Veeco
Metrology Group

di SCANNING THERMAL MICROSCOPY (STHM) FOR DIGITAL INSTRUMENTS CP-II

Operation & Reference Guide

Part Number: 85-10336

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Preface

INTRODUCTION

The Scanning Thermal Microscopy (SThM) package for Digital Instruments CP-II can be used in two different operating modes: conductivity contrast mode and temperature contrast mode, allowing imaging of thermal conductivity and sample temperature, respectively.

The heart of the SThM package is a thermal probe with a resistive element. Thermal monitoring and control are carried out by the Thermal Control Unit (TCU).

In conductivity contrast mode, the thermal probe is kept at a constant temperature. Changes in sample thermal conductivity affect the heat flow between the self-heating probe and the sample. This heat flow is monitored by measuring the voltage necessary to keep the probe at a constant temperature.

In temperature contrast mode, temperature is monitored using a bridge circuit to measure the probe resistance.

Read these instructions carefully before installing the package. This manual assumes that you are already familiar with the operation of your Veeco instrument.

OPERATING SAFETY

The safety warnings and caution statements in your CP-II User's Guides are valid when using the Scanning Thermal Microscopy (SThM) package and should be read carefully and strictly observed.

Additional caution statements specific to the use of the SThM package appear throughout this manual and should be observed in order to prevent damage to the SThM thermal probes.

Chapter 1

SThM Package

Package Contents

The SThM package consists of the following:

- Box of SThM probes
- Thermal Control Unit (TCU)
- BNC cable to connect the Thermal Control Unit to the CP-II stage base or M5 Interface Module.
- BNC-type cable to connect the Thermal Control Unit to the thermal probe
- 15V Power supply
- Cable to connect the power supply to Thermal Control Unit
- Test sample for conductivity contrast imaging

Cable Connections

The instructions in this section describe how to connect the SThM components to your CP-II or M5 system. The connections are illustrated in Figure 1-1 and Figure 1-2. When connecting the SThM components, there is no need to turn off the power to the AEM.

Note that if you will be taking temperature contrast data, you may need to calibrate the probe tip prior to connecting the probe to the TCU (see *Probe Calibration* on pg. 2-5).

Step 1 Connect the ECU BNC connector on the rear panel of the TCU to either the **CN2** BNC connector on the M5 Interface Module or the **AUX 4** BNC connector on the CP-II base, using the supplied cable.

CAUTION: ***Care must be taken when handling the SThM thermal probes because the cantilever arms are easily bent, and the resistive filament is easily broken. Rough handling can bend the cantilever arms, requiring adjustment before proper laser beam alignment is possible.***

Step 2 Load the thermal probe on the probe cartridge and load the probe cartridge on the Digital Instruments CP-II head.

Step 3 Secure the probe cable to the Digital Instruments CP-II head with adhesive tape.

Step 4 Connect the **PROBE** BNC on the rear panel of the TCU to the thermal probe, using the supplied BNC-type cable.

Step 5 Connect the SThM power unit to the 15 V connector on the rear panel of the TCU, using the supplied cable.

NOTE: For optimal performance, place the power unit away from TCU; do not stack these components.

Step 6 Turn on the power to the SThM power unit.

Step 7 Turn on the power switch on the back of the TCU.

Step 8 If power to the AEM is off, turn on the power to the AEM by using the computer on/off switch on the rear panel of the computer unit.

Removing the SThM Package

To remove the SThM components, do the following:

Step 1 Set the **OPERATING MODE** switch to **STANDBY**.

Step 2 Turn off the power to the TCU.

Step 3 Turn off the power to the SThM power unit.

Step 4 Disconnect the BNC-type cable from the thermal probe.

Step 5 Disconnect the cable from either the **CN2** BNC connector on the M5 Interface Module or the **AUX 4** BNC connector on the CP-II base.

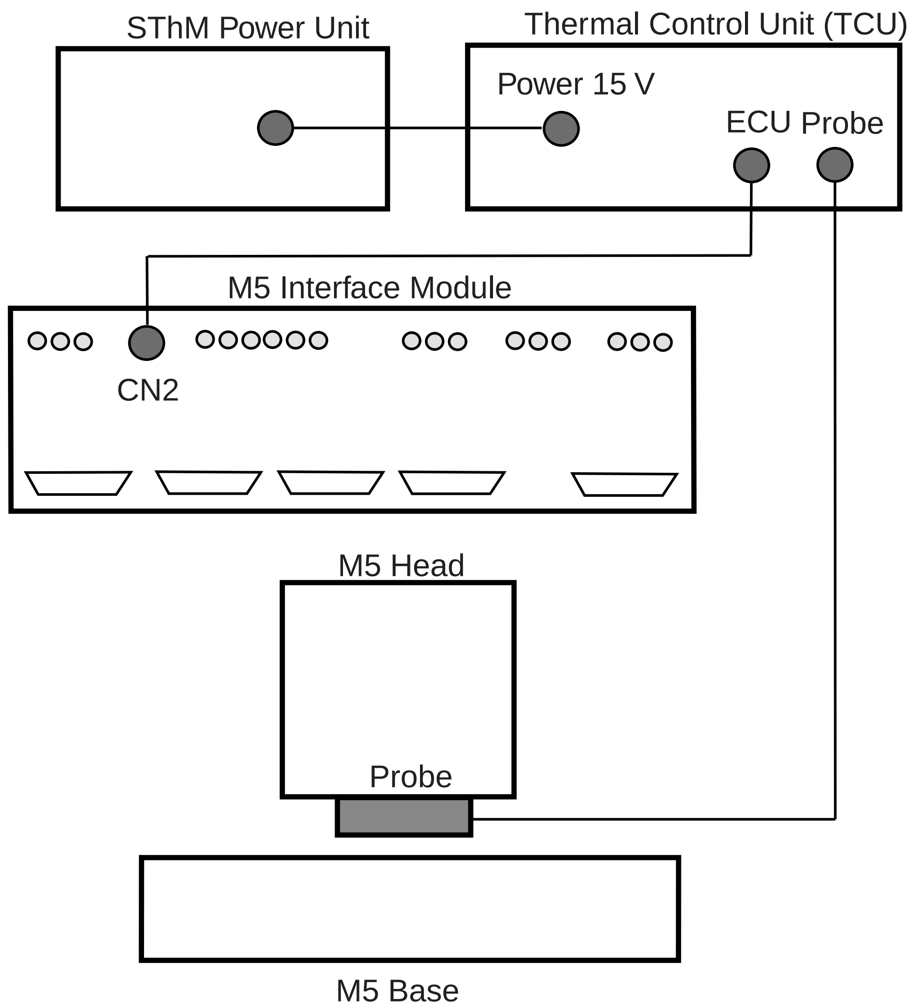

Figure 1-1. M5 Cable connections.

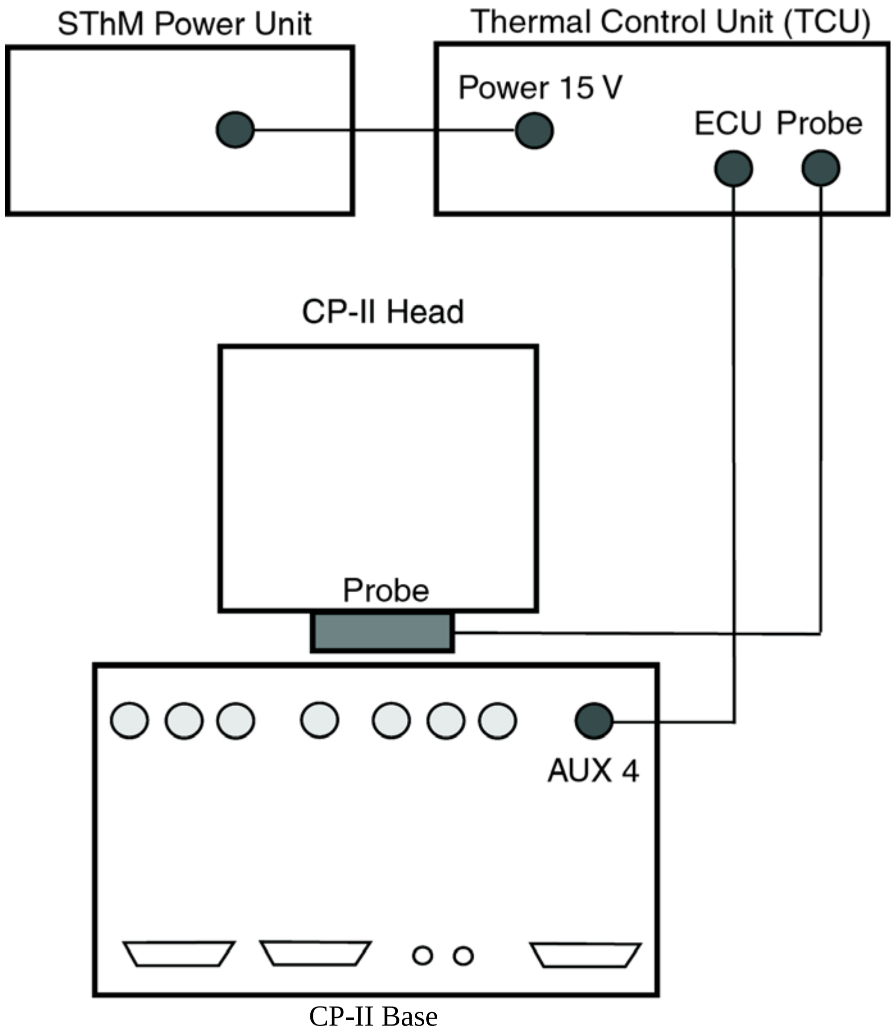

Figure 1-2. CP-II Cable connections.

SThM Probes

CAUTION: ***Thermal probes can be easily damaged or destroyed if the instructions in this manual are not followed carefully.***

The thermal probe used for SThM incorporates the resistive thermal element at the end of a cantilever (see Figure 1-3). The arms of the cantilever are made of a Wollaston wire (silver sheath with a Platinum/Rhodium core), 75 µm in diameter. The resistive element at the end, which forms the thermal probe, is made of a filament (5 µm diameter) of platinum or platinum/10% rhodium alloy. A mirror is cemented to the cantilever arms to reflect the laser to the photodetector. An epoxy bead is added near the end of the cantilever to minimize the risk of breaking the filament. The probe is mounted on a standard half washer which is in turn mounted on a cantilever chip carrier.

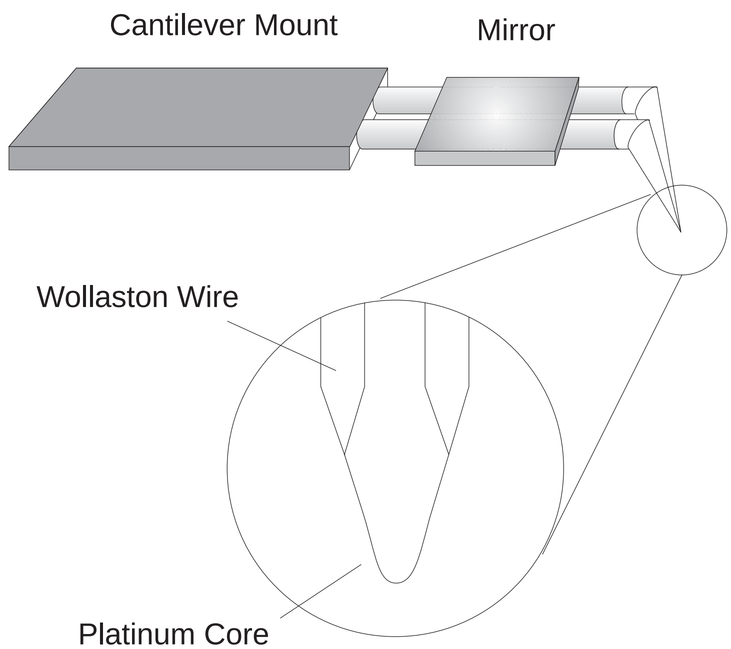

Figure 1-3. Thermal probe.

Thermal Control Unit

This section describes the controls and switches on the Thermal Control Unit (TCU). Operation of the TCU in the conductivity contrast and temperature contrast modes is described in further detail in Chapter 3 and Chapter 4, respectively.

Rear Panel

CAUTION: ***The* OPERATING MODE *switch should be in the* STANDBY *position, or the power switch should be turned to the OFF (0) position BEFORE interrupting the power to the power supply. Failure to do so while operating in conductivity contrast mode can destroy the SThM thermal probe.***

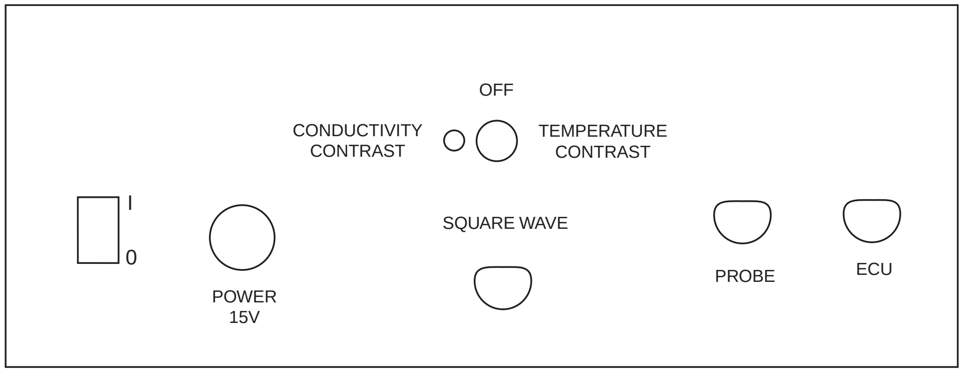

Figure 1-4.   Thermal control unit rear panel.

<u>POWER</u> The rocker switch on the far left controls power to the SThM TCU. The power is turned on by depressing the side marked "|." When the power is turned on, power is supplied to the temperature contrast and conductivity contrast circuits as well as the panel meter. When the power is on, and the **OPERATING MODE** switch is in the **STANDBY** position, the panel meter should read `0.000`.

<u>ECU</u> This BNC provides the output of the TCU. It is connected to either the M5 Interface Module or CP-Research base to collect thermal data. When the **OPERATING MODE** switch (see below) is in the **STANDBY** position, the ECU connector is grounded.

<u>PROBE</u> This BNC-type connector connects the TCU to the thermal probe. When the **OPERATING MODE** switch (see below) is in the **STANDBY** position, the **PROBE** connector is disconnected from the circuitry of the TCU.

<u>SQUARE WAVE</u> This toggle switch applies a square wave to either the conductivity contrast or the temperature contrast circuit. This signal is used to measure the frequency response of the probe in temperature contrast mode, and to measure and optimize the frequency response in conductivity contrast mode. The signal applied to the circuit (100 Hz, 150 mV p-p) is input at the BNC connector located below the **SQUARE WAVE** toggle switch. The toggle has a lock to prevent the signal from being accidentally applied to the circuitry.

Front Panel

CAUTION: ***The OPERATING MODE switch should be in the STANDBY position, or the power switch should be turned to the OFF (0) position before interrupting the power to the power supply. Failure to do so while operating in conductivity contrast mode can destroy the SThM thermal probe.***

<u>OPERATING MODE</u> This rotary switch controls the operating mode of the TCU. In the **STANDBY** position, the thermal probe is isolated from the TCU's circuitry. When **TEMPERATURE** is selected, the thermal probe, the Digital Instruments CP-II instrument, and the panel meter are connected to the temperature contrast circuit.

When **CONDUCTIVITY** is selected, the thermal probe, the M5 Interface Module or CP-II base, and the panel meter are connected to the conductivity contrast circuit.

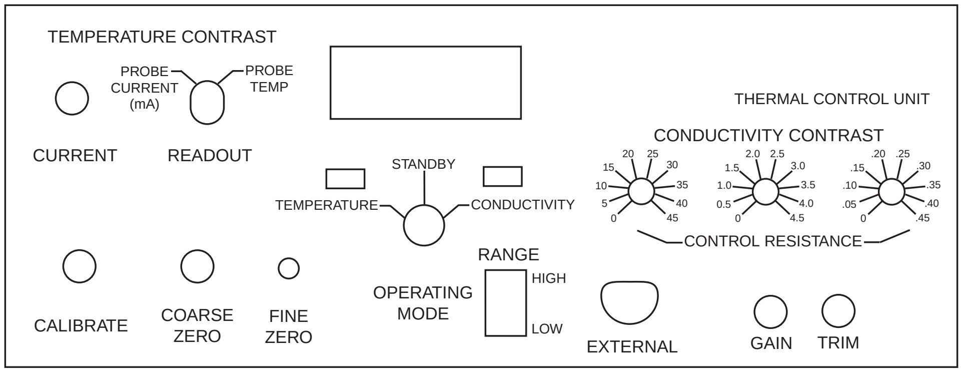

Figure 1-5. Thermal control unit front panel.

RANGE This switch toggles the panel meter range between **LOW** (2V max.) and **HIGH** (full scale). The range switch does not change the output to the M5 or CP-Research instrument.

CONTROLS IN CONDUCTIVITY CONTRAST MODE

The controls used for conductivity contrast mode are located on the right side of the TCU.

CAUTION: ***The* OPERATING MODE *switch should be in the* STANDBY *position, or the power switch should be turned to the OFF (0) position BEFORE interrupting the power to the power supply. Failure to do so while operating in conductivity contrast mode can destroy the SThM thermal probe.***

CONTROL RESISTANCE Note that the control resistance is five times (5X) greater than the desired probe resistance.

These three rotary switches select the operating temperature of the probe in conductivity contrast mode with a series of internal resistors. Three decades of resistors are available, which allow the control resistance to be varied from 0.00 to 49.95 Ω with a resolution of 0.05 Ω.

Due to the 1:5 bridge used in the conductivity contrast circuit, the **CONTROL RESISTANCE** value must be set to a resistance five times (5X) greater than the desired probe resistance.

EXTERNAL If the range of the internal control resistors is not great enough to achieve the 5:1 ratio, the control resistance can be supplemented with the use of an external resistor attached to the **EXTERNAL** BNC connector, located on the front panel. The external resistor is placed in series with the internal control resistors. When using an external resistor, the control resistance is the sum of the internal plus the external control resistance.

NOTE: The **EXTERNAL** BNC connector must have either a shorting cap or a resistor connected to it for proper operation in conductivity contrast mode.

GAIN AND TRIM These controls—which are used to optimize the frequency response of the conductivity contrast circuit—have been optimized in the factory. If you make adjustments to these controls, it will change the response time of the TCU. **GAIN** adjusts the gain of the conductivity contrast circuit's feedback loop. **TRIM** is a variable inductor.

Controls in Temperature Contrast Mode

The controls used for temperature contrast mode are located on the left side of the TCU.

READOUT This switch selects which circuit output (probe current or probe temperature) is displayed on the panel meter. This switch is only active in temperature contrast mode and does not affect the signal recorded by the Digital Instruments CP-II electronics (AEM). The **CURRENT** setting of the **READOUT** is used when verifying or changing the probe current.

CURRENT The current to the probe can be adjusted with this 10-turn potentiometer. The probe current can be adjusted from 0.1 to 2.0 mA. The current can be monitored with the panel meter by turning the **READOUT** switch to the **CURRENT** position. Once the probe current is set at the desired level, the lock on the multi-turn counter can be used to hold the position.

CALIBRATE The gain of the probe monitoring portion of the temperature contrast circuit can be adjusted with this 10-turn potentiometer. The circuit gain can be adjusted from 20 to 1000. The gain is set when calibrating the thermal probes. The circuit gain also controls the dynamic range of the thermal probe. Once the circuit gain is set at the desired level, the lock on the multi-turn counter can be used to hold the position.

COARSE ZERO This control adjusts a 10-turn potentiometer and is used with the **FINE ZERO** control to balance the bridge of the temperature contrast circuit. The **COARSE ZERO** is set when calibrating the thermal probes (see *Probe Calibration* on pg. 2-5). Once the **COARSE ZERO** is set at the desired level, the lock on the multi-turn counter can be used to hold the position.

FINE ZERO This control adjusts a multi-turn potentiometer and is used with the **COARSE ZERO** control to balance the bridge of the temperature contrast circuit. The **FINE ZERO** is set when calibrating the thermal probes (see *Probe Calibration* on pg. 2-5). Once the **FINE ZERO** is set at the desired level, the lock on the multi-turn counter can be used to hold the position.

NOTE: The **FINE ZERO** potentiometer does not have any stops, but it will idle when it reaches its limits.

Chapter 2

Operating Principles

Conductivity Contrast Mode

In conductivity contrast mode, the resistive element of the thermal probe is used as a resistive heater. The self-heated thermal probe is one of the legs of a Wheatstone bridge (see Figure 2-1).

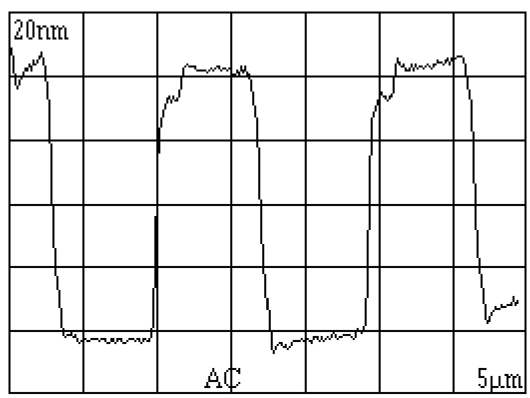

Figure 2-1. Simplified conductivity contrast circuit.

In conductivity contrast mode, the control circuit uses a feedback loop to adjust the voltage applied to the bridge in order to keep the probe at a constant temperature. As the self-heated probe scans the sample surface, heat flows from the probe to the sample. Changes in the thermal conductivity of the sample will affect the flow of heat to the sample. In the absence of the thermal feedback circuit, a change in the flow of heat to the sample would cause the temperature of the probe to change, altering its resistance and shifting the balance of the bridge. The feedback loop senses this shift in the bridge balance, adjusting the voltage applied to the bridge to

control the heating of the thermal probe and maintain a constant resistance. The probe temperature therefore remains constant because the feedback loop keeps the probe resistance constant.

Changes in the heat flow between the probe and the sample are measured by monitoring the bridge voltage. This flow of heat is controlled by the following three factors:

- Thermal conductivity of the sample
- Temperature difference between the probe and the sample
- Contact area of the probe

Changes in any of these variables will create contrast in the thermal image, changing the heat flow to the sample and altering the amount of heating necessary to keep the probe at a constant temperature. If the sample is smooth (the contact area of the probe remains constant) and the sample is at a constant temperature (the probe temperature remains constant), contrast in the thermal image will only be caused by changes in thermal conductivity of the sample.

Frequency response in conductivity contrast mode is limited by the response time of the feedback circuit in the TCU. With proper adjustment of the **GAIN** and **TRIM** controls (these controls are optimized at the factory), this response time is approximately 25 µs. Faster response times are possible than in temperature contrast mode (350 µs) because it is not necessary to establish a new temperature equilibrium within the probe.

The temperature of the probe as a function of its resistance is given by:

$$T = T_0 + \frac{R_P - R_0}{R_0 \alpha} \qquad \text{(Equation 1)}$$

where: T = the probe temperature

T0 = the reference temperature

R0 = the probe resistance at the reference temperature T0

RP = the probe resistance at T

a = the temp. coefficient of resistance of the probe material (see Table 1)

TABLE 1

Probe Material	Properties of Probe Material	
	TEMPERATURE COEFFICIENTS OF RESISTANCE (Ω / Ω -°C)	RESISTIVITY (Ω /CM)
PLATINUM/10% RHODIUM	0.00165	18.9

Rearrangement of Equation 1 gives the probe resistance as a function of its temperature:

$$R_P = R_0[(T - T_0)\alpha + 1] \qquad \text{(Equation 2)}$$

The thermal feedback circuit keeps the bridge balanced. The operating temperature of the thermal probe is controlled by the control resistor (R_C) of the bridge circuit.

Because of the 1:5 bridge ratio (R1=20 Ω, R2=100 Ω), the bridge is balanced when the control resistance is five times greater than the probe resistance. Selection of the proper control resistance (R_C) for a desired probe temperature is given by:

$$R_C = 5R_P = 5R_0[(T - T_0)\alpha + 1] \qquad \text{(Equation 3)}$$

LEAD RESISTANCE

Because of the sensitivity of the bridge circuit and the low resistance of the thermal probes, the resistance of leads (R_L) and internal resistance in the TCU should not be ignored. The procedure for determining the control resistance necessary to compensate for the lead resistance is given on page 3-3.

$$R_C = 5R_P + R_L = 5R_0[(T - T_0)\alpha + 1] + R_L \qquad \text{(Equation 4)}$$

The value of the lead resistance should be 0.6-0.9 Ω. The actual lead resistance is 20% of this value, due to the 1:5 bridge ratio. The lead resistance should be added to the control resistance (of Equation 3) to determine the correct control resistance (R_C):

CONTROL RESISTANCE

CAUTION: ***To avoid "burning up" thermal probes, step to the maximum value of the 0.5 Ω control resistors (4.5 Ω) and then set it back to 0 BEFORE advancing to the next 5 Ω control resistor. Also, when operating the probe at elevated temperatures, bring the thermal probe up to its operating temperature by increasing the control resistance to the desired setting while in conductivity contrast mode.***

The control resistance is selected using the switches on the right side of the TCU's front panel (see the procedure under *Setting the Control Resistance* on pg. 3-3). These controls provide a resistance range over three decades; up to 49.95 Ω, in 0.05 Ω increments. This minimum 0.05 Ω increment of the control resistance defines the minimum change in operating resistance of the thermal probe as 0.01 Ω (due to the 1:5 bridge ratio). For a platinum/10% rhodium probe with a room temperature resistance of 2.5 Ω, the minimum increment in the operating temperature of the thermal probe can be found using Equation 1:

$$\Delta T = \frac{R_P - R_0}{R_0\alpha} = \frac{0.01 \quad \Omega}{2.5 \times 0.00165[\Omega][\Omega/(\Omega - °C)]} \qquad \text{(Equation 5)}$$

The control resistance can be adjusted while the conductivity contrast circuit is active. However, care must be taken when changing the control resistance of an active conductivity contrast circuit. This is especially true when changing the 5 Ω control resistors. Increasing the R_C by 5 Ω will change the temperature of a 2.5 ohm platinum/10% rhodium thermal probe by 250°C! (Equation 1).

Temperature Contrast Mode

In temperature contrast mode, the thermal probe is used as a resistance thermometer. Changes in the probe temperature cause the probe's resistance to change. As in conductivity contrast mode, the thermal probe is one of the legs of a Wheatstone bridge (see Figure 2-2). A constant current passes through the bridge. As the temperature of the probe changes, the corresponding change in probe resistance (R_P) will change the voltage balance of the bridge, changing the output voltage (V_{Out}) of the circuit.

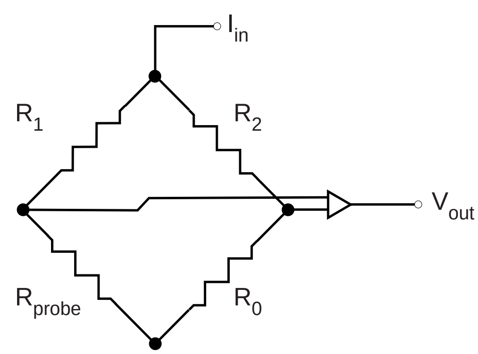

Figure 2-2. Simplified temperature contrast circuit.

The controls for the temperature contrast circuit allow the current applied to the bridge to be adjusted and the output amplification to be varied. These controls allow the output of the circuit to be adjusted so it can be read directly as temperature. The current passed through the probe in temperature contrast mode is set to be small enough that no self-heating of the probe occurs. (Note that self-heating of the probe could cause errors in temperature measurement.)

The frequency response in temperature contrast mode is limited by the response time of the resistive element of the thermal probe: approximately 350 µs. This response time is limited by the time necessary to establish a new thermal equilibrium in the thermal probe.

The output of the temperature contrast circuit is proportional to the probe resistance. This output can be adjusted by using the **CALIBRATE**, **CURRENT**, **COARSE ZERO**, and **FINE ZERO** controls, located on the front panel of the TCU (described above). The relationship between circuit output and probe temperature is given by:

$$V_{Out} = (R_0 \alpha C_i)(T - T_0) + V_0 \quad \text{(Equation 6)}$$

where: V_{Out} = temperature contrast circuit,

R_0 = resistance of the thermal probe at the reference temperature T0

α = temperature coefficient of resistance of the probe material

C = calibration gain

i = current applied to the probe

T = probe temperature

T_0 = probe resistance at the reference temperature

V_0 = bridge voltage at the reference temperature

In practice, the calibration of the system can be reduced to:

$$T - T_0 = S(V_{Out} - V_0) \qquad \text{(Equation 7)}$$

where: S = slope = $(1/ R_0 \alpha Ci)$

SETTING THE BRIDGE CURRENT

The current through the probe can be adjusted with the **CURRENT** potentiometer found on the front panel of the TCU. The current applied to the bridge circuit can be monitored with the panel meter by turning the **READOUT** switch to the **CURRENT** position. The current is displayed in milliamps (mA). The **READOUT** switch only affects the signal to the panel meter; the **ECU** connector on the rear panel of the TCU remains connected to the temperature contrast circuit. The selection of the operating levels of the **CALIBRATE** and **CURRENT** controls will be a balance between the sensitivity desired and the dynamic range required. It should be remembered that adjustment of these controls also affects the signal-to-noise ratio of the temperature contrast circuit.

NOTE: The response time of the thermal probe in the temperature contrast mode is approximately 350 µs.

LEAD RESISTANCE

The resistance of the leads and other contact resistance have essentially no effect in temperature contrast mode since they are compensated for when balancing the bridge with the **COARSE ZERO** and **FINE ZERO** controls.

PROBE CALIBRATION

It is necessary to calibrate the absolute temperature of the thermal probe tip, which requires holding the probe at two different temperatures. Because the output is linear, i.e., the resistance change in the tip is proportional to the change in sample temperature, only two points, T_1 and T_2, are needed to calibrate the probe tip. A suggested calibration procedure is given on page 4-1.

Using Equations 5 and 6, the slope can be calculated by:

$$S = \frac{T_1 - T_2}{V_1 - V_2} \qquad \text{(Equation 8)}$$

Where V_1 and V_2 are the corresponding output voltages at T_1 and T_2. At T_2 the output voltage is set to 0 by adjusting the **COARSE ZERO** and **FINE ZERO** potentiometers (see page 1-8). Therefore the probe temperature can be calculated by:

$$T - T_2 = SV_{Out} \qquad \text{(Equation 9)}$$

Chapter 3

Taking a Conductivity Contrast Image

CAUTION: ***The* OPERATING MODE *switch should be in the* STANDBY *position, or the power switch should be turned to the OFF (0) position BEFORE interrupting the power to the power supply. Failure to do so while operating in conductivity contrast mode can destroy the SThM thermal probe.***

CAUTION: ***As a precaution against burning out thermal probes, it is recommended that you turn the* OPERATING MODE *switch to the* STANDBY *position each time you adjust any of the control resistance knobs.***

Determining the Lead Resistance

First you need to set the lead resistance:

Step 1 Attach a shorting plug to the probe cable.

Step 2 Set the **CONTROL RESISTANCE** to 0.00 Ω.

Step 3 Turn the **OPERATING MODE** switch on the TCU to **CONDUCTIVITY**.

Step 4 Increase the control resistance value until the bridge voltage increases.

Step 5 The value of the control resistance at this point balances the lead resistance.

The value of the lead resistance should be 0.6-0.9 Ω (see *Lead Resistance* on pg. 2-5 for more details).

SETTING UP TO TAKE AN IMAGE

To take a conductivity contrast image, you first need to set up your Digital Instruments CP-II instrument for AFM imaging. Most of the following steps are described in greater detail in your Digital Instruments CP-II User's Guide:

Step 1 Load the shipped test sample on the sample holder.

Step 2 Position the probe tip over an area of the sample where the grating is.

Step 3 Steer the laser beam onto the cantilever mirror.

Step 4 Align the laser spot on the PSPD.

Step 5 Add the thermal signal channel, with a low-pass filter, to the input configuration.

- a) Open the Input Configuration dialog box by selecting **SETUP→INPUT CONFIGURATION**, or click the Input Config icon.
- b) For M5: Select **CN2** from the Available listbox, and then click [Add→].

  For CP-II: Select **AUX4** from the Available listbox, and then click [Add→].
- c) Set the low-pass filter to 1.
- d) Make sure that the Topography channel is selected, and click [OK] to close the dialog box.

Step 6 Perform an approach.

Step 7 Set the scan size to 35 µm.

Step 8 Set the scan rate to 0.5 Hz.

Step 9 Set the gain to 4.

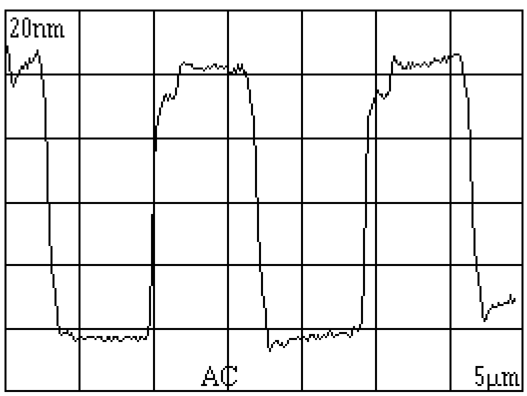

Figure 3-1. Topography signal of test sample grating

Step 10 View the Topography signal on the oscilloscope. It should be a square wave as shown in Figure 3-1. If you do not see this pattern, move the tip to another part of the sample until you do. To move the tip, either use the Offset function, or use the z direction pad to lift the tip and then move to a new location on the sample. Then approach the sample again.

SETTING THE CONTROL RESISTANCE

CAUTION: ***To avoid "burning up" thermal probes, step to the maximum value of the 0.5 Ω control resistors (4.5 Ω) and then set it back to 0 BEFORE advancing to the next 5 Ω control resistor. Also, when operating the probe at elevated temperatures, bring the thermal probe up to its operating temperature by increasing the control resistance to the desired setting while in conductivity contrast mode.***

CAUTION: ***As a precaution against burning out thermal probes, it is recommended that you turn the*** **OPERATING MODE** ***switch to the*** **STANDBY** ***position each time you adjust any of the control resistance knobs.***

Step 1 Set the oscilloscope to display the thermal signal:

For M5: Select **CN2**.

For CP-II: Select **AUX4**.

Step 2 Turn the **OPERATING MODE** switch on the TCU to **CONDUCTIVITY**, connecting the thermal probe to the conductivity contrast circuit. The red LED above **CONDUCTIVITY** indicates that the thermal probe is connected to the conductivity contrast circuit and the circuit is active.

Step 3 Heat the probe tip in order to see conductivity contrast in the sample by first observing the panel meter on the TCU and slowly increasing the control resistance by starting with the middle knob. Heating of the probe is indicated by a change in value on the panel meter.

Step 4 When you see a change in value on the panel meter, indicating that the probe is heating up, observe the oscilloscope trace and increase the resistance until the pattern is like the one shown in Figure 3-2. If you step to the maximum value (4.5 Ω) and still do not see this pattern, turn the knob all the way back down to 0.

Step 5 Now use the left-hand knob to bring the resistance up to 5.0 Ω.

Step 6 Continue with the middle knob by stepping up 0.5 Ω, for a total of 5.5 Ω.

Step 7 Bring the middle knob up slowly until the oscilloscope trace is like the one shown in Figure 3-2. Typically this should happen between 13 Ω and 15 Ω. At this point, the panel meter should read about 0.8 V.

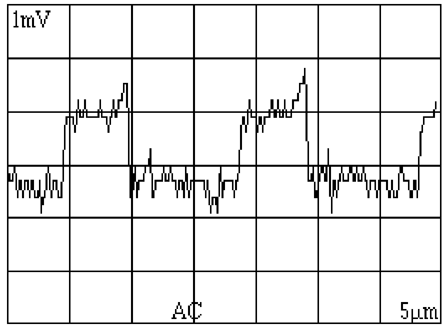

Figure 3-2. Thermal signal of test sample grating

Now you are ready to take a conductivity contrast image.

Diagnostic Voltages in Conductivity Contrast Mode

When the thermal probe resistance is greater than the balance point of the Wheatstone bridge, the bridge voltage is approximately 0.5 V. At this value, the temperature of the probe is not yet self-heating. If the probe is not connected to the TCU, or if the probe filament is broken, the bridge voltage is approximately 0.1 V. See *Control Resistance* on pg. 2-3.

Chapter 4

Using Temperature Contrast Mode

In order to use temperature contrast mode, you will need a means for calibrating the absolute temperature of the probe tip, and a means to heat (or cool) your sample.

Probe Calibration

Calibration of the probe requires holding the probe at two different temperatures (see *Probe Calibration* on pg. 2-5). Below is one possible method of calibrating thermal probes:

Step 1 Set the temperatures of two chambers, one at 0°C and the other at 100°C.

Step 2 Place the probe in the 0°C chamber.

Step 3 Adjust the **COARSE ZERO** potentiometer and the **FINE ZERO** potentiometer until the panel meter reads 0.000 volts.

Step 4 Place the probe in the 100°C chamber. Adjust the **CALIBRATE** potentiometer until the panel meter reads 10.00 V.

NOTE: This procedure should be repeated to ensure accuracy.

Heating the Sample

Two possible ways to heat your sample would be: to use a Peltier element, or to heat it resistively by running a current through it. If the desired sample temperature is at or below 0°C, or at or at elevated temperatures (≥100°C), you need to provide a water-free environment to insure that no ice formation occurs on the sample

surface or that there is no unwanted reaction. Typically, this is accomplished by placing the microscope in a glove box and then purging the glove box with N_2 or Ar, for example.

Setting Up to Take Data

Once the probe has been calibrated and you have set up a means for heating the sample, you need to set up your Digital Instruments CP-II instrument for AFM imaging. Most of the following steps are described in greater detail in your Digital Instruments CP-II User's Guide:

Step 1 Load your sample on the sample holder.

Step 2 Position the probe tip over an area of the sample where you want to take data.

Step 3 Steer the laser beam onto the cantilever mirror.

Step 4 Align the laser spot on the PSPD.

Step 5 Add the thermal signal channel to the input configuration.

a) Open the Input Configuration dialog box by selecting **SETUP→INPUT CONFIGURATION**, or click the Input Config icon.

b) For M5: Select **CN2** from the Available listbox, and then click [Add→].

For CPII: Select **AUX4** from the Available listbox, and then click [Add→].

c) Make sure that the Topography channel is selected, and click [OK] to close the dialog box.

Step 6 Perform an approach.

Step 7 Set the scan size.

Step 8 Set the scan rate to 1 Hz or slower. This range is necessary due to the limited response time (350 µs) of the resistive element of the thermal probe.

Taking Temperature Contrast Data

Step 1 Turn the **OPERATING MODE** switch to **TEMPERATURE**, connecting the thermal probe to the temperature contrast circuit. The red LED above **TEMPERATURE** indicates that the thermal probe is connected to the constant current circuit, and that the circuit is active.

Step 2 Heat the sample to the desired temperature.

Now you are ready to take a temperature contrast data.