



Comments on the paper “How to calculate crystallite size from x-ray diffraction (XRD) using Scherrer method” by Siti Fatimah et al. published in ASEAN Journal of Science and Engineering 2 (2022) 65

Paweł E. Tomaszewski*

¹ Institute of Low Temperature and Structure Research, Polish Academy of Science, Wrocław, Poland

Correspondence: E-mail: petomasz1@wp.pl

ABSTRACT

My comments concern the error in the crystallographic analyses presented in the commented paper “How to calculate crystallite size from x-ray diffraction (XRD) using Scherrer method” by Siti Fatimah et al. published in ASEAN Journal of Science and Engineering 2 (2022) 65.

© 2023 Universitas Pendidikan Indonesia

ARTICLE INFO

Article History:

Submitted/Received 02 Jan 2023

First Revised 24 Feb 2023

Accepted 11 Apr 2023

First Available online 12 Apr 2023

Publication Date 01 Dec 2023

Keywords:

Errors,

Powder diffraction,

X-ray diffraction.

1. INTRODUCTION

Fatimah *et al.* (2022) presents a scholarly text on the calculation of the crystallite size from X-Ray diffraction using the Scherrer method. Unfortunately, this paper suffers from some wrong information.

2. METHODS

Giving comments concern the error in the crystallographic analyses presented in the commented paper “How to calculate crystallite size from x-ray diffraction (XRD) using Scherrer method” by Siti Fatimah *et al.* published in ASEAN Journal of Science and Engineering 2 (2022) 65.

3. RESULTS AND DISCUSSION

Several comments are in the following:

- (i) The authors introduced a new, previously unknown, type of angels – *Bragg angels*. Seriously speaking, the small interchange of positions of two letters, *l* and *e*, leads to the interesting result: the confusing change of “angle” to “angel”. The correct term is “Bragg angle”. In Euclidean geometry, an angle is the figure formed by two rays (see **Figure 1**), called the *sides* of the angle, sharing a common endpoint, called the *vertex* of the angle. *Angle* is also used to refer to the measure of an angle. However, an angel, in various theistic religious traditions, is a supernatural spiritual being who serves God (see **Figure 2**).

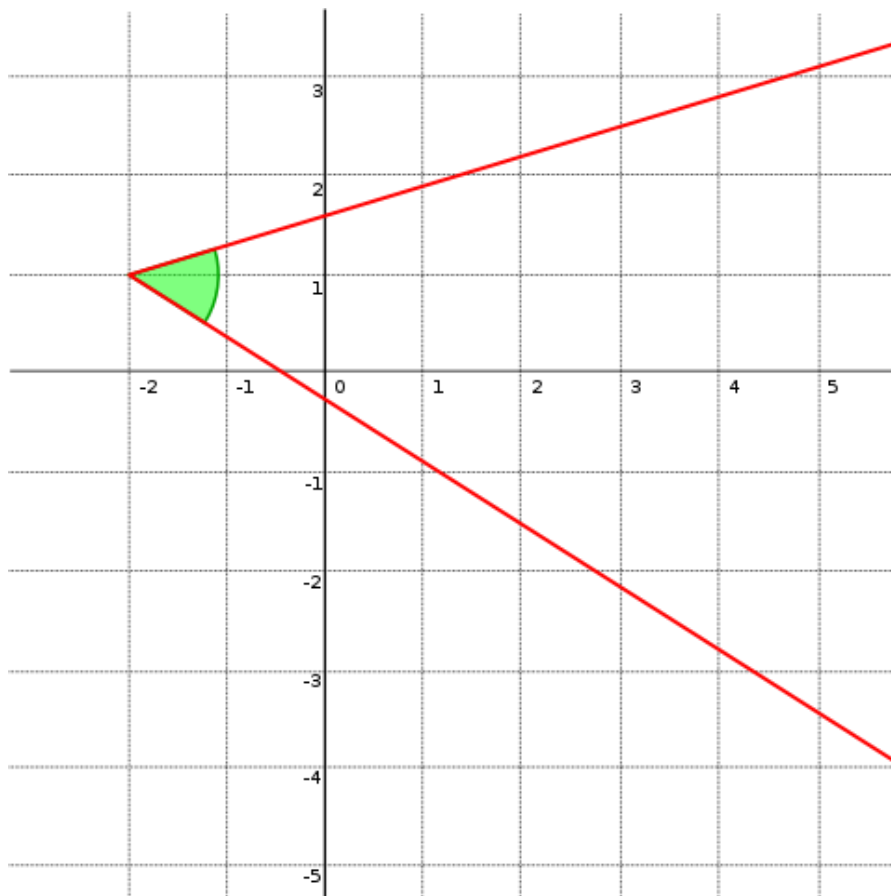


Figure 1. The angle.



Figure 2. The angel.

- (ii) The “crystalline area” is erroneously shown as the triangle goes down to the baseline (see the red oval in **Figure 3** in [Fatimah et al.'s \(2022\)](#) paper). The correct peak area is only the part of the triangle above the “amorphous area”, *i.e.* above the registered diffraction pattern (see the green oval).

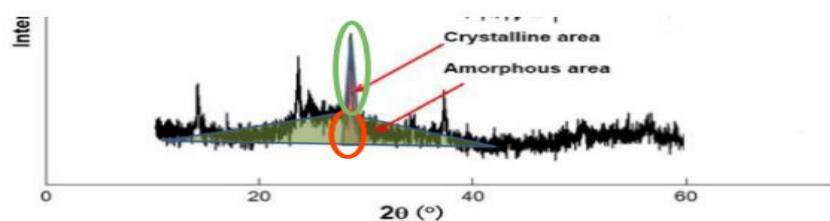


Figure 3. The incorrect identification of “crystalline area” (see the text).

- (iii) The equation on FWHM shown in chapter 2.3, step 5 in [Fatimah et al.'s \(2022\)](#) paper is incorrect (see Eq. 1).

$$\beta = \frac{1}{2} (2\theta_{max} - 2\theta_{min}) \quad (1)$$

The above equation is not correct. The correct definition is in the name FWHM: Full Width at Half Maximum (see **Figure 4**).

$$\beta = \frac{1}{2} (2\theta_{max} - 2\theta_{min})$$

Figure 4. Focused equation.

Thus, the data of β shown in Figures 8, 9, and 19 in [Fatimah et al.'s \(2022\)](#) paper are not correct – they should be two times larger. Thus, the results of subsequent calculations presented in Table 2 are also wrong.

- (iv) Figures 3, 4, and 5 in [Fatimah et al.'s \(2022\)](#) paper are only schematic images of the diffraction peaks. However, the diffraction peaks in Figures 8, 9, and 10 in [Fatimah et al.'s \(2022\)](#) paper are not correctly analyzed. The diffraction pattern is obtained using the $\text{CuK}\alpha$ radiation. The main feature of this radiation is that the diffraction peak contains two parts derived from $\text{CuK}\alpha_1$ ($\lambda=0.15406$ nm) and $\text{CuK}\alpha_2$. The part of the peak from this second wavelength has half intensity of the main peak. Thus, this contribution must be separate before measuring the FWHM of a given diffraction peak. That is why, the correct FWHM is smaller than those presented in the figures. The resulting value of crystallite size is greater than those presented in Table 2.

Note, that for the Bragg angles of about 30° such splitting of the Bragg peak is not visible but it exists and must be taken into account.

- (v) There is one mistake in Chapter 2.3 of [Fatimah et al.'s \(2022\)](#) paper. The angle Θ should be taken in *degrees* and not *radians*. The β is in radians.
- (vi) There is another error. Figure 7 of [Fatimah et al.'s \(2022\)](#) paper contains two diffraction patterns obtained using two different wavelengths of X-rays: the reference data are of cobalt X-ray (see on the left bottom corner) while experimental data (Figure 7a of [Fatimah et al.'s \(2022\)](#) paper) are of copper X-ray. The similarity of both patterns is very good, but the Bragg angles are different due to the different wavelengths used. If we compare both patterns, as made in Figure 4 of [Fatimah et al.'s \(2022\)](#) paper, it is clear that the studied peaks (presented in Figures 8-10 of [Fatimah et al.'s \(2022\)](#) paper) are from the reference pattern and not the experimental pattern. Therefore, the data shown in Table 2 of the commented paper are not correct. The angles are from the data based on $\text{CuK}\alpha$ while the wavelength used is from $\text{CoK}\alpha$ (see [Figure 5](#)). Thus, the subsequent calculation is wrong.

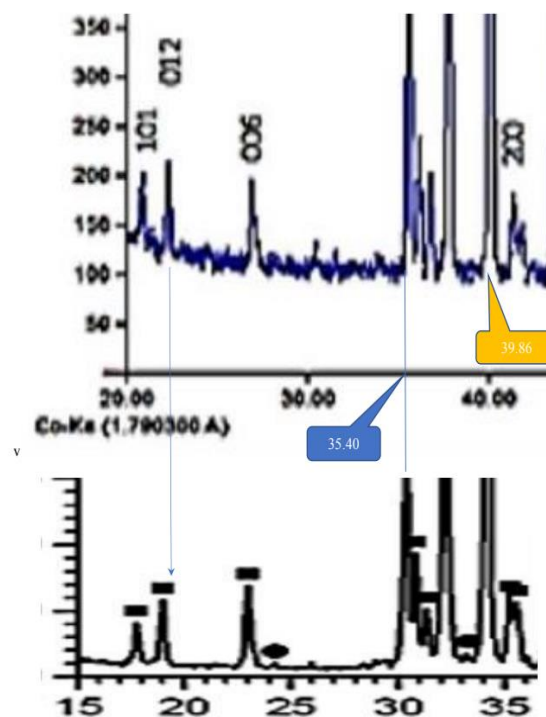


Figure 5. Some parts of the diffraction pattern obtained by two different wavelengths.

4. CONCLUSION

Finally, it is necessary to correct the above erroneous parts of the commented paper.

5. AUTHORS' NOTE

The authors declare that there is no conflict of interest regarding the publication of this article. Authors confirmed that the paper was free of plagiarism.

6. REFERENCES

- Fatimah, S., Ragadhita, R., Al Husaeni, D. F., and Nandiyanto, A. B. D. (2022). How to calculate crystallite size from x-ray diffraction (XRD) using Scherrer method. *ASEAN Journal of Science and Engineering*, 2(1), 65-76.