Pyrometry Lab

Objective: The objective of this laboratory is for you to explore the physics and practical aspects of non-contact temperature measurement by infrared pyrometry.

Preparation

- Read: Speyer Chapter 8 "Heat Transfer and Pyrometry" on Compass
- Review the theory of black body radiation and Boltzmann's law
- Look up reference values as follows:

Melting temperature of Stainless Steel:________________

Melting temperature of Graphite:____________________

Emissivity of various types of graphite (list graphite types and sources):________________

Emissivity of various types of stainless steel (list types and sources):____________________

Emissivity of rough material (you choose the material):______________________________

Emissivity of smooth material (same material as chosen for rough material):______________

Equipment and Samples

- Optical pyrometry station: small tube furnace, chopper, pyroelectric detector
- Sample cylinders made of stainless steel and graphite. Both are 3" long and have 0.9" outer diameter. The graphite tubes have a hole on one end (with inner diameter 0.6"), and are closed on the other end. The stainless steel rods have a polished end and a rough end.
- Lock-in amplifier and control software

Laboratory Safety:

Required Personal Protective Equipment

- Safety glasses with side shields
- Long pants
- Closed toed shoes
- Heat resistant gloves - (will be provided in the lab)

Safety Concerns

- High temperatures and hot materials - potential for burns and fire.

    - Samples will be tested at extremely high temperatures in a furnace capable of reaching 1000° C. Severe burns to skin can be a result of touching hot surfaces or samples. High temperature gloves should be worn when touching any part of the furnace or sample during or immediately after a sample run.
    - Heat resistant tongs should be used when handling samples to prevent burns to exposed skin.
    - Hot samples should be placed in heat resistant trays away from any combustible material. This will reduce the risk of starting a fire.
    - All combustible materials should be kept away from the experimental set up. This will reduce the risk of starting a fire.
- Laser beam - potential for eye damage
    - A small laser will be used to align the components of the experiment. Take care that the laser in not pointed in the direction of anyone. This will reduce the risk of eye injury that can be caused by this laser.

Waste Considerations

- No waste is generated in this experiment.

Experimental Procedures:

Introduction

Most materials processing occurs at elevated temperatures and accurate knowledge of the temperature is extremely important. Placing a thermocouple or other physical thermometer in contact with the material is often inconvenient or impossible. Measurements of the intensity and spectrum of electromagnetic radiation emitted by a material are often the only option for thermometry but accurate calibration of the pyrometer and accurate knowledge of the emissivity of materials is often challenging.

In this lab, infrared radiation emitted from a hot surface will be detected by an IR detector. The detector is made of a special type of dielectric crystal, a pyroelectric, that generates a voltage when heated.

Session 1: Calibrating an optical pyrometer Apparatus

Setup:

Before the experiment can begin, the apparatus must be carefully set up in correct alignment. Proper setup is vital in order to get good data.

- The tube furnace should be positioned against the side of the optical table. Mark or fix the position of the furnace so that it does not move.
- Mount the chopper and pyroelectric detector on the optical table. The chopper should be between the furnace and the detector and must be a minimum of 10cm away from the furnace to prevent it from absorbing heat during the experiment.

- The detector should be positioned after the chopper. Note the distances between the apparatus elements. You must maintain the same distances in the setup throughout the entire experiment.
- Attach the chopper and the detector to the optical table with screws. Hand tighten only.
- Ensure that the furnace, chopper, and detector are lined up both horizontally and vertically. Look through the center of furnace hole. You should see the chopper and detector centered in the field of view. Alignment can also be checked using a laser pointer.
- Once the positioning and alignment is correct, proceed to the calibration step.

Calibration:

The chopper frequency and the lock-in amplifier settings are the two most important elements in getting a good, clean signal from the detector. The lock-in amplifier itself is a complicated instrument but the purpose it serves is simple: it allows us to pick a very small amplitude signal at a known, single frequency (such as the chopped sample signal emitted from the furnace), out from a much higher-amplitude, noisy multi-frequency background such as all the *other* possible sources of light the detector can see- the fluorescent lights, the sunlight outside, the glow of the computer screen, etc.

In order to utilize the chopper and lock-in amplifier to make such a sensitive measurement in such a noisy environment, a calibration run must be done for reference. We will calibrate the setup in two stages: one run that explores the effect of the chopper frequency on the detector signal with the sample at a fixed temperature and another that explores the effect of different sample temperatures on the detector signal with the chopper set at a fixed frequency. Both of these calibration runs are done using the graphite sample with the well side facing the detector. This is our reference 'black-body'.

- Run the chopper frequency calibration first. Once the furnace temperature has stabilized, use the chopper controller to change the frequency of the blades. Frequencies that are available to us are between 10Hz and 110Hz. Choose about 10-12 frequencies to test and record the sample temperature, frequency, and lock-in reading for each chopper frequency tested. Auto zero the lock-in amplifier after each frequency change but there is no need to wait to record a reading (provided it is stable!).
    - Plot the data as it is collected and examine the trend. What is the expected relationship between frequency of the chopper and the voltage read by the detector?

- Next, based on your knowledge of black body radiation and information about the equipment, determine an appropriate temperature range and temperature intervals to investigate. Heat up the sample and collect data as you vary the temperature. This portion of the calibration heats up the sample and determines the luminescence measured by the detector as a function of temperature. Assume the detector voltage read via the lock- in amplifier is linearly proportional to luminescence on the detector.
    - Set the furnace to the appropriate temperature and wait for the temperature to equilibrate. It is vital that the temperature of the sample and the furnace be stable when the voltage measurement is performed. For this reason, it is good to wait at

least 5-10 minutes after the display on the furnace reaches the desired temperature before any readings are done. When the furnace temperature is stable and enough time has passed for it to equilibrate, auto-zero the lock-in amplifier and record the voltage and the furnace temperature. Repeat for frequencies between 10 and 110 Hz, as before.

- Repeat this process for the other experimental temperatures chosen. In your report make a plot of luminescence vs. furnace temperature and fit the result. Include in your report the function that fits your data and compare the result with the Stefan Boltzmann law. Include an analysis of the errors in your measurement and include an uncertainty in the form of an error value.
- From your data for the graphite well, select an appropriate chopper frequency to use for the remainder of the experiment. Be sure to justify your selection.

Session 2: The black body spectrum and emissivity

Now that the pyrometry setup is calibrated and a reference black-body spectrum has been measured, it is possible to explore the effects of different sample materials such as graphite and stainless steel, as well as different sample surfaces - smooth and rough stainless steel and flat-sided graphite with no well. The effect of the different samples and different surfaces is captured by the emissivity, ε.

- First, replace the graphite well-sided sample with a stainless steel rod (oriented with the polished surface facing the detector) and placed 1" into the furnace as before. Determine the emissivity, ε, as a function of temperature by assuming that the temperature displayed on the furnace is accurate (after the appropriate equilibration time), and that the emissivity is the proportionality coefficient between the luminosity and the sample temperature. The data is collected in an identical manner to the procedure in the calibration section; with the only difference being which sample is in the furnace. It is a good idea to use the same chopper frequency for all of the samples and temperatures measured.
- Repeat with the rough stainless steel surface and the flat-sided graphite surface. In your report, if there are changes in emissivity as a function of temperature, plot these values against each other. If appropriate, fit the data and report the functional form. Consider how sensitive this value is to the alignment of the optical system. When you analyze your result you will need to account for the blackbody radiation intensity variation that you would expect for the given temperature change.
- Explain why differences may be observed between the rough and smooth surfaces, and between the two different materials (graphite and stainless steel).

Instrument procedures

Optical chopper

The SDC-5000 optical chopper creates pulses of infrared radiation from a continuous emission source. This is essential for the operation of the pyroelectric detector. The chopper operates by rotating fan blades at a desired speed; the frequency range is 4 to 500 Hz for the **Low Freq** setting (which uses the inner aperture, closer to the center) and 40 to 5000 Hz for the **High Freq** setting (which requires outer aperture, further away from the center). The outer aperture has finer fan blade spacing to achieve the higher chopping frequency.

To operate the chopper, turn on the power switch (at the back, next to power cable) and check the controller to make sure **Int** mode is active. Choose an aperture and select the corresponding speed mode. Press and release the frequency set knob once and the least significant digit on the clock will blink. Turning the knob clockwise increases that digit by one for every click. Pressing and releasing the knob once again will cause the next least significant digit to blink. Continue and adjust each digit as necessary. Once you reach the most significant digit allowed, pressing the knob one more time will set the motor to the speed displayed and also store the value in memory. The Sync Output should be connected to the oscilloscope to provide the trigger.

Detector and oscilloscope

We use the SPH-CM-Test pyroelectric detector to measure the power of radiation emitted. The heart of the detector test box is a 5 mm diameter $LiTaO_3$ detector. The detector generates a voltage signal by detecting a temperature change due to incoming radiation with linear response up to 40,000 V/W. The detector test box has a BaF_2 window to block airflow that is transparent to visible and IR radiation (up to 17.5 μm).

To collect the data, we use a DS1M12 oscilloscope. Turn on the detector and start the EasyScope II program. You want to trigger **ChB** with the sync output of the chopper. Adjust the T/Div and V/Div knobs to see a clear image of the signal. Adjust **Gnd** level if necessary. Pressing **Meter A** button will display useful readings, which you can customize by pressing configure.

Furnace

The Carbolite MTF 10 furnace is used to heat up the test samples to the desired temperatures. All of the samples are 3" long rods, and some have a hole on one end. To ensure uniform temperature, you want to place the sample at the center of the heating tube, except when measuring emissivity. Before you start the heating, align the furnace with the lens/chopper/detector. Then, turn on the furnace with the green switch, press the button once so it displays SPoC (sample temperature), press the up and down arrow buttons to select the desired temperature, then press three more times to set the temperature. The LCD now displays the current temperature of the heating tube. To start the heating, flip the orange switch below the green power switch. The orange light will turn on indicating that the heating coil is on. The temperature will reach the set limit fairly quickly, and overshoot (amount of over-shoot depends on heating rate) then come down to help the system to stabilize. The sample inside the furnace will take 10 to 15 minutes to stabilize; use the oscilloscope reading as a guide. To change the heating rate, press the button twice so it displays SPrr (ramp rate) and then use the arrows to adjust, similar to setting the temperature.

Note: Cooling the furnace down takes a long time. Using a fan will help, but not by much. Therefore, it is best to choose appropriate temperature steps and constantly increase the temperature during the experiment. Also, be gentle when changing temperature, as pushing the buttons too hard might shift the furnace and ruin the alignment.