Fabrication of Bismuth Absorber Arrays for NTD-Ge Hard X-ray Microcalorimeters

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SCIENTIFIC CONTEXT

Many efforts were already performed to observe the universe in hard (10 – 100 keV) X-ray band [1]. CdTe/CdZnTe hard x-ray detectors present moderate spectral resolution (several hundred eV @ 60 keV). A resolution of few tens of eV at such energies could open new frontiers in the study of nuclear processes and high temperature plasma dynamics in energetic processes, such as energetic flares in the Sun.
Microcalorimeters are currently investigated for the detection of soft X-rays from astrophysical sources [2, 3] with high spectral resolution (e.g. TES sensors in the Athena X-ray Integral Field Unit instrument [4]).
We started a feasibility study for a balloon-borne experiment (MISTER-X), aimed to observe the solar corona in the hard X-ray band (20 – 100 keV) at high resolution (about 50 eV @60 keV) by using NTD-Ge microcalorimeters.

AIM OF THE WORK

The aim of this research was to study the electroplating growth process of bismuth structures to be used as absorber pixels for NTD-Ge hard X-ray microcalorimeter arrays.
In the past we developed a process to deposit by electroplating uniform tin layers for soft X-ray absorption [5], but these are not usable for higher energies. Bismuth, conversely, is a well suited material for hard X-ray absorber fabrication, due to:

- high Z material for efficient X-ray absorption in a wide spectrum range;
- very low heat capacity;
- simple growth technology (electroplating);
- high growth thicknesses, giving high quantum efficiencies also at energies up to 100 keV;
- moderate cost.

High thickness bismuth layers were already deposited by our group on suitable substrates by the simple electroplating technique [6], and a specific process was developed for this purpose.

SUBSTRATE PATTERNING

A photoresist test pattern was applied to glass substrates to delimit the bismuth growth areas and to partially guide the metal deposition during the electroplating. The process was performed in a class 10000 clean room at INAF-OAPA X-ray Astronomy Calibration and Testing (XACT) Microtechnology laboratory with the following procedure:

- Glass slices (2.5x1.25x0.1 cm^2) were at first optical grade cleaned by a multi step procedure with de-ionized water, acetone and ethanol.
- The cleaned substrates were coated by a Varian VT114A electron-beam evaporation system with 20 nm Ti and 20 nm Au.
- MA-P1225 photoresist was deposited ot the substrates by spin coating at 4000 rpm for 60 s and oven dried at 90°C for 10 minutes.
- The photoresist was finally exposed using a Suss MA6 mask aligner (hard contact mode, 10 mW/cm^2, 30 s) and developed with ma-D 331 for 1 m at room temperature.

PATTERNED BISMUTH GROWTH

The experimental setup (Figure 1) employed to implement the bismuth electroplating process was based on a Parstat 2263 potentiostat; the deposition cell had a suitable holder for substrates (working electrodes) on which bismuth layers grow, an Ag/AgCl with KCl 3M reference electrode, and a DSA (dimensionally stable anode) counter electrode. The cell was placed on a stirring hot plate.
The process was developed at the Electrochemical Material Science Laboratory of the Dipartimento di Ingegneria of the Università di Palermo.

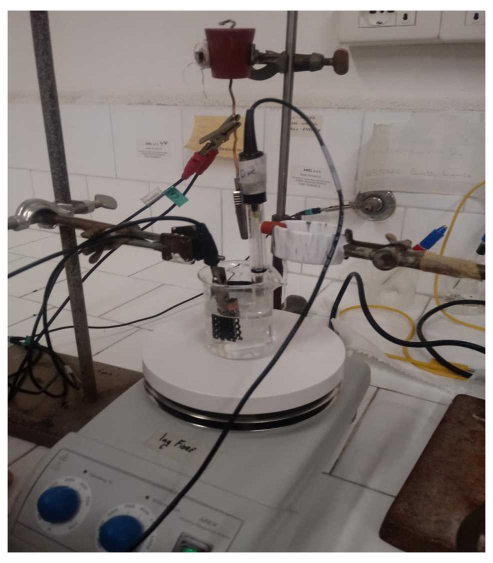


FIGURE 1. Electroplating setup

The electroplating bath composition is reported in Table 1.

Table 1. Bath composition

Bismuth nitrate pentahydrate	$Bi(NO_3)_3H_2O$	3.75 g
Potassium hydroxyde	KOH	3.81 g
Nitric acid	HNO_3	5.7 ml
Glycerol	$CH_2OHCHOHCH_2OH$	6.25 g
Tartaric acid	$HOOC\ (CHOH)_2\ COOH$	2.5 g
Water (bidistilled)	H_2O	50 ml

The solution pH was 0.10, measured by a Hanna laboratory pH-meter; E_S=-0.05V was the chosen potential between working and reference electrodes, according to results of continuous Bi layers electroplating experiments. A magnetic stirrer was in use during the deposition.
The deposition current I(t) diagram is shown in Figure 2. Its smooth trend and the slowly varying regime indicate that the growth process was uniform and regular in time. Figure 3 shows the SEM images of the resulting bismuth deposition.

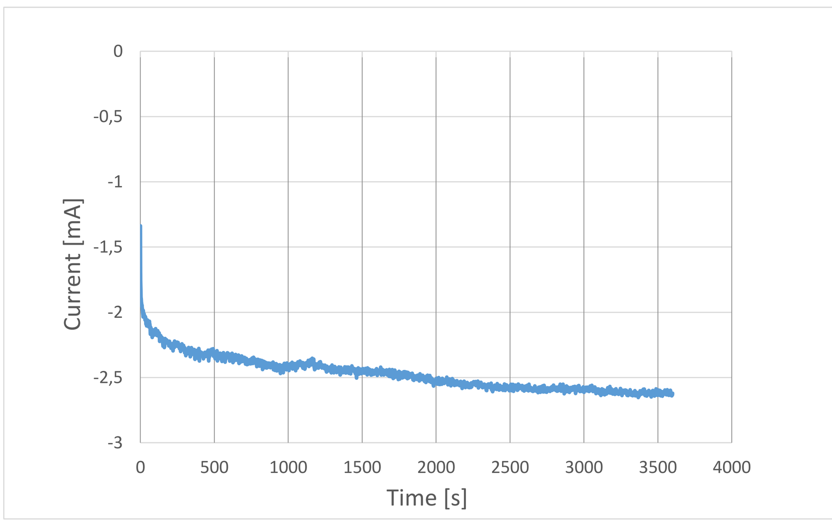
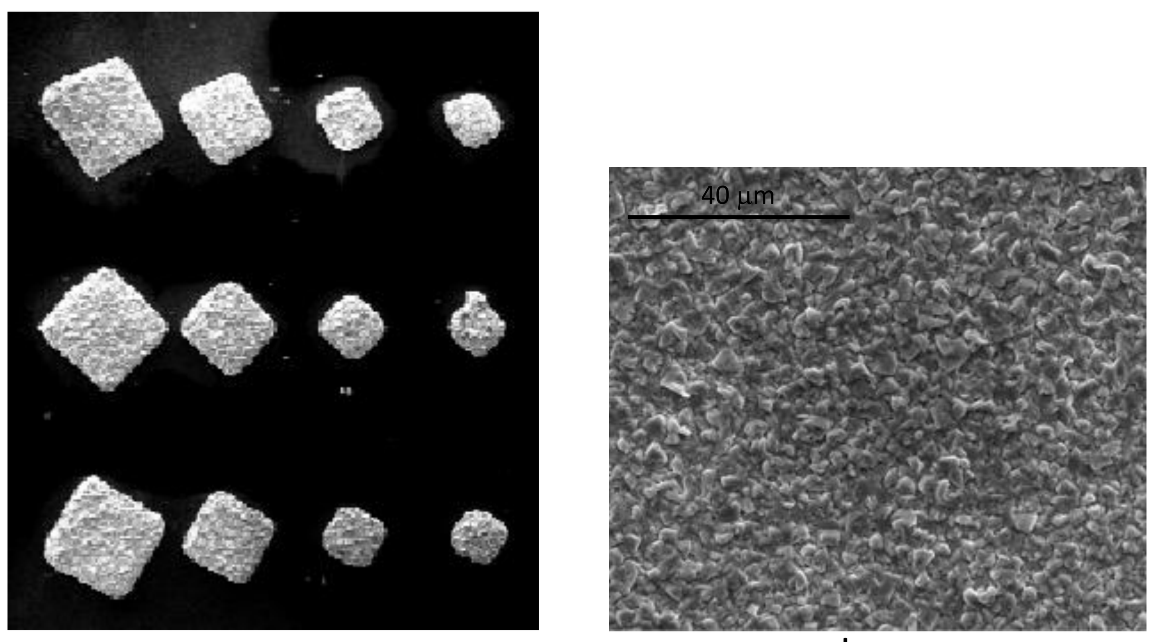


FIGURE 2. Deposition current I(t)



a b

FIGURE 3. SEM images of patterned bismuth structures; a) squares with different sizes and tilt angles; b) morphology of a uniform Bi layer.

Scanning electron microscope observations by a FEI Quanta 200F SEM at the Dipartimento di Ingegneria of the Università di Palermo on growth samples evidenced that:

- the structure surface area increases with height (lateral growth);
- a moderate influence of stirring speed on the geometrical features can be supposed.

Lateral growth depends from the levelling properties of the specific electroplating process and has proved to be relevant. Stirring speed was noted to be relevant on the deposition of continuous layers only in case of very low or high speeds, but only with patterned depositions we we able to appreciate deformations on the geometries.

FUTURE WORK

Both lateral growth and stirring effects have to be better studied. We have therefore designed a new photolithographic mask (Figure 4) with several patterns with different size and separations to better characterize and tune the deposition process, including the effects of different thicknesses of photoresist guiding patterns. The mask also includes arrays with different pixel size and pitch to fabricate absorbers arrays with different effective detection area. The optimization of the process and the fabrication of the arrays is already in progress.

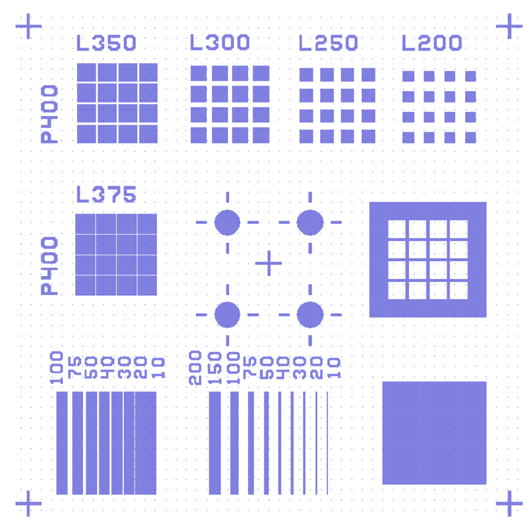


FIGURE 4. Array test pattern

SUMMARY

We have started an investigation to fabricate bismuth absorbers for arrays of hard X-ray microcalorimeters. Preliminary structures have been fabricated allowing us to identify the critical bismuth growing parameters.
The manufacturing of arrays with represantive number of pixels, filling factor and absorber thickness is in progress.

ACKNOWLEDGEMENTS

We thank prof. Giuseppe Lullo of Dipartimento di Ingegneria of the Università di Palermo for relevant support in the realization of the microlithographic absorber array pattern mask. We acknowledge partial support to the feasibility study of MISTER-X by the Italian National Institute for Astrophysics (INAF) and the Italian Space Agency (ASI).

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