

# STUDY & ANALYSIS OF ALUMINIUM FOIL AND ANATASE TITANIUM OXIDE (TiO<sub>2</sub>) USING TRANSMISSION ELECTRON MICROSCOPY

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**Abstract** - Transmission Electron Microscopy (TEM) operates on the same basic principle as the light microscope but uses electrons and is capable of displaying magnified images of a thin specimen. The different types of images were obtained like bright field image and dark field image. In this report, the results from Selected Area Diffraction (SAED) and High-Resolution Transmission Electron Microscopy (HRTEM) of Aluminium foil and Anatase TiO<sub>2</sub> have been analyzed. The zone axis for the SAED is found out to be [02-2]. The presence of silicon and iron in the foil is also confirmed by bright field and dark field images. Lattice space between two fringes is calculated for the anatase TiO<sub>2</sub> crystal and it is found to be 0.545 nm. More better TEM techniques are also available which can predict the better results than regular TEM.

**Key Words:** Transmission electron microscopy, Aluminium Foil, Anatase TiO<sub>2</sub>, Selected Area Diffraction, High-Resolution Transmission Electron Microscopy, Bright Field, Dark Field, High-Angle Annular Dark-Field Scanning Transmission Electron Microscopy

## 1. INTRODUCTION

Transmission Electron Microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through a specimen to form an image. The instrument is used to produce electron-diffraction patterns which are useful for analysing the various properties of a crystalline specimen [1]. The specimen is most often an ultrathin section less than 100 nm thick on a grid and the electrons typically have an energy of about 100 to 400 kiloelectron volts. TEM generally finds the application in cancer research, virology and material science as well as pollution, nanotechnology and semiconductor research.

Selected Area Diffraction Pattern (SAED) is a crystallographic experimental technique that is performed inside a TEM to obtain diffraction patterns that result from the electron beam scattered by the sample lattice. Obeying the Bragg's law, the electrons are scattered elastically by the lattice, therefore, indexing of diffraction spots in the pattern is possible [2]. High Angle Annular Dark Field Scanning Transmission Electron Microscopy (STEM-HAADF) image is a STEM method which receives inelastically scattered electrons or thermal diffuse scattering at high angles using an Annular Dark Field (ADF) detector.

High-Resolution Transmission Electron Microscopy (HRTEM) is an imaging mode of the TEM that allows for direct imaging of the atomic structure of the sample [3]. One of the difficulties with HRTEM is that image formation relies on phase contrast. In phase contrast imaging, contrast is not necessarily intuitively interpretable, as the image is influenced by aberrations of the imaging lenses in the microscope. The largest contributions for uncorrected instruments typically come from defocus and astigmatism. Astigmatism can be referred to as the defect in the lens caused due to the deviation from spherical curvature, which results in distorted images because the light rays are prevented from meeting at a common focus.

## 2. MATERIALS AND METHODS

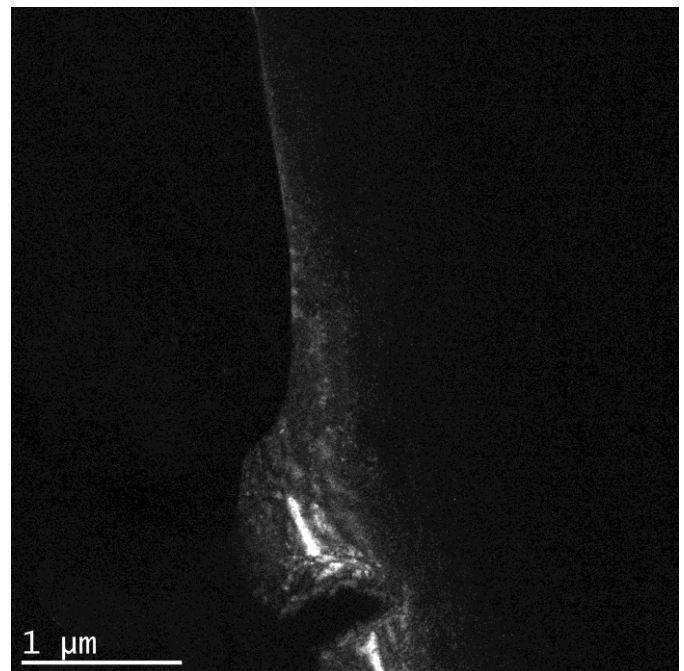
A light source at the top of the microscope emits the electrons that travel through vacuum in the column of the microscope. The electron beam then travels through the specimen. Depending on the density of the specimen, some of the electrons are scattered and disappear from the beam. At the bottom of the microscope, the unscattered electrons hit a fluorescent screen, which gives rise to a shadow image of the specimen with its different parts displayed in varied darkness according to the density. The image is then displayed on the computer using a high-resolution camera.

A sample of aluminium (Al) foil and anatase TiO<sub>2</sub> were analyzed using TEM. Aluminium sample is made from commercial purity aluminium alloy (purity > 99 wt%) and iron and silicon are the inevitable impurities in the alloy. Some dark field images and bright field images were captured. Also, some SAED patterns were obtained which were indexed to know the zone axis of the crystal. The aluminium foil sample which was analyzed has cubic structure with a volume of 66.4 cm<sup>3</sup>. Table 1 shows the crystal information of aluminium foil.

The most common image generated using a TEM is a bright field image. Some areas of the sample scatter or absorb electrons and therefore, appear darker. Other areas transmit electrons and appear brighter. In the bright field image, the objective aperture is used to select the unscattered electron beam. This aperture enhances the contrast in the image. Darker regions generally represent the impurities present in the bright field image of any sample. Figure 1 shows a bright field image of aluminium foil obtained during TEM.

**Table -1:** Crystal information for Aluminium Foil

Sr. No.	Miller Indices (hkl)	Lattice space (d), Å	Intensity (f)
1	111	2.338	100
2	200	2.024	47
3	220	1.431	22
4	311	1.221	24
5	222	1.169	7
6	400	1.0124	2
7	331	0.9089	8
8	420	0.9055	8
9	422	0.8266	9
10	511	---	---
11	440	---	---
12	531	---	---

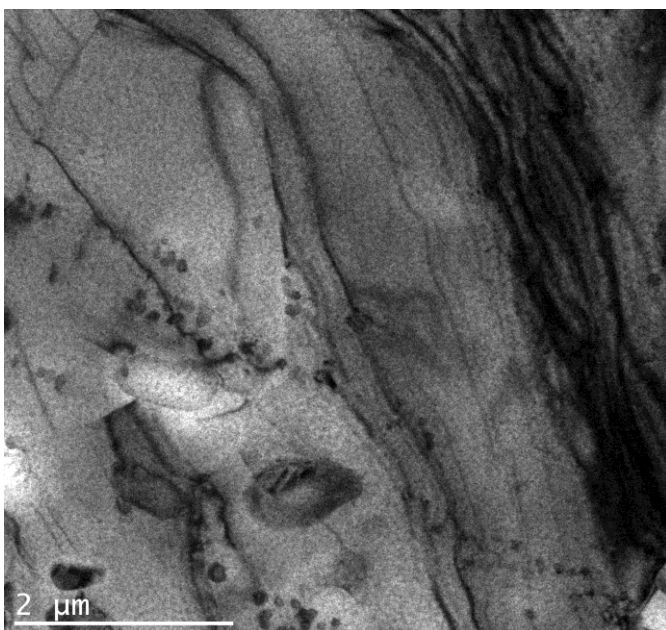


**Fig -2:** Dark Field image of Aluminium Foil

The powder sample, Anatase TiO<sub>2</sub>, was analyzed and some of the results for HRTEM were obtained. The powder sample has tetragonal crystal structure with a volume of 136.3 cm<sup>3</sup>. Table 2 shows the detailed crystal info for Anatase TiO<sub>2</sub>. The lattice space was calculated using the HRTEM images which is further discussed in section 3 of results and discussions under sub heading of Anatase TiO<sub>2</sub> analysis.

**Table -2:** Crystal information for Anatase TiO<sub>2</sub>

Sr. No.	Miller Indices (hkl)	Lattice space (d), Å	Intensity (f)
1	101	3.52	100
2	103	2.431	10
3	004	2.378	20
4	112	2.332	10
5	200	1.892	35
6	105	1.6999	20
7	211	1.6665	20
8	213	1.493	4
9	204	1.4808	14
10	116	1.3641	6
11	220	1.3378	6
12	107	1.2795	2



**Fig -1:** Bright Field image of Aluminium Foil

Dark field images are produced using the primary aperture. The image is produced by scattered electrons. Regions where no scattering occurs, such as where the primary electron beam passes straight through the sample, appear black. This technique is useful in studying crystal defects. Figure 2 shows a bright field image of aluminium foil obtained during TEM.

13	215	1.2649	10
14	301	1.2509	4
15	008	1.1894	2
16	303	1.1725	2
17	224	1.1664	6
18	312	1.1608	4
19	217	1.06	2
20	305	1.0517	4

Philips CM 12 TEM equipment was used to analyze all the images at 120 kV and it can work with various holders such as single tilt, double tilt and cryo specimen holders.

### 3. RESULTS & DISCUSSIONS

#### 3.1 Aluminium Foil Analysis

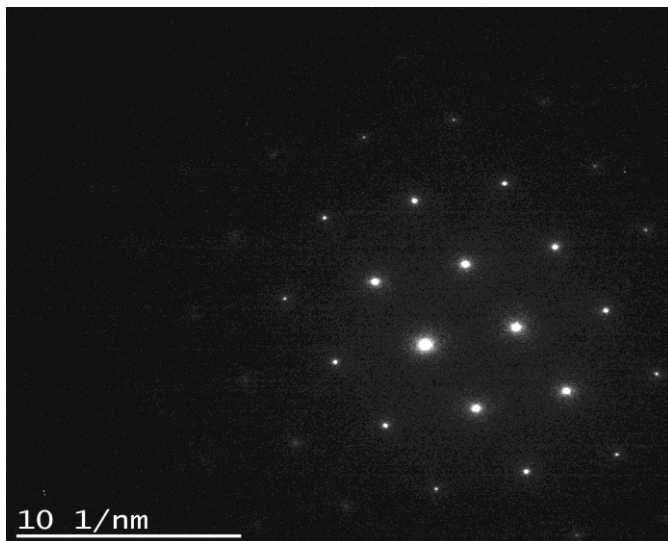


Fig -3: Selected Area Diffraction Pattern of Aluminium Foil

Figure 3 shows a selected area diffraction pattern obtained from an aluminum foil. Consider the marked areas in the figure as R<sub>1</sub>, R<sub>2</sub>, and R<sub>3</sub> and angles φ<sub>1</sub> and φ<sub>2</sub> for the indexing as shown in figure 4.

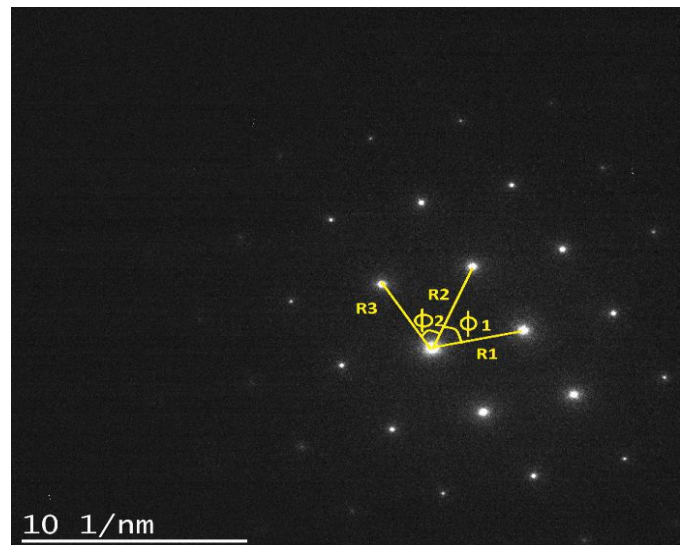


Fig -4: Selected Area Diffraction Pattern of Aluminium Foil Representing Marked Areas

First, the measured values for R<sub>1</sub>, R<sub>2</sub>, and R<sub>3</sub> are 2.85 cm, 3.3 cm, and 2.85 cm respectively with measured angles, φ<sub>1</sub> and φ<sub>2</sub>, as 53.8° and 55.3° respectively. From these values, the ratios are calculated, R<sub>1</sub>/ R<sub>2</sub>, R<sub>2</sub>/ R<sub>3</sub>, and R<sub>1</sub>/ R<sub>3</sub> as 0.866, 1.155 and 1 respectively. On comparing these ratios with the interplanar spacing table, the values of {hkl} indices were obtained which are 111 for R<sub>1</sub> & R<sub>3</sub>, and 200 for R<sub>2</sub>.

Assume h<sub>1</sub>k<sub>1</sub>l<sub>1</sub> as 111 and h<sub>2</sub>k<sub>2</sub>l<sub>2</sub> as 200. From the crystal information, the lattice space for 111 is 0.2338 nm and for 200 is 0.2024 nm. As image has scale 10 nm<sup>-1</sup> so the values of lattice space should be converted. The values are converted on the scale as 7 cm is equal to 10 nm<sup>-1</sup>. So, values of lattice space become 2.99 cm for lattice plane 111 and 3.45 cm for lattice plane 200. The rest of the lattice planes are calculated by using vector addition and the result is shown in figure 5.

Calculating angles mathematically by using equation 1:

$$\cos\phi = \frac{h_1h_2 + k_1k_2 + l_1l_2}{\sqrt{h_1^2 + k_1^2 + l_1^2}\sqrt{h_2^2 + k_2^2 + l_2^2}}$$

Equation 1-Calculation of angle

Putting the values of lattice plane in equation 1 and calculating to find out correct angles.

$$\phi_1 = \phi_2 = \cos^{-1} \frac{0 + 0 + 2}{2\sqrt{3}} = 54.7^\circ$$

Equation 2-Calculation of φ<sub>1</sub> and φ<sub>2</sub>

Finally, we need to calculate the zone axis using equation 3.

$$[UVW] = [k_1l_2 - k_2l_1, l_1h_2 - l_2h_1, h_1k_2 - h_2k_1]$$

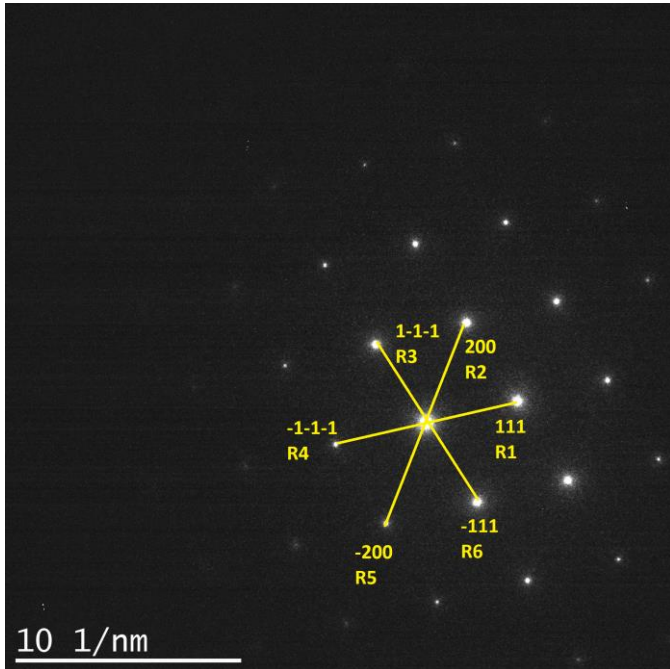
Equation 3- Calculation of zone axis



$$[111] [200] = [02-2]$$

Equation 4-Calculation of zone axis

Zone axis for the SAED in figure 3 is  $[02-2]$ .



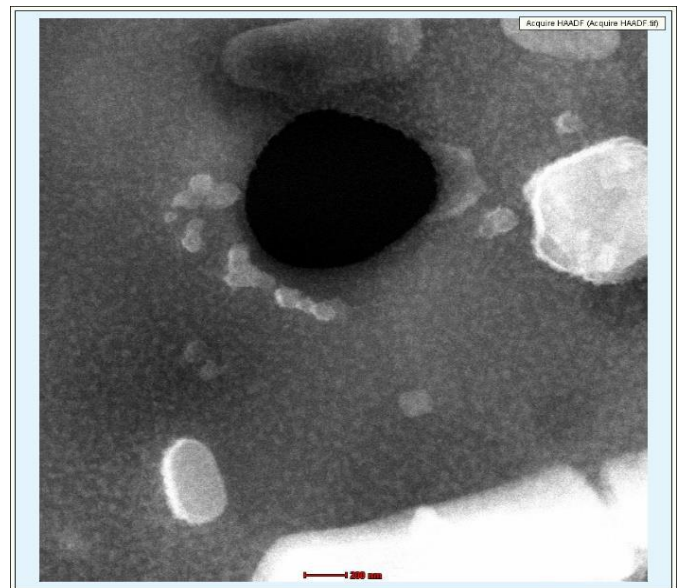
**Fig -5:** Selected Area Diffraction Pattern of Aluminium Foil Representing Lattice Space Calculated Using Vector Addition

Figure 6 shows a HAADF image of Al foil. It is dark field image. The dark hole shown in the figure is due to the defect or empty space inside the aluminium foil. The brighter regions in the space are confirms the presence of some of the impurities in the cubic structure of the foil.

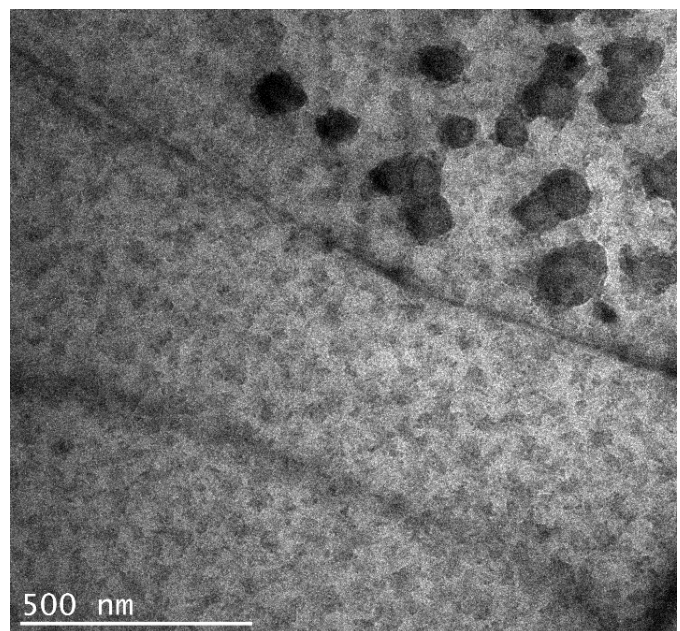
Figure 7 shows a bright field image of the foil. The dark grain like structure is due to the presence of iron and silicon impurities present on the surface of the foil whereas the bright region shows the structure of aluminium foil.

Figure 8 shows a dark field image. In the figure, the dark space represents the structure of the aluminium while the bright phase can be due to the dislocation of the aluminium atoms.

Figure 9 shows a bright field image with the presence of iron and silicon impurities. The difference between bright field and dark field image is that the surface of foil appears to be bright in bright field image whereas the surface appears dark in dark field image. Also, in the dark field image, the defect or impurities in the main crystal appear bright due to which the analysis of the defects in the crystal becomes easy.

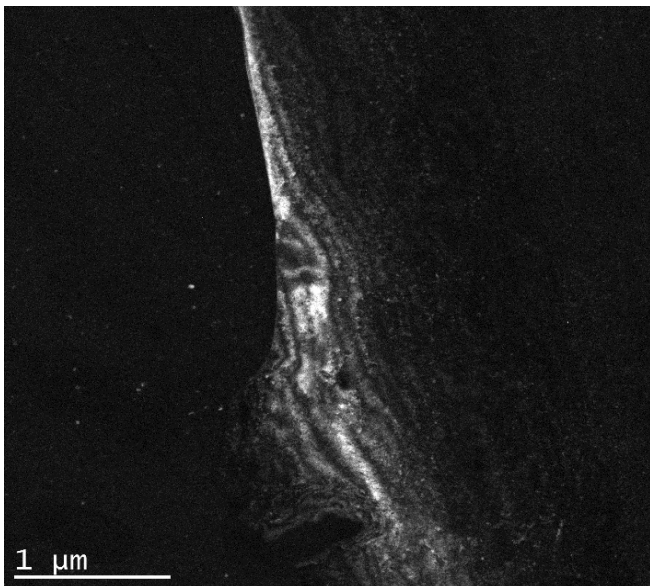


**Fig -6:** High Angle Annular Dark Field Scanning Transmission Electron Microscopy Image of Aluminium Foil

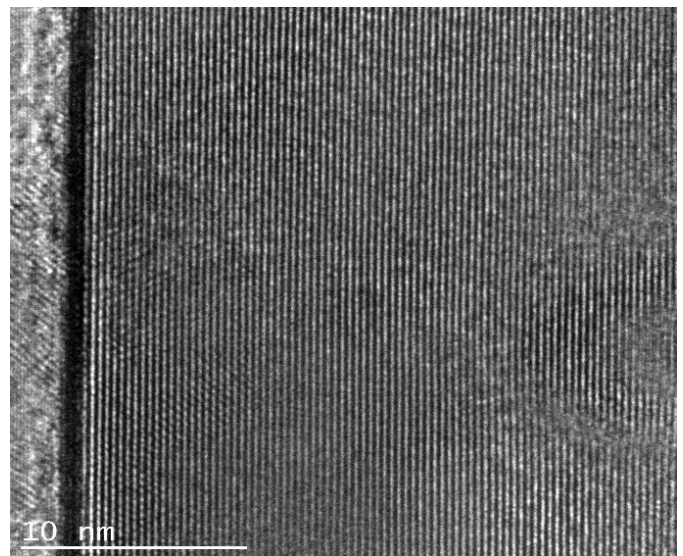


**Fig -7:** Bright Field Image of an Aluminium Foil

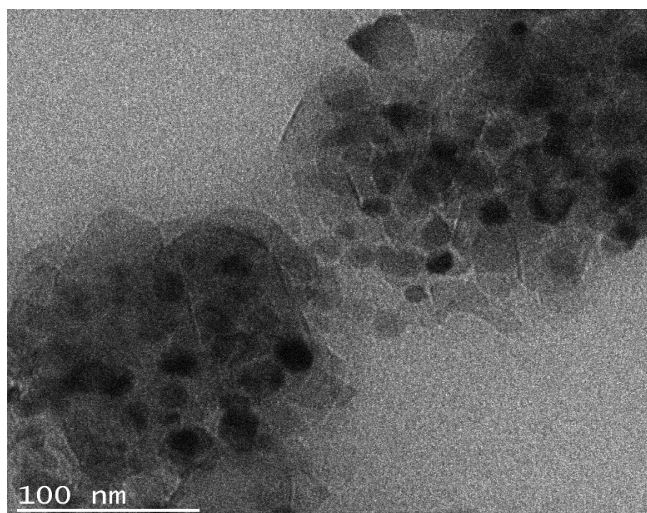




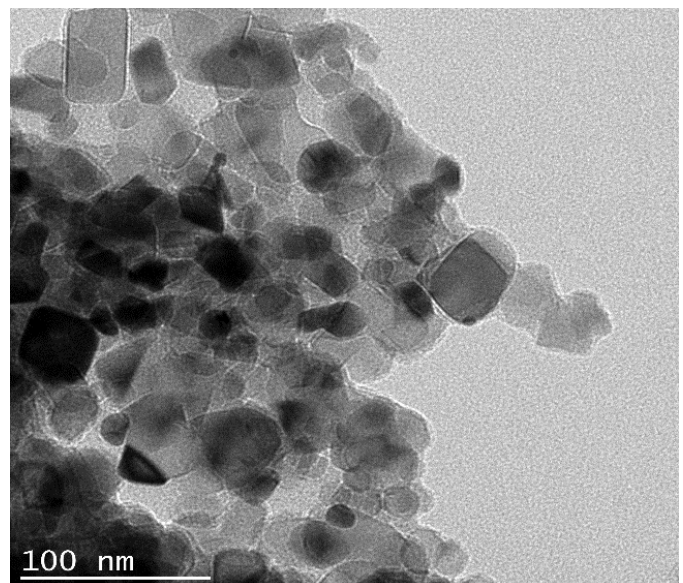
**Fig -8:** Dark Field Image of an Aluminium Foil



**Fig -10:** One Dimensional Fringe Image of Anatase TiO<sub>2</sub> in High Resolution Transmission Electron Microscopy (HRTEM)



**Fig -9:** Bright Field Image of an Aluminium Foil



**Fig -11:** High Resolution Transmission Electron Microscopy (HRTEM) Image of Anatase TiO<sub>2</sub>

### 3.2 Anatase TiO<sub>2</sub> Analysis

Figure 10 shows a one-dimensional fringe image obtained from anatase TiO<sub>2</sub> under HRTEM. The lattice space (d-space) was calculated using the scale of 5.5 cm as 10 nm. The lattice space was found out to be 0.545 nm.

Figure 11 shows another HRTEM image. The black crystals show the different orientation and is present due to the reaction of oxygen with impurities present in the atom. The bright part corresponds to the atom position.

STEM can be used to obtain more better results. STEM detectors can be inserted in the back focal plane of the objective lens. Nano-beam illumination is used for STEM which gives better results.

### 4. CONCLUSIONS

The first bright spot on SAED pattern for lattice plane 111 at diffraction angle 54.7° and lattice space 0.2338 nm was found at 2.99 cm. The second bright spot for lattice plane 200 at diffraction angle 54.7° and lattice space 0.2024 nm was found at 3.45cm. The zone axis was also determined for the Al foil. The impurities of iron and silicon were confirmed by TEM in Al foil. The lattice space was calculated in HRTEM. Calculated value of lattice space was found to be 0.545 nm.

More better technique or instruments like STEM can be employed to study the crystals which can give very high resolution and better contrast images as obtained by regular TEM. This technique can be helpful to analyze the crystal in a more precise way. Cryo TEM could also have been the more precise and better way to analyze the crystals. By implementing the above methods used for calculating the lattice space, results from cryo TEM could also be determined.

## REFERENCES

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## BIOGRAPHIES



Patience, attitude and discipline are the traits that completely define. Hardwork and enthusiasm to work is my nature. Exploring and working on new things and gifting them to the world in the form of writing is my passion.