Replication of Surface Structures with Polydimethylsiloxane (PDMS Soft Lithography)

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Purpose:

The object of this module is to transfer micron-scale features from a hard solid surface onto a soft transparent surface, which can then be used as a transmission diffraction grating to produce a laser diffraction pattern for analysis.

Learning Objectives:

- Through an understanding of Fraunhofer diffraction students will be able to measure and verify the wavelength of a laser pointer.
- Through an understanding of Fraunhofer diffraction students will be able to measure density of grooves on a variety of surfaces.

Introduction:

Polydimethylsiloxane (PDMS) is a widely used silicone-based polymer cured by an organometallic crosslinking reaction that provides the material with its elasticity. PDMS may be produced in the form of a colorless, transparent elastomer with the ability to reproduce surface features.

This lab examines the ability of PDMS to reproduce surface features. The polymer is cured in contact with a diffraction grating and the features on a CD-R. These surface features are thereby imprinted into the elastomer and can be distorted mechanically.

The results of these distortions are then examined by Fraunhofer diffraction, which is based on the splitting of a wave into several outgoing waves after it has passed through slits or holes. The distance between any two of these outgoing waves depends on the wavelength of the wave, as well as the density of grooves, or the distance between the previously mentioned slits or holes. In this lab, this concept is used to determine the density of grooves in both the diffraction grating and the CD-R.

Materials:

- Laser pointer
  Innomate Technology Co., Ltd, #204826
- Optical Bench Set
  Nasco Science, #SB12243M, $8.60 ea.
  Note: Contains 2 metal supports for a meter stick, a lens support, and a lens screen support.
- 2 Replica Diffraction Gratings with 530 Grooves/MM
  The Welch Scientific Co., # 3813
- 8.5” x 11” piece of copy paper
- Ring stand

- Clamp
- Test tube holder
- Small ruler
- (2) metal dishes (1cm deep and 4.5cm in diameter)
  Note: The end caps used on some tube shaped shipping cartons work well.
- CD-R disk
- Scissors
- Large weighing boat
- Spoonula
- Wooden spoon
- Slygard 184 Silicone Elastomer Base
- Slygard 184 Silicone Elastomer Curing Agent
- Disposable pipette
- Oven (130° C)
- Tweezers

Procedure:

Preparing the Lithography Masters:

1) Obtain 2 metal dishes, a diffraction grating, and a CD-R.

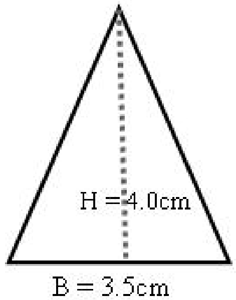
2) Using a high power microscope examine the diffraction grating, determine which side contains the grooves.

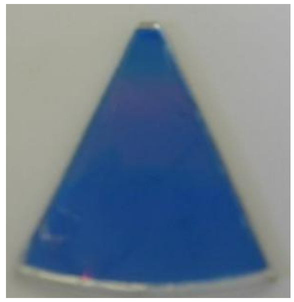
Note: Place a mark on the frame of this side of the grating for future use.

3) With the grooved side of the grating facing up, cut the frame off and place the remaining piece flat in one of the metal dishes.

4) Next, cut a “wedge” from the CD-R the equivalent of a triangle with a base of about 3.5cm and a height of about 4.0cm.

Note: Use regular scissors. They work best.



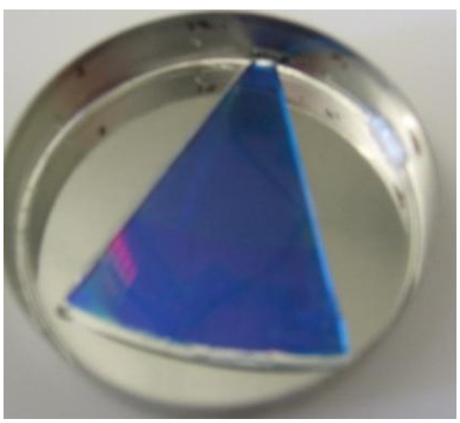


5) Carefully separate the aluminum foil layer from the polycarbonate support of the CD-R.

Note: Either may be used as the lithography master. It is recommended that the foil be used because the foil can be examined using a high power microscope in order to determine the actual groove density which can then be compared with experimental results.

6) Place one of the two layers into the remaining metal dish, with the grooved side up.

Note: The grooved sides of the two layers will be the two that were in contact with each other prior to separation.



Preparing and Using the PDMS:

7) Obtain a large weighing boat, a spoonula, and a wooden stick.

8) Weigh out about 8.00g of Sylgard polymer base into the weighing boat. Transfer the base to the weighing boat by placing the container next to the weighing boat and using the spoonula to scoop the base out of the container and into the weighing boat.

9) Then, using a disposable pipette, add approximately .80g of curing agent drop-wise (~ 40-45 drops) to the same weighing boat.

Note: The polymer base will settle across the bottom of the weighing boat. Distribute the drops of curing agent evenly across the entire surface of the polymer base.

10) Using the wooden stick, stir the base and curing agent in a smooth back and forth motion as to avoid bubbles.

Note: Bubbles will degrade the optical qualities of the cured PDMS.

11) Again using the spoonula, slowly scoop half of the PDMS (about 2.5g) into each of the metal dishes containing the lithography masters.

Note: Leave any remaining PDMS sticking to the walls of the weighing boat; too many bubbles will be created during attempts to remove it.

12) Let the set-ups sit at room temperature for a few minutes to allow any bubbles to rise to the surface. Using the wooden stick push these bubbles to the side and out of the PDMS.

13) Place the metal dishes in the oven at 130° C for 20 minutes.

14) Remove the dishes from the oven and allow them to cool.

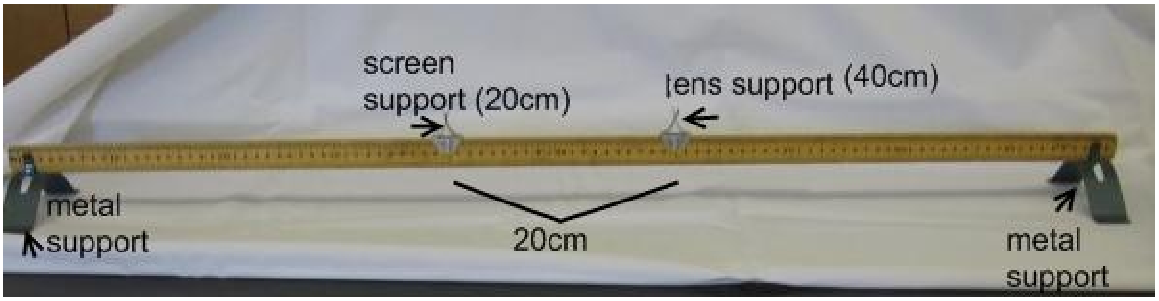
15) Clip the side a dish with a pair of tweezers. Move the tweezers around the edge of the dish while applying even pressure and keeping in contact with the PDMS layer. Continue this process until the PDMS becomes loose and is able to be removed from the dish.

Determining a Laser's Wavelength:

16) Obtain a laser pointer, a meter stick, an optical bench set, a diffraction grating and an 8.5" x 11" piece of copy paper.

17) Set the meter stick up on the two metal supports.

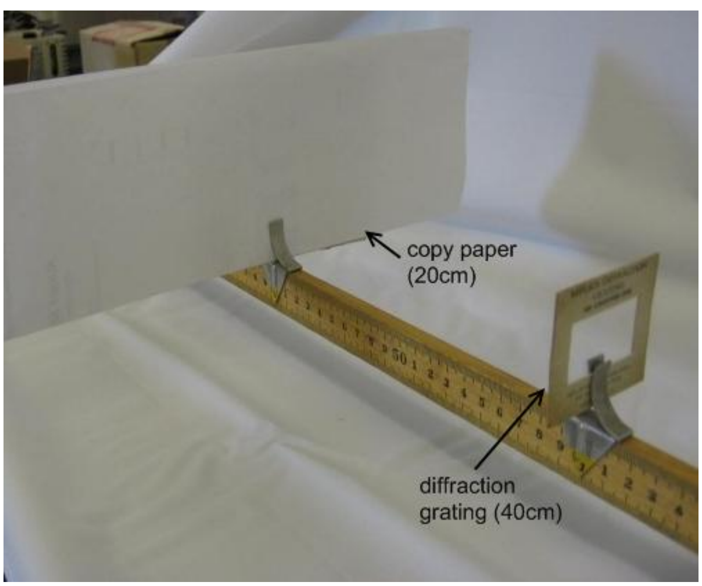
18) Place the lens support clip and the screen support clip on the ruler 20cm apart (i.e. screen support clip at the 20 cm mark and the lens support clip at the 40cm mark).



19) Fold the copy paper in half width-wise (so it's approximately 4.25" x 11").

20) Insert this folded piece of paper horizontally into the clip furthest to the left (i.e. the clip at the 20cm mark or the screen support clip).

21) Now insert the diffraction grating into the other clip (i.e. the clip at the 40cm mark or lens support clip). Make a note of the density of grooves in the diffraction grating. This information will be located on the diffraction grating frame.



Density of grooves: ________grooves/cm.

Now calculate the spacing between individual grooves:

Distance between two grooves: ________

22) Next obtain a ring stand, a clamp, and a test tube holder. Clamp the test tube holder onto the ring stand and place the laser pointer in the test tube holder.

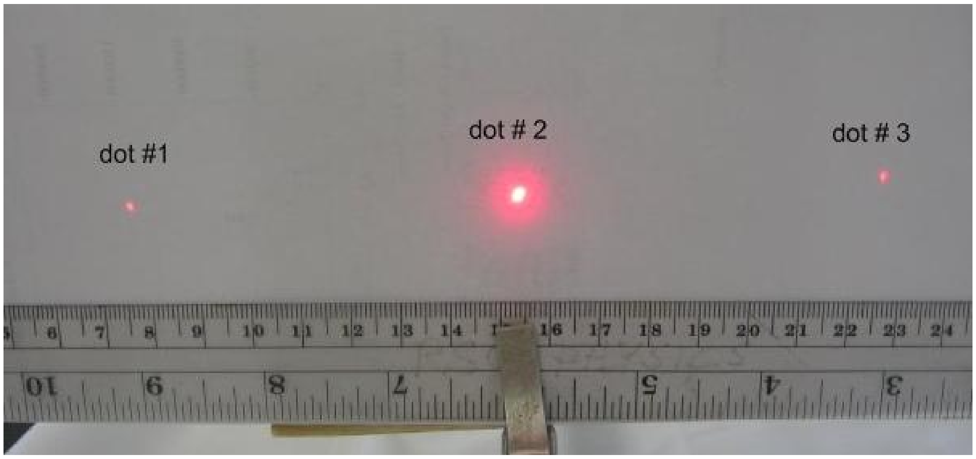
23) Place this set-up 20cm to the right of the clip furthest to the right (i.e. the clip at the 40cm mark, so place the ring stand at 60cm.)

24) Position the laser pointer in the test tube holder so that the holder itself presses the power button and keeps the laser turned on.

25) Finally, move the clamp and test tube holder until the light from the laser passes through the diffraction grating and can be seen on the copy paper.

Hint: You should see an array of 3 red dots.

26) Once the laser is correctly positioned, slip a ruler in front of the copy paper in the same clip.



27) Record the location (in cm) of the 3 dots on the ruler.
Note: The starting point does NOT matter.

Location of dot #1: ________cm

Location of dot #2: ________cm

Location of dot #3: ________cm

28) The distance between each dot can then be determined by simply finding the difference between each location.

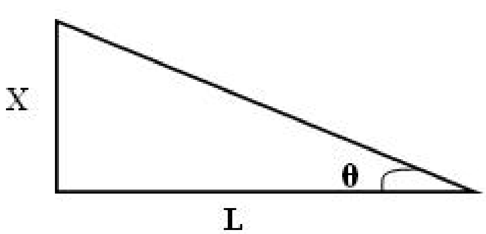
Distance between dots #1 and #2: ________cm

Distance between dots #2 and #3: ________cm

29) Next find the average of those two distances.

Average distance between two dots: _________cm

30) Now the angle of diffraction, θ, must be determined. This can be done by creating a triangle with the length (L) being equal to the distance between the two clips (20cm) and the height (X) being equal to the average distance between two dots as recorded in Step 13.



Note: $\tan \theta = X/L$

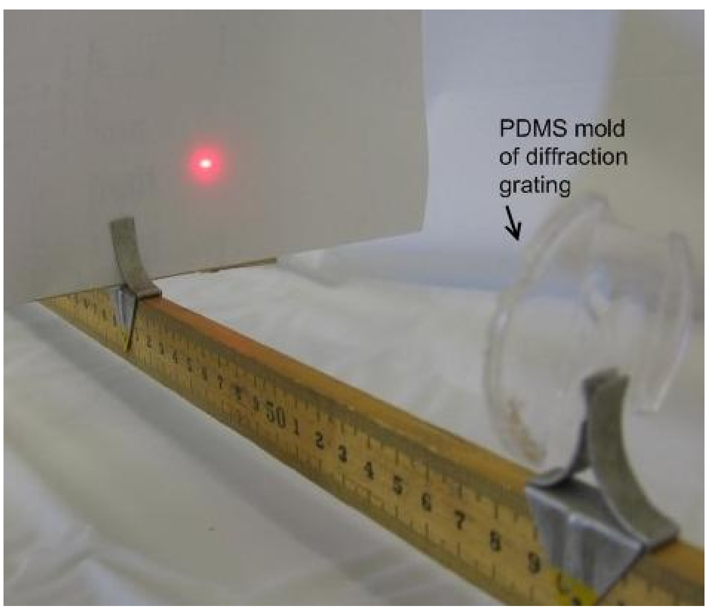
Calculated θ: ________°

31) Use the Fraunhofer equation, $d \sin \theta = \lambda$, to solve for the wavelength (λ). The distance (d) is the distance between grooves in the diffraction grating and was recorded in Step 21. The angle θ is the diffraction angle calculated in the previous step.

Calculated wavelength (λ): ________

Determining Density of Grooves:

32) Create the same set-up as described in the procedure for Determining a Laser's Wavelength (Steps 16-31). However, this time the PDMS mold of the diffraction grating will replace the diffraction grating itself.



33) Once everything is in place record the location (in cm) of the 3 dots on the ruler just as before.

Location of dot #1: ________cm

Location of dot #2: ________cm

Location of dot #3: ________cm

34) The distance between each dot can then be determined by simply finding the difference between each location.

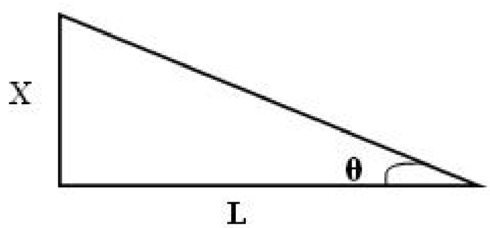
Distance between dots #1 and #2: ________cm

Distance between dots #2 and #3: ________cm

35) Next find the average of those two distances.

Average distance between two dots: _________cm

36) Now the angle of diffraction, θ, must be determined. This can be done by creating a triangle with the length (L) being equal to the distance between the two clips (20cm) and the height (X) being equal to the average distance between two dots as recorded in Step 35.



Note: Tan θ = X/L

Calculated θ: ________°

37) Use the Fraunhofer equation, $d \sin \theta = \lambda$, to solve for the density of grooves (*d*). The wavelength (λ) is the wavelength of the laser pointer as calculated in Step 31. The angle θ is the diffraction angle calculated in the previous step.

Density of grooves (*d*): ________

38) Slowly and gently stretch the PDMS mold.

    How does the diffraction pattern change?

39) Hold the PDMS mold up to the light as you would with a diffraction grating. Does is work the same way?

40) Replace the mold of the diffraction grating with the mold of the CD-R and repeat steps 32 – 38.

    Location of dot #1: ________cm

    Location of dot #2: ________cm

    Location of dot #3: ________cm

    Distance between dots #1 and #2: ________cm

    Distance between dots #2 and #3: ________cm

    Average distance between two dots: _________cm

Calculated θ: ________°

Density of grooves (d): ________

41) Slowly and gently stretch the PDMS mold.

How does the diffraction pattern change?

Discussion Questions:

1) The accepted value for the wavelength of the laser pointer is 645nm. Based on the observed (or experimental) value calculate the percent error in this procedure.

2) Using the actual value for the density of grooves of the diffraction grating noted in Step 21 and the observed (or experimental) value found in Step 38 calculate the percent error.

3) Explain the changes in diffraction grating patterns that occur when the PDMS molds of the diffraction grating and the CD-R surface patterns are stretched.

4) The same set-up described in Steps 31 – 36 was used to calculate the groove density of a PDMS mold of a diffraction grating. The laser pointer used had a wavelength of 633nm. The distance from the diffraction grating to the sheet was 20cm and the average distance between two spots was measured to be approximately 6.82 cm. What is the density of grooves?

References:

Fishbane, P. M., S. G. Gasiorowicz, & S. T. Thorton (2005). *Physics for scientists and engineers with modern physics*. Upper Saddle River, New Jersey: Pearson Education Inc.

Lisensky, G. (2007, May, 15). *Replication of surface structures with polydimethylsiloxan. Interdisiplinary Education Group*, Retrieved June 4, 2008, from http://www.mrsec.wisc.edu/Edetc/nanolab/PDMS/text.html

Teachers Key:

Discussion Questions:

1) Answers may vary.
   Percent error = $\frac{\text{Actual} - \text{Experimental}}{\text{Actual}} \times 100$

2) Answers may vary.
   Percent error = $\frac{\text{Actual} - \text{Experimental}}{\text{Actual}} \times 100$

3) As the molds of the patterns are stretched, the observed spots get closer and closer together. This is because as the mold is stretched it causes the grooves to get further apart thereby lowering the density of grooves (*d*). The lower the density of grooves, the smaller the angle of diffraction (θ), which causes the spots to be closer together.

4) Tan θ = X/L
   Tan θ = 6.82cm/20.0cm
   Tan θ = 0.341
   θ = 18.8° = angle of diffraction

   $d \sin \theta = \lambda$
   $d \sin (18.8°) = 633\text{nm} = 6.33 \times 10^{-5}$ cm
   $0.322\, d = 6.33 \times 10^{-5}$ cm
   $= 1.97 \times 10^{-4}$ cm = density of grooves

Suggestions for lab placement:

It could be used in the Organic Chemistry lab because you're synthesizing a polymer and examining its optical properties.

However, if the PDMS were to be pre-made, it would fit better into the Physics 101/102 lab due to the concepts of density of grooves and calculations based on the Fraunhofer equation.

Supplies List (For 10 Students):

- 10 Laser pointers
  Innomate Technology Co., Ltd, #204826
- 10 Optical Bench Sets
  Nasco Science, #SB12243M, $8.60 ea.
  Note: Contains 2 metal supports for a meter stick, a lens support, and a lens screen support.
- 20 Replica Diffraction Grating with 530 Grooves/MM
  The Welch Scientific Co., # 3813
- 10 8.5" x 11" piece of copy paper
- 10 Ring stands
- 10 Clamps
- 10 Test tube holders
- 10 Small rulers
- 20 metal dishes (1cm deep and 4.5cm in diameter)
  Note: The end caps used on some tube shaped shipping cartons work well.
- 2-3 CD-R disks
- Scissors
- 10 Large weighing boats
- Spoonulas
- Wooden spoons
- Slygard 184 Silicone Elastomer Base
- Slygard 184 Silicone Elastomer Curing Agent
- Disposable pipette
- Oven (130° C)
- Tweezers