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RESISTIVITY AND CARRIER LIFETIME IN GOLD-DOPED SILICON

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Electronic Technology Division
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National Bureau of Standards
Washington, D. C. 20234

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This report describes the present status of an on-going study of the properties of gold-doped silicon. Resistivity, Hall effect, and carrier lifetime are being measured at room temperature in silicon wafers doped with varying amounts of gold and either phosphorus or boron. Reasons are being sought for the apparent discrepancy between total and electrically active gold, for the discrepancy between calculated and observed resistivity in both *n*-type and *p*-type specimens with very large gold concentration, and for the diversity in capture cross section data reported in the literature. Although many questions still remain, progress has been made in resolving these discrepancies. There is considerable evidence that the low resistivity observed at large gold concentrations is associated with the introduction of shallow acceptor states in concentrations strongly dependent on the gold concentration. The origin of these states is being sought. Initial studies of the application of the surface photovoltage method to the measurement of carrier lifetime in gold-doped silicon have been completed. This method also appears to be suitable for use on gallium arsenide specimens. A bibliography of the literature on properties of gold-doped silicon that contains 136 entries is also included as an appendix.

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FOREWORD

The work reported here is a part of the Joint Program on Methods of Measurement for Semiconductor Materials, Process Control, and Devices. This program is carried out at the National Bureau of Standards; it receives support from a number of other agencies of the Federal Government in addition to the NBS. The program was undertaken in 1968 to focus NBS efforts to enhance the performance, interchangeability, and reliability of discrete semiconductor devices and integrated circuits through improvements in methods of measurement for use in specifying materials and devices and in control of device fabrication processes. These improvements are intended to lead to a set of measurement methods which have been carefully evaluated for technical adequacy, which are acceptable to both users and suppliers, which can provide a common basis for the purchase specifications of government agencies, and which will lead to greater economy in government procurement. In addition, such methods will provide a basis for controlled improvements in essential device characteristics, such as uniformity of response to radiation effects. During the period 10 May to 31 December 1971 the portion of the program concerned with gold-doped silicon received partial support from the Air Force Cambridge Research Laboratories (LQD) under project order Y-71-906. The contract monitor was D. E. Davies.

Technical personnel who contributed to this effort during the period covered were:

- W. M. Bullis, supervisory physicist, principal investigator
- M. Cosman, mechanical engineering technician, specimen preparation
- T. E. Gills, research chemist, neutron activation analysis
- J. Krawczyk, physical science technician, diffusion
- T. F. Leedy, physicist, diffusion
- W. E. Phillips, physicist, surface-photovoltage measurements
- A. W. Stallings, mechanical engineering technician, electrical measurements
- W. R. Thurber, physicist, electrical measurements

Preliminary reports of the work have been reported in joint program quarterly progress reports that have been issued in the NBS Technical Note series. NBS Technical Notes 702 and 717 cover periods during which support was received under the above noted project order. Earlier work is reported in NBS Technical Notes 488, 495, 520, 527, 560, 571, 592 and 598.

RESISTIVITY AND CARRIER LIFETIME IN GOLD-DOPED SILICON

W. Robert Thurber and W. Murray Bullis

INTRODUCTION

Gold is an amphoteric recombination center in silicon. Its addition to either *n*-type or *p*-type silicon will affect the carrier lifetime. No other impurity addition has been found that is as effective for controlling carrier lifetime in silicon; hence, gold is widely used in device fabrication.

The basic electrical model for the impurity states associated with gold was clearly enunciated in early work on gold-doped silicon [1], and the diffusion technology for introducing gold into the silicon lattice, though largely empirical, is under sufficient control that device manufacturers can successfully employ it on a large-scale. Nevertheless, unresolved problems remain in both areas. In particular, clarification of the details of the electrical model is necessary before concentrations of gold centers can be determined unambiguously from measurements of resistivity or carrier lifetime as might be desired, for example, in before-and-after studies of radiation effects in gold-doped silicon.

In the work reported here, reasons are being sought for the apparent discrepancy between total and electrically active gold, for the discrepancy between calculated and observed resistivity in both *n*-type and *p*-type specimens with very large gold concentration, and for the diversity in the capture cross section data reported in the literature. To investigate these problems, a series of silicon wafers to which various amounts of gold and phosphorus or boron have been added is being characterized by measuring resistivity, Hall coefficient, and carrier lifetime at room temperature. Gold concentrations are determined directly by neutron activation analysis after gold has been diffused into the wafer; shallow dopant (phosphorus or boron) concentrations are determined by measurement of wafer resistivity before gold diffusion. In addition, ancillary experiments and computations to develop the diffusion technology necessary to prepare the specimens and to elucidate certain aspects of proposed models are being carried out.

The period covered by this report is 10 May to 31 December 1971. During this period, considerable emphasis has been placed on the establishment of facilities and procedures for measuring the short carrier lifetimes associated with gold-doped silicon. In the continuing study of resistivity and Hall effect, principal effort has been devoted to the study of heavily gold-doped *p*-type silicon. For completeness, some work completed before the beginning of this period is also included. A bibliography of the literature on properties of gold-doped silicon is appended to the report.

SPECIMEN PREPARATION

Both *n*-type and *p*-type crystals are being studied. About ten resistivity values of each type cover the range 0.01 to 2500 Ω·cm as listed in table 1. Wafers are cut from single crystals of silicon with a diamond saw and lapped to a final thickness of about 1.1 mm with 12-μm alumina. The resistivity of representative wafers is measured by the four-probe [2] or van der Pauw [3] method. To test for radial homegeneity of the shallow doping impurities, the resistivity profile is measured by the four-probe method on one or more wafers from each crystal. After completion of the initial resistivity measurements both sides of each wafer are chem-mechanically polished.*

Immediately prior to insertion in the diffusion furnace, the wafers are cleaned and plated with gold. The cleaning procedure consists of the following steps: ultrasonic agitation in detergent for one minute, rinses in distilled water and methanol, ultrasonic agitation in trichloroethylene for one minute, rinse in methanol, ultrasonic agitation in methanol, soaking in chromic acid for at least one minute, rinses in distilled water and methanol, three rinses in nitric acid of one minute each, rinses in distilled water and methanol, soaking in hydrofluoric acid for one minute, and final rinses in distilled water and methanol. A 40-nm thick layer of gold is then evaporated on both sides of the wafer and the wafer is loaded into the quartz tube of the diffusion furnace.

Diffusion takes place in an open-tube system. Most of the diffusions have been done in an atmosphere of high-purity oxygen, which flows through the quartz diffusion tube at a rate of 4×10^{-6} m^3/s. Diffusions are made at temperatures of 850, 950, 1050, 1150, and 1250°C. The times at 1050°C and above are long enough to reach the solid solubility limit of gold in silicon. The diffusion time at 850°C is 12 to 14 days and at 950°C it is 6 to 7 days. Even these long times are not generally sufficient for the gold concentration to reach its solid solubility; more variability in concentration for a given time has been found for diffusions at these temperatures than for diffusions at the higher temperatures. In all cases the gold concentration in the center portion of the wafer is relatively flat [4, 5] so 0.12 mm is removed from each face by lapping before the wafers are analyzed further. In some cases difficulty has also been encountered because of accumulation of gold at the rim of the wafer. Some of the early activation analysis specimens included rim portions and anomalously high gold concentrations were obtained. Subsequent experiments confirmed that this was due to the large concentration of gold near the rims of the wafers. This effect appears to be more significant for diffusions made at temperatures below 1250°C.

Hall bars with four side arms and expanded ends are cut from the lapped wafers for electrical measurements. Aluminum contact pads are

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* In the early parts of the study, this step was omitted in preparing specimens for diffusion. No differences between the diffusions made on lapped wafers and those made on polished wafers have yet been observed.

Table 1 — Silicon Crystals for Gold Diffusion

Initial Resistivity ρ_i ($\Omega \cdot cm$)	Type	Pulled or Float Zoned	Status
0.01	*n*		
0.01	*p*		
0.08	*n*	pulled	available
0.08	*p*	pulled	diffused
0.3	*n*		
0.54	*p*	pulled	diffused
1.2	*n*		
1.2	*p*	pulled	diffused
5.3	*n*	pulled	see Table 2
11	*p*	pulled	see Table 2
20	*n*		
20	*p*	f-z	see Table 2
75	*n*	f-z	see Table 2
93	*p*	f-z	see Table 2
380	*n*	f-z	see Table 2
300	*p*	f-z	diffused
1000	*n*		
1080	*p*	f-z	diffused
2300	*n*	f-z	see Table 2
2000	*p*	pulled	diffused
2400	*p*	f-z	diffused

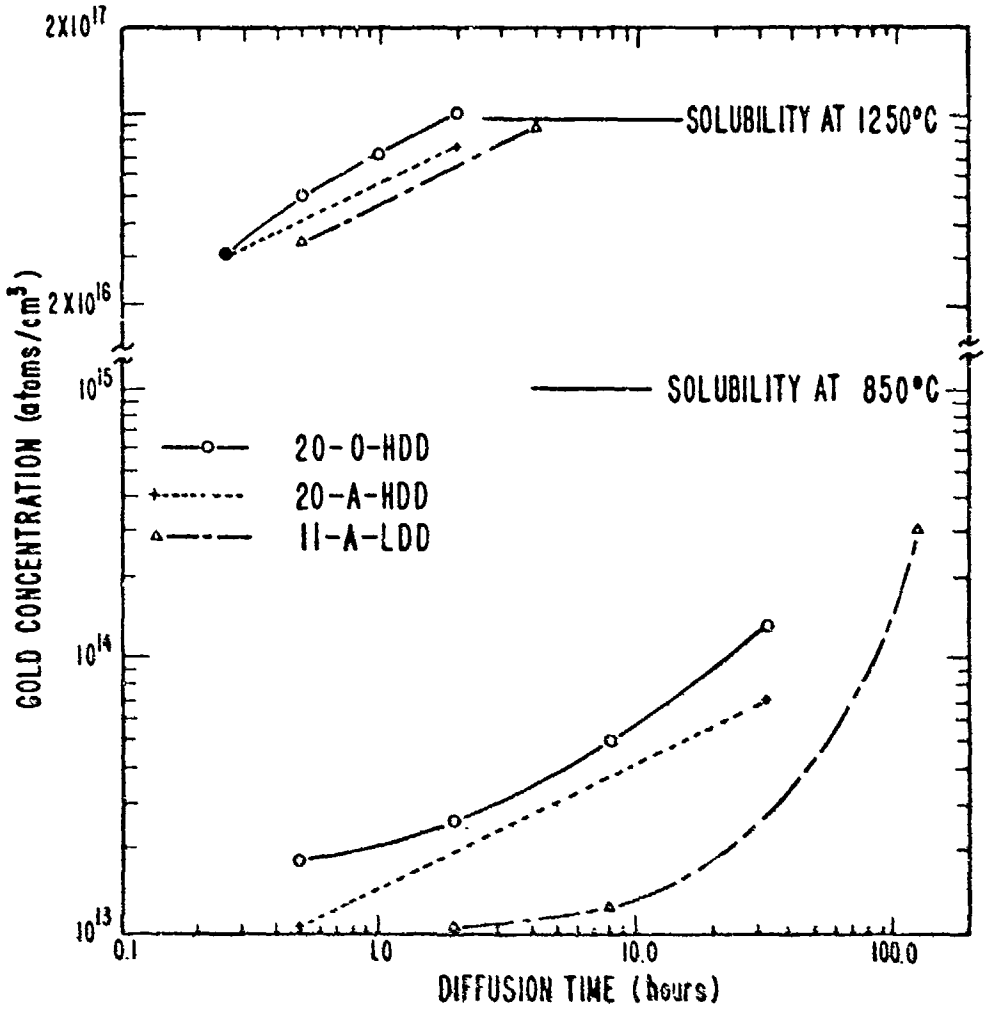


Figure 1. Gold concentration as determined by neutron activation analysis as a function of diffusion time for 11- and 20-Ω·cm, boron-doped, gold-diffused, silicon wafers. The upper curves are for wafers diffused at 1250°C, the lower for wafers diffused at 850°C. The diffusion atmosphere was either oxygen (O) or argon (A). The 11-Ω·cm wafers had low dislocation density (500 cm^{-2}) and are indicated by LDD; the 20-Ω·cm wafers had high dislocation density (17,000 cm^{-2}) and are indicated by HDD. As diffused, the wafers were 1.1 mm thick; before activation analysis, 0.12 mm was lapped from each side and the rim was removed.

evaporated on one surface of the side arms and ends. Hall effect and resistivity measurements are made in a light-tight holder at room temperature (25 ± 1°C). Pressure contact to the aluminum pads is made with flat phosphor-bronze springs.

Pieces from the remainder of the lapped wafer are used to determine the gold concentration by means of neutron activation analysis as described in the next section. The wafer rim is cut away from the pieces before they are analyzed.

During the initial phases of the work, several experiments were conducted to study the effects of atmosphere and dislocation density on the gold diffusion process. Diffusions were carried out in 11- and 20-Ω·cm, boron-doped silicon wafers at 850 and 1250°C in oxygen or argon for various times. The 11-Ω·cm wafers were cut from a pulled crystal with low dislocation density and high oxygen concentration. The 20-Ω·cm wafers were cut from a float-zoned crystal with high dislocation density and low oxygen concentration. The gold concentration in the flat region was determined by neutron activation analysis. The results, shown in figure 1, suggest that gold diffuses slightly faster in an oxygen atmosphere than it does in an argon atmosphere. The effect of a variety of atmospheres has been reported in the literature with conflicting claims as to which is the most suitable one. It was also found that gold diffused faster in the high-dislocation wafers (20 Ω·cm) than it did in the low-dislocation wafers (11 Ω·cm). This result is in agreement with that reported in the literature by others.

ACTIVATION ANALYSIS

The gold concentrations were determined by neutron activation analysis using one of the following procedures. In one procedure, the specimens, along with gold comparator standards, were irradiated at a thermal neutron flux of 2×10^{12} $cm^{-2} \cdot s^{-1}$. The specimens were allowed to sit for two days to allow interfering elements to decay away, after which the specimens and standards were counted using a sodium iodide well-type scintillation detector coupled to a multi-channel analyzer. The gold concentration calculation was based on the 0.142-MeV gamma ray of 2.7-day gold-198.

In the other procedure, the silicon specimens and standard were encapsulated in polyethylene vials and irradiated for 30 min at a neutron flux of 1 to 6×10^{13} $cm^{-2} \cdot s^{-1}$. Copper foils were attached to each specimen and to the standard to normalize for flux variation. The standard consisted of a filter paper approximately the shape and size of the silicon specimens on which was pipetted a known solution of gold in aqua-regia. After irradiation the specimens were allowed to decay for two or three days to minimize short-lived interferences and then counted using a 22 cm^3 lithium-drifted germanium gamma-ray detector in conjunction with a multi-channel analyzer. The results were calculated using the standard-comparator method.

RESISTIVITY AND HALL EFFECT MEASUREMENTS

A summary of the specimens on which resistivity and Hall effect measurements have been completed is given in table 2. The apparent discrepancy between total and electrically active gold is most dramatically observed in *n*-type specimens with initial resistivity lower than 5 Ω·cm. This effect has been well documented in the literature [6]; it has not yet been repeated in the present series. In *p*-type specimens, the increase of resistivity with increasing gold concentration is less abrupt than it is in *n*-type specimens. The results of resistivity measurements on Hall bars cut from 11-, 20-, and 93-Ω·cm *p*-type wafers with varying amounts of gold are shown in figure 2. For comparison, the resistivity calculated from a model for the impurity state in gold-doped silicon is also shown. The calculation is based on a solution to the charge balance equation to find the Fermi level and, hence, the hole concentration. Lattice mobility [7] and impurity mobility [8] were combined reciprocally to obtain the hole mobility used in the calculation of the resistivity. In these calculations the energies of the gold donor and acceptor states were taken as 0.35 eV above the valence band and 0.54 eV below the conduction band, respectively [1], the degeneracy factors were taken as 0.25 for the donor and 1.5 for the acceptor [6], and the energy gap and effective masses were taken from the work of Barber [9]. The above discrepancy can be observed in these results as a displacement of the experimental curves to the right of the theoretical curves by a factor of about 1.5.

There is an even greater discrepancy at the higher gold concentrations. The measured resistivity reaches a maximum and then decreases as the gold concentration increases while the theoretical curves slowly rise to a common limiting value. Hall effect measurements showed that this decrease in resistivity was due to an increase in hole density rather than an increase in carrier mobility. Hall effect measurements made as a function of temperature on several 20-Ω·cm wafers to which different amounts of gold had been added showed that the effect could not be explained by a dependence of the ionization energy of the gold donor state on the gold concentration. Efforts to fit the observation by changing the degeneracy factor of the gold donor state in the model were also unsuccessful.

The possibility that shallow acceptor impurities were being unintentionally introduced during the diffusion was considered. Four 11-Ω·cm *p*-type wafers were diffused at 1250°C for times of 8, 16, 32, and 64 hours. Since the gold reaches its solid solubility concentration in 8 hours, no further changes in gold concentration or in resistivity should occur with increasing diffusion times if gold is the only impurity affecting the electrical properties. Activation analysis results and electrical measurements on the set of four wafers showed that both the gold concentration and the resistivity were essentially the same for all wafers. The result of this experiment therefore indicates that observable amounts of additional shallow acceptor impurities are not being introduced during the high temperature gold diffusions.

Table 2 — Gold-Doped Silicon Specimens

Specimen Number	ρ_I (Ω·cm)	Type	DD (cm^{-2})	Oxygen Conc. (atoms/cm^3)	Diff. Atm.	Diff. Temp. (°C)	Diff. Time (hours)	Gold Conc. (atoms/cm^3)	ρ_f (Ω·cm)	Type	R_H (cm^3/C)	μ_H (cm^2/V·s)	L (μm)	τ (μs)
10P1250-15	11	p	500	1×10^{18}	Oxygen	1250	0.25	1.0×10^{16}	4.90×10^{2}	p	1.79×10^{5}	366		
10P1250-30							0.5	1.7×10^{16}	8.93×10^{2}	p	3.17×10^{5}	355		
10P1250-60							1	2.9×10^{16}	1.18×10^{3}	p	4.40×10^{5}	373		
10P1250-120							2	4.3×10^{16}	1.45×10^{3}	p	5.35×10^{5}	369		
10PA850-2					Argon	850	2	9.3×10^{12}						
10PA850-8							8	1.2×10^{13}						
10PA850-33							33	4.1×10^{12}						
10PA850-128							128	3.2×10^{14}	1.28×10^{1}	p	5.03×10^{3}	393		
10PA1250-0.5						1250	0.5	3.5×10^{16}	1.01×10^{3}	p	3.75×10^{5}	371		
10PA1250-4							4	9.2×10^{16}	8.46×10^{2}	p	3.09×10^{5}	354		
10P850-288					Oxygen	850	288	4.9×10^{14}	1.35×10^{1}	p	5.30×10^{3}	392	9	23
10P950-144						950	144	4.5×10^{15}	1.47×10^{2}	p	5.57×10^{4}	379	2.3	1.5
10P1050-72						1050	72	1.3×10^{16}	5.28×10^{2}	p	1.93×10^{5}	366		
10P1150-24						1150	24	5.2×10^{16}	1.57×10^{3}	p	5.79×10^{5}	369		
10P1250-2						1250	2	5.9×10^{16}	1.25×10^{3}	p	4.46×10^{5}	352		
10P1250-8						1250	8	10.0×10^{16}	1.03×10^{3}	p	3.56×10^{5}	346		
10PS1250-8						1250	8	9.0×10^{16}	1.19×10^{3}	p	4.06×10^{5}	342		
10PS1250-16							16	[illegible]	1.08×10^{3}	p	3.54×10^{5}	327		
10PS1250-32							32	10.0×10^{16}	1.22×10^{3}	p	4.10×10^{5}	335		
10PS1250-64							64		1.21×10^{3}	p	4.06×10^{5}	337		
20P0850-0.5	20	p	17,000	$<2\times10^{16}$	Oxygen	850	0.5	1.8×10^{13}						
20P0850-2							2	2.7×10^{13}						
20P0850-8							8	5.2×10^{13}						
20P0850-32							32	1.3×10^{14}						
20P01250-0.25						1250	0.25	3.1×10^{16}	2.29×10^{3}	p	8.77×10^{5}	383		
20P01250-0.5							0.5	5.1×10^{16}	1.86×10^{3}	p	7.09×10^{5}	381		
20P01250-1							1	7.1×10^{16}	1.43×10^{3}	p	5.30×10^{5}	371		
20P01250-2							2	9.9×10^{16}	9.26×10^{2}	p	3.26×10^{5}	352		
20PA850-0.5					Argon	850	0.5	1.1×10^{13}						
20PA850-33							33	7.6×10^{13}						
20PA1250-0.25						1250	0.25	3.1×10^{16}	1.74×10^{3}	p	6.50×10^{5}	374		
20PA1250-2							2	7.8×10^{16}	1.15×10^{3}	p	4.55×10^{5}	394		
20P850-32					Oxygen	850	32	1.2×10^{14}	2.15×10^{1}	p	8.22×10^{3}	382	28.5	232
20P850-288						850	288	3.1×10^{14}	2.95×10^{1}	p	1.09×10^{4}	370	14.8	63
20P950-144						950	144	3.9×10^{15}	2.69×10^{2}	p	1.00×10^{5}	372		
20P1050-72						1050	72	2.1×10^{16}	1.48×10^{3}	p	5.62×10^{5}	390		
20P1150-24						1150	24	4.1×10^{16}	2.75×10^{3}	p	9.39×10^{5}	342		
90P650-288	93	p	15,000	$<2\times10^{16}$	Oxygen	850	288	3.2×10^{14}	1.73×10^{2}	p	6.95×10^{4}	402		
90P950-144						950	144	2.7×10^{15}	9.30×10^{2}	p	3.40×10^{5}	366		
90P1050-72						1050	72	1.6×10^{16}	5.60×10^{3}	p	2.15×10^{6}	384		
90P1150-24						1150	24	5.5×10^{16}	1.83×10^{3}	p	6.99×10^{5}	382		
90P1250-8						1250	8	10.8×10^{16}	1.02×10^{3}	p	3.42×10^{5}	335		
5N850-304	5.3	n	0	6×10^{17}	Oxygen	850	304	1.3×10^{14}	6.32×10^{0}	n	-1.08×10^{4}	1700		
5N950-144						950	144	3.3×10^{15}	3.81×10^{4}	n	-6.24×10^{7}	1640		
5N1050-72						1050	72	1.3×10^{16}	2.09×10^{5}	n	-3.05×10^{8}	1460		
5N1150-24						1150	24	5.1×10^{16}	3.58×10^{5}	n	-5.20×10^{7}	145		
5N1250-8						1250	8	10.3×10^{16}	2.43×10^{3}	p	8.61×10^{5}	355		
80N850-304	75	n	0	$<3\times10^{15}$	Oxygen	850	304	3.6×10^{14}	5.62×10^{4}	n	-1.04×10^{8}	1850		
80N950-144						950	144	2.2×10^{15}	1.95×10^{5}	n	-1.60×10^{8}	821		
80N1050-72						1050	72	1.0×10^{16}	1.52×10^{5}	n	4.17×10^{7}	274		
80N1150-24						1150	24	4.8×10^{16}	5.25×10^{3}	p	2.09×10^{6}	390		
80N1250-8						1250	8	10.5×10^{16}	1.73×10^{3}	p	6.00×10^{5}	347		
400N850-304	380	n	0	2×10^{17}	Oxygen	850	304	1.9×10^{14}	1.75×10^{5}	n	-2.60×10^{8}	1490		
400N950-144						950	144	2.6×10^{15}	1.95×10^{5}	p	4.50×10^{7}	220		
400N1050-72						1050	72	1.3×10^{16}	1.08×10^{5}	p	3.70×10^{7}	342		
400N1150-24						1150	24	5.3×10^{16}	6.17×10^{3}	p	2.29×10^{6}	371		
400N1250-8						1250	8	9.8×10^{16}	1.81×10^{3}	p	6.36×10^{5}	352		
2200N950-144	2300	n	70,000	$<2\times10^{16}$	Oxygen	950	144	4.4×10^{15}	5.57×10^{4}	p	2.15×10^{7}	386		
2200N1050-72						1050	72	1.2×10^{16}	4.10×10^{4}	p	1.50×10^{7}	366		
2200N1150-24						1150	24	4.4×10^{16}	8.15×10^{3}	p	3.19×10^{6}	392		
2700N1250-8						1250	8	10.6×10^{16}	1.60×10^{3}	p	5.59×10^{5}	349		

ρ_I = Initial Resistivity
DD = Dislocation Density
Diff. Atm. = Diffusion Atmosphere
Diff. Temp. = Diffusion Temperature
Diff. Time = Diffusion Time
ρ_f = Final Resistivity
R_H = Hall Coefficient
μ_H = Hall Mobility
L = Diffusion Length
τ = Minority Carrier Lifetime

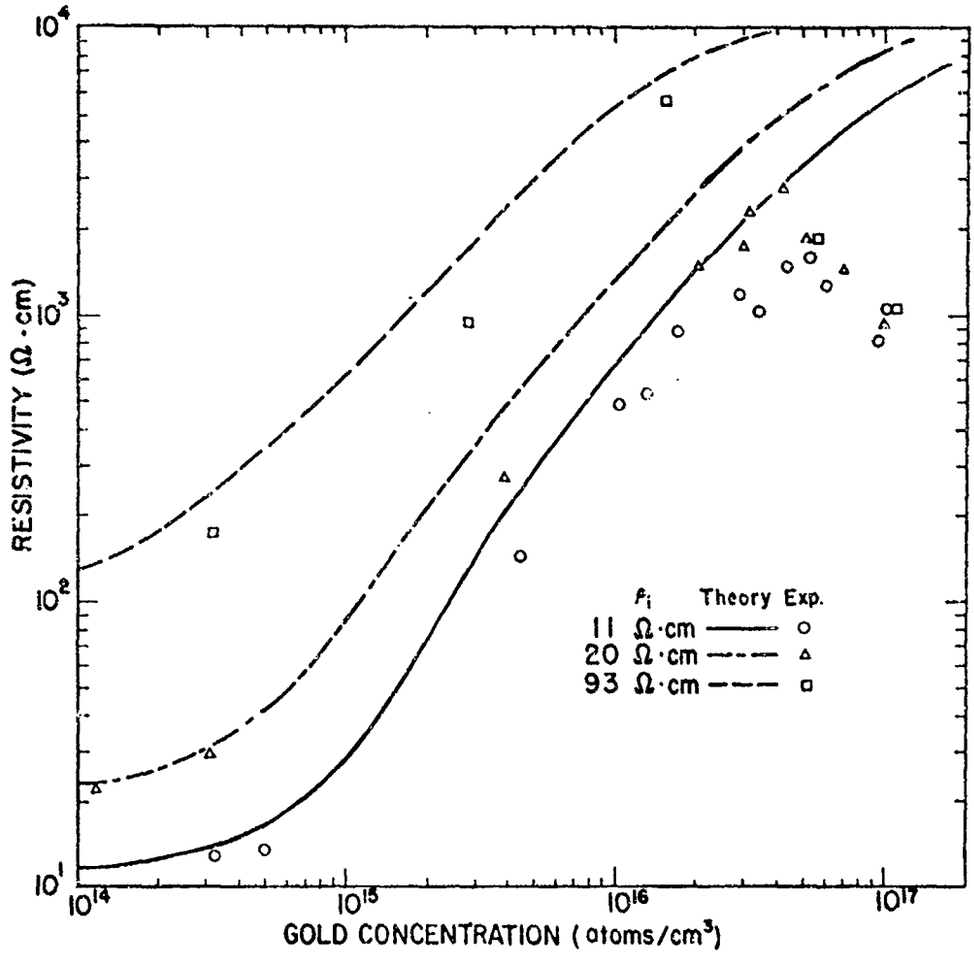


Figure 2. Resistivity as a function of gold concentration in *p*-type silicon with initial resistivity ρ_i. Error bars are not indicated. The standard deviation of the gold determination is estimated to be about 10 percent. Resistivity values are reproducible within about 5 percent.

Brückner [10] has recently observed an impurity state that is located 0.033 eV above the valence band and has suggested that it is an electrically active complex of gold with other defects. The concentration of these acceptors increases rapidly as gold concentration increases. An analysis indicated that the introduction of an energy state between the valence band edge and the gold donor state with a concentration that depends on gold concentration will cause the observed decrease of resistivity as gold concentration increases. The curves in figure 3 were generated by assuming an acceptor concentration that varies as the third power of the gold concentration with an acceptor concentration of 4.5×10^{15} cm^{-3} at a gold concentration of 1×10^{17} cm^{-3}. Exponential functions for the acceptor concentration can also be used to fit the experimental data. More knowledge about the origin of the acceptor center must be developed to aid in determining its true dependence on gold concentration. The agreement of the calculated curves to the data at low gold concentrations is sensitive to the degeneracy factor for the gold donor level, and the value of 0.125 used in the calculations gives a good fit to all three resistivity groups. Values of 0.25 (used in the calculations of figure 2) and 0.0625 give significant shifts of the computed curves to the left and right, respectively, of the experimental data. The other parameters for the curves of figure 3 are the same ones used for figure 2.

These experiments and analysis suggest that the discrepancy between calculated and observed resistivity in *p*-type silicon doped with large amounts of gold is related to the presence of as yet unidentified shallow acceptor complexes. A similar discrepancy can be observed in *n*-type silicon doped with large amounts of gold. Hall effect and resistivity measurements were made at room temperature on Hall bars cut from 5.3-, 75-, and 380-Ω•cm *n*-type wafers that had been diffused with gold at 850, 950, 1050, 1150, and 1250°C and from 2300-Ω•cm *n*-type wafers diffused at 950, 1050, 1150, and 1250°C. In all cases except the 5.3-Ω•cm wafer diffused at 850°C, the hole concentration exceeds the shallow donor concentration. As the gold concentration is increased, the resistivity is expected to reach a maximum and then decrease. Shortly before the maximum the specimens become *p*-type. At large values of gold concentration the resistivity should approach a constant value characteristic only of the gold impurity. The resistivity maximum should occur at the intrinsic value where the product of electron concentration and mobility is equal to the product of hole concentration and mobility, and except for possible differences in mobility be independent of the resistivity of the wafer before gold is added.

The experimental results, shown in figure 4, are generally consistent with theoretical expectations although they do not agree in a number of important details. The dashed lines associated with the data points suggest the form the resistivity-concentration curves would have to take in order to reach a common resistivity maximum near the value of about 3.5×10^5 Ω•cm calculated for a temperature of 25°C (intrinsic carrier concentration of 1.10×10^{10} cm^{-3}, electron mobility of 1300 $cm^2/V \cdot s$, and

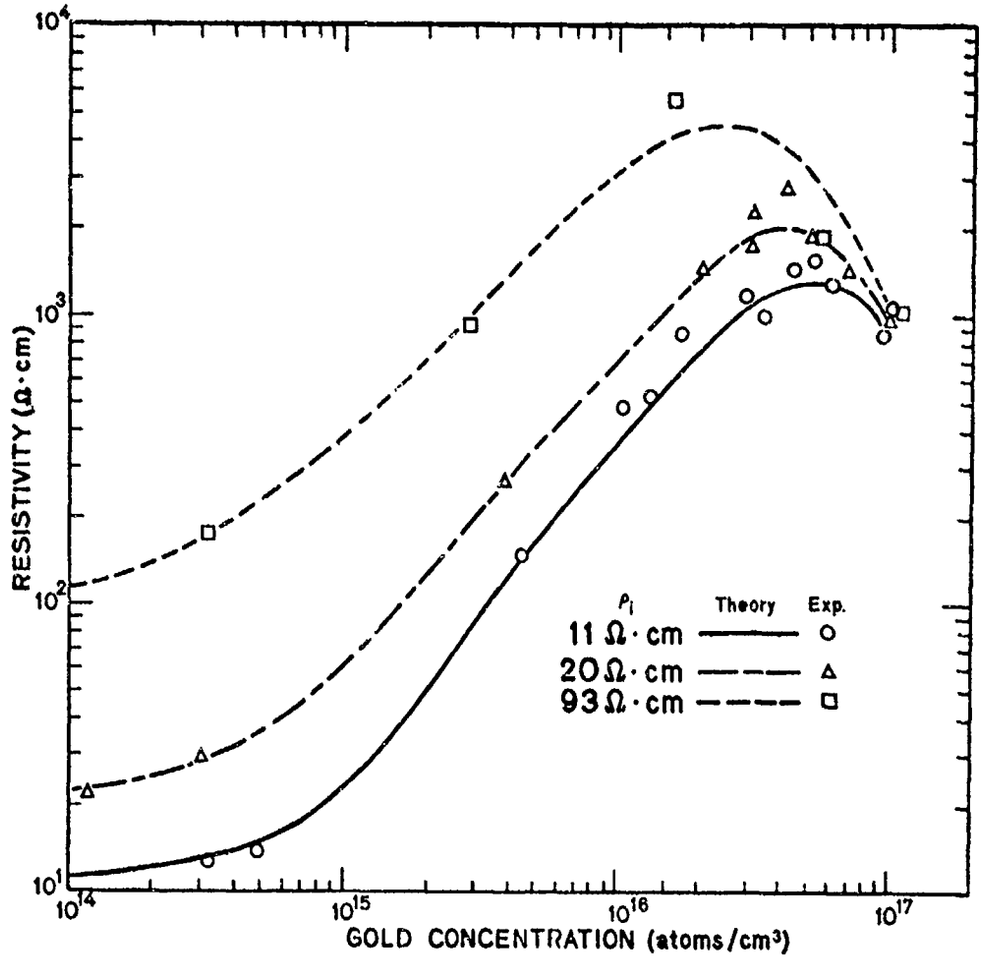


Figure 3. Calculated curves which fit the resistivity as a function of gold concentration for p-type silicon with initial resistivity ρ_i. The experimental points are the same as those in figure 2. The parameters used to generate the curves are given in the text.

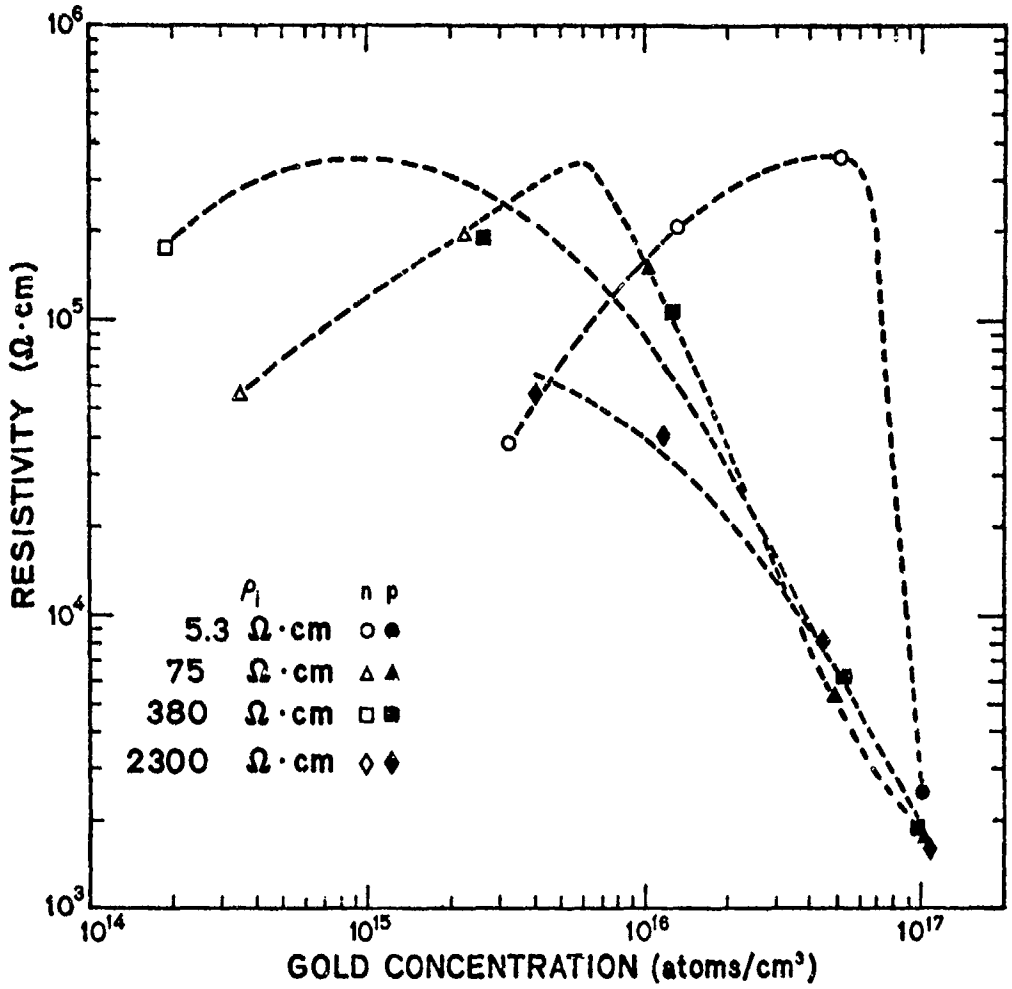


Figure 4. Resistivity as a function of gold concentration in initially *n*-type silicon with starting resistivity ρ_i. Resistivity of the specimens prior to gold doping is given. Open symbols represent specimens which remained *n*-type after gold diffusion; solid symbols, specimens which were converted to *p*-type by the addition of gold. The dashed curves indicate the form of the relationship between resistivity and gold concentration required by the existence of a common maximum as discussed in the text. Error bars are not indicated. The standard deviation of the gold determination is estimated to be about 10 percent. Resistivity values are reproducible within about 10 percent.

hole mobility of 500 $cm^2/V \cdot s$). The differences in shapes of these curves, the poor fit of the data for the 380-Ω·cm specimens, and the *n*-type nature of the 5.3-Ω·cm specimen diffused at 1150°C (which appears at the maximum) all are indicative of either experimental or interpretative uncertainties. The resistivity at a gold concentration of 10^{17} cm^{-3} is consistent with the resistivity measured on *p*-type wafers. Evidently the same mechanism is affecting the resistivity in both types of specimens, but this has not yet been verified by detailed analysis of the data for *n*-type silicon.

There are a number of complicating factors in the analysis of the electrical data. Experiments are now in progress to separate the influence of high-temperature heat treatment from the effects of gold-doping, to examine the effects of interstitial gold on the electrical properties of gold-doped silicon, and to study the effects of gold precipitation and redistribution. These experiments have not yet reached the point where conclusions may be drawn from their results.

CARRIER LIFETIME MEASUREMENTS

When the gold concentration is smaller than the concentration of the shallow doping impurity in either *n*-type or *p*-type silicon, the presence of the gold does not measurably affect the resistivity. However, electrically active gold does affect the carrier lifetime.

Past studies of carrier lifetime in gold-doped silicon have generally been made by means of the reverse recovery technique in diodes [11, 12]. Because of uncertainties encountered in relating carrier lifetime in silicon diodes as measured by reverse recovery and open-circuit voltage decay and because of the desire to make measurements on gold-doped wafers without the necessity of forming *p-n* junctions, the steady-state surface photovoltage (SPV) method [13] was selected for use in the present work.

With this method measurements can be made directly on the Hall bars. The minority carrier lifetime (τ) is calculated from the measured diffusion length (L) by use of the expression $\tau = L^2/D$, where D is the diffusion constant of the minority carrier. A diagram of the experimental apparatus is shown in figure 5. The specimen surface is illuminated with chopped monochromatic radiation of energy slightly greater than the band-gap of the semiconductor. Electron-hole pairs are produced and diffuse to the surface where they are separated by the electric field of a depletion region to produce a surface photovoltage. The SPV signal is capacitively coupled into a lock-in amplifier for amplification and measurement. The photon intensity is adjusted to produce the same signal at various wavelengths of illumination. The photon intensity required to produce this constant SPV signal is plotted against the reciprocal absorption coefficient for each wavelength. Since the reflectivity of silicon varies

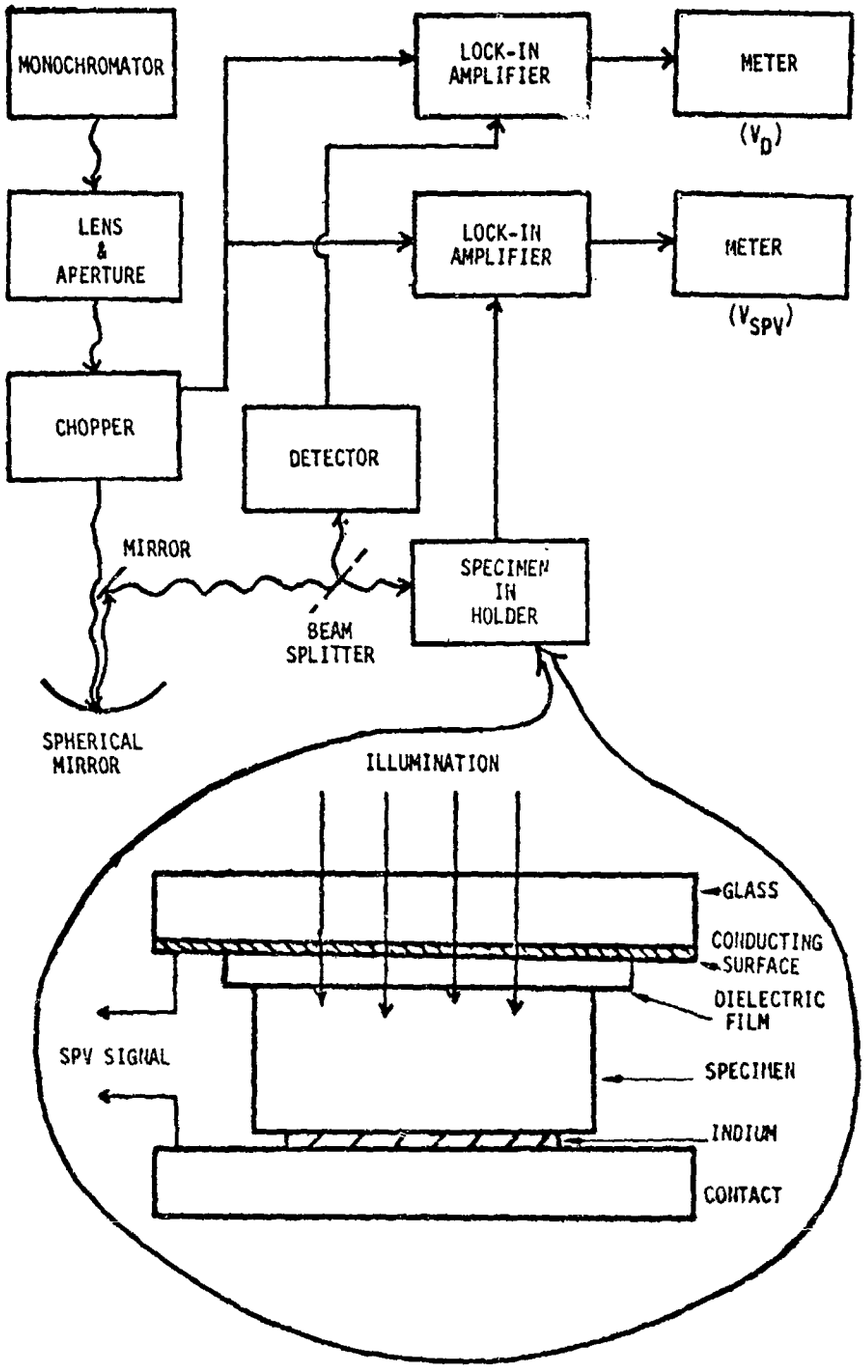
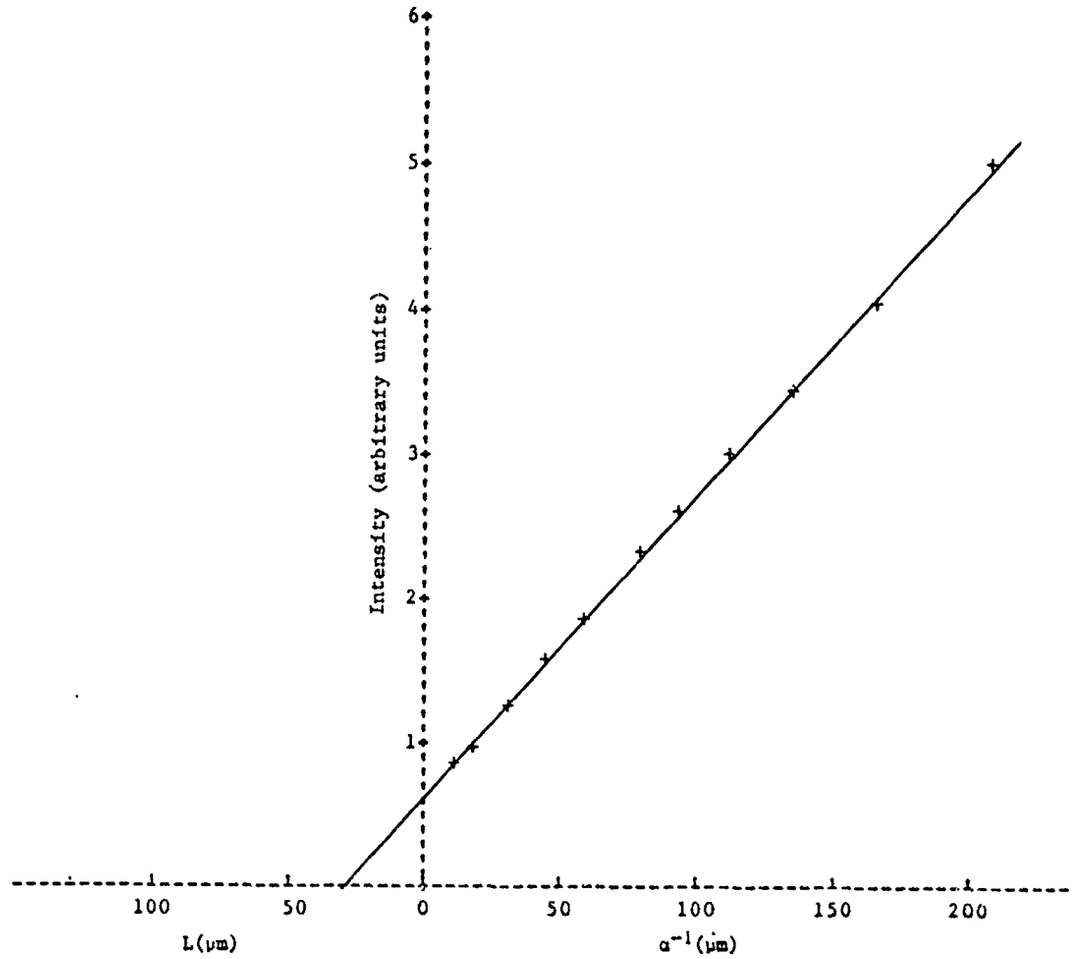


Figure 5. Schematic of the specimen holder for the SPV measurements and block diagram of the equipment.



WAVELENGTH	T C VOLTAGE	RECIPR ABS COEFFI	INTENSITY
1.020 μm	8.40 μV	206.329 μm	5.854
1.010	6.85	164.864	4.725
1.000	5.90	134.306	4.028
.990	5.20	111.159	3.513
.980	4.55	93.225	3.041
.970	4.10	79.063	2.711
.950	3.35	56.450	2.168
.930	2.90	44.494	1.835
.900	2.40	30.797	1.467
.850	1.95	18.032	1.122
.800	1.85	11.269	.997

Lo = 28.9236 μm SIGMA = .9716 μm SPECIMEN: 20P850-32 DATE: 12/27/71

SPV SIGNAL = 2.5 mV

Figure 6. Typical plot and printout of the SPV data. The quantity in the column labeled INTENSITY is the product of WAVELENGTH, TC VOLTAGE, and a factor which accounts for the reflectivity of the specimen surface. To simplify the plotting, the intensity calculated for the longest wavelength is assigned a value 5.000 and the other values are scaled appropriately.

slightly with wavelength, the intensity determined by a thermocouple detector is corrected to correspond to the illumination actually absorbed by the specimen. A typical example of the plot that is obtained is shown in figure 6. The linear portion of the plot is extrapolated to zero intensity; the magnitude of the (negative) intercept value is equal to the effective diffusion length.

In order to plot the SPV data, it is necessary to know the absorption coefficient as a function of wavelength. Many reports of the absorption coefficient of silicon at room temperature in the wavelength range of interest have appeared in the literature. Selected data obtained on high-purity silicon under a variety of experimental conditions are plotted against wavelength in figure 7. The data of Runyan [14] were obtained on stress-relieved silicon, and since the gold diffusion process relieves any stresses in the material, it is thought that use of these data is appropriate for the gold-doped specimens. SPV measurements are made for wavelength values between 0.80 and 1.05 μm. Since the Runyan data extends only to 1.0 μm, an extrapolation was made resulting in values which are in between those obtained by Braunstein, *et al.* [15] and Fan, *et al.* [17] in the range 1.0 to 1.05 μm. Plots made using this data generally are linear in the wavelength range from 0.85 to 1.02 μm. Deviations from linearity at longer wavelengths are expected unless the specimen thickness is greater than four times the reciprocal absorption coefficient at the wavelength in question. On wafers of the starting material, which receive no heat treatment, it was found that use of the Dash and Newman [16] absorption coefficient data, which were measured on silicon that was not stress-relieved, gives a more linear plot than is obtained using the Runyan data.

With the SPV method, accurate and reproducible measurements of short diffusion lengths are much more difficult than measurements of long diffusion lengths. The standard deviation due to scatter in the data of an SPV plot is typically 0.5 μm, independent of diffusion length; the corresponding uncertainty in the accuracy of the diffusion length is 1 to 2 μm. Thus the percentage error can be very significant for a diffusion length of 5 μm, while it is negligibly small for a diffusion length of 100 μm. The most obvious sources of error include noise and calibration uncertainty of the thermocouple detector and lock-in amplifiers, drift of the SPV signal with time, and incorrect values of the absorption coefficient and reflectivity.

Most of the measurements to date have been made on *p*-type specimens in the form of Hall bars. Specimen preparation consists of masking the side of the Hall bar containing the contacts and then removing about 25 μm from the other surface by a polishing chemical etch that consists of a mixture of nitric, hydrofluoric, and acetic acids. This treatment usually results in an SPV signal of a few millivolts which is adequate for the measurement.

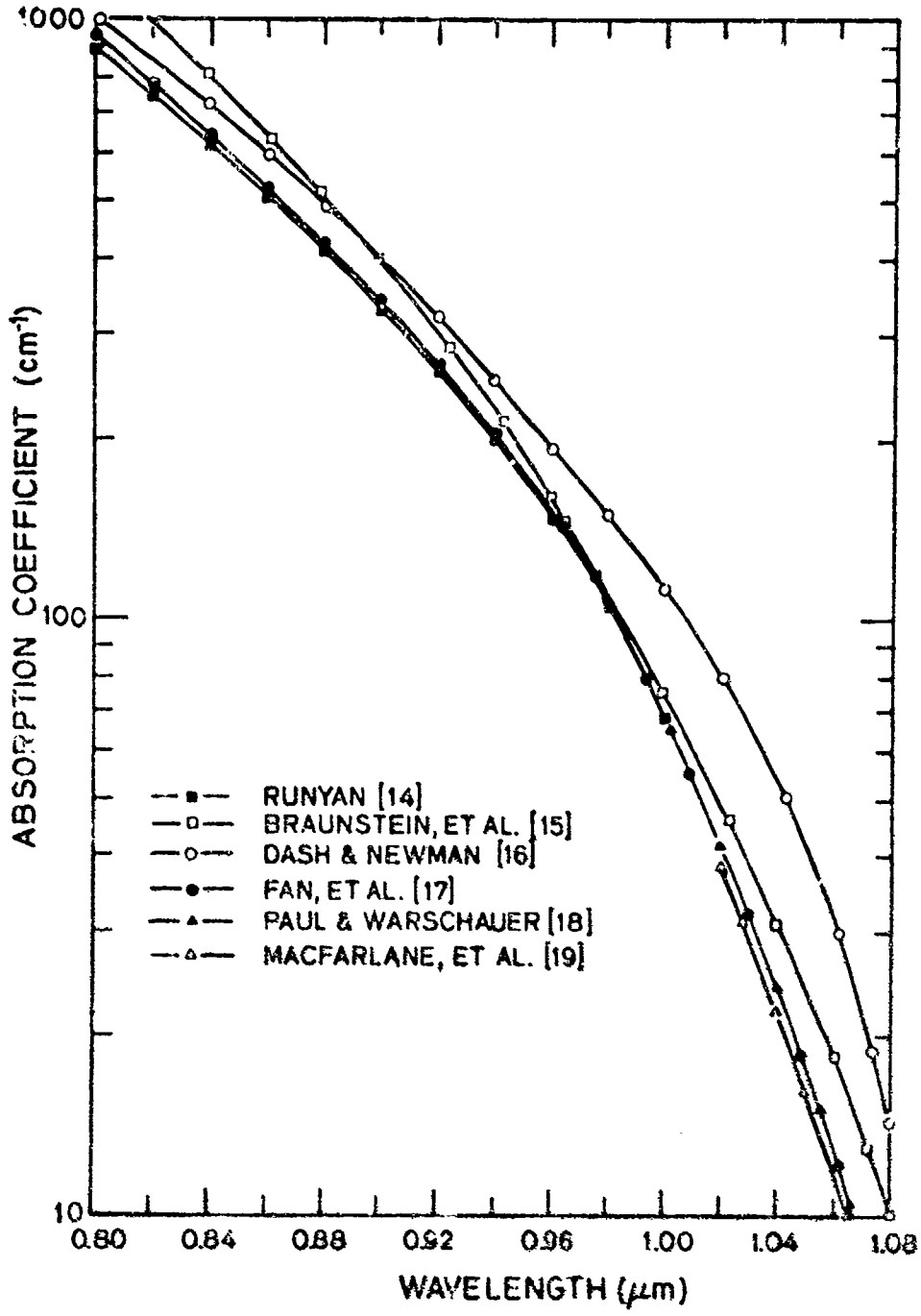


Figure 7. Absorption coefficient results for silicon at room temperature as a function of wavelength. The curve for Macfarlane, *et al.* was obtained by interpolation from their data at higher and lower temperatures.

In p-type silicon where the shallow acceptor concentration is much greater than the gold concentration the lifetime, τ, in seconds, is approximately

$$\tau = \frac{1}{\sigma_{e02} \, v_e \, N_{Au}}$$

where σ_{e02} is the capture cross section for the positively charged gold donor in square meters, v_e is the thermal velocity for electrons in meters per second, and N_{Au} is the gold concentration in atoms per cubic meter. The values for capture cross sections in gold-doped silicon reported in the literature are compiled in table 3. For the positive gold donor the cross sections found by Bemski [20] and by Fairfield and Gokhale [21] are in better agreement than for the other centers. Lifetimes calculated using a cross section of 3.1×10^{-19} m^2 and a thermal velocity of 2×10^5 m/s are given in table 4 for comparison with the lifetime values computed from the measured diffusion lengths of the gold-doped specimens. Measurements on more specimens are needed before definite conclusions can be reached regarding the dependence of lifetime on gold concentration or the appropriateness of the available cross section data.

CARRIER LIFETIME MEASUREMENTS IN GALLIUM ARSENIDE

The feasibility of using the SPV method to measure carrier lifetimes in gallium arsenide was also considered as a related task under this contract. Measurements on gallium arsenide made by Goodman [13] and by Bergmann, Fritzsche, and Riccius [26] yielded diffusion lengths predominately in the range 5 to 20 μm. In gallium arsenide, the carrier lifetime is shorter than it is in silicon of the same type with the same diffusion length. The electron mobility in gallium arsenide is about six times as large as that in silicon while the hole mobility is a little less than twice as large. Lifetimes in n-type gallium arsenide are about ten times larger than those in p-type for the same diffusion length since the diffusion coefficient for holes is about ten times smaller than that for electrons. Since the error in the SPV measurement is nearly independent of the magnitude of the carrier diffusion length, it follows that measurements of electron lifetime in p-type gallium arsenide specimens should be relatively easy to make down to 0.5 to 1 ns while the lower limit in measurements of hole lifetime in n-type gallium arsenide specimens is likely to be 5 to 10 ns. Measurements on gallium arsenide require illumination with 0.8- to 0.9-μm radiation which is easily obtained by installing the appropriate grating in the present monochromator. Values of absorption coefficient as a function of wavelength are available in the literature [26, 27].

Table 3 — Capture Cross Sections for Electrons and Holes in n-Type and p-Type Gold-Doped Silicon

	Capture Site	300 K				162 K	77 K	
		(a)	(b)	(c)	(d)	(e)	(f)	(g)
n-type								
σ_{e01} (cm^2)	Au^0	$5 \cdot 10^{-16}$	$8 \cdot 10^{-17}$	$2.0 \cdot 10^{-15}$	$2.5 \cdot 10^{-15}$	$1 \cdot 10^{-15}$	$\sim 3 \cdot 10^{-15}$	$5 \cdot 10^{-16}$
σ_{h01} (cm^2)	Au^-	$1 \cdot 10^{-15}$	$6 \cdot 10^{-15}$		$1.3 \cdot 10^{-14}$	$2 \cdot 10^{-14}$	$\sim 1 \cdot 10^{-13}$	$2.3 \cdot 10^{-13}$
p-type								
σ_{e02} (cm^2)	Au^+	$3.5 \cdot 10^{-15}$	$3.1 \cdot 10^{-15}$				$\sim 6 \cdot 10^{-14}$	$1 \cdot 10^{-13}$
σ_{h02} (cm^2)	Au^0	$\geq 10^{-14}$	$1.2 \cdot 10^{-15}$				$\sim 3 \cdot 10^{-15}$	

(a) Benski [21], from lifetime data. Calculated by the author assuming thermal velocities of about $1 \cdot 10^7$ cm/s.
(b) Fairfield and Gokhale [22], from capture probabilities obtained from photoconductivity measurements. Computed assuming a thermal velocity of $2 \cdot 10^7$ cm/s for both electrons and holes.
(c) Senechal and Basinski [23], from emission rate data. Calculated by the authors assuming $E_C - E_{Au} = 0.545$ eV, $v_e = 2 \cdot 10^7$ cm/s, and a degeneracy factor for the gold level of unity. The value at 300 K is based on the authors' extrapolation of data obtained at 250 K to room temperature.
(d) Tasch and Sah [24], from emission rate data. Calculated by the authors assuming $E_C - E_{Au} = 0.545$ eV, $E_{Au} - E_V = 0.588$ eV, and thermal velocities of about $2 \cdot 10^7$ cm/s. The degeneracy factors were arbitrarily assigned a value of unity.
(e) Colligan and van Vliet [25], from measurements of generation-recombination noise. Calculated by the authors but the values used for the thermal velocities could not be extracted.
(f) Davis [26], from lifetime measurements using alpha particle irradiation. Calculated by the author assuming thermal velocities of $1.3 \cdot 10^7$ cm/s for electrons and $1.0 \cdot 10^7$ cm/s for holes.
(g) Benski [21]. Computed from 300 K values using temperature dependence given. No temperature dependence was quoted for σ_{h02}.

Table 4 — Measured and Calculated Minority Carrier Lifetimes in p-Type Gold-Doped Silicon

Specimen Number	ρ_i (Ω·cm)	Diff. Temp. (°C)	Diff. Time (hours)	ρ_f (Ω·cm)	Gold Conc. (atoms/cm^3)	L (μm)	D (cm^2/s)	τ (ns) Measured	τ (ns) Calculated
10F850-288	11	850	288	13.5	$4.9 \cdot 10^{14}$	9	35	23	33
10F950-144	11	950	144	142	$4.5 \cdot 10^{15}$	2.3	35	1.5	*
20F850-32	20	850	32	21.5	$1.2 \cdot 10^{14}$	28.5	35	232	134
20F850-288	20	850	288	29.5	$3.1 \cdot 10^{14}$	14.8	35	63	52

*Not calculated because the gold concentration exceeds the shallow acceptor concentration.

DIRECTIONS OF FUTURE WORK

The tasks immediately ahead include completion of resistivity and Hall effect measurements on the silicon crystals listed in table 1. On completion of the experiments concerned with interstitial gold, gold precipitation and redistribution, and effects of heat treatment, it is expected that the data analysis can proceed and that considerably greater understanding concerning the relationship between total and active gold concentration can be developed. A new series of experiments will be designed to investigate the origin and characteristics of the acceptor state introduced at high gold concentrations.

Some additional improvements to the surface photovoltage system are scheduled; these are expected to improve the reproducibility still further and lower the limit of measurable diffusion length. Once these are complete, measurement activity will be increased in order to collect the information necessary to resolve the conflicts in capture cross section data.

Investigation of the relationship between the reverse recovery and voltage decay methods for measuring carrier lifetime in silicon diodes has been resumed in order to develop the capabilities necessary to extend the study of gold-doped silicon to junction structures.

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