The Design and Construction of the Houghton X-ray Diffractometer

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I. Abstract

A Braggs-Brentano θ-2θ X-ray diffractometer is being constructed at Houghton College to map the microstructure of textured, polycrystalline silver films. A Phillips-Norelco X-ray source will be used in conjunction with a 40kV power supply. The motors for the motion of the θ and 2θ arms, as well as a Vernier Student Radiation Monitor and other safety monitors, will all be controlled by a program written in LabVIEW.

II. History of X-ray Diffraction

In 1914, Max Von Laue received the Nobel Prize for Physics for his discovery of the diffraction of X-rays on crystals. In his Nobel lecture titled "Concerning the detection of X-ray interferences", he discussed his idea of the ability for X-rays to penetrate a crystalline lattice structure. This is possible because the spacing between the atomic layers of a crystal is on the same order of magnitude as the wavelength of the X-rays. If the wavelength of the X-rays was larger than the spacing between the atomic layers of the crystal, then they would simply reflect off the surface. Von Laue constructed a mathematical theory of diffraction by a 3D crystal lattice. While analyzing his theory further, he tried to account for the fact that out of a large number of possible diffraction directions only certain directions occurred. He suggested that the X-ray source may be the reason for this, where as William Henry Bragg and William Lawrence Bragg believed the answer to be in the crystalline structure. The father-son duo theorized that at certain angles the x-rays would reflect and constructively interfere, otherwise they would destructively interfere. This gave way to Bragg's law. Since the contribution of Bragg's family, diffraction has mainly been used to find the plane spacing in various crystals. This distance can be used to determine the orientation of the different crystals in a sample, furthering the understanding of the structure of a crystal. A few different methods of diffraction have been engineered to properly calculate this distance, one of them including the Braggs-Brentano θ-2θ X-ray diffractometer.

III. Theory

Bragg's Law

Since X-rays are on the same order of magnitude as the spacing of the crystalline planes they can be used to resolve the plane spacing. If a wave front of X-rays of wavelength λ reflects off of a crystalline structure at an angle θ, θ being the incident angle and the reflected angle, some X-rays reflect off of the first plane while others reflect off of the second plane, and so on. If the X-rays constructively interfere, the X-rays that travel to the second plane travel an integer multiple of the wavelength, $n\lambda$, farther than the X-rays that reflect off the first plane. Figure 1 gives a basic representation of what is described above. From this, Bragg's law is derived as

$$n\lambda = 2dsin\theta \tag{1}$$

where n is an integer, λ is the wavelength of the X-rays, d is the spacing of the crystalline planes, and θ is the angle, up from the horizontal, that the x-rays strikes the crystal lattice at. This shows that the extra distance traveled by the X-rays that hit the second plane is dependent on the spacing of the planes and the angle at which the X-rays enter the crystalline structure at.

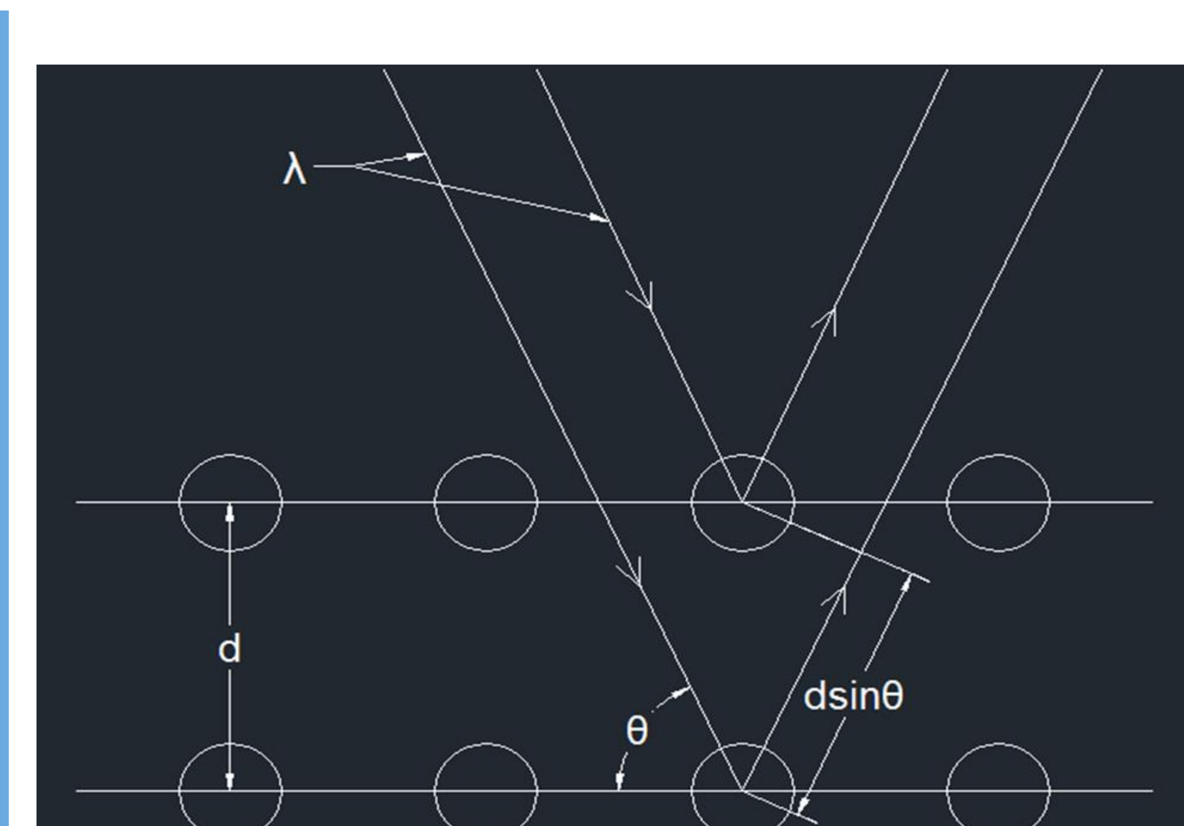


Figure 1- This is a basic visual representation of Bragg's Law. λ is the wavelength of the X-rays, d is the spacing of the crystalline planes, and θ is the angle, up from the horizontal, that the x-rays strikes the crystal lattice at.

IV. X-ray Diffraction at Houghton

The design of the Houghton diffractometer is known as a Braggs-Brentano θ-2θ X-ray diffractometer. Two arms are attached to an axle at the center of a semi-circle. The X-ray source is aimed horizontally along the base of the semi-circle, in line with the axle. One arm is at an angle θ from the horizontal. This arm holds the sample mount. The sample mount consists of a micrometer that holds the silver thin film over the center of the axle. The second arm is at an angle of 2θ from the horizontal. This arm holds the Vernier Student Radiation Monitor. The apparatus is depicted in figure 2. Both arms are rotated around the semi-circle by motors run by a program written in LabVIEW. The program keeps the θ: 2θ ratio between the two arms while moving the arms from θ=0º to θ=90º. An example of the arm motion can be see below in figure 3, where θ=45º. This setup allows the radiation monitor to pick up peaks of intensity where the X-rays constructively interfere. Bragg's law can then be used to find the spacing between the crystalline planes in the silver thin film. This distance gives the orientation of the crystals showing the texture of the film.

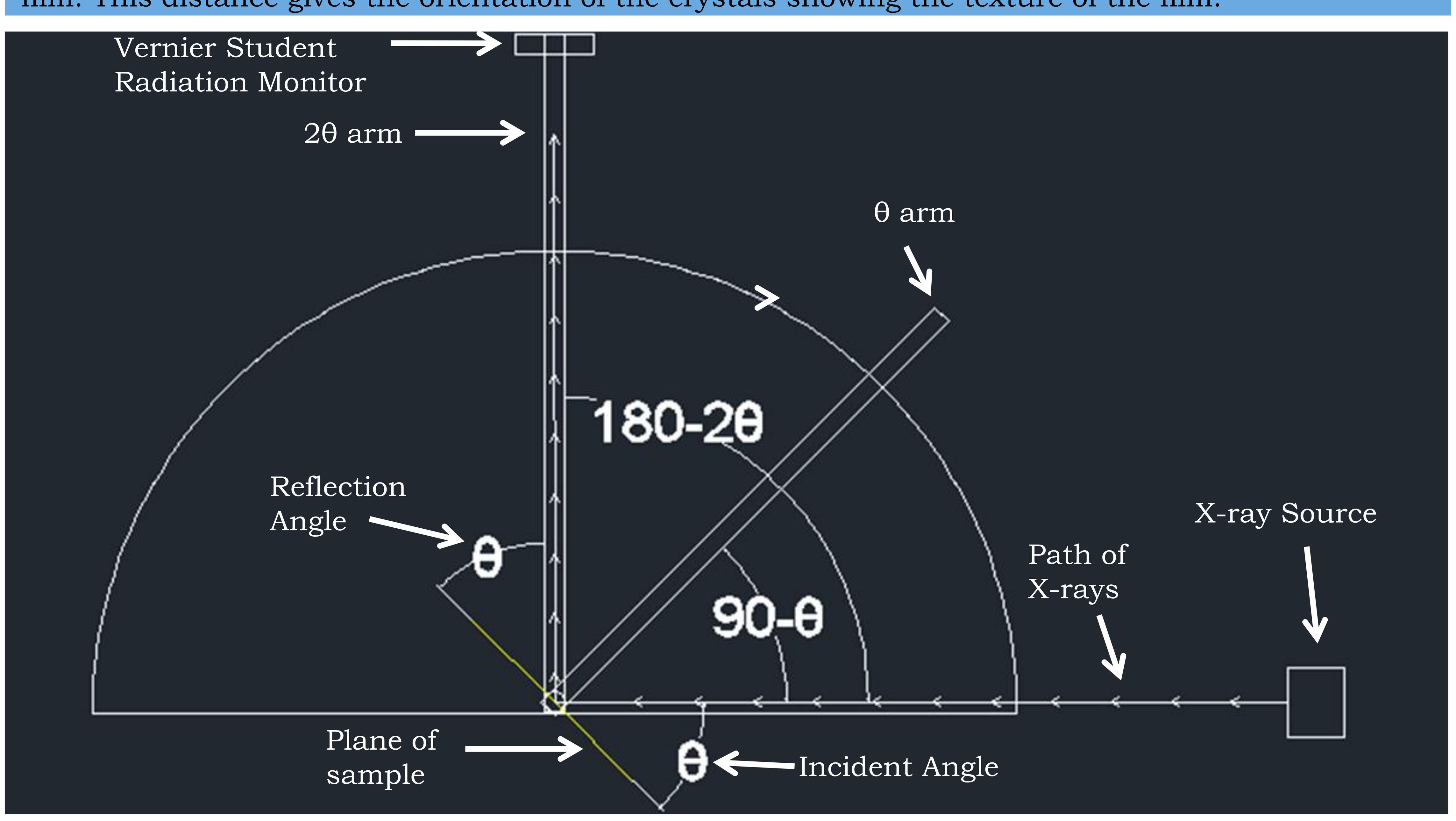


Figure 2 (above)- A basic diagram of a Braggs-Brentano θ-2θ X-ray diffractometer. In this specific case, θ=45º . The X-rays travel along the horizontal axis until they reflect off the plane of the sample. The X-rays that constructively interfere will reflect at a 90º.

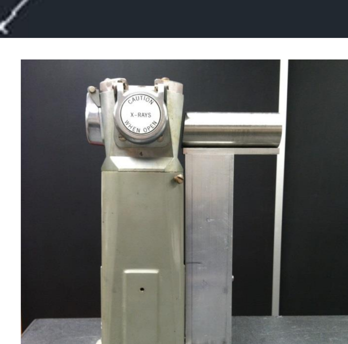
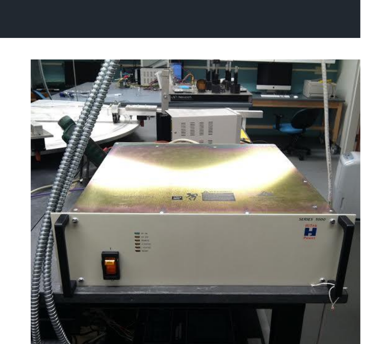


Figure 4 (left)- pictured is the Phillips-Norelco X-ray source. The target in the diffraction tubes is copper. The cylinder in front of the X-ray sources is a steel collimator with a ¼" hole drilled down the center.

Figure 5 (right)- pictured is the 40kV power supply that's used to power the X-ray source. It's hooked up to a circuit that includes a battery and a potentiometer.



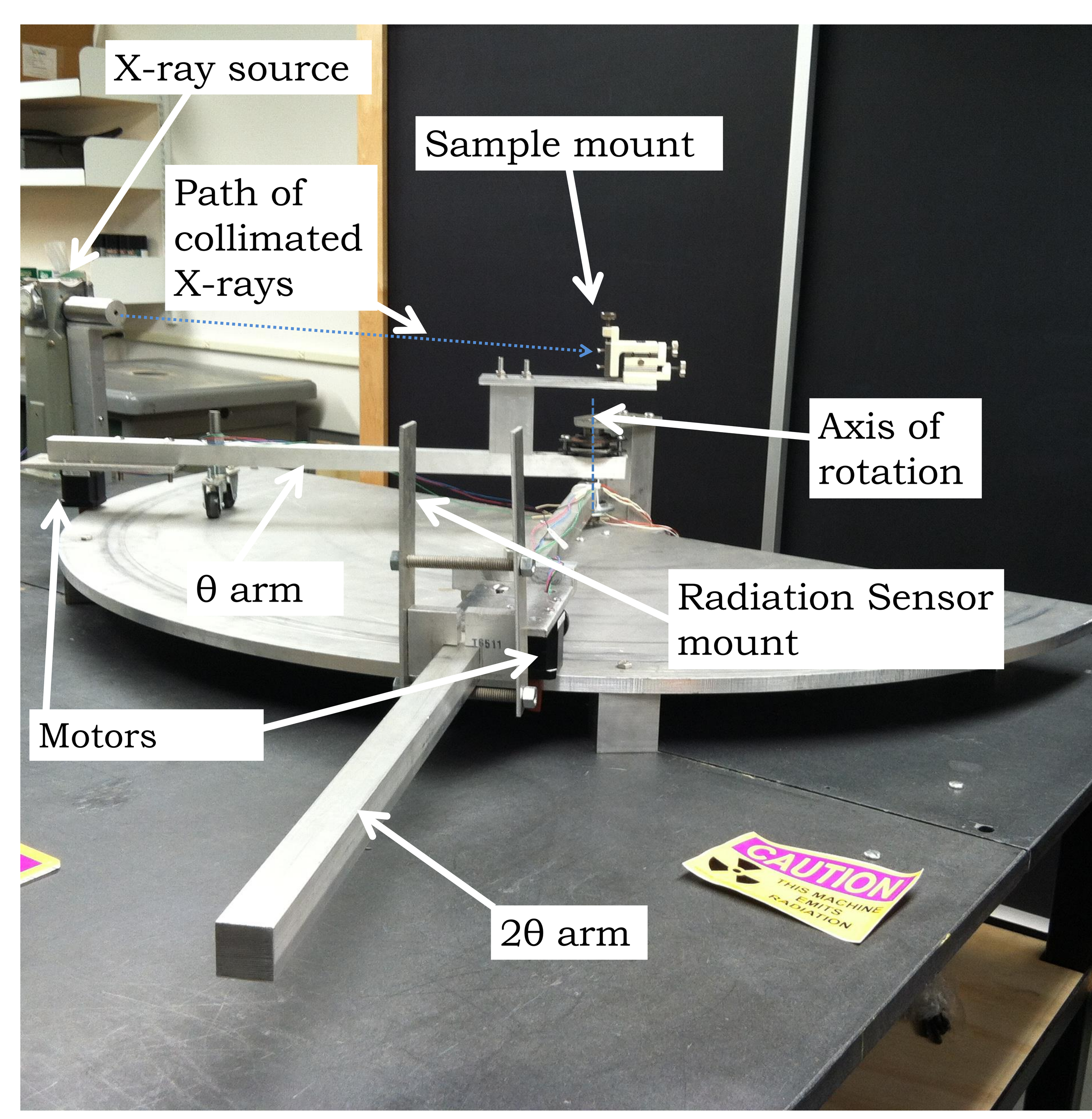


Figure 3 (above)- A labeled picture of the apparatus of the Houghton X-ray diffractometer. The motors rotate the arms from θ=0º to θ=90º while keeping the θ: 2θ ratio between the arms in order to determine the angle at which the X-rays constructively interfere

V. Future Plans

The Houghton diffractometer is still under construction at this point in time. As a safety precaution, the entire apparatus will be encased in a steel box. The box will be certain thickness such that the maximum intensity of X-rays that escape will be 0.05 mRem per hour. A flowmeter will be added to the cooling system for the X-ray source. If the flow of water out of the source is less than the flow in, then the meter will shut down the experiment, saving the source from overheating. There will also be a radiation detector outside of the steel box that will shut down the experiment if the radiation level outside of the box is above 0.05 mRem/hr. Once all of the proper safety measures are in place, data can be collected on the texture of silver thin films.