Planar Array Technology for the Fabrication of Germanium X-Ray Microcalorimeters

Ugo Lo Cicero, Claudio Arnone, Marco Barbera, Alfonso Collura, Giuseppe Lullo, Salvatore Varisco

***Abstract*–Several technologies are presently competing for measuring the temperature increase in cryogenic micro-calorimeters used as high resolution energy-dispersive X-ray detectors. Doped germanium, whose resistivity depends on temperature, is a promising material for this purpose, because of its comparatively low specific heat and the possibility of making wafers with high doping uniformity by neutron transmutation. Presently, Ge-based microcalorimeters are still micro-machined and manually assembled. Here we present a planar approach to the fabrication of 2-D arrays of microcalorimeters and show the preliminary technological results.**

I. Introduction

THE increasing requirement for improved energy resolution in X-Ray spectrometry has driven research efforts toward the design of high sensitivity detectors. Among the different proposed devices [1], micro-calorimetric detectors have shown relevant application possibilities. Microcalorimeters quantify the energy of the impacting photons by measuring the temperature increase of a radiation absorber, which converts the X photons into low energy phonons through a thermalization process. The temperature increase is measured by a sensor, thermally linked to the absorber. In order to maximize the temperature increase, the sensor and the absorber must exhibit very small heat capacity, and thus micrometric size. A thermal link to a cryogenic thermal bath provides a way to dispose of the exceeding heat and maintains the average detector temperature below 100 mK.

There are several types of microcalorimeters, according to the thermometric sensor they are based on. Transition Edge Sensors (TES), Metallic Magnetic Calorimeters (MMC) and semiconductor microcalorimeters are the most popular [1], each having specific advantages and drawbacks. A recent goal in microcalorimeters research is the fabrication of two-dimensional arrays, in order to achieve both imaging and spectral analysis capabilities.

In the last years, a great interest has been devoted to TES, which are based on the strong variation of electrical resistance with temperature in a superconducting material kept in close proximity to its transition temperature. These microcalorimeters have reached a spectral resolution better than 3 eV [2] and have already been manufactured as 2D arrays [3]. A critical aspect in TES technology is the fabrication of the absorber, characterized by a mushroom shape and a high aspect ratio [4]. Another drawback of TES devices is their very low impedance, from a few mΩ to hundreds of mΩ, that requires the use of expensive Superconducting Quantum Interference Devices (SQUID) for signal readout.

In semiconductor microcalorimeters the resistivity change is related to a change in the temperature, according to the variable range hopping conduction model (holding at temperatures below 100 mK) [5]:

$$\rho(T) = \rho_0 \exp(T_0 / T)^p , \qquad (1)$$

where ρ is the resistivity, T is the temperature, ρ_0 and T_0 are constants depending on the semiconductor doping and its properties, and p is a constant usually taken as 1/2.

When cooled to cryogenic temperatures, semiconductor microcalorimeters exhibit an electrical resistance in the MΩ range, allowing direct interfacing to simple low noise FET amplifiers.

Germanium is a fairly sensitive semiconductor material for microcalorimeter fabrication. Single pixel devices based on doped germanium have been built with a FWHM (Full Width at Half Maximum) energy resolution of 3.1 eV at 6 keV and a quantum efficiency greater than 95% [6]. Germanium has some advantages over silicon, the other main semiconductor used to build microcalorimeters. It has a lower heat capacity at cryogenic temperatures, resulting in higher sensitivity [7]. Moreover, the process used for doping the germanium, the NTD (Neutron Transmutation Doping), that leads to very

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Ugo Lo Cicero is with the Dipartimento di Ingegneria Elettrica Elettronica e delle Telecomunicazioni, Università degli Studi di Palermo, 90128 Palermo, Italy, and with the Osservatorio Astronomico di Palermo G. S. Vaiana, Istituto Nazionale di Astrofisica, 90134 Palermo, Italy (email: locicero@astropa.unipa.it).

Claudio Arnone is with the Dipartimento di Ingegneria Elettrica Elettronica e delle Telecomunicazioni, Università degli Studi di Palermo, 90128 Palermo, Italy.

Marco Barbera is with the Dipartimento di Scienze Fisiche ed Astronomiche, Università degli Studi di Palermo, 90123 Palermo, Italy, and with the Osservatorio Astronomico di Palermo G. S. Vaiana, Istituto Nazionale di Astrofisica, 90134 Palermo, Italy.

Alfonso Collura is with the Osservatorio Astronomico di Palermo G. S. Vaiana, Istituto Nazionale di Astrofisica, 90134 Palermo, Italy.

Giuseppe Lullo is with the Dipartimento di Ingegneria Elettrica Elettronica e delle Telecomunicazioni, Università degli Studi di Palermo, 90128 Palermo, Italy.

Salvatore Varisco is with the Osservatorio Astronomico di Palermo G. S. Vaiana, Istituto Nazionale di Astrofisica, 90134 Palermo, Italy.

uniformly doped wafers [8], is not viable for fabricating silicon microcalorimeters[1].

II. Planar approach

To the authors' knowledge, no planar fabrication process has been proposed yet for germanium microcalorimeter arrays. Presently, germanium microcalorimeters are built as single-pixel detectors, and arrays are still manufactured by assembling together more single-pixel elements. Such approach cannot provide the scalability, uniformity and repeatability required to implement microcalorimeters with imaging capabilities. This is the main limitation to the use of germanium detectors which has recently determined, to some extent, the prevalence of the other technologies mentioned before.

Planar technology would provide an easy way to build scalable arrays, with a greater uniformity among detector pixels and higher degree of reproducibility.

The main steps of the planar approach proposed here are:

- doping of the Germanium wafers,
- fabrication of the absorbers,
- shaping of the Germanium sensors,
- electrical connections to the read-out electronics,
- thermal connections to the thermal bath.

Figure 1 shows the structure of the microcalorimeter array, while Fig.2 summarizes the main steps of the proposed process flow.

A. Wafer Doping

Neutron Transmutation Doping of germanium wafers is a well proven method to achieve a very uniform doping throughout the whole wafer [8]. This eventually results in a homogeneous pixel-to-pixel electrical response.

Various germanium isotopes naturally occur in native wafers (Table I). When these are exposed to a flow of thermal neutrons, some isotopes undergo a nuclear transmutation [9]. Among them, ^{70}Ge becomes ^{71}Ga, that acts as an acceptor and gives p-type doping, while ^{74}Ge becomes ^{75}As, a n-type donor atom that partially compensates the p-type doping.

TABLE I
NEUTRON TRANSMUTATION DOPING OF GERMANIUM[8]-[10]

Ge isotope	**^{70}Ge**	**^{72}Ge**	**^{74}Ge**	**^{76}Ge**
Isotopic abundance [%]	20.4	27.3	36.7	7.8
Neutron capture cross-section [cm^2 $\times 10^{-24}$]	2.74	-	0.482	-
Product	^{71}Ga	^{73}Ge	^{75}As	^{77}Se
Doping type	p	-	n	n

The doping level depends on the neutron fluence, the irradiation time, the isotopic abundances and the neutron capture cross-sections of the isotopes. Although ^{70}Ge is less abundant than ^{74}Ge, it has a much bigger capture cross-section (Table I), which results in a prevalent p-type doping.

Nonetheless, ^{74}Ge provides a doping compensation that is fundamental to the hopping conduction mechanism [5].

After finding the optimal neutron fluence and irradiation time, the transmutation induced doping process is very reproducible.

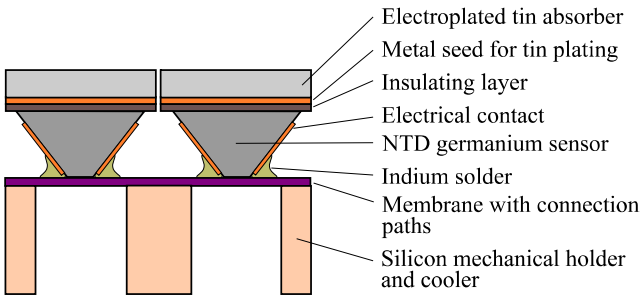

Fig. 1. Schematic structure of the microcalorimeter array.

B. Absorber fabrication

In our planar approach the single absorbers are built first as a continuous absorbing layer, and later separated by mechanical / chemical processes. This technique is different from the traditional approach, where the absorbers are made by gluing a thin square of absorbing material on each sensor. Making the absorbers on a single flat surface allows to achieve a high uniformity thus minimizing the differences among the pixels of the array.

The absorbing layer is made of tin, which has good thermalization properties and absorbs X-Ray photons within a few microns from the surface, thanks to its relatively high density [11]. It is also a superconducting material, therefore it has a low specific heat at cryogenic temperatures.

An insulating layer is required between the germanium sensors and the superconducting absorbers, in order to prevent a short-circuit between the sensor electrical contacts. Therefore, before creating the absorbers, an insulating film must be deposited on the germanium surface. For this purpose, spin-on glass is being evaluated as possible candidate. It is a dielectric that can be spin-coated uniformly with a thickness down to 100 nm.

Tin has been grown by electroplating, which allows to easily obtain pure films with well controlled thickness uniformity.

The conductive seed for the electrochemical tin growth is made with a thin metal film, vacuum deposited above the insulating layer. A double deposit of chrome and copper proved well suited to this purpose. A first film of about 10 nm of chrome is sufficient to give good adherence to the underlying dielectric. The copper film (100 nm thick) is the high conductivity base for the plating process and results chemically compatible with this electrochemical treatment.

[1] Although Silicon can be doped by NTD, its isotopic composition doesn't allows to obtain a doping compensation, which is necessary to the operating mechanism of microcalorimeters as explained below.

The next step concerns the growth of the tin absorber. We experimented a film growth up to 8 µm, with fairly good adherence to the Cr-Cu film. We used an alkaline plating solution of 100 g of potassium stannate trihydrate ($K_2SnO_3 \cdot 3H_2O$) and 15 g of potassium hydroxide (KOH) in 1 l of deionised water [12]. Tin could also be grown using an acid solution, but in that case some additives must be added to the plating solution to obtain a good deposit. Such additives would constitute impurities in the absorber that could affect its performances. Conversely, the alkaline solution we used does not require any additive.

An inert stainless steel anode has been used, therefore the tin atoms that build up the film came directly from the potassium stannate ions in the solution. With a current density of 10 mA/cm^2, 8 µm films where obtained in 30' at a temperature of 70 °C.

C. Germanium sensors and thermal bath

In order to handle the structure during the next fabrication steps, a support must be temporarily glued to the absorber side of the wafer. A silicon wafer can be used for this purpose. Next, the germanium wafer is back-lapped, until its thickness reaches the desired height for the sensors, that is in the range of 50 to 100 µm. After this step, the planar process proceeds on the Ge surface.

Standard photolithography was used for creating pyramid shaped sensors. Windows were opened in photoresist in the areas where germanium was to be removed. To this purpose, exposure through a photomask with an array of 120 µm × 120 µm transparent squares was used, on the resist-coated Ge surface. Anisotropic wet-etching followed. Several formulations for Ge etch are reported in the literature [13]. The one we selected is composed of phosphoric acid (H_3PO_4), ethanol (C_2H_5OH) and hydrogen peroxide (H_2O_2) in a 1:1:1 ratio.

The achieved etch speed in the direction perpendicular to the surface was measured as 15 µm/h. A pyramids height of 60 µm was obtained in about four hours.

The acid etch formulation allows the direct use of standard Shipley 1818 resist as a wet etch mask, as this photoresist has an acceptable stability in the acid bath, at least for the time requested for the etch process.

This anisotropic etch solution produces the truncated pyramidal structures depicted in Fig. 3, with base of 150 µm × 150 µm and flat top areas of 75 µm × 75 µm, 60 µm high. The orientation of the Ge wafers used in this process was <100>.

This etch process has already been studied in the past in the authors' laboratory [14].

The shape of the Ge sensors has a twofold advantage. It provides a large thermal contact area with the absorber through the large base of the pyramid, and offers the possibility to vacuum deposit electrical contacts on the tilted side faces of the pyramids.

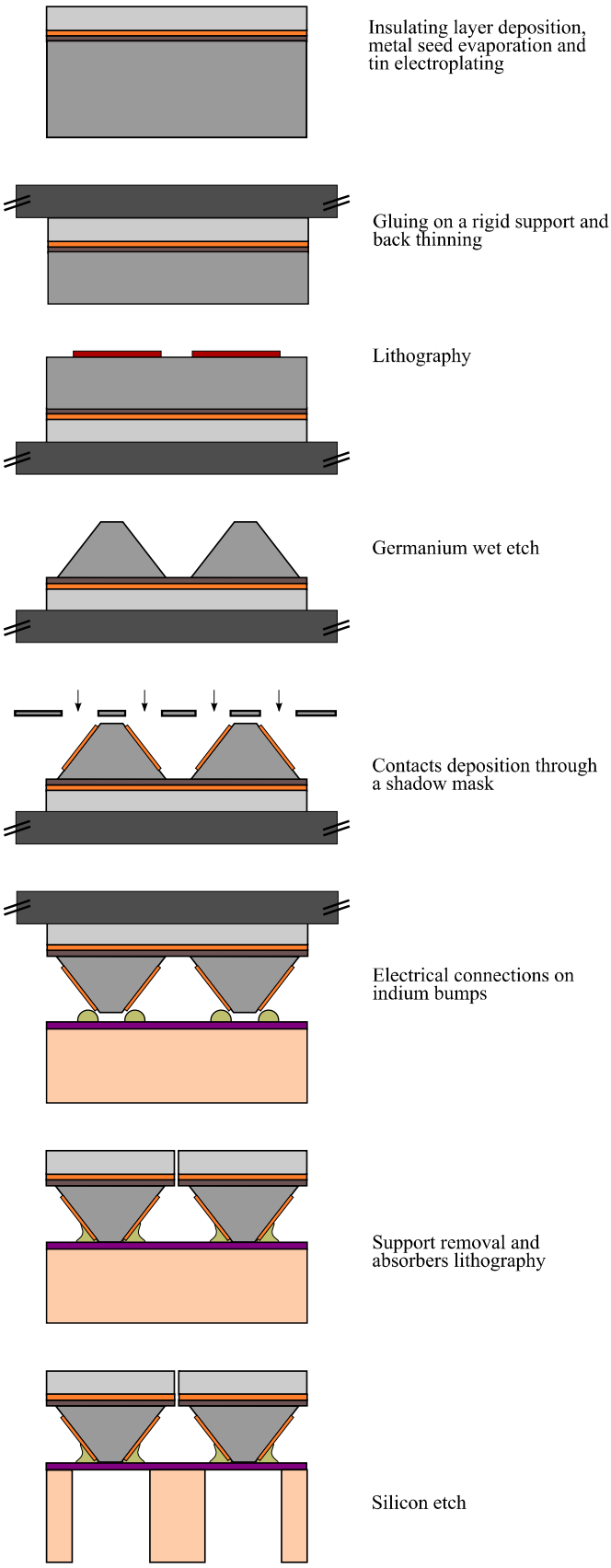

Fig. 2. Schematic view of the main steps of the proposed planar process.

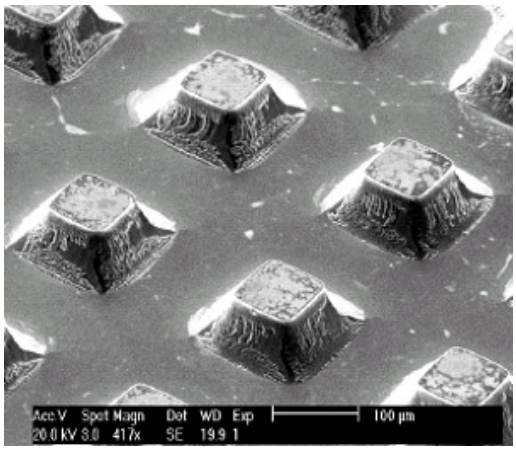

Fig. 3. Germanium square base truncated pyramids obtained by patterned wet etch.

This contact deposition technique is being implemented through a properly shaped shadow mask and will be described in future communications.

The next step includes contacting on indium bumps made on an electrically patterned layer of silicon nitride or polyimide grown on a silicon substrate. This substrate is later back-etched in order to leave free-standing isolating membranes among thermally conductive silicon pillars that will be the path to a mK cryocooler. This is quite standard Micro-Electro-Mechanichal (MEM) technology [15] and is not described here.

The membranes represent the thermal link between the Ge sensors and the cooled thermal silicon substrate, and limit the heat transfer rate to the underlying thermal bath. This allows the sensors to reach a quasi-equilibrium state soon after the arrival of the X photon, and to perform the measurement before the excess heat is transferred to the bath.

The final planar step concerns the removal of the glued mechanical support and a microlithographic process for separating the absorbers.

III. Conclusions

Bulk Germanium microcalorimeters have reached high performances in terms of spectral resolution and quantum efficiency. They exhibit some advantages on other technologies such as the TES and the silicon-based microcalorimeters, while their major drawback is the lack of a planar processing technology. In this paper we propose an innovative planar process leading to the fabrication of Ge-based X-Ray imaging devices with high spectral resolution, well suited for astronomical and laboratory applications, and competitive with other current technologies. Here we focused our attention to the fabrication of the absorbers and the Ge sensors.

The next step will be aimed at the fabrication of full working small arrays.

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