

# Towards Supersensitive Bolometers and Electron Coolers Based on Carbon Nanotubes

MODELING AND FABRICATION OF MICRO/NANO DEVICES

Terese Kraft  
terese@student.chalmers.se

Johan Ek Weis  
ekweis@student.chalmers.se

Mattias Wessling  
wessmatt@student.chalmers.se

Mikhail Tarasov

Supervisors  
Johannes Svensson

Leonid Kuzmin

Chalmers University of Technology  
Göteborg, 27th May 2006



## Abstract

A bolometer is a device for measuring electromagnetic radiation. In this report we present the research we have done investigating the production and behavior when the absorber of the bolometer is made of bundles of carbon nanotubes. We also varied the composition of the electrodes and investigated the effect of varying their widths. Our results showed that the contact resistance can be decreased by using bundles of single walled carbon nanotubes instead of individual nanotubes when replacing a normal metal as absorber in a cold electron bolometer. Our work has also confirmed that the contact resistance is lower when using electrodes composed of titanium/palladium/aluminum instead of pure aluminum. We could also conclude that the width of the electrodes does not affect the contact resistance.



## Contents

<b>1</b>	<b>Introduction</b>	<b>1</b>
<b>2</b>	<b>Background</b>	<b>1</b>
2.1	Bolometer . . . . .	1
<b>3</b>	<b>Production</b>	<b>5</b>
<b>4</b>	<b>Experiments</b>	<b>9</b>
4.1	Setup . . . . .	9
4.2	IV-Measuerments . . . . .	10
<b>5</b>	<b>Results</b>	<b>10</b>
5.1	Chip4 - Aluminum electrodes . . . . .	10
5.2	Chip3 - Titanium/Palladium/Aluminum electrodes . . . . .	12
<b>6</b>	<b>Analysis</b>	<b>13</b>
<b>7</b>	<b>Discussion</b>	<b>15</b>
<b>8</b>	<b>Conclusion</b>	<b>16</b>
	<b>Appendix</b>	<b>18</b>



# 1 Introduction

A bolometer is a device for measuring electromagnetic radiation which was invented in 1880 by Samuel Langley. The word comes from the Greek words *bole* and *metreo* which means beam and measuring respectively.

A bolometer is a device for measuring electromagnetic radiation which can be used for detection of for example weapons and explosives but the most interesting application may be the possibility to illuminate the dark universe. With this device it could be possible to measure the cosmic background radiation which could give information about the temperature distribution in an early universe. Which then may give a further understanding of the 96 % of our universe that consists of dark matter and dark energy and also for example about the expansion speed of the universe.

There is quite a lot of research needed to achieve the sufficient sensitivity of this device but it is also a lot of research going on in this area and our investigation presented in this report is a small step that we hope contributes to this development. Previous attempts have been made using a single nanotube as absorber but the contact resistance turned out to be too high. In this report we present the research we have done investigating the production and behavior when the absorber of the bolometer instead is made of bundles of nanotubes. We also varied the composition of the electrodes and investigated the effect of varying their widths.

## 2 Background

### 2.1 Bolometer

A bolometer is essentially made by an absorber which is connected to a heat sink and something that can measure a temperature difference. The basic principle is that the absorber will absorb incoming radiation which will raise the temperature. This increase in temperature will change the resistance of the absorber, which can be measured by running a current through it and measure the voltage over it (or vice versa).

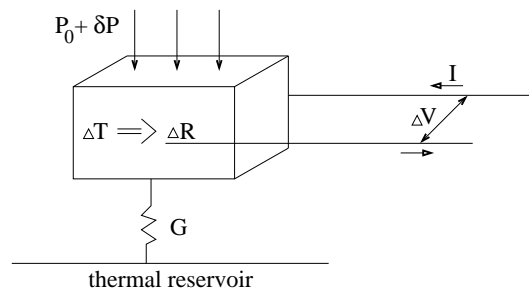


Figure 1: *Schematic picture of a basic bolometer, which is current biased.*

There are a few different ways to realize a bolometer, but we will focus on a cold electron bolometer, CEB. The absorber is a normal metal, in our case a bunch of carbon nanotubes, connected to two superconductors with an insulating layer in between, that is SINIS junctions. The readout can also be done in a few different ways, for example with a JFET or with a SQUID, which is shown in figure 2.

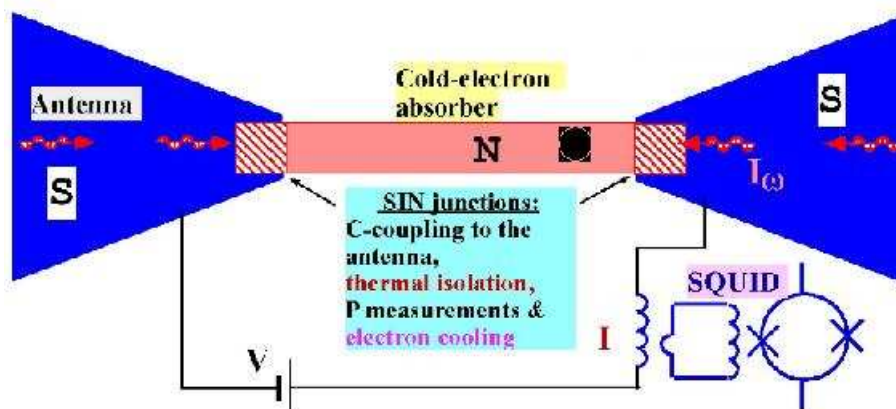


Figure 2: *Schematic picture of a voltage biased bolometer with SQUID readout.[1]*

The physics behind the bolometer is that the energy of an incoming photon is absorbed by an electron in the absorber. The energy of the electron will thus increase by  $\hbar\omega$ , where  $\omega$  is the angular frequency of the incoming photon. This electron will then relax down to



the  $kT_e$ -level, that is the Fermi distribution will change (see fig. 3 from the blue to the red line). The electron will then either interact with a phonon or tunnel through the insulating barrier. The timescale for interactions with phonons ( $10\mu\text{s}$ ) is considerably longer than for tunneling ( $10\text{ns}$ ), which is good since we want the electron to tunnel through the barrier without losses to phonons.

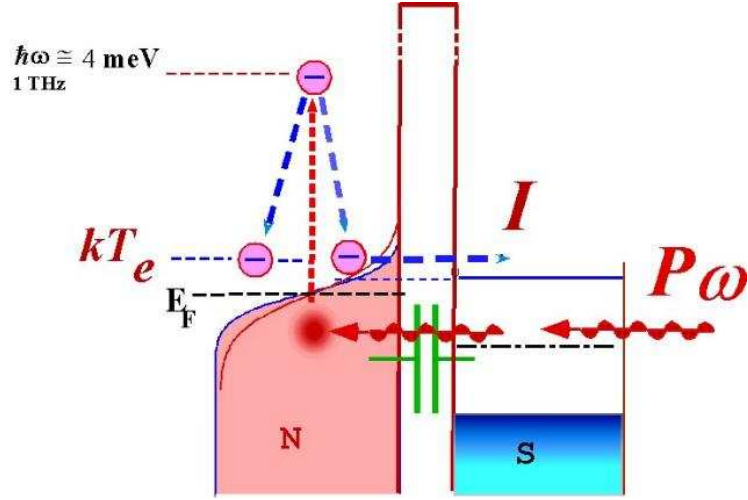


Figure 3: Schematic picture of the tunnel barrier, the energy distribution and basics of the physics.[1]

If the electron tunnel through the barrier it will then be trapped in an electron trap outside the absorber. This means that the absorber will be in the same state as before it absorbed the radiation. That is the temperature will go up when it absorbs a photon but it will then lose the same energy when the electron tunnels out of it. An advantage of this electron cooling is that the background power load can be removed without much noise. This is in contrast to other systems, for example to a transition edge sensor (TES), where the background power has to be removed by the electron phonon coupling.

The energy transfer from the electrons to the phonons can be calculated by[1]:

$$P = \Sigma V (T_e^5 - T_{ph}^5) \quad (1)$$

where  $\Sigma$  is a material constant,  $V$  is the volume of the absorber,  $T_{ph}$  is the phonon temperature and  $T_e$  is the electron temperature which thus can be calculated as:

$$T_e = \left( T_{ph}^5 + \frac{P}{\Sigma V} \right)^{1/5} \quad (2)$$

The electron cooling and the efficiency of increasing the temperature by the radiation (which would increase the sensitivity) can thus be improved by decreasing the volume, see equations 1 and 2.

An alternative way to see this is by calculating the thermal conductance from electrons to phonons, using equation 1:

$$G_{e-ph} = \frac{dP}{dT_e} = 5\Sigma VT_e^4 \quad (3)$$

It is desirable to have this thermal conductance as low as possible since we otherwise would lose energy to the phonons. It can thus again be seen that we want to make the volume of the absorber as small as possible.

In conventional bolometers with an absorber of a normal metal, of for example copper, the volume is  $\sim 10 \times 0.2 \times 0.05 \mu\text{m}^3$ . By using a carbon nanotube instead can this be greatly reduced, even down to  $\sim 10 \times 0.001 \times 0.001 \mu\text{m}^3$ , that is four orders of magnitude smaller!

The noise properties of the CEB are characterized by the total noise equivalent power, NEP, and can be calculated by[1]:

$$\text{NEP}_{\text{total}}^2 = \text{NEP}_{\text{e-ph}}^2 + \text{NEP}_{\text{amp}}^2 + \text{NEP}_{\text{SIN}}^2, \quad (4)$$

where the different contributions comes from electron-phonon interactions

$$\text{NEP}_{\text{e-ph}}^2 = 4kT^2G_{\text{e-ph}}, \quad (5)$$

the amplifier

$$\text{NEP}_{\text{amp}}^2 = \frac{\delta I_{\text{amp}}^2}{S_I^2} \quad (6)$$

and from the SIN junctions

$$\text{NEP}_{\text{SIN}}^2 = \frac{\delta I_{\omega}^2}{S_I^2} + \delta P_{\omega}^2 - 2\frac{\delta P_{\omega}\delta I_{\omega}}{S_I}. \quad (7)$$

The first term in equation 7 is shot noise, the second term comes from the fluctuations of heat flow through the tunnel junctions and the last term is the correlation between these two processes. If we have a strong electron cooling, that is when the process of electron tunneling through the barrier dominates over the electron-phonon coupling, will the NEP be dominated by the contribution from shot noise, which then can be calculated by:

$$\text{NEP}_{\text{shot}}^2 = (2P_0k_B T_e)^{1/2}, \quad (8)$$

where  $P_0$  is the background power load. If we estimate that  $P_0 = 10\text{fW}$  and that we have cooled the electrons to 50mK, would this give a  $\text{NEP}_{\text{shot}}^2$  of  $10^{-19}\text{W/Hz}^{1/2}$ , which is close to what is wanted for future space telescopes.

### 3 Production

The layout of the chip was as can be seen in figure 4. The chip design consists of 16 large contact pads, 18 smaller gold electrodes with wires that creates a number of gaps where the nanotubes are being placed. There is also a network of wires that are used for measurements, for example the four electrodes deposited on the nanotubes that are used for read out.

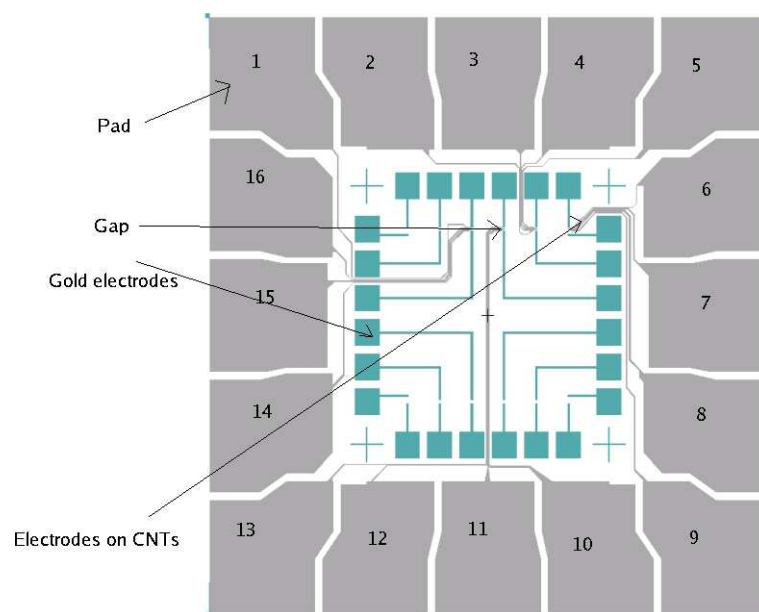


Figure 4: *The design of the chip. Enumerated pads and the main different parts are showed.*

The chip was made in the following way. A 7x7mm silicon substrate with 400 nm silicon dioxide was used. An electron beam made mask was first used to make the small electrodes and their connected wires with photolithography. The electrodes and wires are made of first a thin, 5 nm thick, chromium layer that reacts with silicon dioxide and act as a contact layer between the silicon dioxide and the gold that was placed on top of the chromium. The gold layer was made 120 nm thick. This photolithography and the electron beam lithography process used later were both made using a two resist layer method. This method makes it possible to do a lift-off process. The bottom layer (the LOL 2000) acts as the lift off layer, this will be completely removed by a solvent. So to get the best result the lift off should be developed with a little bit undercut. Because then a deposited film does not get continuous (deposited on the walls) and therefore stays while removing the resist.

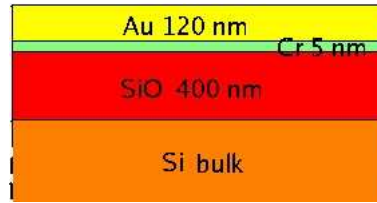


Figure 5: *The vertical design of the chromium - gold electrodes.*

The next step was to bond tiny gold wires to the gold electrodes. Then some drops of a single walled nanotube (with an average size of 1.5 nm) solution was put on the chip. This solution is bought and the concentration of it is unfortunately not known since it changes during the purification process (which also may cause damage and defects to the nanotubes as a consequence of ultrasound baths and acid treatments). The concentration of the solution can only be approximated by looking at the color, the darker solution the higher concentration of tubes. The attached gold wires is then used to apply an AC-voltage of  $2 \text{ V}/\mu\text{m}$  over one gap at a time in approximately 3-10 seconds. And through a process called dielectrophoresis (further explanation see below) the nanotubes will be pulled down to the substrate, and into the gap (which is  $5 \mu\text{m}$  wide), where they stick through Van der Waals forces. So the number of attached nanotubes is hard to know because it depends on the unknown concentration, the time and the applied voltage. But the number of nanotubes will be large, several thousands, and of these a third are theoretically said to be of metallic nature. Most of these however have a very small bandgap which usually doesn't affect much but at very low temperatures this might be important. Approximately 1/20 of the tubes are truly metallic.

The process of dielectrophoresis is used to control and speed up the attachment of nanotubes. They would reach the substrate even if the voltage was not applied but it would take much longer time and they would not be arranged aligned in the gap. The basics of dielectrophoresis is that an AC-voltage is applied between two electrodes, see figure 6. Since the nanotubes are able to be polarized will they act as strong dipoles. If the applied field would be static would the nanotubes follow a field line down to the electrodes. But now since the electric field is non-uniform will the force be different on each side of the nanotube, and therefore will they wiggle back and forth and this will make the nanotubes align in the direction of the electric field. Due to these movements will a dipolemoment be induced, which will interact with the electric field and create a force downwards. The nanotubes will thus move down to the electrodes and stick there due to van der Waals forces.

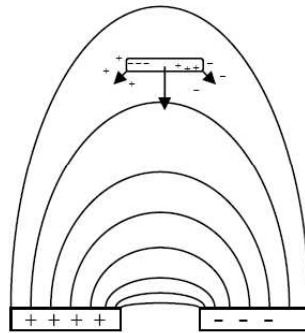


Figure 6: A nanotube and the electric field from the electrodes. The forces acting on the the nanotube resulting in a force towards the electrodes are also shown.

The next step is to create the large contact pads and the network of electrodes that are used for measurements. We worked with two different samples, one with electrodes of Aluminum and one with electrodes made of three layers of metal; 0.5 nm Titanium, 5 nm Palladium and 100 nm Aluminum. The Titanium is used as an adhesion layer, the Palladium to make a good contact with the nanotubes since it has a similar work function and a good wetting interaction to the carbon nanotubes and the aluminum because it is superconducting at the temperatures we are working with. This Ti - Pd - Al chip also had different sizes of the electrodes on the nanotubes, two were 1  $\mu\text{m}$  wide and two were 400 nm wide.

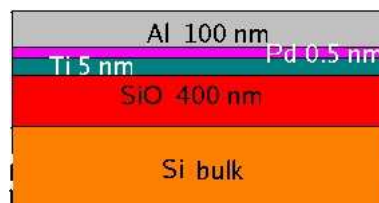


Figure 7: The vertical design of the titanium - palladium - aluminium structures.

The structures described above were in detail done with electron beam lithography as follows. The chip was cleaned with acetone, covered with the LOL 2000 resist through a spin of 1 min at 3000 rpm with an acceleration time of 500 ms and deaccelaration time of 1 second. The sample was then baked at 180 degrees Celsius for 5 min. The second resist layer was UV5-06 that was spun for 1 min at 4000 rpm at an acceleration time of 3000 ms and deacceleration time of 1 second. This second layer was baked at 130 degrees Celsius for 2 min. This two spinning procedured resulted in first a LOL layer that was 180 nm thick and on top of that an UV5 layer with a thickness of 650 nm. The sample was then placed in

an electron beam lithograph and a current of 10nA was used to make the desired pattern. When the patterning was ready the sample was postbaked for 1.5 minutes at 130 degrees Celsius and then developed with MF24A developer for 30 seconds. The result showed that the rests of the bonded gold wires probably damaged the resist layers a bit. The last step was to deposit with an e-gun evaporator (AVAC HVC 600) the three metal layers one by one.

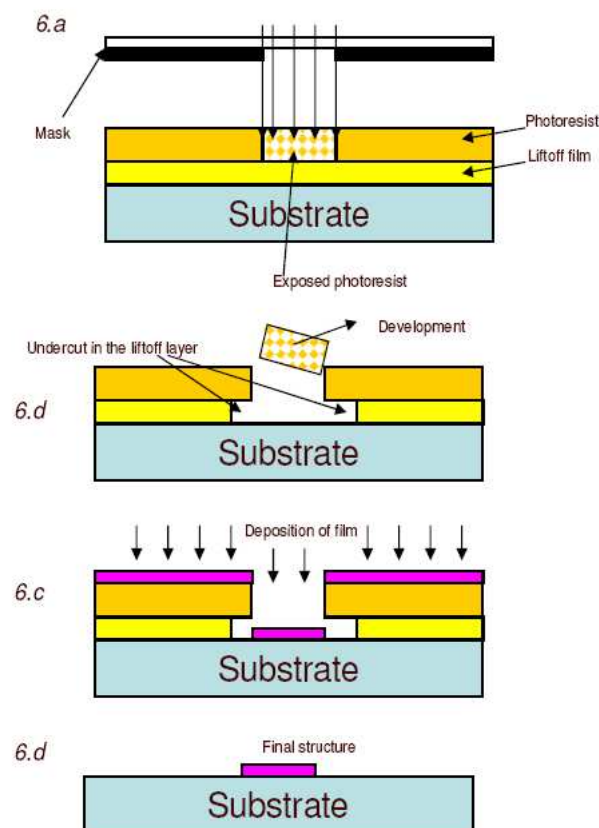


Figure 8: *Lift-off process with two layers of resist. Undercuts are created that helps the deposit to not form a continuous film.*

After these fabrication steps were the chips investigated in a scanning electron microscope, SEM, see figure ??nd appendix ??.

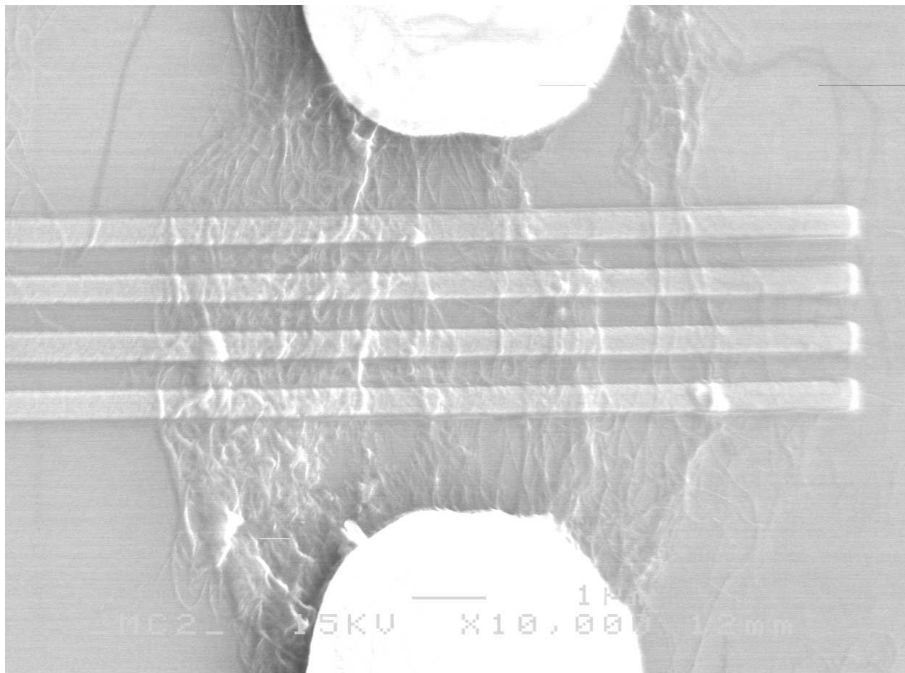


Figure 9: ....

## 4 Experiments

To be able to characterize and analyze our samples we did a number of IV-measurements. This was done for both our chips at room temperature, 4.2K and 300mK.

### 4.1 Setup

During the measurements the sample was put in a sampleholder to secure a good contact and to ease the switching between the 16 electrodes. Depending on temperature this was then connected to a "switch-box" either directly for room temperature, on a deepstick for 4.2K in liquid  $^4\text{He}$  or in the closed-cycle cryostat (HelioxAC- $V^{TM}$  from Oxford Instruments) for measurements at 300mK. The switch-box was then connected to an amplifier stage where gain and bias resistances could be set. The amplifier stage in turn was fed by a sweep-source which could be set to run on an external signal. We used two multimeters to read out the voltage and current as well as an oscilloscope to display the IV-curves live. To control the measurements and to store the output data we used a computer with LabView and a digital-analog-converter.

## 4.2 IV-Measuerments

We did a number of IV-measurements for all possible combinations of electrodes in each gap in order to analyze our samples. The IV-characteristics were measured in voltage biased mode and performed in two, three and four probe mode, even though it was not always possible to do all these. The four probe mode was of course desirable to be able to measure the resistance of individual barriers and pieces of nanotubes.

The software tool Origin was used to handle our data and to plot the IV-curves.

We investigated the general characteristics and any differences in resistance when using bundles of nanotubes compared to using a single one. Also the questions whether the composition of the electrodes or their width would have any influence on the resistance was examined.

# 5 Results

## 5.1 Chip4 - Aluminum electrodes

In the measurements of the chip with just aluminum as electrodes we were only able to use one gap, gap 4. This was due to of the four possible gaps (just four because of the design, we only have 16 pads) the electrodes were damaged somewhere in three of them. But in the "undamaged" gap were we able to do four probe, three probe and two probe measurements.

The general electrical behavior of these two and three probe measurements can be seen in figure 10.



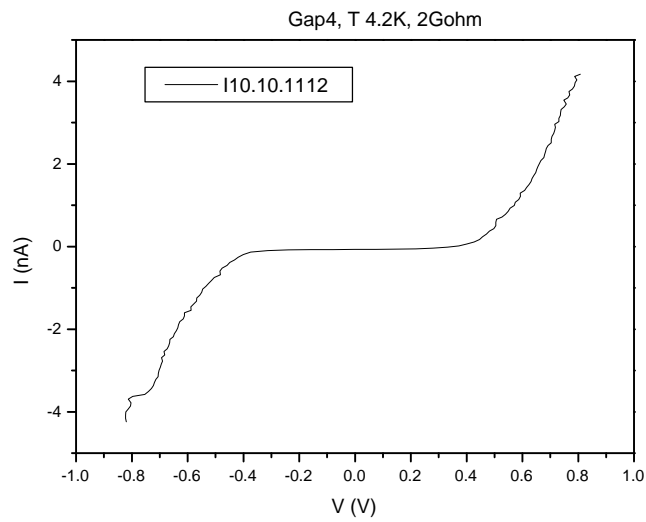


Figure 10: ...

The IV-curves for the four probe measurements were similar, see figure 11.

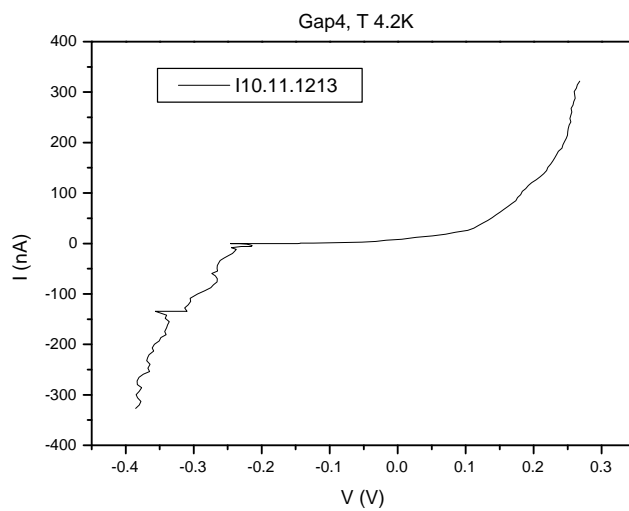


Figure 11: ...

The contact resistance was mostly about  $1\text{ M}\Omega$  and the energy gap in the  $1\text{ V}$  range, see appendix ??.

## 5.2 Chip3 - Titanium/Palladium/Aluminum electrodes

Of the four possible functioning gaps at chip 3 none were totally undamaged, which means that we could not do any four probe measurements of this. The gaps used were number 3,4,5 and 6. At gap 3,4 and 5 one electrode was broken and on gap 6 two electrodes were broken. This means that we were only able to do three probe measurements of gap 3, 4 and 5. The reasons to the not functioning electrodes were in one gap a shortcut and in the other three gaps the resist had been damaged due to that some of the bonded gold wires were not completely removed.

The general electrical behavior of these two and three probe measurements can be seen in figure 12.

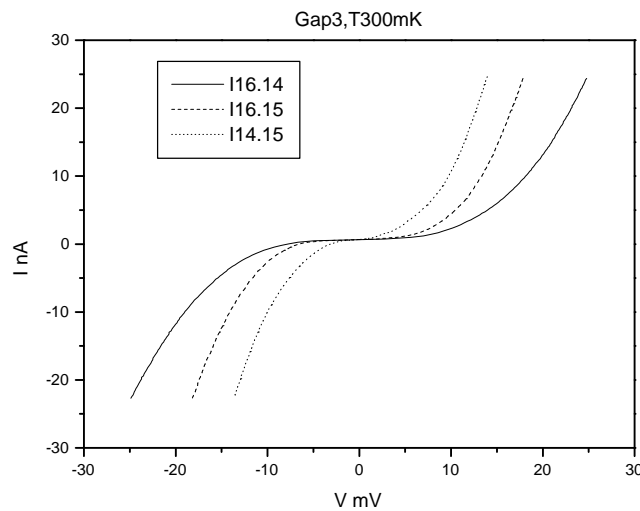


Figure 12: ...

But in the measurements of this chip at  $300\text{ mK}$  we also got two other behaviors, as follows:

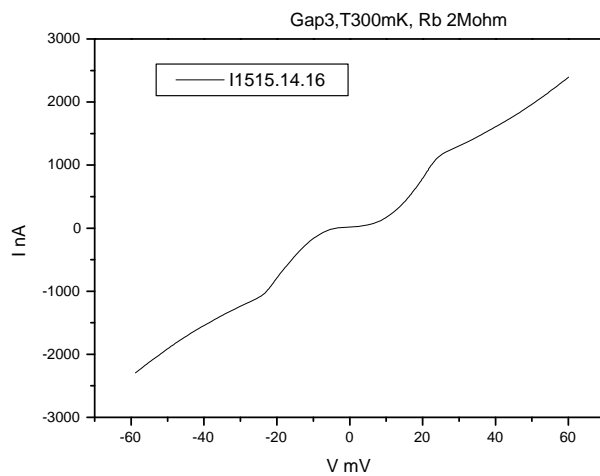


Figure 13: ...

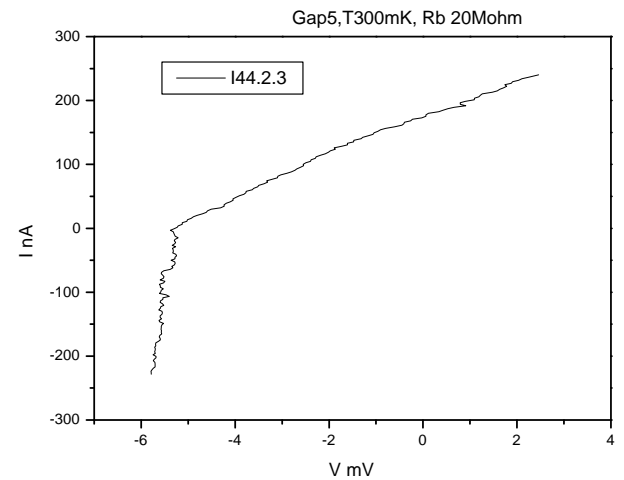


Figure 14: ...

The resistances of these contacts and nanotubes was in the  $k\Omega$  range and the energy gaps are in the 10 mV range.

## 6 Analysis

The general behavior of our measurements, for both composition of electrodes, indicates that we have a Schottky barrier between the normal metal electrodes and the nanotubes, see figure ???. For temperatures below the superconducting transition temperature of the electrodes we usually have a super-Schottky barrier, see figure ??.

The resistance with pure aluminum contacts ( $M\Omega$  range) was too high to be useful in applications. By also including Palladium in the electrodes this resistance could be decreased ( $k\Omega$  range), see appendix ??.

We could not find any difference in resistance between wide and narrow electrodes contacting the nanotubes, see table 1 (for more detailed table see appendix ??).

Table 1: Comparison of resistance of narrow and wide electrodes in chip 3 at 300 mK, illustrating that there is no difference in resistance between them.

Junction	resistance ( $k\Omega$ )	narrow/wide electrode
15/15/14/16	24	narrow
16/16/15/14	60	wide
14/14/15/16	53	narrow
12/12/13/11	4	wide
13/13/12/11	18	wide
11/11/12/13	19	narrow
3/3/2/4	130	wide
2/2/3/4	36	wide
4/4/2/3	32	narrow

The current-voltage characteristics in figure 13 is very similar to a Josephson junction with suppressed supercurrent. The energy gap is about 20 mV even though the graph is quite smeared, which could be due to that we have many nanotubes in series.

We found the behavior in figure 14 interesting, would the left part continue to decrease linearly? So we made the same measurement but with lower bias resistance and found that this was not the case, see figure 15. Instead we found that the behavior was similar to the behavior in figure 13.

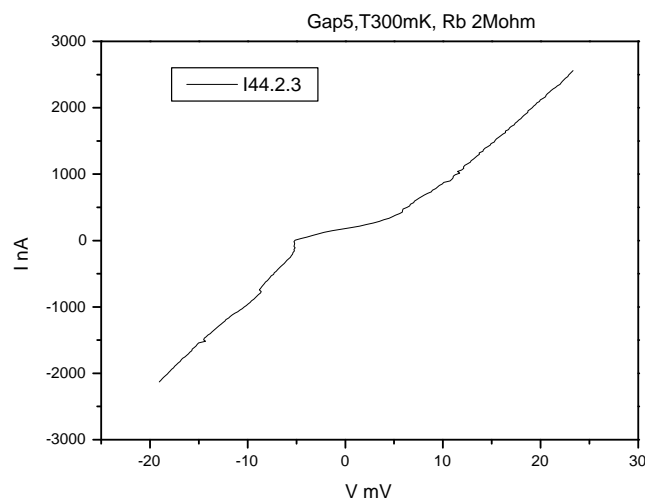


Figure 15: ...

4-PROBE

## 7 Discussion

Unfortunately we could only do one four probe measurement and this limited our results. If we had been able to do more we could have measured the resistance of bundles of tubes and contacts individually. This would have given us more information of how the tubes influenced our measurements. It is difficult to get a clear understanding of the electrical properties when we have so many nanotubes lying around and probably many of them with defects. The defects themselves can form quantum dots which makes the results even more hard to interpret.

Carbon nanotubes are simply not a normal metal and it is therefor not as easy as just to replace the copper absorber. For instance since nanotube are one dimensional objects we will always have an inevitable resistance at the interface to the three dimensional bulk [5]. For an ideal 1D-conductor this resistance can not be lower than  $\sim 12.9k\Omega$  per mode. Since the electrons can have spin up and spin down this means that the resulting lowest possible resistance of an ideal 1D-conductor, including the contacts, is  $\sim 6.5k\Omega$  [4]. One will also have to consider other effects when changing from normal metal to carbon nanotube, as for example how the optical properties will differ.

If the bolometer will be voltage biased it is not an advantage with two SIN-junctions. In this case it would be better to turn one into an Andreev interface since the electrons then would tunnel through only one barrier improving the sensitivity. This is not a problem if the bolometer instead will be current biased.

It would be better if one were able to control the number of nanotubes in the gap better in order to make this device really reproducible. It would also be good if one could manufacture the nanotubes more accurately to avoid defects and to be able to control their electrical properties.

The remaining gold wires from the bonding causes damage to the resist which sometimes ruins the fabrication of the electrodes. This could be avoided by using needles instead of bonding during the dielectrophoresis. Changing the design would also rectify this problem.

Sända på ljus

## 8 Conclusion

We have found that the contact resistance can be decreased by using bundles of single walled carbon nanotubes instead of individual nanotubes when replacing a normal metal as absorber in a cold electron bolometer. Our work has also confirmed that the contact resistance is lower when using electrodes composed of Ti/Pd/Al instead of pure Al. We could also conclude that the width of the electrodes does not affect the contact resistance.

This is a small step towards realizing a cold electron bolometer based on carbon nanotubes

## References

- [1] Lectures by Leonid Kuzmin, Modeling and Fabrication of Micro/Nano Devices, Spring 2006.
- [2] Editors E. Burstein, S. Lundquist "*Tunneling Phenomena in Solids*", Lectures presented at the 1967 NATO Advanced Study Institute at Risö, Denmark.
- [3] .....
- [4] K.S.Yngvesson, "*Very wide bandwidth hot electron bolometer heterodyne detectors based on single-walled carbon nanotubes*", Applied Physics Letters 87, 043503 (2005).
- [5] Yoseph Imry, Rolf Landauer "*Conductance viewed as transmission*", Reviews of Modern Physics, Vol. 71, No. 2, Centenary 1999.

## Appendix