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### Neutron Transmutation Doped (Ntd) Germanium Thermistors for Sub-Mm Bolometer Applications

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#### NEUTRON TRANSMUTATION DOPED (NTD) GERMANIUM THERMISTORS FOR SUB-MM BOLOMETER APPLICATIONS

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#### ABSTRACT

We report on recent advances in the development of Neutron Transmutation Doped (NTD) semiconductor thermistors fabricated from Germanium of natural and controlled isotopic composition. The near ideal doping uniformity which can be achieved with the NTD process, the device simplicity of NTD Ge thermistors and the high performance of cooled junction field effect transistor (FET) preamplifiers have led to the widespread acceptance of these thermal sensors in many radiotelescopes operating on the ground, on high altitude aircraft and on spaceborne satellites. These features also have made possible the development of efficient bolometer arrays which are beginning to produce exciting results.

#### 1. INTRODUCTION

Detection of electromagnetic radiation in the mm and near mm wavelength range continues to pose a challenge, especially for high performance applications in low photon backgrounds. The invention of the semiconductor thermistor [1] and subsequently of broadband composite bolometers by Coron [2], which were optimized by Nishioka et al. [3], has led to widespread use of these sensors in recent years on many radiotelescopes.

The introduction of Neutron Transmutation Doped (NTD) Germanium thermistors [4] has put thermistor research and development on a firm scientific and solid engineering footing. This has stimulated the development of bolometer arrays reaching pixel numbers of close to 100. There are no fundamental limitations to further improvement of bolometer performance. Lower operating temperatures, a reduction of the bolometer heat capacity, improved bolometer array mounting schemes and efficient coupling of the radiation to the bolometer are among the major ingredients for higher performance devices.

In the following we will review the NTD process and point out its advantages over conventional doping techniques. The availability of isotopically controlled Germanium offers new opportunities for thermistor design. The major aspects of array developments will be mentioned, but for further details the reader is referred to the relevant papers in these proceedings (W. Gear et al., E. Kreysa et al.). We will not discuss preamplifier issues which are complex and require most careful attention. One can state that in optimized systems the preamplifiers should not limit the performance of single element bolometers or of arrays.

#### 2. THE NTD PROCESS AND THERMISTOR FABRICATION

The low operating temperatures required for high performance bolometers make it necessary to use rather highly doped semiconductors for the sensing element, the thermistor. At typical operating temperatures of below 1K, thermal activation of donor (acceptor) bound electrons (holes) no longer occurs at a rate sufficient to produce carrier concentrations which lead to resistivities of a few M  $\Omega$  cm. Such resistivities are required for the fabrication of optimized thermistors. Rather, the semiconductor becomes an excellent insulator when lightly doped. A new conduction regime is observed when doping in Ge reaches concentrations of  $\ge 10^{15}$  cm<sup>-3</sup>. In this mode, bound carriers hop from one dopant atom to a nearby empty neighbor dopant atom. The probability for hopping to occur depends exponentially on the inter-dopant distance since we deal with a tunneling process.

This distance shows a normal distribution in a truly randomly doped semiconductor. Any fluctuations in the average local dopant concentration (often referred to as "dopant striations" in melt doped crystals [5] ) will lead to a change in the distribution of the inter-dopant distance which in turn will lead to fluctuations in the local hopping probability. Resistivity fluctuations over many orders of magnitude can easily result from these doping striations as has been shown in experiments with Si thermistors [6]. What is required is a doping process which leads to a truly homogenous random distribution of donors and acceptors. Neither melt doping, doping during epitaxial growth, dopant diffusion nor ion implantation, all commonly used semiconductor doping processes, lead to the required uniformity.

The only process which guarantees this homogeneity is based on a process in which individual Ge atoms in a very pure and structurally perfect crystal are transmuted into dopant atoms. Natural Ge is poly-isotopic and of its five stable isotopes (A = 70, 72, 73, 74, 76) three transmute to dopants after they have captured a thermal neutron in their nucleus [7]:

$${}^{70}_{32}\text{Ge} + n \rightarrow {}^{71}_{32}\text{Ge} \xrightarrow{\text{EC}}_{\text{Ti}_{2} = 11.2 \text{ d}} \rightarrow {}^{71}_{31}\text{Ga} + v_{e} \qquad (1)$$

$${}^{74}_{32}\text{Ge} + n \rightarrow {}^{75}_{32}\text{Ge} \xrightarrow{\beta}_{\text{T1/}_2 = 82.8 \text{ min}} \rightarrow {}^{75}_{33}\text{As} + \overline{\nu}_e \qquad (2)$$

$$\frac{76}{32} \text{Ge} + n \rightarrow \frac{77}{32} \text{Ge} \quad \frac{\beta}{\text{Ti}_{2} = 11.3 \text{ hrs}} \rightarrow \frac{77}{33} \text{As} + \overline{\nu}_{e}$$

$$\frac{\beta}{\mathrm{Ti}_{l_2} = 38.8 \mathrm{\,hrs}} \rightarrow \frac{77}{34} \mathrm{Se} + \overline{\mathrm{v}}_{\mathrm{e}} \qquad (3)$$

In reaction (1) Ga acceptors form while reactions (2) and (3) lead to donors and double donors, respectively. Homogeneity of doping is intrinsic to the NTD process because the following three conditions are fulfilled:

- The stable isotopes in natural Ge are randomly distributed because they are chemically identical and none of the standard crystal growth processes are sensitive to the small nuclear mass fluctuations between the various isotopes.
- The thermal neutron sources used in NTD are very large compared to the size of semiconductor crystals.
- The absorption cross sections for neutron capture are very small (a few barns (10<sup>-24</sup> cm<sup>2</sup>)) resulting in negligible "self" shadowing of the neutron flux inside the semiconductor.

Any neutron flux fluctuations vary very slowly with distance resulting in quasi-constant doping through rather large semiconductor crystals. Indeed, the Silicon power device industry uses several hundred tons of NTD Floating Zone (FZ) Si in order to obtain high yields in the production of silicon controlled rectifiers (SCR) and other high voltage devices. [8]

The obvious problems related to NTD are radiation damage and nuclear transformation caused by residual high energy neutrons. The former problem is solved by appropriate thermal annealing (in Ge 400°C for 6 hrs) while the latter is hard to avoid. Fortunately a thorough study by Alessandrello et al. [9] has shown that the production of the radioactive  $^{68}$ Ge and  $^{65}$ Zn is totally negligible for radioastronomy applications of NTD Ge thermistors in composite bolometers. Equations (1)-(3) show that control of the isotopic composition allows the control of the net-doping concentration  $N_A$ - $N_D | (N_A = acceptor concentration, N_D = donor concentration), the compensation <math>(N_A/N_D \text{ for } N_D > N_A \text{ and } N_D/N_A \text{ for } N_A > N_D)$  and the conduction type (n or p). We have obtained highly enriched <sup>70</sup>Ge, <sup>73</sup>Ge, <sup>74</sup>Ge and <sup>76</sup>Ge from the Kurchatov Institute in Moscow and we have grown crystals with various isotopic compositions and studied the dependence of the resistivity on temperature of these isotopically controlled materials [10].



Sample	Hole Conc. $(x10^{16} \text{cm}^{-3})$	K	ρ <sub>0</sub> (Ω cm)	Т <sub>о</sub> (К)
NTD 2	0.03	0.32	200	82.9
NTD 5	1.5	0.32	0.47	77.6
NTD 14	2.7	0.32	0.11	49.0
NTD16	4.2	0.32	0.1	26.5
NTD 18	5.3	0.32	0.15	15.9
NTD 28	6.3	0.32	0.09	7.84
NTD 12	6.8	0.32	0.02	7.84
NTD 25	8.6	0.32	0.008	2.74
<sup>70</sup> Ge-3.30	3.02	<0.001	0.34	364.8
<sup>70</sup> Ge-2.98	8.00	<0.001	0.0074	247.6
<sup>70</sup> Ge-1.90	9.36	<0.001	0.0019	201.4
<sup>70</sup> Ge-1.65	14.5	<0.001	0.0006	100.3
<sup>70</sup> Ge-2.15	17.7	<0.001	0.0215	20.7

Fig. 1 Temperature dependence of resistivity of various NTD <sup>Nat</sup>Ge:Ga and NTD <sup>70</sup>Ge:Ga samples: <sup>70</sup>Ge-3.30 ( $\blacklozenge$ ), <sup>70</sup>Ge-2.98 ( $\blacktriangle$ ), <sup>70</sup>Ge-1.90 ( $\blacksquare$ ), <sup>70</sup>Ge-1.65 ( $\triangledown$ ), and <sup>70</sup>Ge-2.15 ( $\blacklozenge$ ).

Fig. 1 shows the log ( $\rho$ ) versus the inverse absolute temperature dependence of both natural and of enriched NTD Ge. The enriched crystal had a composition of 96.3% <sup>70</sup>Ge and 3.7% <sup>72</sup>Ge with the other Ge isotopes below the detection limit of 0.1% estimated for the secondary ion mass spectrometer (SIMS) used for this analysis. Whereas the natural NTD Ge crystals are p-type and have a compensation ratio of ~ 0.32 [11], only Ga

acceptors will form in  $^{70}$ Ge during NTD. The compensation will be dominated by residual chemical donors which are in the material before NTD. This donor concentration is of the order of  $10^{12}$  cm<sup>-3</sup> as determined by variable temperature Hall effect measurements of the undoped  $^{70}$ Ge crystals. The major interesting finding is that the resistivity appears to obey the simple dependence derived by Shklovskii and Efrös [12]:

$$\rho = \rho_0 \exp\left(\Delta/T\right)^{1/2} \tag{4}$$

for the very wide range of compensation from the natural NTD Ge (0.32) to the heavily-doped NTD <sup>70</sup>Ge samples (0.001). The resistivity of all <sup>70</sup>Ge samples depends more strongly on temperature than the corresponding natural NTD Ge crystals. This difference is especially pronounced for the lightly doped samples with resistivities between 10<sup>4</sup> and 10<sup>7</sup>  $\Omega$  cm in the 0.5 to 1K range.

In order to make full use of the properties of NTD Ge crystals, thermistors with excellent Ohmic contacts and passive bare surfaces have to be formed. Boron ion implantation followed by annealing and the deposition of a thin (200Å) Pd adhesion layer and a thicker (1000-2000Å) Au contact layer reliably leads to low noise contacts on p-type crystals [13]. The uncovered surfaces are polish-etched in a 3:1 mixture of nitric and hydrofluoric acids for 30 to 60s and quenched in pure methanol. The native oxide growing on the surface leads to minimal bandbending and no excess surface currents.



Fig. 2 New NTD Ge thermistor "flat-pack" design with approximately five times smaller thermal mass and electrical contacts on one surface for easier integration into arrays. All dimensions are given in  $\mu$ m.

In order to further reduce the total thermal mass of NTD Ge bolometers and to facilitate the application of electrical

connections, we have developed and tested a new bolometer design. We have drastically reduced one dimension of our typical  $(250 \ \mu m)^2$  thermistors to 50  $\mu m$  and have moved both contacts onto one surface measuring 200  $\mu m \times 300 \ \mu m$ . (Fig. 2) Our tests show that the electric field inhomogeneities introduced by this design do not appear to cause any problems in typical applications. This so-called "flat-pack" geometry will be used in future arrays described in the next chapter.

#### 3. NTD GERMANIUM BOLOMETER ARRAYS

Array development is actively pursued by many radioastronomy groups worldwide. We have given a brief summary of the state of bolometer arrays in 1994 [14]. At this symposium we have seen the first pictures taken with SCUBA (Submillimeter Common User Bolometer Array) which was recently installed on the James Clerk Maxwell radiotelescope. It features two impressive arrays: the long wavelength array with 37 pixels operates in the 600, 750 and 850  $\mu$ m atmosphere windows while the short wavelength array has 91 pixels and is optimized for 350 and 450  $\mu$ m. [15]

The great interest in arrays is based on the expectation that the telescope output does not increase linearly with the number n of pixels but with  $n^2$ . An array allows us to distinguish clearly between astronomical sources and signal fluctuations caused by the so-called "sky noise." Single pixel detectors when scanned cannot differentiate between photon fluctuations from various sources. An astronomical source scanned with an array produces a predictable two-dimensional scan pattern while random sky noise fluctuations remain uncorrelated. Krevsa et al. have demonstrated this power of noise rejection with their 7 element NTD Ge array composed of individual bolometers [16]. A new approach is currently being taken which uses silicon nitride membranes and micromachining. Details of this new design are being reported at this Symposium [17]. The optimization for the NTD Ge bolometers used in the new arrays are also discussed. Once the technical problems concerning optical coupling, mechanical mounting of the thermistors and electrical contact formation have been solved satisfactorily, we expect to see the widespread use of bolometer arrays operating at the background limit on all radiotelescopes.

#### 4 SUMMARY

We have reviewed the properties and fabrication of NTD Ge thermistors. The availability of isotopically controlled Ge lets us control separately both the acceptor and donor concentrations by the appropriate choice of the percentages of the various Ge isotopes. We have shown that highly enriched  $^{70}$ Ge when doped moderately by NTD shows a stronger dependence of the resistivity on

temperature than natural NTD Ge thermistor material of similar resistivity. This results in an increase of the sensitivity of the thermistor.

The doping uniformity provided by NTD and the reliability of thermistor fabrication have led many groups to the adoption of NTD Ge thermistors in bolometer arrays. The largest array in SCUBA has 91 pixels and has started to produce results on the James Clerk Maxwell telescope on Mauna Kea. Novel array designs using integrated circuit techniques and micromachining promise to revolutionize the use of large bolometer arrays on all radiotelescopes for broadband observations of extended sources. The potential advantages of arrays with n pixels over single detector instruments may be as large as n<sup>2</sup>!

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#### REFERENCES

- 1. F. Low, J. Opt. Soc. Am. 51, 1300-04 (1961)
- N. Coron, G. Dambier and J. Leblanc, in *Infrared* Detector Techniques for Space Research, eds. V. Manno and J. Ring (Reidel, Dordrecht 1971), pp. 121-131
- 3. N. S. Nishioka, P. L. Richards and D. P. Woody, *Appl. Optics* 17, 1562 (1978)
- E. E. Haller, N. P. Palaio, M. Rodder, W. L. Hansen, and E. Kreysa, in *Neutron Transmutation Doping of Semiconductor Materials*, Ed. R.D. Larrabee, Plenum Press, New York, pp. 21-36 (1984)

E. E. Haller, K. M. Itoh, J. W. Beeman, W. L. Hansen and V. I. Ozhogin, SPIE Symp. Astron. Telescopes & Instrum. for the 21<sup>st</sup> Century, 2198, 630 (1994)

- J. R. Carruthers and A. F. Witt, Crystal Growth and Characterization, Eds. R. Ueda and J. B. Mullin, pp. 107-54, North Holland Publ. Co., Amsterdam (1975)
- P. M. Downey, A. D. Jeffries, S. S. Meyer, R. Weiss, F. J. Bachner, J. P. Donnelly, W. T. Lindley, R. W. Mountain, and D. J. S. Silversmith, *Appl. Opt.* 23, 910 (1984)
- Table of Isotopes, Seventh Edition, Eds. C. M. Lederer and V. S. Shirley, John Wiley & Sons, New York (1978)

 M. S. Schnöller, *IEEE Trans. Electr. Dev.* ED-21, 313 (1974); see also:

Narain G. Hingorani and Karl E. Stahlkopf, Scientific American, 269, No.5, pp. 78-85, November 1993

- A. Alessandrello, C. Brofferio, D. V. Camin, O. Cremonesi, et al., Nucl. Instr. & Meth. B, vol. B93, 322 (1994)
- Kohei Itoh, W. L. Hansen, E. E. Haller, J. W. Farmer, V. I. Ozhogin, A. Rudnev and A. Thikomirov, J. Mat. Res. 8, No. 6, 1341, (1993); see also:

K. Itoh, W. L. Hansen, E. E. Haller, J. W. Farmer and V. I. Ozhogin, *Materials Science Forum* 117 & 118, 117 (1993); and:

K. M. Itoh, W. L. Hansen, J. W. Beeman, E. E. Haller, J. W. Farmer, and V. I. Ozhogin, J. Low Temp. Physics 93, Vol. 3/4, 307 (1993)

- K. Itoh, W. L. Hansen, E. E. Haller, J. W. Farmer and V. I. Ozhogin, *Materials Science Forum* 117 & 118, 117 (1993)
- B. I. Shklovskii and A. L. Efrös, *Electronic* Properties of Doped Semiconductors, Solid State Series Vol. 45, (Springer-Verlag, Berlin, 1984)
- E. E. Haller, K. M. Itoh, J. W. Beeman, W. L. Hansen and V. I. Ozhogin, SPIE Symp. Astron. Telescopes & Instrum. for the 21<sup>st</sup> Century, 2198, 630 (1994)
- 14. E. E. Haller, Infrared Physics and Technology 35, No.2/3, 127 (1994)
- W. S. Holland, P. A. R. Ade, M. J. Griffin, I. D. Hepburn, D. G. Vickers, C. R. Cunningham, P. R. Hastings, W. K. Gear, W. D. Duncan, T. E. C. Baillie, E. E. Haller and J. W. Beeman, *Intl. J.Infrared and MM Waves* 17, 669 (1996); see also:

W. K. Gear et al., Proc. 30<sup>th</sup> ESLAB Symp. on 'Submillimetre and Far Infrared Space Instrumentation,' Noordwijk, NL, 9/24-7/96, these proceedings

- E. Kreysa, E. E. Haller, H. -P. Gmünd, G. Haslam, R. Lemke and A. Sievers, Proc. Fourth Intl. Conf. on Space Terahertz Technology, pp. 692-7 (1993)
- 17. E. Kreysa, J. Beeman and E. E. Haller, Proc. 30<sup>th</sup> ESLAB Symp. on 'Submillimetre and Far Infrared Space Instrumentation,' Noordwijk, NL, 9/24-7/96, these proceedings.

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