Electro-diffractive Measurements of Graphite Molecular Spacings

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We investigated the molecular spacings of Carbon atoms in a Graphite crystal through analyzing interference patterns produced via electron diffraction. We accelerated electrons toward a Graphite grating using an accelerating voltage, V_A. The electrons diffracted off the Graphite and toward a fluorescent screen, producing a visible interference pattern characterized by concentric circular bands, each of a distinct radius. We measured the radii of the pattern's bands and used a transfer function derived from Bragg's law to determine the corresponding molecular spacing in relation to V_A. We observed 6 modes of a fundamental wavelength corresponding to an average molecular spacing of 0.176 ± 0.013 nm, confirming Bragg's Law for electron diffraction, but failing to discriminate between the two theoretical molecular spacings.
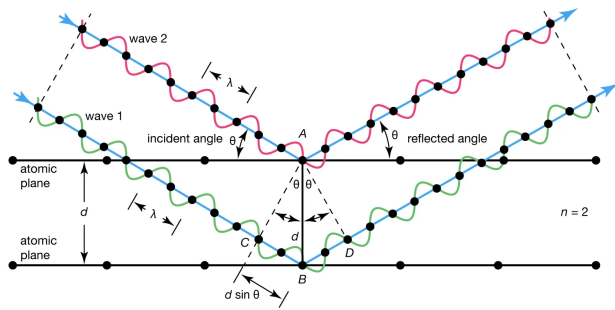
I. INTRODUCTION

Father and son, Sir William Henry Bragg and Sir William Lawrence Bragg shared the 1915 Nobel Prize in physics for describing the interference pattern of x-rays diffracting off a crystal [1]. They related the wavelength of x-rays (λ) with the molecular spacing of the crystal (d) and the observed diffraction angle (θ) as shown in Figure 1. Since these rays reflect off atoms in many "molecular planes" — quantified as the nth plane, $n \in \mathbb{N}$ — Bragg's law is written $n\lambda = 2d\sin\theta, n \in \mathbb{N}$. Since electrons can act as waves, we can observe Bragg diffraction when firing electrons toward a uniform crystal.

In this experiment, we will quantify the spacing of Graphite by accelerating electrons using an applied voltage, V_A, which we refer to as the accelerating voltage. One can find the kinetic energy, KE, of these accelerated electrons through the relation, $\text{KE} = p^2/2m_e = eV_A$, where p is the momentum and m_e is the mass of an electron, and e is the charge of an electron. Using DeBroglie's wavelength law, $\lambda = h/p$, where h is Planck's constant, we can produce an equation for relating the electron wavelength to V_A:

FIG. 1: As waves of wavelength λ, encounter a crystal, of uniform molecular spacing d, they reflect at an angle θ. However, they can penetrate further and reflect off atoms in the nth molecular plane (if we define the outermost plane as $n = 1$.) This results in Bragg's law. Note that the diffraction angle is multiplied by 2 since the equation describes the total angular deflection of the beam, equal to the sum of the angles of incidence and reflection [2].

$$\text{KE} = \frac{p^2}{2m_e} = eV_A$$

$$p^2 = 2m_e eV_A \quad \therefore \quad p = \sqrt{2m_e eV_A}$$

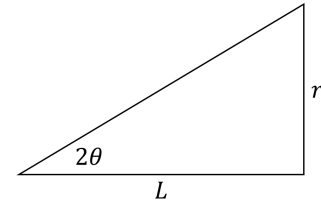$$\lambda = \frac{h}{p} = \frac{h}{\sqrt{2m_e eV_A}}$$

Using this expression for λ in Bragg's law, along with the small angle approximation $\sin\theta \approx \theta$, we can find a relationship between molecular spacing (d) and our observed interference pattern, characterized by i concentric circles of radii, r_i.

$$2d\sin\theta = n\lambda$$

$$2d(\theta) = n\frac{h}{\sqrt{2m_e eV_A}}$$

$$d = n\frac{h}{\sqrt{2m_e eV_A}}\frac{1}{2\theta}$$

$$\tan 2\theta = \frac{r}{L}$$

$$2\theta = \frac{r}{L}$$

$$d = n\frac{h}{\sqrt{2m_e eV_A}}\frac{L}{r} \tag{1}$$

Since we are interested in using electron diffraction to measure the molecular spacing of Graphite, a layered crystal of pure carbon, we must first understand its crystal structure. Ideal Graphite crystals have hexagonally positioned Carbon atoms in a one-atom thin "molecular plane." These planes are stacked upon each other to form the bulk crystal as shown in Figure 2.

Depending on the orientation of the crystal, there are several molecular planes we can consider, each with a corresponding molecular spacing, d, as illustrated in Figure 1. The theoretical molecular spacings we expect are $d_1 = 0.123$ and $d_2 = 0.213$ nm [4].
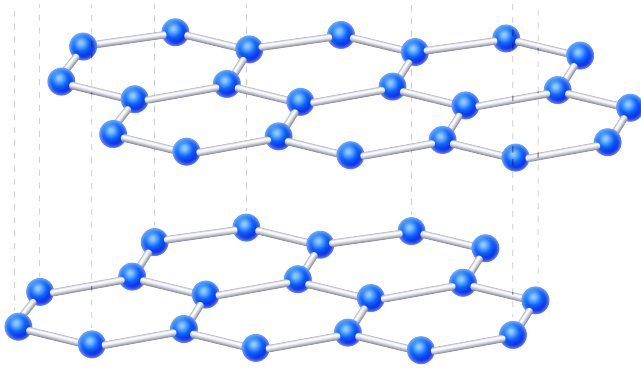
FIG. 2: Bulk Graphite crystal. Notice that layers are not perfectly hybridized (aligned). Each coplanar Carbon atom is covalently bonded to its neighbors. Carbon atoms in different layers are attracted via Van der Waal's forces which are much weaker [3].
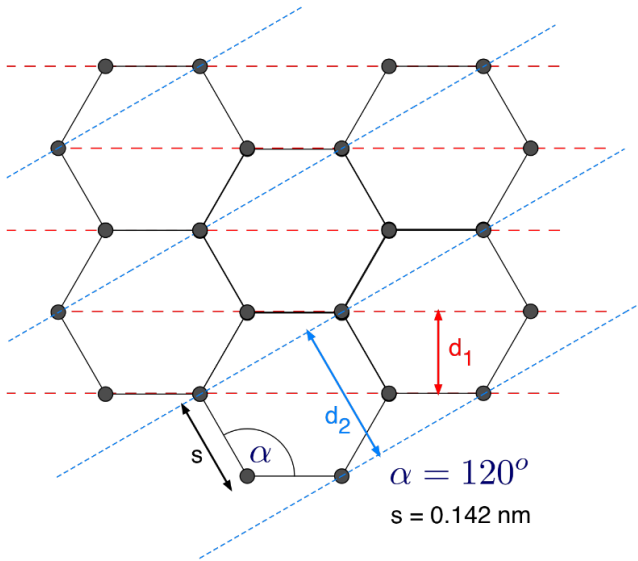

FIG. 3: Illustrating the lattice spacing of a single layer of ideal graphite. The theoretical observed spacings for our experiment are $d_1 = 0.123$ nm and $d_2 = 0.213$ nm, calculated from the distance between Carbon atoms, $s = 0.142$ nm, and the angle at which they bind to form the crystal, $\alpha = 120°$ [4].

II. EXPERIMENTAL SETUP

A. Apparatus

To investigate the molecular spacing of Graphite via electron diffraction, we use a Leybold 55517 Electron Beam Diffraction Tube, powered with a Tetron Model 813 kV Power Unit. This produces V_F, a 6.3 V AC voltage necessary to heat a filament which undergoes thermonic emission (boiling an oxide-coated cathode) to "produce" our electrons. Then we accelerate the electrons using $V_A \sim 2000$-5000 V. We test voltages only within this range; any voltages below around 2000 V does not produce
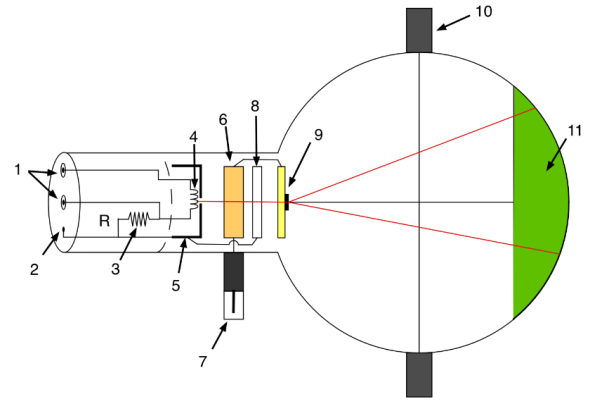

FIG. 4: A detailed illustration of the geometry of the electron diffraction tube. (1) Filament heating supply voltage socket, (2) cathode socket, (3) internal resistor, (4) filament, (5) cathode, (6) anode, (7) high voltage anode connection, (8) focusing electrode, (9) graphite grating, (10) boss (a mount to rest the tube), (11) fluorescent screen [4].

an interference pattern, and the upper limit of our supply was 5000 V. We also use a Precision Model 1601 Regulated Power Supply to provide a biasing voltage, V_B, negative relative to the cathode. This centers the accelerated electrons toward the target Graphite sample so that fewer collide with the sides of the tube. Once the electrons reach the sample, they diffract and continue toward a fluorescent screen which produces a visible interference pattern by absorbing the electrons and emitting photons. This process is shown in Figure 4.

B. Data Collection

Since we have shown via Equation 1 that the molecular spacings we will observe are dependent upon the radii of the interference pattern, r, and the accelerating voltage, V_A, we will collect measurements of r for each concentric circular "band" at many values of V_A.

First, we used a caliper to measure the radii as the distance from the center of the interference pattern to the center of each band. However, upon repeated measurements of the same radius, we could not produce repeatable, accurate results. Therefore, we reran the experiment taking high quality images of each interference pattern which required high exposure time. To limit the blurring effects of this high exposure time, we stabilized the camera using a tripod. This allowed us to post process the images to accurately determine the radii. However, to implement this method, we first had to provdie a reference length, so we used the caliper to measure the diameter of the fluorescent screen. Then, using an image measurement software, IC Measure, we calibrated the size of each pixel by inputting the diameter of the fluorescent screen as our reference length. Finally, we measured the radii of each concentric circular band by measuring the distance from the center of the interference pattern to the center of the band. We stored the
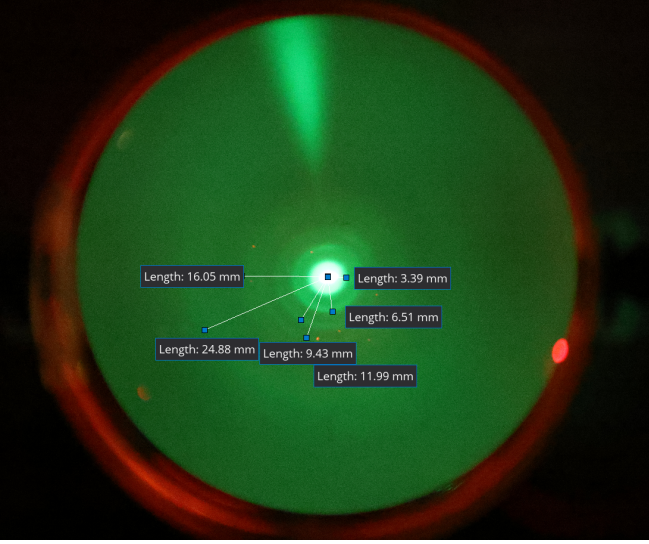
FIG. 5: Illustrating an example measurement from the $V_A = 3000$ V trial. Notice distinct bands of light with a radius measured from the center of the diffraction pattern to the center of the band.

radii values and corresponding accelerating voltages into a csv file. An example of this image with the measurements overlaid is shown in Figure 5.

When collecting data, we noticed that we did not observe a consistent number of bands for each V_A. Upon viewing all measurements, we hypothesized that some of the bands were so close together that we could not discern the them. Therefore, each band had a different number of observations which will impact our uncertainty. Figure 6 illustrates our collected data graphically.

C. Data Analysis

To analyze our data, we read our csv document using `Pandas`, a Python package for managing data-frames. For each radii and V_A pair, we calculated theoretical spacings using Equation 1. We then compared the average and standard deviation of the resultant calculations for each radii and noticed that they were not identical. To determine whether these differences in measured spacings resulted from each band being a multiple of a fundamental mode — as Bragg's law predicts — or a result of different molecular spacings, we compared pairwise ratios of elements in the list of mean spacings. Since Equation 1 is linear in n, this will produce a ratio of mode numbers for each pair of bands.

When performing this analysis, we noticed that the 2nd spacing calculation was roughly double that of the 1st spacing calculation; we therefore hypothesized that the 1st and 2nd bands were 1st and 2nd order modes respectively. We repeated this process for each pairwise combination and determined that all ratios of mode numbers were approximately rational, indicating that the bands are all modes of the same fundamental wavelength ($n = 1$). For each of the 6 bands, we hypothesized

corresponding mode numbers of 1, 2, 3, 4, 5, 7. This indicates that we observed several modes of one molecular spacing.

We then scaled each calculated spacing value by multiplying it by its respective mode number to produce a set of approximately identical spacing values. After propagating our errors, we determined one molecular spacing of 0.176 ± 0.007 nm. However, we then propagated our uncertainty in V_A, L, and r to determine our true uncertainty.

Since we calibrated V_A using the analog voltage meter on the Tetron Power Unit; we estimate our uncertainty in V_A at most 100 V. L was specified as 140 mm in the documentation for the diffraction tube; therefore, we didn't include an uncertainty in L. Since we measured r using an image processing system, our dominant uncertainty relied in the measurement of the fluorescent coating diameter, which we estimate to have an uncertainty of 0.5 mm. Therefore, uncertainty can be calculated as follows:

$$d = n \frac{h}{\sqrt{2m_e e V_A}} \frac{L}{r} \equiv n \frac{\alpha}{\sqrt{V_A}} \frac{L}{r}$$

$$\sigma_d = \sqrt{\left(\frac{\partial d}{\partial V_A}\right)^2_{n,V_A,L,r} \sigma_{V_A}^2 + \left(\frac{\partial d}{\partial r}\right)^2_{n,V_A,L,r} \sigma_r^2}$$

$$\frac{\partial d}{\partial V_A} = n \frac{-\alpha}{2V_A^{3/2}} \frac{L}{r} \qquad \frac{\partial d}{\partial r} = n \frac{\alpha}{\sqrt{V_A}} \frac{-L}{r^2}$$

$$\sigma_d = \sqrt{\left(n \frac{-\alpha}{2V_A^{3/2}} \frac{L}{r}\right)^2_{n,V_A,L,r} \sigma_{V_A}^2 + \left(n \frac{\alpha}{\sqrt{V_A}} \frac{-L}{r^2}\right)^2_{n,V_A,L,r} \sigma_r^2}$$

$$\sigma_d = 0.013 \text{ nm}$$

III. DISCUSSION

We observed several bands on our diffraction pattern, each of which corresponded to a molecular spacing of 0.176 ± 0.013 nm, confirming Bragg's Law can represent the interference pattern resultant from electron diffraction. However, we expected to see two distinct spacings of 0.123 and 0.213 nm corresponding to d_1 and d_2 on Figure 3. We believe our method lacked the resolution to discern the two unique spacings, instead observing their average of 0.168, which falls within our confidence interval (mean $\pm$ one sigma).

However, a change in methodology could resolve this. When consulting others who have performed this experiment, several sources indicated that one can observe two unique molecular spacings by using the inner and outer edge of the bands. Therefore, each band would correspond to an inner and outer radius. Repeating the same analysis method we performed, you measure several modes of two unique spacings [4, 5].

IV. CONCLUSIONS

We used electron diffraction to determine the molecular spacing of Graphite. By applying an acceleration voltage, V_S,
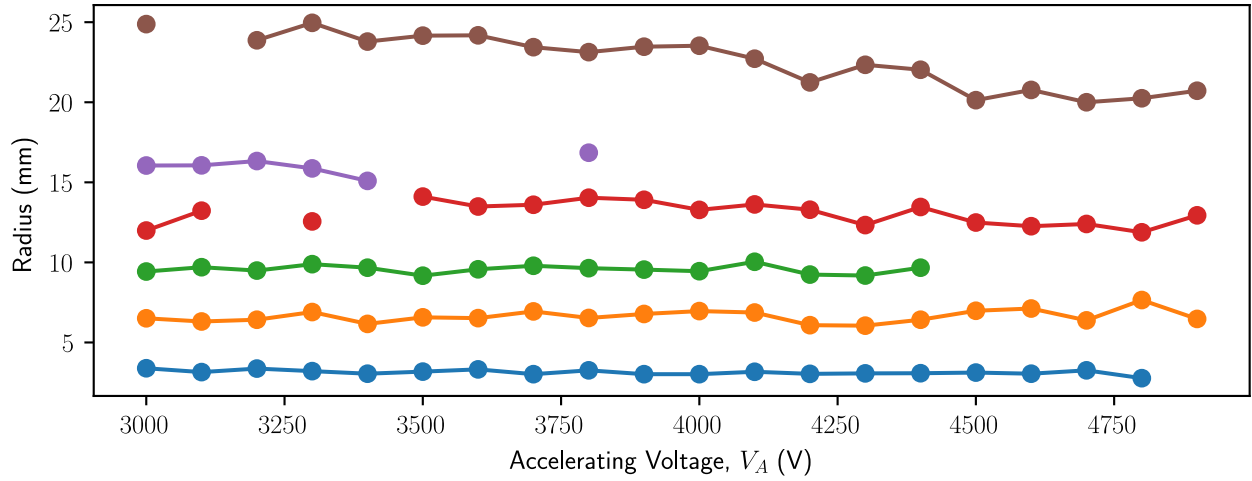
FIG. 6: Graphing band radii measurements as we increased the accelerating voltage, V_A. Each band radius is denoted by a separate color. Notice that we did not observe every band for each accelerating voltage, especially the 5th band which is illustrated in purple and only contains 6 observations.

to an electron source, we fired electrons toward a Graphite grating, creating an interference pattern on a fluorescent screen. We used image processing to accurately measure the radii of several diffraction bands which we defined as the distance from the center of the interference pattern to the center of the band. We measured these radii for several values of accelerating voltage to test the repeatability of the theoretical transfer function from radius and voltage to molecular spacing. We observed 6 modes of the same fundamental wavelength, confirming Bragg's Law.

We found the molecular spacing of Graphite to be 0.176 ± 0.013 nm, corresponding to the average of the two spacings we sought to verify, $d_1 = 0.123$ and $d_2 = 0.213$ nm. We believe that we can recover the individual spacings by changing how we measure the radius of the band. In future experiments, we

can measure an inner radius from the center of the interference pattern to the inner edge of the diffraction band. Then, measure an outer radius from the center of the interference pattern to the outer edge of the diffraction band. This will produce two radii from which two spacings can be calculated. Since V_A would be fixed, the difference in radii must either yield a difference in calculated molecular spacing or an integer difference in mode number, n, as shown in Equation 1.

[1] L. Bragg, *The Crystalline State: A General Survey* (G Bell and Sons, 1949).

[2] T. D. of Encyclopaedia Britannica, X-ray diffraction, `https://www.britannica.com/science/X-ray-diffraction` (2023), Accessed: 3 March 2024.

[3] K. Griffin, What are the structure and properties of graphite?, `https://mwi-inc.com/blog-post/what-are-the-structure-and-properties-of-graphite/` (2022), Accessed: 3 March 2024.

[4] W. Boeglin, Electron diffraction, `https://wanda.fiu.edu/boeglinw/courses/Modern_lab_manual3/Electron_diffraction.html` (2024), Accessed: 3 March 2024.

[5] D. Smith, Electron diffraction lab (2017).