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Molybdenum–gold proximity bilayers as transition edge sensors for microcalorimeters and bolometers

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Abstract. Mo/Au proximity bilayers as transition edge sensors (TESs) are promising candidates for low-temperature thermometry. The transition temperature of the bilayers can be easily tuned between 50 and 600 mK, yielding sensors which can be used in a variety of calorimetric and bolometric applications. With phase transition widths of less than 1 mK, Mo/Au TESs show very high temperature sensitivity ($\mathrm{d}(\log R)/\mathrm{d}(\log T) \sim 2500$). Also, Mo/Au TESs show improved thermal and chemical stability compared to most other bilayer configurations. Fabrication issues and detector performance of Mo/Au TESs on Si_3N_4 membranes are discussed.

1. Introduction

In general, a microcalorimeter or bolometer consists of an absorber with heat capacity $C(T)$ and a temperature dependent resistance (thermometer) linked to a heat bath through a thermal conductance $G(T)$. The incoming radiation heats the absorber which causes a change in the resistance of the thermometer. The resistance change can be measured as a voltage or current change, depending on the biasing method. The thermometer is characterized by its fractional resistance change with temperature, α, which is defined by $\alpha = \mathrm{d}(\log R)/\mathrm{d}(\log T)$.

As a measure of the temperature sensitivity of the thermometer, α plays a key role in the detector performance: a high value of α leads to a high detector responsivity, and noise performance near the thermal fluctuation limit.

Our transition edge sensors (TESs) show maximum α values of 2500, which is an improvement over commonly used standard semiconductor thermistor thermometers by a factor of 400. Given such a high sensitivity, our TES can operate in extreme electrothermal feedback (ETF) which dramatically improves detector stability, dynamic range and response time [1].

Conceptually, a TES is a superconducting material biased within the transition from normal to superconducting state. By using normal-superconducting metal bilayers instead of elementary superconductors, the transition temperature (T_c) of a bilayer can be tuned to the desired operating point and very small transition widths ($\mathrm{d}T_c$) are achievable [2].

Within the last year, we started to investigate a new type of TES, a molybdenum/gold (Mo/Au) proximity bilayer, which shows improvements in chemical, electrochemical and thermal stability compared to previous bilayer combinations [3]. Therefore, the Mo/Au bilayer TES promises long-term stability. Furthermore, a TES microcalorimeter or bolometer can be produced using standard microlithographic techniques without any degradation of its detector performance.

In ETF operation, our Mo/Au TES sensor is voltage biased within its transition and temperature biased well above the bath temperature T_b (i.e. $T_b \ll T_c$). At the bias point the electrical resistance of our TES is typically 10 mΩ–100 mΩ. Any resistance change is detected by a highly sensitive SQUID via the current change through the TES.

ETF effectively counteracts any temperature (and therefore resistance) excursion from the bias point by introducing or reducing power via the electrical circuit. Therefore, our TES has the ability to self-bias within its superconducting transition and can operate on a much shorter effective time scale than its intrinsic thermal time constant ($\tau_0 = C/G$). The relationship between the effective time constant (τ_{eff}) and τ_0 is given by $\tau_{eff} = (n/\alpha)\tau_0$, where $n = 4$ for silicon nitride (Si_3N_4) [4].

2. TES applications

There are several ongoing investigations at NASA/Goddard employing Mo/Au TES on Si_3N_4 membrane detector technology. We present the following applications: a TES as a thermometer for x-ray calorimetry, a TES as a thermometer for far-IR bolometry and a monolithic stripline bolometer with a TES thermometer. The choice to fabricate the TES on Si_3N_4 is to reduce the $G(T)$ and $C(T)$ of the detector configuration. The $G(T)$ is tuned for each application by perforating the Si_3N_4 membrane around the device.

The parameters for the TES detector element are chosen by performing an optimization between the sensitivity of the

Table 1. Design parameters for presented TES detectors.

	$C(T_c)$ [J K^{-1}]	$G(T_c)$ [W K^{-1}]	τ_{eff} [ms]	T_c [K]
A	1×10^{-11}	1×10^{-9}	0.1	0.1
B	1×10^{-12}	5×10^{-12}	1–10	0.3–0.4
C	5×10^{-12}	1×10^{-11}	2	0.3

device, its speed and its thermal environment. The sensitivity is related to the achievable value of α and the conductivity $G(T)$ between the thermistor and the bath—higher α and lower $G(T)$ generally implies a more sensitive detector. The time constant of the device is related to $C(T)$, α and $G(T)$. A lower bath temperature usually allows the design of sensitive detectors.

Since a TES is to be operated within its transition, a given α and energy range set the upper limit to the total $C(T)$, whereas the $G(T)$ is given by the total $C(T)$ and the desired response time of the detector.

2.1. X-ray TES

High-resolution x-ray spectroscopy is an important tool in the understanding of some of the most energetic phenomena in the universe, involving, for example, quasars and active galactic nuclei. The design of our TES microcalorimeters is targeted for high energy sensitivity and fast detector response for energies ranging between 1 and 10 KeV. In order to obtain the necessary $C(T)$ and provide effective x-ray absorption and thermalization, bismuth/copper multilayers are added (2 μm to 10 μm) as absorbers on top of the TES devices. In addition, operation temperatures of less than 0.1 K boost the detector sensitivity. For design parameters, refer to row A in table 1.

2.2. Far-IR TES

Over the last few decades, millimetre-wave and far-infrared astronomy have increased our understanding of galactic processes, large-scale structure and the early history of the cosmos. This has been enabled by continuing improvements in observational methods and in instrumentation. One area in which instrumentation can be further improved is detector sensitivity—many measurements are not photon noise limited. The most sensitive detectors at present for far-infrared broadband measurments are bolometers. The sensitivity of these devices can be improved by a factor of 30 using TES thermometers instead of traditional doped semiconductor thermistors. For design parameters, refer to row B in table 1.

2.3. Stripline TES

More than 99% of the photons in the universe lie within the millimetre and sub-millimetre wavelength bands. Emitted by diffuse sources, the radiation can be characterized by three properties: its frequency spectrum, its brightness and its polarization. Of these three properties, the spectrum of the sources is the best known. The intensity power spectra of the sources at small angular scales where the most interesting cosmological information is to be found is less known. The polarization state of each of the sources remains unknown at any angular scale.

The monolithic stripline bolometer is composed of the following elements: a polarization selective 'launcher'—designed to couple the TE_{10} mode from a square or rectangular waveguide to a stripline transmission line, a band-defining filter realized in the stripline, a resistive termination in a thermally isolated section of stripline and an integrated TES microbolometer which measures the temperature rise in the termination. For design parameters, refer to row C in table 1.

3. Fabrication

Fabrication begins with the deposition of 0.5 μm of low-stress Si_3N_4 on both sides of a double-sided polished $\langle 100 \rangle$ silicon wafer by low-pressure chemical vapour deposition. The starting silicon substrate ranges in thickness between 200 and 400 μm. Holes the size of the desired free standing area of the Si_3N_4 membranes are etched using reactive ion etch (RIE) in the nitride on the back side of the water. This is done by photolithographically patterning a photoresist (PR) etch mask, etching the nitride through the PR and removing the PR in oxygen plasma. The wafer is then placed in anisotropic KOH chemical etch (80 °C, 45% solution) to form the free standing Si_3N_4 membranes.

In an ultra-high-vacuum deposition system, the Mo/Au bilayers are thermally deposited by electron beam evaporation at a base pressure of 1×10^{-10} Torr. The molybdenum (Mo) layer is deposited first at a rate of 0.5 nm s^{-1} on the Si_3N_4 water at a temperature of 500 °C. Under these conditions, our thin Mo films (40–70 nm) show low film stress and sharp transitions at about 700 mK. Without breaking vacuum, the gold (Au) layer (100–300 nm) is deposited next at a rate of 0.1 nm s^{-1} on the Mo layer at temperatures between 10 and 200 °C. We intend to investigate other methods of bilayer deposition including sputtering both Mo and Au and sputtering Mo and e-beam Au.

The Mo/Au bilayer structure is defined on the Si_3N_4 membrane using photolithographic techniques aligning to the back of the water and chemically etching the Au in gold etch ($KI{:}I_2{:}DIW$) and the Mo in molybdenum etch ($HNO_3{:}H_2SO_4{:}DIW$). The contact leads and the bonding pads are produced in a series of masking, deposition and lift-off steps. For lift-off, we use Futurrex NR5-6000PY PR which produces overhang structures of 3 μm and removes in acetone. Currently, the contact leads are aluminium (Al) which when in extended contact with Au corrodes to form a compound known as the 'purple plague'. We intend to investigate using niobium (Nb) or granular Al as the contact material. In the case of Nb, the leads will be sputtered and formed using lift-off. See figure 1 for a TES device fabricated as described.

Perforations are made in the free standing Si_3N_4 membrane to control the $G(T)$ of the device using RIE. The absorber is placed on top of the TES structure using photolithography and lift-off techniques. The absorbing material and the thickness of absorber are dependent on the specific application.

Heat treatment studies on Mo/Au bilayers were conducted in order to test and prove their processibility.

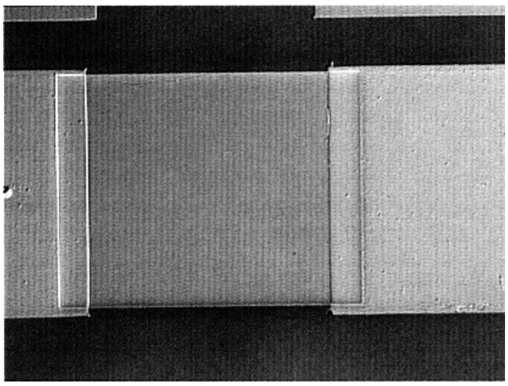

Figure 1. A 500 × 500 μm^2 Mo/Au TES with Al leads on an Si_3N_4 membrane.

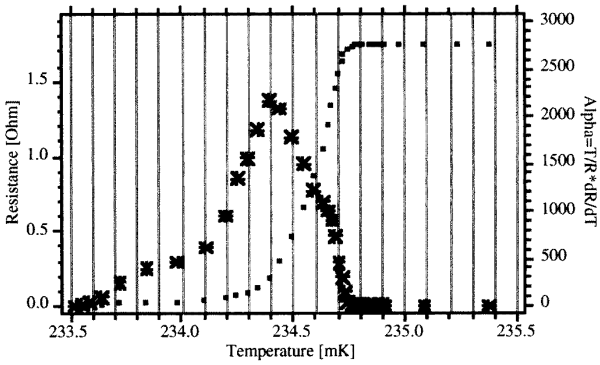

Figure 2. The RFT curve and corresponding α of an Mo/Au superconductive phase transition.

The Mo/Au bilayer system was annealed at temperatures in excess of the actual temperature range and duration within a standard microlithographic process. Comparisons of test results before and after the heat treatment revealed no significant change in the T_c and dT_c of the Mo/Au bilayers.

4. Readout and results

Mo/Au bilayer characterization and detector test runs are carried out in an Oxford dilution refrigerator and in an IR Lab ^{3}He refrigerator. The dilution refrigerator is used for experiments in the temperature range between 50 and 300 mK, while the ^{3}He refrigerator covers the range between 300 mK and 1 K. As part of the bilayer characterization, the T_c and dT_c of a bilayer strip are obtained by recording its resistance as a function of temperature (RFT). The resistance of an Mo/Au bilayer strip is read out in a four-wire scheme by an ac resistance bridge during a temperature sweep. With the current experimental setup, resistance changes down to a fraction of a milliohm and temperature changes of about 0.1–0.2 mK can be resolved.

Test results on our Mo/Au bilayer strips indicate very good tunability and sufficient reproducibility of T_c and very steep transitions of 1 mK and smaller. We have produced bilayers with T_c values between 100 and 600 mK and maximal values of α of about 2500. Figure 2 presents an RFT of a typical phase transition with a dT_c of less than 1 mK, and an α value of 2300 at the steepest part of the transition. For T_c ranging between 200 and 300 mK, the total distribution of T_c on a 4 in wafer was found to be less than ±8 mK. In addition, critical current measurements were carried out. First results showed values for the critical current density J_c between 100 and 160 A cm^{-2} at $T/T_c = 0.9$, which is in good agreement with theoretical J_c models of very thin superconducting films.

To date, our TES detectors are read out by dc SQUID systems. First detector tests have already been performed in the dilution refrigerator using commercially available dc SQUID sensors (from Conductus) which have input inductances of 600 nH. Two sensors are installed in the ^{4}He bath of the dilution refrigerator. A magnetically shielded twisted pair of superconducting NbTi wires provide the electrical connection between the TES circuit and the SQUID sensor which is inside a superconducting Nb shield.

A typical I–V curve of a TES microcalorimeter with Al leads shows two distinct voltage ranges where the slope dI/dV is negative, representing bias conditions within phase transitions. The range at the higher bias voltage is related to the phase transition of the Mo/Au bilayer. The one at the lower voltage represents the phase transition of the contact areas of the Al leads and the TES Au layer. This region is presumably due to the formation of the 'purple plague'. Preliminary results on first x-ray irradiation experiments indicated an energy resolution of about 115 eV at 3 keV for that type of detector (i.e. an Mo/Au TES with Al leads). The detector was voltage biased within the Mo/Au transition.

5. Conclusion

Mo/Au proximity bilayers as transition edge sensors are ideal for bolometric and calorimetric applications requiring low-temperature thermometry. Fabricating the TES on patterned Si_3N_4 membrane substrate reduces the thermal conductance and the heat capacity of the devices. Mo/Au bilayers have shown very sharp transitions of less than 1 mK and are, therefore, highly temperature sensitive. Our goal is to proceed with the development of TES bolometers and microcalorimeters suspended by silicon nitride membrane structures.

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