Phase equilibria

Objective

The objective of this laboratory is for you to observe in real-time the microstructures that form during the solidification of a two-component alloy with a eutectic phase diagram.

Preparation

Review sections of the textbook you used for MSE 182 or 201 that discusses eutectic phase diagrams and the microstructures that form during solidification. A few pages from the introductory book by Smith are posted at the Compass site. Read the introduction to the 1981 paper by Glicksman. The phase diagram of SCN-camphor is given in the 2004 article by Witusiewicz that is posted at the Compass site.

Equipment and samples

- Optical microscope, Linkham hot-stage.
- Succinonitrile (SCN) and camphor for making transparent organic alloys; hot plate; microscope slides and cover slips.
- Computer, video camera, image acquisition and analysis software, software for controlling the hot-stage.

Introduction

Understanding and controlling the microstructures formed during the solidification of multi-component alloys is one of the foundational concepts of materials science and engineering. Essentially all metal alloys owe their strength to the microstructures that are created during solidification and heat treating. Details vary between systems but many aspects of the solidification of alloys are common to metals, ceramics, semiconductors, and small-molecule organics. In this lab, you will use a "transparent organic alloy" of succinonitrile (SCN) and camphor as a model system to observe the formation of microstructures in real-time in an optical microscope. SCN-camphor has a eutectic phase diagram with extremely small solubility in the solid state.

Sessions 1 and 2: Thermodynamics, kinetics and microstructures of two-component "transparent organic alloys"

- This lab is more open-ended than the other labs in MSE 307. We encourage you to explore whatever aspects of phase equilibria and microstructures of two-component alloys strikes your interest. Some ideas:
    - Using pure SCN, measure the super-cooling of liquid as a function the cooling rate. Upon heating, does the crystal ever superheat?

- Add small amounts of camphor to SCN and observe the microstructures that form during solidification. Is the shape of the dendrites sensitive to the composition? The radius of the tip of the dendrite and the spacing between side branches are two possible lengths to measure.

- Add small amounts of camphor to SCN and measure the volume fractions of the phases. Are the volume fractions consistent with the assumption that the solubility in the solid-date is negligible?

- Make a eutectic composition and observe the solidification at different cooling rates. How does the characteristic length scale of the eutectic microstructure vary with cooling rate? If you reheat the alloy and hold the temperature of the sample just below the eutectic temperature, can you observe a coarsening of the microstructure? If so, how does the characteristic length scale vary as a function of time?

- Vary the composition by small amounts on either side of the eutectic (hypo-eutectic and hyper-eutectic compositions) and relate the microstructures you observe to the composition.

Instrument procedures

Reflected Light Microscope

This experiment utilizes a reflected light microscope. We will be using the 10x objective to observe the melting and solidification of materials, as controlled by a hot-stage. Remember to focus the image by first lowering the stage and then slowly raising it toward the objective, always paying close attention to the position of the hot-stage relative to the objective. Coarse tuning knob (larger) should be used first, and then fine tuning. Sample position can be adjusted using the stage controls, as seen in this picture as the vertical rod with two knobs.

Infinity© Camera

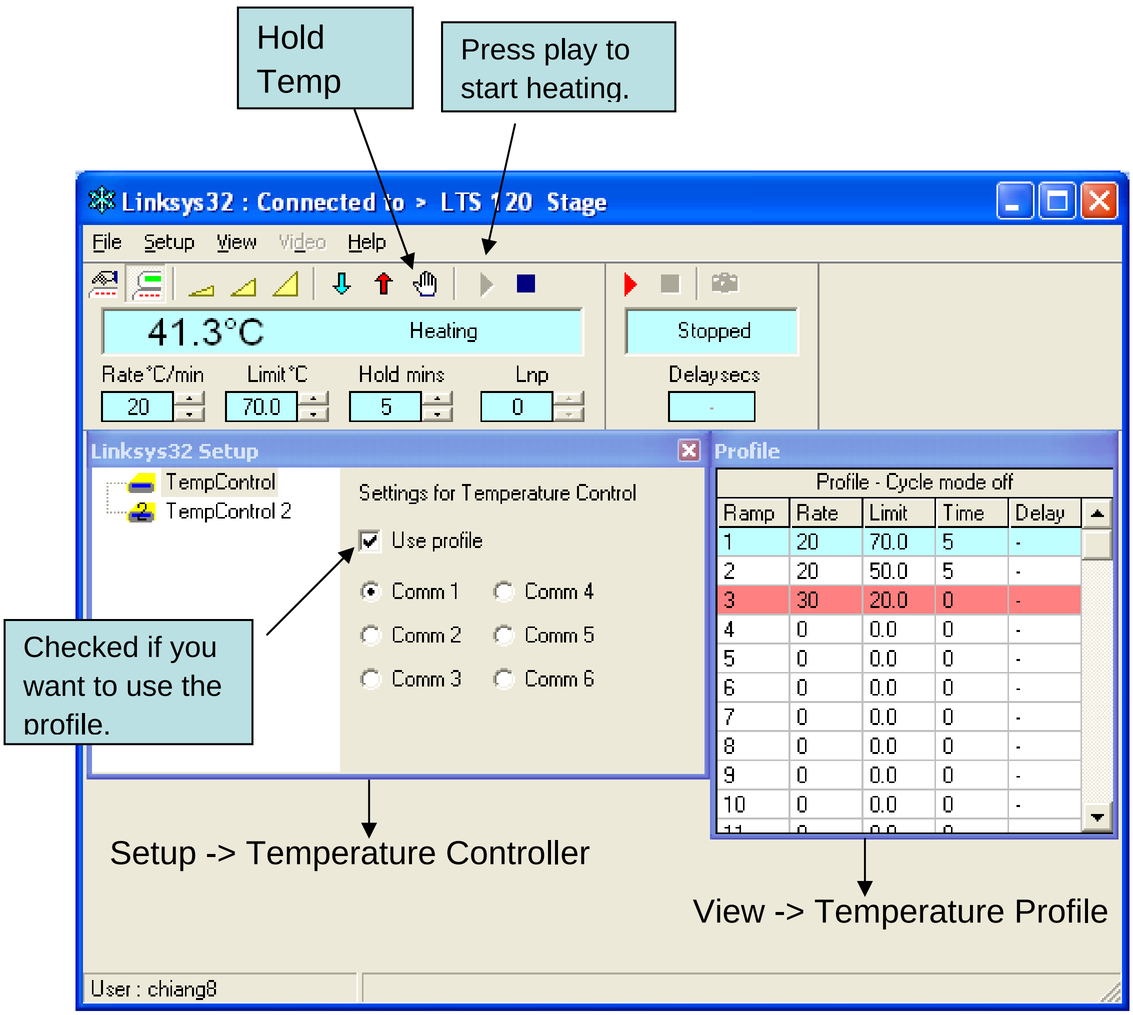
A camera is attached to each microscope, which can be used to view the image on the computer stations. The program INFINITY ANALYZE can be used to view the image, adjust image properties, and record the image. Scale bars can be added by selecting "10x Objective" under the Capture Control tab and then selecting Micrometer under the Annotation tab. Measurements of linear distance, angle, radius and more can be done under the Measurement tab. A screen shot is included below.

Hot-stage

A hot-stage is used to melt and solidify the alloys of interest under programmable temperature control. The hot-stages hold a standard-sized microscope slide, on which you will place your sample and cover slip. After the sample is in place, the hotstage can be placed on the microscope stage with the glass window underneath the chosen objective. The long working distance restriction imposed by the hotstage limits the possible objectives to 10 and 20x.

Linksys32 software

Linksys32 (shown below) is the program which controls the temperature profile for the hot-stage. After turning on the control box and opening the program, click File -> Connect to establish communications between the computer and the control box. Temperature control can be done in real-time by manipulating the values in the boxes, or a temperature profile can be set up.



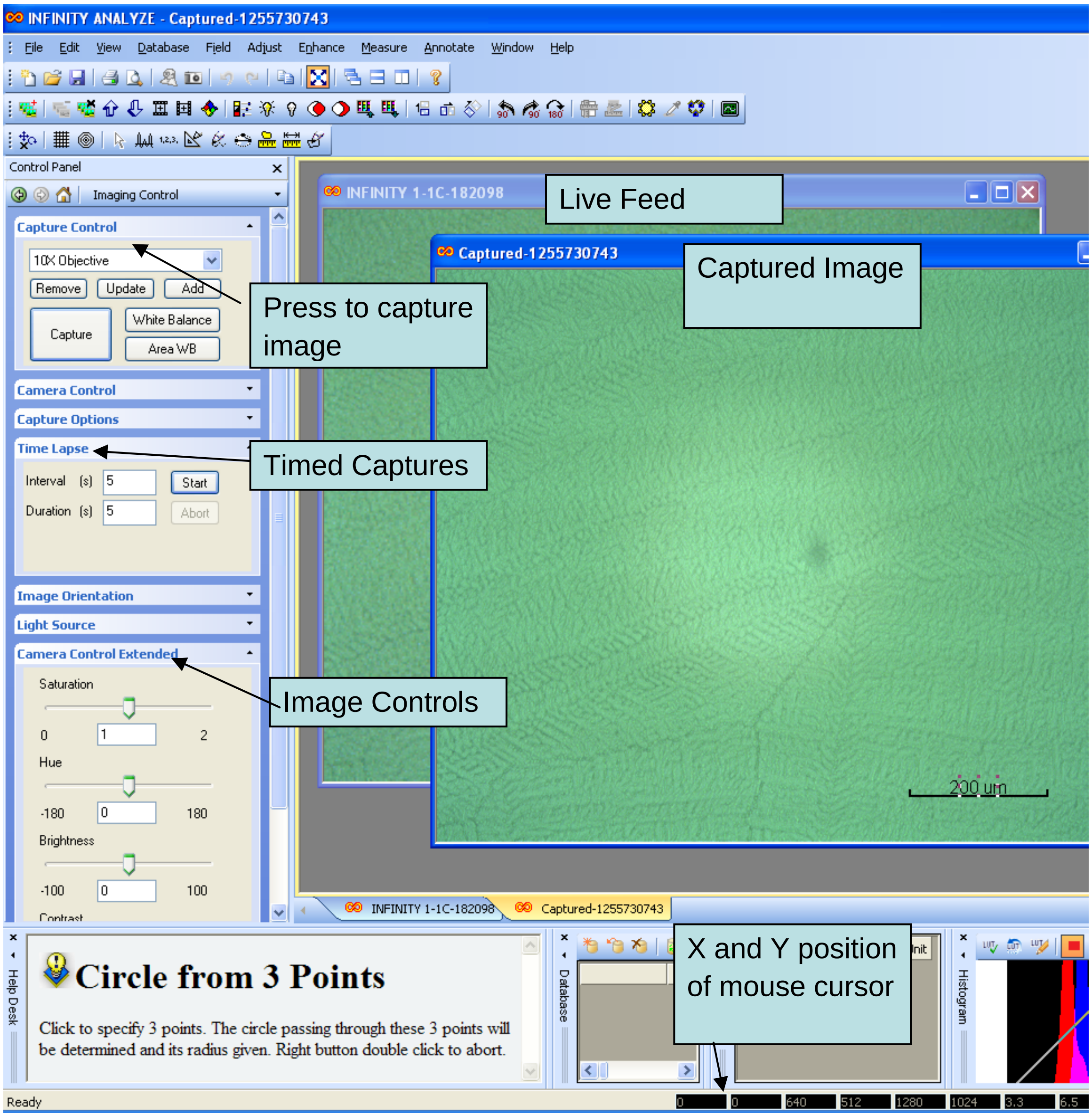
<u>Profile Box</u>

Rate = Temperature ramp rate in °C/min. No need for negative values for cooling if the Limit is set appropriately.

Limit = Target Temperature.

Time = Amount of time to hold at Limit Temp in minutes

Infinity Analyze Program



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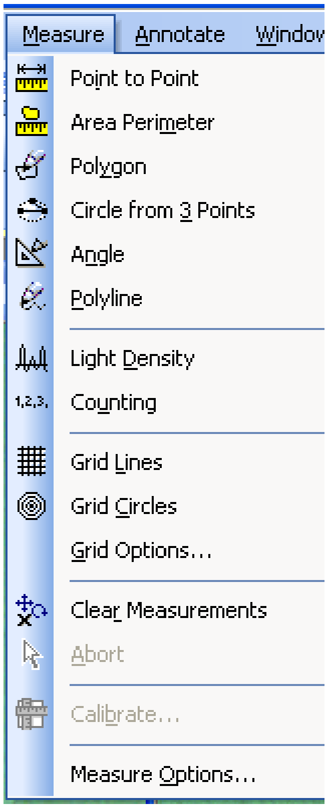

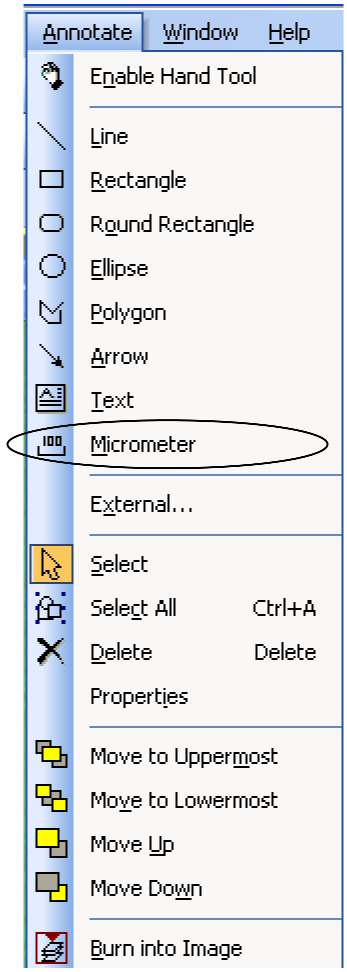

These functions can only be performed on captured images, not on the live feed.