# NBS TECHNICAL NOTE 570

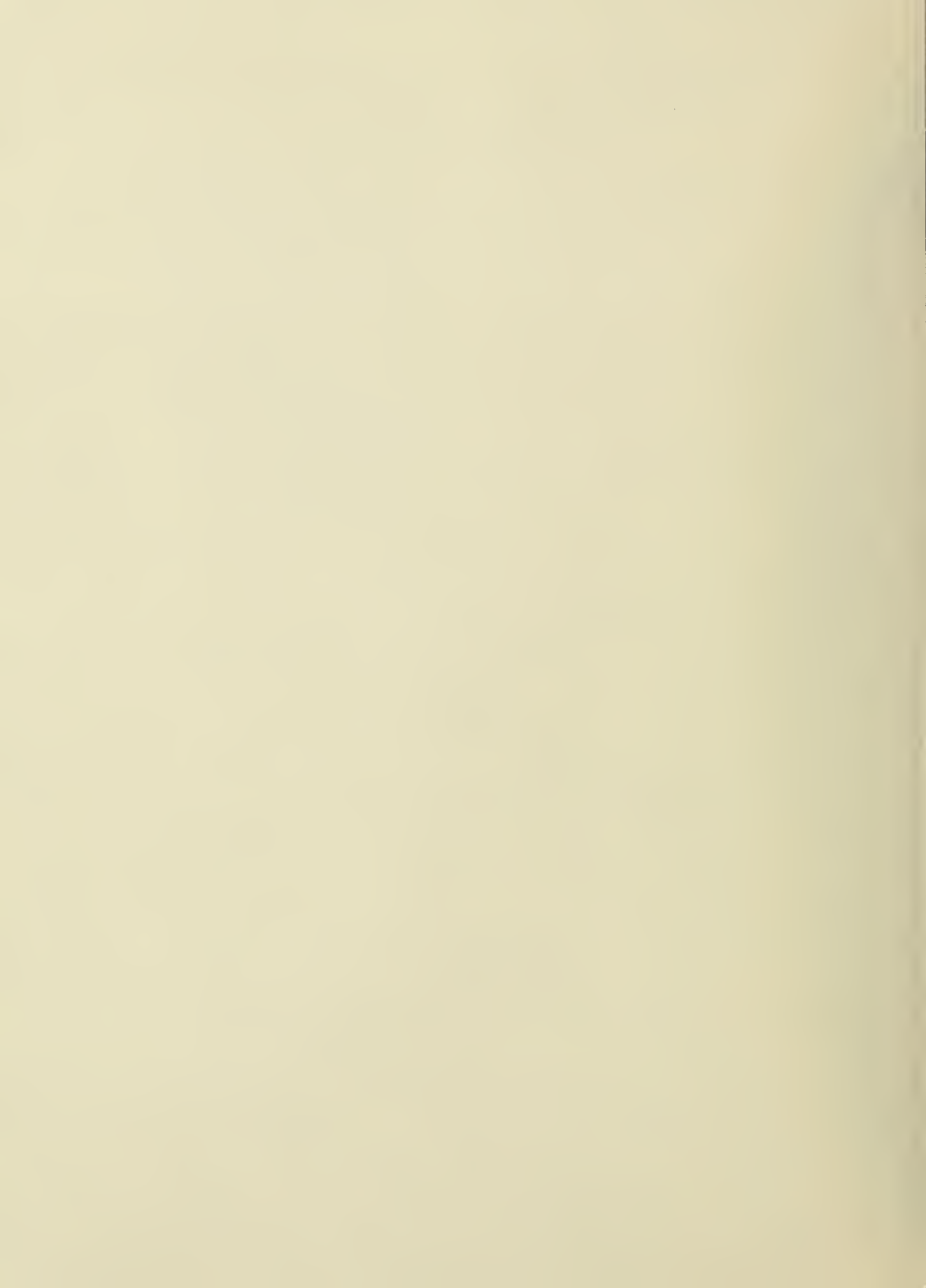# Determination of Deep Impurities in Silicon and Germanium by Infrared Photoconductivity

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# Determination of Deep Impurities in Silicon and Germanium by Infrared Photoconductivity

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# Determination of Deep Impurities in Silicon and Germanium by Infrared Photoconductivity

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The feasibility of using infrared photoresponse and photo-conductivity measurements to study deep impurities in germanium and silicon is examined by reviewing the literature. It is concluded that photoconductivity is useful in detecting the presence of specific impurities because each impurity has a long wavelength cut off in response associated with its ionization energy. However, when there are several deep impurities in the same specimen, it is difficult to be certain of detecting each one because some have broad cut offs and many have nearly the same ionization energies. Photoconductivity as a general technique has serious limitations for determining the total concentration of deep impurities. The equations for determining impurity concentration from the magnitude of the photoconductivity signal depend on the relative influence of deep and shallow centers. Equations are derived for several situations and experimental results from the literature are discussed for each one. Only uncompensated centers are available for photoionization and therefore the total concentration can not be obtained directly. In some situations the response due to a deep center is independent of its concentration. Other techniques for studying deep impurities are discussed briefly.

Key words: Deep impurities; germanium; infrared; photoconductivity; photoresponse; semiconductors; silicon.

## 1. Introduction

This report discusses the following two questions: (1) Can a specific impurity in germanium or silicon be identified from photoconductivity? (2) Does the photoconductivity vary with impurity concentration in a systematic way?

Before proceeding to answer the questions, some experimental aspects will be discussed. The electrical circuit for photoconductivity measurements is very simple. The specimen and a load resistor are connected in series with a voltage source. Chopped, monochromatic radiation is incident on the specimen and the resulting change in specimen conductivity gives rise to a voltage change which is detected by a phase-sensitive

amplifier.  A more detailed treatment of the experimental method can be found in Ryvkin [1].  For most measurements, specimens must be cooled to 77 K or below to eliminate thermal ionization of the impurity under study.  Since a small change in conductivity resulting from photoionization is most easily detected in a high resistance specimen, the lowest readily available temperature is often used to freeze-out uncompensated shallow impurities.  However, since the lifetime and mobility of the photoionized carriers are functions of temperature, there is usually a maximum in the response at some particular temperature.

A deep impurity is one that has an energy level near the middle of the band gap.  To obtain quantitative information on a deep impurity it is necessary to know the optical absorption and capture cross sections of the impurity at the temperature used for the photoconductivity measurements.  These values may be available in the literature, but if they are not, it is necessary to determine them experimentally.  For some impurities there are large discrepancies in the published data, and additional measurements are needed to obtain reliable values.

The most direct method for determining the optical absorption cross section of a particular impurity is measurement of the transmission of a specimen containing a known concentration of the impurity.  Emmons [2] gives a discussion of the procedure with specific application to gallium in silicon at 20 K.  Data are usually obtained as a function of wavelength and at the temperature used for the photoconductivity measurements as the cross sections are generally wavelength and temperature dependent.

Ryvkin [3] has discussed the determination of the absorption cross section from the initial slopes of the photoconductivity rise and decay curves.  Putley [4] has compiled absorption cross sections of some deep impurities in germanium.

The most common method of obtaining data on capture cross sections is carrier lifetime measurements.  Blakemore [5] has discussed in detail the relation between lifetime and capture cross section.

Capture cross sections can also be determined from measurements of the generation-recombination noise as a function of frequency.  This technique is discussed by Schibli and Milnes [6] and by Neuringer and Bernard [7] who determined the cross sections of free holes in *p*-type gold-doped germanium.

References to other techniques such as the pulsed field effect and frequency-dependent capacitance methods are given by Schibli and Milnes [6] who have also compiled capture cross section data for impurities in silicon.  Bonch-Bruevich and Landsberg [8] have published a review article on recombination mechanisms which includes tables of cross sections for impurities in both silicon and germanium.

## 2. Identification of Specific Impurities

That photoconductivity as a function of wavelength for each impurity in a given material is unique makes possible the identification of an unknown impurity from its photoconductivity spectrum. The most significant feature for each impurity is the long wavelength cut off of the response. At this wavelength the energy of the radiation is equal to the ionization energy of the impurity. For longer wavelength radiation, photoionization is not possible and photoconductivity ceases. Some impurities in a given material have a sharp long wavelength cut off whereas other impurities have a broad and irregular one. This variation can be an advantage when identifying impurities with nearly the same ionization energies. However, when two or more impurities with about the same ionization energy are present in the same specimen, broad long wavelength cut offs are a definite disadvantage.

A further complication is that the thermal ionization energies of various impurities, as determined by Hall effect measurements for example, do not correspond to the same relative position on the photoconductivity curves. Thus if unknown impurities are to be identified from photoconductivity measurements, it is important to have photoconductivity spectra for the likely impurities. Several impurities in the same specimen can be identified by photoconductivity if each is present in sufficient concentration to give a sharp drop in response at its respective long wavelength cut off. If two or more of the impurities have about the same ionization energies, then photoconductivity may be attributed to fewer impurity species than are actually present. Graphs of photoconductivity spectra for many deep impurities in germanium and silicon have been collected in data sheets prepared by the Electronic Properties Information Center [9].

## 3. Dependence of Photoconductivity on Impurity Concentration

### 3.1 Concentration of Deep Impurities Exceeds that of Shallow Impurities

First the basic equations will be reviewed. Putley [4] has derived the expression

$$\Delta\sigma = \frac{q\mu\eta\tau I}{Vh\nu} \tag{1}$$

where $\Delta\sigma$ is the change in conductivity,
   I is the intensity of radiation falling normally on the surface,
   q is the electronic charge,
   $\mu$ is the mobility of the generated carriers,
   $\tau$ is the lifetime of the free carriers,
   $\eta$ is the fraction of incident photons absorbed by the impurity centers,

V is the specimen volume,
h is Plank's constant, and
v is the frequency of the radiation.
With the assumption that each quantum absorbed liberates one free
carrier, $\eta$ is determined by the fractional amount of energy absorbed.
For a specimen with polished, parallel sides

$$\eta = \frac{(1-R)(1-e^{-\alpha x})}{1-Re^{-\alpha x}} \qquad (2)$$

where R is the reflectivity, $\alpha$ is the impurity absorption coefficient,
and x is the specimen thickness. Further manipulation will relate the
change in conductivity to impurity density for $p$-type material containing
a single deep acceptor whose concentration exceeds that of the shallow
donors. The quantity $\alpha x$ is usually less than one and an approximation for
$\eta$ good to 20 % for $\alpha x$ between 0.1 and 0.7 and R = 0.3 (appropriate for
silicon) can be obtained by neglecting $Re^{-\alpha x}$ in the denominator and ex-
panding the numerator to give

$$\eta \cong (1-R)(\alpha x) = (1-R) \; \sigma_a \; (N_A-N_d)x \qquad (3)$$

where $\sigma_a$ is the photoionization (absorption) cross section of an un-
occupied center, $N_A$ is the density of deep (capital subscript) acceptors,
and $N_d$ is the density of shallow donors available for compensating the
deep acceptors.

In general free carrier recombination occurs by the capture of car-
riers by ionized impurities. At temperatures low enough so that thermal
ionization is unimportant, the number of recombination centers in mate-
rial for which the deep impurity concentration exceeds that of the shal-
low impurities is determined by the concentration of compensating im-
purities. The carrier lifetime is

$$\tau = \frac{1}{vN_a\sigma_n^+} \qquad (n\text{-type samples}) \qquad (4)$$

and

$$\tau = \frac{1}{vN_d\sigma_p^-} \qquad (p\text{-type samples}) \qquad (5)$$

where v is the thermal velocity of the charge carriers, $\sigma_n^+$ is the capture
cross section for an electron by a positively charged (before capture)
deep center, and $\sigma_p^-$ is the capture cross section for a hole by a negative-
ly charged deep center. Deep impurities such as copper or gold are usu-
ally very effective recombination centers if they are charged.

The approximate dependence of the photoresponse, $\Delta\sigma/I$, on impurity
concentration for initially $n$-type material which has been doped with

a deep level impurity to become $p$-type is found by substituting Eqs. (5) and (3) into (1). That is

$$\frac{\Delta\sigma}{I} = \frac{q\mu(1-R)\sigma_a(N_A-N_d)x}{vVh\nu N_d\sigma_p^-} .$$

(6)

The equation for $n$-type material is found by substituting $N_D$ for $N_A$, $N_a$ for $N_d$, and using the electron mobility and appropriate cross sections. The photoresponse has the disadvantage that the concentration of deep impurities can not be found unless the density of compensating impurities is known or can be determined by an independent experiment.

Quist [10] has shown that the relative photoresponse at a given temperature for three copper-doped germanium detectors, each with the same copper concentration ($7 \times 10^{15}$ atoms/cm$^3$), but with different donor concentrations is proportional to the reciprocal of the donor density as predicted by Eq. (6).

Newman and Tyler [11] prepared a series of $p$-type germanium specimens by diffusing nickel at 850°C into crystals with predetermined concentrations of donors (arsenic). The photoconductivity spectra at 77 K had a threshold at 0.23 eV corresponding to the energy of the lower nickel level. The dependence of the photoresponse on the ratio of arsenic to nickel concentrations was in excellent agreement with Eq. (6). Thus compensation plays a dominant role in photoresponse measurements.

Sidorov and Lifshits [12] have studied the dependence of the photoresponse on the impurity concentration and degree of compensation in the systems Ge:Au:Sb and Ge:Zn:Sb. Gold concentrations varied from 0.3 to $4 \times 10^{15}$ atoms/cm$^3$, while zinc concentrations ranged from 0.2 to $5 \times 10^{15}$ atoms/cm$^3$. The amount of antimony in the germanium was chosen to give specimens with different degrees of compensation — from nearly empty levels for Au (0.15 eV) or Zn (0.09 eV) (the 0.03 eV Zn level was completely filled) to complete occupation of these electron levels. For the gold system hole recombination occurs at Au$^-$ ions whereas for the zinc-doped specimens hole capture is by Zn$^{--}$ ions. The photoresponse of both sets of specimens showed the expected dependence on compensation as given by Eq. (6).

To calculate the photoconductivity, $\Delta\sigma/\sigma I$, for $p$-type material we need to divide Eq. (6) by the conductivity $pq\mu$ where p at low temperatures is given by

$$p = \frac{N_A-N_d}{N_d} N_v exp[-(E_i-E_v)/kT].$$

(7)

Eq. (7) holds for partially compensated material where $N_v$ is the density of states in the valence band and $E_i - E_v$ is the ionization energy of the deep impurity. The photoconductivity is

$$\frac{\Delta\sigma}{\sigma I} = \frac{(1-R)\sigma_a x exp[(E_i-E_v)/kT]}{vVh\nu\sigma_p^- N_v} \ .$$  (8)

Thus if the dark conductivity and the light induced change in conductivity are determined by impurity atoms of one species, the photoconductivity is independent of the concentration of active centers and their compensation and is determined only by the ratio of the cross sections of these centers for light absorption and charge carrier recombination. This type of behavior has been observed for copper-doped germanium by Adams, Beyen, and Petritz [13]. For copper concentrations from 1.7 to $14.0 \times 10^{15}$ atoms/cm$^3$, the photoconductivity they observed at 5 K was independent of copper concentration.

It is interesting to note that when the photoconductivity is given by Eq. (8), cross section data can be obtained without measuring the impurity concentration. Thus from photoconductivity data and knowledge of one cross section, the value of the other cross section can be calculated.

3.2 Concentration of Shallow Impurities Exceeds that of Deep Impurities

If the shallow impurities outnumber the deep ones, then the recombination center density depends on the characteristics of the specific impurities. For example, gold has both donor and acceptor levels in silicon, and in *n*-type material the Au$^-$ ions are effective capture centers whereas in *p*-type material the Au$^+$ ions have large capture cross sections. Thus for either *n*- or *p*-type silicon the number of recombination centers is equal to the gold concentration.

Consider the response of a deep acceptor level in a specimen where the shallow donor concentration exceeds the deep acceptor density. The light absorption and carrier lifetime are both proportional to the deep impurity concentration so the photoresponse is

$$\frac{\Delta\sigma}{I} = \frac{q\mu(1-R)\sigma_a x}{vVh\nu\sigma_p^-} \ .$$  (9)

At low temperatures the conductivity is

$$\sigma = q\mu \ \frac{(N_d-N_A)}{N_A} \ N_c exp[-(E_c-E_i)/kT],$$  (10)

where $N_c$ is the density of states in the conduction band and $E_c - E_i$ is the ionization energy of the shallow donor. Then the photoconductivity due to the deep acceptor is given by

$$\frac{\Delta\sigma}{\sigma I} = \frac{(1-R)\sigma_a x N_A \, exp[(E_c - E_i)/kT]}{vVh\nu\sigma_p^-(N_d - N_A)N_c} . \qquad (11)$$

The shallow donor will also give rise to photoconductivity and may mask the contribution of the deep acceptor. For this case there is no reason to measure the photoconductivity as the equation for the dark conductivity gives the dependence on impurity concentration. To obtain $N_A$ from the conductivity it is necessary to know $N_d$. Hall effect measurements as a function of temperature can be used to get both $N_d$ and $N_A$, but considerable effort is required.

Small concentrations of deep impurities have been detected in silicon by photoconductivity measurements made by Plotnikov and co-workers [14]. They observed shoulders in the spectra at wavelengths corresponding to the energy levels of copper and gold. Concentrations were estimated to be $10^{10}$ to $10^{11}$ atoms/$cm^3$ using literature values for the capture cross sections, but the equations used were not given. For these low densities the dependence of photoconductivity on impurity concentration may be approximately linear. The absorption of light by a particular deep impurity is proportional to its uncompensated concentration whereas the carrier lifetime is determined by other centers when there are so few deep ones. In addition the deep impurities may have very little influence on the specimen conductivity so in very pure material it may be appropriate to replace Eq. (7), which holds only for partially compensated material, with an equation involving only the majority impurity concentration. Silicon crystals intentionally doped with gold and copper to $5 \times 10^{15}$ atoms/$cm^3$ were also investigated. The results supported the conclusion that the photoconductivity structure observed in the undoped material was due to gold and copper.

The previous equations and experimental results indicate the complexities of determining deep impurity concentrations from photoresponse and photoconductivity measurements. There is no circumstance in which the deep impurity concentration can be obtained without knowing the density of compensating impurities. Since the applicable equations depend on the situation, it is necessary to know whether the deep impurity acts as an acceptor, donor, or both, and to know its concentration relative to that of the shallow impurity. The equations were written for a single deep impurity in the presence of a single shallow impurity. The influence of additional impurities can be readily included, but there remains the practical problem of estimating the concentrations of these impurities so that their effect on the impurity under study can be determined.

# 4. Summary of Other Methods for Determining Deep Impurities

Since photoconductivity spectra generally do not yield directly the needed information on impurity density, it seems worthwhile to briefly examine other techniques which may be more useful or which can supplement or confirm information obtained by photoconductivity.

Deep impurities introduce recombination centers which reduce the carrier lifetime. For example gold acts as a recombination center in silicon with an observable room temperature effect on lifetime in both $n$- and $p$-type silicon at the $10^{12}$ atoms/cm$^3$ level [15]. At low temperatures even fewer impurity atoms may influence the lifetime in pure single crystals. Experimental data show the expected inverse linear relation between lifetime and recombination center density for center densities covering many orders of magnitude. Thus, for a single impurity, if the capture cross section is known, the center density can be calculated. From lifetime measurements at one temperature it is not possible to identify the specific impurity involved. However, when measurements are made as a function of temperature, it is possible to obtain the position of the recombination level in the band gap from the slope of the lifetime vs. temperature plot as discussed by Streetman [16]. Since ionization energies of most deep impurities in silicon and germanium are well known, it is often possible to correlate an observed energy with a specific impurity. The photoconductive decay (PCD) method is commonly used for lifetime measurements on bulk specimens. Two ohmic contacts are generally used to pass current through the specimen, but contactless techniques can also be used. Ryvkin [3] has discussed the determination of the impurity concentration from the initial slope of the photoconductivity rise curve.

The diode recovery method has been popular for measuring lifetime in $p$-$n$ junction structures. In this method, a diode which is forward biased is abruptly reverse biased. The time before the onset of the normal reverse current is called the recovery time and can be related to the lifetime. There is an inverse linear relation between reverse recovery time and deep impurity concentration in many systems. A good example is gold-doped silicon [17].

Sah and co-workers [18] have described capacitance-voltage and transient capacitance experiments with and without light which give information on deep impurities. Concentrations as low as $10^{11}$ atoms/cm$^3$ were said to be detectable. The C-V measurements allow the determination of impurity concentration as a function of position if data are obtained on a control specimen with no impurities and on a specimen with a known impurity density.

Armantrout [19] has explored an infrared photoresponse technique for determining impurity ionization energies and concentrations in a

lithium compensated germanium detector. From a plot of photoresponse as a function of incident radiation energy, it may be possible to identify various impurities and defects because of their characteristic spectra. No quantitative relations are given between the photoresponse signal and the concentration of defects and impurities.

Another method for determining deep centers is thermally stimulated conductivity measurements. In this technique excess carriers are excited into the traps of a reverse biased *p-n* junction diode, either by light or by a small forward bias. By heating the specimen, the thermally stimulated current is obtained as a function of temperature. Peaks in the spectrum are due to the release of charge carriers from a particular trapping level. The integrated area between the current peak and the dark conductivity is proportional to the trap concentration. This method has detected nickel, silver, copper, and iron in silicon with concentrations of 0.4 to $3 \times 10^{12}$ atoms/cm$^3$ [20].

Hall effect measurements as a function of temperature are another source of information about deep impurities. Concentrations of ionized impurities as low as $10^{11}$ atoms/cm$^3$ can be detected providing the shallow impurity density does not exceed $10^{13}$ atoms/cm$^3$. It is possible to identify a specific deep impurity from a measured ionization energy. However, since several impurities have about the same energy levels, other information may be necessary for the positive identification of a specific impurity.

Electron spin resonance combined with infrared absorption has been used by Kravitz [21] to study deep impurities. Ionization and trapping processes which change the impurity population are detected by monitoring the spin resonance signal of an impurity while simultaneously illuminating the specimen with monochromatic infrared radiation.

Neutron activation analysis is a widely used technique for determination of many elements simultaneously in a specimen. The total chemical concentration is obtained in contrast to the other techniques discussed which determine only the electrically active atoms. Activation analysis is generally non-destructive, but the electrical properties of semiconductors may be affected by the irradiation. Annealing will usually restore the original properties. Activation analysis is very sensitive for many deep impurities. For example, as few as $1 \times 10^{12}$ gold atoms/cm$^3$ can be detected in a one gram specimen of silicon.

## 5. Conclusions and Recommendations

Infrared photoresponse and photoconductivity are useful techniques for identifying specific impurities because the response due to each impurity has a long wavelength cut off associated with the ionization energy of the impurity. When a single species is present in sufficient

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concentration to give a strong signal, the technique gives a result which is easily interpreted. However, if two or more species are present in the same specimen, then identification is more difficult since long wavelength cut offs are frequently not sharp and a number of impurities have about the same ionization energies. For determining impurity concentrations, the techniques are limited to well-characterized situations as the applicable equations depend on the combination of impurities present. Only neutral impurities can be photoionized so information about compensation is necessary if the total deep impurity concentration is desired.

In specimens used for most applications, the deep impurity concentration is several orders of magnitude smaller than the shallow impurity density. If both impurities are acceptors with comparable absorption and capture cross sections, then the signal due to the deep impurity will generally be masked by that due to the shallow impurity. It is assumed that the temperature of the specimen is low enough so neither impurity is thermally ionized. If the shallow impurity is thermally ionized, then the specimen conductivity is high and the fractional change due to photoionization of the deep impurity may be difficult to detect. Likewise, a deep acceptor level compensated by a large concentration of shallow donors would contribute little to the photoconductivity since photoexcitation of the donors would predominate at all wavelengths.

Photoconductivity measurements may be helpful in a research situation as one of several techniques for determining the concentration of deep impurities. As a single technique, photoconductivity appears to have no advantages over several other methods.

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