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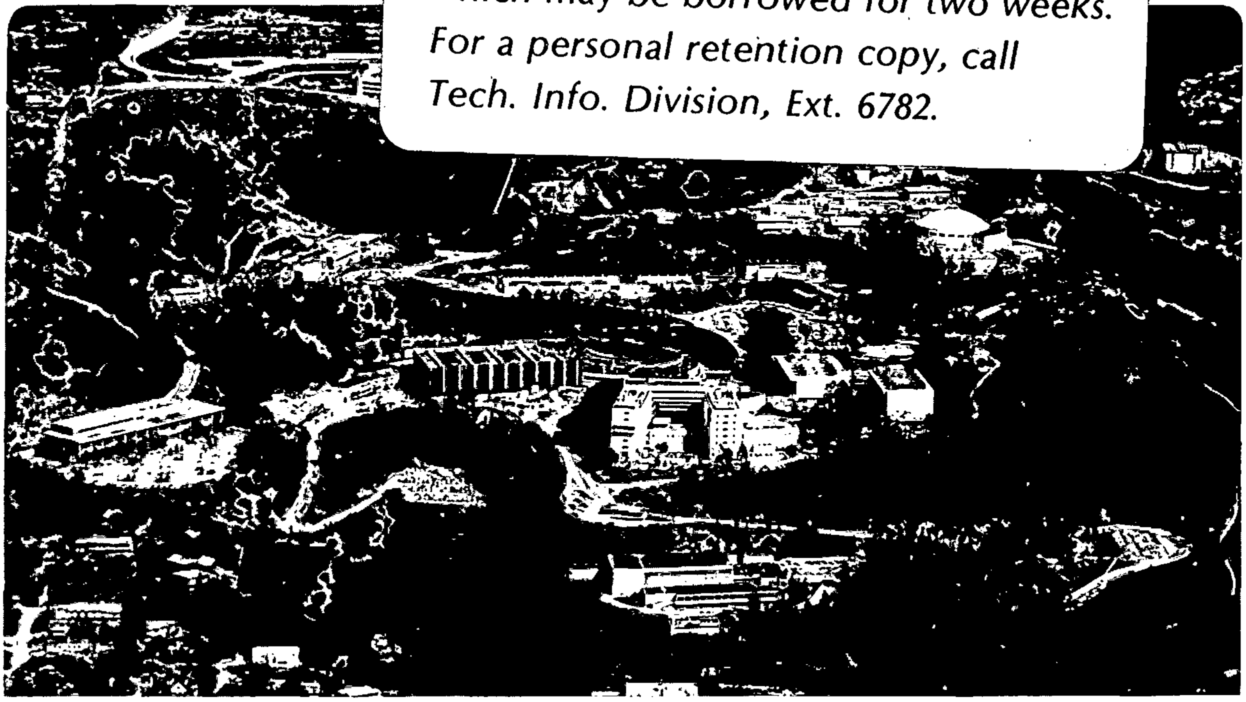

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DISCLAIMER

NTD GERMANIUM: A NOVEL MATERIAL FOR LOW TEMPERATURE BOLOMETERS*

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ABSTRACT

Six samples of ultra-pure ($|N_A - N_D| \leq 10^{11}cm^{-3}$), single-crystal germanium have been neutron transmutation doped with neutron doses between 7.5 x 10^{16} and 1.88 x $10^{18}cm^{-2}$. After thermal annealing at 400°C for six hours in a pure argon atmosphere, the samples have been characterized with Hall effect and resistivity measurements between 300 and 0.3 K. Our results show that the resistivity in the low temperature, hopping conduction regime can be approximated with $\rho = \rho_0 \exp(\Delta/T)$. The three more heavily doped samples show values

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for ρ_0 and Δ ranging from 430 to 3.3 Ω cm and from 4.9 to 2.8 K, respectively. The excellent reproducibility of neutron transmutation doping and the values of ρ_0 and Δ make NTD Ge a prime candidate for the fabrication of low temperature, low noise bolometers. The large variation in the tabulated values of the thermal neutron cross sections for the different germanium isotopes makes it clear that accurate measurements of these cross sections for well defined neutron energy spectra would be highly desirable.

INTRODUCTION

Low temperature semiconductor bolometers have gained in importance as very long wavelength photon power sensors in recent years.[1] The possibility of performing IR astronomy outside the atmosphere from satellites, space probes and the space shuttle has opened the medium and long infrared part of the electromagnetic spectrum. Until recently, the vast amount of information contained in this part of the spectrum has not been actively sought by man. This is in part because the atmosphere does not transmit these photons, but also because man's vision has naturally given great emphasis to the visible light range of the electromagnetic spectrum.

The potential use of doped semiconductors as photon power detectors has been recognized some forty years ago at Bell Labs. The photon absorption in semiconductor material raises the temperature which, in turn, leads to a resistivity change, a parameter which can be measured electrically. The first liquid helium temperature germanium bolometers were designed and operated by Low.[2] These low temperature bolometers have found a wide range of applications because they are sensitive and rugged. One major problem however, has remained unsolved--the reliable fabrication of highly doped and highly compensated semiconductor single crystals bolometer material. This problem becomes more important with decreasing bolometer operating temperature. As we shall show in the following chapter, the interest in going to ever lower operating temperatures persists because of signal-to-noise considerations. The small doping fluctuations which one encounters in all melt-doped and grown crystals[3] become important in the low temperature conduction regime. At temperatures below 1 K, dopant concentration fluctuations of a few percent lead to resistivity fluctuations of more than an order of magnitude. This strong detrimental effect has stimulated the search for improved doping techniques. Ion implantation together with planar technology have been used to fabricate thin silicon bolometers.[4] Though some outstanding bolometers have been fabricated with this technology, one would like to obtain homogenously doped semiconductor single crystals in quantities which allow the fabrication of large numbers of identical bolometers. Further disadvantages of the ion implantation approach are the high level of technology and the excellent control of surface cleanliness which are required. The large number of high temperature processing steps together with the delicate structure of these bolometers are factors which may be responsible for the low yield of useful devices obtained wth this technology. In contrast, neutron transmutation doping of germanium yields bulk-doped single crystal material. A few low temperature processing steps which will be described in a later chapter yield large numbers of identical bolometers.

The low temperature resistivity dependence of neutron-transmutation-doped germanium has been explored by Fritzsche[5] over twenty years ago but surprisingly, nobody has published the application of this material to low temperature bolometer fabrication.

SEMICONDUCTOR BOLOMETERS

The thermal equivalent circuit of a low temperature bolometer is illustrated in Fig. 1. The conservation of energy requires that the photon power which is absorbed in the bolometer $\eta\bar{P}$ is equal to the heat flowing through the thermal link of thermal conductance G (typically a very fine wire) and the heat required to warm up the bolometer $H(d\Theta/dt)$:

$$\eta\bar{P} = H(d\Theta/dt) + G\Theta \qquad (1)$$

The temperature of the bolometer in the dark is T and Θ is the bolometer temperature increase under illumination. An additional term which can be neglected at low temperature is the energy dissipated by the bolometer through radiation. Typically the incident power consists of a constant background part P_0 and a signal part

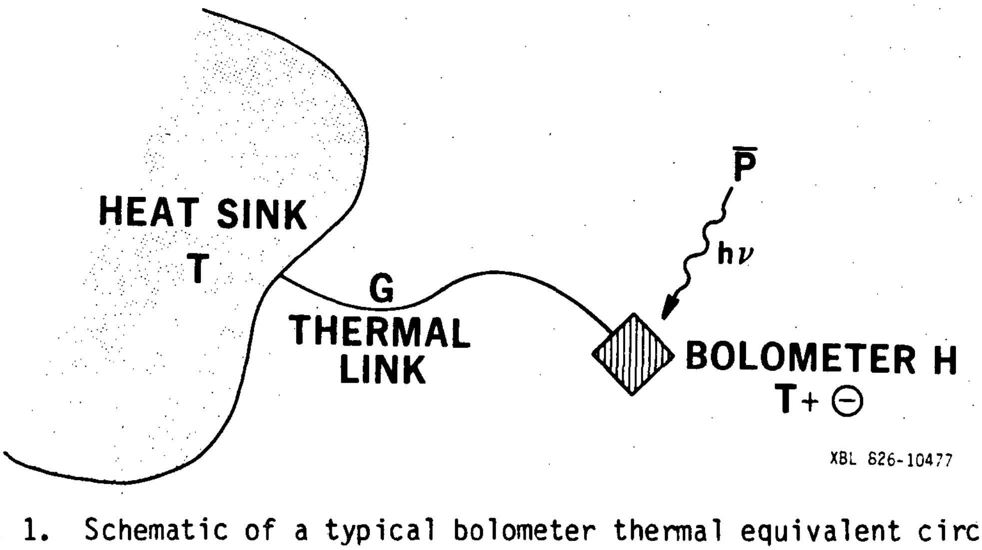

Fig. 1. Schematic of a typical bolometer thermal equivalent circuit.

$P_S = A \exp(i\omega t)$. Solving Eq. (1) for Θ_S we find:

$$\Theta_S = \eta A \, (G^2 + \omega^2 H^2)^{-\frac{1}{2}} \; . \exp(i\omega t + \psi) \qquad (2)$$

with the phase shift :

$$\psi = \arctan\,(\omega H/G) \qquad (3)$$

In order to get a large signal temperature fluctuation one wants to minimize G and ωH. The thermal response time constant τ can be defined as the inverse of ω:

$$\tau = \omega^{-1} \qquad (4)$$

at

$$G = \omega H \qquad (5)$$

It follows that:

$$\tau = H/G \qquad (6)$$

Equation 6 shows that H should be made as small as possible, certainly much smaller than G in order to get short response times and the highest possible signal temperature fluctuation Θ_S. The electrical circuit for the measurement of the resistivity fluctuations caused by the temperature fluctuations is shown in Fig. 2. A constant current I leads to a voltage V across the bolometer. The signal voltage V_S is caused by the resistance fluctuation δR. The DC component of the voltage drop across the bolometer is separated from the signal part by a capacitor C. Using the conventional definition of the temperature coefficient α:

$$\alpha = \frac{1}{R}\,(dR/dT) \qquad (7)$$

we find for V_S:

$$\begin{aligned} V_S &= I \, \alpha \, R \, \Theta_S \\ &= I \, \alpha \, R \, \eta \, P_S \, (G^2 + \omega^2 H^2)^{-\frac{1}{2}} \qquad (8) \end{aligned}$$

The responsivity RESP, a figure of merit for a bolometer is given by:

$$\text{RESP (V/W)} = V_S/P_S = \eta I \alpha R (G^2 + \omega^2 H^2)^{-\frac{1}{2}} \qquad (9)$$

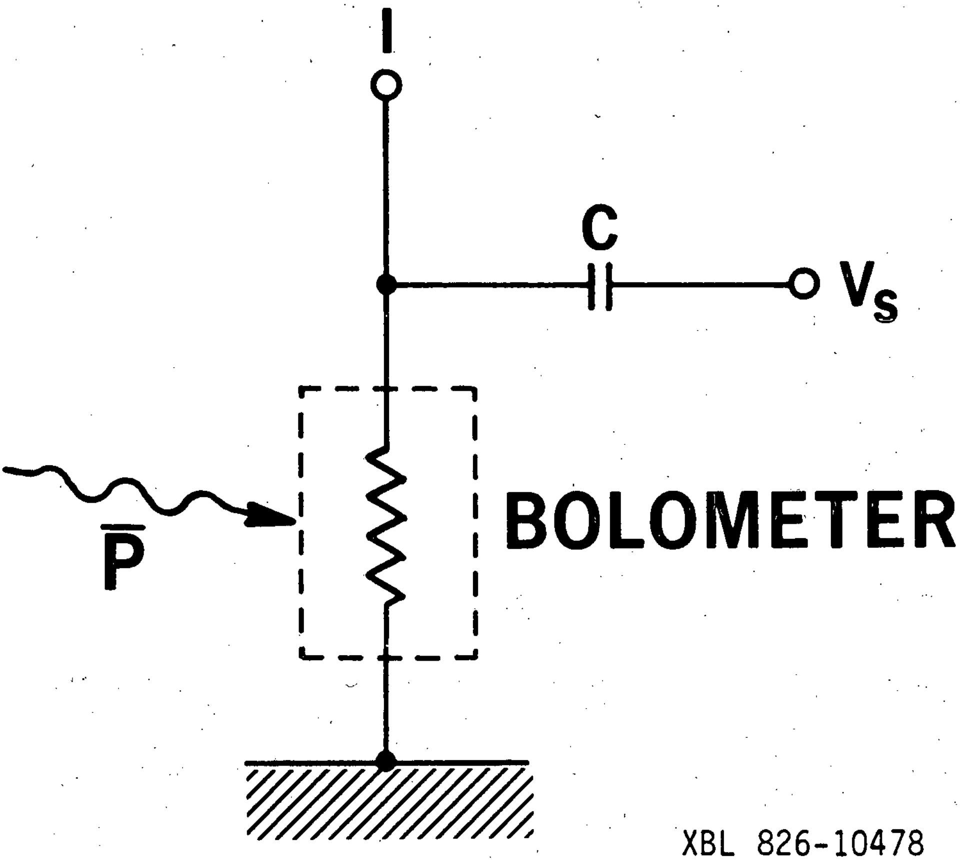

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Fig. 2. Electrical circuit for bolometers.

Contrary to the simple interpretation of Eq. (9), one obviously cannot choose I and R to be any large value. What we have neglected is the fact that the current through the bolometer leads to an internal source of power:

$$P_{int.} = I^2R \qquad (10)$$

The proper current I is chosen so that RESP becomes a maximum. Furthermore, a careful analysis of all the noise sources discloses that the optimum electrical amplifiers show the lowest excess noise when they operate with a source input resistance of 10^5 to $10^7 \Omega$. It is not the aim of this paper to review all the aspects of the complicated and interrelated noise terms for a given bolometer. Such analyses have been published recently.[6] The foregoing equations have made it sufficiently clear that a large value of α, a small thermal mass H and an optimum resistance between 10^5 and $10^7 \Omega$ are the basic requirements for a high performance low temperature semiconductor bolometer.

We should also mention that the ideal bolometer approaches a fundamental noise limit, the fluctuations which are inherently present in any photon stream. In the Poisson statistics approximation one can describe the fluctuation ΔN in the photonstream N by:

$$\Delta N = N^{\frac{1}{2}} \tag{11}$$

with

$$N = \overline{P}/h\nu \tag{12}$$

and for a detection system of band width B and of quantum efficiency η, we obtain:

$$\Delta P = (h\nu\overline{P}B/\eta)^{\frac{1}{2}} \tag{13}$$

Equation 13 describes the photon fluctuations in a photonstream in terms of power fluctuations.

ELECTRICAL CONDUCTION MECHANISMS IN SEMICONDUCTORS

Four mechanisms which lead to electrical conduction in a semiconductor crystal are schematically illustrated in Fig. 3. Mechanism 1 shows the thermal generation of electrons and holes across the bandgap. In silicon and germanium this term becomes negligible at low temperatures where $kT << E_{gap}$. Mechanism 2 illustrates the generation of free charge carriers (in this particular example--electrons) by ionization of shallow donors. This mechanism can also be neglected at very low temperatures because $kT << E_C - E_D$. Mechanisms 3 ("banding") and 4 ("hopping") occur in semiconductors which are heavily doped, and heavily doped and compensated respectively. The charge carrier moves from one impurity to the next without reaching the band. Mechanism 3 is characterized by an impurity concentration which is high enough to lead to substantial overlap between neighboring wave functions. The individual impurity states form an impurity band. The average interimpurity distance and its fluctuations are the critical parameters. The transition at very low temperatures from the insulator regime at large interimpurity distances to the metallic conduction regime at small interimpurity distances occurs abruptly. It is called the metal-insulator transition[7,8] or somewhat incorrectly, Mott-transition. Hopping conduction (mechanism 4) occurs when compensating or minority impurities create a number of majority impurities which remain ionized down to T = OK. In this case, charge carriers can "hop" from an occupied majority impurity site to an empty site. It is this mechanism which has been proven to be useful for low temperature bolometer applications. As in the case of "banding" conduction, the interimpurity distance and its fluctuations are of

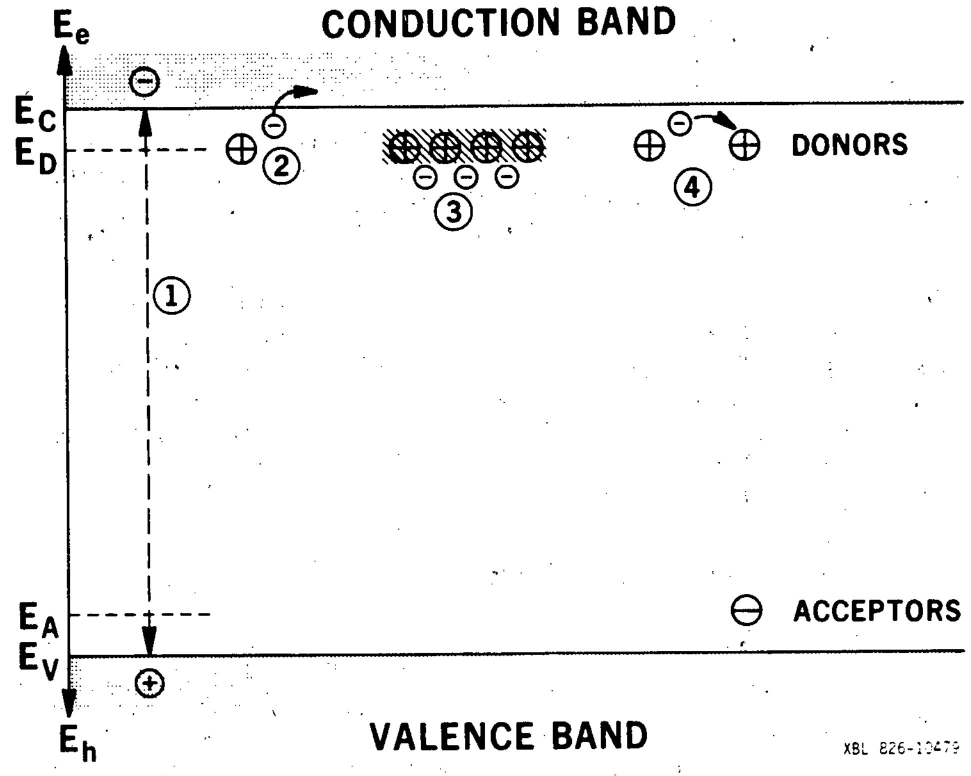

Fig. 3. Electrical conduction mechanisms in semiconductors.

critical importance. It is almost impossible to obtain large crystals with sufficiently good impurity distribution homogeneity by conventional doping techniques. Trial and error have been the approach to solving the problem. Extensive and costly testing of many single crystal samples has yielded some useful material which is carefully guarded by the owner. In view of the fact that Fritzsche and coworkers have used neutron-transmutation-doped germanium to study the fundamental conduction mechanisms in semiconductors[5] already in the early 60's , it is surprising that to our knowledge, there exists no record in archival journals of the use of NTD Ge for low temperature bolometer fabrication.

NEUTRON TRANSMUTATION DOPING OF ULTRA-PURE GERMANIUM

The advantages of neutron transmutation doping over conventional doping techniques have been discussed in great detail.[9,10] For the specific application of NTD Ge discussed in this paper, the basic advantage of extreme homogeneity of the dopant distribution becomes more important than in typical NTD Si applications. To illustrate this point it may be useful to mention that near the onset of "hopping" conduction, an increase in doping concentration of slightly more than one order of magnitude leads to resistivity changes at $T = 4$ K of ten orders of magnitude. This shows that not only the homogeneity but also the net-dopant concentration itself is extremely critical.

The transmutation of the stable germanium isotopes via the capture of thermal neutrons is well understood. Table 1 contains all the information relevant to NTD of Ge. The listed values are well known with the exception of the thermal neutron capture cross sections. We quote the values of σ_n of three different sources.[10,11,12]

The information in Table 1 permits the computation of the acceptor and donor concentrations for a known neutron exposure. Not only are these concentrations important but the ratio of the sum of all minority dopants (donors) and the sum of all majority dopants (acceptors) i.e., the compensation K is crucial for the low temperature conduction. For the case of germanium, one obtains K from the following equation:

$$\begin{aligned} K &= (\Sigma\,\text{donors/cm}^3)/(\Sigma\,\text{acceptors/cm}^3) \\ &= (N_{As} + 2N_{Se})/N_{Ga} \end{aligned} \tag{14}$$

The substitutional selenium impurities are double donors providing two electrons for compensation. Therefore they are counted twice in the sum of donors. Using the different values for σ_n, one finds K ranging from 0.322 to 0.405. It would be of great help for both the basic understanding of the hopping conduction as well as for the application of neutron-transmutation-doped germanium as bolometer material, if these cross sections could be accurately evaluated in one or more well characterized nuclear reactors.

In order to obtain the full advantage of NTD, one should choose the purest available crystals as a starting material. Germanium is, in this respect, ideally suited for NTD because it can be purified today down to concentrations of $\lesssim 10^{11}\text{cm}^{-3}$.[14] Such a low concentration is negligible when compared with the dopant concentrations after NTD in the low 10^{16}cm^{-3} range. The concentrations of electrically neutral impurities such as hydrogen, carbon, oxygen and silicon can be as high as 10^{14}cm^{-3}. Of all the isotopes of these impurities only ${}^{30}_{14}\text{Si}$ transmutes to an electrically active impurity, phosphorus, a shallow donor. With only one silicon atom in every 4.4×10^8 Ge atoms and only 3% of all Si atoms being ${}^{30}_{14}\text{Si}$ which has a neutron capture cross section much smaller than the Ge isotope cross sections, we can estimate that less than one phosphorus donor is produced for every 10^{11} Ga majority acceptors during the NTD process. In other words: ultra-pure germanium crystals are virtually perfect starting material. For the NTD experiment, we have chosen an ultra-pure Ge single crystal which we have grown at our crystal growth facility at the Lawrence Berkeley Laboratory.[15]

TABLE 1.

Isotope	Abundance (%)	Neutron Capture Cross Sections (barn)			Neutron Capture and Decay Reactions	Dopant Type
		(11)	(12)	(13)		
${}^{70}_{32}\mathrm{Ge}$	20.5	3.4	3.2	3.25	${}^{70}_{32}\mathrm{Ge}(n,\gamma){}^{71}_{32}\mathrm{Ge} \xrightarrow[11.2d]{EC} {}^{71}_{31}\mathrm{Ga}$	P
${}^{72}_{32}\mathrm{Ge}$	27.4	0.98	1.0	1.0	${}^{72}_{32}\mathrm{Ge}(n,\gamma){}^{73}_{32}\mathrm{Ge}$	
${}^{73}_{32}\mathrm{Ge}$	7.8	14.0	14.0	15.0	${}^{73}_{32}\mathrm{Ge}(n,\gamma){}^{74}_{32}\mathrm{Ge}$	
${}^{74}_{32}\mathrm{Ge}$	36.5	0.62	0.5	0.52	${}^{74}_{32}\mathrm{Ge}(n,\gamma){}^{75}_{32}\mathrm{Ge} \xrightarrow[82.8m]{\beta^-} {}^{75}_{33}\mathrm{As}$	n
${}^{76}_{32}\mathrm{Ge}$	7.8	0.36	0.2	0.16	${}^{76}_{32}\mathrm{Ge}(n,\gamma){}^{77}_{32}\mathrm{Ge} \xrightarrow[11.3h]{\beta^-} {}^{77}_{33}\mathrm{As} \xrightarrow[38.8h]{\beta^-} {}^{77}_{34}\mathrm{Se}$	n

Our ultra-pure Ge crystals are typically grown for nuclear radiation detectors i.e., large volume p-i-n junctions which are operated at liquid nitrogen temperature.[16] The specific crystal which was chosen for NTD (LBL #516) has been grown in a hydrogen atmosphere (1 atm) from a melt contained in a pyrolytic carbon-coated quartz crucible using the Czochralski technique.[17] Six 2 mm thick slices of 36 mm diameter were cut, lapped and chemically etched. They were irradiated with thermal neutrons at the University of Missouri Research Reactor facility[18] with neutron doses ranging from 7.5 x 10^{16} to 1.88 x $10^{18} cm^{-2}$. After more than ten half lives of $^{71}_{32}Ge$ ($T_{1/2}$ = 11.2d), the samples were annealed at 400°C for six hours in a pure argon atmosphere (1 atm).

ELECTRICAL MEASUREMENTS

A few small square specimens (7 x 7 mm^2) were cut from each slice. The Van der Pauw geometry[19] was chosen for Hall effect and resistivity measurements. This geometry is very insensitive to the shape, the position and the size of the electrical contacts. In order to obtain degenerately doped p^{++} contacts on all four corners of both faces, we used the well established contact fabrication technology which was developed for Ge:Ga photoconductors.[20] These contacts are doped so highly that they are truly metallic and can inject and extract holes down to the lowest measurement temperatures. The following is an abbreviated description of the contact fabrication process.

Both square faces were implanted with 25 and 50 keV boron ions at doses of 10^{14} and 2 x $10^{14} cm^{-2}$ respectively. Annealing at 250°C for one hour was followed by RF sputtering of 400 Å of Ti and 8000 Å of Au. The stress in the metal film was relieved by a second annealing step at 220°C for 0.5 hours. Areas of ~2 - 3 mm^2 were covered with an etch resistant wax in all four corners on both faces. The Au layer was etched off with a KI + I_2 solution. The Ti layer was removed with a brief soaking in 1% HF in water. The implanted layer was removed by a 20 sec etching cycle in a HF:HNO_3 (1:3) mixture. After washing off the wax in trichloroethylene and rinsing in transistor grade methanol, the samples were ready for measurement.

Resistivity (ρ) and Hall effect (R_H) measurements were performed in a liquid helium flow type cryostat.[21] The results of both measurements yield the mobility μ:

$$\rho = (p\mu e)^{-1} \quad \text{(p = free hole concentration, e = charge of the electron)} \tag{15}$$

$$R_H = (pe)^{-1} \tag{16}$$

$$\mu = R_H/\rho \tag{17}$$

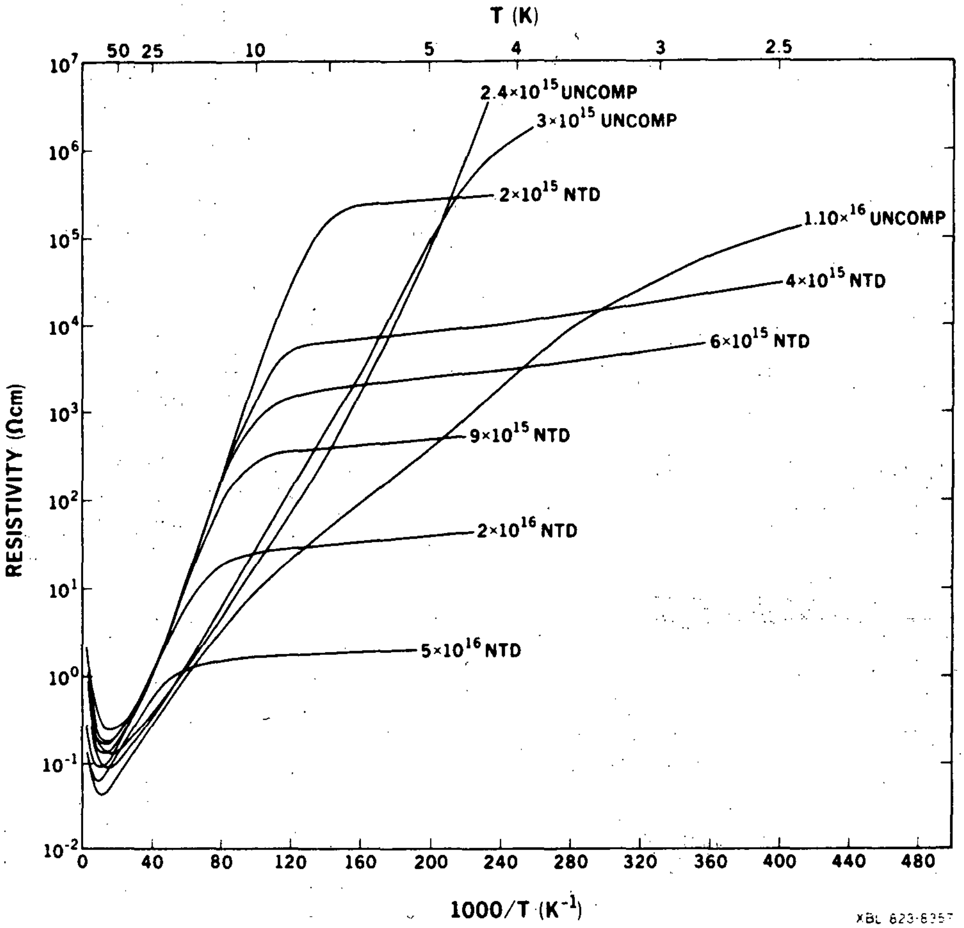

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Fig. 4. Resistivity as a function of 1000/T for NTD and uncompensated Ge samples.

The mobility values are only useful down to the temperature where hopping conduction sets in. Our mobility values agree with published values in the temperature range above the hopping regime very well which means that the concentration of residual radiation damage or other free carrier scattering centers must be very small. Figure 4 shows the log (resistivity) versus 1/T dependence for the six NTD Ge samples. The number next to each NTD curve corresponds to the acceptor (Gallium) concentration in each sample. For comparison we have measured gallium doped Ge samples which were exhibiting extremely small values of K. These so-called uncompensated samples were cut from crystals which were doped in the melt and were grown in the ultra-pure germanium crystal growing equipment. The compensating donor concentration in these crystals is estimated to be less than 10^{11} to $10^{12} cm^{-3}$. The resulting K is of the order of 10^{-4} to 10^{-5}.

The resistivity-temperature dependence of the NTD samples is characterized by three regimes. At high temperatures (room temperature down to ~50 K), the resistivity decreases because the carrier mobility increases. Below ~50 K carrier freeze out begins and reduces the free hole concentration rapidly. The slope of the freeze out in highly compensated material (as NTD Ge is!) is proportional to the acceptor binding energy $E_A - E_V \cong 11$ meV. Below the onset of hopping conduction the resistivity increases only very slowly. All six NTD Ge samples show these three resistivity regimes very clearly. The low compensation samples show different log (ρ) versus 1/T dependences. In these samples the concentration of ionized acceptors is low, and hopping from neutral to ionized centers is improbable. A third conduction mechanism has been proposed for such material.[8] It is based on the idea that carriers can "hop" from a neutral to a neighboring neutral acceptor thereby forming a positively charged acceptor. The carrier binding energy for such positive acceptors is very small, leading to the characteristic conduction range between 10 and 3.5 K in the sample 1.1×10^{16} uncomp.

The samples which show a resistivity dependence which is of interest to bolometer applications are the three most highly doped NTD crystals. They were measured to temperatures as low as T ~ 0.3 K. Figure 5 shows the same dependence as Fig. 4 but the 1/T axis is about tenfold compressed. The resistivity can be expressed empirically with the following equation:

$$\rho = \rho_0 \exp(\Delta/T) \qquad (17)$$

The following values for ρ_0 and Δ have been fitted to the experimental curves:

NTD Sample	$\rho_0(\Omega\text{cm})$	Δ(K)
9×10^{15}	430.0	4.9
2×10^{16}	34.0	4.4
5×10^{16}	3.3	2.8

Depending on the desired bolometer operating temperature, the bolometer dimensions and the optimum resistance value, one can virtually produce the ideal doping concentration for any case. So far, NTD sample 5×10^{16} has been used for the fabrication of bolometers. These devices have been field tested and have been found to work as reliably as any conventional bolometer. The noise for these field tests was limited by effects not related to the bolometer. Critical tests on the noise as a function of the bolometer measuring current have not been performed so far but are planned for the near future.

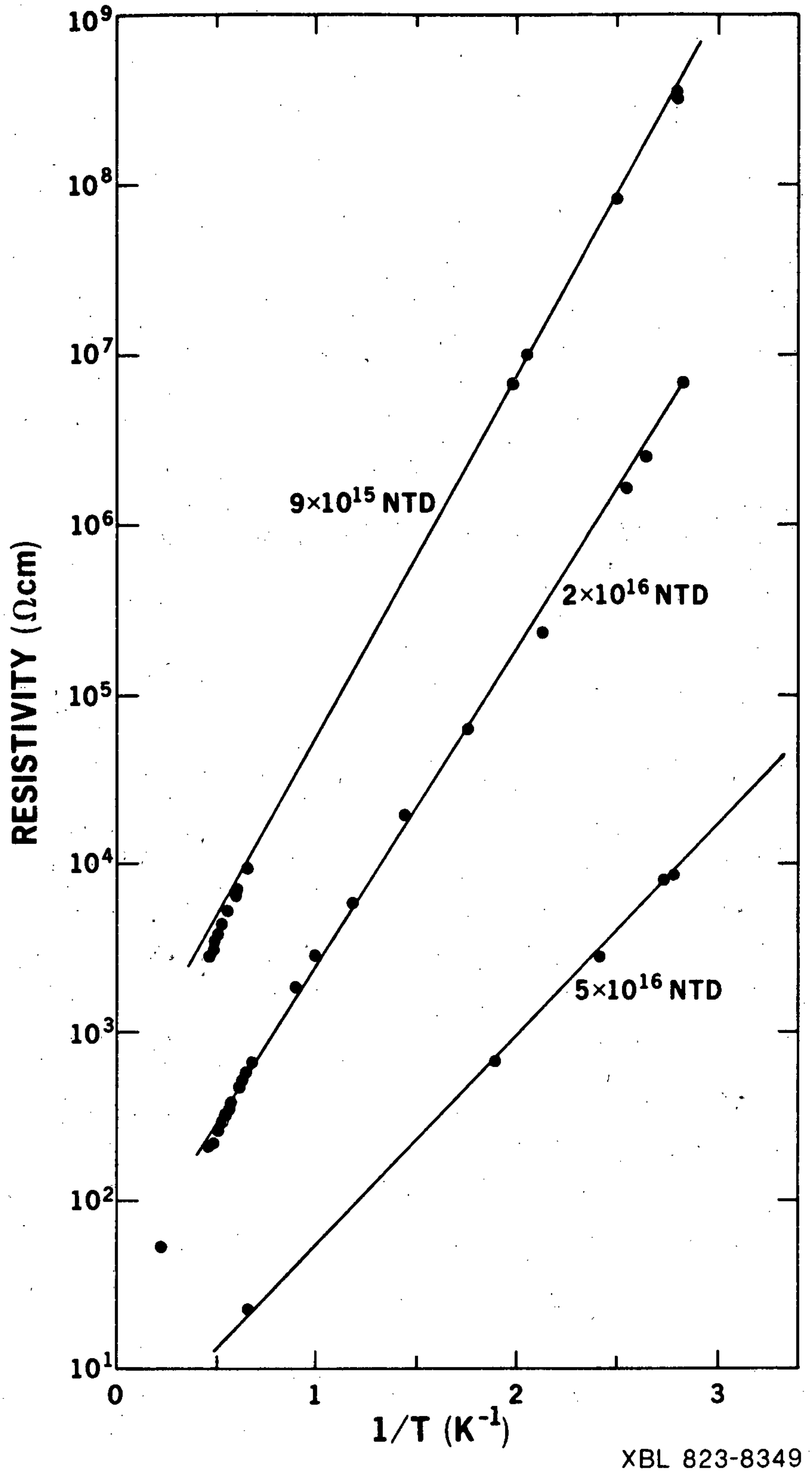

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Fig. 5. Resistivity as a function of 1/T for the most heavily doped NTD Ge samples.

CONCLUSIONS AND SUMMARY

Neutron transmutation doping seems to be the ideal doping technology for germanium which is used for low temperature bolometers. The excellent homogeneity of the doping concentration produced by NTD is for this particular application even more important than in the typical application of NTD Si for high-voltage, high power devices. In the low temperature range conduction mechanisms begin to dominate which rely on quantum mechanical tunneling (i.e., "hopping") of charge carriers from one acceptor site to an empty neighbor site. The tunneling probability is exponentially dependent on the interimpurity distance which explains the extreme sensitivity of hopping conduction on the homogeneity of the dopant distribution.

The NTD process leads to a fixed compensation which in turn results in a certain slope of the log (ρ) versus 1/T dependence (see Fig. 5) for a given neutron exposure. This slope represents the temperature coefficient α of the material. Using Eq. (7) and (17) we find:

$$\alpha = \frac{1}{R}\,(dR/dT) = \frac{1}{\rho}\,(d\rho/dT) = -\Delta/T^2 \qquad (18)$$

Because the responsivity is directly proportional to α (Eq. 9), we see that a large value of Δ and a small value of T are desirable. The Δ values are a factor of 1.5 to 2.0 smaller than the values of the best bolometer material for T = 4.2 K applications. The noise equivalent power--the most important figure of merit--depends only on the square root of Δ which renders the difference between NTD material and the conventional material negligible. At lower temperatures, the difference vanishes and at the lowest temperatures no conventional material has been found which would be useful. These facts together with the possibility of producing reliably large quantities of predictably, homogenously doped Ge using the NTD process seem to have created a good solution to the fabrication of low temperature bolometer material.

The two tasks which remain to be performed for NTD Ge are on the one hand, the accurate determination of the thermal neutron cross sections and on the other hand, the fine tuning of the NTD process for the production of material which is ideally suited for several typical bolometer operating temperatures (e.g. 3, 1.5, 0.3 and 0.1 K). A careful series of noise tests have to be performed at these temperatures as a function of excitation current I in order to determine if there are any excess noise sources present in this material.

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