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DOI

[10.1117/12.2562100](https://doi.org/10.1117/12.2562100)

Publication date

2020

Document Version

Final published version

Published in

Space Telescopes and Instrumentation 2020

Citation (APA)

Wang, G., Bruijn, M., Nagayoshi, K., Ridder, M., Taralli, E., Gottardi, L., Akamatsu, H., Den Herder, J. W., Gao, J. R., & Cui, W. (2020). Developing X-ray microcalorimeters based on TiAu TES for HUBS. In J.-W. A. den Herder, S. Nikzad, & K. Nakazawa (Eds.), *Space Telescopes and Instrumentation 2020: Ultraviolet to Gamma Ray* Article 114449J (Proceedings of SPIE - The International Society for Optical Engineering; Vol. 114444). SPIE. <https://doi.org/10.1117/12.2562100>

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SPIE.

Event: SPIE Astronomical Telescopes + Instrumentation, 2020, Online Only

Developing X-ray microcalorimeters based on TiAu TES for HUBS

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ABSTRACT

Hot Universe Baryon Surveyor (HUBS), a Chinese space mission, is proposed to find a large fraction of the so-called missing baryons, which would help us to understand more about the structure formation and evolution of the universe. Both theoretical and experimental results show that developing a highly efficient soft X-ray spectrometer over a large field of view and with a high energy resolution is the key to detect the “missing baryons”. X-ray microcalorimeters based on a transition-edge sensor (TES) array is required for HUBS, which aims to have 1 deg² field of view (FoV) with 1' angular resolution and 2 eV energy resolution optimized around 0.6 keV. Taking the high throughput X-ray optical focusing system on HUBS into account, the TES array is designed to have 60 x 60 pixels with an area of 1 mm² for each pixel. The microcalorimeter consists of a TES, a weak thermal link to a heat bath, and a semi-metal or normal metal absorber to increase the X-ray absorption efficiency. When an X-ray photon with a given energy is absorbed, the temperature of the absorber increase, that can be monitored by measuring the resistance change of the TES. A bilayer of a superconductor and a normal metal is used to fabricate a TES with a critical temperature (T_c) of ~100 mK. The latter is set for the required energy resolution. For HUBS, both MoCu and TiAu TES technologies are considered in its development phase. Here we will focus on TiAu TES calorimeters designed and partially fabricated at SRON for HUBS. Recent demonstration of a resolution of 2.5 eV at 5.9 keV in an AC readout at SRON for X-IFU on board of Athena illustrates the promising of this technology. However, the challenging for the HUBS array is the large pixel size. We will report the design and fabrication of prototype HUBS calorimeters.

Keywords: HUBS, X-ray microcalorimeter, Transition edge sensor, TiAu bilayer, BiAu absorber

1. PIXELS FOR HUBS

Hot Universe Baryon Surveyor (HUBS) is proposed as a Chinese space mission to find a large fraction of the so-called missing baryons^{1,2}, which would help us to understand more about the structure formation and evolution of the universe. Both theoretical^{3,4} and experimental results show that developing a highly efficient soft X-ray spectrometer over a large field of view and with a high energy resolution is the key to detect the “missing baryons”. X-ray microcalorimeters based on a transition-edge sensor (TES) array is required for HUBS.

1.1 Science requirements

Since a large amount of the baryons is not “seen” in the previous observations, HUBS is designed to focus on soft X-ray detection. It should have 1 deg² field of view (FoV) with 1' angular resolution and 2 eV energy resolution optimized around 0.6 keV. Taking the high throughput X-ray optical focusing system on HUBS into account, the TES array is designed to have 60 x 60 pixels with an area of 1 mm² for each pixel. Table 1 shows the key parameters of the design⁵.

Table 1. HUBS Specifications*.

Parameter	Minimum	Expectation	Goal
Detector array			
Regular grid		60 × 60	
Central grid		12 × 12	
Energy resolution (eV) at 0.6 keV			
Regular pixels	2.5	2.0	1.5
Central pixels	1.0	0.8	0.6
Lower energy (keV)	0.2	0.2	0.1
Higher energy (keV)	1.0	2.0	2.0
Effective area ^a (cm ²) at 0.6 keV	400	500	600
FoV (deg ²)	0.8	1.0	1.2
Grasp (cm ² deg ²) at 0.6 keV	320	500	720
Angular resolution (HPD) (arcmin)	1.3	1.0	0.7

* Taken from ref 5.

1.2 Pixel design

Each pixel of the microcalorimeter consists of a TES, a weak thermal link to a heat bath, and a semi-metal or normal metal absorber to increase the X-ray absorption efficiency. When an X-ray photon with a given energy is absorbed, the temperature of the absorber increases, which can be monitored by measuring the resistance change of the TES. A bilayer of a superconductor and a normal metal is used to fabricate a TES with a critical temperature (T_c) of ~100 mK. The latter is set for the required energy resolution.

Both MoCu and TiAu TES technologies are considered in its development phase. In SRON, we focus on TiAu TES calorimeters designed and partially fabricated for HUBS. TiAu is developing in Tsinghua University as well. Recent demonstration of a resolution of 2.5 eV at 5.9 keV in an AC readout at SRON for X-IFU on board of Athena illustrates the promising of this technology^{6,7}. However, the challenging for the HUBS array is the large pixel size. We focus on the design and fabrication of prototype HUBS calorimeter absorber.

Briefly, for HUBS, the camera consists of a 60x60 detector array, possibly with central smaller pixels with improved energy resolution for absorption-line spectroscopy. They might have the capability to capture almost all X-rays from a point source.

2. ABSORBER DESCRIPTION

For large pixel design, absorber is the key issue. Gold and bismuth bilayer is selected as absorber when considering quantum efficiency and energy resolution, as well as microfabrication.

2.1 energy and quantum efficiency calculation

2.1.1 Quantum efficiency

According to law for absorption of x-rays in film, $I(x) = (1 - I_0 e^{-\mu x}) * 100\%$. Where $Loss = I/I_0 = e^{-\mu x}$, I_0 is incident intensity, x is film thickness. Both gold and bismuth monolayer thickness calculation results are shown in table 2.

Table 2. thickness (nm) requirement of Au and Bi for different quantum efficiency for such energy x-ray

Au ($\rho=18.85$)				Bi ($\rho=9.73$)		
$[\mu/\rho](\text{cm}^2 \text{g}^{-1})$	4652	425.3	118.1	5441	485.5	136
Energy	1keV	6keV	10keV	1keV	6keV	10keV
Loss=0.01	525	5744	20686	870	9749	34801
Loss=0.02	446	4880	17573	739	8281	29563
Loss=0.05	342	3737	13457	566	6342	22639
Loss=0.1	263	2872	10343	435	4874	17401
Loss=0.2	184	2008	7230	304	3407	12162
Loss=0.3	137	1502	5408	227	2549	9098
Loss=0.4	104	1143	4116	173	1940	6924
Loss=0.5	79	865	3114	131	1467	5238

For Bi/Au bilayer: $I = I_0 e^{-\mu_B t_B} = I_0 e^{-\mu_A t_A} e^{-\mu_B t_B}$, Loss = $e^{-\mu_A t_A} e^{-\mu_B t_B}$. 1 keV, suppose Loss ≤ 0.1 :

If $t_A=100$ nm, loss_A=0.42, loss_B ≤ 0.24 , $t_B \geq 270$ nm;

If $t_A=20$ nm, loss_A=0.84, loss_B ≤ 0.12 , $t_B \geq 400$ nm.

Where t_A and t_B are the thickness of gold and bismuth. μ_A and μ_B are constants calculated from μ/ρ ⁸.

2.1.2 Energy resolution

Energy resolution can be determined by the following equation.

$$\Delta E = \sqrt{\frac{\xi k_B T^2 C}{\alpha}} \tag{1}$$

For a certain TES device working in a certain temperature, delta E is a function of heat capacitance C, where

$$C(T) = \frac{\rho}{A} \gamma V T \tag{2}$$

Comparing to ESA TES devices, ignoring contribution of other structures, the heat capacitance of HUBS absorber should be equal (9.50×10^{-10} mJ/K with 2 eV energy resolution).

For gold and bismuth bilayer, according to parameters from table 3, if $t_A=132$ nm, $t_B \leq 0$; if $t_A=100$ nm, $t_B \leq 6.23$ μm .

Table 3. Table of values for calculating TES properties.

	ρ_{el}^1 $\mu\Omega \cdot \text{cm}$	n^2 $10^{28}/\text{m}^3$	v_F^2 $10^6/\text{m/s}$	γ^1 $\text{mJ}/\text{mole} \cdot \text{K}^2$	ρ/A mole/cm^3	T_c^1 K	$\lambda_L(0)$ nm
Al	2.74	18.1	2.03	1.35	0.100	1.140	16 ¹
Ti	43.1	10.5	0.041 ⁴	3.35	0.094 4	0.39	310 ⁴
Mo	5.3	38.6	0.60 ⁴	2.0	0.107	0.92	
W	5.3	37.9	0.70 ⁴	1.3	0.105	0.012	82 ⁵
Ir	5.1	63.4	0.36 ⁶	3.1	0.117	0.140	29 ⁶
Nb	14.5	27.8	0.62 ³	7.79	0.092 2	9.5	39 ¹

Cu	1.70	8.47	1.57	0.695	0.141
Ag	1.61	5.86	1.39	0.646	0.097 4
Au	2.20	5.90	1.40	0.729	0.098 3
Bi	116.0	14.1	1.87	0.008	0.046 8

As a result, gold only film thickness should be less than 130 nm. If Bismuth layer is added, thickness of gold can be reduced accordingly.

Considering both quantum efficiency and energy resolution, gold and bismuth bilayer can be an option. Here, for HUBS requirements, set loss = 0.1, Au (100 nm) + Bi (5 μm) for 2 eV energy resolution combination is selected.

2.2 Strength analysis

In order to minimize thermal conductance between absorber and bath, mushroom like absorber is expected. Thus a new problem arises: strength. The model of absorber can be simplified to one dimensional cantilever and beam as shown in figure 1. For cantilever, the end is fixed. For beam, the ends are fixed or freestanding.

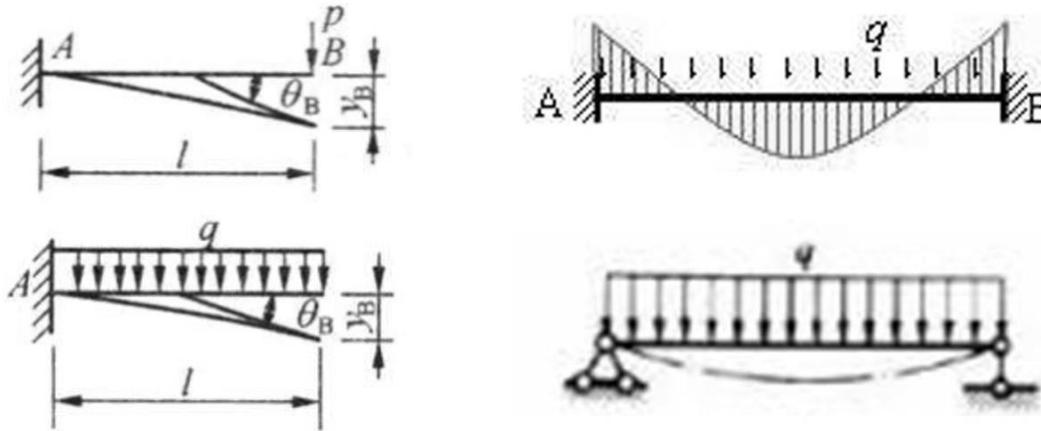


Figure 1. Model of cantilever and beam.

Three kinds of loads are considered: acceleration force, capillary force of drying liquid and surface adhesion. According to previous calculation⁹, if gap between absorber and substrate is 2 μm and bismuth thickness is 5 μm , critical length 120 μm for cantilever and 370 μm for beam. On the other hand, Critical length of doubly clamped beams is $2\sqrt[4]{3} \approx 2.6$ (2.5 or 2.9) times larger than that of cantilever when load is acceleration force (capillary force or surface adhesion). These numbers give us a clue to design patterns for absorber. Another conclusion from the calculation is that capillary force which is induced in microfabrication process is much larger than gravity.

2.3 Absorber design

2.3.1 Test structures

Each chip contains absorbers and a set of test structures. Test structures consists of 3 groups:

1. Side wall check for absorber foot and hat pattern. These structures are a series of sloped edge connected by film on substrate and float. If the sloped edge is not fully covered by Au/Bi film, an open circuit can be detected. As shown in figure 2a.
2. Large meander (200 μm wide, 70 squares long) of the electroplated absorber for resistivity check. Shown in figure 2b.

3. 1D beam and cantilever of different size (100 μm wide, 50~800 μm long) for strength test. 0 deg, 45 deg and 90 deg. Shown in figure 2 c and d.

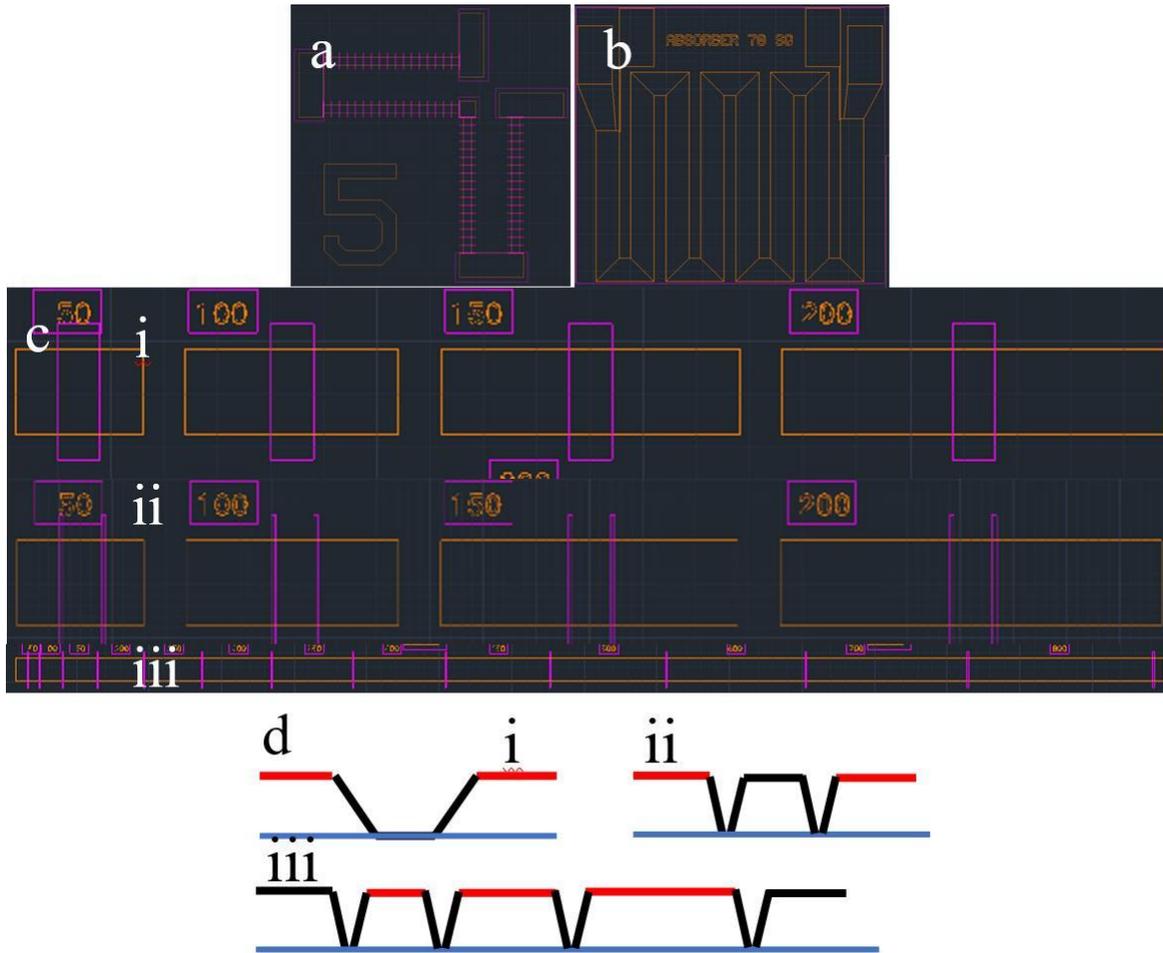


Figure 2. Test structures for absorber. a) Side wall check for absorber foot and hat pattern. b) Large meander (200 μm wide, 70 squares long) of the electroplated absorber for resistivity check. c) 1D beam and cantilever of different size (100 μm wide, 50~800 μm long) for strength test. d) schematic of beam and cantilever.

2.3.2 Absorber foot, hat and pattern

Four types of absorber hat pattern are designed to increase strength of absorber hat as shown in figure 3: Flat, a #, b *, c |-. Flat is not shown. These patterns may also affect noise response and thermal conductivity differently.

Absorber foot patterned to minimize thermal conductance and increase mechanical strength. A series of foot pattern is set as shown in figure 3 d ~ g: 5 triangle, 6 square, 10, 14.

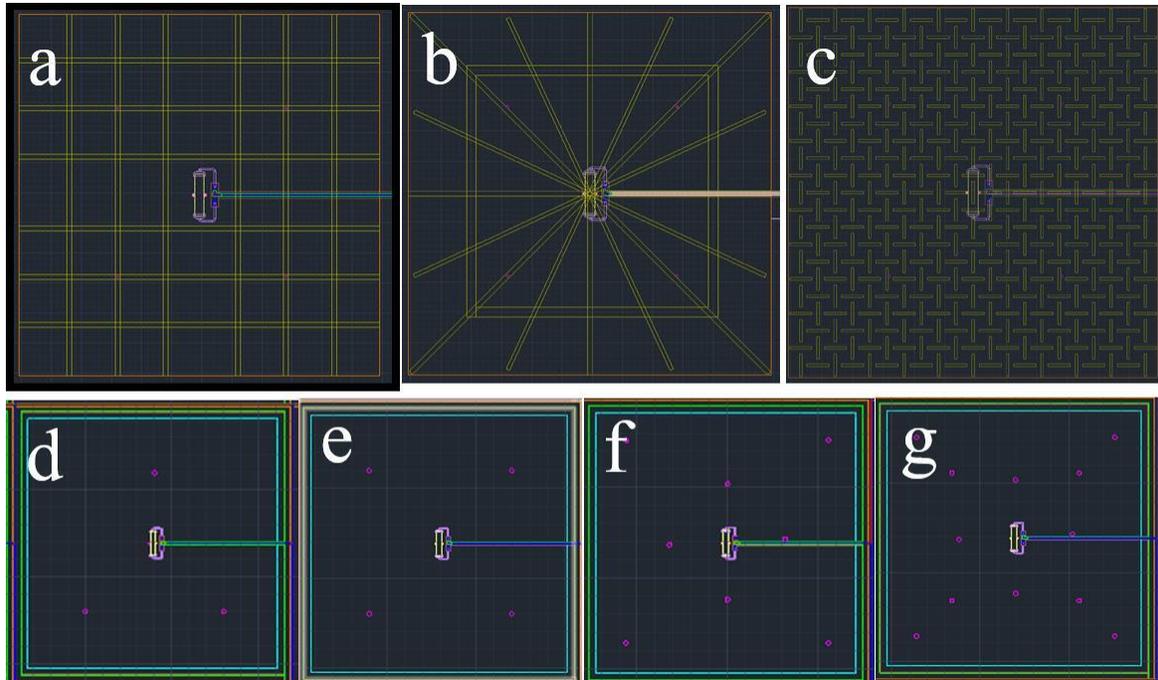


Figure 3. Absorber design. The magenta circles (3, 5, 10, 15 μm diameter) are the coupling points of the overhanging X-ray absorber (in brown). The yellow patterns are absorber hat patterns which are for strength enhancement. The two center points contact the virtual TES. The other absorber contact points rest on the substrate. Blue square is defined as DRIE back etched Si₃N₄ membrane.

2.4 Microfabrication

2.4.1 Electroplating bismuth

100 nm thick gold is easily deposited by e-beam evaporation. The best performance bismuth film for absorber should be electroplated on gold seed layer. Generally there are two methods for electroplating: Aqueous or non-aqueous solution. We use non-aqueous solution¹⁰ to achieve the electrodeposition. The grain size can be controlled by current as shown in figure 4. Large grain with size of more than 1 micro is expected to have less energy tail phenomenon.

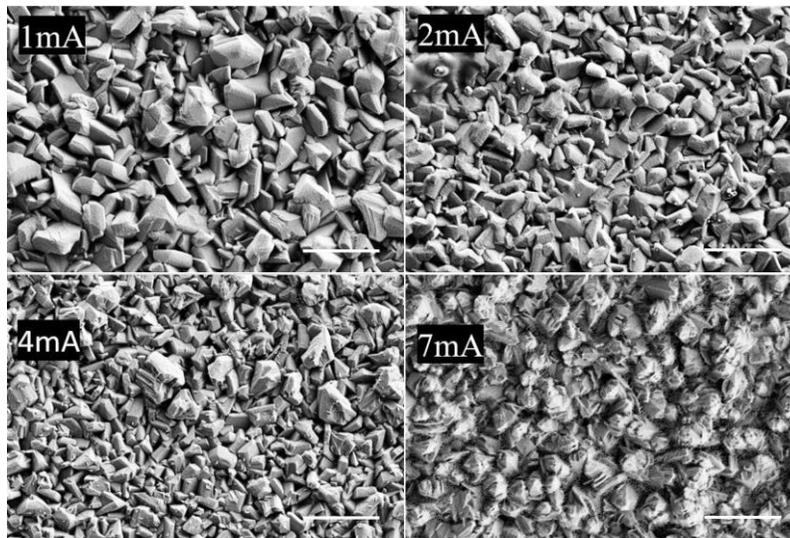


Figure 4. SEM images of electroplated bismuth film surface. With increasing current, the grain size decreases and even some pores present. Scale bars are 2 μm .

Etching of Bismuth and gold bilayer film is necessary because non-aqueous solution we chose is reactive with photoresist. Thus lift-off process can not be applied. Both wet and dry etching method were tested. A combination of wet etching of bismuth by nitrite followed by dry etching of gold by argon ion beam give us well patterned bilayer. Figure 5 is an example of etched bismuth and gold pattern.

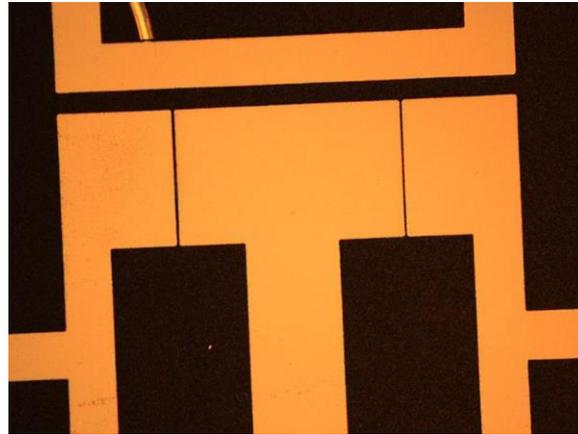


Figure 5. Optical image of BiAu pattern.

2.4.2 Microfabrication process for absorber

3 layers of photoresist are designed to fabricate mushroom absorber, which is shown in table 4 and figure 6. Top photoresist for defining absorber size is not shown. Foot fill and pattern fill will only be added if nesesity.

Table 4. microfabrication steps.

Step	Description
1	Resist pattern absorber foot
2	Resist pattern absorber hat pattern
3	Deposition electroplating base gold
4	Electroplating of absorber bismuth
5	Resist pattern absorber hat
6	Ion beam etching absorber hat (bismuth and gold)
7	Remove all photoresist

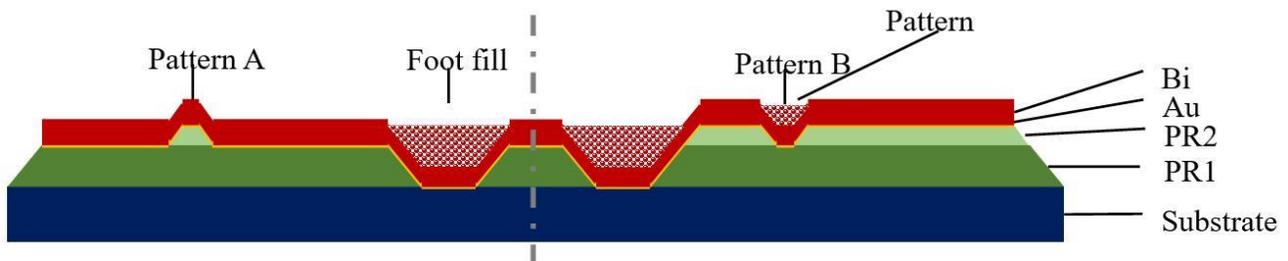


Figure 6. Absorber structure. A) Ultrathin Au layer might be deposited by angular rotating the wafer or thick then thinning. B) Hard bake PR1 for PR2 pattern or use proximal exposure method. C) There is also a possibility to fill foot holes by applying another mask and deposition. It might be tested in further process. D) Pattern B fill will only be applied when necessary, a different mask (larger holes) is required.

3. CONCLUSION

Absorber of a prototype calorimeter for the proposed Chinese HUBS mission based on TiAu TES fabricated at SRON in a collaboration with Tsinghua University is designed and partially fabricated. According to energy resolution and quantum efficiency calculation, the absorber consists of 100 nm thick gold and 5 micrometer thick bismuth. Mechanical strength and thermal conductance determine its supporting foot and pattern. Test structures are designed and the patterned BiAu bilayer structures will be tested and measured. HUBS will have a highly efficient soft X-ray spectrometer, which will have 1 deg² field of view with 1' angular resolution and 2 eV energy resolution around 0.6 keV. The sensor consists of an pixel array of 60 x 60 with an area of 1 mm² for each.

Acknowledgements The development of absorber for HUBS is supported by the Chinese Academy of Sciences through the Strategic Priority Research Program, Grant No. XDA15010400, the National Natural Science Foundation of China through Grants 11927805 and 11821303, the Ministry of Science and Technology of China through the National Key R&D Program of China, Grant No. 2018YFA0404502, the Netherlands Institute for Space Research and Tsinghua University.

REFERENCES

- [1] Fukugita, M., Hogan, C. J., and Peebles, P. J. E., "The cosmic baryon budget, " *Astrophys. J.*, 503, 518 (1998).
- [2] Bregman, J.N., "The search for the missing baryons at low redshift, ", *Ann. Rev. Astron. Astrophys.*, 45, 221 (2007)
- [3] Cen, R., and Ostriker, J. P., "Cosmic chemical evolution, " *Astrophys. J.*, 519, L109 (1999).
- [4] Dave, R., Cen, R., Ostriker, J.P., Bryan, G.L., et al., "Baryons in the Warm-Hot Intergalactic Medium, " *Astrophys. J.*, 552, 473 (2001).
- [5] Cui, W., Chen, L.-B., Gao, B., Guo, F.-L., et al., "HUBS: Hot Universe Baryon Surveyor," *J. Low Temp. Phys.*, 199, 502 (2020).
- [6] Taralli, E., Gottardi, L., Nagayoshi, K. et al. "Characterization of High Aspect-Ratio TiAu TES X-ray Microcalorimeter Array Under AC Bias," *J Low Temp Phys* 199, 80–87 (2020).
- [7] Nagayoshi, K., Ridder, M.L., Bruijn, M.P. et al. "Development of a Ti/Au TES Microcalorimeter Array as a Backup Sensor for the Athena/X-IFU Instrument," *J Low Temp Phys* 199, 943–948 (2020)
- [8] <https://physics.nist.gov/PhysRefData/XrayMassCoef/ElemTab>
- [9] Tas, N., Sonnenberg, T., Jansen, H., Legtenberg, R., Elwenspoek, M., "Stiction in Surface Micromachining," *Journal of Micromechanics and Microengineering* 6(4), 385–397 (1996).
- [10] Cerededa-Company, P., and Costa-Kramer, J. L., "Electrochemical Growth of Bismuth for X-ray Absorbers," *Journal of The Electrochemical Society*, 165 (5) 167-182 (2018)