A Mo-Cu superconducting transition-edge microcalorimeter with 4.5 eV energy resolution at 6 keV

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Abstract

We describe a superconducting transition-edge microcalorimeter with 4.5 ± 0.1 eV Full Width at Half Maximum (FWHM) energy resolution for Mn Kα x-rays from an ^{55}Fe source. The thermometer consists of a photolithographically patterned Mo-Cu superconducting proximity bilayer with a 93 mK transition temperature. The device is fabricated on a Si_3N_4 membrane patterned as a “flyswatter” to control the thermal conductance to the heat bath. The sensor is voltage biased with an on-chip shunt resistor, and the current is measured by a two-stage SQUID amplifier consisting of a single first-stage SQUID and a series-array second-stage SQUID.

We are developing x-ray microcalorimeters based on transition-edge sensors (TES) for x-ray astronomy and materials analysis. These devices consist of a TES thermometer, a weak thermal link to a heat bath, and a semimetal or normal-metal absorber to increase the x-ray absorption efficiency. Our TES thermometers are a bilayer of a superconductor and a normal metal. Due to the proximity effect, the bilayer acts as a single superconducting film with a transition temperature lower than that of the superconductor. The weak thermal link to the heat sink is created by fabricating the TES on a silicon nitride membrane.

We voltage bias our TES thermometers, and measure the current with a Superconducting Quantum Interference Device (SQUID) amplifier. Due to the voltage bias and sharp superconducting transition, these microcalorimeters operate in the strong negative electrothermal feedback limit, which provides many benefits. Operating in this mode makes the response faster, more linear, and less sensitive to bath temperature fluctuations. It also makes it possible to simultaneously bias large arrays of microcalorimeters with slightly different transition temperatures [1].

In the past, using Al-Ag bilayers and a shadowmask fabrication process, we have demonstrated an energy resolution of 3 eV FWHM at 1.5 keV [2] and 7 eV FWHM at 6 keV [3]. While this energy resolution is sufficient for many practical materials analysis applications, improvement is needed to meet the requirements of next-generation x-ray observatories such as Constellation-X. Further, while useful for making single-pixel devices, shadowmask fabrication is not practical for making the large-format arrays required in x-ray astronomy – photolithography is preferable. While our Al-Ag bilayers have proven to be stable when fabricated with a shadowmask process, we have found that Al-Ag bilayers that are photolithographically patterned degrade over time.

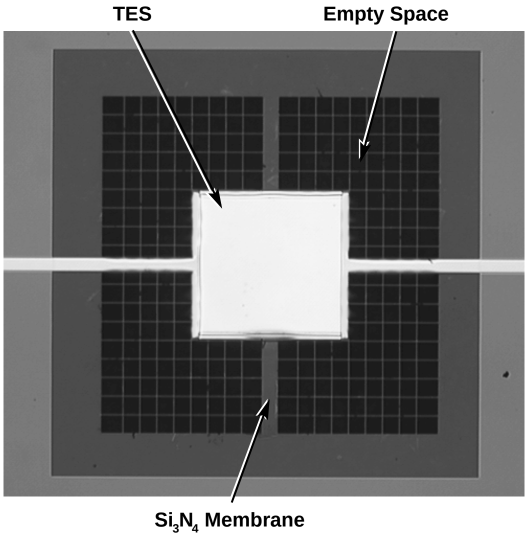

Fig. 1. A Mo-Cu TES microcalorimeter on a Si_3N_4 membrane patterned as a flyswatter. The TES has dimensions 400 μm x 400 μm. The large bars are 50 μm wide, and the small bars are 5 μm wide on 50 μm centers.

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To achieve the objectives of higher energy resolution and compatibility with photolithographic processing, we have implemented two key changes in our microcalorimeter fabrication. First, in order to lower the thermal conductance to the heat bath through the silicon nitride membrane, we have patterned our silicon nitride into a "flyswatter" pattern (Fig. 1). The flyswatter is similar to the "spiderweb" patterns used in infrared bolometers [4], but with a square geometry for convenience of fabrication. Second, we have replaced our Al-Ag bilayers with Mo-Cu bilayers, which we have shown to have good long-term stability to interdiffusion and corrosion, compatibility with photolithography and high temperature processing steps, and long electron mean-free paths in the copper, which provides fast diffusion of heat.

The devices are fabricated on double-side polished, 250 μm thick, [100] Si wafers that have had 250 nm of low-stress Si_3N_4 deposited on both sides by LPCVD. Alignment marks are fabricated on both sides of the wafer to permit front-to-back registration. Windows are then opened in the Si_3N_4 film on the wafer backside, and the wafer is etched in a one-sided etching apparatus in a KOH solution (50% Transene PSE 200 [5] and 50% H_2O) at 95 C to remove all but 50 μm of Si under the windows.

The bilayer is deposited by dc sputter deposition on the wafer front side. First 60 nm of Mo are deposited followed by 200 nm of Cu. The bilayer is patterned by two successive wet etches. The Cu is etched using an ammonium-persulfate-based etch (50% APS Cu Etchant 100 [5] and 50% H_2O). The Mo is etched using a solution of 80% HPO_3, 5% CH_3COOH, 5% HNO_3, and 10% H_2O. Normal-metal bias resistors and side banks on the TES are fabricated by e-beam evaporation of 5000 nm of Cu through a lift-off stencil. The Cu TES banks are used to create fully normal edge boundary conditions along the TES: calculations indicate that superconducting boundary conditions, while preferable, are difficult to reliably achieve with simple fabrication methods. In the future, a bismuth absorber will be deposited directly on top of the TES to increase the x-ray absorption cross-section, but this step was not yet implemented in the device described in this paper. The bismuth does not significantly affect the electrical properties or heat capacity of the device.

Photoresist is applied to the front of the wafer and patterned for the flyswatter Si_3N_4 etch. The wafer is returned to the one-sided etching apparatus and the KOH etch is repeated until the Si is completely removed, leaving suspended membranes. The wafer is then diced. The flyswatter is formed by removing the Si_3N_4 by reactive ion etching of single detector chips. The photoresist is removed by ashing the chip in an oxygen plasma.

The low impedance of our TES thermometers (normal $R \approx 17$ mΩ) allows them to couple efficiently to SQUID amplifiers, which have extremely low noise, operate conveniently at the base temperature, and generate extremely low power. Because the electrical bandwidth of the detector is limited by the L/R time constant set by the SQUID and stray inductances and the detector impedance, the stray inductance in the circuit must be kept small. We improve the speed and stability of our detectors by mounting a low inductance first-stage SQUID chip adjacent to the TES chip. As mentioned previously, the shunt resistor is fabricated on the TES chip, and the TES chip is connected to the first stage SQUID chip by wirebonds (fig. 2). The output of the first-stage SQUID is further amplified by a series-array SQUID [6] operated at 4 K. The large output voltage (5 mV) and high output impedance (100 Ω) of the series-array SQUID permits the use of simple room temperature amplifiers. Further, this two-stage SQUID configuration can be extended to a SQUID multiplexer design which uses only one output channel to instrument a large number of TES devices and first-stage SQUIDs [7].

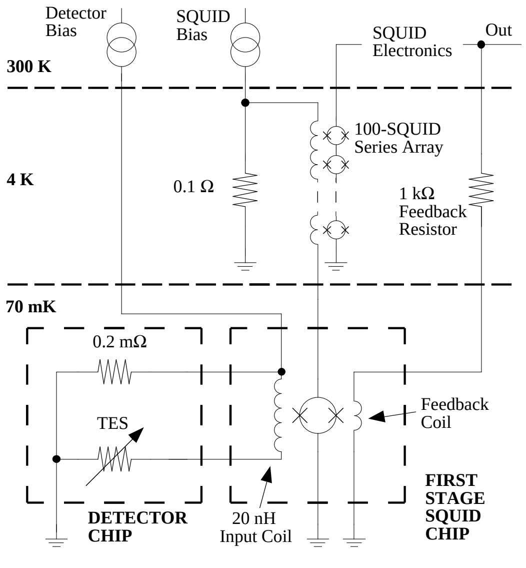

Fig. 2. Electrical bias circuit of the TES microcalorimeter.

The detector was heat sunk to the cold finger of an adiabatic demagnetization refrigerator, and cooled to 60 mK. When biased, the detector self-heated to its 93 mK transition temperature. Mn Kα and Kβ x-rays from a room-temperature ^{55}Fe source were incident on the film through a series of infrared-blocking x-ray windows and a 150 µm aperture centered on the 400 µm x 400 µm TES. The resulting pulses of reduced current were measured by the SQUID and digitized. When the bias conditions were optimized for speed, pulse 1/e recovery times of 700 µs were observed, with energy resolution of about 5 eV FWHM. When the detector was optimized for energy resolution without regard to response time, 1/e recovery times of about 2 ms were observed. The digitized x-ray pulses were analyzed using a Wiener optimal filter. In Fig 3(a), the resulting spectrum is shown with clear Mn Kα and Mn Kβ peaks. Below the Mn Kα peak, an artifact peak is seen which is caused by x-rays absorbed directly in the Si_3N_4 membrane below the TES. We have previously shown that this artifact is eliminated when a bismuth layer is added on top of the TES. In previous detectors, our energy resolution has been approximately the same with and without the bismuth absorber.

In Fig. 3(b), the measured Mn Kα lines are fit to the line profiles measured by Holzer et. al. [8], convolved with a Gaussian microcalorimeter response by a weighted least squares method. The Mn Kα1 and Kα2 lines are clearly separated, and a Gaussian instrument energy resolution of 4.5 ± 0.1 eV FWHM is determined.

In summary, Mo-Cu TES microcalorimeters have been fabricated in a fully lithographic process. The Si_3N_4 membrane was patterned as a flyswatter to control the thermal conductance. An energy resolution of 4.5 ± 0.1 eV FWHM was measured at 6 keV.

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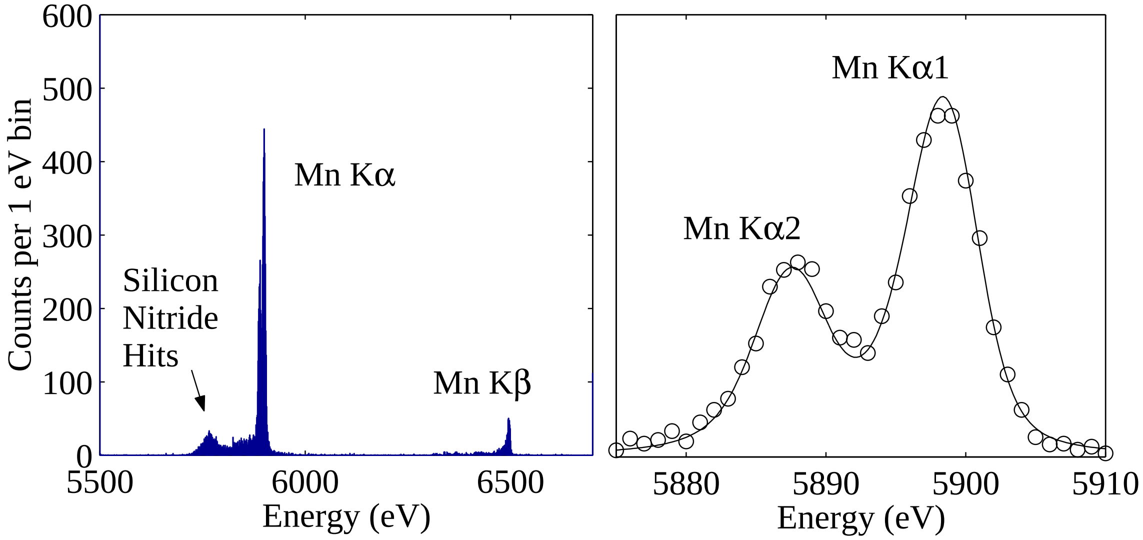

Fig. 3. Spectrum of an ^{55}Fe source. (a) Wide spectrum (b) a weighted least-squares fit of the Mn Kα x-rays to a convolution of the theoretical line profile and a Gaussian instrument response. An instrument resolution of 4.5 +- 0.1 eV FWHM is determined.