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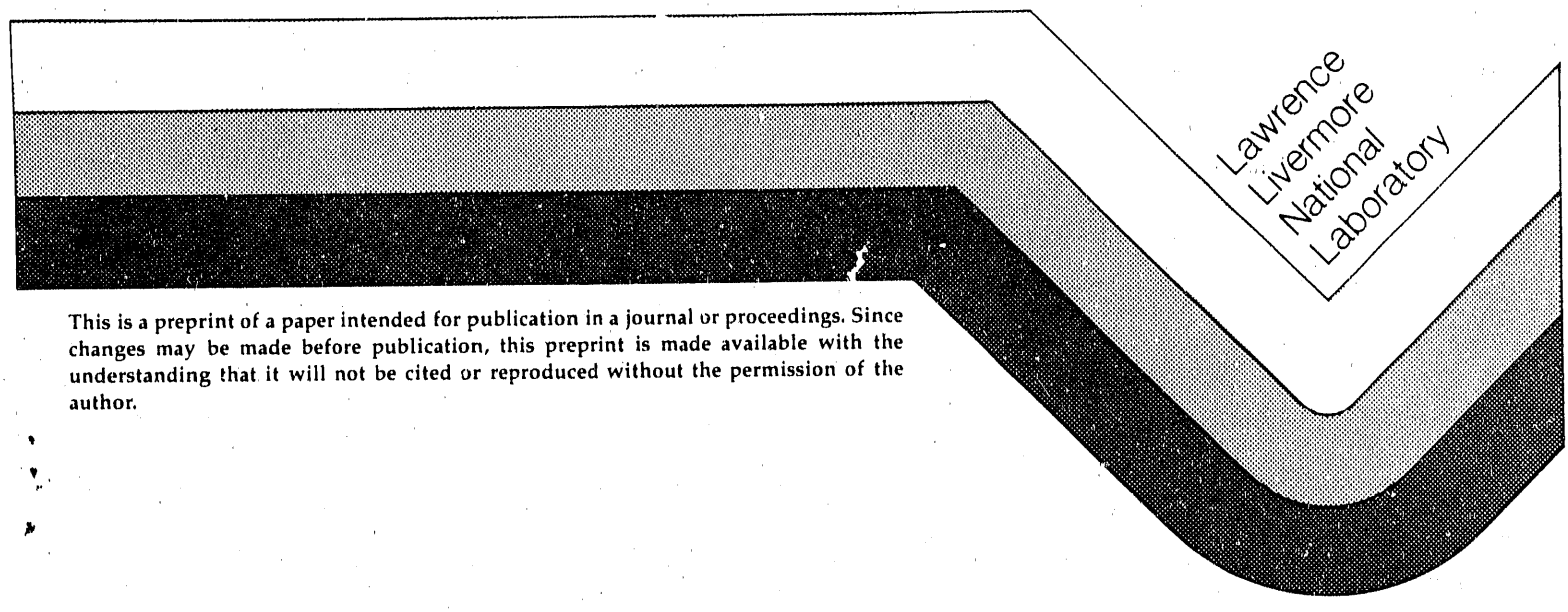

X-ray microcalorimeters with germanium resistance thermometers

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ABSTRACT

We report on the current status of our work on x-ray microcalorimeters for use as high resolution x-ray spectrometers. To maximize the x-ray collecting area and the signal to noise ratio, the total heat capacity of the device must be minimized. This is best achieved if the calorimeter is divided into two components, a thermal sensor and an x-ray absorber. The thermal sensor is a neutron transmutation doped (NTD) germanium resistor made as small as possible to minimize the heat capacity of the calorimeter. The thermistor can be attached to a thin x-ray absorber with large area and low heat capacity fabricated from superconducting materials such as niobium. We discuss results from our most recent studies of such superconducting absorbers and present the x-ray spectra obtained with these composite microcalorimeters at a temperature of 0.1 K. An energy resolution of 19 eV FWHM has been measured.

1. INTRODUCTION

We are developing microcalorimeters for high resolution x-ray spectroscopy. When an x-ray photon is absorbed in one of these detectors, a rise in temperature results which can be measured with great accuracy. In principle, an x-ray microcalorimeter can detect x-ray photons between 0.1 and 10 keV with nearly 100% efficiency and resolution of a few eV.[1] This high resolution and high efficiency over a broad range of energies is not available in other x-ray spectrometers. The potential applications of these detectors include atomic physics, plasma physics and x-ray astrophysics.

The x-ray induced temperature pulse of a calorimeter is measured by a thermal sensor. While capacitive[2,3] and inductive[4,5,6] thermal sensors are under development, most high resolution measurements to date have been obtained with resistive sensors. Detector arrays with ion-implanted silicon thermistors have been produced at Goddard Space Flight Center which yield 7 eV FWHM resolution[7] when operated at 0.065 K. We report here on developments utilizing neutron transmutation doped (NTD) germanium thermistors. Although NTD germanium has a larger specific heat than ion-implanted silicon, the doping in NTD germanium is more reproducible. Once the material for a given neutron dose has been characterized, one can routinely fabricate devices with the desired resistance at a chosen operating temperature.

This paper discusses the operation of these NTD germanium thermistors and their response to x-rays absorbed in the germanium, in the gold electrical contacts, and in a superconducting film deposited directly on the sample. The paper also describes our plans for improving the resolution and increasing the area and efficiency of such detectors.

2. NTD GERMANIUM CALORIMETER PERFORMANCE

2.1 Calorimeter geometry

The results presented in this section were obtained by direct irradiation of an NTD germanium calorimeter 100 μm x 100 μm x 250 μm long, operating at a temperature of 0.1 K. The two ends of the sample have been implanted with boron ions and coated with 200 nm of gold for electrical contact. Graphite fibers which are 7 μm in diameter and have been coated with 4 μm of indium, are silver epoxied to the samples. These indium/graphite wires are the electrical leads and also serve as the mechanical support for the device. The length of each wire is typically ~150 μm. The detector is biased at a voltage which is held constant for the duration of the x-ray pulse. The absorbed x-ray heats the thermistor and reduces its resistance. The resulting current pulse is amplified by a JFET negative feedback amplifier.[8,9]

2.2 Non-ohmic behavior

A thermistor is used to convert the x-ray induced temperature change into a current pulse. The resistance of the thermistor, however, does not depend only on the temperature. As the voltage across a semiconductor resistor at cryogenic temperatures is increased, the resistance falls even if the temperature of the device is fixed. We have observed this nonlinear relation between voltage and current in NTD germanium at temperatures between 0.08 K and 1.2 K.[9] It has also been observed by others in ion-implanted silicon[7] as well as NTD germanium between 4 K and 1 K[10] and at 0.02 K.[11]

This nonlinear behavior directly effects the performance of the calorimeter. As the bias voltage is increased, not only does the resistance fall, but the gradient of the resistance as a function of temperature (dR/dT) flattens severely. The detector's signal to Johnson noise ratio is therefore reduced, and the resulting energy resolution is much worse than the predictions based on the low power measurements of resistance as a function of temperature.

The nonlinear behavior also makes it difficult to characterize the basic parameters of the system. Under normal operating conditions, the calorimeter is weakly coupled to the cold sink. In this case, the resistance will fall as the bias voltage is increased due to a combination of the voltage effect and ohmic heating of the device. The usual technique for calculating the thermal conductance between the calorimeter and the cold sink, is to apply power to the device and measure the resulting change in resistance. One then assumes that all the change in resistance is due to ohmic heating, and then calculates the change in temperature using the low power resistance-temperature relation of the device. At typical operating powers and applied voltages, however, the electrical properties of the device do not follow the low power behavior, and not all of the resistance change is due to ohmic heating. Since the temperature of the device is uncertain, special test devices are required to accurately measure dR/dT at the operating power. Preferably two thermistors thermally connected to each other should be used, one monitoring the temperature of the other. Since the non-ohmic behavior varies with the volume and geometry of the resistor, numerous test devices are required to fully characterize the nonlinear effect. We have not yet undertaken such an effort, but it will be necessary to do so in order to properly optimize the thermistor and achieve the best possible signal to noise ratio.

To obtain an estimate of the resistance-temperature slope (dR/dT) encountered at operating powers, we measure dR/dT of the device with a moderate amount of power, in between the low power limit and the higher powers required for calorimeter operation. In this measurement, we apply enough bias voltage to change the resistance, but not enough to significantly heat the device. Rough estimates of the thermal conductance are adequate to determine that the device is not being heated with these moderate power levels. The resistance is measured in this way at several cold sink temperatures to determine dR/dT. The resulting dR/dT is much closer to the actual dR/dT encountered under operating conditions than the dR/dT found with measurements in the low power limit. With this improved estimate of dR/dT, we can calculate the thermal conductance, and using the decay time constant of the pulse, calculate the heat capacity.[9]

2.3 Estimation of operating parameters

The detector described above was cooled in an adiabatic demagnetization refrigerator which has a temperature stability of 2 μK rms.[9,12] Operating at a base temperature of 0.1 K, we measure a decay time constant of 0.7 msec, a thermal conductance of 3×10^{-10} W/K and a heat capacity 2×10^{-13} J/K. The fractional change in resistance per degree K with 3.5 mV across the 2 MΩ thermistor is $1/R\ dR/dT = 28\ K^{-1}$.

From these numbers, 5.89 keV of energy absorbed in the detector should produce a voltage signal of 0.18 V at the output of the preamplifier. The peak voltage measured from the gold absorption of a 5.89 keV x-ray is 0.175 V, in close agreement with the prediction. This confirms the approximate values of the heat capacity and other parameters given above, but the analysis is not sufficient to accurately predict the absolute pulse height. A much more thorough analysis of the voltage-resistance-temperature characteristics of the thermistor would be necessary to determine the exact amount of energy required to produce a given pulse height.

2.4 Pulse height analysis

The 0.1 K detector was irradiated with an ^{55}Fe source producing Mn Kα (5.89 keV) and Kβ (6.49 keV) x-rays,

and the resulting spectrum is shown in Figure 1. An electronic pulser was used to simultaneously measure the electronic noise, and the resulting line profile appears in Figure 1. As described previously,[9] these NTD germanium detectors produce two different pulse heights for each x-ray energy. The Kα, Kβ pair with lower pulse heights (and large number of counts) arise from x-rays absorbed in the germanium. When x-rays are absorbed in the gold contacts, a larger pulse height results. For x-rays absorbed in the germanium, some of the energy does not appear as thermal energy within the electronic processing time of about 1 msec.

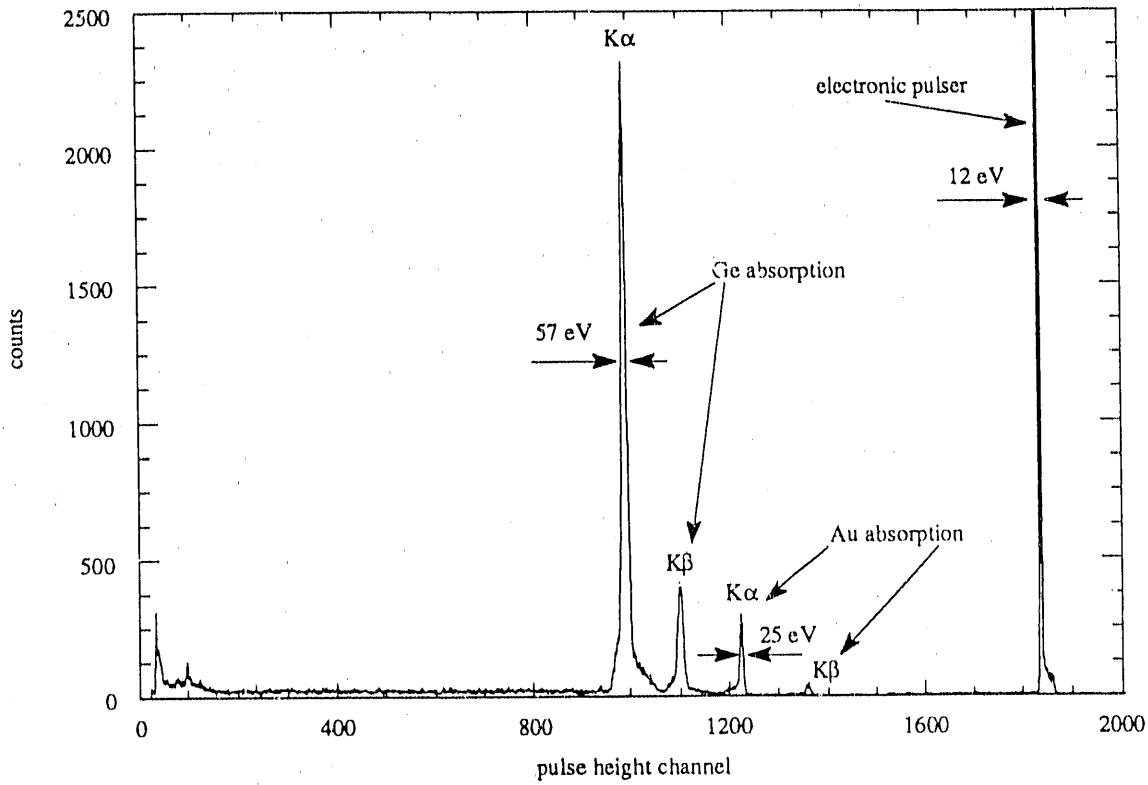

Figure 1. An x-ray spectrum obtained with an NTD germanium calorimeter 100 μm x 100 μm x 250 μm long operating at 0.1 K. The detector was illuminated with Mn Kα and Kβ x-rays giving rise to the four peaks in the center of the figure. The peak on the far right is from an electronic pulser which is used to measure the electronic noise. The 25 eV FWHM resolution indicated by the Au absorption peak includes contributions from the Mn $K\alpha_1$ and $K\alpha_2$ lines. Accounting for these two lines within the peak shown yields an actual energy resolution of 19 eV.

In solid state ionization detectors where x-rays are absorbed in a semiconductor, some of the energy goes into creating electron-hole pairs. At 77 K, 2.96 eV of x-ray energy is required, on average, to produce one electron-hole pair in germanium. Since the band gap of germanium is 0.67 eV, only 22.6% of the x-ray energy goes into electron-hole pairs. The remaining 77.4% of the x-ray energy is converted to heat during the cascade of electron energies which is initiated by the photoelectric absorption. At 0.3 K, the ratio of the germanium absorption pulse height, to the gold absorption pulse height is 0.778.[9] This strongly suggests that the electron-hole pairs in the germanium are not recombining within the duration of the signal pulse processing time, whereas, when x-rays are absorbed in the gold the energy rapidly appears as thermal energy. At 0.3 K, we have found that the fraction of the x-ray energy appearing as a thermal signal in the germanium is always 77%, regardless of detector voltage, and pulse shape filtering. This same fraction was observed at 0.3 K with two different detector sizes and germanium doped at two different levels.

In contrast, at 0.1 K the fraction of the x-ray energy which appears as a thermal signal in the germanium varies from 79% to 85% depending on detector voltage and pulse shape filtering time scale. Possible explanations for this variation include partial electron-hole recombination, a change in the band gap or in the average energy required to create an electron hole pair. The lower operating temperature and/or higher germanium doping concentration could be responsible for some of

these effects. Another explanation is suggested by the preamplifier pulse profile at 0.1 K shown in Figure 2. The pulse does not exhibit a simple exponential decay, but appears to exhibit an extremely short duration spike followed by a much longer exponential decay. The height of the spike relative to the longer exponential decay signal varies with detector bias voltage. The post amplifier peaking time determines how much the spike contributes to the output pulse used for pulse height analysis. This spike could therefore explain the change in the ratio of pulse height due to x-rays absorbed in germanium to those absorbed in gold. This spike may originate from a decoupling of the electrons from the germanium lattice; such a decoupling model might also explain some of the nonlinear current-voltage behavior.[11] In the calculation of the detector parameters in Section 2.3, the spike was ignored, and it was assumed that the signal with the longer exponential decay arises from the thermalization of the entire detector.

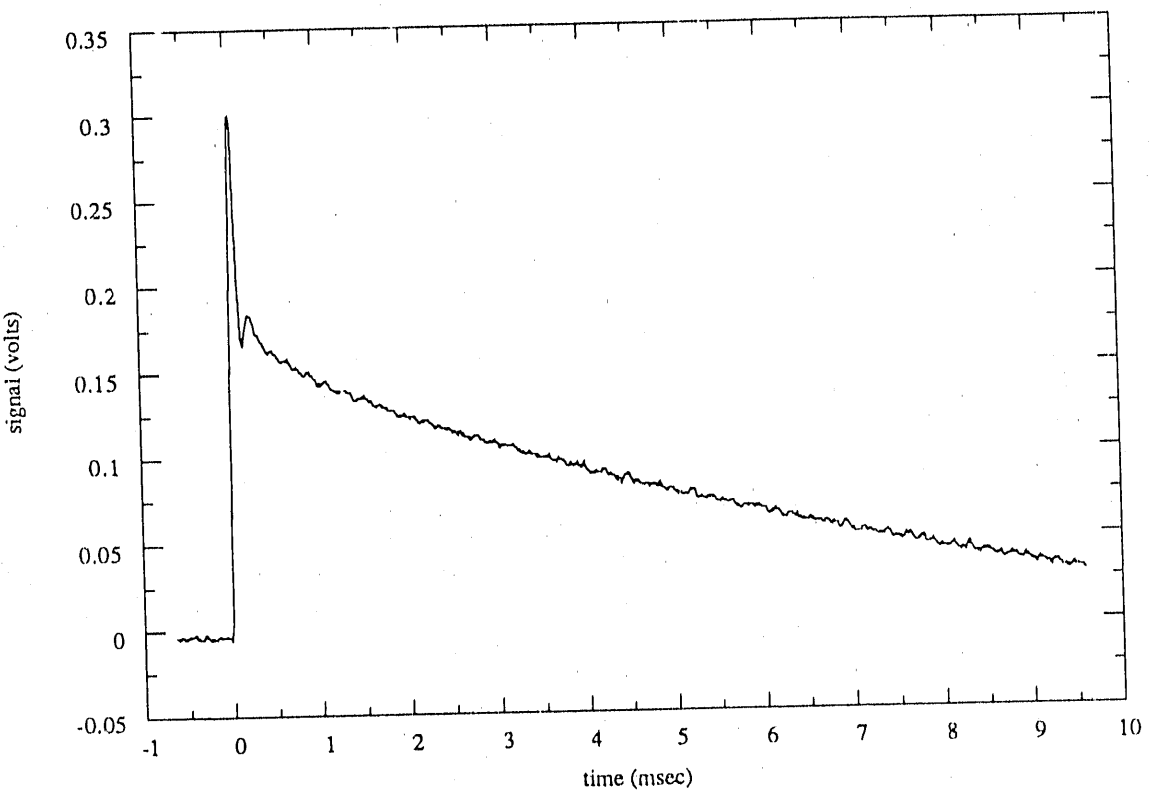

Figure 2. The response of the NTD germanium calorimeter to a 5.89 keV photon. The detector base temperature is 0.1 K.

2.5 Spectral widths

As shown by the relative width in channels produced by the electronic pulser and shown in Figure 1, the electronic noise in this detector is 12 eV. Spectral analysis of the electronic noise reveals significant excess noise at frequencies below 100 Hz, which is only present when there is a voltage on the detector. After the pulse shaping filter, the total integrated noise in the bandpass of interest is about twice what is expected. While excess low frequency noise has been observed in ion-implanted silicon[7], we are not convinced the excess noise we observe is intrinsic to the NTD germanium. Rather, it may arise in contacts or elsewhere in the circuit. Further investigation of the noise is underway.

Since gold is a metal, one expects the resolution to be limited only by electronic noise, and the convolution of the $K\alpha_1$ and $K\alpha_2$ peaks which are 11 eV apart. This does not account for the 25 eV width observed in the gold absorption peak. After deconvolving the $K\alpha_1$ and $K\alpha_2$ peaks, the spectral resolution of our system is 19 eV, still above the 12 eV electronic noise. Additional spreading may be due to x-rays which are absorbed near the surface of the gold layer where energy can be lost by electron escape. We are planning to construct a device with a thicker gold layer to see if a resolution closer to the electronic noise can be produced. The x-rays absorbed in the germanium produce a 57 eV width peak. It is not

surprising that the germanium peak is broader than the electronic noise since one expects at least 30 eV due to statistical fluctuations in the number of electron-hole pairs created.

3. X-RAY ABSORPTION IN A SUPERCONDUCTOR

While thicker gold would improve the x-ray absorption in the layer, gold has too much heat capacity to be used as an efficient x-ray absorber with a reasonable (at least 0.5 mm x 0.5 mm) area. Zero band gap materials such as HgTe have proven to be good absorbers, but they have a relatively large specific heat.[7] As an alternative, we are investigating absorbers of superconducting materials that exhibit much smaller heat capacity than gold. For our first test, an insulating SiO_2 layer 100 nm thick was sputtered on an NTD germanium sample identical to the one described in Section 2. A 1 μm thick niobium layer was then sputtered over the insulator, covering a 0.1 mm x 0.1 mm area. This sample was cooled to 0.1 K and the ^{55}Fe spectrum is shown in Figure 3.

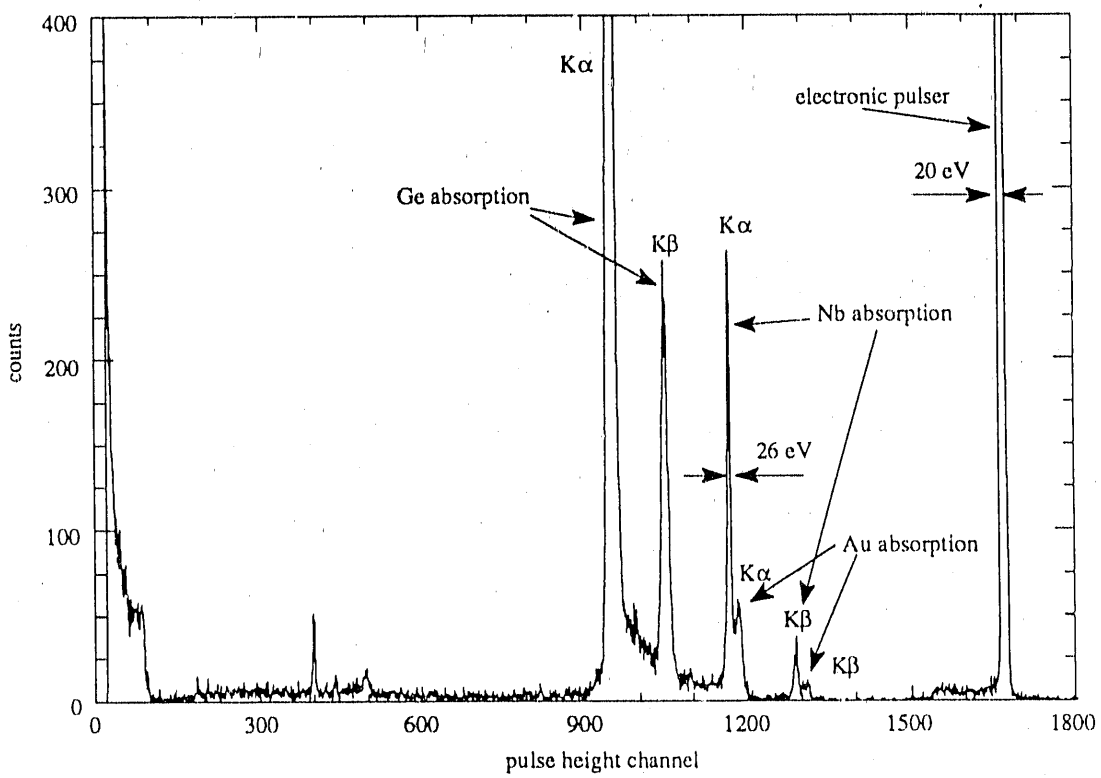

Figure 3. An x-ray spectrum obtained with a calorimeter identical to that used to obtain the spectrum in Figure 1, with the addition of a 100 μm x 100 μm x 1 μm thick niobium absorber. The 26 eV FWHM resolution indicated by the Nb absorption peak includes contributions from the Mn $K\alpha_1$ and $K\alpha_2$ lines. Accounting for these two lines within the peak shown yields an energy resolution of 20 eV.

Instead of the two pairs of lines observed by direct irradiation of the Ge calorimeters, we now observe three pairs. Furthermore, the relative position of the peaks changes relative to the gold peak when the peaking time of the pulse shaper is changed. If we assume that 100% of the x-ray energy absorbed in the gold is converted to heat, then 95% of the energy absorbed by the niobium is converted to heat within a 0.4 msec pulse shaping time. As the pulse shaping time (integration time) is increased, the niobium peaks move closer to the gold absorption peaks. The data displayed in Figure 3 were obtained with a peaking time of 0.8 msec, in this case 98% of the energy absorbed by the niobium is converted to heat. At pulse shaping times of 1.2 msec and longer, it appears that 100% of the energy absorbed in the niobium is converted to heat.

As demonstrated above, this device is a useful tool for studying how energy is partitioned in different superconducting and semiconducting materials. For example, measuring the fraction of x-ray energy which is converted to heat is important for the development of superconducting tunnel junction detectors, another type of cryogenic x-ray detector.[13,14] Superconducting tunnel junction detectors rely on converting the x-ray energy into quasiparticles rather than heat. Measurements of the fraction of the x-ray energy converted to heat on time-scales similar to the typical junction tunneling time (10 and 100 μsec) permits calculation of the number of quasiparticles the junction detectors should produce. The performance of tunnel junction detectors can then be more accurately predicted.

The width of the niobium peak in Figure 3 is 26 eV, or 20 eV after deconvolving the $K\alpha_1$ and $K\alpha_2$ peaks, similar to the width of the gold peak in Figure 1. Since the electronic noise in this device is 20 eV, the niobium absorber does not contribute to the total noise observed. It is not yet known why the electronic noise in this device is larger than the device without niobium discussed in Section 2; this is currently under investigation. It may be possible to alter the absorber material or deposition process to give faster recombination and hence 100% of the energy converted to heat within shorter time scales. We plan to study the absorption properties of niobium in more detail by depositing thicker films on a thin diamond substrate to which the NTD germanium thermistor is attached to the other side.[9] Superconductors other than niobium will also be tested.

4. CONCLUSIONS

We have demonstrated that high resolution x-ray calorimeters can be constructed by absorbing the x-rays in the gold contact of NTD germanium thermistors. An energy resolution of 19 eV FWHM at 6 keV have been observed with electronic noise of only 12 eV. The thermistor has not been completely optimized, and further improvement in energy resolution should be possible.

A sputtered layer of niobium appears to be a good x-ray absorber, and most of the x-ray energy is converted to heat within the time scales of interest. An energy resolution of 20 eV FWHM at 6 keV has been observed for x-rays absorbed in niobium. Furthermore, these calorimeters can be used to study the thermalization process in different materials. To fabricate larger area x-ray detectors with high spectral resolution, more experimentation with superconducting absorbers is necessary. The results shown here are very encouraging.

5. ACKNOWLEDGEMENTS

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