Fabrication of Superconducting Bilayer Transition Edge Thermometers and their Application for Spaceborne X-ray Microcalorimetry

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***Abstract*—The transition between normal conduction and superconductivity in superconducting materials can be exploited as a highly sensitive thermometer. Transition temperatures can be tailored through the selection of materials, their component material thicknesses, and the comparative ratios in cases of more than one material. Two bilayer configurations, Ag/Al and Au/Mo, are examined, including details of preparation, testing, and encountered difficulties. Proposed designs for spaceflight detector applications are discussed.**

I. Introduction

Fourteen years after the invention of X-ray calorimeters [1], we are approaching the practical limits of our semiconductor thermistors. Employing HgTe absorbers on silicon substrates containing ion implanted thermistors, the resolution has been steadily improved to a limit of about 7 eV @ 6 KeV [2].

To further improve energy resolution and to use absorbers with high heat capacities, we recently started to investigate superconducting transition edge sensors (TES) as thermometers. In comparison with thermistors, transition edge sensors are able to boost the temperature sensitivity by a factor of 100, where the sensitivity is defined by $\alpha = T/R \times dR/dT$ [3], [4].

Our transition edge sensors consist of thin normal conductor/ superconductor bilayers running at temperatures within their transitions. Exploiting the proximity effect between its normal layer (N) and its superconducting layer (S), the transition temperature of a superconducting bilayer can be specifically designed, and, furthermore, sharp transitions can be expected [5].

However, material aspects of a NS bilayer are of great importance. For instance, atomic interdiffusion and electrochemical reactions can observably alter or deteriorate the detector performance of a TES. In this paper, we will concentrate on this critical issue. Two material combinations, Ag/Al and Au/Mo, will be presented. Lastly, we will give a brief overview of the current status of our detector development efforts.

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II. Proximity Bilayer Configurations

Samples of Ag/Al bilayers were produced in an ultra-high vacuum deposition system at a base pressure of 1×10^{-8} Torr. Silver and aluminum were thermally deposited on silicon wafers by using an electron beam gun. The Si substrates were not actively heated or cooled during deposition. The deposition rates and thicknesses were accurately monitored and controlled. Best results were obtained at deposition rates of about 1 nm/sec for silver and 1.5 nm/sec for aluminum. The film thickness range for silver was between 50 and 120 nm, and for aluminum, between 25 and 50 nm. At T=4K, the maximum electron mean free path in the Ag layer is 250 nm, and in the Al layer 65 nm. Depending on their total thicknesses and thickness ratios (N/S), the transition temperatures of our Ag/Al bilayers were between 50 mK and 300 mK. For example, a critical temperature (Tc) of 300 mK was measured for a bilayer of total thickness 150 nm and thickness ratio N/S=2. For the same total thickness and a ratio of N/S=3, the transition temperature dropped to about 70 mK. The transition widths, dTc, of our Ag/Al bilayers were between 1 mK and 0.4 mK, yielding high values of α. The maximum value of α was determined to be 400.

To investigate the transition behavior induced by the proximity effect, photolithography was used to pattern the proximity bilayers. The geometry of that pattern was a strip with dimensions 80 micron by 2.5 mm. Transition curves were taken by a resistive four point measurement using an AC resistance bridge. The same readout technique was used for all our bilayer configurations.

Since aluminum and silver thin films are easily prepared and the transition temperature of aluminum is easily obtained and highly reproducible (Tc(Al)=1.19 K), Ag/Al bilayers have become one of the most popular proximity bilayer configurations. TES microcalorimeters using Ag/Al bilayers have already achieved an energy resolution of about 3 eV for 1.5 KeV X-ray photons [6].

Unfortunately, under certain circumstances, our Ag/Al bilayer configuration has shown a dramatic deterioration of its detector performance. Possible causes for degradation are:

- Elevated temperatures - increased atomic interdiffusion and eventual compound formation.
- Electrochemical reactions - water (humidity) acts as an electrolyte, causing corrosion of the bilayer.
- Chemical exposures - most photoresists contain sulphur which can enhance corrosion. Furthermore, aluminum is also attacked by many photoresists and resist developer solutions.

In order to develop more stable and robust transition edge sensors, we started to investigate gold/molybdenum proximity bilayers on silicon substrates. It has been shown that the Au/Mo configuration is less diffusive even at higher temperatures [7]. For temperatures below 300°C, calculations predict no diffusion at all. In addition, both Au and Mo are highly resistant to corrosion.

The first part of our investigations was to study the superconducting behavior of pure molybdenum thin films (target purity 99.999%). Mo was evaporated from an electron beam gun at a background pressure of 3 - 4 x 10^{-9} Torr. The deposition rate was 0.5 nm/sec. The silicon substrate was heated to 500°C, which is the maximum temperature we can achieve at the moment. Reference [8] indicates a stress minimum in thin Mo films at substrate temperatures between 800°C and 900°C.

Mo films were also prepared at 200°C for general comparison. In order to avoid molybdenum silicide formation at the interface between Mo and Si, a 300 nm thick SiO_2 layer was thermally grown on the Si substrate as a diffusion barrier. A 500 nm thick Si_3N_4 surface layer (applied via chemical vapor deposition) on a silicon substrate was used as our second choice for a diffusion barrier. Fig. 1 shows the resistance-temperature behavior (R(T)-curve) for thin Mo films on SiO_2 and Si_3N_4 deposited at 500° and 200°C. For substrate temperatures of 500°C, the figure shows curves of two independent samples. Mo films deposited at 500°C showed transition temperatures between 700 and 710 mK on SiO_2 and between 580 and 590 mK on Si_3N_4. The transition widths were sharp (dTc=3-5 mK) and reproducible. The difference in substrate deflection before and after the deposition at 500°C was negligible, indicating low stress films. The residual resistance ratio (RRR) values are 3 for Mo on SiO_2 and 2 for Mo on Si_3N_4. Compared to SiO_2, the Si_3N_4 surfaces seem to induce lower transition temperatures also at lower substrate temperatures.

Next, we studied the behavior of Au/Mo bilayers. On a 50 nm thick Mo film on SiO_2 prepared at 500°C, a gold film was deposited after the Mo film had been cooled to 200°C, in order to rule out any thermally activated diffusion between the two metal layers during Au deposition. The gold film thicknesses were 120 nm and 130 nm for two samples. The Au/Mo bilayers were prepared without breaking vacuum. First results on our Au/Mo bilayers on SiO_2 are given in Fig.2. It shows two Au/Mo bilayers both with a total thickness of 180 nm and N/S=2.6. The transitions are about 0.5 mK wide.

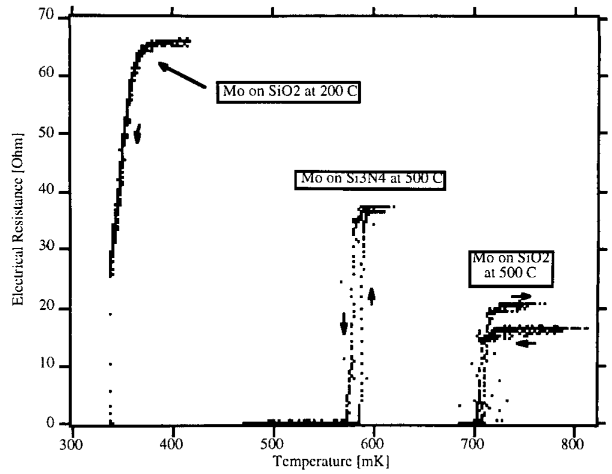

Fig. 1. Transition curves of 50 nm thick Mo films prepared on different substrates at 200°C and 500°C.

III. DETECTOR CONCEPTS AND SPACEBORNE APPLICATIONS

As a first concept, our microcalorimeter consists of a several micron thick copper/bismuth multilayer as the X-ray absorber. It completely covers the transition edge thermometer whose sensor area ranges between 200 μm^2 and 600 μm^2. The microcalorimeter is deposited on a 500 nm thick Si_3N_4 membrane which thermally decouples the microcalorimeter from the heat sink at 50 mK. A submicron thick gold strip bridges the membrane and connects the TES to the sink. This thermal link can be easily tailored, by altering the gold strip width, to provide a thermal conductance (G) varying between 10^{-9} and 10^{-10} W/K. For optimal detector performance, with minimal thermal noise, the transition edge sensor has to have its transition temperature between 70 mK and 120 mK. This is based on an available heat sink temperature of 50 mK.

The realization of this concept has been almost completed. Both presented bilayer configurations are currently being implemented.

The above described detector scheme is a potential detector for a soft X-ray spectrometer in a future NASA mission which requires an energy resolution of 2 eV for 6 KeV X-rays. This future spaceborne spectrometer requires an arrayed detector of at least 30 by 30 pixels which are closely packed to 98% of the detector filling factor.

The membrane design does not scale well to this requirement due to the complexity of bringing out electrical contacts and providing the necessary thermal conductance. This scheme is solely useful for producing, testing, and optimizing single TES microcalorimeter detectors. Therefore, 3-dimensional micromachined silicon pixel structures are currently under investigation. These microstructures are designed to mechanically suspend each microcalorimeter element in a closely packed array, providing each pixel with electrical contacts and an independent thermal link to the heat sink.

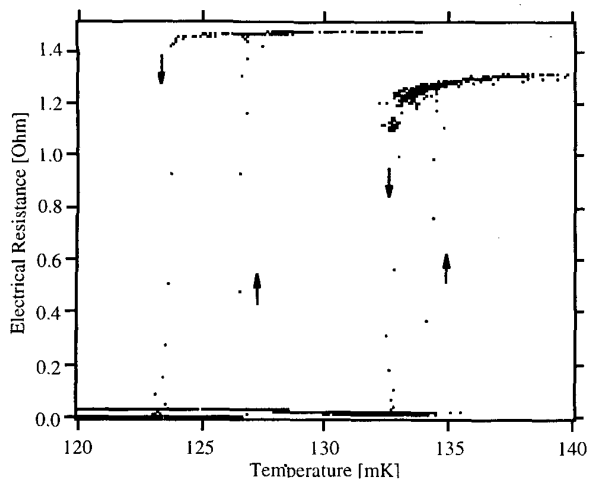

Fig. 2. Transition curves of two Au/Mo bilayers of total thicknesses 180 nm and N/S=2.6 . The hysterese was caused by the RT-measurement.

IV. CONCLUSION

Proximity N/S bilayers are ideal candidates for tailoring transition temperatures for TES applications. Aluminum/silver bilayers have shown very sharp transitions of less than 1 mK and are, therefore, highly temperature sensitive. However, our Ag/Al bilayers seem to lack thermal, electrochemical, and chemical stabilities. For spaceborne applications, more stable and robust proximity bilayer configurations, like Au/Mo, are desirable. Our goal is to proceed with the development of robust TES microcalorimeters suspended by silicon structures.

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