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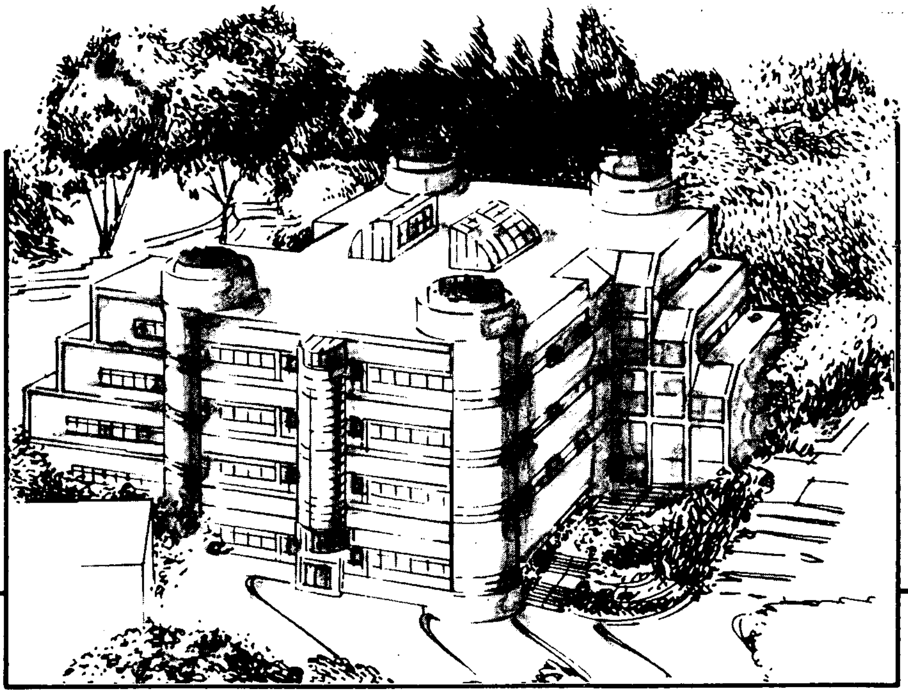
July 1988



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DISCLAIMER

Bias Induced Nonlinearities in Neutron Transmutation Doped Germanium at ^{4}He Temperatures.

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(Received

Abstract

Non-thermal nonlinearities have been studied in Neutron Transmutation Doped germanium resistance thermometers at ^{4}He temperatures. The temperature dependence of the low-field resistance indicates that the conduction is due to variable range hopping, which dominates at the lowest temperatures, and ionized impurity conduction, which becomes important at the highest temperatures. The observed nonlinearities of DC I-V curves are consistent with the predictions for variable range hopping. In addition, a carrier relaxation time significantly larger than the thermal and RC time constants has been observed in the more lightly doped thermometers. Implications of these phenomena for the use of germanium thermometers in absolute measurements of temperature, infrared radiation, exotic particles and heat capacity are discussed.

Introduction

Doped single-crystal germanium is widely used in the fabrication of resistance thermometers, which operate near ^{4}He temperatures.[1, 2] Under proper conditions, the resistivity of these materials varies rapidly with temperature, and there is little or no excess noise. Small thermometers can be made with relatively low heat capacity and high internal thermal conductance. These properties alone qualify doped germanium as excellent thermometer material. Highly uniform dopant concentrations can be achieved through Neutron Transmutation Doping (NTD), in which Ge atoms are transmuted by nuclear reactions with thermal neutrons.[3] By changing the doping density, the resistivity at a given temperature can be controlled, allowing the fabrication of sensitive thermometers with impedances that are compatible with low-noise amplifiers. Such thermometers have been successfully used as the thermal sensing elements of bolometric infrared detectors[4] as well as differential microcalorimeters.[5]

Typical bolometric radiation detectors consist of an energy absorbing element and a current-biased electrical resistance thermometer. These components are connected to a heat sink by a known thermal conductance. The thermal response time of the device is determined by its heat capacity and the thermal conductance. Characterization of such bolometers often begins with the measurement of a DC I-V curve.[6] For a given thermal conductance to the heat sink, there exist bias currents small enough that the temperature rise due to dissipated electrical power can be neglected. For these small currents, the thermometer resistance should be independent of the bias current and the I-V curve is said to be ohmic. As the bias power is increased, a temperature difference appears between the thermometer and the heat sink, giving rise to a change in its resistance, and a non-ohmic, thermal nonlinearity in the I-V curve. In practical applications, a small amount of signal power is dissipated in the detector, leading to an additional temperature rise, and a correspondingly small change in the voltage across the thermometer. To first order, the change in voltage is linearly related to the applied signal power.

The absolute sensitivity of the bolometer can be determined from the shape of the I-V curve[6, 7] if the electrical power dissipated in the thermometer produces the same result as signal power dissipated in the absorbing element, and if all of the nonlinearity in the I-V curve is of thermal origin. However, the changes in resistance observed in an I-V curve cannot always be attributed to temperature changes. Conduction in the thermometers under study is expected to be due in part to variable range hopping.[3] For this conduction mechanism, electric fields can induce nonlinearities,[8, 9] and many theoretical discussions have been published.[10-13]

It usually is advantageous to modulate the incoming signal in order to reduce the effect of low frequency noise. The response of the detector as a function of modulation frequency must be known in order to obtain quantitative results. The frequency dependence of the response usually is ascribed to the thermal time constant of the detector or, occasionally to the electrical time constant of the measurement circuit. We have found that some thermometers have an effective internal relaxation time that is longer than either the thermal time constant or the electrical time constant. In order to isolate internal relaxation times from external thermal or electrical relaxation times, a number of different thermometers were measured in the same thermal and electrical circuits.

In this work, the resistance of several NTD germanium resistance thermometers has been measured as a function of temperature, bias voltage, thermal oscillation frequency and dopant concentration. The results have been compared to the theoretical predictions found in the literature and to existing experimental data. The importance of these effects to absolute measurements of temperature, infrared radiation, exotic particles and heat capacity is discussed.

Experimental Technique

The NTD thermometers used in this experiment were fabricated from ultrapure Ge single crystals, which were doped to the acceptor and compensating donor concentrations as shown in Table 1 by exposure to a thermal neutron source.[3] This procedure takes advantage of the uniformity of the neutron source and the small transmutation cross-sections to produce very uniform dopant concentrations. In contrast, both implantation doping and melt doping of semiconductors can produce uneven dopant concentrations, which greatly complicates the problem of obtaining a suitable thermometer. The crystals are sliced into 300 μm wafers, fine-lapped, and etched in a 4:1 HNO_3:HF solution to remove lapping damage, provide clean surfaces for ion implantation and to insure contact adhesion. Both surfaces are step-implanted with B^+ ions (1×10^{14} cm^{-2} at 25 keV, 2×10^{14} cm^{-2} at 50 keV), which produces a metallic region in the Ge approximately 2000 Å deep. A 200 Å layer of Pd and a 4000 Å layer of Au are argon sputtered onto the wafers, which are then annealed for one hour at 300 C. Annealing helps activate the implanted ions, and relieves any stress resulting from the metallization. The wafers were cut into chips 0.265 mm on a side, and rinsed briefly in the 4:1 solution to etch the damaged surfaces and thus remove surface electronic states, which might irreproducibly contribute to conduction at low temperatures. The final size after all processing is approximately $(0.25\ \text{mm})^3$.

Several different NTD thermometers were mounted in each test apparatus as is shown in Fig. 1. The thermometers were attached to evaporated NiCr films on a sapphire substrate with an Ag-filled conductive epoxy.[14] Cu wires with a diameter of 0.001" were also attached to the thermometers and to the films with conductive epoxy. The other ends of the Cu wires were soldered to Cu heat sink posts. This assembly, which was designed for other purposes[5], is a useful test bed for the comparison of nonlinearities of two thermometers because of the ability to do simultaneous tests in nearly identical thermal surroundings.

A 9 volt mercury battery and a ten-turn 10 kΩ potentiometer produced a variable DC bias voltage across a room temperature carbon load resistor in series with a cold thermometer. Both the total bias voltage and the voltage across the thermometer were measured with digital voltmeters with input impedance > 1 GΩ and recorded manually. Because of the large thermometer impedances studied, two-wire measurements of resistance were sufficiently accurate, and the lead resistances have been neglected. To check for thermal EMFs, the polarity of the bias was periodically reversed.

Measurements of the dependence of the resistance on thermometer lattice temperature and DC bias voltage were carried out with the test apparatus submerged in liquid ^{4}He. The thermal conductance to the heat sink is then limited by the thermal boundary resistance between the Ge surfaces of the thermometers and the liquid ^{4}He, and is of order 10^{-3} W/K.[15] The vapor pressure of the helium bath was measured with a capacitance manometer and was used to calculate the bath temperature. The temperature of the helium bath was controlled by observing the vapor pressure and manually adjusting the valve to the vacuum pump. Drifts in the bath temperature were less than 5% during these measurements. The errors introduced by these drifts do not affect the major conclusions of this study.

The phase and amplitude of the voltage resulting from an AC thermometer bias were measured as a function of frequency for several different thermometers with different resistances. The results were consistent with an RC rolloff due to a capacitance of approximately 300 pF.

Measurements of the response to an applied thermal oscillation were carried out with the apparatus mounted on a cold finger in a conventional UHV system, where the thermal conductance to the heat sinks is limited by the copper wires and is of order 10^{-5} W/K. The amplitude and phase of the thermometer resistance oscillation were monitored with a two-phase lock-in amplifier and recorded manually.

Experimental Measurements

As the applied electric field approaches zero, the resistivity of these materials approaches a constant value, which depends on the temperature. For variable range hopping in a disordered semiconductor, one expects to find low-field resistivities which vary as[16]

$$R(0,T) = R_0 \exp (T_0/T)^{\alpha}, \qquad (1)$$

where α is equal to $\frac{1}{4}$ if one assumes that the medium is three-dimensional and that the carrier density of states near the Fermi surface is constant. However, it has been shown that long range Coulomb interactions between localized electrons can give rise to an energy gap between the filled and empty states.[17, 18] The resulting quadratic variation in the density of states near the Fermi surface leads to a low-temperature resistivity as in Eq. 1, but with $\alpha = \frac{1}{2}$. At higher temperatures, where k_BT is comparable to the Coulomb gap, deviations from this temperature dependence are expected. Temperature dependent resistivities as in Eq. 1 with $\alpha = \frac{1}{2}$ have been observed experimentally,[19, 20] and a review of all these considerations for the case of doped Ge has been given by Efros and Shklovskii.[21] The low-field resistivity of the four samples studied is shown in Fig. 2a as a function of $T^{-1/2}$. At the lowest temperatures studied, the resistivities of all four samples as plotted in Fig. 2a approach straight lines. This is consistent with previous low temperature measurements on similar samples[3], and with the predictions for variable range hopping in the presence of a Coulomb gap. As the temperature increases, the deviations from straight lines indicate the presence of a change in the conduction mechanism. At the highest temperatures, the data are better fit by Eq. 1 with $\alpha = 1$, as shown in Fig. 2b.

This suggests that conduction due to ionization of bound carriers is becoming important.

As the electric field is increased, deviations from ohmic behavior become apparent. A series of I-V curves for a typical thermometer measured at different temperatures in the liquid helium bath are shown in Fig. 3. Since the resistance of these thermometers depends on temperature, these curves might be interpreted as evidence for increases in thermometer temperature due to dissipated bias power. However, quantitative measurements do not support this hypothesis. For example, in the I-V curve measured at 1.25 K for sample NTD-2, a nonlinearity of a few percent is observed at an applied bias power of less than 10^{-9} W. Since the thermal conductance between the thermometers and the bath is of order 10^{-3} W/K, the expected temperature rise is 10^{-6} K. This should produce a change in resistance of about 1 part in 10^6, which is only 10^{-4} of the observed effect. This leaves only the possibility that the resistance depends on the applied bias voltage as well as the temperature.

Much theoretical work has gone into the understanding of electrical nonlinearities in hopping conduction, and the predictions[10-13] are that the resistance depends on the electric field

$$R(E,T) = R(0,T) \exp(-eEL/k_BT), \qquad (2)$$

where E is the electric field, k_B is Boltzmann's constant, and L is a characteristic length for the hopping process. Different authors predict additional numerical factors in the exponent, but all find a form similar to that in Eq. 2. To check for such a field-dependent nonlinearity, the I-V curves in Fig. 3 were re-plotted as shown in Fig. 4. At each bath temperature, the data fit straight lines, indicating that the measured nonlinearities are consistent with Eq. 2. The non-zero slopes indicate that there is an electric field-induced nonlinearity in all of the samples throughout the range of temperatures and electric fields investigated. The slight deviations from straight lines in Fig. 4 are within the drifts expected from bath temperature variations during a particular measurement, and do not recur.

As the bath temperature is changed, the slope of the lines through the data in Fig. 4 also changes, implying that the characteristic hopping length L has a temperature dependence. Fig. 5 shows a plot of the temperature dependence of L for thermometers with three different doping densities. The nonlinearity in Eq. 2 is most important when the characteristic hopping length is large and the temperature is low. Therefore, measurements below 1 K will be most seriously affected. Surprisingly, the samples with the largest average impurity separation show the smallest characteristic hopping lengths. The characteristic hopping length in variable range hopping in the presence of a Coulomb gap is expected[21] to vary as $T^{-1/2}$ at low temperatures. This prediction is also plotted in Fig. 5 and is not in good agreement with the data.

When the test apparatus is placed in a vacuum and an oscillating current is applied to the heating strip, there is an oscillation in the resistance of the thermometer. If the thermometer response is sufficiently fast, the amplitude of the resistance oscillation is determined by the modulation frequency, heat capacity of the test apparatus, and the thermal conductance to the heat sink.[22] Two thermometers mounted on the same test apparatus would then have the same thermal response. Fig. 6 shows the frequency dependence of the amplitude of the voltage oscillation of four current-biased thermometers mounted on the same substrate. The data for NTD-4 fit a thermal relaxation model with a time constant $\tau = C/G = 8$ ms up to 2 kHz. This value of the thermal time constant is consistent with the known values of C and G for the test apparatus. Above 2 kHz, the rolloff steepens because of the electrical time constant of 0.6 ms. Additional measurements on very heavily melt-doped thermometers are coincident with those for NTD-4, the most heavily doped thermometer in this study. Since the curve for NTD-4 shows the fastest response and is coincident with that of a completely different thermometer, it reflects the thermal response of the test apparatus. The data for NTD-1 through 3 show an additional internal relaxation time $\tau = 36$ ms, which is longer than the thermal C/G time of 8 ms and the electrical time constant of 1 to 7 ms. Measurements on a given thermometer at different temperatures, and therefore different resistances, but the same capacitance, produce virtually the same frequency dependences. This is further indication that the observed rolloff is not simply due to the time constants of the electrical circuit.

Discussion

At low applied electric fields, the low-temperature resistivity for conduction by variable range hopping in the presence of a Coulomb gap is expected to follow Eq. 1 with $\alpha = \frac{1}{2}$. The measured resistivities follow this dependence at the lowest temperatures investigated as predicted by Efros and Shklovskii[21]. At higher temperatures, thermal ionization of carriers becomes important, as seen by the approach to an $\alpha = 1$ dependence.

The electric field dependence of the resistance at any given temperature as shown in Figs. 3 through 5 is in agreement with the form predicted for variable range hopping. The rough consistency between the values of the hopping length and the average separation of uncompensated acceptors is encouraging, if not particularly illuminating. Agreement between the measured characteristic hopping length and average impurity separation has not always been found in other materials[9]. The hopping length decreases more rapidly with temperature than is expected. This discrepancy may be taken as further evidence for a transition in the conduction mechanism from variable range hopping to another mechanism, such as thermally ionized carrier conduction.

The appearance of an internal relaxation time greater than the thermal time constants of these detectors is not expected. Simple arguments[23] can be used to set a tight upper limit on the thermal relaxation time of the carriers in this material as follows. The heat capacity of the carriers must be less than that of an ideal gas of carriers whose number density equals that of the uncompensated acceptors in the material. If we interpret the observed nonlinearity in the I-V curves as a change in the electron temperature, we can estimate the thermal conductance from the electrons to the lattice. This procedure has been used elsewhere to estimate the thermal conductance between the electrons and phonons in doped Ge thermometers.[2] From these models, the estimated heat capacity is $C = 10^{-12}$ J/K and the estimated thermal conductance is $K = 10^{-8}$ W/K. The resulting time constant is 10^{-4} s, which is considerably shorter than the observed internal relaxation time. Since the changes in resistance are more likely to be due to the field dependent nonlinearity, this represents an underestimate of the the true thermal conductance present in this system. Since the heat capacity is grossly overestimated, and the thermal conductance is probably underestimated, the true thermal relaxation time for the electrons should be much faster. The mechanism for the internal time constant is not known.

McCammon and coworkers have observed discrepancies between expected and measured responses of x-ray detectors made from antimony doped silicon[24]. In their experiments, individual Mn Kα x-rays were absorbed, producing pulses in the temperature of the detector. The shapes of the measured pulses were not consistent with a single exponential thermal response, and may have been distorted by an internal time constant. They also report a field-induced nonlinearity whose form is consistent with our observations. The appearance of both the internal time constant and the field induced nonlinearity in these two different materials suggests that these effects may be fundamentally related. Similar measurements by Silver and Labov[25] on heavily doped NTD Ge at lower temperatures than in this study have been carried out. Their results indicate the presence of both a field induced nonlinearity, and an internal time constant similar to the effects reported here and by McCammon et. al..

Loponen and coworkers[26] have made extensive measurements of the time-dependent heat capacity of amorphous semiconductors below 1 K. Their measurements at ms time scales showed that the heat capacity had a component with a logarithmic time dependence. A time dependent heat capacity of this form is theoretically expected[27] for glasses which exhibit tunneling excitations with a wide distribution of relaxation times and energies. A similar distribution in relaxation times could be responsible for the distortions in pulse shapes observed by others[24,25], as well as the internal time constant reported here.

There have been many reports in the literature of frequency dependences in the AC conductivity of materials in the hopping conductivity regime.[28, 29] One explanation of these results is that polarizability of the bound carriers increases with frequency and adds to the total AC conductivity of the material. A more complicated model, which includes the effects of spatial fluctuations on the ionization probability of deep levels appears to fit more of the observed details.[30] In the present work, measurements of the voltage response to an AC thermometer bias were consistent with an electrical time constant, and showed no clear evidence of any other a frequency dependent contribution to the AC conductivity up to 10^5 Hz.

The implications of these results for the use of NTD thermometers for absolute measurements of temperature, absorbed power, or heat capacity can be summarized as follows:

1) The electric field dependence of the resistance of these materials complicates their use as ordinary thermometers. In particular, great care must be taken to perform calibrations and measurements at identical voltages, or, preferably, in the low voltage limit, where the field dependence is not important. Another approach is through the use of electrical readouts that measure thermometer current at constant bias voltage. The electric field dependence of the resistivity is especially important at lower temperatures, where the hopping lengths can be quite large.

2) Calibrations of bolometric radiation detectors obtained by calculations based on measured I-V curves will be distorted by the electric field dependence. This is because the observed changes in resistance will not be entirely due to sample heating. Correction procedures for electrical nonlinearities have been proposed.[31] However, the observed temperature dependence of the hopping length shows that more complicated corrections may be required. Therefore, the only reliable calibrations of bolometric radiation detectors are those which deposit the energy in the same place in the thermal circuit as in the actual measurement.

3) The appearance of an internal relaxation time larger than the thermal relaxation time of the apparatus and the RC time constant of the electrical circuit complicates any AC measurement made with these materials. In particular, the sensitivity of bolometric detectors to modulated or pulsed signals will be reduced by this effect. This implies that calibrations of absolute bolometric detectors will have to be performed at the same frequency as the actual measurement. Furthermore, the sensitivity of bolometric detectors being designed for x-ray detection[24, 25, 32] and dark-matter searches[33, 34] depends on fast response and so will be seriously reduced by the observed internal time constant. Finally, the application of these thermometers to AC calorimetry[4] is seriously affected, since accurate measurements require that the slowest time constant be due to thermal relaxation of the entire calorimeter. The presence of an internal time constant longer than the thermal time constant of the detector makes quantitative AC heat capacity measurements very difficult.

For a given bias power, the electric field-induced nonlinearity and the internal time constant decrease in more heavily doped material. This suggests that optimum performance can be obtained through the use of relatively low impedance thermometers. The detailed optimizations depend sensitively on the properties of amplifiers and are beyond the scope of this work.

Conclusion

A series of measurements have been made of the electrical properties of doped germanium thermometer material at ^{4}He temperatures. The temperature dependence of the low-field resistivity is in agreement with the predictions for variable range hopping in the presence of a Coulomb gap at the lowest temperatures studied. At higher temperatures, deviations from this prediction arise due to conduction by thermally ionized carriers. The electric field dependence of the resistivity has a form consistent with predictions for variable range hopping, but the magnitude and temperature dependence of the hopping length deduced are not correctly predicted. Finally, an internal relaxation time is observed which is longer than both the thermal time constant of the test apparatus and the RC time constants in the electrical circuit. The observed internal time constant can seriously distort the response of absolute thermal detectors.

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Table 1

Dopant	NTD-1	NTD-2	NTD-3	NTD-4
Ga	2.21×10^{15}	4.41×10^{15}	6.62×10^{15}	9.94×10^{15}
As	6.28×10^{14}	1.26×10^{15}	1.89×10^{15}	2.83×10^{15}
Se	9.2×10^{13}	1.6×10^{14}	2.5×10^{14}	3.8×10^{14}
Na-Nd	1.49×10^{15}	2.99×10^{15}	4.48×10^{15}	6.73×10^{15}

Table 1 Dopant concentrations in the NTD samples used in this study. All other impurity concentrations are below 10^{13}. All concentrations in cm^{-3}.

Figure Captions

1) Schematic diagram of the test apparatus used in the measurements. All contacts to the wires, thermometers and NiCr films were made with Ag-filled conductive epoxy. As many as four thermometers can be mounted on a single assembly and tested simultaneously.

2) Plots of the log of the resistivity of all four thermometers measured as a function of (Temperature)$^{-\alpha}$ with $\alpha = \frac{1}{2}$ in (a) and $\alpha = 1$ in (b).

3) A series of I-V curves for thermometer NTD-2 submerged in ^{4}He at 6 different bath temperatures. Nonlinearities are evident for applied power as low as 10^{-9} W. Heating of the thermometer by the bias power is significant only above 10^{-5} W.

4) Data from Fig. 3 re-plotted as the log of the resistance versus the ratio of electric field to temperature. Straight lines are consistent with theoretical predictions for electric field assisted hopping.

5) Temperature dependence of the characteristic hopping length L extracted from I-V curves for all of the thermometers in this study. The lines drawn through the data are included as a guide to the eye. The dashed line corresponds to the predicted $T^{-1/2}$ dependence of the characteristic hopping length upon temperature, and has been fitted to the low temperature data.

6) The measured amplitude of the voltage oscillation of the thermometer plotted as a function of the applied thermal oscillation frequency for four

thermometers mounted on the same substrate. Measurements with highly melt-doped thermometers as well as calculations based on estimated values of C and G are consistent with the rolloff for NTD-4. The other more lightly doped thermometers show an additional internal time constant longer than the external thermal or electrical time constants. Electrical RC times become important in all curves after the first decade of rolloff.

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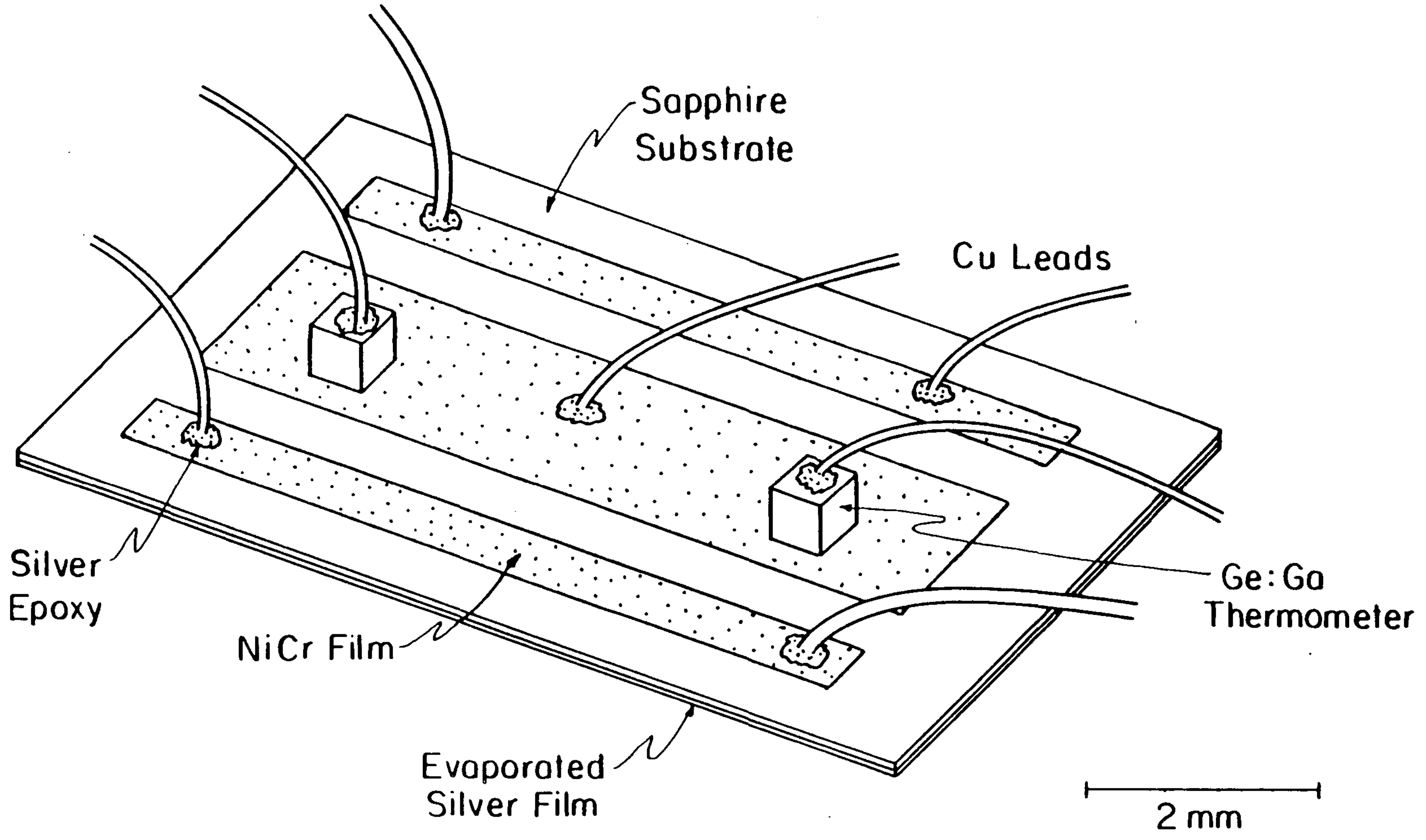
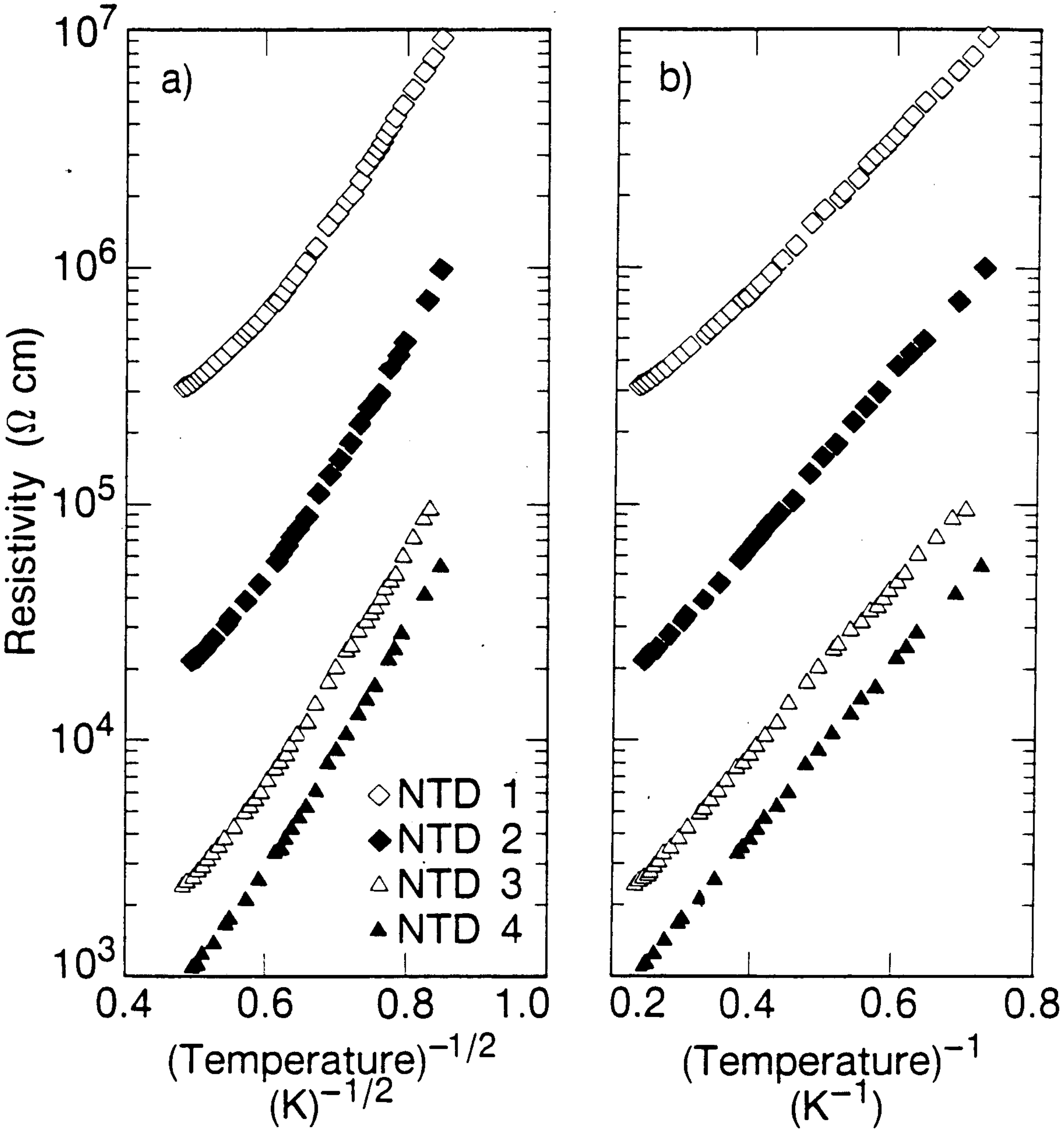


XBL 861-7480

FIGURE 1



XBL 885-7424

FIGURE 2

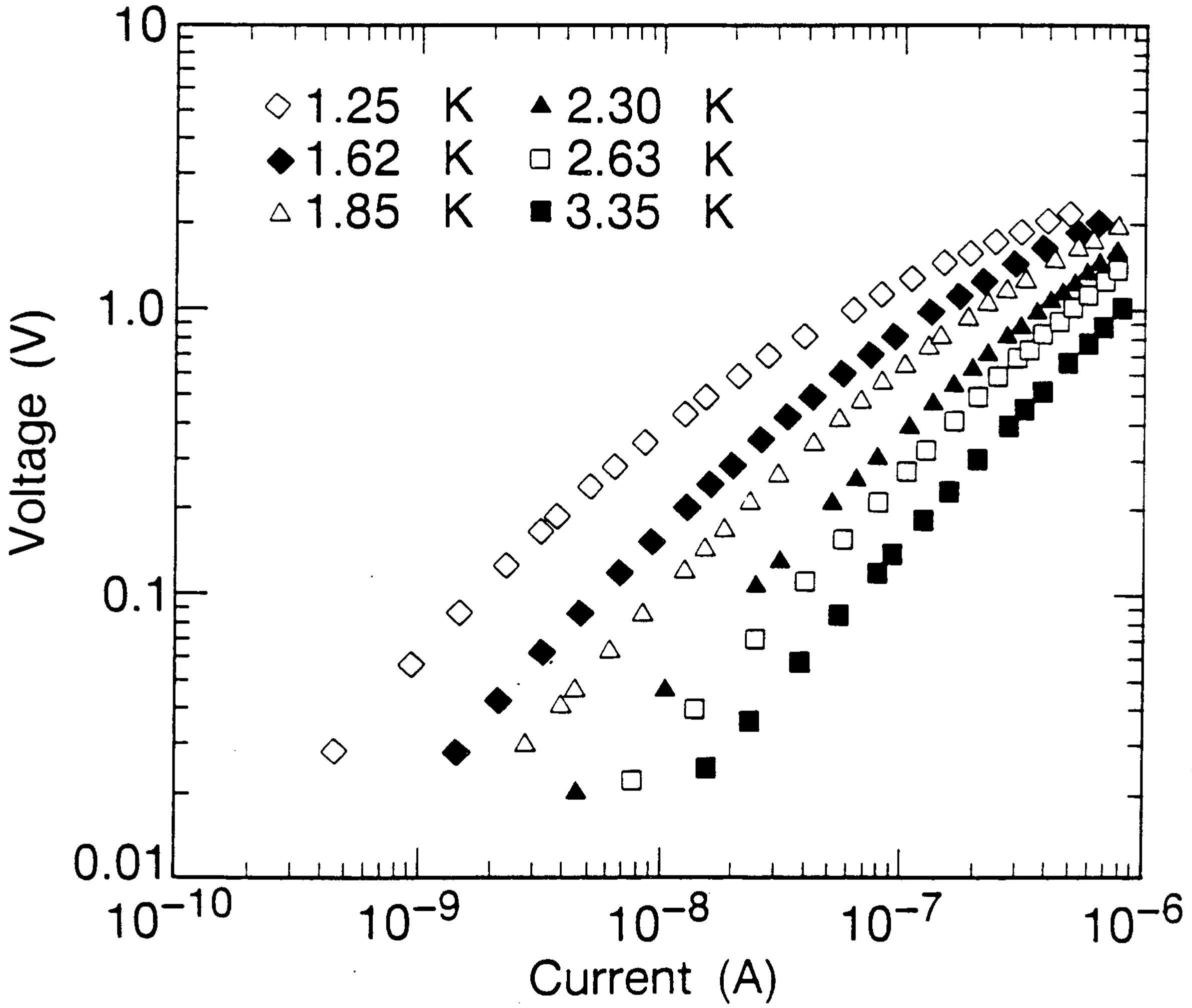


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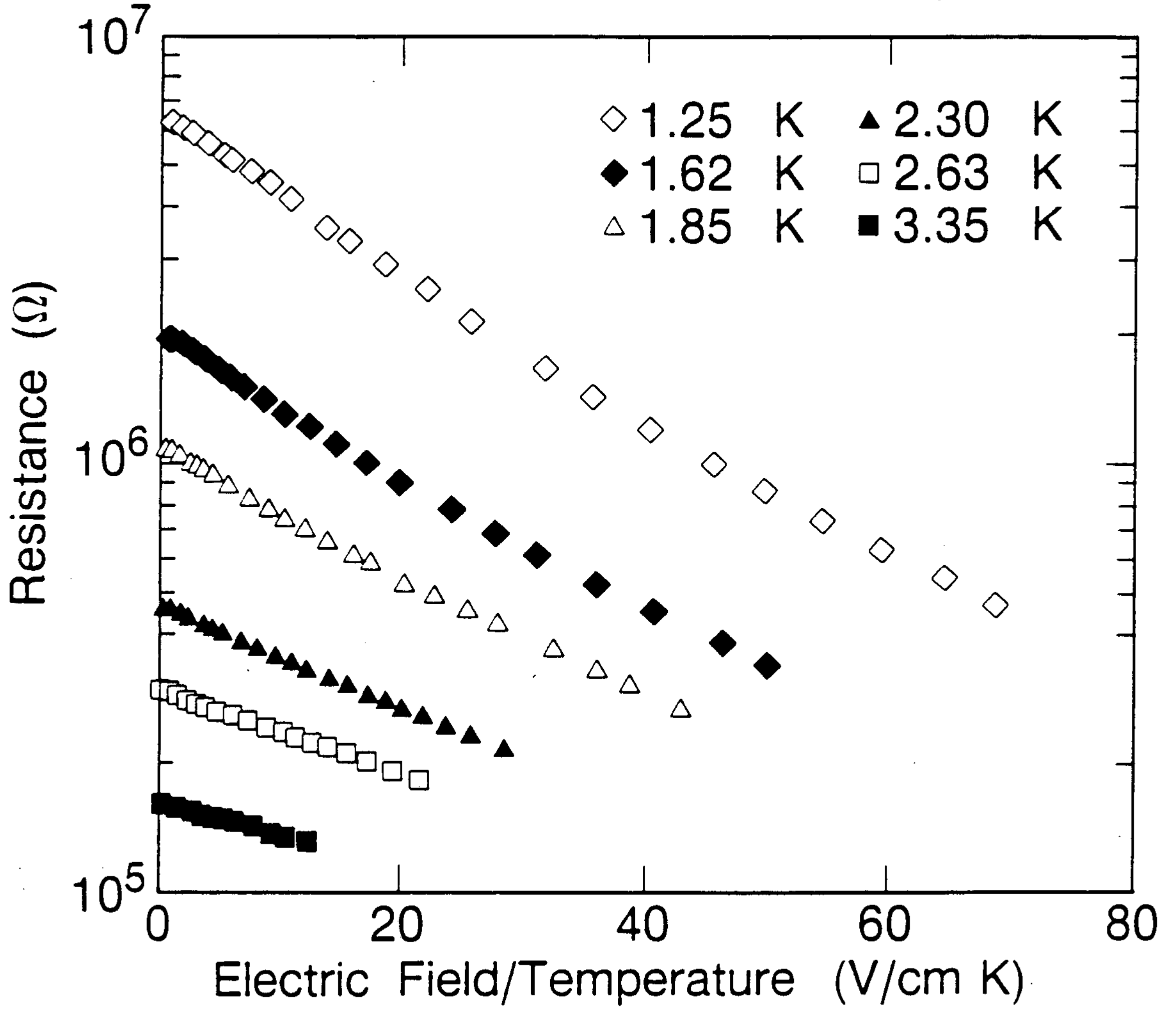
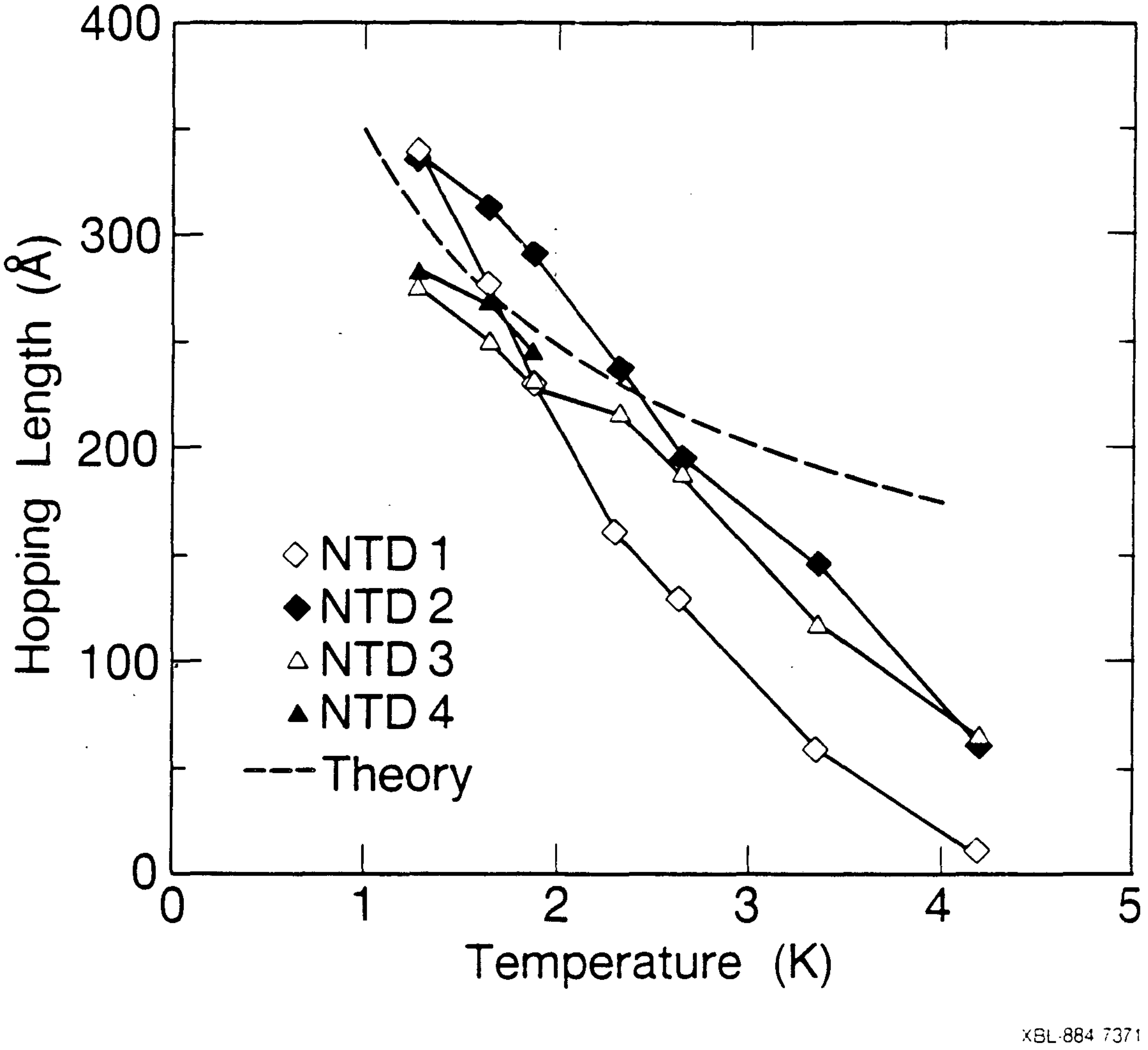
FIGURE 3



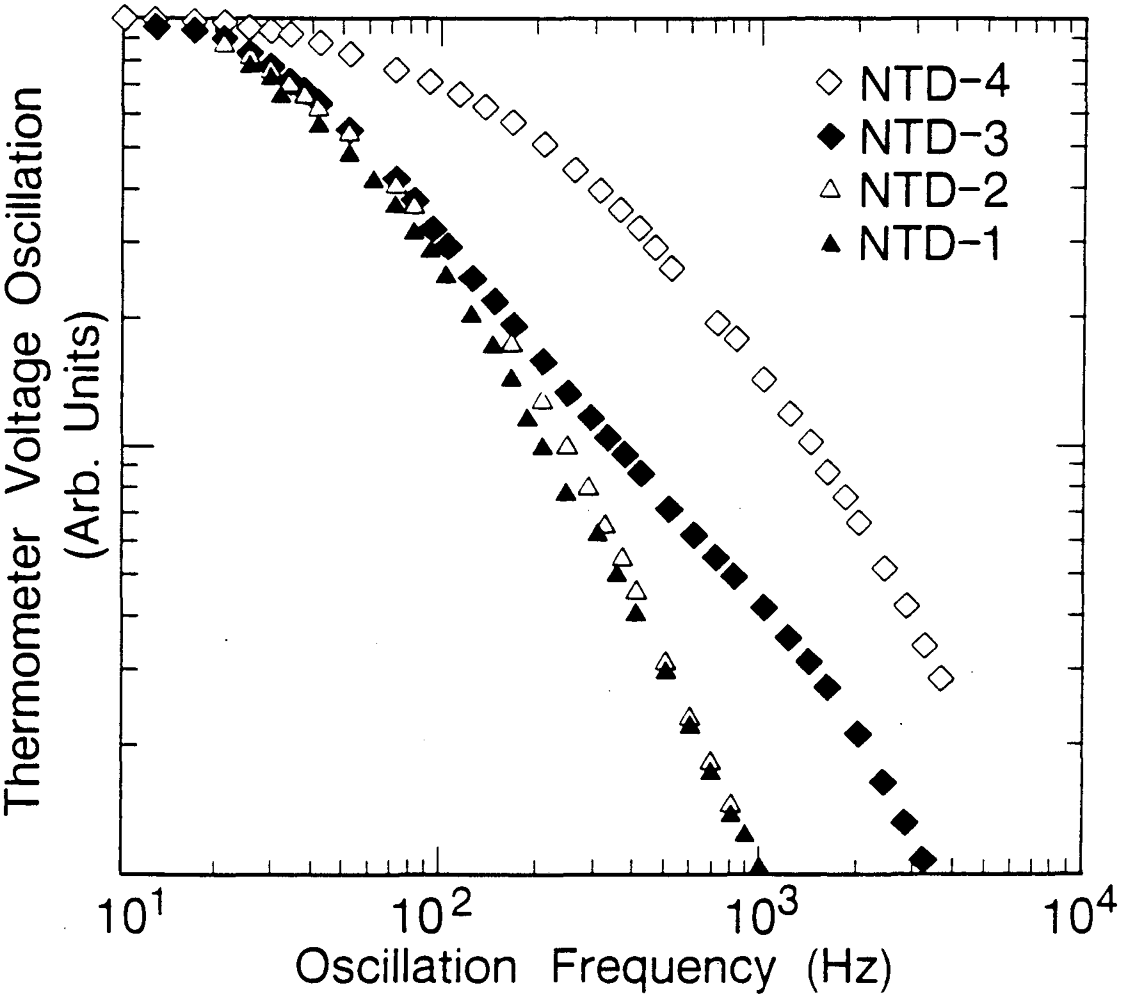
XBL 884-7367A

FIGURE 4



400
300
200
100
0
Hopping Length (Å)
0
1
2
3
4
5
Temperature (K)
◇ NTD 1
◆ NTD 2
△ NTD 3
▲ NTD 4
--- Theory
XBL 884 7371

FIGURE 5



Thermometer Voltage Oscillation
(Arb. Units)
◇ NTD-4
◆ NTD-3
△ NTD-2
▲ NTD-1
10^1
10^2
10^3
10^4
Oscillation Frequency (Hz)
XBL 884-7368

FIGURE 6

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