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Bismuth X-ray absorber studies for TES microcalorimeters

J.E. Sadleir[a,b,*], S.R. Bandler[a], R.P. Brekosky[a], J. Chervenak[a], E. Figueroa-Feliciano[a], F. Finkbeiner[a], N. Iyomoto[a], R.L. Kelley[a], C.A. Kilbourne[a], J.M. King[a], F.S. Porter[a], I.K. Robinson[b], T. Saab[a], D.J. Talley[a]

[a]*NASA Goddard Space Flight Center, Greenbelt, MD 20771, USA*
[b]*University of Illinois Physics Department, Urbana, IL 61801, USA*

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Abstract

Bismuth's large atomic number and low carrier density makes it an attractive X-ray absorber material for microcalorimeters. Bismuth's long fermi wavelength and long mean free paths have motivated much interest in the fabrication of high quality bismuth films to study quantum size effects. Despite such incentives, fabrication of high quality bismuth films has proven difficult, and measured properties of such films are highly variable in the literature. Implementing a bismuth deposition process for TES (superconducting Transition Edge Sensor) device fabrication presents additional challenges particularly at interfaces due to the inherent granularity and surface roughness of its films, its low melting point, and its tendency to diffuse and form undesired intermetallic phases. We report observations of Bi–Cu and Bi–Au diffusion in our devices correlating with large shifts in T_c (superconducting transition temperature). Using SEM and in situ R vs T annealing experiments we have been able to study these diffusion processes and identify their activation temperatures.
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Keywords: Diffusion; In situ resistance annealing; Multilayer strip resistance; X-ray absorber; Superconducting transition edge sensor (TES); Microcalorimetry

1. Introduction

Bismuth's low heat capacity and X-ray stopping power are excellent for microcalorimeter absorber applications. High conductivity is desirable for fast devices without positional dependence. To aid in lateral thermalization, multiple Cu layers have been sandwiched inside Bi absorbers [1] at the cost of larger heat capacity. This paper examines three major issues regarding Bi absorbers and their impact on Cu layer continuity and the TES–absorber interface: (1) Bi films surface roughness, (2) Bi–Cu diffusion, (3) Bi–Au diffusion.

2. Bi surface roughness and Cu layer discontinuity

Our Bi films are thermally evaporated at pressures $<1e-6$ torr at 2 nm/s onto a water cooled stage. As depicted in Fig. 1, our Bi film surface morphology consists of weakly connected individual Bi grains (~200 nm in size). Bi surface roughness and Cu diffusion contribute to the Cu layer being discontinuous in our Bi/Cu multilayer absorber. To investigate the conductivity of our Bi/Cu multilayer structures, four-wire resistance strips of Bi deposited on Si with Cu top layers of various thickness were fabricated and their resistances measured from room temperature down to 4 K. Using measured R–T curves from Bi-only and Cu-only strips in various lumped circuit configurations, we modeled the measured R–T dependence for our Bi/Cu bilayer structures. The best fit to the data came from the simple model of the Bi strip connected in parallel to the Cu strip with a multiplicative parameter to increase the Cu layer resistance. This model predicted an effective Cu layer thickness absent from the conduction that agrees with the

*Corresponding author. NASA Goddard Space Flight Center, Greenbelt, MD 20771, USA.
E-mail address: sadleir@milkyway.gsfc.nasa.gov (J.E. Sadleir).

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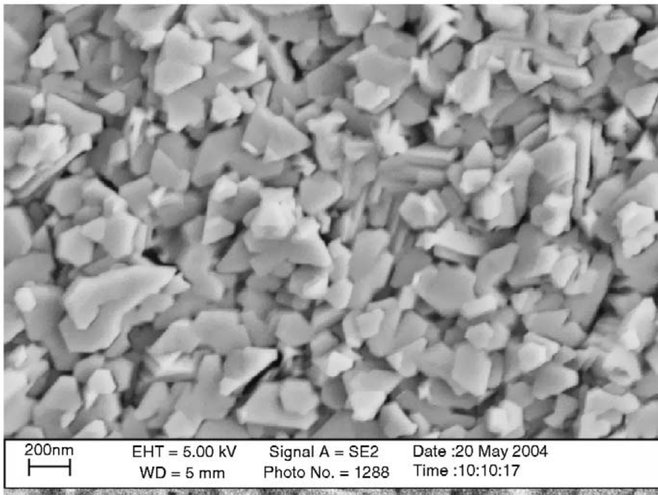

Fig. 1. Bi film surface of 2.25 μm thickness—typical surface morphology for Bi films 1.0 μm and thicker.

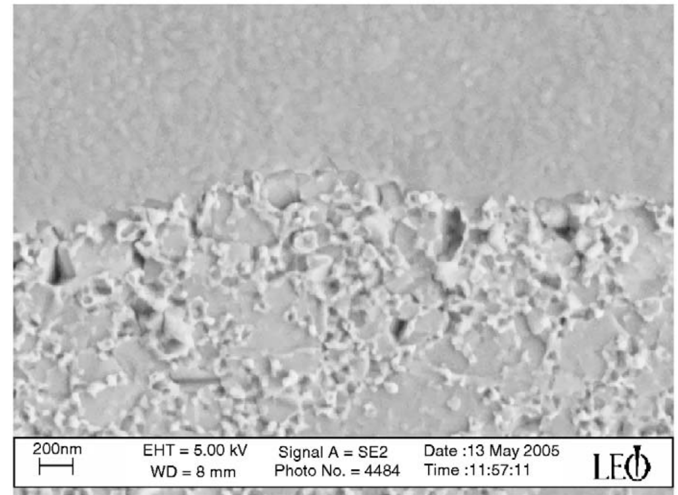

Fig. 2. Bi/Cu/Bi absorber removed by hand, Scanning Electron Microscope (SEM) image of underlying Au TES surface. At top the Au TES surface protected by photoresist layer, bottom half-Au surface contacted Bi mushroom absorber. Notice pits in Au surface and raised Bi–Au compound.

measured Bi surface roughness. The modeling showed that all the Cu is contributing to the conduction when the Cu thickness reaches 300 nm. Applying this result, our absorber design changed from multiple couple layers to a single thick Cu layer to maximize lateral thermalization per volume of Cu [2].

3. TES–absorber interface chemistry

We have made six separate fabrication runs using the same design and fabrication procedure: Mo/Au TES thermometer (50/300 nm, respectively) and Bi/Cu/Bi mushroom absorber (0.1/0.6/6.5 μm, respectively). Despite identical fabrication steps the six different wafers show markedly different Au–Bi (TES–absorber) interfaces. Three out of the six wafers have pristine Au–Bi interfaces—no evidence of diffusion. One wafer showed a Cu–Au solid solution when the Au TES surface was exposed (the Cu layer diffused through the 0.1 μm thick Bi layer and the Cu–Au mixed together). The Cu mixing was extensive enough to cause the Au TES layer to delaminate from the Mo layer showing exposed Mo layer sections when the mushroom absorber was removed. The other two wafers showed considerable Bi–Au diffusion, Fig. 2. In SEM images, pits in the Au TES surface are clearly visible from where the Bi/Cu/Bi absorber stem contacted the Au. All pixels with Bi/Cu/Bi mushroom absorbers from both wafers with Bi attacking the Au surface had a measured T_c of 170 mK, devices measured from the same chip without absorbers had T_c's of 100 mK (the designed target T_c).

Possible explanations for interface variation between wafers are: (1) there is an unintentional diffusion barrier on the pristine Au–Bi interface wafers, (2) the wafers with attacked surfaces were subjected to higher temperatures.

When in our fabrication are the diffusion processes occurring? After monitoring the temperature of a wafer going through the entire process we found that the maximum temperatures reached are 100 °C for 20 min (photoresist soft bake) and 123 °C for 3 min (waxing down a backing wafer to support the absorber structures during the back etch).

To investigate further, chips from all six wafers described were annealed at 123 °C for 15 h in vacuum followed by Bi/Cu/Bi mushroom absorber removal and SEM with Energy Dispersive Spectroscopy (EDS) examination of the exposed surfaces. For all six wafers the backside of the mushroom absorbers had small dark Cu spots (Cu–Bi diffusion). We found that all three wafers that had prior surface attacks were attacked completely through the 300 nm Au layer and Bi reached the Mo TES layer. In all three wafers with perfect interfaces neither the Bi nor Cu spots mixed with the TES Au. A similar annealing test was performed at 170 °C and the results were the same. Completely mixed Bi–Au eutectic-type structure was seen for the three wafers with attacked surfaces, and the three pristine interface samples still showed no signs of Bi–Au diffusion or Au–Cu diffusion despite nearly complete Cu spot coverage on the back surface of the mushroom absorbers.

Our working hypothesis to explain this behavior is that the three wafers that have diffusion resilient TES Au surfaces have a diffusion barrier layer of unknown origin. EDS has been unable to identify an impurity layer. Attempts of intentional diffusion barriers have been made using Ti and Ge, both tests showed Au mixing with the barrier layer.

To further study the temperature effects on our devices we have done in situ R vs T annealing tests on multilayer strips. Resistance changes in time when the temperature is held constant correspond to compositional changes in the strip, and when the sample is cooled the R vs T curve does not retrace. Our tests consist of ramping to a set point

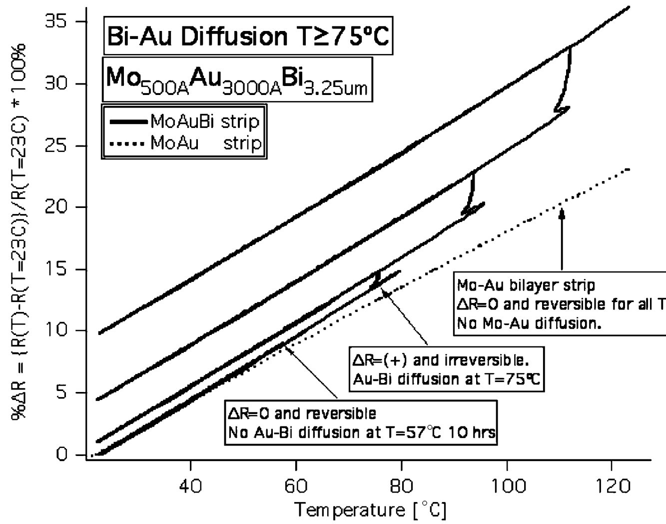

Fig. 3. Onset of Bi–Au diffusion.

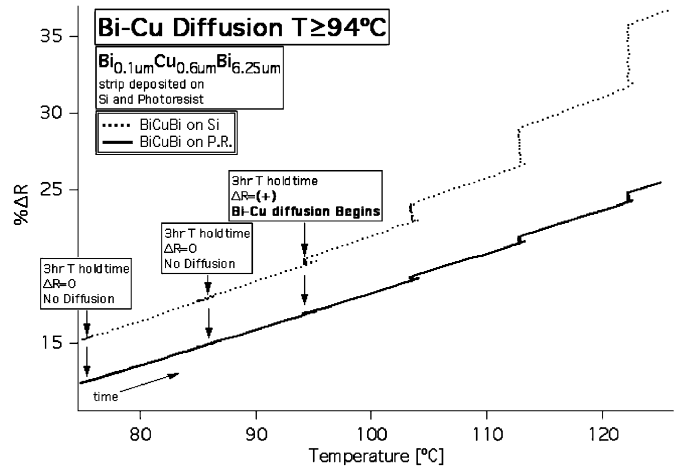

Fig. 4. Onset of Bi–Cu diffusion. Temperature soaks of 3 h at 75, 86, 94, 104, 113, and 122 °C. A positive resistance change at constant temperature and Bi–Cu diffusion observed at 94 °C and above.

temperature soaking for 3–10 h and measuring the resistance change. When Au only, Cu only, Bi only, or Mo/Au only strips are annealed their resistance changes at constant temperature are either negative or zero. Positive resistance changes at constant temperature in multilayer strips have been an unambiguous signature of interlayer diffusion, confirmed repeatedly by post-annealing SEM investigations of interfaces.

The six previously mentioned wafers, three with perfect Au interfaces and three with chemically attacked interfaces, each have chips on the wafer with resistance test strips consisting of the entire multilayer stack (0.05 μm Mo, 0.3 μm Au, 0.1 μm Bi, 0.6 μm Cu, 6.5 μm Bi). The *R–T* data are consistent with the observed SEM tests, wafers with perfect interfaces remained perfect even up to 170 °C. To isolate Bi–Cu from Bi–Au diffusion, *R–T* annealing experiments on Mo/Au/Bi and Bi/Cu/Bi strips were conducted. For Mo/Au/Bi strips the *R–T* traces are reversible up to 75 °C, the onset temperature of Au–Bi diffusion, Fig. 3. The Bi/Cu/Bi curves are reversible up to 90–95 °C, the onset temperature of Bi–Cu diffusion, Fig. 4.

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