Chapter Two - Probing the Structure of Materials at the Nanoscale

Building or disassembling an instrument built from a familiar material such as LEGO® bricks provides a revealing view of the instrument and the principles behind it.

**NOTE:** LEGO® brick dimensions will often be described by numbers of pegs on their top surfaces. For example, a 2x4 brick is 2 pegs wide and 4 pegs long.

### 2.1 - Scanning Probe Microscopy

Scanning probe microscopy (SPM) is a method for mapping surface forces of materials on the atomic scale. By mapping these forces, much can be learned about the surfaces of materials, where many interesting and complex phenomena occur. For example, many chemical reactions involving solids are dependent on the nature of their surfaces. Scanning probe microscopy includes the methods of atomic force microscopy (AFM), magnetic force microscopy (MFM), and lateral force microscopy (LFM). Most force microscopy techniques are variations of the same basic principle, illustrated in Figure 2.1. Forces between the surface and a cantilever tip cause the tip to deflect up and down. Deflection of the cantilever shifts the position of a laser beam that reflects off the top of the cantilever onto a photodiode array. The movement of the beam between the photodiodes is used to calculate the cantilever deflection.

![Diagram of scanning probe microscopy](image)

**Figure 2.1** - The general principle behind force microscopy: Forces between the surface and the cantilever tip cause the tip to be deflected upward and downward. Deflection of the cantilever shifts the position of a laser beam that reflects off the top of the cantilever onto a photodiode array. The movement of the beam is tracked by the photodiodes and used to calculate the cantilever deflection.

**Figure 2.2** shows a model of an atomic force microscope built from LEGO® bricks. The AFM contains a laser pointer and a cantilever with a triangular LEGO® probe on its underside and a mirror atop it. As in a real AFM, the cantilever is held in place and the surface is moved back and forth underneath the probe. In this model, the cantilever tip is
in physical contact with the substrate, so the pegs on the LEGO® substrate surface can be taken to represent a square array of surface atoms. The interaction between the probe tip and the surface results in deflection of the cantilever. Light from the pocket laser reflects from the mirror on the cantilever and shines onto a wall or screen. The farther the wall is from the AFM, the more the cantilever deflection will shift the beam spot on the wall.

![Figure 2.2 - A LEGO® AFM. Substrate motion is perpendicular to light path. See Section 2.1 Appendix for building instructions.](image)

Modification of the model by addition of LEGO® magnets or other magnets converts it to a magnetic force microscope model, Figure 2.3. Here, the refrigerator magnet at the end of the cantilever interacts with a refrigerator magnet taped to the LEGO® surface to alternately attract and repel the cantilever.

![Figure 2.3 – A LEGO® MFM. Substrate motion is perpendicular to light path. See Section 2.1 Appendix for building instructions.](image)
2.2 – Charge-Coupled Devices

A charge-coupled device (CCD) is a camera that produces digital images instead of conventional film images. The digital format allows the images to be manipulated by a computer, which can electronically sharpen, modify, or copy them.

CCDs are used to take pictures of very faint stars, and to make accurate measurements of position and therefore of speed and acceleration. They also serve as detectors in X-ray diffraction experiments, which provide information on the relative positions of atoms in materials and the sizes of nanoscale particles. In each case, the large detection area allows a great deal of information to be collected simultaneously.

In filmmaking, CCD cameras are useful for adding special effects. Previously, scenes were shot on conventional film and then scanned into a computer. This process is slow, and image quality is lost in the process. With a CCD, the scene is placed directly onto a computer for editing.

A CCD relies on semiconductor properties for its operation. Basically, photons of certain frequencies of light (electromagnetic radiation) strike a layer of silicon, breaking electrons away from chemical bonds. A CCD collects and counts these electrons to determine how many photons were absorbed by the silicon.

Every CCD starts with a backing of some sort, usually glass. The backing is then covered with metal electrodes, as shown in Figure 2.4. The bottom row of electrodes is designated as the readout register.

![Figure 2.4 – Electrode array of a CCD.](image-url)
A thin layer of silicon dioxide is placed over the electrodes, followed by two layers of silicon. The purpose of the silicon dioxide is to separate electrically the silicon from the electrodes and keep the electrons in the silicon. The two silicon layers contain small concentrations of other elements. This creates an electronic boundary called a p-n junction, allowing an electrical current to travel efficiently through the silicon. Between each column of electrodes in the silicon layer there is a channel stop, indicated by dashed lines in Figure 2.4. Channel stops prevent electrons from flowing horizontally across the CCD array. There are no channel stops in the readout register; in this row, charges are free to move horizontally to the detection device. A cross section of a portion of a CCD is shown in Figure 2.5.

![Figure 2.5 – Cross section of a portion of a CCD.](image)

Each CCD is divided into many groups of electrodes called pixels. The exact number of pixels depends on the individual pixel size and the cost of the array. The CCD cannot differentiate between a photon of red or blue colored light, because it can only count numbers of electrons. In order to provide color images, the silicon above a group of electrodes is covered with red, green, and blue colored filters and combined to make a single color pixel.

Many photons of light are absorbed by the silicon layer as they enter the CCD. Each absorbed photon breaks electrons away from chemical bonds. Next, all of the collected electrons are sequentially moved to the detection device where they are counted. This is accomplished by changing the charge of the electrodes under the silicon layers in a timed, sequential manner.

To illustrate using Figure 2.6, electrodes 2 and 5 are initially positively charged (the red electrodes in the left-hand part of diagram) and therefore collect all the electrons photogenerated around them. Next, electrodes 3 and 6 gradually begin to acquire positive charge while 2 and 5 gradually become negatively charged. This causes the electrons to move to electrodes 3 and 6 (Figure 2.6, middle). Then, electrodes 1, 4, and 7 gain positive charge while 3 and 6 become negatively charged This causes the electrons to move once more (Figure 2.6, right-hand part). The electrons from the bottom-most pixel (pixel #2) are now in the readout register, electrode 7.
The readout register now begins to deliver the electrons along the bottom of the CCD to the detection device by the same process of varying the charge of its electrodes. The detection device counts the number of electrons by applying a known voltage across the final electrode. (The final electrode is located inside the detection device, not on the CCD array.) The voltage will increase slightly as electrons arrive from the CCD. By subtracting the background voltage, the voltage from the CCD electrons can be found. The number of electrons can then be calculated by a computer.

![Diagram of electrode charges](image)

Figure 2.6 - An illustration of moving electrons by changing charges within a single column of electrodes in a CCD.

A LEGO® model of a CCD is shown in Figure 2.7. The table tennis ball simulates an electron while the colored bars simulate the electrodes. In this model, bar height is inversely proportional to voltage: as the bar drops down the voltage becomes more positive. By pushing and releasing the levers in sequence the bars drop and rise in sequence, moving the ball to the ramp at the left that represents the readout register.

![LEGO® model of a CCD](image)

Figure 2.7 - This CCD model demonstrates how changing charge on the electrodes causes the electrons to move. See Section 2.2 Appendix for building instructions.
2.3 - Photometry

Photometry is a widely used tool for characterizing the electronic structure of materials and determining the amount of matter present. Electrons are the "glue" that holds atoms together at the nanoscale, and their properties often become evident through the interaction of matter with light.

Photometry refers to qualitative and quantitative aspects of the interaction of matter with light. Qualitatively, absorption of light corresponds to inducing electronic transitions between energy levels of an atom, ion, molecule or material. The separation in energy levels corresponds to the portion of the electromagnetic spectrum that is absorbed. This technique is thus used for solid materials as well as for liquids and gases. Quantitatively, a larger path length and/or higher concentration of matter will lead to absorption of a greater fraction of incident light.

The essential components of a photometer are a light source, a sample, and a light detector, shown in Figure 2.8. Most photometers also contain some means to select a relatively narrow spectral region of light such as orange light. The detector measures the intensity of the light that is reaching it. Without the sample in the light beam to absorb some of the light, the light intensity reaching the detector is defined as $I_0$. When the sample is in place to absorb some of the light, the intensity of light reaching the detector is defined as $I$. Therefore the transmittance ($T$), which is the fraction of original light that passes through the sample, is defined as:

$$T = \frac{I}{I_0}$$

Another unit of measurement used in photometry is absorbance ($A$), defined as:

$$A = -\log T$$

Figure 2.8 – General schematic of a photometer.

Figure 2.9 below shows a working photometer built from LEGO® bricks. It can be built or taken apart to provide an understanding of the basic principles of spectroscopy. This photometer may be built with light sources and sensors available through the LEGO®
Corporation. The sensor may be interfaced with a computer to read the light transmitted through the sample. The only non-LEGO® piece in this model is the sample cell, which consists of a conventional plastic spectrophotometer cuvette glued to a standard 2x2 LEGO® brick in order to fasten it to the LEGO® board underneath. Notice that this model also includes a shutter to regulate the beam intensity and shape. The components are placed in a casing to minimize stray light from other sources. The dummy sensor marks an alternate position for the light sensor.

One way in which photometers may be used to find chemical concentrations is through a calibration curve. To make the calibration curve, the absorbances of a number of samples with known concentrations of chemicals are measured. These samples are called standards. The absorbances of these standards are plotted as a function of their known chemical concentration, resulting in a line or curve like that shown in Figure 2.10. When an unknown sample is measured, its absorbance may then be used to calculate its chemical concentration.

Figure 2.9 - A LEGO® photometer. See Section 2.3 Appendix for building instructions.

Figure 2.10 - A calibration curve. The points are calibration data used to construct the curve.
A calibration curve can be modeled with LEGO® bricks. Stacks of different numbers of bricks can be arranged in order of height. The height of each stack can be correlated to the different numbers of bricks. This can be plotted with height on the y-axis and number of blocks on the x-axis. Then when another stack (of a different color to signify that it is an unknown) is given to the students, they can see that it is the same height (y value) as the stack with the same number of bricks (x value). Examples of LEGO® graphs are shown in Figure 2.11. The left curve describes a linear graph and its use as a calibration curve. The middle curve describes a first-order decay, where each column of bricks is half the height of the previous column of bricks. This can be representative of processes such as radioactive decay, in which the concentration of a particular radioactive element decreases by a factor of two for constant time intervals (half-lives). The right curve describes an exponential growth, where each column of bricks is ten times the height of the previous column of bricks. This has been used as a demonstration of solution pH, in which each change in pH by one unit represents a ten-fold change in hydrogen ion concentration in solution.

![Image of LEGO brick setup](image)

Figure 2.11 – (left) A linear calibration curve modeled with 55 1x1 LEGO® bricks. (middle) A curve of 255 1x4 LEGO® bricks describing first-order decay. (right) A curve of 111 1x4 LEGO® bricks describing exponential growth. The top bricks of the columns in the middle and right curves have different-colored bricks for visibility and the tallest column in each is taped to a wall for stability.

Different types of light measurements may be taken if the detector is in the 90° geometry, as shown in Figure 2.12. In this arrangement, the detector measures light leaving the sample at an angle of 90° from the source light beam. Therefore, the detector does not measure any light shining directly through the sample from the source. This geometry allows measurements of effects such as fluorescence and light scattering. These techniques may also use calibration curves such as the one shown in Figures 2.10 and 2.11, although the Y-axis data are light intensity rather than absorbance. The light
detector in the LEGO® model shown in Figure 2.9 above can be mounted in a 90⁰ geometry by switching its position with the dummy sensor.

Figure 2.12 - The 90⁰ geometry for light scattering measurements

One of the many uses of a photometer in a 90⁰ geometry is turbidimetry, which measures turbidity, or cloudiness. When light passes between two substances (such as water droplets in air, or mud particles in water) some of the light is scattered. More particles provide more opportunities for light to pass between substances, and more light is scattered. Therefore, even though water and air are both transparent, light can be scattered so much by the air/water boundary of water droplets that it may be difficult to see on a foggy day. Turbidity also has environmental implications. When soil is washed into a waterway, the water can become turbid, affecting plants and animals. Turbidimetry may be used to monitor water cloudiness due to soil erosion.

### 2.4 – Nuclear Magnetic Resonance

Nuclear magnetic resonance (NMR) is a technique used to determine the structures of chemical compounds. Many atomic nuclei have their own magnetic fields, as if they were tiny bar magnets. These magnetic nuclei are referred to as magnetic “spins”. When the magnetic nuclei are placed in the presence of a magnetic field, they will either align their nuclear spins with or against the magnetic field. By irradiating the nuclei with a pulse of electromagnetic radiation perpendicular to the magnetic field the nuclear spins will first align themselves with the pulse and then rotate in a plane perpendicular to the applied magnetic field. The frequency of this rotation, called resonance, is proportional to the strength of the applied magnetic field that the nucleus experiences. Isolated nuclei of the same element would all experience the same magnetic field and thus have the same resonant frequency. However, each nucleus in a chemical compound is in a slightly different magnetic environment because it is in a slightly different chemical environment. Therefore, magnetic nuclei in chemical compounds may experience different magnetic
fields and thus have different resonant frequencies. The detector in the NMR identifies the different resonances and translates them into a spectrum that allows the user to determine the chemical structure.

A simple model of a pulsed NMR experiment built with LEGO® bricks can be used to illustrate the idea of resonance. The swiveling magnets represent the nuclear spins, and the stationary magnetic array represents the applied external magnetic field. Adjusting the height of the stack of bricks upon which the swiveling magnets are placed can change the distance between the magnets. In particular, increasing the height of the stack of bricks decreases the strength of the stationary magnetic field felt by the swiveling magnets.

The mechanical analogy to NMR described in Figure 2.13 requires 10 flat face-poled LEGO magnets all in the same magnetic orientation, 5 swiveling face-poled magnets, and stacks of one to five conventional 1x1 bricks. Normally, the swiveling magnets will orient themselves in the same direction as the stationary magnets.

Manually rotating the swiveling magnet 90° out of position represents a pulse of electromagnetic radiation that rotates the nuclear spins. When the rotated magnet is released, it will oscillate as it swings back into alignment with the stationary magnetic field. Swiveling magnets that are placed closer to the permanent magnetic array will have higher frequency oscillations as they realign with the stronger applied magnetic field. The oscillation of the swiveling magnets is analogous to the resonant frequency of the nuclear spins, which also increases as the applied magnetic field increases.
2.5 – **Chromatography**

Chromatography is a widely used technique for the separation of chemical components in a mixture. The technique involves moving the mixture, called the mobile phase, over or through a solid, high surface area material called the stationary phase. The stationary phase will slow the rate of flow differently for different chemical components in the mobile phase. Thus two components that are combined at the beginning of the process will leave the stationary phase at two different times. In the LEGO® model of this process, described in Figure 2.15, a column of stationary phase material is represented by a trough with the sides comprised of 2x8 bricks and the back 2x4 bricks. The packing material is represented by 1x8 bricks, the same length as the sides of the column, that are placed randomly along the trough. As you insert molecules, represented by different size bricks, into the top of the column, they are slowed down by the stationary phase. Larger bricks will slow down more and therefore come out of the column last. This is analogous to the adsorption and desorption of real species to the material of the stationary phase. A greater affinity for the stationary phase is modeled by an increase in size of LEGO® bricks. Some shaking of the column may be needed in order to remove all of the bricks from the column. Shaking can be analogous to pressure forcing the mobile phase through a real chromatography column. Along with many other uses, chromatography has been used to separate different sizes of nanoscale particles.

![Figure 2.15](image)

**Figure 2.15** – (left) Two LEGO® “molecules” at the top of a trough representing a chromatography column. (right) The smaller brick leaves the trough first.

<table>
<thead>
<tr>
<th>Type of LEGO® brick</th>
<th>Number of LEGO® bricks</th>
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<tbody>
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<tr>
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<tr>
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</tr>
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</tbody>
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2.6 – **Diffraction and Wave Addition**

A variety of methods for studying patterns in materials is based on the principles of diffraction. These methods are based on the scattering of waves from various locations within the structure of the material. These waves can be those of electromagnetic radiation, such as light or X-rays, or subatomic particles that have some wavelike
character, such as electrons and neutrons. The way the waves scatter from the material and interact with each other can be measured and used to elucidate the material structure. When two or more sets of waves cross paths, they combine together in ways that can produce more intense or less intense resulting waves, sometimes even canceling each other out. Figure 2.16 illustrates this principle with 1 peg x 1 peg bricks arranged on a flat base board. This arrangement uses the base board as a sort of graph paper and the bricks represent the ink for drawing graphs. The oscillating patterns at the left side of the board represent sets of waves that are crossing paths and will be added together. The yellow lines represent the center of the oscillation. On graph paper this would represent the x axis (y = 0) for each wave. The crests of the waves are shown with red bricks and are located above the yellow line. The troughs of the waves are shown with white bricks and are located below the yellow line. A wave crest and trough together constitute one wave. The distance along the x axis from a point on a wave to the same point on the next wave is called a wavelength. The distance between the height and depth of the wave is referred to as its amplitude. Figure 2.16 represents the combination of two sets of waves with the same wavelength and amplitude. These sets of waves are also “in phase” – the waves match crest to crest and trough to trough. The easy addition of bricks to the boards and their easy removal allows for a rather quick summing of the waves to produce the resulting wave pattern at the right side of the board. For each column of pegs in the left side sets of waves, add up the number of red bricks and subtract all of the white bricks. For example, the first column of pegs has four red bricks and no white bricks. Therefore the first column of pegs for the waves on the right side should have four red bricks. The sixth column of pegs has no red bricks and eight white bricks. Therefore the sixth column of pegs for the waves on the right side should have eight white bricks. Note that the resulting set of waves has the same wavelength as the originals, but double the amplitude. This summing of waves to produce waves of larger amplitude is called constructive interference.

<table>
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<tr>
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</thead>
<tbody>
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</tr>
<tr>
<td>1x1 white bricks</td>
<td>96</td>
</tr>
<tr>
<td>2x2 yellow bricks</td>
<td>30</td>
</tr>
<tr>
<td>1x1 yellow brick</td>
<td>1</td>
</tr>
<tr>
<td>32x32 baseplate</td>
<td>1</td>
</tr>
</tbody>
</table>

Figure 2.16 – Combination of two sets of waves that are “in phase” with each other, resulting a set of waves with double the amplitude.
Figure 2.17 represents the combination of two waves with the same wavelength and amplitude that are also completely “out of phase” – the waves match crest to trough and trough to crest. These waves cancel each other out completely, which can be shown by counting the bricks. For example, the first column of pegs has two red bricks and two white bricks. Therefore the first column of pegs for the waves on the right side of the board should have no red or no white bricks. The sixth column of pegs has four red bricks and four white bricks. Therefore the sixth column of pegs for the waves on the right side of the board should have no red or no white bricks. Note that the resulting waves have no amplitude and therefore does not exist. This summing of waves to produce waves of smaller amplitude or to completely cancel each other out is called destructive interference.

<table>
<thead>
<tr>
<th>Type of LEGO® brick</th>
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<tr>
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</tr>
<tr>
<td>1x1 white bricks</td>
<td>48</td>
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<tr>
<td>2x2 yellow bricks</td>
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<td>1x1 yellow brick</td>
<td>1</td>
</tr>
<tr>
<td>32x32 baseplate</td>
<td>1</td>
</tr>
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Figure 2.17 – Combination of two sets of waves that are completely “out of phase” with each other, resulting in the waves completely canceling each other out.

Figure 2.18 represents the combination of two waves with the same wavelength and amplitude that are only partially “out of phase” – the waves do not match completely nor cancel each other out completely, which can again be shown by counting the bricks. For example, the first column of pegs has six red bricks and no white bricks. Therefore the first column of pegs for the waves on the right side of the board should have six red bricks. The sixth column of pegs has no red bricks and six white bricks. Therefore the sixth column of pegs for the waves on the right side of the board should have six white bricks.

<table>
<thead>
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<th>Type of LEGO® brick</th>
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<tbody>
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<td>1x1 red bricks</td>
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</tr>
<tr>
<td>1x1 white bricks</td>
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<td>2x2 yellow bricks</td>
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<td>1x1 yellow brick</td>
<td>1</td>
</tr>
<tr>
<td>32x32 baseplate</td>
<td>1</td>
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</table>

Figure 2.18 – Combination of two sets of waves that are partially “out of phase” with each other, resulting in a new, different set of waves.
Waves that are not in the same plane can also be combined. Circularly polarized light can be considered to be the sum of two electric field waves at 90° angles to each other and partially out of phase with each other. The resulting light can be described as having a spiraling electric field. Figure 2.19 represents the combination of two waves that are at 90° angles to each other and partially out of phase.

<table>
<thead>
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<th>Type of LEGO® brick</th>
<th>Number of LEGO® bricks</th>
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<td>1x2 bricks</td>
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<td>1x4 bricks</td>
<td>16</td>
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<tr>
<td>1x1 flat bricks</td>
<td>8</td>
</tr>
<tr>
<td>2x2 flat bricks</td>
<td>8</td>
</tr>
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</table>

Diffraction methods use waves to find the repeating arrangements of atoms in crystal structures (see Section 1). This is most commonly done by shining a beam of X-rays at a crystal of material. When the X-ray waves encounter the atoms in the crystal they scatter in various directions. When waves scattered from different atoms encounter each other they can interfere constructively in some places and destructively in others to produce a diffraction pattern. This pattern of X-ray variation can be measured with detectors and can be used to calculate the positions of atoms in the crystal structure. Though X-ray diffraction is most common, other waves can be used to produce diffraction patterns. Figure 2.20 shows a visible light diffraction pattern produced by shining a laser through a structure containing regions of polymer spheres in a face-centered cubic arrangement. A relatively simple model of the face-centered cubic structure and its unit cell is described in the Section 1.2 Appendix.
Often water comes to mind when waves are mentioned, and water has long been used in so-called wave tanks to demonstrate principles of diffraction. Figure 2.21 shows a wave tank made from LEGO® bricks in conjunction with a flat pan filled with water. The system uses long straight waves generated by oscillating a flat wall of LEGO® bricks that is dipped partway into the water. The waves produced by the paddle can be scattered by barriers made from LEGO® bricks. The barriers are made from bricks with their pegs oriented sideways, enabling them to be sunken more easily in water.

Figure 2.21 – (top) A wave tank made using LEGO® bricks and a flat pan filled with water. A long, straight wave can be produced by oscillating a flat wall of LEGO® bricks. The wall can be oscillated up and down by using a LEGO® machine or by hand. (bottom) Interference between sets of circular waves scattered from two gaps in the 2x4 LEGO® brick barrier. The water level is lower than the bricks oriented vertically but higher than the bricks in the gaps that are oriented horizontally.
2.7 - Scanning Electron Microscopy

An electron microscope uses electrons, rather than light (photons), to obtain magnified images of a sample. There are many different electron microscopy techniques, including scanning electron microscopy (SEM). In SEM, a beam of electrons is scanned back and forth across a surface, producing a variety of electrons and X-rays that can be used to analyze the structure of that surface. One of the most common SEM techniques involves detecting electrons that have been bumped away from sample atoms by the microscope electron beam. These secondary electrons, as they are called, are produced both at the surface and under the surface of the sample. On areas of the sample that face the electron beam, only the secondary electrons produced at the surface escape the sample and are detected. At the edges of the sample that do not directly face the electron beam, the secondary electrons produced under the surface can also escape the sample and are also detected. Increased numbers of electrons reaching the detector in the SEM become brighter areas of the sample image that the microscope produces. The resulting sample images show features that appear to be lit from the side – brighter at their edges than where they face the electron beam.

This edge effect can be modeled with simple household items:

- firm, flat surface (like a table top)
- adhesive tape
- white crayon
- flat LEGO® bricks
- black paper

Tape the bricks to the flat surface to prevent the bricks from shifting. Place the paper over the bricks. Rub the white crayon lightly over the paper over the area where the bricks are taped. The crayon marks will cover all of the paper to some extent, but will be heaviest at the edges of the object under the paper, similar to secondary electron emission being greatest at the edges of samples in the SEM, Figure 2.22.

In this demonstration, the piece of white crayon represents the electron beam of the microscope scanning across a sample surface. The crayon marks on the paper represent the secondary electrons produced by the sample, and the image on the black paper resembles an image that would be produced by an electron microscope.

Figure 2.22 – (top) Flat LEGO® bricks taped to a flat surface in the pattern of the letters “FNT”. (bottom) White crayon marks rubbed on the black paper picks up the pattern of the pegs.