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NEUTRON-TRANSMUTATION-DOPED GERMANIUM BOLOMETERS*

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Abstract

Six slices of ultra-pure germanium were irradiated with thermal neutron fluences between 7.5×10^{16} and $1.88 \times 10^{18} \text{ cm}^{-2}$. After thermal annealing the resistivity was measured down to low temperatures ($< 4.2\text{K}$) and found to follow the relationship $\rho = \rho_0 \exp(\Delta/T)$ in the hopping conduction regime. Also, several junction FETs were tested for noise performance at room temperature and in an insulating housing in a 4.2K cryostat. These FETs will be used as first stage amplifiers for neutron-transmutation-doped germanium bolometers.

Keywords: Bolometers, Neutron-transmutation-doping, germanium detectors

Introduction

Low temperature ($\leq 4.2K$) semiconducting bolometers, used as infrared detectors in astronomy and spectroscopy, are commonly made from germanium which is highly doped and highly compensated. The traditional method of producing this germanium involves the addition of dopant impurities to the melt prior to Czochralski crystal growth. The distribution of impurities in the resulting crystal is defined by the effective segregation coefficient and the impurity concentration in the melt. In general, impurity atoms have a higher concentration in the liquid phase than in the adjacent solid; therefore, as the crystal is pulled, impurity atoms are continually rejected into the melt, leading to a positive compositional gradient from head to tail of the crystal. Superimposed on this gross segregation are local compositional fluctuations resulting from the dynamics of crystal growth. Oscillations in growth rate, non-planar solidification front, and convection stirring of the melt all lead to longitudinal and radial inhomogeneities of up to 50% on the micron to millimeter scale (1). As will be shown, the electrical resistivity at low temperatures is a strong function of net dopant concentration as well as compensation (ratio of minority to majority dopants). Hence, the described segregation of dopants in melt doped semiconductors makes it extremely difficult to select bolometer material which possesses the doping condition yielding an optimum resistivity. As a result, extensive trial and error testing of the crystal is required, often yielding only a small portion of usable material. It is clear that a method of incorporating dopants producing a predictable, uniform, and reproducible distribution of these impurities would be advantageous. Neutron-transmutation-doping (NTD) provides an alternative to melt doping by meeting all the essential requirements of semiconducting bolometer material and avoiding the problems resulting from impurity segregation. NTD silicon has been used commercially for over a decade in high-voltage rectifiers and thyristors where uniform dopant distributions are essential for smooth breakdown characteristics (2). Similarly, this well developed neutron irradiation technology may be applied to germanium for use as low temperature bolometer material. When germanium is exposed to a flux of thermal neutrons (~ 25 meV), the five

naturally occurring isotopes partake in capture and decay reactions as shown in Table I. The isotopes of interest are ^{70}Ge , ^{74}Ge , and ^{76}Ge since their decay yields gallium, arsenic, and selenium--all of which are electrically active impurities in substitutional positions in the germanium lattice. The gallium acceptors are produced in excess of the sum of arsenic donors and selenium double donors in a constant compensation ratio:

$$K = \frac{N_{\text{As}} + 2N_{\text{Se}}}{N_{\text{Ga}}} = 0.322$$

defined by the relative abundance and capture cross-section of their parent isotopes. Most importantly, since the parent isotopes are distributed uniformly throughout the crystal, the resulting impurities are also uniformly distributed. Since the number of dopants formed is simply the product of isotopic concentration, neutron capture cross-section, and neutron fluence, the net dopant concentration may be controlled over a wide range by varying the total neutron dose. Thus, the two most critical parameters influencing low temperature conduction in bolometers--impurity concentration and compensation--are entirely controllable and uniform throughout the material. As mentioned, NTD has been used to dope silicon for some time and has also been applied to gallium arsenide (4). The applicable transmutation reactions are shown in Table I. After irradiation, sufficient time must be allowed for the completion of the gallium reaction (10 half lives = 3 months). Also, NTD causes structural damage to the semiconductor crystal arising from knock-on displacement of germanium atoms by fast neutrons and β decay recoil. A simple thermal annealing cycle is sufficient to repair this damage and restore all electrical properties critical to bolometer performance to their pre-irradiation values. The annealing characteristics of neutron induced defects in NTD silicon are well documented (5).

Experimental

Ultra high-purity germanium grown at the crystal growth facility at the Lawrence Berkeley Laboratory was used as starting material for a series of neutron irradiations. This material is ideally suited for NTD since

TABLE I. NTU Reactions for Ge, Si, and GaAs.

<u>ISOTOPE FRACTION</u>	<u>REACTION</u>	<u>CROSS SECTION (b)</u>	<u>t_{1/2}</u>
20.5	${}_{32}^{70}\text{Ge}(n, \gamma) {}_{32}^{71}\text{Ge} \rightarrow {}_{32}^{71}\text{Ga} + \text{K}$	3.25	11.2d
36.5	${}_{32}^{74}\text{Ge}(n, \gamma) {}_{32}^{75}\text{Ge} \rightarrow {}_{33}^{75}\text{As} + \beta^{-}$	0.52	82.8m
7.8	${}_{32}^{76}\text{Ge}(n, \gamma) {}_{32}^{77}\text{Ge} \rightarrow {}_{33}^{77}\text{As} + \beta^{-} \rightarrow {}_{34}^{77}\text{Se} + \beta^{-}$	0.16	11.3h
3.1	${}_{14}^{30}\text{Si}(n, \gamma) {}_{14}^{31}\text{Si} \rightarrow {}_{15}^{31}\text{P} + \beta^{-}$	0.108	2.62h
60.1	${}_{31}^{69}\text{Ga}(n, \gamma) {}_{31}^{70}\text{Si} \rightarrow {}_{32}^{70}\text{Ge} + \beta^{-}$	1.7	21.1m
39.9	${}_{31}^{71}\text{Ga}(n, \gamma) {}_{31}^{72}\text{Ga} \rightarrow {}_{32}^{72}\text{Ge} + \beta^{-}$	4.6	14.1h
100.0	${}_{33}^{75}\text{As}(n, \gamma) {}_{33}^{76}\text{As} \rightarrow {}_{34}^{76}\text{Se} + \beta^{-}$	4.4	26.3h

(All values are taken from Ref. 3)

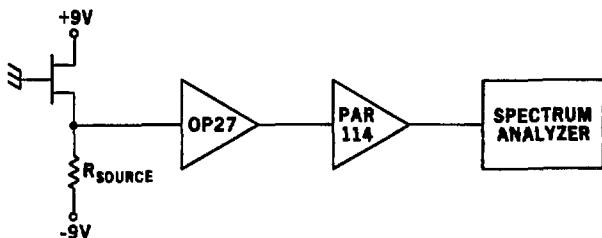
the concentration of residual electrically active impurities is low ($10^{10} - 10^{11} \text{ cm}^{-3}$) compared to the resulting doping levels. Neutral impurities, carbon, oxygen, hydrogen, and silicon, are also present in amounts up to 10^{14} cm^{-3} . Of these neutral impurities, only silicon partakes in a neutron transmutation reaction yielding one phosphorus atom for every 10^{11} gallium atoms produced, a negligible amount. Six slices of ultra high-purity germanium (36 mm in diameter, 2 mm thick) were irradiated with neutron fluences between 7.5×10^{16} and $1.88 \times 10^{18} \text{ cm}^{-2}$ at the University of Missouri Research Reactor (6). After more than 10 half-lives of ^{71}Ge , the material was annealed at 400°C for six hours to repair neutron induced damage. Next, $7 \times 7 \text{ mm}^2$ samples were cut from each slice and prepared in the Van der Pauw geometry (7) for variable temperature Hall effect and resistivity measurements. Ohmic contacts down to low temperature were fabricated by ion implanting boron in high doses to form degenerate p^+ regions. The resistivity of each sample was measured to 4.2K with the three most highly doped samples being measured down to 0.3K. For comparison, three samples doped to different gallium concentrations from the melt ($K \approx 10^{-5}$) were also tested.

In conjunction with development of NTD bolometer material, a low noise amplification system consisting of a cooled ($\sim 70\text{K}$) junction FET in close proximity to the bolometer is being developed. Electrical leads between bolometer and amplifier are the major source of microphonic noise pickup. By stationing the first stage electronics physically close to the bolometer, the output impedance and thus extraneous voltage signals are reduced. This idea has been previously explored by Low (8). Several different types of FETs were tested for their noise performance at room temperature as shown in Figure 1. An FET housing, located adjacent to the bolometer ($\leq 4.2\text{K}$), which keeps the device at an operational temperature ($>60\text{K}$) with a heater resistor was constructed. Two FETs were tested for noise in this housing at low temperatures.

Results

Figure 2 shows the results of variable temperature resistivity measurements for NTD and melt doped (labeled UNCOMP) samples. Each curve is identified by its gallium concentration. NTD samples show three distinct regions of

JFET NOISE MEASUREMENTS



<u>FET</u>	<u>VOLTAGE NOISE @ 10Hz (nV/√Hz)</u>
2N4416	21.0
2N3875	17.6
TCG132	11.4
J230	7.3
2N4867	6.4
2SK147	4.4

XBL 832-8259

Figure 1. JFET noise test circuit.

conduction over the temperature range tested. From room temperature to 50K, conduction is dominated by free holes from ionized gallium acceptors. In this region, resistivity decreases as temperature decreases due to an increase in carrier mobility. Below 50K, free holes begin to freeze out and the resistivity increases with a slope proportional to the acceptor binding energy (11 meV). At lower temperatures, a third conduction mechanism is observed. At these low temperatures, the concentration of free holes is so low that they contribute a negligible amount to conduction. Instead, conduction is accomplished through a thermally assisted hopping mechanism. In hopping, a hole which is bound to an acceptor may travel by tunneling to a neighboring vacant acceptor which has been ionized through compensation. The number of net acceptors

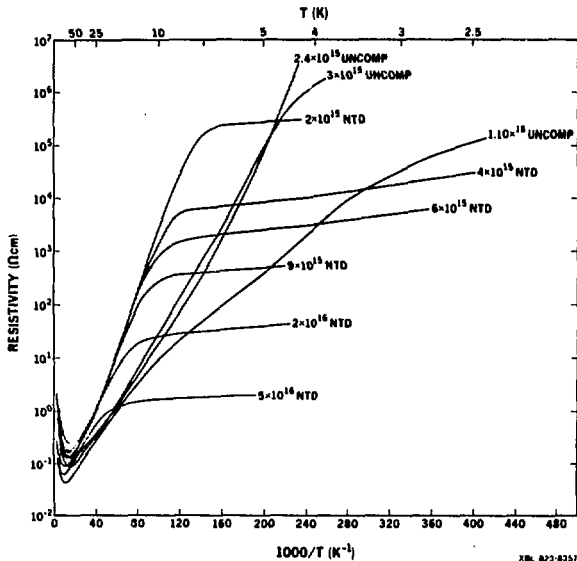
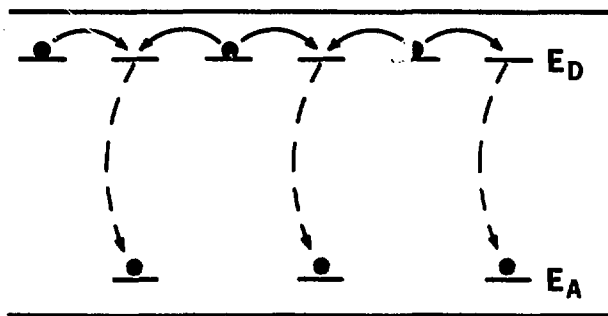


Figure 2. Resistivity as a function of $1000/T$ for NTD and uncompensated Ge samples.

directly affects the number of holes available for tunneling while compensation directly affects the number of sites to which these holes may tunnel. Figure 3 shows this hopping mechanism in the case of donors, for simplicity. This phenomenon has been previously observed in silicon (9), gallium arsenide (10), and NTD germanium (11). The influence of net impurity concentration on conductivity can be appreciated by noting that almost six orders of magnitude of resistivity are spanned by a little more than a ten fold increase in dopant concentration. The effect of compensation can be seen by comparing NTD ($K = 0.322$) and melt doped ($K = 10^{-5}$) samples of similar gallium concentration. The three most highly doped NTD samples were measured for resistivity down to 0.3K as shown in Figure 4.



XBL 836-10207

Figure 3. Hopping conduction mechanism in an n-type semiconductor.

Data for all NTD samples indicated that resistivity in the hopping conduction regime adheres to the temperature relationship:

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

where ρ_0 and Δ are experimentally derived constants listed in Table II for each NTD sample.

TABLE II. ρ_0 and Δ Values for NTD Ge.

SAMPLE	ρ_0 (Ω -cm)	Δ (K)
2×10^{15}	1.4×10^5	8.95
4×10^{15}	4000	6.90
6×10^{15}	1230	6.72
9×10^{15}	430	4.90
2×10^{16}	34.0	4.39
5×10^{16}	3.3	2.82

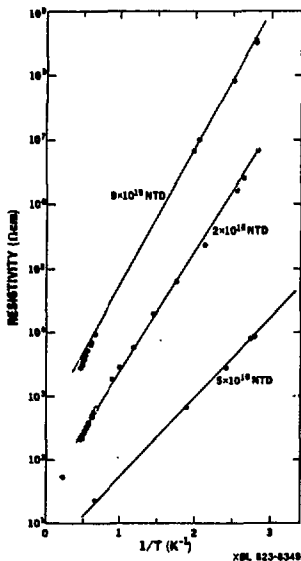


Figure 4. Resistivity as a function of $1/T$ for the most heavily doped NTD samples.

Room temperature noise testing of several types of FETs yielded three device types suitable for use as cooled first stage bolometer signal amplifiers. The Siliconix* J230 and 2N4867, and the Toshiba** 2SK147 possess voltage noise values of 7.3, 6.4, and 4.4 nV/ $\sqrt{\text{Hz}}$ at 10 Hz, respectively. When housed in an insulating package and tested in a 4.2K cryostat, the J230 and 2SK147 measured voltage noise of 5.8 and 6.0 nV/ $\sqrt{\text{Hz}}$ at 10 Hz, respectively. The $1/f$ noise components of each transistor rose considerably when tested in this configuration. The J230 is currently being used in NTD germanium bolometer noise and sensitivity measurements.

*Siliconix Inc., Santa Clara, Ca., 95054.

**Toshiba America Inc., Tustin, Ca., 92680.

Conclusion

Ultra pure germanium may be neutron-transmutation doped at different neutron doses to produce material covering a wide range of low temperature resistivities. Six slices were irradiated in such a manner in order to provide optimum resistivity bolometer material at any temperature between 4.2K and ~0.3K. Six more slices have been irradiated to further fine tune the low temperature resistivity to the optimum value for different cryostat temperatures, 4.2, 1.5, 0.3, and 0.1K. Bolometers have been made from NTD germanium ($5 \times 10^{16} \text{ cm}^{-3}$) and used successfully in astronomical observation (12). Additional studies in progress include bolometer noise measurements as a function of current using the cooled amplifier concept and annealing effects of neutron induced damage in NTD germanium.

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