Fast Resistive Bolometer

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Prepared by
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Abstract
Resistive bolometry is an accurate, robust, spectrally broadband technique for measuring absolute x-ray fluence and flux. Bolometry is an independent technique for x-ray measurements that is based on a different set of physical properties than other diagnostics such as x-ray diodes, photoconducting detectors, and P-I-N diodes. Bolometers use the temperature-driven change in element resistivity to determine the total deposited energy. The calibration of such a device is based on fundamental material properties and its physical dimensions. We describe the use of nickel and gold bolometers to measure x-rays generated by high-power z pinches on Sandia's Saturn and Z accelerators. The Sandia bolometer design described herein has a pulse response of ~1 ns. We describe in detail the fabrication, fielding, and data analysis issues leading to highly accurate x-ray measurements. The fundamental accuracy of resistive bolometry will be discussed.
Fast Resistive Bolometry*

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I. INTRODUCTION

The broadband measurement of absolute x-ray fluence and flux with nanosecond time response from intense sources has long been a serious challenge. We describe the design, fabrication, and use of fast resistive bolometry [1,2] as an absolute measure of x-ray fluence and flux, covering the spectral range from 5 – 4000 eV. These bolometers have been used to measure the outputs from z-pinch x-ray sources on Sandia National Laboratories' Saturn and Z accelerators. [3,4]

Other x-ray diagnostics suffer from one or more problems when compared with resistive bolometry when used to determine total x-ray flux or fluence.
Calorimetry,[5] considered a very accurate, broadband measure of x-ray fluence provides no temporal information. X-ray diodes (XRDs)[6] have excellent time response but their variable spectral sensitivity makes accurate spectral unfolds difficult and XRDs require careful calibration due to the strong dependence of quantum efficiency on surface metrology. Diamond photoconducting detectors (PCDs)[7] have excellent time response, stable sensitivity, and relatively flat spectral response above 1 keV but suffer from significant sensitivity variation and non-linear response due to large surface energy deposition below 1 keV. Silicon-based detectors such as P-I-N diodes[8] can have adequate time responses and have relatively flat spectral responses but can suffer from dead layer effects and have excessive sensitivity.

II. BOLOMETERS

A bolometer resistive element consists of a thin metallic film with dimensions of order ~ 1-μm thick, ~ 1- to 2-mm wide, and ~ 2-cm long. We typically fabricate bolometer elements onto low conductivity substrates such as fused silica for ruggedness. X rays are normally incident on the bolometer film and are absorbed via the photoelectric effect. The resulting energy in the primary photoelectrons and secondary electrons ends up heating the element. The energy deposition profile is determined by the x-ray absorption profile and the range of the primary photoelectrons. The heat absorbed by the bolometer films results in a very rapid increase in the temperature of the element that is determined by the specific heat of the material. Resistive bolometers make use of the temperature dependence of the resistivity to measure this change in temperature. The thinness of the bolometer film together with the depth-averaged measurement of the element resistance gives fast detector time response. The element is immersed in a ~ 0.05-T magnetic field to suppress secondary photoelectron emission and preclude current shunting.
We commonly use nickel as the element material when designing bolometers for sub-keV operation and gold as the element material for > 1 keV operation. Nickel is used because of the nearly linear relationship of resistivity with temperature. Gold was chosen for the > keV bolometers because of the ease of fabrication and the high x-ray opacity. High-Z materials are inappropriate for sub-keV usage due to the preferential absorption of soft x rays at the surface of the detector and the resultant strong heating of the surface material. (Other materials are possible bolometers candidates.)

We have designed a bolometer driver that passes a pulsed current of 40-100 A through the element. This bias current provides the voltage change across the element when the temperature changes due to x-ray heating. This bias current is delivered to the bolometer element via a 50-Ω coaxial cable. A second 50-Ω cable is placed in parallel with the bias cable, also in series with the element, to measure the change in voltage across the element. The driving current pulse has a rise time < 0.5 μs and a pulse width of 5-10 μs. The short pulse width of the bias current is required to minimize the resistive heating of the element and subsequent damage.

The absorbed fluence can be related to the electrical characteristics of the circuit through the following relations. The total energy absorbed by the element is simply,

\[ \text{Energy} = \Delta R \left( \frac{dR}{dE} \right) \]

Here \( \Delta R \) is simply \((\Delta V / I)\) from the electrical circuit measurements, \( dE \) is \((\rho M \cdot v \cdot c_p \cdot dT)\), \( dR \) can be expressed as \((\ell / wt) \cdot d\rho\). Where \( v \) is the element volume, \( w \) is the element width and \( t \) is the element thickness, \( \rho_M \) is the element mass density \( c_p \) is the specific heat at constant pressure (J/g-K), and \( \ell \) is the element length. This gives the following relation:
\[ \Delta E = \left( \frac{\Delta V}{I} \right) \left( \frac{w}{\ell} \right) \rho M^2 \left( \frac{c_p}{\frac{dp}{dT}} \right) \]

Where \( \rho \) is the material resistivity.

Converting to fluence and replacing the change of resistivity with temperature with enthalpy we obtain the classic bolometer result:

\[ F = \left( \frac{\Delta V}{I} \right) \left( \frac{w}{\ell} \right) \frac{\rho M^2}{\frac{dp}{dT}} \]

Where \( F \) is the fluence onto the element and \( \frac{dp}{dT} \) is the measured change in the resistivity with enthalpy. In the case of nickel \( \frac{dp}{dT} \) is 9.0 \( \times 10^{-10} \) \( \Omega \)-cm-g/J and in the case of gold \( \frac{dp}{dT} \) is 6.5 \( \times 10^{-8} \) \( \Omega \)-cm-g/J.

III. ELEMENT FABRICATION

A. Mask selection and fabrication

The masks were fabricated with an EDM wire process to maintain precision tolerances of the critical dimensions. The first mask dimensions include the 2-mm wide (1-mm wide for gold bolometers) by 12.7-mm long primary strip with 5-mm by 5-mm squares at the ends to define the contact pads. A second mask is made with only the 5-mm by 5-mm square electrical contacts that will allow the 2-mm by 12.7-mm strip to be masked while depositing the copper contacts. The mask material is 304L stainless steel and is solvent cleaned followed by vacuum firing at 450 °C for eight hours prior to use. The masks were machined to fit a resistive substrate heater to maximize thermal contact with the substrate. The substrate heater design is made for Ultra High Vacuum use. It is critical that the resistive heat fixture be selected carefully because outgassing sources are fatal to the process.

B. Substrate selection and preparation

The dimensions of the element are 6.223-mm wide by 23.241-mm long by 1.016-μm thick with two electrical lead bores with 1.397-mm diameters centered in
the width dimension and spaced evenly 18.288-mm apart along the length. The current substrate material of choice is Pyrex. Its coefficient of thermal expansion is better than that of fused silica and maintains a better surface figure. We specified that the surfaces be laser quality with a scratch-dig specification of 10-5 with a surface figure of tenth wave or better. There is a direct correlation between initial surface figure and the film's nucleation, growth, and microstructure evolution which in turn has yielded the lowest baseline resistivity per unit area. Two additional substrate species have been ordered for investigation that have lower coefficients of thermal conductivity and thermal expansion. The substrates are ULETM ("ultra low expansion" made by Corning) and ZerodurTM (made by Schott). We believe that these new materials will aid the surface figure to maintain better form, as the surface figure after coating degrades to quarter wave or worse due to film stress.

The substrate surface is inspected for scratches and pits over the critical clear aperture. It is critical that any surface defects in the central region be on the submicron level. Any significant defects have huge effects on the nucleation, growth, and microstructure evolution of the film and therefore drastically effect the film performance.

The surface is then scrubbed with acetic acid and cleaned with pure ethyl alcohol. The substrates are then placed in a UV/Ozone cleaner for 24 hours. The UV light source converts ambient oxygen into ozone that attacks organic compounds on the surface. This protocol is under investigation because while the oxide layer that forms aids in passivation of the surface, it is unclear how this affects the performance of the film in the bolometer circuit.

The substrates are carefully inserted into the masks and resistive heat source. Painstaking care must be made make sure that the surface remains clean. After the elements are placed in the fixture, the components are dusted with an ultra pure carbon dioxide gas duster and loaded into the process chamber.
C. Thin Film Deposition Protocols

The main elemental materials utilized for bolometer production are nickel, gold, copper, and chromium. All materials are selected for 99.999% purity. The protocol outlined is for nickel but is applicable to gold bolometer production as well.

The process chamber is fitted with an e-beam gun with a rotating turret that allows for up to six crucibles to be loaded per run. The first run defines the bolometer element's nickel strip but also forms the foundation for the electrical contacts as well. The chromium and the nickel are loaded into the appropriate crucibles. Standard contamination avoidance is employed, as contamination in the materials is also fatal to the process.

The geometry of the system is configured with a source to substrate distance of 480 mm. This distance maximizes uniformity over the 7.5-cm diameter of the work heater in which the elements are placed. A shutter was installed to shield the substrates during ramp up of the emitter current. This serves two main purposes. The first is to provide a mechanism of immediate, constant deposition rates to ensure maximum deposition rate. The high arrival energy of the deposited atoms aids the nucleation, growth, and microstructure evolution of the film to form films that are not columnar. If the initial growth of the film begins columnar, there is no way to correct for it in process and the result will be failure. The second purpose for the shutter is to mitigate "spit" from the source. Contamination or trapped gas mass that is released will shoot particulate to the elements. This will cause defects in the film and can be a primary failure mechanism to the process. A thermocouple lead is attached to the substrate heater at the surface to monitor fixture temperature and another is attached to the chamber to measure the process chamber temperature. The resistive heater has bare leads to through a ceramic electrical feed through. An array of quartz lamp radiant heat sources is also configured for system bake out.
The thickness measurement system is an Inficon\textsuperscript{TM} crystal quartz deposition rate monitor.

The system is then evacuated with a two-stage rotary-vane mechanical pump to crossover at maximum crossover pressure to a cryopump. Diffusion pumps and turbomolecular pumps are not as desirable due to contamination issues, but can be used if appropriate liquid nitrogen traps are employed. Once the chamber reaches $5 \times 10^6$ Torr, the resistive substrate heaters are employed and monitored to 125 °C. Once the temperature reaches equilibrium, the quartz lamp radiant work heaters are employed to heat the process chamber to 125 °C as well. The objective is not only to bake out the system for molecular water removal, but also to thermalize the substrates for thermal uniformity. The system (still at 125 °C) is pumped to an ultimate pressure of approximately $5 \times 10^8$ Torr. Our process chamber achieves this in 24 to 36 hours.

The deposition process begins with the shutter positioned to shield the components. The first layer will be a chromium adhesion layer of less than 10 nm. The chromium is heated with an emitter current of 0.050 A to achieve a deposition rate of approximately 0.5 nm/s. Once the source is thermalized at equilibrium and out of danger of shooting particulates, the shutter is opened and deposition is monitored to 10 nm. The shutter is closed, the emitter current is cut, and the gun turret rotates to the nickel source. The current is ramped up to 0.180 A to yield a deposition rate of approximately 2.5 nm/s. It is important to not greatly exceed the deposition rate of 2.5 nm/s because the film degrades in performance when faster deposition rates are employed, perhaps by increased film stress and the resulting microcracks. The shutter is opened and the thickness is monitored to 1.00 μm when the shutter is closed and the emitter current is cut. Once the targeted film thickness is achieved, all heaters are shut down and the heat is allowed to completely radiate out. All heat must be allowed to completely radiate out to avoid thermally shocking the film. This will result in microcracks and in high resistivity of the film. The vent
gas used is UHP argon. The vent gas is allowed to continue to flow while the fixture is changed for the copper electrical contact pad mask. This helps to minimize adsorption and/or reaction of undesirable materials with the nickel. This will aid in mitigation of contact resistance at the nickel/copper film-to-film interface. Time should be minimized to maximize pumpdown speed and to minimize contamination. Great care is taken to protect the nickel strip. Once the components are fixtured and reloaded, the chamber is quickly evacuated again. Pump down and heating procedures are identical to the first run. The copper crucible is indexed and the emitter current is ramped to 0.320 A with the shutter positioned again to shield the components to achieve a deposition rate of 2.5 nm/s. The targeted thickness is monitored to a nominal 1.75 μm, the shutter is closed, the current is cut, and all heaters are shut down. Again, all heat must be allowed to completely radiate out before venting.

D. Film measurement and characterization

The film thicknesses are measured via a Dektak 3st profilometer. Five points along the length of both sides of the nickel strip are measured. The average deviation of these points is usually within 2.5 to 5.0 nm. The copper pads are measured from the nickel strip. The baseline room temperature resistance measurement is made utilizing a Hewlett Packard 4275A multi-frequency LCR meter. Measurements of the element tabulated versus calculated theoretical resistance have been characterized to within 1% deviation. Current efforts are underway to characterize this further at the Sandia Primary Standards Lab, and initial tests show the deviation to track the intrinsic resistance curves of nickel in a linear fashion. The goals of the effort are precision calibration factors, process temperature versus resistance, QC instruments, and ultimately, an in-situ accelerator element viability/calibration instrument that can verify the performance of the element on a shot to shot basis.
IV. CALIBRATION AND ERROR ANALYSIS

Ideally, one could rely on the fundamental material properties of the
bolometer element to obtain accurate measurements. Realistically, care must be
taken to characterize the element. We use microscopy and profilometry to
characterize the physical dimensions of the bolometer elements. The resistivity of
the element is measured in a controlled vacuum oven and compared against the
intrinsic properties found in the 77th Edition of the CRC Handbook. The tabular
values for nickel are \( \rho(25 \, ^\circ\text{C}) = 7.12 \, \mu\Omega\cdot\text{cm} \) with a temperature dependence of
0.006416 \( ^\circ\text{C}^{-1} \). Similarly the tabular values for gold are \( \rho(25 \, ^\circ\text{C}) = 2.255 \, \mu\Omega\cdot\text{cm} \)
with a temperature dependence of 0.003545 \( ^\circ\text{C}^{-1} \). These values are known to
\( \sim 2.2\% \).
Fig. 1 shows a picture of a bolometer element. The size reference for all physical measurements is a NIST-traceable standard. For this element the width is 2±0.15 mm and the length is 12.7±0.1 mm. The thickness of the nickel film is 1.00±0.05 μm.

As mentioned in the fabrication section the most difficult material property to obtain is the intrinsic elemental resistivity. Fabrication improvements have resulted in thin films that have the resistivity of intrinsic nickel (and gold). The resistivity of nickel (and gold) elements have been measured as a function of temperature in a vacuum oven. Figure 2 shows the resistance of a nickel bolometer element as a function of temperature. Plotted against the data is the tabular resistivities obtained from the 77th Edition of the CRC Handbook. The measured values are ~ 3.5% larger than the tabular values. All electrical and temperature measurements are based on NIST-traceable standards.

Sources of systematic measurement and analysis errors exist. These include heat loss to the insulating substrate, energy loss via primary photoelectrons, heat flow along the element, and x-ray transmission through the element. (See Fig. 3.)

Measurement errors are real concerns for any detector. With bolometry it is necessary to simultaneously measure two quantities, the current passing through the element and the voltage developed across the element, in order to obtain the element resistance. The current must be measured with a carefully cross-calibrated device such as a Pearson Probe (a self-integrating Rogowski coil) taking into account such things as the time constant and bandwidth of the detector. Both the voltage and current must be measured using calibrated, high-bandwidth (> 500 MHz) data acquisition with sufficient resolution to maintain good signal-to-noise or bit-noise levels. Typically, we find that the accuracy of a signal measurement using commercial digitizers such as a Tektronix TDS640 is ~ 2-5% in our environment. The sum of calibration and fielding errors gives us confidence at the 10% level today.
V. FIELDING ISSUES

The bolometers are fielded on 20-m long, vacuum lines-of-sight with auxiliary vacuum pumps near the detector. Alignment is done with optical telescopes. The detectors are protected from debris with pneumatically-driven fast closing valves. The line-of-sight pipes are carefully apertured to minimize internal x-ray reflections off the walls of the pipes. The detectors are cabled with low-loss heliax (Andrews LDF4-50A) with an additional ground shield of flexible Breeze Tubing™.

The bolometer pulsers are placed in a Lindgren double-wall shielded enclosure together with the digitizers. Each bolometer requires two cables: a current drive cable and a signal return cable. The presence of two cables acting as a ground/pickup loop at each detector increases the problem with electrical noise. Fig. 4 shows a current and voltage data set for a bolometer from Z shot 140. The figure shows the signal to noise and the overall data quality.

VI. CONCLUSIONS

We routinely use bolometers as absolute x-ray fluence and flux diagnostics for z-pinch experiments on the Saturn and Z accelerators. These detectors provide a valuable reference against which other x-ray diagnostics are compared. Bolometers provide a bounding measurement on the total x-ray fluence and flux emitted by any x-ray source. Careful fabrication, characterization, testing provides an x-ray detector with an absolute accuracy of ±10%.
ACKNOWLEDGEMENTS

We wish to gratefully acknowledge the support of the Sandia Primary Standards Laboratory for their detailed characterization and measurement of the bolometer elements. The bolometer pulsers were designed by Team Specialty Products of Albuquerque, NM.

*Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

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REFERENCES


5. D. L. Fehl, et al., this proceeding.


Figure Captions

Fig. 1 A photograph of a nickel bolometer element is shown. Also shown are the copper current contact pads on either end.

Fig. 2 The measured resistance (open circles) of a nickel bolometer element is plotted as a function of temperature. The theoretical value (solid line) for the resistance is plotted as a reference. The measured values of resistivity are ~ 3.5% above the tabulated values.

Fig. 3 The fractional absorption of x rays in a 1-μm thick nickel bolometer element is plotted as a function of photon energy. The spectral response of the element is nearly flat up to 1.5 keV.

Fig. 4 Data from Z shot 140 is plotted vs. time. The current waveform (solid line) shows the nearly constant drive for the duration of the measurement. The voltage (dashed line) clearly shows the effect of heating on the element resistance.
Fig. 1

Fig. 2
Fig. 3

Fig. 4
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