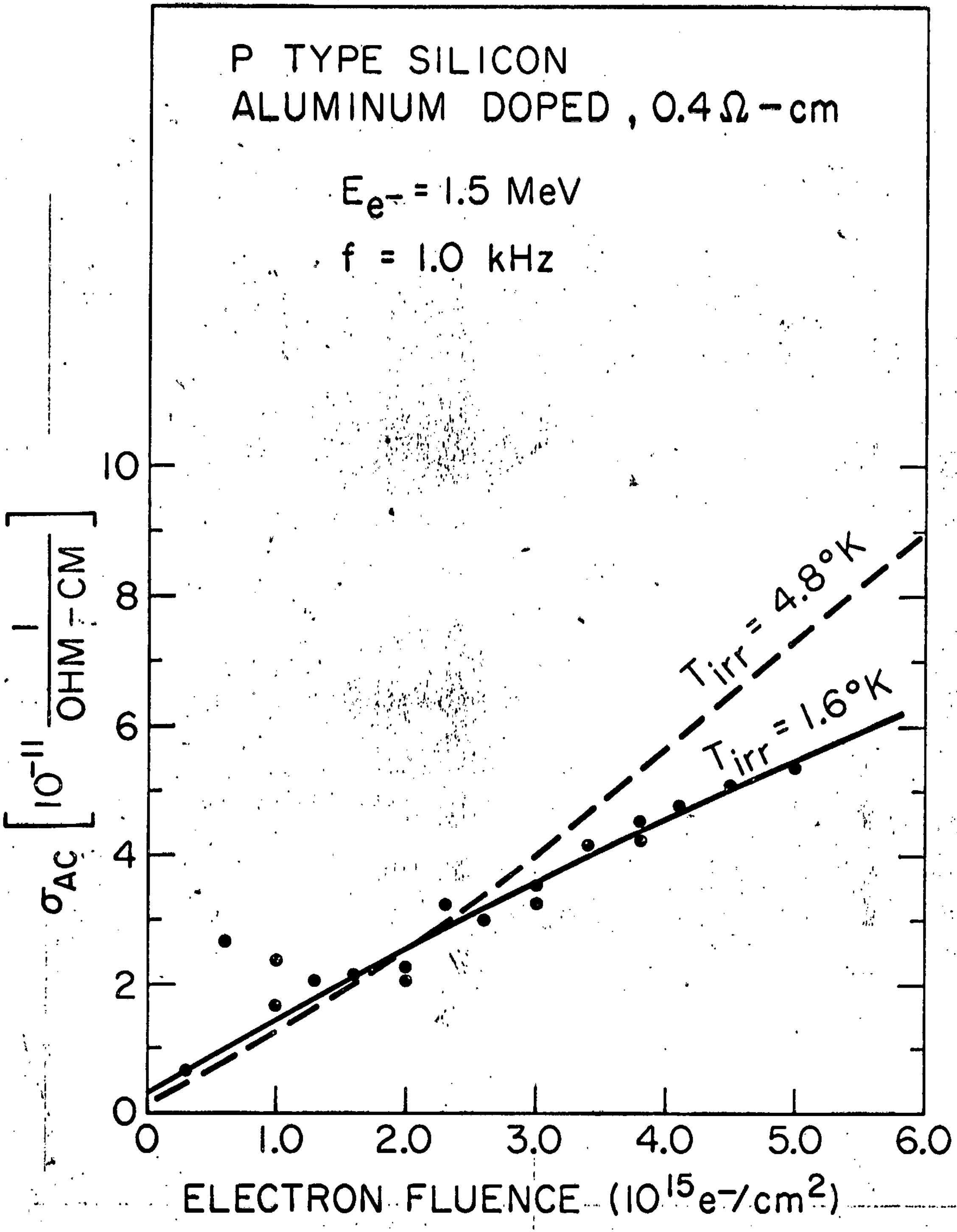

HIGH PURITY SILICON
500-1000 Ω-cm
E_{e^-}=1.0 MeV
T_{irr}=5.2°K
f=1.0 kHz
σ_{AC} $\left[10^{-13}\frac{1}{\text{OHM-CM}}\right]$
16
14
12
10
8
6
4
2
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
ELECTRON FLUENCE (10^{15} e⁻/cm²)

Fig. 5



P TYPE SILICON
ALUMINUM DOPED, 0.4Ω-cm
E_{e^-} = 1.5 MeV
f = 1.0 kHz
T_{irr} = 4.8°K
T_{irr} = 1.6°K
$\sigma_{AC}\left[10^{-11}\frac{1}{\text{OHM-CM}}\right]$
0
2
4
6
8
10
0
1.0
2.0
3.0
4.0
5.0
6.0
ELECTRON FLUENCE (10^{15}e⁻/cm²)

Fig. 6

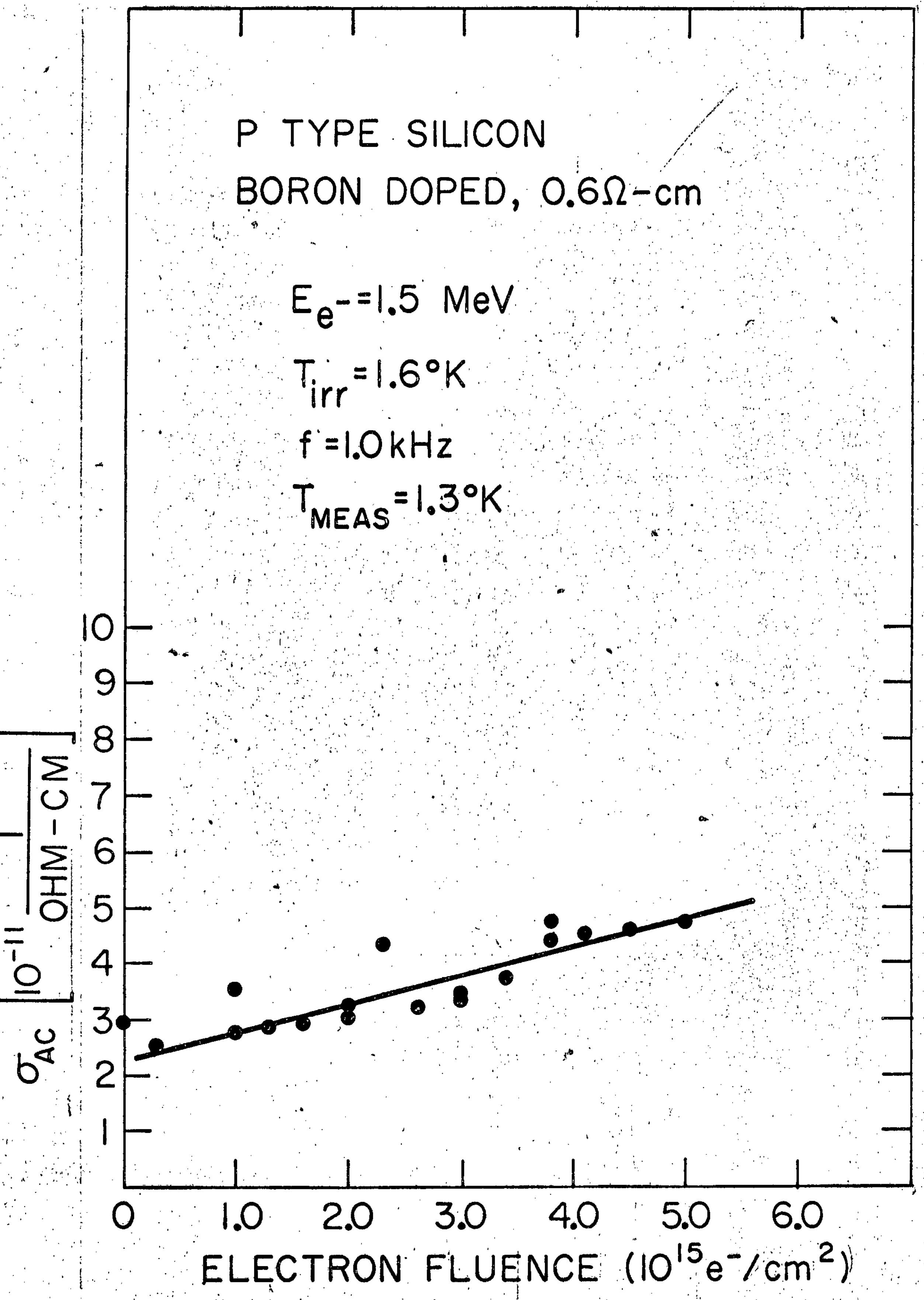

P TYPE SILICON
BORON DOPED, 0.6Ω-cm
E_e- =1.5 MeV
T_irr=1.6°K
f =1.0 kHz
T_MEAS=1.3°K
10
9
8
7
6
5
4
3
2
1
σ_AC [10^-11 1/OHM-CM]
0
1.0
2.0
3.0
4.0
5.0
6.0
ELECTRON FLUENCE (10^15 e^-/cm^2)

Fig. 7

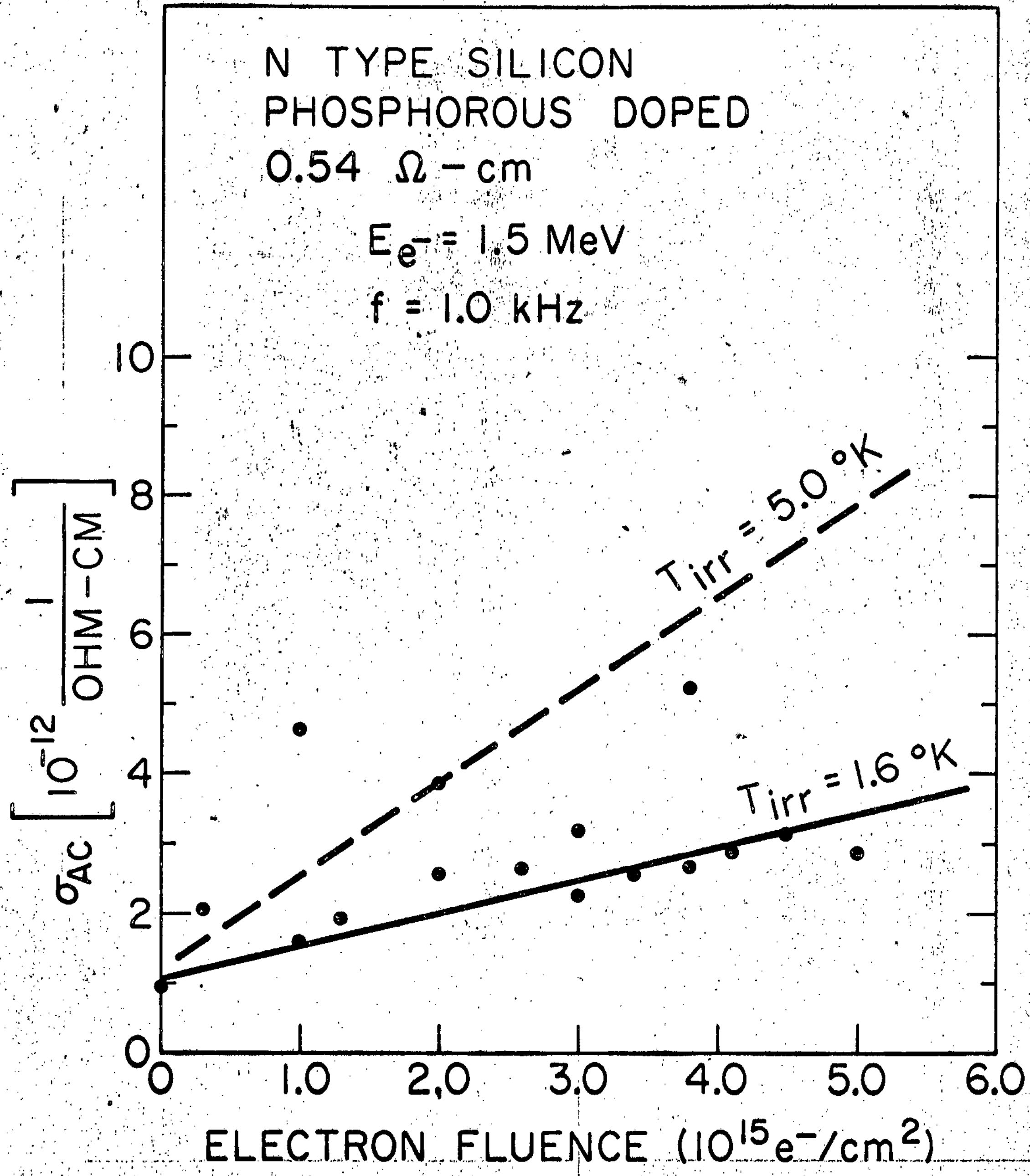
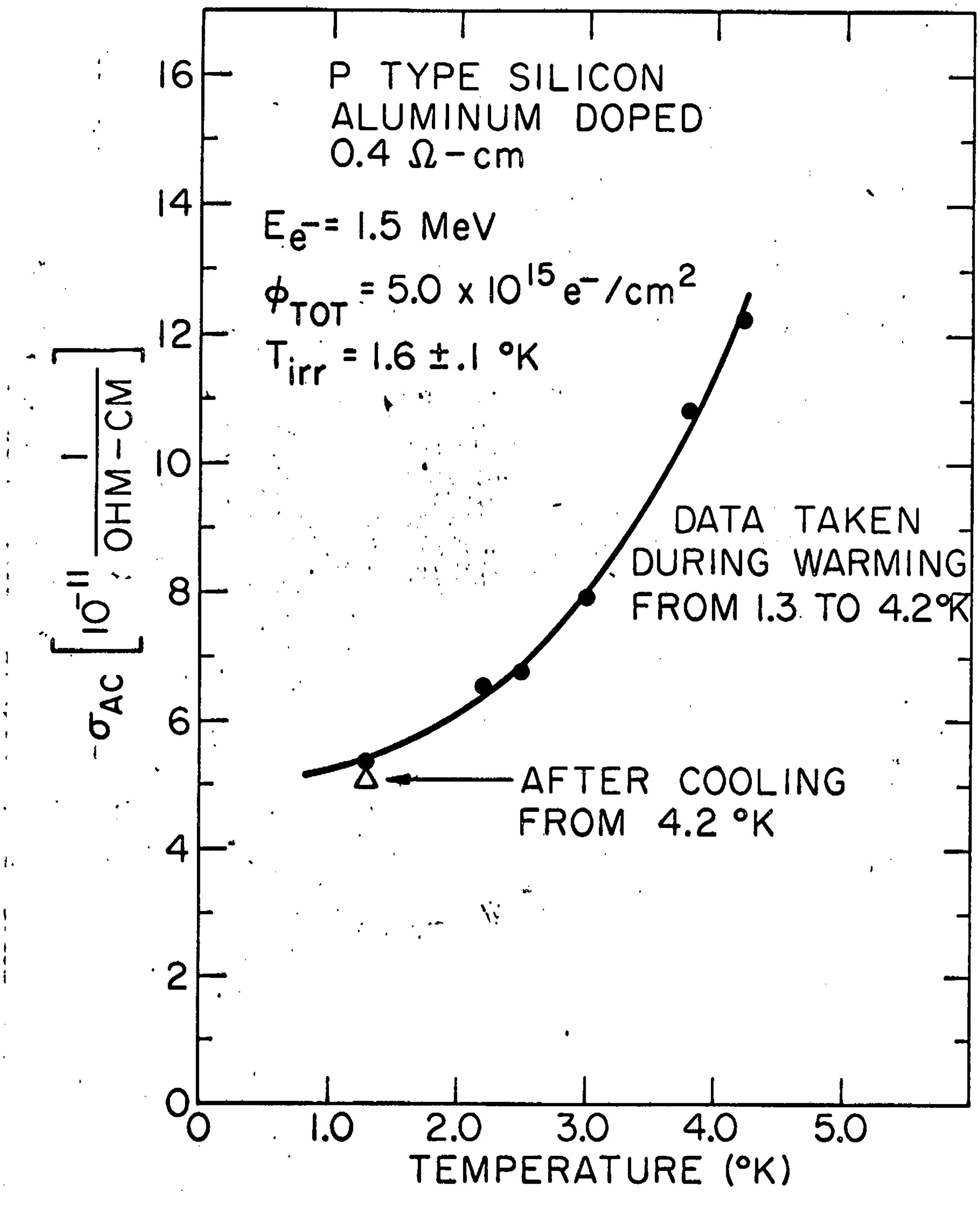

N TYPE SILICON
PHOSPHOROUS DOPED
0.54 Ω - cm
E_{e^-} = 1.5 MeV
f = 1.0 kHz
T_{irr} = 5.0 °K
T_{irr} = 1.6 °K
σ_{AC} $\left[10^{-12} \frac{1}{\text{OHM-CM}}\right]$
0
2
4
6
8
10
0
1.0
2.0
3.0
4.0
5.0
6.0
ELECTRON FLUENCE (10^{15} e^-/cm^2)

Fig. 8



P TYPE SILICON
ALUMINUM DOPED
0.4 Ω-cm
$E_{e^-} = 1.5$ MeV
$\phi_{TOT} = 5.0 \times 10^{15} e^-/cm^2$
$T_{irr} = 1.6 \pm .1$ °K
DATA TAKEN DURING WARMING FROM 1.3 TO 4.2°K
AFTER COOLING FROM 4.2 °K
$-\sigma_{AC} \left[10^{-11} \frac{1}{\text{OHM-CM}}\right]$
0
2
4
6
8
10
12
14
16
0
1.0
2.0
3.0
4.0
5.0
TEMPERATURE (°K)

Fig. 9