Effect of Slit Configuration and Optical Design on XRD Data Quality: A Comparison of FDS and BBHD Systems

Sandeep Jape, Tejaswini Ghadyale, Poonam Phagare, Chandrakala Madichetty
The Bombay Textile Research Association, LBS Marg, Ghatkopar (W), Mumbai 40086, India.

Abstract

Instrumental optics and slit configurations play a major role in defining the quality of powder X-ray diffraction (XRD) data. In this study, the effect of divergence slit and Antiscatter Slit variation on XRD pattern was systematically investigated using a PANalytical Empyrean X-ray diffractometer equipped with Bragg-Brentano HD (BBHD) optics and fixed divergence slit (FDS) optics, using zinc oxide (ZnO) as an experimental sample. Divergence and anti-scatter slits were varied, while all other instrumental parameters, including scan conditions,were kept constant, enabling a controlled and reproducible methodological comparison. The diffraction data were quantitatively analysed in terms of background intensity (IB), highest peak intensity(IP), peak-to-background intensity ratio (IP/IB), and full width at half maximum (FWHM). The results demonstrate that divergence slit selection strongly influences background and peak intensity, while relatively marginally influencing FWHM, revealing a clear trade-off between intensity and resolution. Using optimum slit configuration and BBHD optics we identified the zinc oxide phase from the crystallographic open database (COD) with high degree of accuracy. On the basis of this study, we are providing a practical approach for optimizing instrumental settings for reliable phase identification.

Key words:

Optics, fixed divergence slit (FDS), Bragg-Brentano HD (BBHD), full width at half maximum (FWHM)

Citation

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1. Introduction:

X-ray Diffraction (XRD) serves as the primary analytical tool for evaluating phase purity, calculating lattice parameters, and determining crystallite size.[1]However, the quality of the resulting data is highly influenced by the specific optical configuration of the diffractometer, making the choice between fixed slit and mirror-based systems a critical experimental consideration.[2]

The PANalytical Empyrean XRD uses a ϴ–2ϴ Bragg–Brentano geometry in which the X-ray source is kept fixed, while the sample and detector move. In this geometry, the incident beam makes an angle ϴ with the sample, while the detector is placed at an angle 2ϴ with respect to the incident beam as shown in Fig:1a. The fixed source and the receiving slit (placed before the detector) lie on a circle (diffractometer circle) of fixed radius, with the sample at the center. A divergent beam falls on the surface of the sample. A focusing circle is formed by the source, sample surface, and receiving slit, whose radius varies with the incidence angle to ensure that a convergent beam reaches the detector.[3-5]

The standard Fixed Slit System (FDS), based on Bragg–Brentano geometry, uses physical slits to control the X-ray beam. The divergence slit controls how wide the beam spreads in the equatorial direction and how much of the sample is illuminated. Soller slits reduce unwanted beam spreading in axial direction to help maintain proper peak shape. The beam mask controls the height of the beam.[3, 6]This setup provides high intensity, which is suitable for bulk powder samples. The system also uses physical filters, such as a nickel filter, to remove K-beta radiation.[7]

An important improvement in focusing geometry is provided by the Malvern PANalytical Bragg–Brentano High Definition (BBHD) optics. Unlike the FDS system, which controls the beam mainly by blocking it with slits, the BBHD optics uses a high-definition multilayer mirror [8]. This mirror reduces K-beta radiation and background intensity while still keeping a strong, focused beam.[8-10]Comparing BBHD with the FDS system is scientifically meaningful, as

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*Corresponding author,
E-mail: polyfrlab@btraindia.com

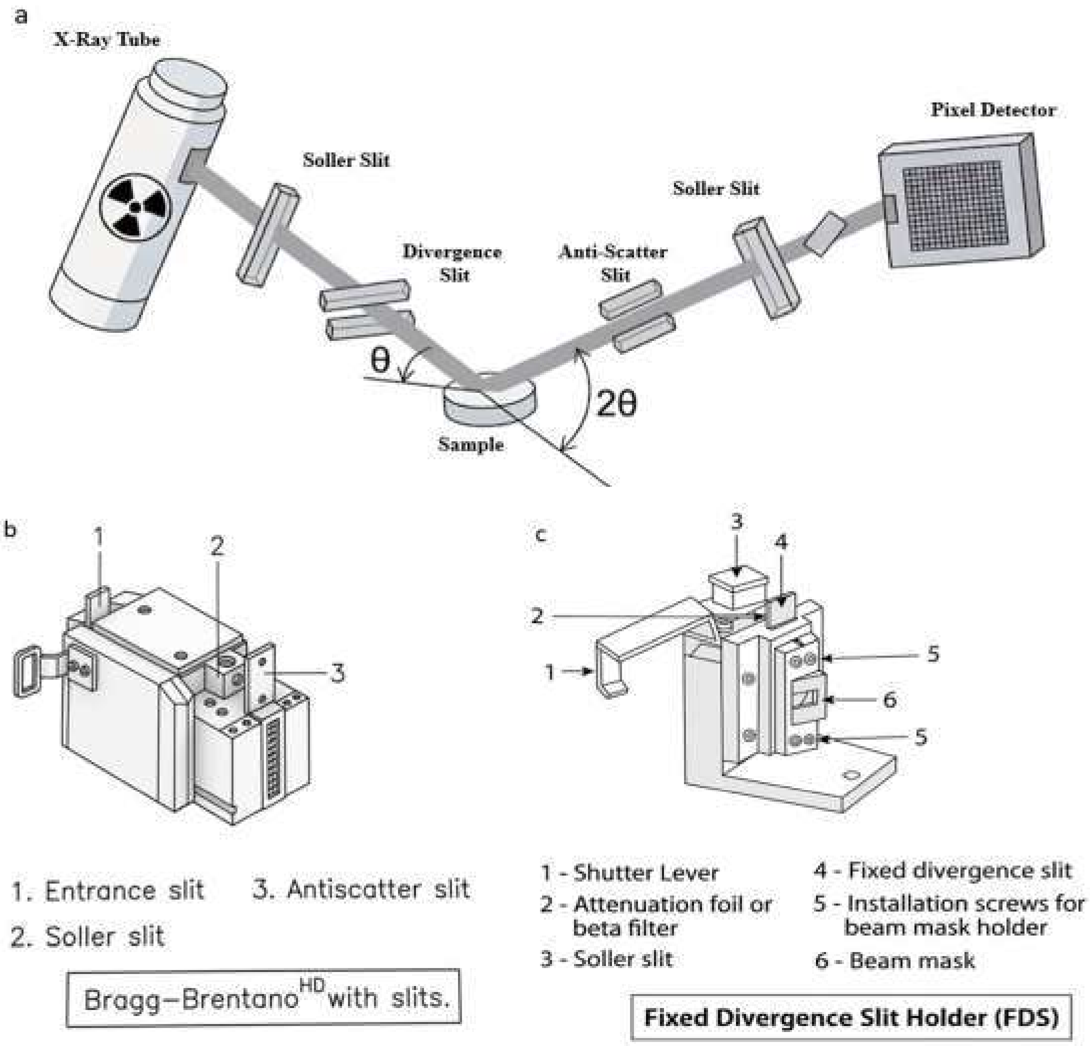

Fig:1 :- a) XRD Instrumental Layout[6],b)Bragg-Brentano HD(BBHD) with slits and c) Fixed Divergence Slit Holder (FDS). Adapted from reference[7].

it helps evaluate how changes in optics influence signal parameters, which can result in more accurate material identification.

Zinc Oxide (ZnO) is a highly versatile inorganic material with widespread applications ranging from industrial rubber vulcanization and UV-protective coatings to sophisticated optoelectronic devices such as varistors and solar cell buffer layers [11].To study the comparison of BBHD and FDS system, ZnO was used as an experimental sample. By analysing ZnO powders using both FDS and BBHD configurations, this study aims to measure the improvements in peak shape and intensity achieved with high-definition optics. While the earlier studies such as that by Zhang et al[2] focussed on studying the influence slitson the peak background, intensity and resolution using calcium carbonate, the current study seeks to expand the work to find out whether changing from traditional fixed-slit systems to multilayer focusing optics is really needed to achieve reliable quality control and accurate structural analysis in routine work.

Experimental Details:-.

To study how instrumental parameters affect peak intensity, resolution, and background intensity in XRD patterns, this experiment was carried out by changing the divergence slit, anti-scatter slit, and optic configuration. The experiment was performed using the EMPYREAN diffractometer with HighScore Plus software and Cu Kα radiation. The operating conditions were 40 kV voltages and 30 mA current. The scan range was from 25° to 80° (2θ), with a step size of 0.013° and a scan speed was 0.067(°/s). ZnO was purchased from MERCK. The detailed experimental parameters are listed in the table 1, 2 below.

	Incident Beam Side		**Diffraction Beam Side**
Beam Mask (mm)	Antiscatter Slit (°)	Divergence Slit (°)	Receiving Slit(mm)
4	0.125	0.03125	7.5
4	0.25	0.0625	7.5
4	0.5	0.125	7.5
4	1	0.25	8

Table-1: Details of Slits and Beam Mask with BBHD Detector:

The slit combinations were chosen to match the incident beam to a standard 16 mm diameter sample holder, following the guidelines provided by Malvern PANalytical.[12]. The beam mask on the incident beam side and the receiving slit on the diffracted beam side were kept constant in most of the measurements except for the 0.25° diversion slit in the BBHD experiments, where the receiving slit on diffraction side was increased to 8mm in order to fit beam within the sample holder. The machine and optics outline are presented in Fig.1 (a, b and c). Soller slits of 0.04 rad were used in both incident and diffracted beam side.

Incident Beam Side			Diffraction Beam Side
Beam Mask (mm)	Antiscatter Slit (°)	Divergence Slit (°)	Receiving Slit (mm)
10	0.0625	0.03125	7.5
10	0.125	0.0625	7.5
10	0.25	0.125	7.5
10	0.5	0.25	7.5

Table-2: Details Of Slits and Mask with FDS Detector

Results and Discussion: -

The XRD graphs obtained from BBHD and FDS are in fig. 2 and fig.3.The displayed scan range has been shortened for clarity. ZnO powder showed clear diffraction peaks with the highest peak at around 36.18° corresponding to the (101) plane. [11]. From the scans, the following data were collected, minimum background intensity (IB), maximum peak intensity (IP), the ratio of peak to background (IP/IB) intensity, and the full width at half maximum (FWHM) and the results are presented in fig.4 & 5.

From the BBHD graphs, it can be seen that both the background and the highest intensity of the ZnO peak at 36.18° increase as the divergence slit width (beam size) increases. (Fig.4 a and b). The IP/IB ratio which can be an indication of the quality of the diffractogram. [5]after an initial increase decreases consistently with increasing slit width because the background rises faster than the peak intensity. (Fig.4-c) The most suitable slit width for BBHD, balancing peak intensity without losing low-intensity peaks due to background rise, is 0.0625°, as larger slit widths do not significantly improve the signal.

For the FDS system, both the background and peak intensity also increase with slit width. (Fig.5 a and b).Compared to BBHD, FDS shows lower peak intensity at small slit widths,it displays higher values for higher slit widths. In addition, FDS displays higher background intensity for comparable slit width as compared to BBHD . The IP/IB ratio for FDS becomes nearly constant beyond 0.0625° slit width. (Fig.5-c). Considering both peak and background intensity, the optimal slit width for FDS is 0.0625°.Overall, BBHD provides a higher IP/IB ratio at comparable slit widths, and its peaks start from a very low background level, making it more suitable for detecting low-intensity peaks. (Fig.2)

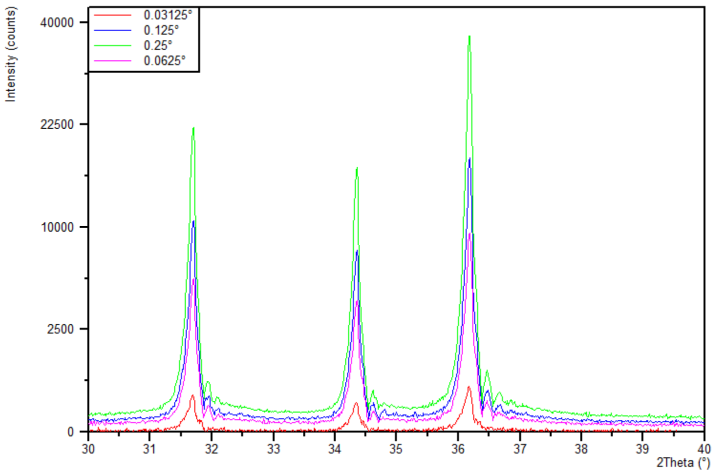

Fig.2: - XRD patterns obtained from BBHD and different divergence slits widths

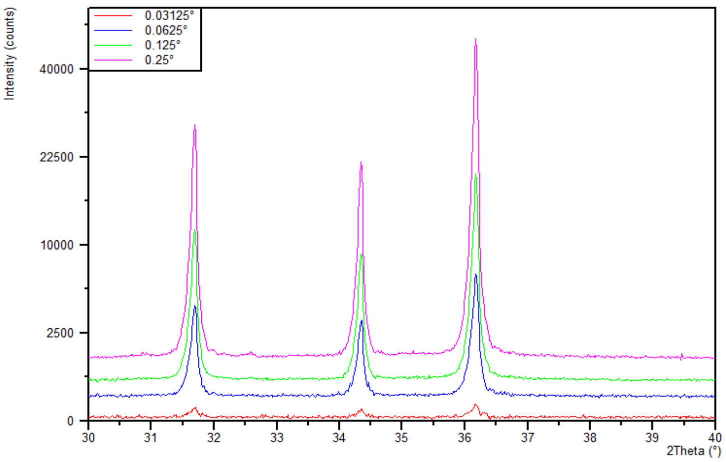

Fig.3: - XRD patterns obtained from FDS and different divergence slits widths

The FWHM and highest peak intensity were taken from 36.18°peak.

From the graphs, it can be seen that the FWHM values tend to level off at higher slit widths in both systems. BBHD generally shows slightly higher FWHM compared to FDS. A smaller FWHM indicates less peak broadening, meaning the peak widths are closer to their true values. Although BBHD exhibits slightly more peak broadening, the difference from FDS is not significant. (Fig.4-d and Fig.5-d).

Using the optimizedslit configuration using the BBHD optics we have identified our sample from crystallographic open database (COD) database as Zincite having ref. code no 96-900-4180 which shows the validity of the slit combination.(Fig.6). For the same slit combination, the reference has a score match of 60 as compared to FDs which has a score match of 51 for the same, which would suggest that the selection of the optics led to a higher degrees of accuracy of identification of the material in the BBHD system.

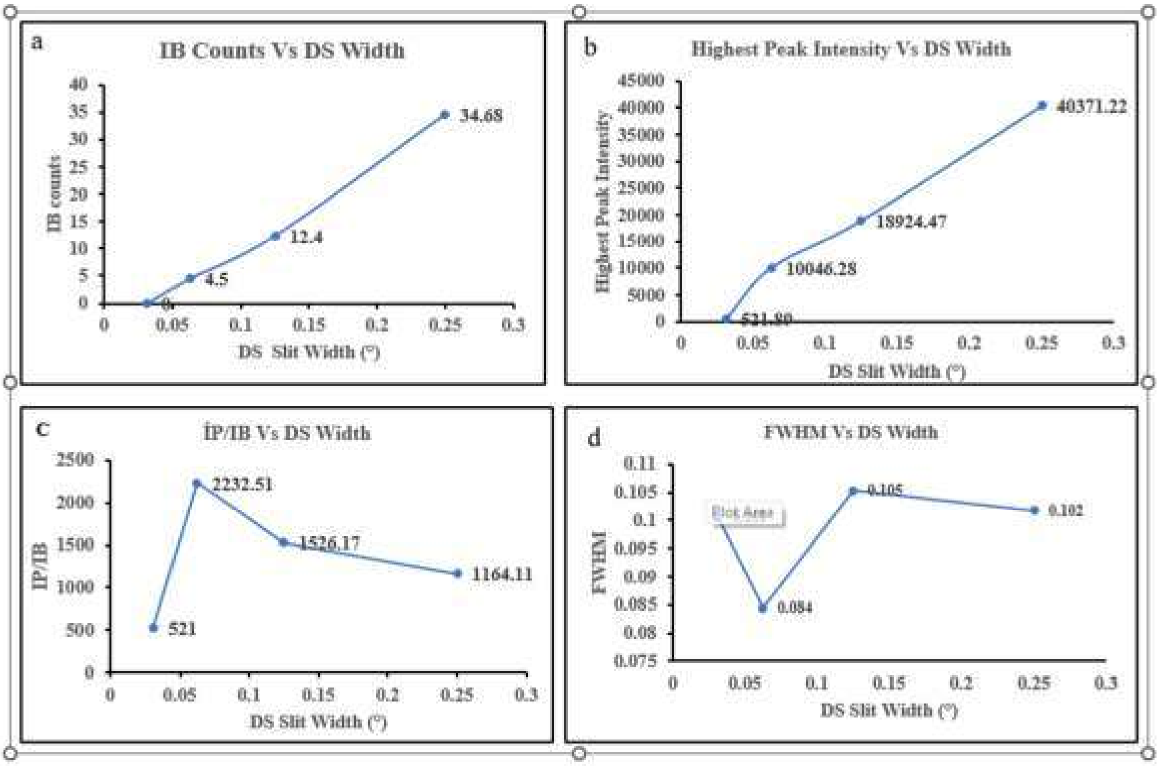

Fig-4:- For the BBHD optics the effect of Divergence Slit width on a) Background intensity b) Highest Peak Intensity c) IP/IB i.e. ratio of Highest peak intensity/Background Intensity and d) FWHM, The FWHM and highest peak intensity were taken from 36.18°peak

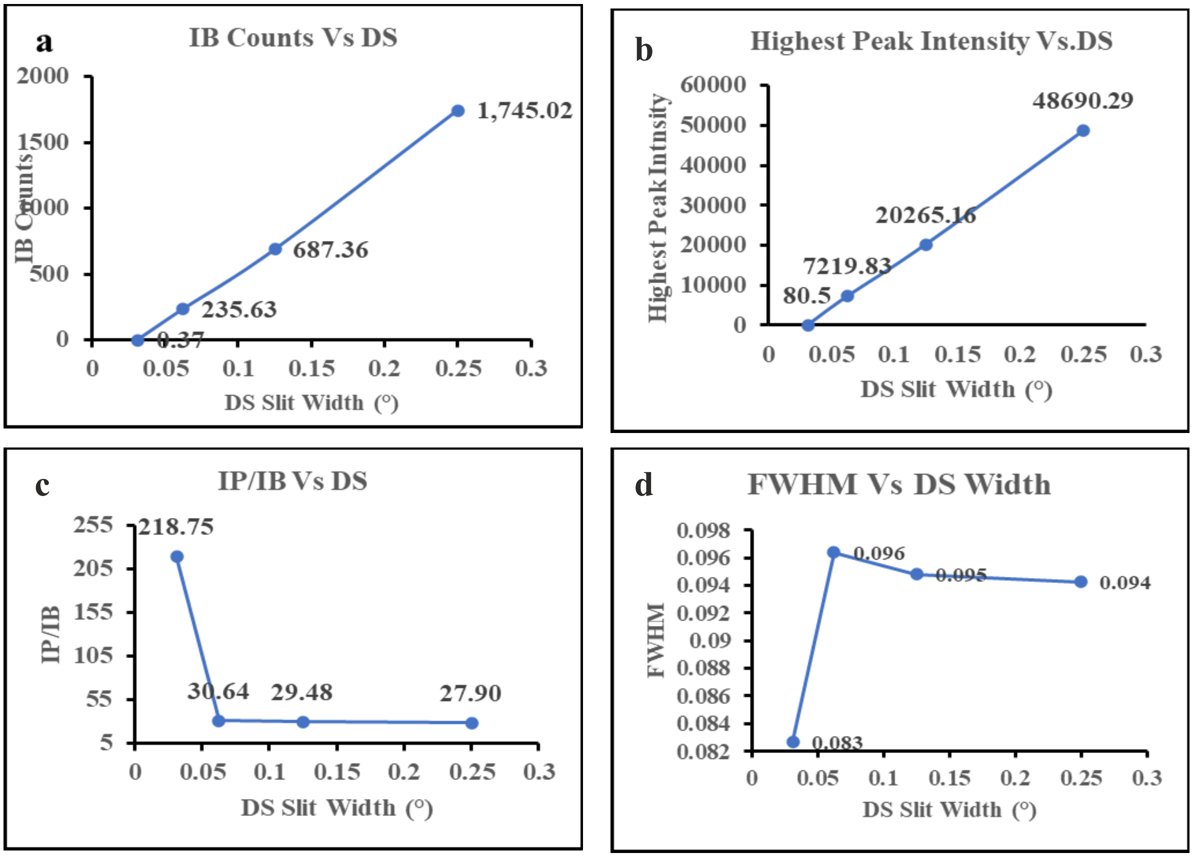

Fig.-5:- For the FDS optics the effect of Divergence Slit width on a) Background intensity b) Highest Peak Intensity c) IP/IB i.e. ratio of Highest peak intensity/Background Intensity and d) FWHM,

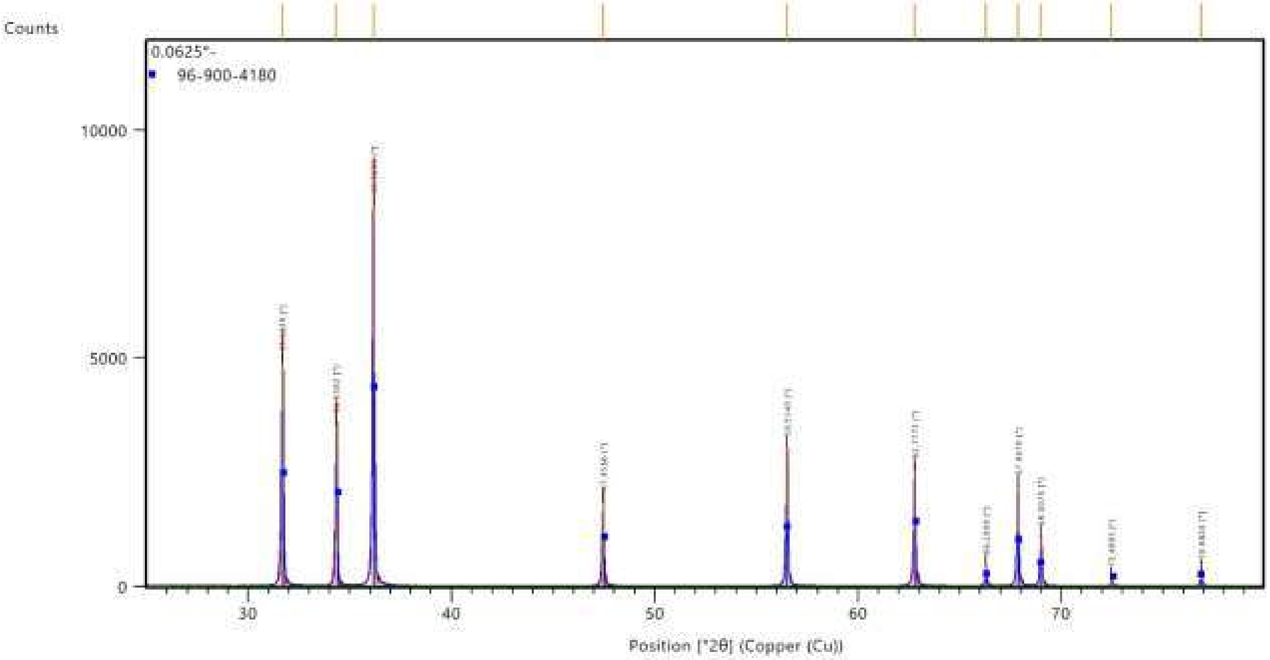

Fig-6. Phase match ZnO sample with Zincite having ref. code no 96-900-4180 in COD data base.

Conclusion:

In this study systematically evaluated the influence of divergence and anti-scatter slits in both Bragg–Brentano HD (BBHD) and Fixed Divergence Slit (FDS) optics, using ZnO as a sample, clear trends were observed in the obtained XRD diffractograms. The use of BBHD optics, combined with appropriate slit selection, enhances data quality by improving peak to background intensity ratio. For BBHD, a divergence slit of 0.0625° offered the best balance between intensity and background and for FDS, a divergence slit of 0.0625° was also most suitable, yielding reliable peak intensity without excessive noise.Although BBHD showed slightly higher FWHM values (indicating minor peak broadening), the difference from FDS was not significant and did not compromise structural analysis.

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