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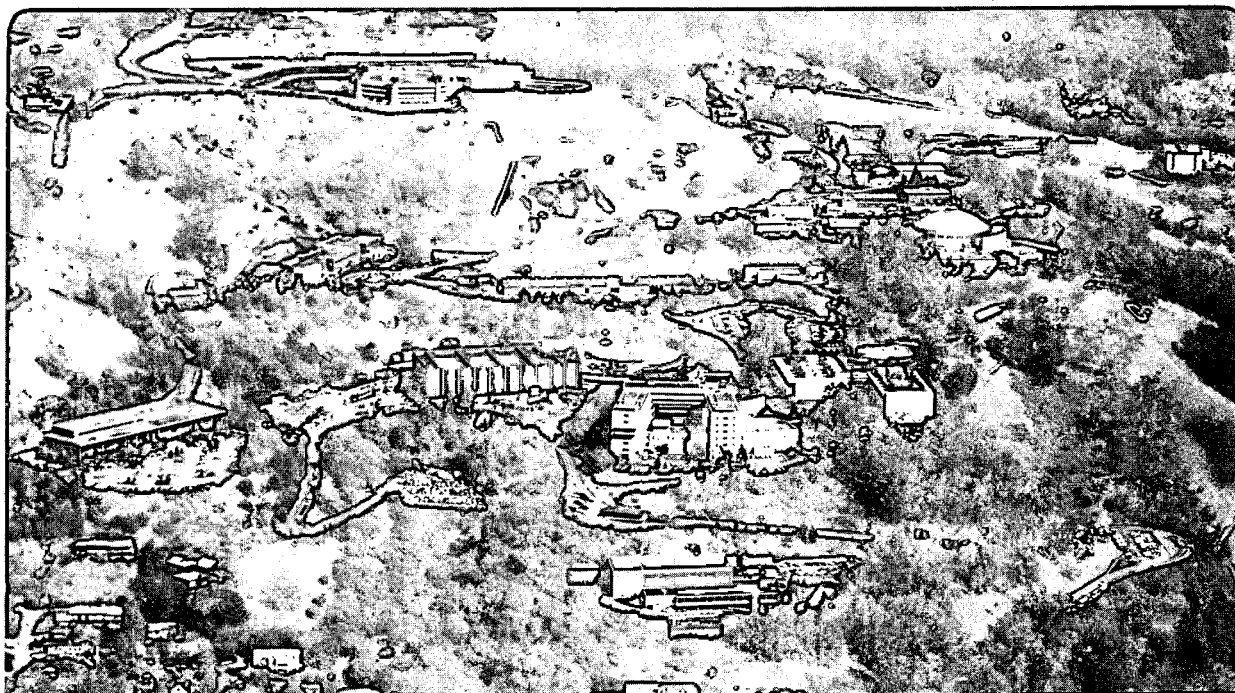
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NEUTRON TRANSMUTATION DOPED NATURAL AND ISOTOPICALLY ENGINEERED GERMANIUM THERMISTORS

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Neutron transmutation doped natural and isotopically engineered germanium thermistors

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ABSTRACT

We report on the development, fabrication and performance of a new class of thermal sensors for far infrared and millimeter wave detection. These devices consist of small single crystal samples of ultra-pure, natural or isotopically engineered germanium which have been doped by the neutron transmutation doping (NTD) technique. The concentrations of the acceptor and donor dopants (N_A, N_D) can be accurately controlled with this technique. They depend on the thermal neutron fluence, the neutron absorption cross sections and the atomic fractions of ^{70}Ge (for the Ga acceptors) and ^{74}Ge (for the As donors), respectively. The values of N_A and N_D and their ratio result in a predictable resistivity of the Ge crystals down to temperatures of a few milliKelvin. The excellent control of the resistivity down to very low temperatures, together with the development of ohmic contacts working at the lowest temperatures, allows the fabrication of high sensitivity bolometer arrays with over 100 pixels and highly uniform response.

1. INTRODUCTION

The development and construction of a number of advanced far infrared and millimeter wave ground based telescopes as well as space borne instruments requires a parallel development of optimal detectors and detector arrays. For direct detection beyond the operating range of photoconductors ($\lambda_{\text{max}} \approx 250 \mu\text{m}$) heavily doped semiconductor chips which exhibit a strong temperature dependence of their resistivity have been used as thermal detectors for the past 30 years.¹ Only recently has the art of making high quality bolometers become a science.^{2,3}

Semiconductor temperature sensors (hereafter called "thermistors") have a number of attractive features. Their electrical resistance can be controlled through doping and geometry over a very wide range of values for temperatures ranging from a few K down to a few milliKelvin. The size of thermistors can be made so small that their thermal mass (heat capacity) can be made comparable to the heat capacities of all the other elements of the detector (bolometer) leading to high sensitivity and good response times.

The principle limitations with semiconductor thermistors are related to the uniformity of the distribution of acceptors and donors in the thermistor chip. Because such sensors are typically operated at rather low temperatures in order to achieve the necessary sensitivity, one relies on electrical conduction mechanisms which are depending exponentially on the interdopant distances inside the crystal. Hopping conduction is the principle mechanism which is used in thermistors working at temperatures below 4 K. In this conduction mode electrons (or holes) tunnel thermally assisted from an occupied (neutral) donor (acceptor) to a nearby unoccupied (ionized) donor acceptor. Much theoretical work has been performed to explain the experimentally observed resistivity versus temperature dependencies. It is surprising and convenient that in all of our Ge thermistors working between 4 K and 10 mK we find the results of Shklovskii and Efros⁴ to describe accurately our experimental findings. The resistivity ρ depends on the temperature T as:

$$\rho = \rho_0 \exp (T_0/T)^{1/2} \quad (1)$$

The constants ρ_0 and T_0 can be easily determined from plots of $\log \rho$ versus $1/\sqrt{T}$.

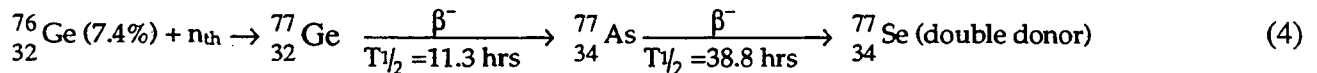
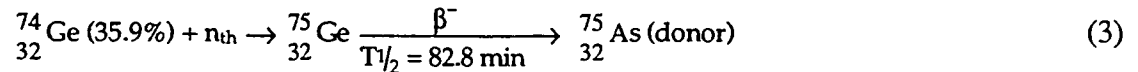
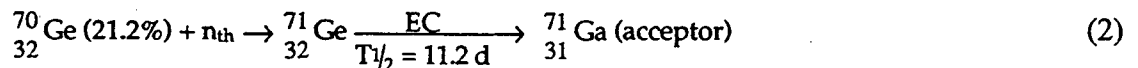
The question then arises as to how one can achieve truly homogeneous doping. The ideal case, which at this point in time is completely out of reach, would be to position the dopants on a regular sublattice of the Ge crystal lattice. This would guarantee accurate inter-dopant distances. The next best solution is a perfectly randomly distributed dopant population with a constant average concentration. Unfortunately there are many processes which lead to deviations from a truly random dopant distribution. Dependence of the dopant impurity segregation coefficient on natural and driven thermal convection during growth of a semiconductor from a melt generates the well known dopant striations.⁵ Interaction between dopants and residual impurities (e.g. oxygen, carbon) or native defects (e.g. vacancies, interstitials) or local temperature fluctuations prevent perfectly homogeneous doping involving thermal diffusion. Ion implantation has achieved the best uniformities which approach fluctuations of $\pm 0.5\%$ over large silicon wafers. Unfortunately, one has to thermally anneal the implanted crystals in order to activate the implanted dopant ions (i.e., the implanted dopants have to move to crystal lattice sites and the lattice damage has to be repaired). The fluctuations in concentration of the implanted dopants and the thermal annealing following often lead to unacceptable dopant fluctuations. Regardless of these problems the development of ion implanted Si microbolometers has made much progress since the early work by Downey et al.⁶ and very high resolution X-ray spectrometers have been fabricated.⁷

A doping technique which relies on the random distribution of the various stable isotopes in natural Ge uses the nuclear transmutation of certain Ge isotopes into dopant elements. This process is described in the following section.

2. NTD OF NATURAL AND ISOTOPICALLY CONTROLLED Ge

The introduction of dopants with NTD was first practiced with semiconductor silicon.⁸ This doping process has evolved into a mature technology. Though not much noticed by the low power electronics community the high power device experts make use of several hundred tons of NTDs per year. High power thermistors and silicon controlled rectifiers are widely used in electric power transmission (AC to DC and reverse conversion) and in the control of large electric traction motors (railways, etc.).^{9,10}

Whereas in Si only one isotope (³⁰Si) present at a rather small atomic concentration (3.1%) can be converted into phosphorus donors, Ge offers a more interesting situation. Out of the five stable isotopes (⁷⁰Ge, ⁷²Ge, ⁷³Ge, ⁷⁴Ge and ⁷⁶Ge), three transmute to dopants after capture of a thermal neutron:¹¹



Only a very small concentration of Se double donors are produced compared to the acceptors and donors described by reactions (2) and (3) respectively. We therefore neglect the generation of Se in germanium of natural isotopic composition.

In order to remove crystal lattice damage produced by unavoidable energetic neutrons and to activate the dopant atoms (i.e., move them to crystal lattice positions), one typically thermally anneals the Ge crystals after NTD and after the radioactivity has cooled down to safe levels at 400°C for 1 hr in an inert gas ambient (Ar, He,

N₂). The first systematic application of NTD Ge to low temperature bolometers has been reported by Haller et al.¹² Park and Haller¹³ have performed a quantitative study on the formation of the Ga acceptors and As donors in NTD Ge. Consistent with earlier radiation damage studies they found that the Ga acceptors are electrically active directly after NTD while the donors are inactive presumably because they form complexes with mobile point defects produced by the fast neutrons. Annealing removes the point defects and the As donors become visible in IR absorption spectroscopy. The University of Missouri Research Reactor facility at Rolla, Missouri, provides accurate thermal neutron doses required for the NTD process.

The end of the cold war has increased the availability of stable, highly enriched isotopes of many elements from Russia. In particular, all the Ge isotopes can be obtained in macroscopic quantities with enrichments up to 95% and higher. Collaborating with Professor V. Ozhogin at the Kurchatov Institute for Atomic Energy in Moscow we have acquired ⁷⁰Ge and ⁷⁴Ge. After chemical and physical purification (zone purification) we have obtained ultra-pure, highly enriched Ge isotopes. We have grown a number of highly enriched, small single crystals of ⁷⁰Ge and ⁷⁴Ge.¹⁴ We have doped these crystals with NTD and characterized them with variable temperature Hall effect and far IR absorption spectroscopy.¹⁵ These studies allowed us to obtain very accurate thermal neutron capture cross sections for our specific NTD conditions. It should be pointed out that these cross sections depend on the thermal neutron spectrum at the irradiation site and for a specific reactor power level. Accurate calibrations of the cross sections for ⁷⁰Ge and ⁷⁴Ge for the specific irradiation conditions are necessary for precision doping. Our interest in isotopically engineered NTD Ge lies in the tunability of certain parameters which control the electrical conductivity at very low temperatures.

3. NTD Ge THERMISTORS AND THEIR APPLICATIONS

We will focus in this section on the temperature sensing part of composite bolometers, the thermistor element. Thermistors of both doped Si and Ge have been used for some time. The very large technology development effort which has been spent on Si may lead one to conclude that this semiconductor material may offer certain advantages over other semiconductors in regard to thermistor fabrication. Unavoidable dopant fluctuations have so far prevented the full realization of these advantages.

As bolometer operating temperatures drop to ever lower values, a fundamental limitation related to the dopant homogeneity in the thermistor crystal rapidly begins to dominate. The change in resistivity with temperature, a measure of the sensitivity of a thermistor, is a consequence of thermally assisted hopping conductivity. Hopping conductivity is due to tunneling processes of charge carriers. Tunneling in turn depends exponentially on the width of the potential barrier through which the carrier tunnels. These distances are closely related to the dopant impurity interdistances. Any fluctuations in the local doping concentrations, equivalent to fluctuations in inter-impurity distance, cause large resistivity fluctuations. All the traditional doping techniques including doping in the melt during crystal growth, ion implantation followed by thermal annealing and thermal diffusion of dopants exhibit inherent homogeneity problems. Very large efforts have been directed at overcoming these doping fluctuations. The only doping technique which overcomes all the problems inherent to the introduction of dopants with standard techniques is the NTD technique which has been discussed in the previous section. Unfortunately Si can only be doped with phosphorus, a shallow donor, via NTD. None of the three stable Si isotopes transmutes into acceptors. Furthermore, the atomic concentration of ³⁰Si which forms P via beta decay is very small, putting the high concentrations necessary for hopping conductivity at sub Kelvin temperatures out of reach.

Ge, on the other hand, can be doped by NTD with acceptors and donors right up to the point where it conducts metallically. The resistivity ρ in NTD Ge is well described by equation (1). Fig. 1 shows a plot of the logarithm of the resistivity ρ as a function of the inverse of the square root of the temperature T for a number of differently doped NTD Ge thermistors.¹⁶ The dashed lines were obtained with Ge crystals of natural composition. Many groups worldwide have reported their measurements to us for the numerous samples of crystals NTD 2 through NTD 25. All these measurements produce this very consistent picture. In some cases it

took several attempts at stress-free mounting of the crystals and at accurately measuring the resistivity at the very low test temperatures before mutual agreement between the various results could be achieved.

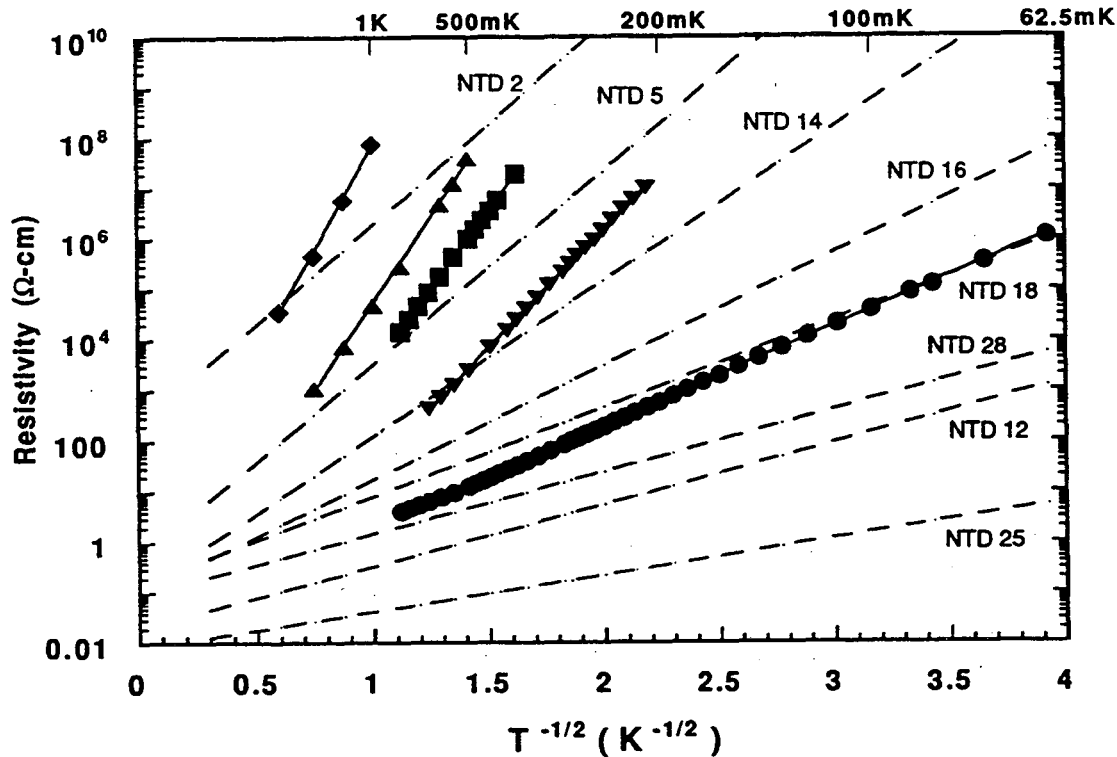


Fig. 1. Temperature dependence of resistivity of various NTD $^{nat}\text{Ge}:\text{Ga}$ and NTD $^{70}\text{Ge}:\text{Ga}$ samples: ^{70}Ge -3.30 (\blacklozenge), ^{70}Ge -2.98 (\blacktriangle), ^{70}Ge -1.90 (\blacksquare), ^{70}Ge -1.65 (\blacktriangledown), and ^{70}Ge -2.15 (\bullet).

In addition to the natural Ge samples we show five curves obtained with isotopically enriched ^{70}Ge crystals doped with NTD. With the exception of the most heavily doped NTD ^{70}Ge sample (filled circles) the isotopically engineered crystals show stronger temperature dependencies indicating higher sensitivity.

The NTD process with natural and isotopically engineered Ge single crystals leads to a completely reliable and reproducible production of thermistor material for temperatures down to a few milliKelvin and possibly lower. Measuring the resistance of a thermistor at such low temperatures, however, becomes increasingly difficult. The coupling between the crystal lattice vibrations and the mobile charge carriers decreases with a high power of temperature. Self-heating and nonlinear effects caused by the electric field used to measure the resistance are further effects which begin to influence accurate measurements at the lowest temperatures.¹⁷⁻¹⁹

A critically important component of high quality thermistors are the electrical contacts. Such contacts must consist of a very thin layer ($\approx 1000\text{\AA}$) of heavily doped material which is metallically conducting and a combination of metal layers which guarantee good mechanical adhesion and chemical inertness. We have developed boron ion implanted contacts for all our p-type thermistors (all natural Ge and ^{70}Ge crystals doped by NTD). Two implants at B^+ ion energies of 30 keV and 50 keV at doses of 10^{14} cm^{-2} and $2 \times 10^{14}\text{ cm}^{-2}$, respectively, followed by thermal annealing at 300°C for one hour lead to boron acceptor concentrations exceeding the insulator to metal transition threshold concentration by close to three orders of magnitude. This large margin of safety has made this type of contact completely reliable. A very thin adhesion layer of Pd (100\AA

to 200Å) followed by a 2000Å to 4000Å thick Au layer form rugged metal contacts. For thermistors operating at 100 mK and lower, the contributions of the Pd and the Au layers to the total heat capacity become significant and their thickness must be carefully optimized.

4. BOLOMETERS AND BOLOMETER ARRAYS USING NTD Ge THERMISTORS

The excellent reproducibility of the NTD process and the ion implanted contact formation has transformed bolometer design and construction with NTD Ge thermistors from an art into a science. Modern composite bolometers can be precisely optimized for their specific application.

The basic design of composite bolometers for far IR and sub mm detection has not changed much over many years²⁰ (Fig. 2). Bolometers operating at 300 mK have been described in detail by Alsop et al.²¹ More recently Tanaka et al.²² have analyzed the operation of a similar composite bolometer at an operating temperature of 100 mK. These composite bolometers typically consist of an absorber (also called antenna) made from a small platelet of sapphire, silicon or diamond with an absorbing metal film (e.g. bismuth). This absorber element is thermally coupled to a low temperature bath with thin brass wire and is mechanically supported by polymer strands (e.g. nylon). The temperature sensor, also called thermistor, is mounted on an absorber element with epoxy. The contact leads consist of metallized, low thermal conductivity graphite leads.

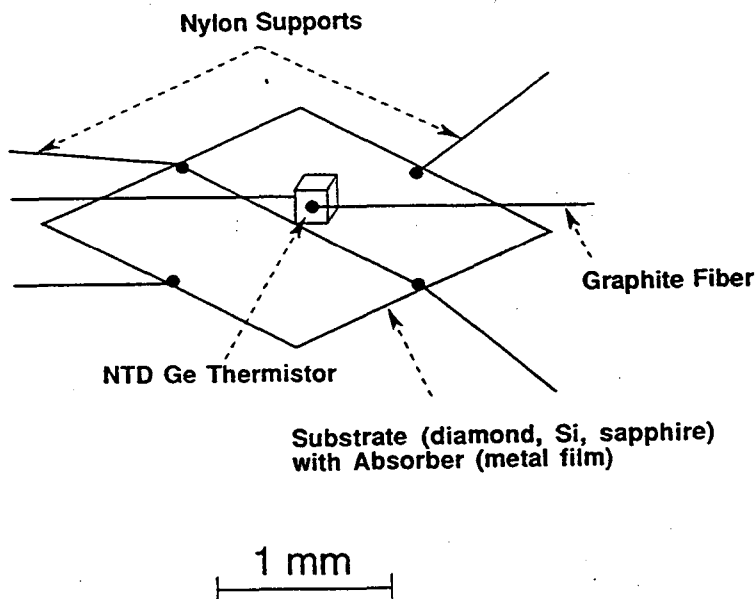


Fig. 2. Sketch of the design of a typical composite bolometer using an NTD Ge thermistor. (Adapted from P.L. Richards, Ref. 2.)

Tanaka et al.²² have reported the following characteristics for individual composite bolometers for a bath operating temperature near 100 mK. The thermistor consisted of a 200 μm cube of our NTD 17 Ge crystal with Boron ion implanted contacts. The responsivity (signal voltage/input photon power) is approximately 10⁹ V/W over a thermal time constant range of 25 to 60 ms. Noise Equivalent Power (NEP) values near 2x10⁻¹⁷ W/√Hz were determined. These values constitute the state-of-the-art in 100 mK composite bolometers.

The reproducibility of NTD Ge thermistors has strongly encouraged the development of bolometer arrays for broad band observations with mm and μm wave telescopes. The first array consisting of seven bolometers which has been used in real observations has been designed, built and described by Kreysa.^{23,24} The beam pattern of the seven bolometer array obtained on the planet Uranus with the IRAM 30 m telescope is shown in Fig. 3. In addition to the expected gains in signal quality, a very large reduction in sky noise was achieved. Sky noise appears in the array as noise highly correlated between the individual bolometers. Removal of all the

correlated noise with a sophisticated computer routine leads to extraordinarily clean signal traces from the individual bolometers.

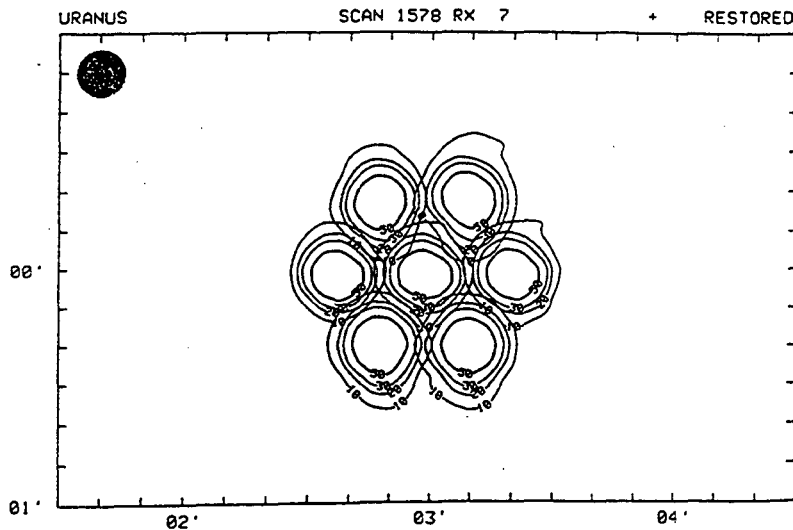


Fig. 3. Beam pattern of the seven bolometer array obtained on Uranus with the IRAM 30 m telescope. Contour levels are in percent of peak intensity. (Courtesy E. Kreysa, Ref. 23.)

A similar seven bolometer array using NTD Ge thermistors has been developed by Kuno et al.²⁵ for use at the Nobeyama 45 m telescope working at 150 GHz.

Perhaps the most ambitious bolometer arrays using NTD Ge thermistors are those under construction for the James Clerk Maxwell telescope on Mauna Kea. These so-called Submillimeter Common-User Bolometer Arrays (SCUBA) consist of a 91 horn array optimized for 438 μm and a second 37 horn array optimized for 855 μm .²⁶ An update on the status of these arrays is given in these proceedings. The designers and builders of SCUBA expect improvements of the order of 10^4 with SCUBA over a single channel bolometer.

A major handicap in the construction of large arrays is the lack of a mass production process. Currently individual bolometers are constructed and tested. They are then combined into an array. It appears feasible to adapt mechanical array and mounting designs developed in silicon micromachining. The very high stability and strength of single crystal silicon together with the highly developed lithography process should provide unique advantages in the design of bolometer arrays. The ultimate goal is an all silicon bolometer array. The difficulties regarding uniformity in doping of the thermistor part appear to remain, however, a serious problem for some time. Hybrid designs using Si as mechanical support, SiN-membranes as thermal links, thin metal strips as contact leads and NTD Ge chips as thermistors are likely to become the next generation of semiconductor bolometer arrays.

5. SUMMARY AND CONCLUSIONS

This paper gives a brief survey of the state of NTD Ge thermistors and their use in high responsivity bolometers and bolometer arrays working at low photon backgrounds and low operating temperatures. We have demonstrated that the NTD process is a well understood doping process which leads to uniform doping simply because the various isotopes of natural Ge have been thoroughly mixed over very long times. The thermal neutron spectrum and the neutron dose lead to accurate dopant concentrations. In natural Ge the donor to acceptor ratio is fixed at ~ 0.32 but isotopically controlled Ge crystals can now be grown which allow us to change

this ratio over a very wide range. NTD of ^{70}Ge crystals leads to thermistor material with larger temperature coefficients than the values obtained with natural Ge.

The formation of ohmic contacts through ion implantation and subsequent metallization with Pd and Au has become totally reliable. Contact noise is no longer observable down to very low electrical frequencies (≤ 0.5 Hz).^{27,28}

The high reproducibility of NTD Ge thermistor fabrication has led to the successful design and fabrication of bolometer arrays. The early results from small arrays are most promising, making us anxious for better results to be reported with the large arrays which are under construction.

6. ACKNOWLEDGMENT

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