Thermal Conductivity of Indium Bonded Silicon

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Abstract

The Einstein Telescope will have a xylophone configuration of interferometers. One will be suited for high frequency detection, the other for low frequencies. The low frequency interferometer will be cryogenically cooled, and will use silicon for its test mass. One reason for the use of silicon is its high thermal conductivity at low temperatures. The suspension wires will also be composed of silicon, and the bond between the wires and the test mass must also have a comparable thermal conductivity. One feasible candidate is indium bonding. Our results show that it has negligible effects on the thermal conductivity of silicon, and an indium bonded silicon can be viewed as quasi-monolithic.

1 Introduction

The sensitivity of gravitational wave detectors is limited by many sources of noise, including seismic noise, quantum noise, shot noise, and thermal noise. The Einstein Telescope, a third generation GW detector, is designed to be a whole power of magnitude more sensitive than Advanced LIGO and Advanced VIRGO . The Einstein Telescope will be composed of two types of interferometers (a xylophone configuration), each suited for a different frequency regime [See Figure 1]. One will use a 3 MW laser cavity to lower quantum noise and will be used for high frequency detection. The other interferometer will be cryogenically cooled to lower thermal noise and will be used for low frequency detection. [1]

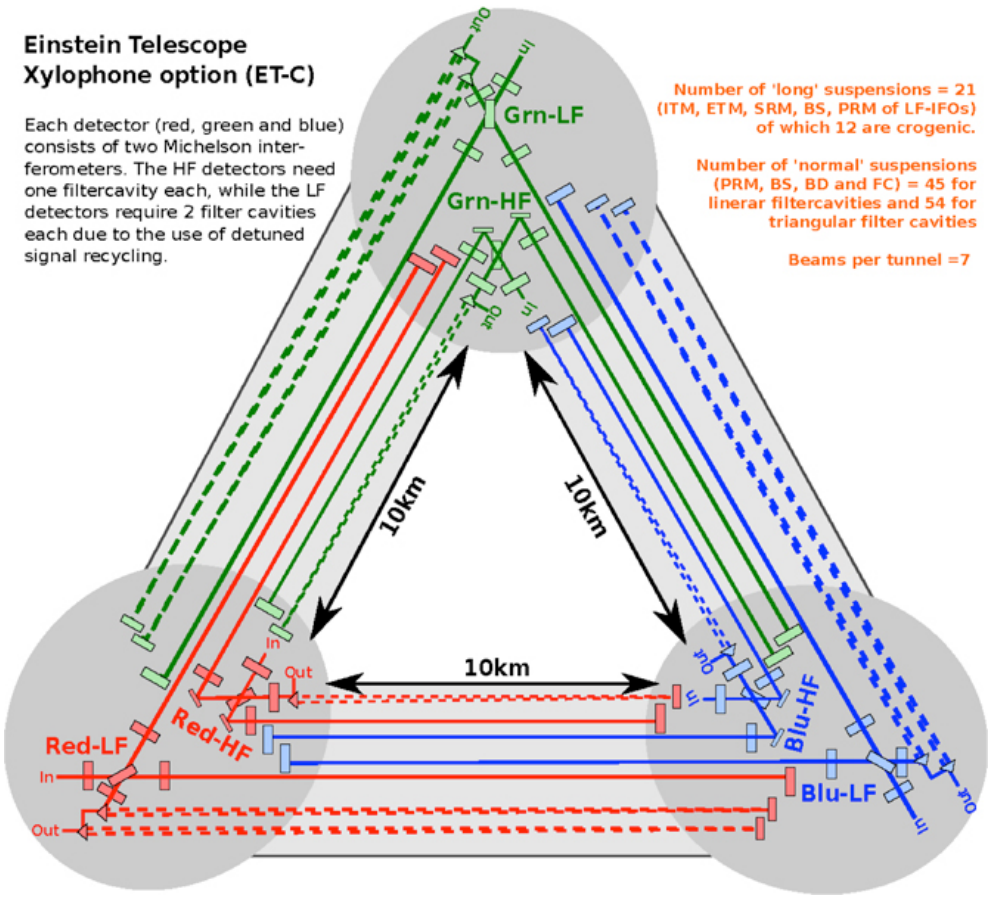

Figure 1 Diagram of the Einstein Telescope. The blue, green, and red lines each represent a single detector. Each detector is made of two interferometers.

Although cryogenically cooling the test mass is a good solution to lowering thermal noise, it brings with it many problems and material requirements for the mirror. Viable materials must have, at low temperatures, very high thermal conductivity, low thermal expansion rate, good optical properties, and low mechanical loss. Silicon meets all of these requirements and will be used for mirror and suspension cables in the Einstein Telescope's low frequency interferometer. Furthermore, mirror suspensions consist of multiple parts that must be welded together. These welds must have little impact on the necessary properties of the mirror. [1][2]

Some bonding options are already being implemented in the third generation GW detector, KAGRA. Its test mass and suspension fibers are made of sapphire, and it uses a combination of indium and hydroxide-catalysis bonding. Although research has shown that hydroxide-catalysis meets all of the needed requirement, the bond is too permanent to weld together the mirror and suspension fibers. If a fiber were to break it could not be

easily repaired and may result in a total replacement of the mirror. On the other hand, an indium bond is easy to undo. The bond is made by melting a thin strip of indium (melting point is 156.6 °C) while it is simultaneously squeezed between the two surfaces. The bond is completed by letting the indium cool back to a solid. If a suspension fiber were to break, the bond could be undone by applying heat and melting the indium. Research for KAGRA has shown that the thermal conductivity of an indium bond between two sapphire substrates is sufficient; however, the thermal conductivity of indium bonding between two silicon substrates has yet to be tested. [2]

2 Methods

2.1 Experimental Setup

Measuring thermal conductivity is straightforward, and can be solved for using the equation

$$\kappa = PL/(A\Delta T) \quad (1)$$

Where ΔT is the temperature gradient (T1 - T2), L is the distance between T1 and T2, A is the area of a fiber, P is the power being supplied to the sample, and κ is the thermal conductivity.

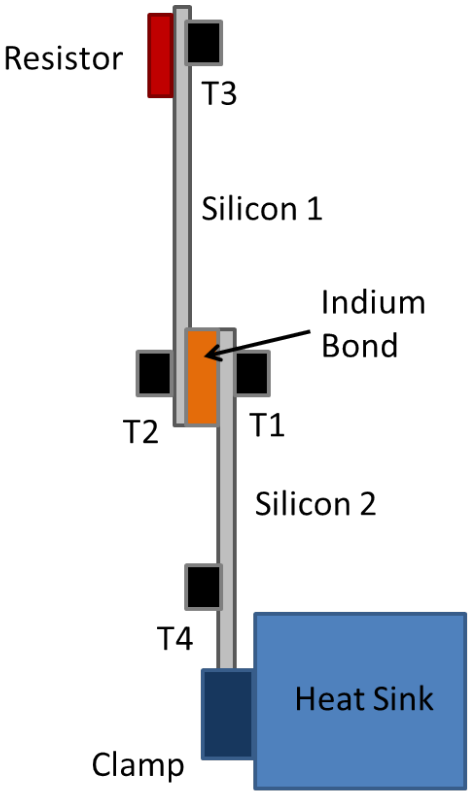

Figure 2 Experimental setup for thermal conductivity measurement of indium bonded sample

The indium bonded sample was attached at one end to a heat sink. The opposite end was attached to a 30 W, 1 kΩ resistor that provided power to the sample. The resistor was attached to an ISO-TECH IPS2302A DC power supply that varied 0 to 60 volts. The voltage was measured by an Agilent 3458A multimeter, and the current was measured with a Hewlet-Packard 3468A multimeter. A copper strip was placed between the thermometer and the sample to provide good thermal contact.

Power was calculated using

$$P=IV \quad (2)$$

Where I is the current and V is the voltage

Four thermometers were attached to the sample in order to measure ΔT. One was placed near the resistor, one near the heat sink, and two on either side of the indium bond. All thermometers and the resistor were attached to the sample with 1" and ¾" binder clips. Two additional thermometers were attached to different parts of the clamp [see Section 2.3] Our setup used two different models of LakeShore thermometers: model DT-670A-CU and model DT-470-CU-12. Both models work identically. The only reason for using two different models was because that was all we had in stock. The measurements were displayed on a LakeShore 218 Temperature Monitor, which sent measurement readouts to a program in LabView that recorded the data to a text file. The time between LabView recording data was adjustable, varying from large time scales to fractions of a second. For the thermal conductivity measurement, we set the time increment to one minute.

2.2 Preparing the indium Bonded Silicon Sample

The silicon substrates used for bonding were approximately 0.5 mm thin, 10 mm wide, and had lengths that varied between the range of 50 to 120 mm. The ends of the silicon pieces were cut with an angle. As a result, the area of the bond was later calculated as

$$W(B1 + B2)/2 = A \quad (3)$$

Where W is the width of the bond, and B1 and B2 are the lengths of the silicon pieces overlap on both sides.

The indium foil had a purity of 99.999% and was 0,1 mm wide. Although thin, the foil was still too thick to mimic the bond thickness (5 micrometers) that would be used for the ET. The indium sheet needed to squeezed thinner during the bonding process.

The materials used for bonding were as follows: three pieces of silicon (two long strips for bonding, and a third small strip for support); a large, smooth, slab of aluminum to provide a flat, stable, and portable surface for the bonding process; two, one inch wide binder clips, used to apply a squeezing pressure to the bond while being heated; four, 20

mm x 60 mm pieces of aluminum that are 3 mm thin: two to sandwich the silicon and indium, and two for support; an oven capable of reaching temperatures of 250 °C; and a strip of indium. Except for the indium foil, all surfaces were cleaned with isopropyl alcohol prior to bonding.

The bonding process took place on a Sterilflow table with the fan speed set to 0.4 m/s to mitigate dust interference. Three of the aluminum pieces were placed length-wise on top of the aluminum slab. A long silicon strip for bonding was placed on two of the aluminum pieces, and a small strip of silicon was laid on the third, unused strip of aluminum.

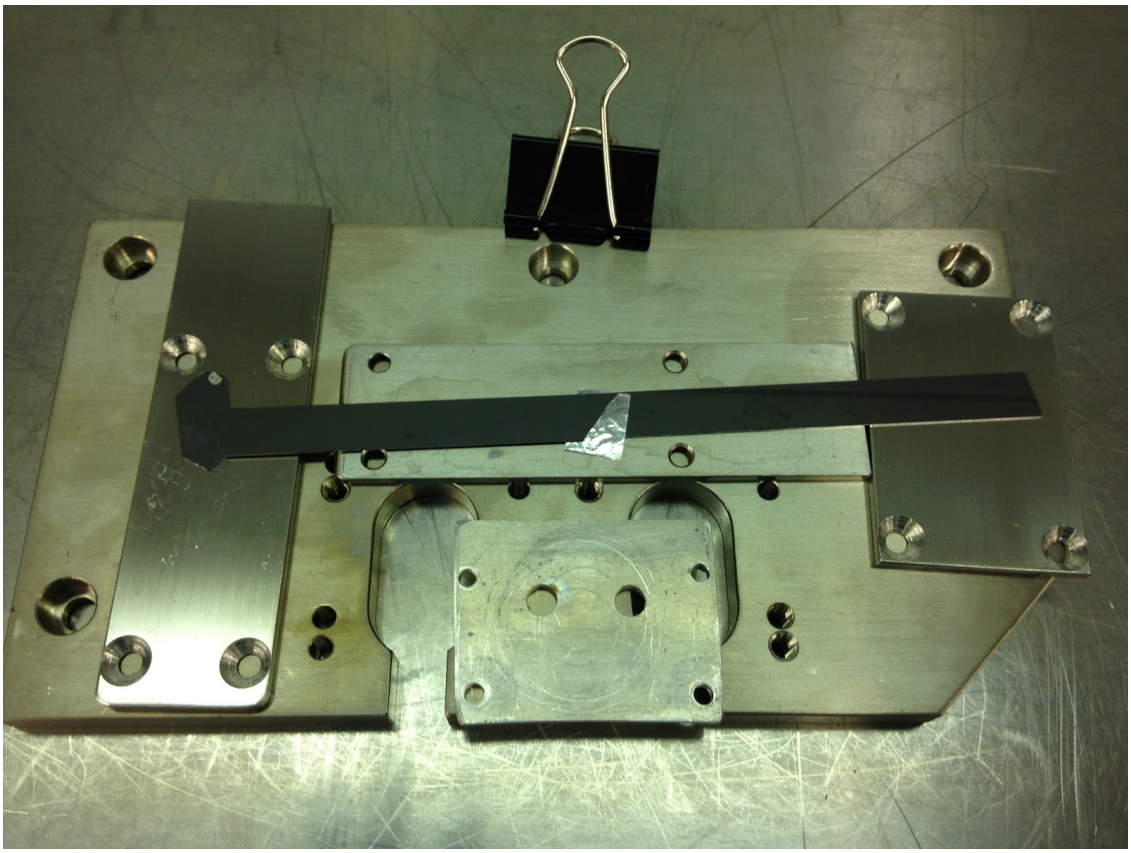

Figure 3 Preparation for indium bonding

Next, we cut and set a small indium sheet down on the end of the first piece of silicon resting between the two aluminum strips. Light pressure was applied to the indium molding it to the shape of the silicon beneath it, making alignment of the second silicon piece easier. The second piece of aluminum was aligned with one end

resting on the indium foil and the other resting on the small silicon strip [See Figure 3]. We placed the fourth strip of aluminum on top of the bonding area and aligned it with the bottom aluminum piece From there, we used manual pressure with our fingers to hold the sandwich together while we slid it to the edge of the aluminum slab. When a sufficient

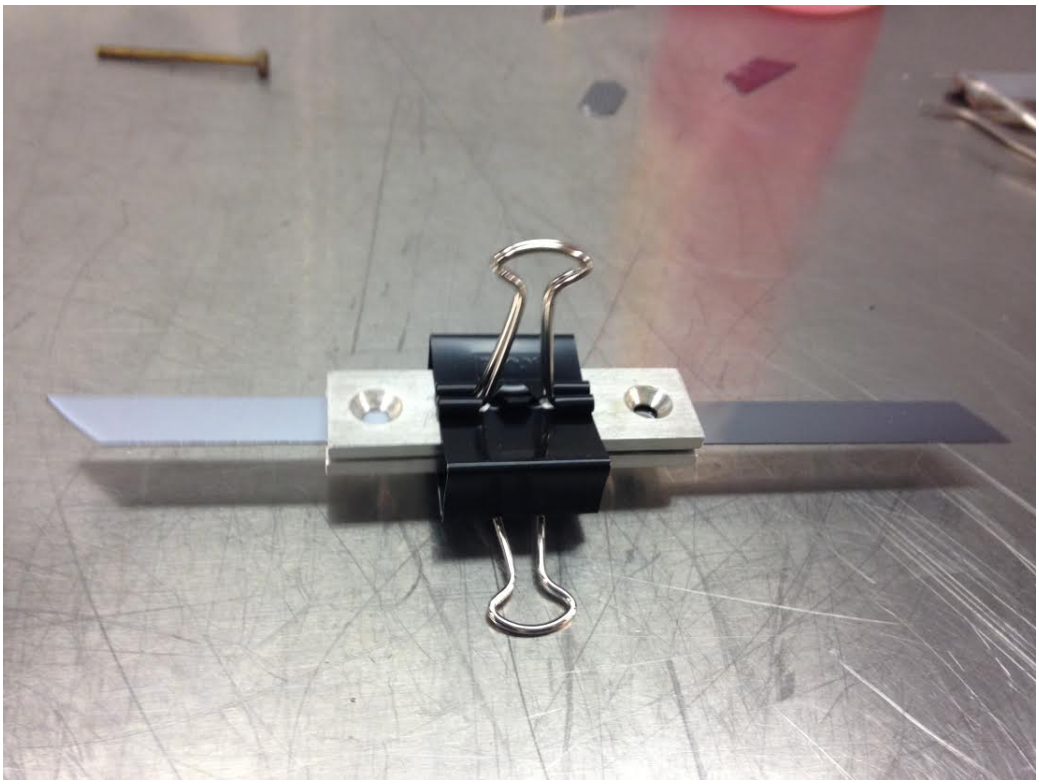

Figure 4 Clamped sample ready for heating

area was hanging off the edge we clamped the sandwich with one of the binder clamps, and then carefully clamped the other side with the second binder clip [See Figure 4]. The clips kept the sample alignment in place and provided pressure to squeeze the foil thinner during the bonding process.

The clipped sample was placed in the center of the aluminum slab, which was then slid into a preheated oven (250 °C). We left the sample in there for two hours, after which we removed it via the slab. In order to keep the bonded sample more isolated from room temperature and less prone to cracking, we covered the sample with a box made of aluminum after removing it from the slab to cool [See Figure 5]. When room temperature was reached, the clips and aluminum strips were removed, leaving only the indium bonded sample.

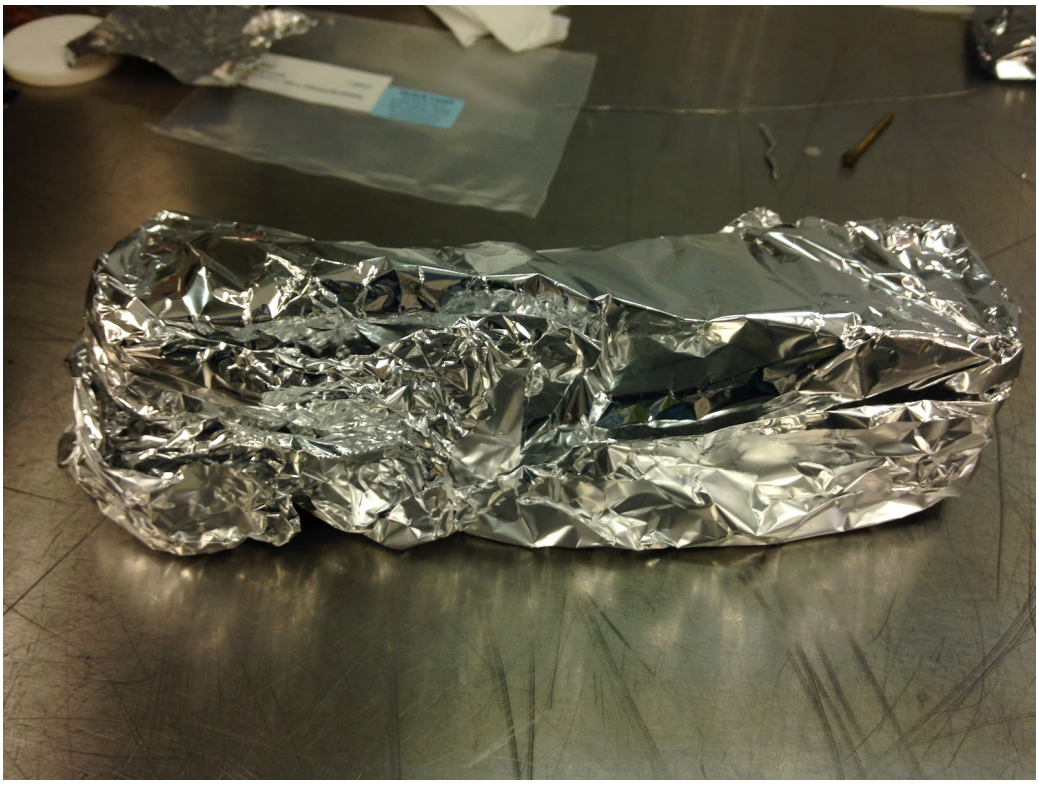

Figure 5 Foil cover used to protect sample during cooling

2.3 Clamping the Silicon Sample

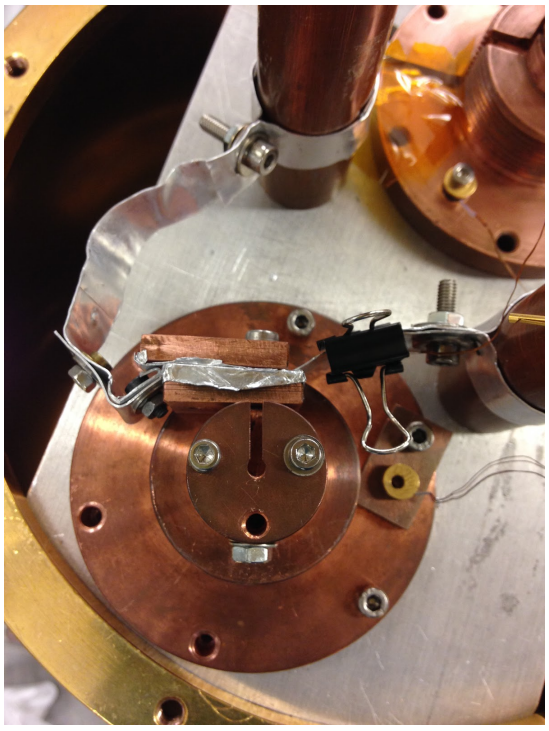

Figure 6 Clamp used to secure sample to heat sink

The sample was held in place by a clamp attached to a copper holder. The clamp consisted of two 30 mm x 20 mm x 10 mm pieces of copper. Each piece had one side covered with 99.9999% pure aluminum foil. Sandwiched in between the aluminum coated sides was 20 mm wide strip of 99.9995% pure aluminum. The two ends of this strip were wrapped around and fastened to the heat conducting tubes attached to the above level. The aluminum strip was placed between the two coated copper pieces and all three were screwed into the copper holder. The screw

holes are off to one side leaving the other side able to act as a clamp. To secure a silicon sample, the clamp screw was slightly loosened and one end of the silicon tip would be placed between the aluminum strip and a copper piece. After in place, the clamp was tightened—carefully and not too much to avoid shattering the silicon—leaving the sample securely fastened to the heat sink. Two thermometers were attached to the clamp, one was screwed into the base of the copper holder and the other was clipped to aluminum strip [see figure 6].

Once fastened, it becomes very easy to apply torque to the sample since one end is fixed. To minimize interactions where torque was easily applied, we attached the resistor and all thermometers prior to clamping.

2.4 Measurement Environment

The cooling system consisted of a CRYOMECH CP2800 Series Helium Compressor, which has two stages during its cooling process. The cryo chamber consisted of three separate levels. The top level was attached to the first stage of cryocooling (reaching a temperature of about 128 °K). Beneath it, the second level was attached to the second

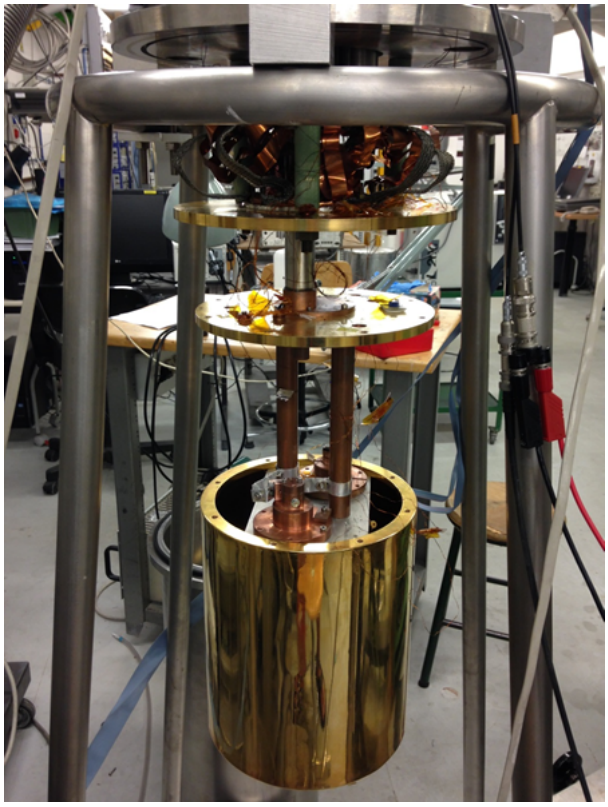

Figure 8 Cryotube with unattached first radiation shield hanging from bottom tier

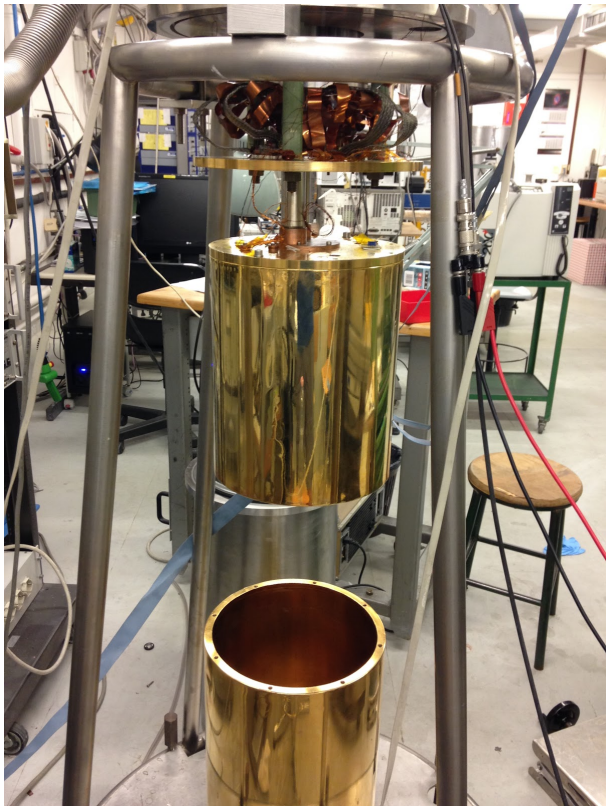

Figure 7 Cryotube with first radiation shield attached and the second shield resting beneath

stage of the cryocooling (cooling to about 5 °K). At the bottom, the third a level was suspended beneath the second level via heat conducing tubes [See Figure 7]. To help mitigate the loss of heat due to radiation, two gold-plated, steel tubes were used to enclose the sample and levels. The first, smaller tube was attached to the underside of the second level, covering the third level. A second, larger tube was attached beneath the first level, encasing the smaller tube and second level [See Figure 8]

After the thermal radiation shields were attached, the whole system was enclosed by a vacuum cover. To attain a high vacuum a standard pump was first used to reach a pressure 10^{-2} milliBar, followed by a turbo molecular pump that created a vacuum of 10^{-3} milliBar prior to cooling. The pressure dropped down to 10^{-4} milliBar after cooling.

2.5 Measurement Procedure

It took less than twelve hours for the system to reach the desired temperature of 5 °K. When sufficiently cooled, a set voltage was fed to the resistor. After waiting twenty minutes for equilibrium to be reached, the voltage, current, and temperatures were manually recorded. The voltage was then increased by three volts and the process was repeated. The same method was used for the monolithic measurement.

2.6 Uncertainties

The relative uncertainty of κ was solved for using

$$\Delta\kappa/\kappa = \sqrt{[\Delta V/V + \Delta I/I + \Delta A/A + \Delta L/L + \Delta(\Delta T)/(\Delta T)]} \quad (4)$$

Voltage was recorded to the fourth decimal place (the last digit that kept a constant value), but measured values were rounded up to three digits and given an uncertainty of ±0.0005 V. The current readout was only accurate to the fifth decimal. The value read was recorded, unless the fifth digit continuously flip-flopped by 1, in which case the average of the two were recorded. Either way, an error of ±0.000005 A was assigned

An electric caliper (Digitrix II) was used to measure both the thickness and width of the indium bonded area, the monolithic piece of silicon, and the first and second bonded silicon pieces. A manual caliper was used to measure all the lengths between thermometers, and the length of base 1 and base 2 of the indium bonded area. All lengths, thicknesses and widths were each measured 10 times. The average was used for the final value and the total error included the standard deviation and the uncertainty in precision.

There is an increased uncertainty of ΔT due to insufficient time between measurements. For the indium bonded sample, the 20 minute measurement time became less and less sufficient as power increased, due to an increasing separation between measured and final possible temperature. At 12 to 15 volt intervals, an extra 10 minutes was given to reach equilibrium to see how drastic the difference was. The temperature of the thermometers could vary by more than 5% after 10 minutes, however, the value of ΔT was not drastically effected by this. Measurements made after waiting even more time still had a ΔT value within 1% of the original. In all cases, extra wait time only increased ΔT. A one percent error was assigned to all ΔT values and was included only in the negative error bars since an increase in ΔT decreases the value of κ.

Another uncertainty in ΔT came from the thermometers. Both models have an known uncertainty of ±12 mK between 1.4 K and 10 K, an uncertainty of ±22 mK at 77 K, and an uncertainty of ±32 mK at 300 K. These uncertainties did not always seem to account for observed variations with more than four standard deviations between thermometers when attached to the same heat sink. In an attempt to explore this phenomenon, a thermometer calibration test was done. Two thermometers and the resistor were attached to the same heat sink. A voltage was applied to the resistor and the measurement of the temperatures was made after 12 minutes. This was repeated multiple times. By assuming the temperature should be the same for both thermometers, a calibration equation can be made by plotting the temperature of the two thermometers versus each other [See Figure 9]. In theory, multiplying the measured values of the thermometer on the x-axis time the slope should create negligible error in the thermometers. A similar thermometer calibration was attempted for all six thermometers. Due to time constraints, after the heat sink reached 5 °K the resistor was turned all the way up to 60 V while the LabView program recorded data ten times a second. This running measurement method was not accurate, which is known because same data recording methods were used while heat sink was cooling down and the calibration slopes using cooling down measurements are not consistent with the warming up slopes.

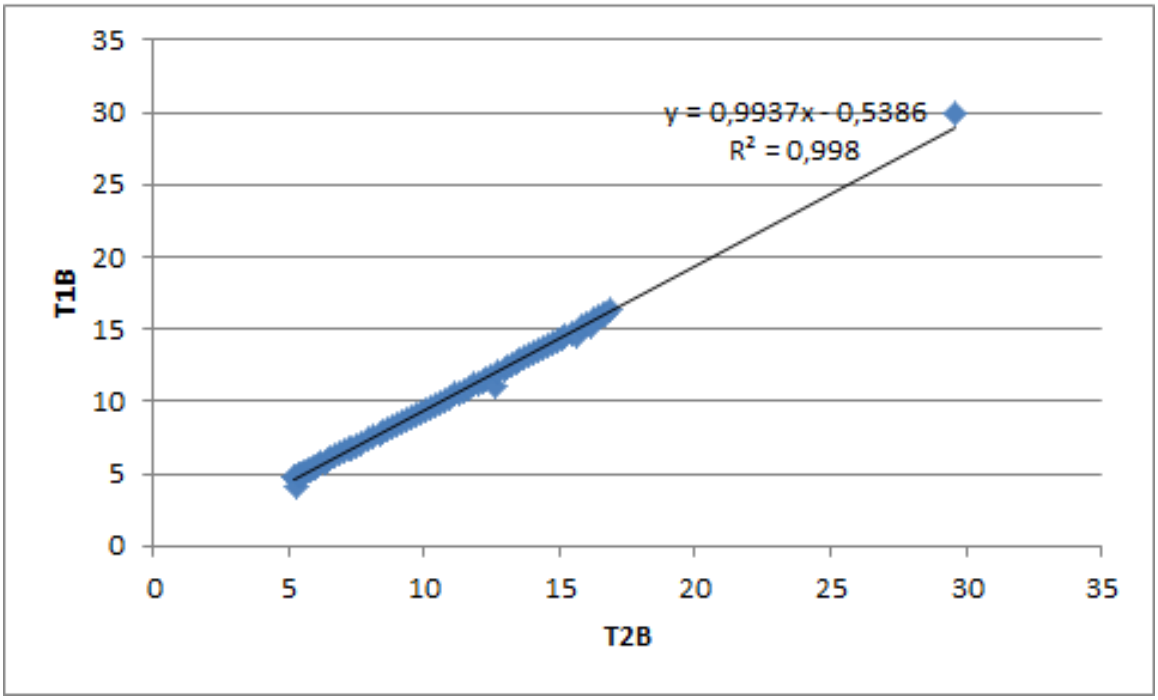

Figure 9 Plot of two thermometers for calibration.

From Figure 9 it is clear that there is a linear relation between T1B and T2B as temperature increases; however, there is insufficient data to prove that this is the result of insufficient calibration or the slight temperature gradient between the two. In short, the original error of ±12 mK was used for temperatures below 10 °K, an error of ±22 mK was used for temperatures between 10+ and 77 Kelvin, and an error of ±32 mK was used for all temperatures above 77 K.

3 Results

The sample used had a bond area of 63 ± 3 mm^2 and a bond thickness of 0.010 ± 0.005 mm. The thickness was at most a factor of three larger than the ideal thickness. The area and length values used to calculate the thermal conductivity of the top piece of silicon attached to the resistor (Silicon 1), the bottom piece of silicon attached to the heat sink (Silicon 2), and the total Bonded sample can be seen in Table 1 below.

	Silicon 1	Silicon 2	Bonded
Area (mm^2)	5.31 ± 0.02	5.30 ± 0.04	5.30 ± 0.04
Length (mm)	73.0 ± 0.7	34.7 ± 0.7	105.3 ± 0.8

Due to placement of the four thermometers on the bonded sample we were able to measure the thermal conductivity of different sections of the sample. As a result, we calculated the thermal conductivity of the monolithic silicon piece above the bond attached to the resistor (Silicon 1), the bottom silicon piece attached to the heat sink (Silicon 2), and the whole bonded silicon sample (Bonded**).** The temperature value on the X-axis is the average of the two temperatures used to calculate ΔT.

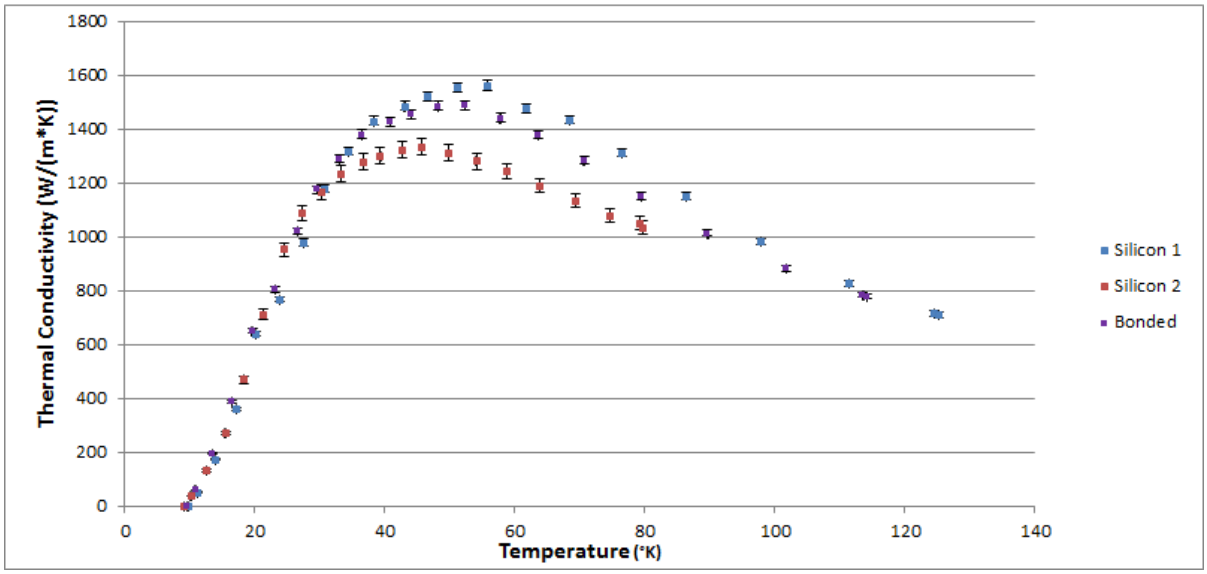

Figure 10 Thermal Conductivity of Silicon 1, Silicon 2, and the Bonded sample versus the average temperature of the thermometers used to calculate ΔT

The thermal conductivity values of Silicon 1, a monolithic measurement, are 15% larger than monolithic measurements made by other research teams after about 30 K. The values of monolithic Silicon 2 are almost identical to the same previous research. This 15% gap occurring after 30 °K between Silicon 1 and Silicon 2 and between Silicon 1 and Bonded is due to different responses to power by the thermometers [see Figure 11].

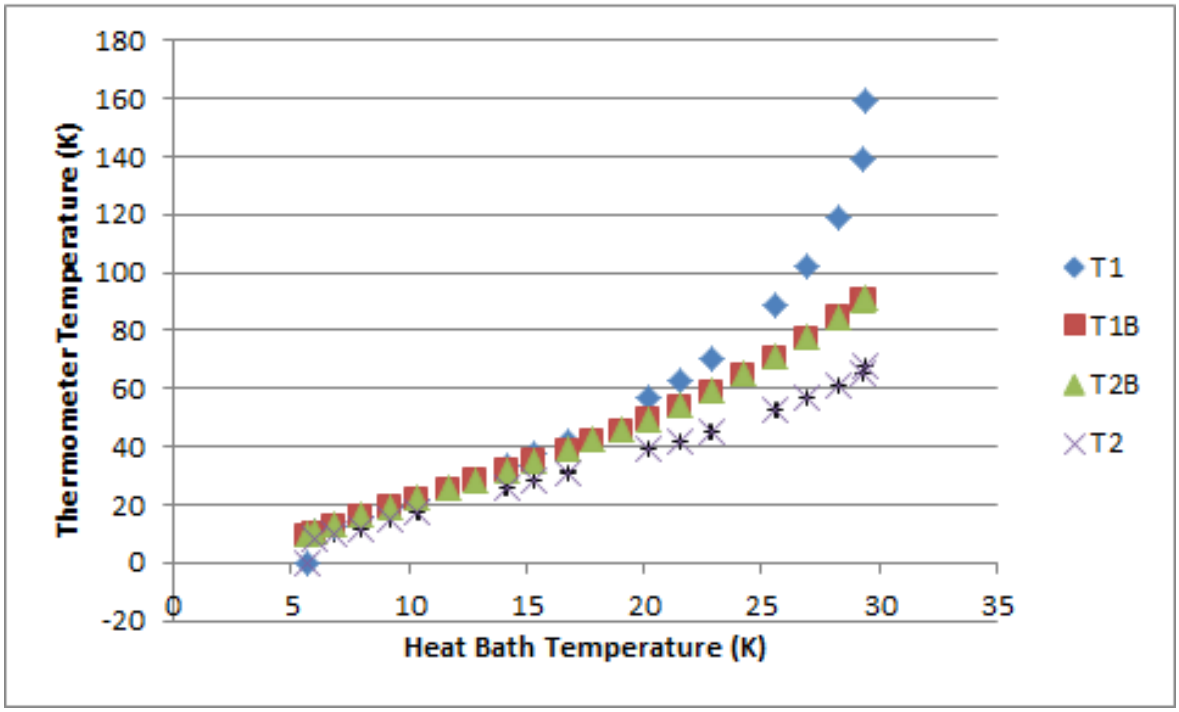

Figure 11 Temperature of all thermometers versus the heat bath temperature

When compared to the temperature of the heat bath, the temperature of T1 has exponential growth. This larger growth rate results in a different derivative of ΔT calculated using T1.

4 Discussion

The thermal conductivity measurement of Silicon 2 are in agreement with measurements made by a previous research groups[2]. Despite the exponential growth issue, the measurements of Silicon 1 and Bonded, which both use T1, show that the indium bond has very little effect on the thermal conductivity of silicon. At the temperature where the ET will be operating, there is no effect from Indium bonding. These results show that the thermal conductivity of indium bonded silicon is sufficient e for the Einstein Telescope.

Future work should try to see if our results can be duplicated, as well as making the same measurements with varied areas of the indium bonded surfaces. It should be noted that the temperatures of the two thermometers nearest the indium bond went against common sense. The thermometer attached to Silicon 1 side of the bond read

lower temperatures than the thermometer attached to the Silicon 2 side of the bond. There is no explanation this. Future research using the same setup and material should see if they witness this same phenomenon.

ACKNOWLEDGEMENTS

I would like to thank the University of Flore the Guido Muller, Bernard Whiting for organizing this amazing research opportunity, and the National Science Foundation for funding. I am also very grateful for the essential guidance of Dr. Majorana and Dr. Puppo for my research.

References

[1] ET Science Team, ET Design Study, 2011

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