

# 1 301 Laboratories

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## Laboratory 1: Crystal Structure, Imperfections, and Diffraction

### Objective

The objectives of this lab are to gain a better understanding of crystal structures, how they are measured and/or identified and the types of defects present in crystalline materials.

### Outcomes

Upon completion of the laboratory, the student will be able to:

1. Distinguish FCC, BCC and HCP structures.
2. Understand how - broadly - diffraction enables qualitative identification and quantitative measurement of repeating structures.
3. Describe edge and screw dislocations and how they might affect material properties.

**Experimental Details** Tennis balls simulate “metal atoms” formed into different structures. A laser pointer, ruler and tape measure are used to measure spacing of a two-dimensional pattern, simulating the application of x-ray diffraction.

### Exercise 1: Benchtop Simulation of Crystal Structures

**A. FCC and HCP space-filling structures** Each group should have a box, several tennis balls, and small wooden balls.

1. Arrange the tennis balls in a single close-packed layer in the bottom of the box. Start with a line of balls parallel to the “Start here” arrows along one side of the box.
2. Add a second (partial) layer. Identify the three-fold symmetry (six atoms around one) in the first and second planes. Compare this to the HCP model, and you will notice that this hexagonal structure occurs in the basal (base) plane with Miller indices (0001).
3. Add a third plane. At this point in building your structure, you must choose between two different sites. What is the difference between picking between one site and another? Which sites correspond to which crystal structure? (FCC vs HCP)
4. Identify the three-fold symmetry associated with the close-packed planes in many directions. In all, there are 4 such planes, perpendicular to the 4 different cube diagonals. The Miller indices are (111), (111), (111) and (111). There is only one set of close-packed planes in HCP, (0001). What are the consequences of FCC having multiple close-packed planes and HCP having only one? Think about the class discussion of dislocations.

- Identify the FCC unit cell. It isn't easy. . . .it is easier to start with an FCC model and find the close-packed planes. They form the diagonals of the cube! That is, the family of {111} planes. (Look for the half-unit cell styrofoam ball models, which you may put together and take apart – carefully!!!)
- Due to applied stress or deformation of a material, “mistakes” in stacking can occur. These “mistakes” are known as stacking faults. Create a stacking fault in the third plane of tennis balls with one side FCC and the other side HCP.

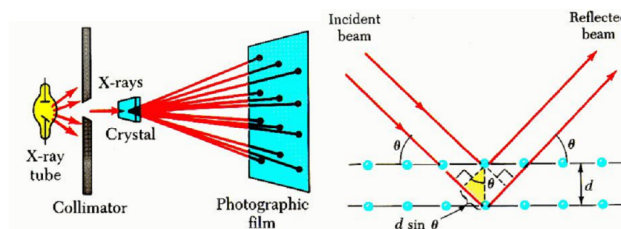
## B. Interstitial Sites

- You will notice gaps, or interstices, between the spherical atoms. These “interstitial” sites leave room for small solute atoms, and reinforce that that atomic packing factor (APF) is less than 1.
- Identify two different interstitial sites: octahedral and tetrahedral. To aid with visualization, you may take the small wooden balls (“solute atoms”) and place them in the interstitial sites. What are the coordination numbers for each of these interstitial sites?

**C. Body Centered Cubic Space Filling Model** Build a BCC lattice with tennis balls. Be careful about which balls touch. How do the interstices of BCC compare to that of the close packed structures from Part A?

## Exercise 2: Diffraction (HW is the last part below)

Diffraction occurs when wavelength of the source and obstacle spacing are similar in size. The wavelength of x-rays,  $\lambda = 0.1\text{-}0.5\text{ nm}$ , is well suited to investigate atoms with d-spacings with the same order of magnitude. Constructive interference occurs when the Bragg law is satisfied:  $n\lambda = 2d\sin\theta$ , where n is an integer and diffraction (3-D sample) occurs at an angle  $2\theta$  from the incident direction. The spacing between atom planes is d.

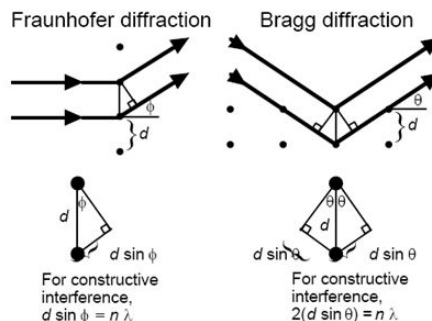


**Figure 1:** Diffraction condition

Visible light with wavelength about a thousand times larger ( $\lambda = 0.4\text{-}0.7\ \mu\text{m}$ ) can be diffracted by small dots on a glass plate with a spacing similar to d. Optical diffraction of dot arrays is thus an analogy to crystal structure determination by x-ray diffraction.

Here, for a two-dimensional array,  $n\lambda = d\sin\theta$ . This equation applies to the lab HW.

Two 35 mm slides are “samples” for this lab. Each slide contains eight patterns as indicated below and are designated A-H. Make sure the ICE text is on the right hand side, facing the laser, as shown below.



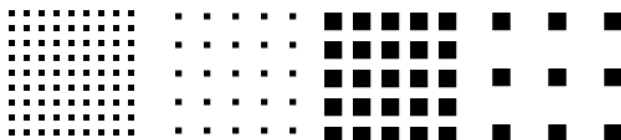
**Figure 2:** Figures from: The optical transform: Simulating diffraction experiments in introductory courses George C. Lisensky , Thomas F. Kelly , Donald R. Neu and Arthur B. Ellis J. Chem. Educ., 1991, 68 (2), p 91 DOI: 10.1021/ed068p91 Publication Date: February 1991

1. Discovery slide = green
2. Unit cell slide = black

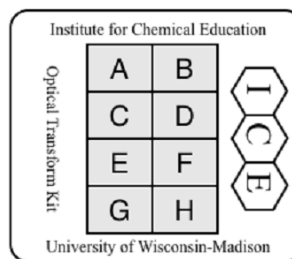
**Warm-up exercise:** Label each of the patterns below with its location (a-h) on the slide to the right. You may check your conclusions by looking at the slide under the stereomicroscope. Don't spend too much time here- make sure you proceed to the quantitative measurements, below.



\_\_\_\_\_



\_\_\_\_\_



## Lab Writeup

Due at the beginning of the next lab session.

For the lab writeup you will be using the black unit cell slide. See note about measurement accuracy, above. Record your measurements and calculations with the appropriate number of significant figures. Estimate the uncertainty in each computed value. (Assume your measurement tools are accurate, but with limited precision.)

1. Generate the diffraction pattern corresponding to unit cell (B) using a red laser. What wavelength is your laser? (How well do you know this value? How will you account for uncertainty? How might you measure the wavelength? )

2. Measure the distance between "sample" and "detector." (Note how changing this distance affects the pattern.)
3. Record the diffraction pattern (trace spots) on a sheet of paper, taped to the wall and use the spacing of your "detector" spots applied to the Fraunhofer equation to determine the spacing of dots on the slide (the spacing in the "sample"). Is this a reasonable value? Be sure to consider the number of significant figures you should report in your answer.
4. Repeat the process to measure the spacing for unit Cell (D) based on the corresponding diffraction pattern. Again, pay careful attention to the significance of your answer.
5. What if you used a green laser? Qualitatively, how would the diffraction pattern (displayed on the paper) change?
6. What type of packing arrangement is observed for Unit Cell (H)?

### **Models: Visualizing Defects**

Look at the models at the front of the room and identify which model represents

- (a) an edge dislocation
- (b) a screw dislocation
- (c) FCC
- (d) HCP
- (e) BCC

### **DEMO: Atomic Packing illustrated with the "Atomix Raft"**

The steel ball raft is a 2-dimensional model of atomic packing. Observe the following features in the demonstration: vacancies, hexagonal-packed grains, square-packed grains, grain and twin boundaries. Similar structures can be seen in (micrographs of) copper and brass samples.

### **DEMO: "Atomic Trampoline" Amorphous vs. Polycrystalline Metal**

The effect of defects and micro-structure on macroscopic properties:

Stacking the tennis balls provides an example of the ordered crystalline arrays that most metals form. Is it possible for metals to have a completely disordered amorphous structure? Yes. But typically these are many-component alloys.

In this demonstration, one base is aluminum (polycrystalline FCC), the second base has a 1/8" thick disc of amorphous metal alloy (Zr<sub>0.42</sub>Ti<sub>0.14</sub>Cu<sub>0.12</sub>Ni<sub>0.10</sub>Be<sub>0.22</sub>) glued to the aluminum. As the ball bearing bounces on the aluminum base, it loses kinetic energy to friction, heat and sound generation, and the formation of crystalline defects - slip and dislocation formation. The amorphous metal plate has a highly disordered structure - it is not a regular crystalline array - and these defects do not form. Hence less energy is transferred from the ball to the base. This explains why these alloys are used in state-of-the-art golf clubs.

## Laboratory 2: Electrical Conductivity & Optoelectronics

### Prelab

Before lab, please READ CAREFULLY both the lab handout and Callister Chapter 18, pages 666-689 & 694-695. The relevant concepts are: Ohm's Law, the difference between resistance ( $R$ ) and resistivity ( $\rho$ ), how conductivity changes with temperature in metals vs. semiconductors, bandgaps, and p-n junctions (pp. 694-695). Come in with questions.

### Exercise 1

**Conductivity and Resistivity** In this exercise, we will determine resistance as a function of temperature for different materials. This behavior distinguishes semiconductors from metals. If the geometry of the sample is known, you may convert measured **resistance ( $R$ )** to the corresponding property, **resistivity ( $\rho$ )**.

#### A. Metals

1. Measure the series resistance of your meter and leads: connect the two leads, then turn the meter to the most sensitive resistance scale, which has a maximum value of 200  $\Omega$ . (Ignore the  $\blacktriangleright+$ , which indicates the diode test function.) Note: the meter will read 1 if  $R$  is greater than the maximum of scale chosen. In that case, turn the dial to the next higher scale.
2. Measure the resistance  $R$  of the steel wire at room temperature. Record  $R$  in the table below.
3. Measure  $R$  at 77K. With the leads attached, immerse the coil in liquid nitrogen ( $T = 77 \text{ K} = -196 \text{ }^\circ\text{C}$ ). Record the value in the table.
4. Calculate resistivity and conductivity. The wire has a length of 1 meter and a diameter of 0.203 mm. Remember to subtract the series resistance from the measured value of  $R$ !
5. Compare your  $\sigma$  to that for "Steel alloy A36" in Table B.9 of the text; some deviation from the "book value" is expected. Why may your result differ from the book's?

	Temperature	$R (\Omega)$	$\rho(\Omega m)$
Series resistance	RT ( $\sim 298\text{K}$ )		
Steel Wire	RT		
	$LN_2(77\text{K})$		
Graphite Composite	RT		
	$LN_2(77\text{K})$		

Table 1: Resistance

#### Semiconductor: Thermistor

1. Measure the resistance of the metal oxide thermistor at three or more temperatures between  $0^\circ\text{C}$  and  $\sim 80^\circ\text{C}$ . Use water baths to expose the thermistor to these temperatures; measure the water bath temperatures with the thermocouple.

- Plot  $\ln(R)$  vs.  $1/T$  in Kelvin; the slope =  $E_g/2k$ . Note that  $R \propto 1/\sigma \propto \exp(E_g/2kT)$ . What is the effective bandgap,  $E_g$  (in eV)? Hint: make sure  $k$  is in appropriate units. Typically  $0.5 < E_g < 3$  eV for semiconductors..

Temperature (K)				
Resistance ( $\Omega$ )				

Table 2: Thermistor Resistance

## B. Graphite Composite of Unknown Composite

- Measure the resistance of the graphite composite pencil “lead” at room temperature. Record in the table on pg. 1.
- Measure the resistance of the graphite composite cooled in liquid nitrogen. Observe what happens immediately when it is dunked into the LN2. If the clips freeze, the measurement will be inaccurate. Record in the table on pg. 1.
- Calculate the resistivity of the graphite composite. The diameter of the rod is 0.7 mm. Measure the length between the leads. Does this material behave like a metal or a semiconductor?

## Exercise 2

**Optoelectronics** In this exercise, we will determine the relationship between the current ( $I$ ) through and voltage across ( $V$ ) a light emitting diode (LED) and a photoresistor. We will correlate the I-V behavior with light emission (LED) or absorption (photoresistor).

### A. Light Emitting Diode

A light emitting diode is an example of a p-n junction. It is based on a solid solution of GaP and GaAs ( $GaAs_xP_{1-x}$ ). The emission wavelength depends on the composition of this solution, as well as any dopants present.

- Set up the measurement apparatus. Attach the voltmeter clips to the alligator clips on the board, so it is in parallel with the diode (p-n junction) that you will also attach there. (Note, red = +) Attach the ammeter in series, across the two bare wire connections on the bottom of the board. (Connect red to red, so you obtain the correct sign for  $I$ .)
- Forward Bias ( $V_{bias} > 0$ ). Do a quick check that as you turn up the voltage (rotate the post on the potentiometer) the diode eventually emits light. Start with the ammeter on the most sensitive scale (200  $\mu A$ ). Take enough data points (about 10 total) to determine the I-V curve from  $0V < V_{bias} < +3V$ . Remember to note the units for both  $V, I$ .
- Reverse Bias ( $V_{bias} < 0$ ) Connect the circuit in reverse bias: take the black lead with the alligator clip and attach it to the opposite side of the potentiometer. Again, measure the I- V response. The magnitude should very small; be sure to use the maximum sensitivity of the ammeter. Please plot the I-V curve based on both forward and reverse bias on one graph (use

graphing software). Note that the voltage where the current increases rapidly, and where the light intensity increases, corresponds (approximately) to the bandgap energy. How would the curve change for a green LED? An IR LED?

	Reverse Bias (-V,-I)				Forward Bias (+V,+I)			
V				0				
I				0				
Light?								

**Table 3:** LED I-V Data

### B. Photoresistor

This photoresistor is made of CdS, a II/VI compound intrinsic semiconductor (not doped). The bandgap  $E_g = 2.59$  eV, which means that conduction electron/holes provided by thermal energy ( $kT$ ) are very rare. However, light with energy  $E(\text{eV}) = hc/\lambda = 1.24/\lambda = E_g$  ( $\lambda \leq 0.48 \mu\text{m}$ ) will promote valence electrons to create conduction electron/hole pairs. Visible light ranges from 0.4 to 0.7  $\mu\text{m}$ . More light will provide more charge carriers ( $n_e = n_h$ ), and increasing conductivity  $\sigma$ . A CdS element is the heart of light meters, etc.

1. Measure R in the dark. Shield the resistor from ambient light with your hand or a cardboard box. Record the resistance.
2. Measure R in ambient light. Take photoresistor out of shadow and record the resistance.
3. Observe how R changes with distance from a light source. Move the photoresistor closer to the ceiling lights. What would happen if you illuminated your photoresistor with monochromatic 1 $\mu\text{m}$  wavelength light?

	Dark	Light
Resistance ( $\Omega$ )		

**Table 4:** Photoresistor resistance

### C. Light Emission vs. Composition

Light emission is possible when charge carriers (electrons and holes) recombine across the bandgap of some semiconductors. The four LED's are solid solutions of GaAs ( $E_g = 1.4\text{eV}$ ) and GaP ( $E_g=2.3\text{eV}$ ). Varying the composition of the solid solution allows us to control the bandgap, and thus vary the color of emission.

1. Observe light emission from the four LEDs. The LEDs have differing ratios of GaAs and GaP- this ratio varies monotonically along the strip. Given what you know, which LED has the highest ratio of GaAs:GaP?
2. Observe the blue LED. This LED is made of a different material. Why might one have to switch to a different material to get the observed light emission? What property would need to be different?



## Lab Writeup

One writeup per person (not per group) is due at the beginning of the next lab session.

Please turn in a full set of data tables, including calculated numbers like resistivity, and answer the questions/do the tasks in italics. The questions are repeated below for your convenience.

1. Compare your result for the  $\sigma$  of Steel alloy A36 to that in the book. Why might your value differ?
2. For the Thermistor, Plot  $\ln(R)$  vs.  $1/T$  in Kelvin; the slope =  $E_g/2k$ . Note that  $R \propto 1/\sigma \propto \exp(E_g/2kT)$ .
3. What is the effective bandgap,  $E_g$  (in eV)? Hint: make sure  $k$  is in appropriate units. Typically  $0.5 < E_g < 3$  eV for semiconductors.
4. Does the graphite composite behave like a metal or a semiconductor?
5. Plot the LED I-V curve based on both forward and reverse bias on one graph (use graphing software).
6. How would the curve change for a green LED? An IR LED? Note that the voltage where the current increases rapidly, and where the light intensity increases, corresponds (approximately) to the bandgap energy.
7. What would happen if you illuminated your photoresistor with monochromatic  $1\mu\text{m}$  wavelength light?
8. Given what you know, which LED has the highest ratio of GaAs:GaP?
9. Why might one have to switch to a different material to get the observed light emission? What property would need to be different?

## Laboratory 3: Mechanical Properties of Metals

### Objective

The objectives of this lab are to explore how crystal structure and defects affect mechanical properties and to understand how these properties are measured.

### Outcomes

Upon completion of the laboratory, the student will be able to:

1. Describe the effects of work-hardening and annealing in metal samples.
2. Describe a ductile-to-brittle transition temperature.
3. Compute the elastic modulus, yield strength, ultimate tensile strength and strain at fracture from load-displacement (stress-strain) curves.

### Experiment

Parts A and B are in Room 2068, while parts C and D are in Room 1034. Safety\*\*

**A. Work hardening and annealing in copper - Use caution with torch and hot tubing - see below.\*** You are supplied a length of copper tubing. Grasp it near one end and bend it slightly (through about 20-30°). Now bend it back to straighten the piece. You should feel considerable additional resistance to straightening. Bend and straighten the same place once again, and perhaps a third time. Resistance will continue to grow. Now bend an "original" section near the other end - notice how easy it is to deform the as-received Cu. You are feeling the effect of "work hardening" or "strain hardening". Plastic deformation is accomplished by dislocation motion. The force required to achieve plastic deformation also creates many additional dislocations that were not present initially.

Dislocations move easily through structurally regular crystals. The dislocations created during the first deformation constitute numerous irregularities, and hence make the material more resistant to further deformation, in this case the straightening step. Dislocations are high energy defects that can be removed by annealing the material at  $T_a \sim 0.5T_m$ . The melting temperature of Cu is 1360 K, so annealing effects are rapid above 680 K or 407 °C. The propane torch has a flame temperature greater than 1000 °C.

\*\*Keep the propane torch on the table at all times. Holding the Cu tubing with tongs or pliers, heat the "cold worked" region until it is red hot. Cool the entire length of copper and the pliers in water!! Now test the copper for yield force; you should be back to the original "soft" condition.

**B. Rolling of brass** As for copper above, brass is capable of significant work hardening. The purpose of this exercise is to quantify the effect of work hardening that was observed qualitatively with the copper tubing. Resistance to yield is here measured by hardness after different extents of plastic deformation by rolling.

You are provided with a set of brass samples that have been rolled to different thicknesses. For each:

1. Measure the thickness of the specimen.
2. Measure its hardness. (Use the Rockwell B scale: 1/16" ball indenter, 100 kg load)

Sample	A	B	C	D	E
Thickness					
%CW					
Hardness, $R_B$					

**Table 5:** Rolling of brass

**C. Charpy impact testing** Impact tests (also called Charpy tests) induce fracture under conditions of high rates (impact) at an intentional flaw (the notch). This method readily demonstrates the effect of temperature upon the fracture energy of A36 steel (carbon content of not more than 0.30 wt%) and on an Al alloy (2024, 4.4 wt. % Cu). Note: steel is a BCC metal, while Aluminum is FCC. Complete the table below.

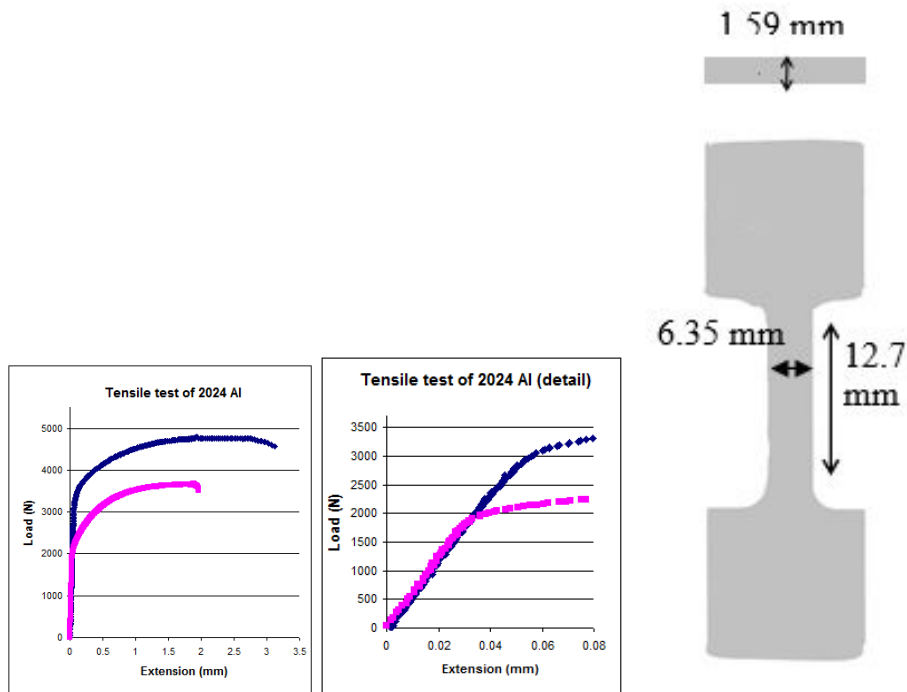
Sample	Temperature ( $^{\circ}C$ )	Impact Energy (ft-lb)
A36 steel, RT	$25^{\circ}C$	
A36 steel, LN	$-196^{\circ}C$	
2024 Al, RT	$25^{\circ}C$	
2024 Al, LN2	$-196^{\circ}C$	

**Table 6**

**D. Tensile test of an Al alloy** You will have an opportunity to observe a tensile test on a sample of an Al alloy. Notice inhomogeneous deformation (necking) before failure. The plot below indicates the tensile test results for two 2024 Al samples, one as-received and a second after heat-treatment. 2024 is an aluminum alloy containing small amounts of Cu. Both test samples had the same dimensions: width ( $w$ ) = 6.35mm, thickness ( $t$ ) = 1.59mm, and gauge length ( $L_o$ ) = 12.7mm.

### Homework Problems

1. (Part A) Copper Work Hardening
  - (a) Given the information in this handout, would you expect to be able to anneal work-hardened copper in boiling water?
  - (b) Would you expect annealing temperatures for molybdenum to be higher or lower? Why?
2. (Part C) Charpy Impact testing



**Figure 3:** Tensile test of an Al alloy

- (a) Why do the high and low temperature steel samples differ in appearance of the fracture region, while they appear similar for the aluminum? Note: this steel is BCC, while the aluminum is FCC.
3. (Part D) Tensile Testing: Convert load and displacement values from the graph to stress and strain to calculate the following. Show your work. Use engineering stress and strain (i.e. assume the cross-sectional area remains the same throughout the test).
- Elastic (Young's) modulus for 2024 Al.
  - The 0.2% offset yield strength for both the as-received sample and the annealed sample.
  - The ultimate tensile strength for the both the as-received sample and the annealed sample.
  - The amount of strain at fracture for both samples.

## Laboratory 4: Phase Diagrams and Transformations

### I. Eutectic salt solidification

Below is part of the phase diagram for the ammonium nitrate-sodium nitrate salt solution. The diagram shows a eutectic reaction at 21wt%  $NaNO_3$ .

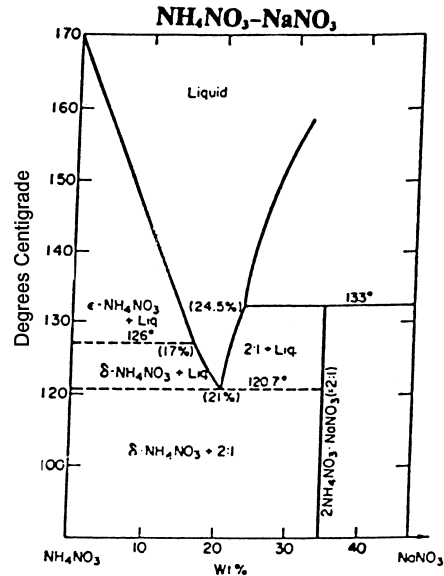


Figure 4:  $NH_4NO_3 - NaNO_3$

NOTE: Phase diagrams are determined assuming equilibrium and thus predict equilibrium concentrations and phases. On heating, transitions occur close to equilibrium temperatures. This is not true for cooling (nucleation).

### II. Steel rod

Many useful properties of iron and steel are determined by the crystal structure of the material, which in turn may be altered by heating and cooling. When steel is liquid, carbon dissolves readily in the iron. Even as a solid, carbon dissolves into the iron, forming a solid solution; however, the solubility of carbon in iron is limited by the crystal structure, based on the size of the interstitial sites available for carbon in the crystal structure. FCC gamma iron (austenite) can accommodate carbon at the edges of the crystal cell with little distortion. BCC alpha iron (ferrite) can accommodate carbon at the edge or face center of a cell; however the sites are smaller, and significant distortion of the crystal lattice occurs.

When slow cooled, the carbon has time to diffuse through the iron and form cementite  $Fe_3C$  at dislocation sites; the steel remains ductile at room temperature.

When quenched, the carbon atoms are not given time to diffuse; they force iron atoms out of the usual places in the unit cell, resulting in a distortion of the crystal. This new structure is called martensite. It results in material that is hard— because dislocations cannot move easily through the distorted lattice, and brittle, for the same reason.

If martensite is tempered, that is, heated to a temperature below the eutectoid transformation (usually this means between 250 – 600°C), the material will remain hard, but ductility and toughness will be enhanced. (Getting this right with the torch is a bit tricky – because the temperature is not well-controlled.)

### III. Shape Memory wires (NiTi)

The wire on the table is NiTi (or NiTiNOL, named for the NiTi alloys developed at the Naval Ordnance Lab).

1. Start with the wire at room temperature.
2. Deform it gently, i.e. curl or bend it.
3. Warm it above the austenite transformation temperature, and observe how it recovers.

The low temperature phase (martensite) is deformable, but the alloy “remembers” its shape when it returns to the higher temperature (austenite) phase at  $T < 100$  °C.

### IV. Bi-Cd binary eutectic alloy

We will examine metallographic sections of different compositions in the Bi-Cd system, which exhibits a eutectic at 39.7 wt.% Cd. Note that the Bi-Cd system has negligible solid solubility. Compare this to the binary eutectic Pb-Sn system which does have solid solubility, shown in Fig.5.7 of your text.

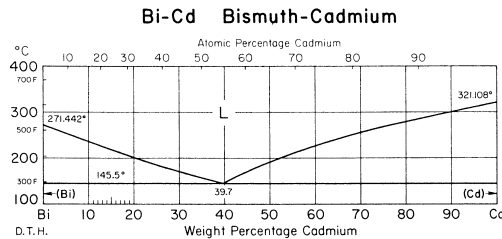


Figure 5: Bi-Cd Bismuth-Cadmium

### HOMEWORK PROBLEM

1. **Sketch** each of the three microstructures corresponding to samples A-C.
2. **Label the composition of the alloy** you were shown.
3. **Label the phases present** (indicate which phase is etched dark, which phase is etched light).
4. For each sample, use the **lever rule** to predict amounts (wt%) of **primary phase vs. eutectic**.

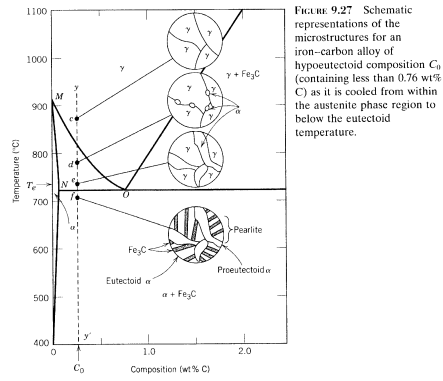


FIGURE 9.27 Schematic representations of the microstructures for an iron-carbon alloy of hypoeutectoid composition  $C_0$  (containing less than 0.76 wt% C) as it is cooled from within the austenite phase region to below the eutectoid temperature.

Figure 6: Microstructures of hypoeutectoid iron-carbon alloy

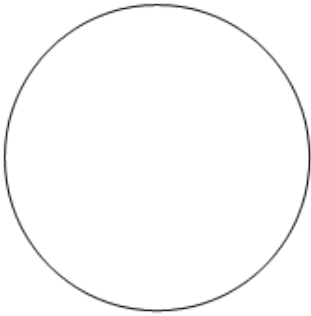


Figure 7: Part A

V. Steel wire/carburized steel samples

A. \_\_\_\_\_ wt% Cd

Amount primary phase: \_\_\_\_\_

Amount eutectic: \_\_\_\_\_

B. \_\_\_\_\_ wt% Cd

Amount primary phase: \_\_\_\_\_

Amount eutectic: \_\_\_\_\_

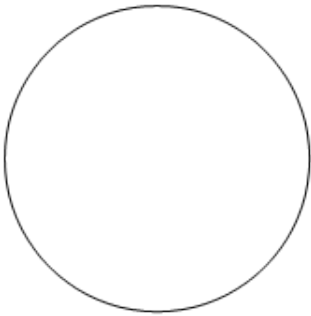
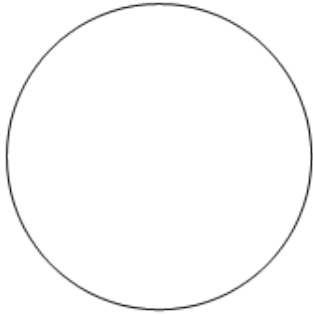


Figure 8: Part B

C. \_\_\_\_\_ wt% Cd

Amount primary phase: \_\_\_\_\_

Amount eutectic: \_\_\_\_\_



**Figure 9:** Part C

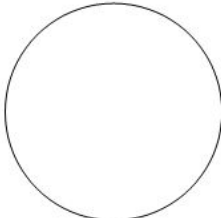
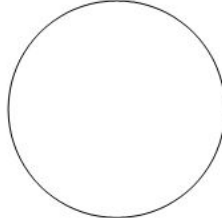
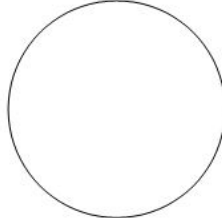
**Phase Transformation** When piano (~0.8% carbon steel) wire is heated (in this case by running a current through it) it transforms from BCC alpha iron to FCC gamma iron. The reverse is true on cooling. Why does the wire sag as it is cooled?



Name: \_\_\_\_\_

### Homework Problems

1. Identify (circle) the eutectic phase transformation on the  $\text{NH}_4\text{NO}_3\text{-NaNO}_3$  phase diagram and indicate the temperature/composition at which it occurs.
2. Explain why steel quenched from high temperature which forms a martensite phase exhibits higher hardness and strength than slow cooled steel which forms a mixture of ferrite/cementite.
3.
  - (a) Sketch each of the three microstructures you observed, corresponding to samples A-C.
  - (b) Label the composition of the alloy you were shown.
  - (c) Label the phases present, which is etched dark and which is etched light?
  - (d) For each of the samples use the lever rules to predict the amounts (wt %) of the primary phase vs. the eutectic.
  - (e) Are the samples hypo- or hyper eutectic?

A. _____ wt% Cd	B. _____ wt% Cd	C. _____ wt% Cd
Amount primary phase: _____	Amount primary phase: _____	Amount primary phase: _____
Amount eutectic: _____	Amount eutectic: _____	Amount eutectic: _____
		

4. When piano (~0.8% carbon steel) wire is heated (in this case by running a current through it) it transforms from BCC alpha iron to FCC gamma iron. The reverse is true on cooling. Why does the wire sag as it cools?

## Laboratory 5: Mechanical Behavior of Ceramics and Polymers

### Polymer 1. Crystallization of Isotactic Polypropylene

When cooled from the melt, this isomer, with R = CH<sub>3</sub> groups all on the same side of the chain, crystallizes into spherulites. Note that crystallization occurs below T<sub>m</sub> = 165 °C (undercooling).

### Ceramics 1. Three-Point bend test (Bonus)

It is difficult to perform conventional tensile tests on most ceramic materials; they are brittle and break in the grips of the test apparatus. It is easier to test these materials in the bending mode, as illustrated below. Note that when loaded, the top of the material is in compression and the bottom is in tension.

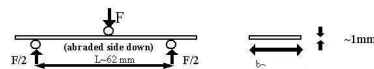


Figure 10: Three-point bend test

- Measure the force  $F$  (weight of volume of water plus container) to fracture a pristine glass microscope slide.
- Abrade a second microscope slide with 180 grit ( $a = 80 \mu\text{m}$ ) paper. Abrasion should be in center of slide, and in the "b" direction (above). BE SURE YOUR HANDS AND SLIDE ARE DRY. Measure fracture force for slide with flaw dimension  $a$ ; flaw on bottom (tensile side).
- If time permits, repeat part b with abraded side up (under compression). This should be close to your answer in part a.
- Glassblowers often use the trick of scribing a mark, then wetting it, to get the glass to break there. If you notice the maximum load is less when the abraded region of the slide is wet, you have observed stress corrosion cracking. The Si-O-Si network in the glass is broken by formation of Si-O-H bonds at the surface, and the material effectively "unzips."

Container weight = 118 g (Add to obtain total load)	Vol (ml)	Total load (g)
A. Pristine slide: vol. (ml)		
B. Flawed slide: vol. (ml)		
C. Compression: vol. (ml)		
D. Repeat b, but WET abraded region		

Table 7: Bend test results

### Lab Homework

- Calculate fracture force for experiments A and B.
- Use equation 12.3a in Callister (see addendum) to calculate the "flexural strength" obtained with this three point bend experiment. Sample dimensions are  $d = 1 \text{ mm}$ ,  $b = 25 \text{ mm}$ , and  $L = 62 \text{ mm}$  between support points. Your answer should be in MPa, and you will need to convert  $F_f$  from g to N.

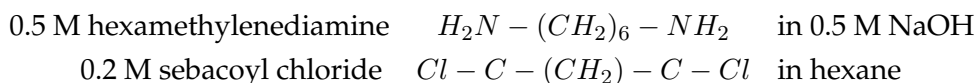
- Use your result from experiment B and the following equation to calculate  $K_{IC}$  for the microscope glass material:  $K_{IC} = Y\sigma_f\sqrt{\pi a}$ , where  $a$  is the flaw size ( $= 80\mu$ ) and  $Y$ , dependent on sample & flaw geometry = 1. Compare your  $K_{IC}$  to the range predicted for ceramic materials in Table 8.1.
- Use your  $K_{IC}$  and the result from experiment A to calculate flaw size  $a$  in the pristine glass slide (the slide you did not intentionally scratch).

## Ceramics 2. Softening glass

Hold the glass rod in the flame of the propane torch. When the rod becomes hot enough, that is when the temperature exceeds the "glass transition temperature,"  $T_g$ , the glass will flow and you can easily bend it. **BE CAREFUL NOT TO BURN YOUR FINGERS. Don't touch the HOT glass.**

## Polymers 2: Nylon Polymerization

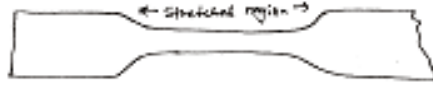
Nylon is formed by the reaction of two liquid monomers, here dissolved in immiscible liquids. These can react with each other, but not with themselves:



The reaction occurs in a thin layer at the interface between the two liquids. By slowly pulling the film in this region, a long, continuous filament can be pulled from the beaker. NOTE THE BYPRODUCT IS HCl – so use gloves.

## Polymers 3. Plastic Deformation of Polyethylene

- With thumbs separated by about 0.5 cm, stretch the strip with a slow, steady force. The polyethylene will yield and deform plastically. Here the sliding of planes of atoms (crystals) is accompanied by uncoiling sections of the chain-like polymer molecules (amorphous regions).
- You will notice that a thin portion or "neck" is formed and propagates during stretching. The plastically deformed polymer is anisotropic with different properties in different directions. With many chains or portions of chains in the stretch direction, this chain orientation means that covalent bonds are deformed or break when the film is further loaded in the stretch direction. Relatively weak van der Waals bonds control properties in the transverse directions.
- Grasp a section of the necked polymer and pull in the stretch direction. The oriented film is stiff and strong (no easy yield or failure) in this direction.
- Now grasp the same necked portion in the transverse direction and pull slowly. The materials will yield and "split", as weak secondary bonds are dominant in this direction.



**Figure 11:** Necked polymer

Conclusion: You can "feel" the difference between strong covalent and weak van der Waals bonds when the chains are oriented.

#### **Polymers 4. Plastic Deformation of Polyethylene**

You are all familiar with "high density" ( $f_c = 0.7$ ) and "low density" ( $f_c = 0.4$ ) polyethylene. As PE at room temperature is a crystal/rubber combination, the elastic modulus of HDPE is larger. As there are more crystals, the yield strength  $\sigma_y$  of HDPE is larger as well.

- With increased yield strength (more crystallinity) comes reduced ductility.
  - Somewhat similar to BCC metals, the yield strength of polymers is temperature dependent.
1. Test with your fingers the two flexure samples. Which is high density polyethylene? Which is low density polyethylene?
  2. Cool the tensile samples in liquid nitrogen and repeat. A substantial change in ductility (and likewise an increase in yield stress) will be noticed.

#### **Polymers 5. Rubber nails (Glass Transition Temperature (a))**

Cool a rubber "nail" by holding in liquid nitrogen (with tongs!!). The material is now a glass. Use the glassy nail to join two pieces of (soft) balsa wood. Hints: grasp the nail with the tongs; align the nail with the grain of the wood; use repeated soft taps to avoid shattering your "nail." Have everyone in your group contribute a nail.

#### **Polymers 6. Rubber band (Glass Transition Temperature (b))**

At room temperature, rubber is above its glass transition temperatures  $T_g = -70^\circ C$ , but at liquid nitrogen temperature, rubber is below  $T_g$ .

A stretched rubber band is cooled with liquid nitrogen. The shape is conserved as the macromolecules are "frozen in", and the band is very brittle. As the temperature increases back toward room temperature and  $T_g$  is reached, the band recovers its natural undeformed shape.

#### **Polymers 7. Happy/ Sad balls (Glass Transition Temperature (c))**

Two polymer balls of different compositions are provided, along with hot water and LN2. DO NOT try to FREEZE the balls completely in LN2. Cool them gently, while moving around in a small amount of liquid, for 10-20 seconds. Otherwise, the balls will fracture.

1. Drop both balls onto a table at room temperature. Ball 1 is above its  $T_g$  and thus bounces back elastically (rubber elasticity). Ball 2 is near its  $T_g$  and dissipates all the kinetic energy without bouncing.
2. Heat Ball 2 and repeat the experiment. The ball is now above its  $T_g$  and bounces back elastically (rubber elasticity)
3. Cool Ball 2 and repeat the experiment. The ball is now below its  $T_g$  and bounces elastically (glass elasticity).
4. Cool Ball 1 and repeat the experiment. The ball will be glass elastic below  $T_g$ , "dead" near  $T_g$  and rubber elastic above  $T_g$ . Very cool!

What practical items are made from such rubbery elastomers? Rubber o-rings, used to seal surfaces – except when they get cold, below  $T_g$ , they become stiff and don't conform – hence resulting in leaks. (The Challenger Disaster was a consequence.)

**E. Viscoelasticity (Strain rate dependence)** Silly Putty is a polymer above its glass transition temperature which is thus viscoelastic at room temp. At high strain rates, it behaves elastically, while at low strain-rates, it is viscous.

**Ceramics Demonstrations**

**III. Thermal Shock** If you have ever poured hot water into a cold glass, or vice versa, you may have experienced the effect of the material's low resistance to thermal shock. ( $TSR = \sigma_c k / E \alpha_1$ ). This, in turn, is due to the large thermal expansion coefficient and low thermal conductivity typical of most ceramic materials. These properties may be varied by changing the composition of the glass, as indicated below. (Of course, the cost changes, too.)

Glass	Comp. (wt%)	Thermal Conductivity, k ( $W m^{-1} K^{-1}$ )	Thermal expansion coefficient (linear), $\alpha_1 (K^{-1})$	Thermal Shock Resistance
Soda-lime (windows, bottles) Borosilicate (Pyrex)	70% $SiO_2$	11	$8.5 \times 10^{-6}$	84
	10% $CaO$			
	15% $Na_2O$			
	80% $SiO_2$		$4.0 \times 10^{-6}$	280
	15% $B_2O_3$			
	5% $Na_2O$			

**Table 8:** Thermal properties of glass

**IV. Toughening plates** As you will see in the laboratory exercise, ceramics have a high probability of fracturing when a tensile force is applied. This is due to the crack propagation through the material. One way to strengthen ceramics, then, is to introduce compressive forces in the outer portion of the piece, where cracks are likely to initiate. The compressive forces act to close surface cracks, rather than cause them to propagate. An example is tempered glass, which is formed by

quickly cooling from above its glass transition temperature: the outside cools faster than the inside, and is put into compression by contraction of the slower cooling core. This technique is used in making car windshields.

A second method for producing ceramics with an outer layer in compression is to use layered materials. In this case, the outside layer has a lower thermal expansion coefficient than the inside, and again is put into compression as the piece forms.

**V. Tough ceramic ball** The material is  $Zr_2O_2/CaO$  "alloy." At room temperature it contains ~10% of nonequilibrium tetragonal phase in a matrix of equilibrium monoclinic phase. Large local tensile stress near a crack tip triggers a tet  $\rightarrow$  mono transition with a 4% volume increase. The larger volume of new monoclinic crystals creates compressive stress that "pinches" the crack shut.  $K_{IC}$  is increased to about 10 MPa  $m^{1/2}$ .

**VI. Other Ceramics** Ceramics are refractory, That is, they maintain structural integrity at high temperature, and may provide thermal and electrical insulation. Some are used as capacitors or resistors on computer motherboards. Some are piezoelectric, that is they may be used to transform mechanical energy into a voltage. Others are magnetic (Hard B  $BaFe_{12}O_{19}$ ,  $SrFe_{12}O_{19}$ , used for small electric motor field magnets, Refrigerator door seals and posting magnets; Soft B  $NiFe_2O_4$ ,  $MnFe_2O_4$ , transformers, inductors) or superconducting.

#### **Addendum:**

For a sample with a rectangular cross-section tested on a three-point bend apparatus, the tensile breaking stress, termed the flexural strength,  $\sigma_{fs}$ , is:

$$\sigma_{fs} = \frac{3F_b L}{2bd^2}$$

Here  $F_b$  is the load at break,  $L$  is the distance between supports,  $b$  is the width and  $d$  is the thickness of the rectangular cross-section.<sup>1</sup>

1. Callister, William D. Jr. Materials Science and Engineering: An Introduction, 6th Edition, ISBN 0-471-13576-3, John Wiley and Sons, Inc., New York, 2003.

## Discovery Lab 1: Cholesteryl Ester Liquid Crystals

### Pre-lab

Important: Be sure that you are wearing long pants and closed-toed shoes for this lab. Lab coats, safety glasses, and gloves will be provided.

**Goal** To investigate the relationship between temperature, composition, and reflected/transmitted colors of a cholesteryl ester liquid crystal.

**Background** Liquid crystals are organic compounds that are in a state between liquid and solid forms. They are viscous, jelly-like materials that resemble liquids in certain respects (viscosity) and crystals in other properties (light scattering and reflection). Liquid crystals must be geometrically highly anisotropic, as opposed to an isotropic liquid. Such properties, however, can be altered through thermal action (heat) or by the influence of a solvent.

Common liquid crystals are composed of derivatives of cholesterol,  $C_{27}H_{46}O$ . Materials that will be used in this lab for making the cholesteryl ester liquid crystals are cholesteryl benzoate,  $C_{34}H_{50}O_2$ , cholesteryl pelargonate,  $C_{36}H_{62}O_2$ , and cholesteryl oleyl carbonate,  $C_{46}H_{80}O_3$ .

Cholesteric-phase liquid crystals contain molecules aligned in layers rotated with respect to one another, resembling helix structures (Figure 12). The distance between layers that have the same orientation is known as the pitch,  $p$ . At high temperature, the rotation angle from one layer to the next increases, hence the pitch is smaller. A color will be reflected when the pitch is approximately equal to the color's wavelength of light.



**Figure 12:** Cholesteric-phase liquid crystals (Figure courtesy of George Lindsey)

### Pre-lab Questions

1. What are the safety concerns with each of the chemicals? What is the proper way to handle the chemical waste? Please read the Materials Safety Data Sheets (MSDS) for each chemical used in this lab beforehand.
2. Liquid crystals have anisotropic structures. In your own words, explain the term “anisotropy” and how will this affect the material properties of liquid crystals.

3. If the reflected color is green and changes to red, chemically/physically explain what has happened to the liquid crystal. Be specific. Does this correspond to a temperature increase or decrease? Why?



## Lab Part 1

**Objective** Build a cholesteryl ester liquid crystals thermometer that covers the temperature range between 17-40°C.

### Laboratory Procedure

1. Based on Table 1, select 5 ratio combinations of the cholesteryl oleyl carbonate, the cholesteryl pelargonate, and the cholesteryl benzoate.
2. Measure out the allotted amounts of each of the cholesterol components and place them in a 10 ml vial.
3. Melt the solid in a sample vial using a hair drier or heat gun. (Make sure the vial lid is off during the melting process because it can ruin the seal on the vial.)
4. Cut one piece of clear packing tape and lay the sticky side up on a table.
5. Take the vial of the cholesteryl ester mixture, open it and transfer a small amount of the gel paste to the tape with a spatula. Spread the gel uniformly in the middle of the tape. Be sure to leave a centimeter clearance from the edge of the tape. Take the other piece of packing tape and place the sticky sides together. Smooth the paste evenly so there are no air bubbles.
6. Submerge the liquid crystal sandwich in a beaker of water and gently heat the water with a hot plate.
7. Measure and record the temperature at which the liquid crystals first change color. What color do you see first?
8. Measure and record the temperature at which the liquid crystals become colorless. What color do you see last?
9. Cool the sample by removing it from the water. Repeat steps 2-3 as necessary to list the order of colors observed.

### Lab Writeup

1. Upon heating, when the liquid crystal sandwich undergoes a color change, what color do you see first (i.e., at coolest temperature)? What color do you see last (warmest)? What is the order of colors you see going from coolest to warmest? Explain what happens to the liquid crystal during this transition at molecular level.
2. What is the relationship between the mass of cholesteryl oleyl carbonate in the mixture and the transition temperature? What do you think might be the reason for this?
3. As the liquid crystal changes temperature it reflects different colors. Is the color change better observed over a black background or a white background? Why?
4. What are some of the applications you can think of with the temperature sensitive cholesteryl ester liquid crystals?
5. What happens if you heat 0.30 g of Cholesteryl oleyl carbonate, Cholesteryl pelargonate, Cholesteryl benzoate by themselves, will you observe the same behavior?

Cholesteryl oleyl carbonate	Cholesteryl pelargonate	Cholesteryl benzoate	Transition range, °C
0.65 g	0.25 g	0.10 g	
0.70 g	0.10 g	0.20 g	
0.45 g	0.45 g	0.10 g	
0.43 g	0.47 g	0.10 g	
0.44 g	0.46 g	0.10 g	
0.42 g	0.48 g	0.10 g	
0.40 g	0.50 g	0.10 g	
0.38 g	0.52 g	0.10 g	
0.36 g	0.54 g	0.10 g	
0.34 g	0.56 g	0.10 g	
0.32 g	0.58 g	0.10 g	
0.30 g	0.60 g	0.10 g	

**Table 9:** Cholesteryl liquid crystal mixtures and their transition temperatures. Different compositions give different color patterns over different temperature ranges.

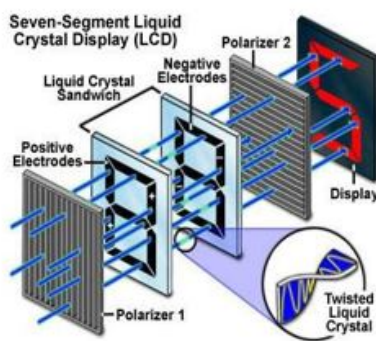
## References

1. Lisensky, G (2007). Preparation of cholesteryl ester liquid crystals. Interdisciplinary Education Group, Retrieved May 26, 2008.
2. Lisensky, G, & Boatman, E (2005). Colors in liquid crystals. *Journal of Chemical Education*, 82, 1360A, Retrieved June 4, 2008.

## Lab Part 2

**Liquid crystal display** (<http://www.microscopyu.com/articles/polarized/polarizedlightintro.html>)

One of the most common and practical applications of polarization is the liquid crystal display (LCD) used in numerous devices including wristwatches, computer screens, timers, clocks, and a host of others. These display systems are based upon the interaction of rod-like liquid crystalline molecules with an electric field and polarized light waves. The liquid crystalline phase exists in a ground state that is termed cholesteric, in which the molecules are oriented in layers, and each successive layer is slightly twisted to form a spiral pattern (Figure 13). When polarized light waves interact with the liquid crystalline phase the wave is "twisted" by an angle of approximately 90 degrees with respect to the incident wave. The exact magnitude of this angle is a function of the helical pitch of the cholesteric liquid crystalline phase, which is dependent upon the chemical composition of the molecules (it can be fine-tuned by small changes to the molecular structure).



**Figure 13:** Liquid crystal display

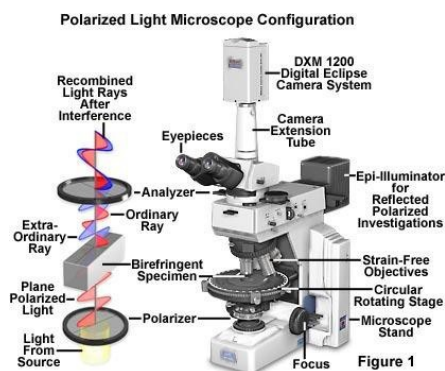
An excellent example of the basic application of liquid crystals to display devices can be found in the seven-segment liquid crystal numerical display (illustrated in Figure 13). Here, the liquid crystalline phase is sandwiched between two glass plates that have electrodes attached, similar to those depicted in the illustration. In Figure 2, the glass plates are configured with seven black electrodes that can be individually charged (these electrodes are transparent to light in real devices). Light passing through polarizer 1 is polarized in the vertical direction and, when no current is applied to the electrodes, the liquid crystalline phase induces a 90 degree "twist" of the light that enables it to pass through polarizer 2, which is polarized horizontally and is oriented perpendicular to polarizer 1. This light can then form one of the seven segments on the display.

When current is applied to the electrodes, the liquid crystalline phase aligns with the current and loses the cholesteric spiral pattern. Light passing through a charged electrode is not twisted and is blocked by polarizer 2. By coordinating the voltage on the seven positive and negative electrodes, the display is capable of rendering the numbers 0 through 9. In this example the upper right and lower left electrodes are charged and block light passing through them, allowing formation of the number "2" by the display device (seen reversed in the figure).

**Procedure** Liquid Crystal Transition Temperature Measurement by Optical Characterization

1. Heat the liquid crystal mixtures prepared in last lab

2. Prepare sample slides for the 5 compositions of liquid crystals (LC) prepared in Lab 1
  - (a) It will be helpful if you heat up your slide so that the LC mixture is still liquid when you carefully put a coverslip on top of it
  - (b) While placing coverslip try to avoid trapping as many bubble as possible in your sample
3. TA/instructor will provide instructions on how to operate the optical microscope and record your data
  - (a) The polarized light microscope is designed to observe and photograph specimens that are visible primarily due to their optically anisotropic character. In order to accomplish this task, the microscope must be equipped with both a polarizer, positioned in the light path somewhere before the specimen, and an analyzer (a second polarizer; see Figure 14), placed in the optical pathway between the objective rear aperture and the observation tubes or camera port. It is a contrast-enhancing technique exploits the optical properties specific to anisotropy and reveals detailed information concerning the structure and composition of materials that are invaluable for identification and diagnostic purposes.
  - (b) The liquid crystalline phase exists in a ground state that is termed cholesteric where the molecules are oriented in layers where each successive layer is slightly twisted to form a spiral pattern. When polarized light waves interact with the liquid crystalline phase the wave is "twisted" by an angle of approximately 90 degrees with respect to the incident wave. This angle is a function of the helical pitch of the cholesteric liquid crystalline phase, which is dependent upon the chemical composition of the molecules (it can be fine-tuned by small changes to the molecules).
  - (c) References: Useful resources are the Nikon web page describing polarized light microscopy and the Olympus page on polarization of light.



**Figure 14:** Polarized light microscope

You will generate movies of transitions observed in the liquid crystals with changing temperatures.

For the report please record observations of transition in structures from the different samples.