

# Analysis of a GaAs Crystal Structure Using TEM

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## Abstract

In this experiment, the Transmission Electron Microscope (TEM) was employed to analyze the structure and diffraction properties of a GaAs sample. Techniques including Selected Area Electron Diffraction (SAED), Convergent Beam Electron Diffraction (CBED), and Energy Dispersive X-ray Spectroscopy (EDS) were utilized to identify the material and examine its crystallographic orientations, on both a simple and complex diffraction pattern of the sample. We found our GaAs was in the [110] viewing axis, and that a complex diffraction pattern came from two distinct growth directions in the crystal. The lattice constant was determined to be  $a = 5.635 \pm 0.0118 \text{ \AA}$ , and the angle  $\theta$  between the two growth zones in our sample was calculated as  $70.5^\circ$ .

However, we note that the calibration method, referencing  $R_{[111]}$ , would yield similar results for any FCC material in the [110] orientation, emphasizing the need for precise calibration of the camera constant. Despite this limitation, our main focus of material identification and the analysis of the two patterns and relative orientations was successfully achieved.

## 1. Introduction

Transmission Electron Microscopy (TEM) is a powerful and important tool that provides detailed insights into the atomic arrangement, crystal orientation, and diffraction patterns of materials, making it an essential tool for characterizing unknown samples. In this report we will explore the structural and orientational characteristics of a Gallium Arsenide (GaAs) sample. We employ TEM techniques like Bright Field (BF) TEM, Selected Area Electron Diffraction (SAED), and Convergent Beam Electron Diffraction (CBED), focusing on analyzing two diffraction patterns recorded with SAED. One simple, and one complex. We identify the material, determine its orientation, and study the angular relationship between our different crystallographic zones.

With indexing diffraction spots and analyzing reciprocal lattice vectors we will determine the crystal orientation and structure, particularly through angular measurements and symmetry analysis.

## 2. Theory

### 2.1. Imaging techniques

To analyze our GaAs sample, some different imaging techniques were used:

**Bright Field (BF) TEM**, provides an overall view of the sample's structure by utilizing the unscattered and weakly scattered electrons to create contrast. Regions with higher thickness or heavier elements appear darker due to increased electron scattering. It is particularly useful for observing grain boundaries, defects, and overall sample structure at lower

magnifications [1].

**High Resolution (HR) TEM**, on the other hand, allows visualization of the atomic arrangement within the crystal. By utilizing the interference between transmitted and scattered electrons, HR TEM produces high-resolution images that reveal lattice fringes and atomic-scale details. [1].

**Convergent Beam Electron Diffraction (CBED)**, is where an incident beam is focused with both the condenser lenses and the pre field of the objective lens working together. In CBED the incident rays come into the specimen at different angles. This angular range is small, however, and in practice all electrons in the incident cone can be diffracted, at least to some degree. We use CBED to check the Symmetry of our sample. [1]

**Selected Area Electron Diffraction (SAED)**, is used to obtain diffraction patterns from specific regions of a sample. We will use it to give us information about the crystal structure, orientation, and lattice parameters of the material. [1]

**Fast Fourier Transform (FFT)**, essentially is a computer simulated way to receive the diffraction pattern some image/pattern would theoretically give. We use this to help the analysis of our sample.

### 2.2. Structure of the GaAs

GaAs has a face-centered cubic (FCC) Bravais lattice with two atoms (Ga and As) per basis, with lattice constant  $a = 5.653 \text{ \AA}$  [2]. It has a zinc-blende structure and is in the space group of  $F\bar{4}3m$ . This means GaAs is non-centrosymmetric, and has the following kinematic diffraction conditions [3]:

$$\begin{aligned}
hkl : h+k, h+l, k+l &= 2n \\
0kl : k, l &= 2n \\
hhl : h+l &= 2n \\
h00 : h &= 2n
\end{aligned}$$

Figure 1: Kinematic diffraction conditions for a  $F\bar{4}3m$  structure.

As our sample is relatively thick, we need to consider dynamic diffraction, but the allowed diffraction points for GaAs don't change. Importantly for this paper, the two lowest allowed orders are: [111] and [200] zone axes.

To analyze the defect in the structure of the crystal, we will use the formula (1) to calculate the angle between the two orientations:

$$\cos \theta = \frac{g_1 g_2}{|g_1||g_2|} \quad (1)$$

### 2.3. Method of Indexing

The allowed diffraction spots must be perpendicular to the orientation of the incident beam of the diffraction pattern, as described by Weiss zone law [1]. An example of the diffraction pattern we would receive for a FCC structure in the [110]-viewing axis, can be seen in figure (2). As we are in the dynamic case (and the diffraction conditions (1) are equivalent) a GaAs sample in a [110]-axis would give the same pattern.

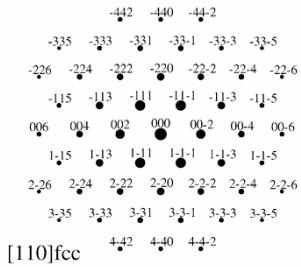


Figure 2: Indexed Single Crystal Diffraction Pattern [110]fcc [1]

We supplement this with the fact that the shortest allowed distance for the GaAs has to be the [111]-indexes (2.2), Weiss law then gives us the freedom to choose the indexing of our [111] vectors, which we use to find the [002]-indexes by vector addition.

### 2.4. Calibration

We have the calibration formula used for a SAED diffraction pattern [1]:

$$Rd = \lambda L, \quad (2)$$

where  $R$  is distance between diffraction spots in the reciprocal space,  $d$  is the distance in real space between the lattice planes under observation,  $L$  is the camera length and  $\lambda$  is the wavelength of the electrons used:

$$\lambda = \frac{h}{\sqrt{2m_e eV \left(1 + \frac{eV}{2m_e c^2}\right)}}, \quad (3)$$

where  $h$  is Planck's constant,  $m_e$  is the electron mass,  $e$  is the elementary charge,  $V$  is the accelerating voltage, and  $c$  is the speed of light.

### 2.5. Energy Dispersive X-ray Spectroscopy (EDS)

We use this technique along with electron microscopy to analyze the elemental composition of materials. In EDS it detects characteristic X-rays emitted when a sample is bombarded with high-energy electrons [1].

## 3. Experimental

To record and image the sample, the Jeal JEM 2100F, Field Emission Gun Transmission Electron Microscope (FEGTEM) was used (see Block diagram of TEM 3).

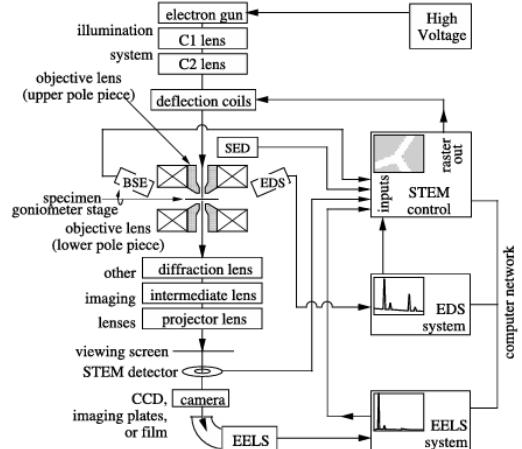


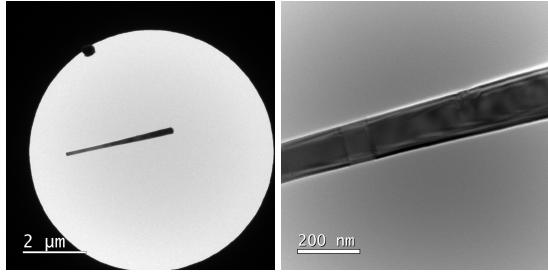
Figure 3: Block diagram of typical TEM with STEM capability [[1]]

In (3) an electron beam is focused by lenses and interacts with the sample to produce transmitted electrons. Detectors collect signals for imaging, diffraction and/or spectroscopy. Results are visualized on a screen or captured by a camera for analysis.

To record this data an acceleration voltage of about 200 kV was used. When the sample was turned to  $x = 1.8^\circ$  and  $y = 3.4^\circ$ , we found a clear SAED diffraction pattern and proceeded to record ED TEM, HR TEM, CBED and SAED of two positions in the crystal. Then to measure the reciprocal distances in the SAED iamges, ImageJ [4] was used.

## 4. Results and Discussion

The particle we study in this paper is seen in the bright field images of figure (4).



(a) BF image of our particle (the long thin object), 25 times magnified,  $x = 1.8^\circ$ ,  $y = 3.4^\circ$ .  
(b) 250 times magnification, into an erroneous area. Notice the "lines" in the particle.

Figure 4: BF images of the particle we study in this paper.

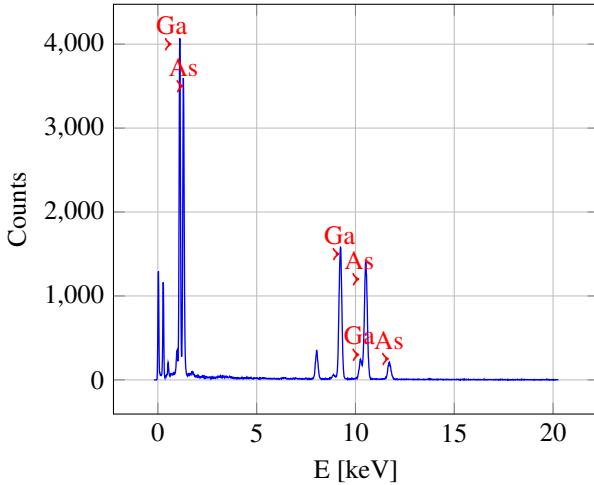


Figure 5: EDS of our sample. The intensity (y-axis) represents the detected signal strength, while the X-axis is the energy of the photon. Arrows indicate peak positions corresponding to Ga and As characteristic x-rays [5]. Unmarked peaks correspond to other elements (such as Cu, Si and O).

To identify the particle/sample we recorded its EDS 5), which shows peaks correspond to characteristic x-rays that reveal a high relative presence of Ga and As in the sample [5], hence this is a GaAs sample. The additional peaks appearing in the plot represent elements that are present either within the sample or as external contaminants. We now look closer into an area in 4b.

Inspecting (6), we observe that the crystal grows in different orientations, as seen by the red and green lines. As the crystal was manufactured/grown, an "error" in growth occurred in the crystal, giving us a clear line separating the two orientations. We will study this later on. To confirm that this is a GaAs sample, we first inspect only Zone 1.

We will argue zone 1 is in the [110]-orientation by the symmetry of the GaAs structure (2.2), as the structure will entail that the [110]-viewing axis will have two fold symmetry, much like a FCC structure. In (7) we observe a two fold symmetry, therefore we are in the [110]-viewing axis.

Further, we indexed the pattern in (8) as described in section

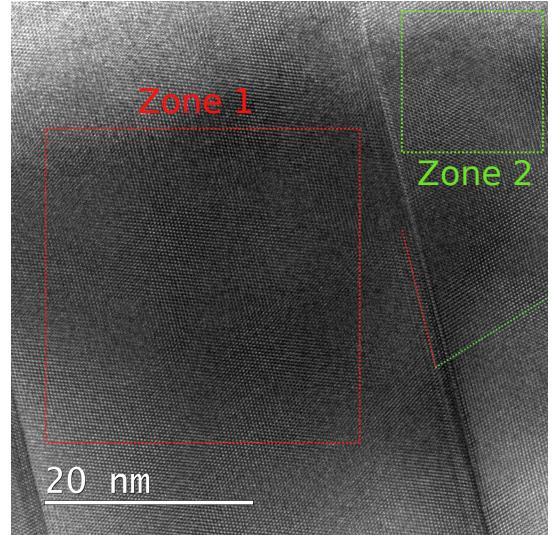


Figure 6: HR TEM of the area in (4b), 400 times magnification of original sample. We have marked "Zone 1" and "Zone 2" as clarification for what areas are studied later on. The red and green line show the relative growth direction of the crystal. Both zones are in an [110]-viewing axis, but at different orientations.

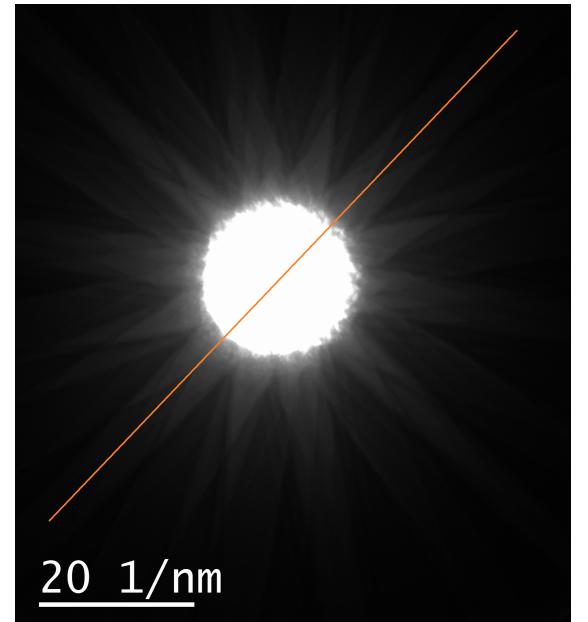


Figure 7: CBED of GaAs in Zone 1 (of figure (6)).  $L = 8$  cm. The Kikuchi pattern shows us a two-fold symmetry, as seen in the lack of repetition in the Kikuchi lines adjacent to the highlighted orange line.

(2.3), taking the liberty of choosing  $\vec{b}_1 = [1\bar{1}\bar{1}]$  and  $\vec{b}_2 = [00\bar{2}]$ .

#### 4.1. Calibrations

By using eq (2) and eq (3) with  $L = 30$  cm and  $V = 200$  kV, we get a value for the camera constant:  $\lambda L = 7.53 \cdot 10^{-13}$ .

Comparing this to  $d_{111}R_{111} = 9.79 \cdot 10^{-19}$  and  $d_{002}R_{002} = 9.75 \cdot 10^{-19}$  that can be found by instead calibrating with the scale bar in (8) and using eq (4) for  $d$ , we see how the value for the camera constant should be much lower

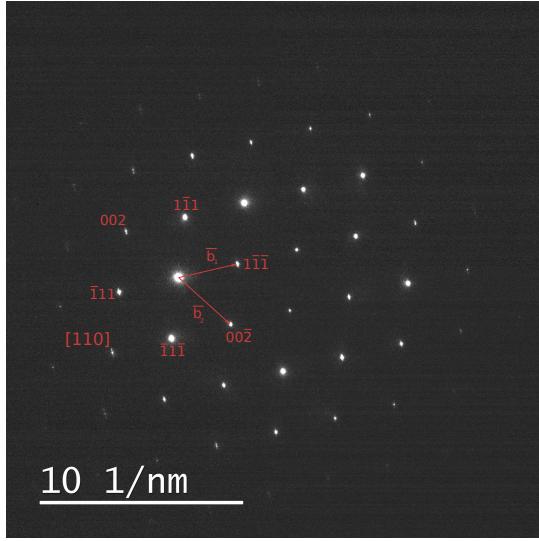


Figure 8: SAED of our GaAs sample in Zone 1 figure 6.  $L = 30$  cm. This was found to be in the [110]-viewing axis, and is indexed as such. Indexing and vectors drawn in Inkscape. [6]

(around  $9.77 \cdot 10^{-19}$ ). This most likely comes from an error of recorded camera length; this value is not in the square root of eq (2) and therefore more significantly changes the resulting camera constant. We still mark our figures with this wrong camera length though, to show consist camera length is used.

Instead we calibrated with the following approach: Given the  $d$ -spacing can be calculated using [1]:

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad (4)$$

Further, we have for a reciprocal distance  $R_{hkl} = \frac{1}{d_{hkl}}$  [1], then with  $a = 5.653 \text{ \AA}$ :

$$R_{hkl} = \frac{\sqrt{h^2 + k^2 + l^2}}{a}, \quad R_{111} = 0.306 \text{ \AA}^{-1}. \quad (5)$$

We used  $R_{111}$  on the distance to  $[1\bar{1}\bar{1}]$  as our calibration for 8.

#### 4.2. Confirming the sample as GaAs

k	[111]	[111]	[111]	[111]	$R_{k[111]}$	$a$ [nm]
1	3.06	3.07	3.10	3.10	$3.08 \pm 0.01$	$0.562 \pm 0.0019$
2	6.17	6.14	6.22	6.16	$6.17 \pm 0.02$	$0.56122 \pm 0.0015$

Table 1: Table of measured distances of the indexes in 8, for  $k \cdot [111]$ , where  $k$  is a scaling parameter that indicates the order of reflection along the [111] direction.  $R_{k[111]}$  is the mean of these values.  $a$  is the calculated lattice constant. All parameters except  $k$  and  $a$  are in  $\text{nm}^{-1}$ .

k [002]	[002]	$R_{k[002]}$	$a$ [nm]
1	3.54	$3.52$	$3.53 \pm 0.01$
2	7.06	$7.12$	$7.09 \pm 0.03$

Table 2: Table of measured distances of the indexes in 8, for  $k[002]$ , where  $k$  is a scaling parameter that indicates the order of reflection along the [002] direction.  $R_{k[002]}$  is the mean of these values.  $a$  is the calculated lattice constant. All parameters except  $k$  and  $a$  are in  $\text{nm}^{-1}$ .

In table (1) and (2) we have the data of measured distances to indexed diffraction points and the corresponding lattice constant  $a$  for the orientational groups, which was found with eq (5). Error was found by using standard error of the mean (SEM) [7]

The statistical error is very small, as expected for work like this where many diffraction points are measured with similar distances. Finding the mean lattice constant  $\bar{a}$  using the values found in (1) and (2) gives:  $\bar{a} = 5.635 \pm 0.0118 \text{ \AA}$  (error found by weighted standard error (WSE) [7]). Ignoring the statistical error, this gives a relative error to the real  $a$  ( $\frac{|\bar{a}-a|}{a}$ ) as 0.34%. This is very accurate, however as discussed later in 4.4, because of the calibration method used this can not be used to confirm whether the sample is GaAs.

#### 4.3. Complex Diffraction Pattern

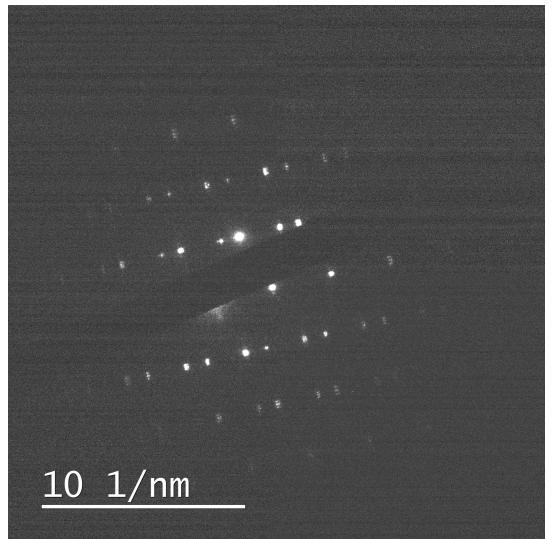


Figure 9: SAED of the sample in the area shown in figure 6.  $L = 30$  cm. We see an interesting pattern of double dots, with a central line that only has single dots.

In (9) we see the diffraction pattern resulting from a SAED of the area in (6). At first it would seem quite daunting to index this pattern as We see single diffraction points in a line along the [111] direction, and "double" diffraction points at all other points. Could this be a strange viewing orientation of the crystal? To get some insight on what is happening here, we took the FFT of (6).

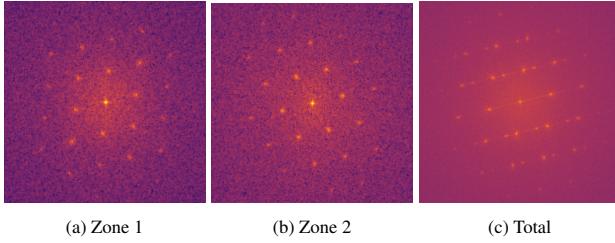


Figure 10: FFTs of figure 6, in zone 1, zone 2 and the whole picture.

The FFTs in (10) helps us in the directional analysis and indexing of our sample and figure (9) respectively, by clarifying where this pattern emerges from. In (10a) we see how the simulated diffraction pattern of Zone 1 corresponds with (8). Further we see that taking the FFT of zone 2 (10b) gives a different diffraction pattern from zone 1. But we recognize that this is also in the [110] orientation, just rotated with some angle. The total FFT is shown (10), which we recognize as the sum of the two diffraction patterns! Hence we can explain that the diffraction pattern emerges from the two different growth directions that were formed on the same crystal, and not some stranger orientation. Thus shining our beam on (6) it gives our diffraction pattern (9). Thus we see that the single diffraction point "line" comes from the fact that these [110]-viewing axis oriented patterns share some points.

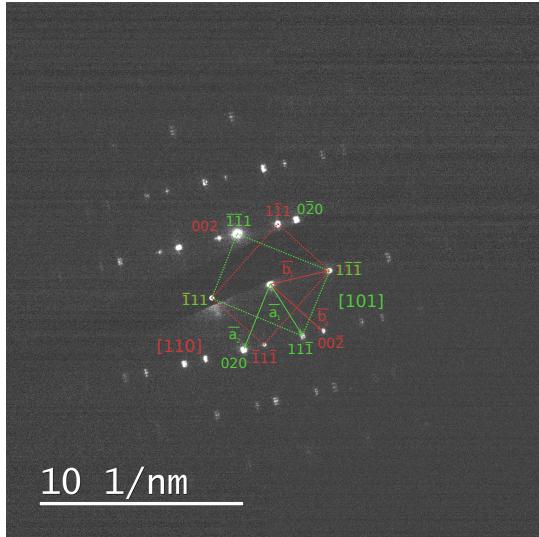


Figure 11: Indexed version of figure (9). The different growth directions of the crystal are represented by the red and green dotted rectangles and vectors  $\vec{b}_1$ ,  $\vec{b}_2$  and  $\vec{d}_1$ ,  $\vec{d}_2$ . Indexing and vectors drawn in Inkscape. [6]

Using this, we see in (11) there should be a point at the shared  $[\bar{1}11]$  vector. This has been artificially added as a white dot for clarity, as a mask is in the way of the full diffraction pattern. Zone 2 (green) was then indexed in the context of zone 1, using that  $\vec{b}_1$  and  $-\vec{b}_1$  is shared between them, as seen by the single diffraction point line and the FFTs. Because of Weiss law we then know zone 2 has to be in the [101]-orientation! From there the indexing was done by Weiss and recognizing

the further spots had to be [020] or  $[0\bar{2}0]$ .

Using eq (1) on  $\vec{b}_1$  and  $\vec{d}_1$  we receive;  $\theta = 70.5^\circ$ . Doing a qualitative measurement on the lines in (6), using a tool like Inkscape [6], we receive the same angle  $\pm 1.5^\circ$ , linking reciprocal to real space.

#### 4.4. Error In Calibration and Camera Constant

The method used for the calibration of our GaAs SAED images would yield similar results if applied to another sample with an FCC structure in the [110] orientation. This comes from the fact that we used  $a$  of GaAs to set the scale for  $R_{[111]}$ . For any FCC lattice, the ratio of  $\frac{R_{[111]}}{R_{[002]}} = \frac{\sqrt{3}}{2}$  is constant. However, this method confirms that the orientation is correct and the indexing is consistent with what is expected for a GaAs crystal.

If the real camera constant were obtained and a reference measurement of the diffraction pattern provided, the exact spacing between the lattice planes could be calculated using eq (2). This highlights the importance of an accurate calibration of the camera constant for precise lattice plane spacing calculations.

## 5. Conclusion

Through the analysis of electron diffraction patterns and high-resolution imaging, it has been demonstrated that the element under investigation is Gallium Arsenide (GaAs) in the [110] orientation. The lattice constant was measured as  $a = 5.635 \pm 0.0118 \text{ \AA}$ . However, the particle does not exhibit uniform growth along the entire direction. As shown in Figure 6, the particle is divided into at least two distinct growth zones. The angle  $\theta$  between the two growth zones was determined to be  $70.5^\circ$ . These findings are consistent with the expected structural properties of GaAs.

However, it is important to note that the methods used to calibrate the diffraction pattern, specifically by referencing the  $R_{[111]}$  vector, would yield similar results for any FCC material in the [110]-orientation. This limitation highlights the need for precise camera constant calibration to further differentiate materials. Despite this caveat, our primary focus of identifying the material, characterizing the simple and complex diffraction pattern, and analyzing their orientations and relative angles were successfully achieved.

For further work, proper recording of the camera constant will allow in finding the lattice parameter with eq (2).

## References

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