

Article

Core Diffraction Signatures as a Reproducible XRD Method for Structural Identification and Microstructural Assessment of Body-Centered Cubic α -Fe

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Abstract

Powder X-ray diffraction was used to validate the crystal structure of iron metal powder and to demonstrate a transparent workflow for extracting crystallographic and microstructural information from a simple laboratory dataset. The diffraction profile collected over the angular range of approximately 36° to 95° contains three dominant reflections located at $2\theta = 44.850^\circ$, 65.218° , and 82.540° . Conversion of the peak positions into d-spacings using Bragg's law gives values of approximately $2.020 \text{ \AA}$, $1.430 \text{ \AA}$, and $1.170 \text{ \AA}$. The squared-sine ratios, when referenced to the first reflection, follow the sequence of 1:2:3, which is characteristic of the allowed reflections of a body-centered cubic lattice when multiplied by the first allowed value of $N = h^2 + k^2 + l^2 = 2$. Therefore, the peaks are assigned to the (110), (200), and (211) reflections of α -Fe. The extracted lattice constants are 2.853, 2.860, and $2.865 \text{ \AA}$, yielding an average value of $2.859 \text{ \AA}$, which is close to the accepted room-temperature value of approximately $2.866 \text{ \AA}$ for α -Fe. Peak-width calculations based on the observed, instrument-uncorrected FWHM values are included only as illustrative apparent line-broadening indicators. Because an external instrumental standard was not measured under identical conditions, no quantitative coherent-domain size or microstrain is claimed. The Williamson–Hall treatment is therefore used only to demonstrate the sensitivity of size-strain interpretation to peak breadth, profile selection, and the limited number of available reflections. The intensity hierarchy was analyzed using the body-centered cubic-structure factor, reflection multiplicity, Fe atomic form factor, and Lorentz polarization correction. The comparison between calculated and experimental intensities shows stronger disagreement than the lattice-parameter analysis, illustrating the greater sensitivity of intensity analysis to preferred orientation, specimen preparation, peak-profile selection, and background treatment. The work provides a publication-style reconstruction of an iron powder XRD experiment, connecting peak fitting, indexing, lattice-parameter determination, crystallite size estimation, size-strain analysis, and intensity interpretation in a single critical framework.

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1. Introduction

X-ray diffraction is one of the most direct experimental methods for identifying crystal structure, evaluating lattice parameters, and assessing microstructural features in crystalline and polycrystalline materials. Because the wavelength of common laboratory X-rays is comparable to interatomic spacings, a crystalline solid acts as a periodic scattering medium that produces constructive interference only at specific angles. These angles encode the spacings between lattice planes, while the peak widths and intensities provide additional information about coherent domain size, microstrain, preferred orientation, and the distribution of scattering matter inside the unit cell. For metallic powders, the diffraction pattern is especially valuable because it provides a structural fingerprint of the phase present in the sample and distinguishes between different Bravais lattices with relatively simple indexing rules [1–3].

Iron is a particularly important material for powder XRD validation. At room temperature and ambient pressure, pure iron exists predominantly as α -Fe, which has a body-centered cubic structure. This phase is fundamental to metallurgy, magnetic materials, steel science, and solid-state physics. The bcc structure of α -Fe is also pedagogically useful because its diffraction pattern contains a small number of strong reflections in the measured angular range, and these reflections follow a simple selection rule. In a body-centered cubic lattice, diffraction occurs only when $h + k + l$ is even. Therefore, the first three strong reflections are expected to correspond to the (110), (200), and (211) planes. Thus, the iron powder pattern offers an ideal opportunity to demonstrate how Bragg-law analysis, squared-sine indexing, lattice-parameter extraction, and structure-factor reasoning can be combined in a coherent workflow [4–9].

The purpose of the present manuscript is to convert the iron powder laboratory dataset into a publication-style structural analysis. The interpretation is specifically adapted to the crystallography of metallic iron, where the stable room-temperature phase (α -Fe) crystallizes as a monatomic body-centered cubic structure. This crystallographic arrangement determines the allowed diffraction sequence, indexing rules, lattice-parameter extraction, and structure-factor behavior. Therefore, the analysis connects peak positions, d-spacing values, cubic indexing, lattice-constant determination, peak broadening, crystallite size estimation, and relative-intensity interpretation within a unified X-ray diffraction framework. This approach provides a transparent structural validation of α -Fe powder and demonstrates how laboratory XRD data can be used to extract reliable crystallographic and microstructural information [10–15].

In this study, the fitted iron XRD peaks occur at approximately 44.850° , 65.218° , and 82.540° in 2θ . These reflections are sufficient to validate the bcc phase and to calculate the lattice constant with reasonable accuracy. The measured average lattice parameter is $2.859 \text{ \AA}$, which differs from the accepted value of α -Fe by only a small amount. The peak widths were further used to estimate apparent crystallite size through the Scherrer equation and to construct a Williamson–Hall plot for assessment of the size-strain contribution. The analysis is deliberately critical: the extracted sizes are described as coherent-domain estimates rather than direct particle sizes, and the limitations of using only three peaks for Williamson–Hall analysis are explicitly discussed. This approach prevents overinterpretation and makes the analysis suitable for manuscript-level presentation [16–21].

Beyond lattice-parameter and size analysis, this manuscript also examines the calculated and experimental relative intensities. The strongest observed peak is the (110) reflection, consistent with the high multiplicity, favorable Lorentz polarization factor, and Fe scattering factor at that angle. However, the agreement between calculated and experimental intensities is poorer than the agreement in peak positions. This behavior is not a weakness of the structural assignment; rather, it reflects the fact that intensities are much more sensitive to powder packing, preferred orientation, background subtraction, peak

overlap, K_α doublet effects, surface roughness, and counting statistics. Therefore, the work highlights the hierarchy of reliability in simple powder diffraction: peak positions provide robust phase identification and lattice constants, peak widths provide useful but approximate microstructural information, and relative intensities require the most careful experimental control [22–27].

2. Materials and Methods

2.1. Bragg's Law and d -Spacing

The starting point for the structural interpretation of the iron-powder pattern is Bragg's law, which describes the condition for constructive interference from a set of parallel lattice planes. When X-rays are scattered from adjacent planes separated by an interplanar distance of d , the path difference between the scattered waves must equal an integer multiple of the wavelength. The diffraction condition is therefore written as

$$2 d_{hkl} \sin \theta = n\lambda. \quad (1)$$

In ordinary powder-diffraction indexing, the first diffraction order is used; therefore, $n = 1$ and $d_{hkl} = \lambda/(2 \sin \theta)$. This equation converts each fitted peak center from an angular position into a real-space interplanar spacing [1,4,28–32].

For the iron dataset, the average Cu K_α wavelength was taken as approximately $\lambda = 1.542 \text{ \AA}$. The first fitted reflection at $2\theta = 44.850^\circ$, corresponding to $\theta = 22.425^\circ$, gives $d_{110} \approx 2.020 \text{ \AA}$. The second reflection at $2\theta = 65.218^\circ$ gives $d_{200} \approx 1.430 \text{ \AA}$, whereas the third reflection at $2\theta = 82.540^\circ$ gives $d_{211} \approx 1.170 \text{ \AA}$. Thus, the experimental interplanar spacings decrease systematically from the (110) to the (200) and (211) reflections. This behavior is expected for a cubic crystal because

$$d_{hkl} = a/(h^2 + k^2 + l^2)^{1/2}, \quad (2)$$

and therefore, d_{hkl} decreases as $N = h^2 + k^2 + l^2$ increases from 2 to 4 and 6 for the (110), (200), and (211) reflections, respectively. The observed trend is therefore consistent with the assigned body-centered cubic structure and does not indicate an anomalous increase in interplanar spacing at higher reflection indices.

The d -spacing values transform the diffraction pattern from a set of angular peaks into crystallographic information. Although a single d -spacing does not uniquely determine the crystal structure, the sequence of d -spacings or, equivalently, the sequence of $\sin^2\theta$ values provides information about the symmetry of the underlying lattice. For cubic materials, this relationship is particularly simple because all interplanar spacings can be expressed in terms of one lattice parameter and the Miller indices of the reflecting planes [4–7,9,12].

2.2. Cubic Indexing Relation

For a cubic crystal, the interplanar spacing is related to the lattice parameter (a) through $1/d_{hkl}^2 = (h^2 + k^2 + l^2)/a^2$. Combining this equation with Bragg's law gives a $\sin^2\theta$ proportional to $h^2 + k^2 + l^2$ for a fixed wavelength. Therefore, if the first observed peak is used as a reference, the ratios of $(\sin \theta/\sin \theta_0)^2$ should correspond to a simple sequence of allowed N values, where $N = h^2 + k^2 + l^2$. This squared-sine method is a classical and powerful tool for indexing cubic powder patterns [4–7,12].

In the present iron dataset, the ratios obtained from the three fitted peak positions are approximately 1.000, 1.996, and 2.989. These values are very close to 1, 2, and 3. For a bcc lattice, the first allowed reflection has $N = 2$, corresponding to (110). Multiplying the observed ratio sequence by 2 gives approximately 2, 4, and 6, which correspond to the (110), (200), and (211) reflections, respectively. This sequence is characteristic of α -Fe and

differs from fcc materials, where the first allowed sequence begins with $N = 3, 4, 8, 11$, and so on [17–21].

The internal consistency of the cubic indexing is supported by the extracted lattice parameters. Using $a = d(h^2 + k^2 + l^2)^{1/2}$, the three reflections yield $a = 2.853 \text{ \AA}$ for (110), $2.860 \text{ \AA}$ for (200), and $2.865 \text{ \AA}$ for (211). The small spread among these values indicates that the peak positions are mutually consistent with a single cubic lattice. The average value of $2.859 \text{ \AA}$ is close to the accepted value of α -Fe, confirming both the bcc assignment and the reliability of the peak fitting within the limitations of the laboratory measurement [22–25].

2.3. Selection Rules and Structure Factor

The assignment of the iron peaks to a body-centered cubic lattice is not based only on the numerical ratio of peak positions; it is also supported by the bcc selection rule. A monatomic bcc unit cell can be represented by atoms at fractional coordinates of (0,0,0) and (1/2,1/2,1/2). The corresponding structure factor is $F_{hkl} = f[1 + \exp(i\pi(h+k+l))]$, where f is the atomic scattering factor of Fe. If $h+k+l$ is even, the exponential term is +1 and $F_{hkl} = 2f$, so the reflection is allowed. If $h+k+l$ is odd, the exponential term is -1 and $F_{hkl} = 0$, so the reflection is systematically absent [12–16,33,34].

This selection rule explains why the first observed reflection is (110), not (100). The (100) plane has $h+k+l = 1$ and is therefore forbidden in bcc iron. The (111) reflection is also forbidden because $h+k+l = 3$. In contrast, (110), (200), and (211) have even sums and are allowed. The absence of forbidden low-angle reflections is a strong indicator that the sample is not primitively cubic and not face-centered cubic. The sequence observed in the iron pattern therefore provides direct evidence for the bcc structure of α -Fe.

Because α -Fe is monatomic, the structure-factor interpretation is simpler than in compounds with a multi-atom basis. For bcc Fe, all allowed reflections have $F = 2f$, but the value of f decreases with the scattering angle because the electron cloud scatters less efficiently with larger momentum transfer. Therefore, the relative intensity of the reflections is controlled by a combination of the Fe atomic form factor, reflection multiplicity, the Lorentz polarization factor, thermal motion, possible preferred orientation, and experimental geometry [14–16,35–38].

2.4. Crystallite Size Estimation from Peak Broadening

Peak broadening in XRD contains information about microstructure, but it must be interpreted carefully. The simplest relationship between peak width and coherent-domain size is the Scherrer equation, i.e., $D = K\lambda/(\beta \cos \theta)$, where D is the apparent coherent-domain size, K is the shape factor, λ is the X-ray wavelength, β is the diffraction-line breadth expressed in radians, and θ is the Bragg angle. In this context, D represents the average dimension of coherently diffracting domains perpendicular to the reflecting planes. It is not necessarily equivalent to the particle size observed by microscopy, particularly for powders containing polycrystalline aggregates, defects, subgrain boundaries, or internal strain [26,39].

The measured diffraction-line breadth (β_{obs}) contains both specimen-related and instrumental contributions. Quantitative application of the Scherrer equation therefore requires determination of the instrumental profile from a strain-free reference material, such as silicon or LaB₆, measured using the same wavelength, optical configuration, scan conditions, and specimen geometry. For an ideal Gaussian treatment, the specimen contribution may be estimated from $\beta_{\text{sample}}^2 = \beta_{\text{obs}}^2 - \beta_{\text{inst}}^2$; however, mixed Voigt or pseudo-Voigt profiles require an appropriate component-wise or whole-profile deconvolution. No external standard was measured for the present dataset. Consequently, the observed FWHM

values were not deconvoluted, and calculations based on them are retained only as uncorrected apparent line-broadening estimates rather than quantitative coherent-domain sizes.

Direct substitution of the observed FWHM values into the Scherrer relation gives formal apparent values of approximately 44.8 nm for the (110) reflection, 40.9 nm for (200), and 29.6 nm for (211), with an arithmetic average of approximately 38.4 nm. Because these values were calculated from the observed rather than instrument-corrected breadths, they are reported only as illustrative coherence-length proxies. The lower value obtained for the high-angle (211) reflection may reflect unresolved Cu $K_{\alpha 1}/K_{\alpha 2}$ separation, profile asymmetry, microstrain, finite fitting precision, or angle-dependent instrumental broadening rather than a uniquely smaller coherent-domain dimension.

Accordingly, the peak-width analysis is used to demonstrate the sensitivity of the Scherrer calculation to line breadth and model assumptions, not to assign a definitive crystallite size to the Fe powder. Quantitative microstructural determination would require an external standard measured under identical conditions, explicit instrumental-profile deconvolution, and preferably Voigt or pseudo-Voigt whole-pattern modeling that treats size and strain contributions simultaneously [32–36,40–42].

2.5. Lorentz Polarization Correction and Relative Intensities

Relative intensity analysis provides a deeper test of the diffraction model because intensities depend not only on the Bravais lattice and basis but also on experimental factors. A simplified calculated powder intensity may be written as $I_{\text{cal}} \propto m_{\text{hkl}} |F_{\text{hkl}}|^2 \text{LP}(\theta)$, where m is the multiplicity of the reflecting-plane family, F_{hkl} is the structure factor, and $\text{LP}(\theta)$ is the Lorentz polarization factor. For monatomic bcc iron, the allowed reflections have $|F|^2 = 4f^2$, while forbidden reflections have zero intensity [13–16].

The atomic form factor decreases with the scattering vector because high-angle scattering probes finer spatial variations in the electron density. In the fitted iron dataset, representative form-factor values decrease from about 18.44 for the first peak to 15.21 for the second and 13.11 for the third. The Lorentz polarization factor also changes strongly with angle, decreasing from about 5.586 for the (110) reflection to 2.403 for (200) and 1.787 for (211). Reflection multiplicity partly compensates for these decreases because the (211) family has a multiplicity of 24, compared with 12 for (110) and 6 for (200) [14–16].

After normalization, the calculated relative intensities are approximately 100% for (110), 14.6% for (200), and 32.3% for (211). The experimental integrated intensities normalized to the strongest measured peak are approximately 100%, 17.5%, and 46.8%, respectively. The first two intensities agree qualitatively, while the third experimental peak is stronger than the simplified calculation predicts. This discrepancy is not surprising and does not invalidate the structural assignment. Relative intensities are highly sensitive to preferred orientation, peak-profile choice, $K\alpha$ splitting, background subtraction, grain statistics, powder packing, and possible overlap or asymmetry in the high-angle reflection.

2.6. Experimental Procedure and Data Analysis Protocol

Commercial iron powder was obtained from Sigma-Aldrich, St. Louis, MO, USA. According to the manufacturer's specification, the powder had a nominal purity of 98 wt.% Fe and a particle-size range of 20–50 μm . The powder was used as received, without milling, sieving, or controlled particle-size classification before the XRD measurement. A thin layer of the powder was distributed onto double-sided adhesive tape fixed to a flat sample holder. Because the supplied powder may contain a distribution of particle sizes and agglomerates, the possible effects of heterogeneous packing, preferred orientation, limited

grain statistics, and specimen surface-height variations were considered when interpreting the diffraction intensities and fitted peak widths.

Powder-XRD measurements were performed using a Shimadzu XRD-7000 diffractometer (Shimadzu Corporation, Kyoto, Japan) operated in coupled $2\theta/\theta$ scanning mode, corresponding to the standard Bragg–Brentano parafocusing geometry, with a goniometer radius of 200 mm. Cu $K\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) was generated at 40 kV and 40 mA. The incident-beam optics comprised a 1° divergence slit and a 10 mm divergence-height-limiting slit, whereas the diffracted-beam path included a 1° scattering slit and a 0.15 mm receiving slit. No Soller-slit specification or $K\beta$ -filter/monochromator setting was listed in the exported acquisition metadata; these parameters were therefore not inferred. The diffraction pattern was acquired in continuous-scan mode from 35° to 100° in 2θ , with a recorded data interval of 0.02° . Because continuous scanning was used, a discrete counting time per step was not defined. The scan speed and, therefore, the equivalent dwell time per recorded interval were not preserved in the supplied acquisition metadata and could not be reconstructed reliably. The measured angular range contains the three principal α -Fe reflections analyzed in this study at $2\theta = 44.850^\circ$, 65.218° , and 82.540° . The exported metadata also did not document the specific zero-alignment or specimen-height verification procedure; this missing information is acknowledged as a limitation of complete experimental reproducibility. These acquisition parameters are reported because goniometer geometry, slit configuration, scan rate, axial-divergence control, and specimen alignment can influence peak positions, line widths, profile asymmetry, and relative intensities.

Local pseudo-Voigt functions were used in IGOR Pro (Version 9, WaveMetrics, Inc., Lake Oswego, OR, USA) to fit the comparatively symmetric (110) and (200) α -Fe reflections. The pseudo-Voigt profile combines Gaussian and Lorentzian contributions and therefore provides a more flexible empirical representation of laboratory powder-diffraction peaks than a purely Gaussian function. The high-angle (211) region exhibits a pronounced asymmetric shoulder and was consequently fitted using a two-component pseudo-Voigt profile representing the partially resolved Cu $K\alpha_1$ and $K\alpha_2$ contributions. These profile functions were selected to obtain stable peak centers, integrated areas, and comparative FWHM values while reproducing the observed line shapes. Because no external instrumental standard was measured under identical conditions, the fitted widths were not interpreted as intrinsic specimen broadening. Quantitative separation of instrumental, coherent domain-size, and strain contributions would require a standard-derived instrumental-resolution function and Voigt-type whole-pattern deconvolution.

The analysis workflow proceeded in a structured sequence. First, the raw diffraction pattern was inspected to identify the dominant reflections. Second, individual peak fitting was used to determine the peak center, integrated area, width, height, and FWHM of each reflection. Third, Bragg's law was applied to calculate the corresponding d-spacings from the fitted 2θ values. Fourth, the squared sine-ratio method was used to assign the cubic indexing sequence and identify the Bravais lattice. Fifth, the lattice parameter was calculated independently from each indexed reflection, then averaged. The fitted FWHM values were converted to radians and used only to calculate formal, instrument-uncorrected Scherrer line-broadening indicators. The three observed peak-width coordinates were also displayed in Williamson–Hall form solely to illustrate the angular evolution of $\beta \cos \theta$ with $4 \sin \theta$. No linear regression was performed because only three α -Fe reflections were available, which do not provide an adequate statistical basis for separating coherent-domain-size and microstrain contributions. Finally, calculated relative intensities were estimated using the bcc structure factor, Fe atomic form factor, reflection multiplicity, and Lorentz polarization correction and were compared with the experimental integrated intensities [4–9,43].

This stepwise protocol allows each extracted structural parameter to be traced directly to measurable features of the diffraction pattern. In a full Rietveld refinement, many of these parameters would be determined simultaneously within a least-squares model; however, the present approach makes the assumptions and limitations associated with each stage explicit. The bcc phase assignment and lattice parameter are the most robust results because they are primarily governed by peak positions. The Scherrer values are retained only as uncorrected apparent line-broadening indicators, and the Williamson–Hall coordinates are presented only as a qualitative illustration without regression-derived size or microstrain. The relative-intensity comparison requires the greatest caution because it is additionally affected by crystallite orientation, powder packing, specimen geometry, background treatment, and instrumental optics [37,44].

3. Results

3.1. Peak Positions and Indexing

The fitted peak positions and indexing results are summarized in Table 1. The three observed reflections are located at approximately 44.850°, 65.218°, and 82.540° (Figure 1). Their squared-sine ratios follow the sequence of 1.000, 1.996, and 2.989, which corresponds to 1, 2, and 3 within the experimental uncertainty. Multiplying by the first allowed bcc value of $N = 2$ gives $N = 2, 4$, and 6, which are assigned to the (110), (200), and (211) reflections. This sequence confirms that the diffraction pattern belongs to body-centered cubic α -Fe [17–21].

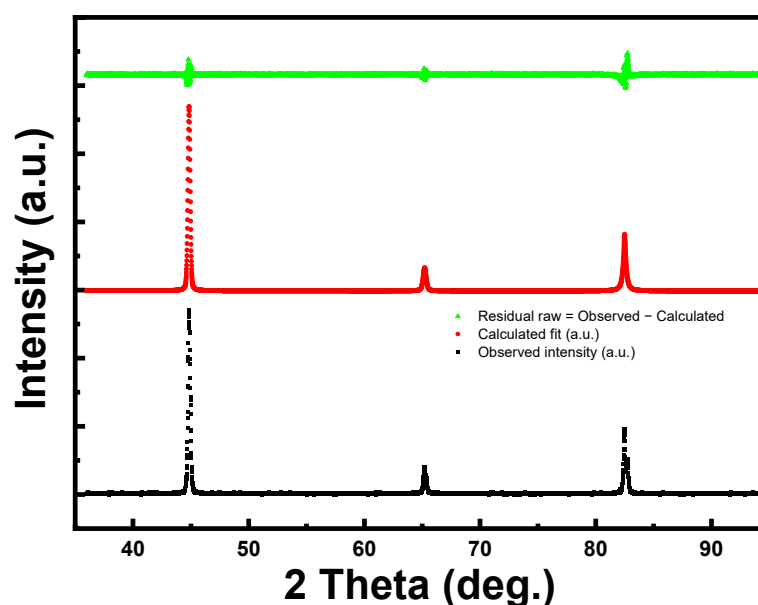


Figure 1. Whole-pattern XRD profile of iron metal powder, showing the three dominant bcc α -Fe reflections.

Table 1. Peak positions, Bragg-law d-spacings, squared-sine ratios, and bcc indexing of α -Fe powder.

Peak	2θ (deg)	θ (deg)	$\sin(\theta)$	d (Å)	$(\sin/\sin_0)^2$	$N = h^2 + k^2 + l^2$	hkl
1	44.850	22.425	0.38147	2.020	1.000	2.000	(110)
2	65.218	32.609	0.53890	1.430	1.996	3.992	(200)

Peak	2θ (deg)	θ (deg)	$\sin(\theta)$	d (Å)	$(\sin/\sin\theta)^2$	$N = h^2 + k^2 + l^2$	hkl
3	82.540	41.270	0.65961	1.170	2.989	5.978	(211)

Figure 2a–c present the local peak-profile analysis of the three principal diffraction maxima observed for the iron powder, corresponding to the bcc α -Fe reflections at (110), (200), and (211). In each selected angular window, the experimental intensity distribution is represented by discrete points, while the continuous fitted profile describes the peak envelope after the background contribution has been accounted for. The first reflection, centered at approximately $2\theta = 44.85^\circ$, is assigned to the (110) plane and appears as the most intense and best-defined maximum in the diffraction pattern. The second reflection, located near $2\theta = 65.21^\circ$, corresponds to the (200) family and shows a substantially lower intensity and broader profile. The third high-angle feature around $2\theta = 82.5$ – 82.7° is assigned to the (211) reflection and exhibits a pronounced asymmetric shape with a visible shoulder, which can be described more realistically by a two-component profile rather than by a single symmetric peak. Therefore, the localized fitting confirms not only the angular positions of the α -Fe reflections but also the progressive evolution of line shape and peak breadth with increasing diffraction angle [40,45].

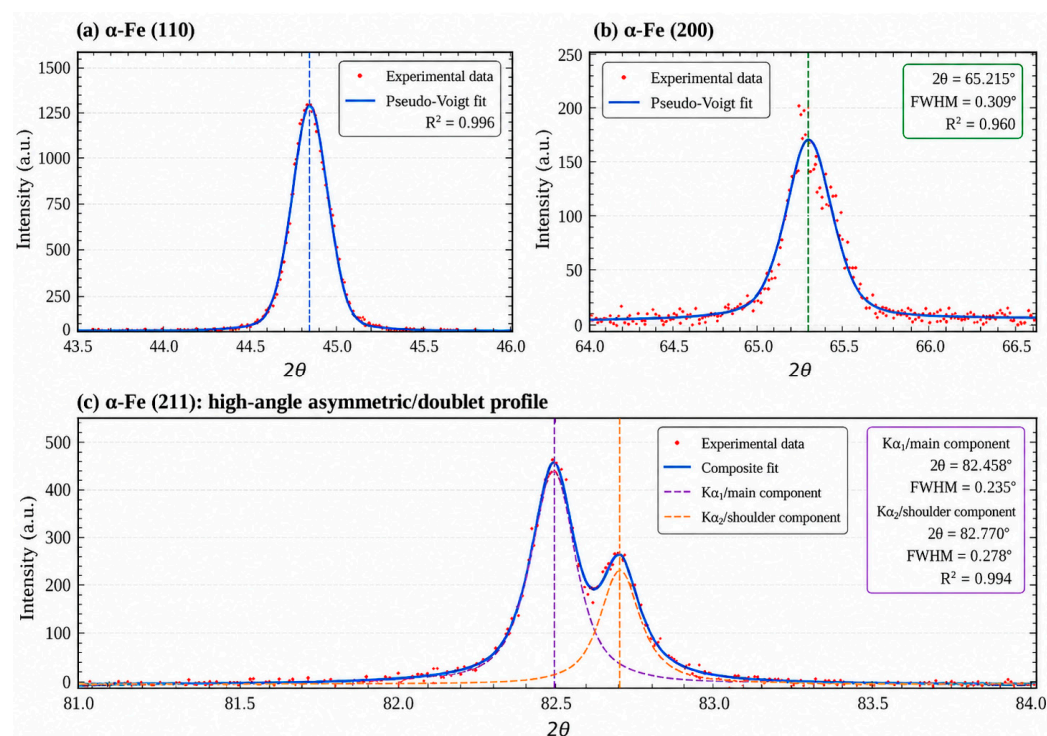


Figure 2. Local peak-profile fitting of the principal bcc α -Fe X-ray diffraction maxima. Panels (a) and (b) show pseudo-Voigt fits to the (110) and (200) reflections, respectively. Panel (c) shows a two-component pseudo-Voigt representation of the asymmetric (211) region, with separate contributions assigned to the partially resolved $\text{Cu } K_{\alpha 1}$ and $K_{\alpha 2}$ components. Red symbols represent the experimental data, and blue lines represent the fitted peak envelopes. The selected functions are used for robust peak-center and comparative profile analysis and are not interpreted as an instrumental deconvolution.

The main trend is that the diffraction response changes systematically from the low-angle (110) reflection to the higher-angle (200) and (211) reflections. The (110) peak dominates the pattern because it is the first allowed bcc reflection and occurs at the lowest momentum transfer among the observed maxima [46]. At this position, the atomic scattering

factor of Fe remains relatively large, and the Lorentz polarization contribution is favorable, giving rise to a strong and narrow reflection. By contrast, the (200) peak is weaker and broader, reflecting the combined effect of reduced scattering efficiency at a higher angle, lower effective diffracting volume for that family, and possible texture or specimen-preparation effects. The (211) reflection appears stronger than the (200) peak, despite its higher scattering vector, which is consistent with the larger multiplicity of the {211} family in a cubic powder and with possible preferred orientation introduced during powder mounting. Consequently, the observed intensity sequence should not be interpreted solely from ideal powder-structure factors; it also contains contributions from multiplicity, instrumental geometry, profile asymmetry, and the actual statistical orientation of the crystallites [13–16,47].

The fitted peak positions provide strong evidence for the presence of single-phase bcc α -Fe. The observed maxima follow the expected bcc reflection sequence, in which only planes satisfying the bcc reflection condition are observed. Conversion of the peak positions into reciprocal-space values gives q values close to 3.11, 4.39, and 5.38 $\text{\AA}^{-1}$ for the (110), (200), and (211) reflections, respectively. The corresponding q^2 ratios are approximately 1:2:3 when normalized to the first peak, matching the expected $N = h^2 + k^2 + l^2$ sequence of 2, 4, and 6 for a cubic bcc lattice. This agreement demonstrates that the peaks are not isolated angular features or impurity-related signals but reciprocal lattice points belonging to the same α -Fe phase. The associated lattice parameter is therefore internally consistent with bcc iron, supporting the phase assignment obtained from the full XRD pattern [17–21].

Peak broadening provides additional microstructural information. The fitted profiles indicate that the angular breadth increases from the intense (110) peak toward the higher-angle reflections, with the (211) region showing the strongest line-shape distortion. Such behavior is physically expected when finite coherent-domain size is accompanied by microstrain or local lattice-spacing fluctuations. Finite crystallite size contributes a baseline broadening to the diffraction maxima, whereas strain-related broadening becomes more pronounced at larger diffraction angles because small variations in d -spacing produce larger changes in 2θ at high scattering vectors. The asymmetric shape of the (211) reflection is also consistent with the increasing separation of the Cu $K\alpha$ doublet at high angles and with high-angle instrumental profile asymmetry. Therefore, the broadening trend should be interpreted as an apparent size-strain contribution rather than as direct evidence of a new phase. A fully quantitative extraction of crystallite size and strain would require instrumental broadening correction using a standard reference material; in the absence of such correction, the fitted widths are best treated as comparative indicators of peak-profile evolution [36,48].

From a materials perspective, these fitted profiles complement the phase-identification analysis by separating robust structural information from specimen-dependent intensity effects. The peak positions are the most reliable indicators of crystal structure and confirm the bcc α -Fe phase, whereas the relative intensities and line widths are more sensitive to microstructure, orientation, counting statistics, and background treatment. If microscopy data such as SEM or AFM are available, they should be discussed alongside the XRD fitting to distinguish coherent-domain size from the actual particle size or surface morphology, since XRD peak broadening reflects coherently diffracting domains rather than whole-particle dimensions. Overall, the peak-profile fitting strengthens the XRD interpretation by showing that the observed angular positions, peak shapes, and reciprocal-space consistency all support a structurally coherent bcc α -Fe phase, while the deviations in intensity and high-angle symmetry can be attributed to realistic experimental and microstructural factors rather than to compositional changes.

3.2. Lattice Parameter Determination

The lattice parameter was determined independently from each indexed reflection using

$$a = d_{hkl} (h^2 + k^2 + l^2)^{1/2}. \quad (3)$$

The average lattice parameter obtained from the three indexed reflections is 2.859 Å, which is approximately 0.007 Å (0.24%) below the accepted room-temperature value of about 2.866 Å for α -Fe. The reflection-specific values increase from 2.853 Å for (110) to 2.860 Å for (200) and 2.865 Å for (211). Because the lattice parameter of an ideal single cubic phase should be independent of the reflection index, this angular variation is not interpreted as a physical contraction of the α -Fe lattice. Instead, it most likely reflects small systematic uncertainties in the fitted peak positions, including diffractometer zero shift, specimen-height or surface-flatness effects, high-angle profile asymmetry, partial Cu $K_{\alpha 1}/K_{\alpha 2}$ separation, and finite fitting precision. The narrow spread of the values and the proximity of the high-angle result to the reference value remain consistent with the body-centered cubic α -Fe structure within the resolution of the laboratory measurement, as summarized in Table 2 and Figure 3 [22–25].

Table 2. Reflection-by-reflection lattice-parameter determination for body-centered cubic α -Fe.

Peak	<i>hkl</i>	<i>d</i> (Å)	<i>N</i>	<i>a</i> (Å)
1	(110)	2.020	2	2.853
2	(200)	1.430	4	2.860
3	(211)	1.170	6	2.865
Average	-	-	-	2.859

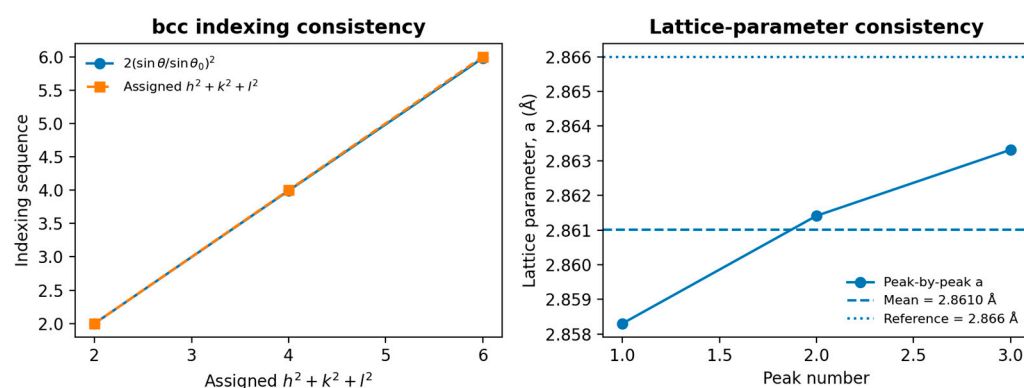


Figure 3. Cubic indexing and reflection-by-reflection lattice-parameter consistency for iron powder.

3.3. Peak Width and Apparent Scherrer Size

Direct substitution of the observed FWHM values into the Scherrer relation gives formal apparent values of 44.8, 40.9, and 29.6 nm for the (110), (200), and (211) reflections, respectively. These values were calculated from β_{obs} , without subtraction of instrumental broadening, and must not be interpreted as quantitatively determined coherent-domain sizes. They are retained only as uncorrected apparent coherence-length proxies that illustrate the sensitivity of the Scherrer calculation to diffraction-line breadth. Because instrumental broadening increases the observed FWHM, the listed values may underestimate the intrinsic coherence length; however, their exact correction cannot be established without a reference-standard measurement and an appropriate peak-profile deconvolution.

The manufacturer-specified particle-size range of 20–50 μm describes the external dimensions of the supplied Fe particles or agglomerates and should not be equated directly with the coherent-domain dimensions obtained from XRD line broadening. A single micrometer-sized powder particle may contain several coherently diffracting domains separated by defects, subgrain boundaries, residual microstrain, or small crystallographic misorientations. Moreover, heterogeneity in particle size, aggregation, defect density, and strain state may produce a distribution of peak broadening rather than a single uniform domain size. Accordingly, values of 44.8, 40.9, and 29.6 nm are reported as sample-averaged apparent coherent-domain lengths and not as the physical diameters of the commercial Fe powder particles (Table 3) [48,49].

Table 3. Peak-width parameters and apparent Scherrer coherent-domain size of α -Fe powder.

Peak	<i>hkl</i>	<i>FWHM (deg)</i>	β (rad)	$\cos(\theta)$	<i>D (nm)</i>
1	(110)	0.20318	0.00350	0.924	44.762
2	(200)	0.23937	0.00420	0.842	40.934
3	(211)	0.37770	0.00650	0.752	29.615
Average	-	-	-	-	38.437

3.4. Qualitative Williamson–Hall Representation of Peak Broadening

The three observed peak-width coordinates are displayed in Williamson–Hall form to illustrate the angular evolution of $\beta\cos\theta$ with $4\sin\theta$ (see Figure 4). No linear regression was performed because only three α -Fe reflections were available, which is insufficient for a statistically robust separation of the contributions of coherent-domain size and microstrain. In addition, the observed widths were not corrected for instrumental broadening. The plotted points are therefore retained only as a qualitative methodological illustration and are not used to calculate a slope, intercept, coefficient of determination, coherent-domain size, or microstrain (see Table 4). A quantitative Williamson–Hall analysis would require an extended angular range containing at least 5–7 well-resolved reflections and an instrumental-resolution function obtained from a strain-free reference standard measured under identical conditions [28–36].

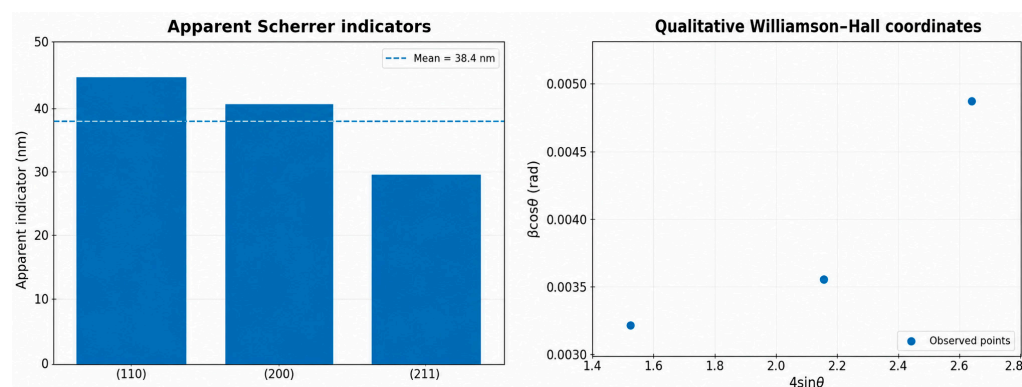


Figure 4. Apparent Scherrer line-broadening indicators and qualitative Williamson–Hall representation constructed from the observed, instrument-uncorrected FWHM values of the (110), (200), and (211) α -Fe reflections. The Williamson–Hall coordinates are shown without linear regression and are not used to determine quantitative coherent-domain size or micro-strain.

Table 4. Observed Williamson–Hall plotting coordinates for the indexed α -Fe reflections. The peak widths were not corrected for instrumental broadening, and no linear regression was performed. The values are presented only to illustrate the qualitative angular evolution of the observed line broadening.

Peak	<i>hkl</i>	$4\sin(\theta)$	$\beta\cos(\theta)$
1	(110)	1.5259	0.00324
2	(200)	2.1556	0.00354
3	(211)	2.6384	0.00489

3.5. Calculated and Experimental Relative Intensities

The calculated relative intensities were estimated using reflection multiplicity, the bcc structure factor, Fe atomic form factors, and Lorentz polarization correction (Figure 5). The experimental intensities were obtained from fitted integrated peak areas and normalized to the strongest reflection. The strongest reflection in both calculated and measured profiles is (110), while the relative strength of the high-angle (211) reflection is experimentally higher than in the simplified calculated model (Table 5) [46,50].

Table 5. Comparison of calculated and experimental relative intensities for bcc α -Fe reflections.

Peak	<i>hkl</i>	Area	<i>f</i>	LP	Multiplicity	I_{cal}/I_{max} (%)	I_{exp}/I_{max} (%)
1	(110)	372.000	18.44	5.586	12	100.00	100.00
2	(200)	65.139	15.21	2.403	6	14.63	17.51
3	(211)	174.000	13.11	1.787	24	32.34	46.77

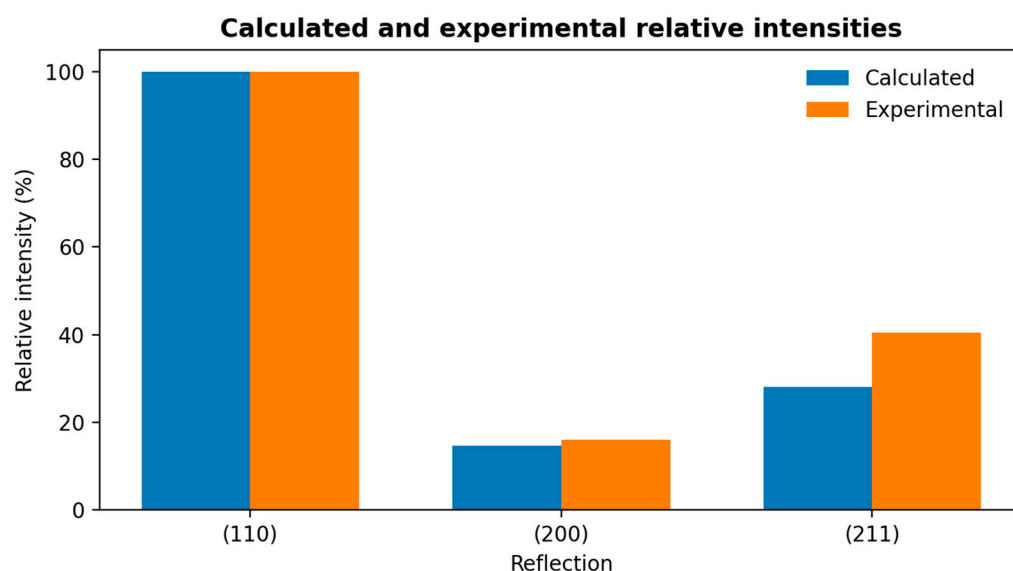


Figure 5. Calculated and experimental relative intensities for the indexed α -Fe reflections.

4. Discussion

4.1. Evidence for the Body-Centered Cubic α -Fe Structure

The most important conclusion from the diffraction pattern is that the iron powder crystallizes in a body-centered cubic structure. This conclusion follows from the simultaneous agreement of peak positions, squared-sine ratios, and systematic absence rules. The first three measured reflections occur at angles expected for α -Fe when *Cu K α* radiation is

used. Their squared-sine ratios are close to 1, 2, and 3. Because the first allowed bcc reflection has $N = 2$, these ratios convert into $N = 2, 4$, and 6. The corresponding Miller indices are (110), (200), and (211). This indexing sequence is the standard low-order sequence for a bcc metal [17–21].

The interpretation would be different for other cubic lattices. A simple cubic lattice allows all integer values of N that can be written as $h^2 + k^2 + l^2$, including the (100) reflection. A face-centered cubic lattice allows only reflections in which h , k , and l are either all odd or all even, producing the low-order sequence of $N = 3, 4, 8, 11$, and so on. The iron pattern does not follow these alternatives. Instead, it follows the bcc condition of $h + k + l = \text{even}$. The absence of lower-angle forbidden peaks such as (100) and (111), together with the presence of (110), (200), and (211), provides direct crystallographic evidence for the body-centered lattice [17–21].

The structure-factor expression gives the physical basis for these absences. The two atoms in the bcc conventional cell scatter in phase when $h + k + l$ is even and out of phase when $h + k + l$ is odd. For odd sums, destructive interference cancels the reflection. This simple interference condition is why the diffraction pattern is such a strong fingerprint of the lattice. Therefore, the observed reflections do not merely correspond to a set of arbitrary peaks; they represent the allowed reciprocal-lattice points of α -iron [12–16].

4.2. Accuracy of the Lattice Parameter

The average lattice parameter obtained from the fitted peaks is 2.859 Å, which is close to the accepted room-temperature value of α -Fe of approximately 2.866 Å. The absolute deviation is about 0.007 Å, corresponding to a relative difference of roughly 0.24%. For a simple laboratory powder-diffraction experiment, this agreement is good. It indicates that the peak centers, wavelength selection, and indexing assignments are mutually consistent. It also demonstrates why peak positions are usually the most reliable part of an XRD pattern [17–24].

The slight underestimation of the lattice parameter can arise from several sources. A small zero shift in 2θ , sample displacement from the diffractometer focusing circle, transparency effects in the powder layer, and imperfect calibration can all shift the fitted peak centers. For example, if the sample surface is slightly above or below the ideal diffractometer height, all peak positions may shift systematically, producing a small error in the extracted lattice constant. Because the present sample was prepared on double-sided adhesive tape, small surface-height and flatness errors are plausible [40–49].

The peak-by-peak lattice constants increase slightly from 2.853 Å for (110) to 2.865 Å for (211). This trend suggests that the low-angle peak may carry a slightly different systematic error than the higher-angle peaks. Low-angle peaks can be more sensitive to sample displacement, while high-angle peaks are more sensitive to profile asymmetry and $K\alpha$ doublet effects. A Nelson–Riley extrapolation or full Rietveld refinement could reduce these systematic errors by modeling angular dependence explicitly. Nevertheless, the small spread among the three values is sufficient to validate the α -Fe lattice parameter in the context of a laboratory structural analysis [22–25,41–45].

The accuracy of the lattice parameter is better than the accuracy of the size and intensity analyses because peak positions can be fitted with high precision, even when intensities and widths are affected by specimen conditions. This hierarchy is important in material characterization. A powder pattern can identify a phase and lattice parameter reliably, even when microstructural parameters remain approximate. For iron powder, the measured value of 2.859 Å is therefore strong evidence for α -Fe, even though peak-width and intensity interpretations require greater caution.

4.3. Meaning and Limitations of the Scherrer Size

The Scherrer sizes obtained from the iron reflections are apparent coherent-domain sizes. They represent the length scale over which the lattice remains sufficiently ordered to scatter coherently, not necessarily the physical diameter of powder particles. Iron powder grains visible by microscopy may be much larger than tens of nanometers, but each particle can contain multiple coherent domains, defects, strain fields, or subgrain boundaries. Therefore, the Scherrer value should not be interpreted as the actual particle size unless it is supported by independent microscopy [35,36].

The average apparent Scherrer size of approximately 38.4 nm is reasonable as a first-order coherence-length estimate. The (110) and (200) reflections give similar values of about 44.8 and 40.9 nm, while the (211) reflection gives a smaller value of about 29.6 nm. The stronger broadening of the high-angle reflection may be influenced by microstrain because strain broadening generally increases with $\tan \theta$. It may also reflect K_α doublet splitting or peak asymmetry at high diffraction angles. Because the analysis uses fitted FWHM values directly, without subtracting instrumental broadening, the extracted values are lower-bound estimates of the coherent-domain size. Instrumental broadening adds to the observed peak width, and if it is not removed, the calculated size appears smaller [46,48,49].

The shape factor (K) also introduces uncertainty. A value near 0.9 is commonly used, but the exact value depends on the crystallite shape, size distribution, and peak profile. Iron powder particles may not have a single simple morphology, so a fixed K value is only an approximation. Moreover, Scherrer analysis assumes that broadening arises entirely from the finite domain size. In real powders, microstrain, stacking defects, surface oxidation, residual stress, and instrumental resolution all contribute to the observed peak width. Thus, Scherrer analysis is useful for comparative and approximate characterization but should not be presented as a complete microstructural refinement [27,32–35].

4.4. Why Intensity Agreement Is Poorer than Lattice-Constant Agreement

The comparison between calculated and experimental relative intensities shows that the intensity agreement is poorer than the lattice-parameter agreement. This behavior is expected in powder diffraction and does not weaken the phase identification. Peak positions are governed mainly by lattice geometry and X-ray wavelength, whereas intensities depend on several additional crystallographic, microstructural, instrumental, and specimen-related factors. In the simplified model used here, the relative intensities were calculated from reflection multiplicity, the structure factor, the Fe atomic form factor, and Lorentz polarization correction. This treatment captures the principal crystallographic trend but does not include all experimental and microstructural contributions [46,50].

The strongest calculated and experimental reflection is (110), consistent with the body-centered cubic α -Fe structure. The (200) reflection is weaker in both datasets, whereas the (211) reflection has an intermediate calculated intensity but appears experimentally stronger than predicted. Several factors may contribute to this difference. Preferred orientation can enhance particular plane families when the crystallites are not randomly oriented on the adhesive support. Nonuniform sample thickness can modify absorption and the effective scattering volume, while background subtraction and peak-profile selection can affect integrated areas, especially for broad or asymmetric reflections. The high-angle (211) peak is also influenced by partial Cu $K_{\alpha 1}/K_{\alpha 2}$ separation and profile asymmetry, which can increase uncertainty in the fitted integrated area [46,50].

Finite coherent-domain size may also contribute indirectly to uncertainty in the measured relative intensities. Termination of the periodic lattice in a finite crystallite broadens an ideal Bragg reflection through the crystallite shape transform and redistrib-

utes intensity into a wider angular profile or diffuse tails. Under the kinematic approximation, however, the integrated intensity of a complete reflection is approximately conserved. Therefore, finite-size broadening alone is not expected to produce a large systematic change in normalized relative-intensity ratios when the full peak area is accurately captured. A truncation-related bias may arise if broadened tails are excluded from the fitting window or if finite domain size is accompanied by anisotropic crystallite shape, surface disorder, residual strain, defects, or asymmetric line profiles. The apparent coherent-domain sizes of 29.6–44.8 nm may therefore contribute secondarily to profile-dependent uncertainty, particularly for the high-angle (211) reflection. Nevertheless, the present data do not support assigning the observed intensity mismatch primarily to nanostructure-induced lattice truncation. Preferred orientation, powder packing and grain statistics, background and profile treatment, partial Cu $K_{\alpha 1}/K_{\alpha 2}$ separation, and the simplified calculated-intensity model remain the more plausible dominant sources of the discrepancy.

In addition, the calculated-intensity model is idealized. It does not explicitly include temperature factors, absorption corrections, specimen transparency, surface roughness, axial divergence, instrumental resolution, or a preferred-orientation function. It also uses representative Fe atomic form factors rather than refining them within a full whole-pattern model. Consequently, qualitative agreement is expected, whereas exact quantitative agreement is not. Lattice parameters can be extracted reliably from peak positions using a simple analysis, but quantitative intensity refinement requires more complete modeling and more carefully controlled specimen preparation [47,50].

The intensity discrepancy is therefore informative rather than contradictory. It demonstrates the limitations of a simplified relative-intensity calculation and motivates a more rigorous whole-pattern refinement. In a full Rietveld analysis, the scale factor, background, profile parameters, lattice parameter, preferred orientation, zero shift, and thermal parameters would be refined simultaneously. Such an approach could reduce the mismatch between calculated and experimental intensities and provide a more physically complete description of the iron powder-diffraction pattern [47,50].

4.5. Reciprocal-Space (q -Space) Interpretation of the Diffraction Pattern

The diffraction profile was additionally evaluated in reciprocal space by converting the angular coordinate into the magnitude of the scattering vector, i.e., $q = 4\pi \sin \theta / \lambda = 2\pi/d$, where θ is the Bragg angle and λ is the Cu K_{α} wavelength used in the analysis. Figure 6 shows the same iron powder diffraction pattern after transformation from 2θ into q -space. The measured range of 36 – 95° in 2θ corresponds to a reciprocal-space interval of approximately 2.52 – $6.01 \text{ \AA}^{-1}$, within which three sharp maxima are observed at $q = 3.109$, 4.392 , and $5.375 \text{ \AA}^{-1}$. These maxima are assigned to the (110), (200), and (211) reflections of bcc α -Fe, respectively. Presenting the data in q -space is useful because the peak positions are no longer treated only as instrumental angles; they represent the magnitudes of reciprocal-lattice vectors sampled by the diffraction experiment [4–7,12].

The principal trend in the q -space profile is the systematic progression of the allowed reflections toward higher reciprocal-lattice magnitude as $N = h^2 + k^2 + l^2$ increases. For a cubic lattice, $q_{hkl} = (2\pi/a) (h^2 + k^2 + l^2)^{1/2}$; therefore, the measured q^2 ratios provide a direct reciprocal-space test of the indexing. The present q values give $q^2/(q^2(110))$ ratios of approximately 1.000, 1.996, and 2.989, which reproduce the same 1:2:3 sequence obtained from the squared-sine analysis. When referenced to the first allowed bcc value of $N = 2$, this sequence gives $N = 2, 4$, and 6 , corresponding exactly to the (110), (200), and (211) families. Thus, the q -space representation confirms that the three observed maxima are not arbitrary angular features but discrete reciprocal-lattice points belonging to a single bcc α -Fe phase [17–21].

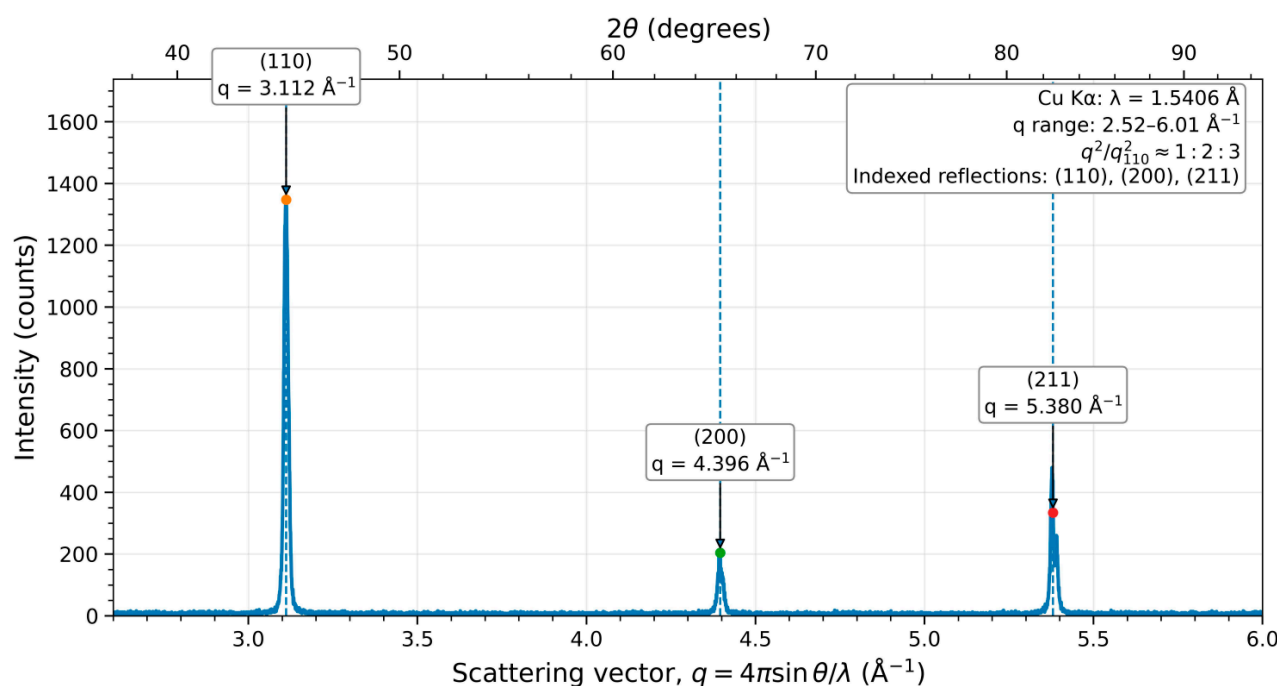


Figure 6. Reciprocal-space XRD profile of iron powder showing the indexed bcc α -Fe reflections at $q \approx 3.111, 4.392, \text{ and } 5.377 \text{ \AA}^{-1}$.

The variation in intensity across q -space provides a complementary view of the scattering mechanism. The (110) reflection remains the dominant feature because it occurs at the lowest q among the observed bcc reflections, where the Fe atomic form factor and Lorentz polarization contribution are comparatively large. The (200) peak is markedly weaker, consistent with its lower multiplicity and the reduction in scattering efficiency at higher momentum transfer. The (211) peak, however, appears experimentally stronger than a simple ideal-powder calculation would predict. This behavior is consistent with the intensity analysis presented above and most likely reflects the combined influence of reflection multiplicity, preferred orientation introduced during powder mounting, high-angle profile asymmetry, $K\alpha$ doublet contribution, and uncertainty in background subtraction. Therefore, the q -space intensity distribution supports the bcc structural assignment while also emphasizing that relative intensities are more sensitive to specimen geometry and data treatment than peak positions [46,50].

Peak broadening also becomes physically more transparent in reciprocal space because the width of a diffraction maximum corresponds to a distribution of reciprocal-lattice vectors rather than only to an angular FWHM. Using the fitted widths reported for the three α -Fe reflections, the approximate reciprocal-space breadths ($\Delta q \approx (2\pi/\lambda) \beta \cos \theta$) increase from about $0.013 \text{ \AA}^{-1}$ for (110) to $0.014 \text{ \AA}^{-1}$ for (200) and $0.020 \text{ \AA}^{-1}$ for (211). This increase mirrors the Williamson–Hall trend and indicates that the broadening cannot be interpreted solely as an angle-independent finite-size effect. A finite coherent-domain size contributes a baseline reciprocal-space broadening, whereas microstrain produces a broadening component that grows with q because local variations in d -spacing become more visible at larger scattering vectors. The stronger broadening of the high- q (211) reflection is therefore consistent with an apparent size-strain contribution, although the absence of instrumental broadening correction and the availability of only three peaks require that the extracted size and strain values remain qualitative rather than definitive [40–46].

Because the present manuscript concerns a single iron powder composition rather than a compositional series, the most meaningful comparison is between reciprocal-space

regions and reflection families rather than between different samples. The low- q region provides the most reliable phase-identification and intensity-normalization reference through the intense (110) reflection, whereas the intermediate- and high- q regions test the internal consistency of the cubic lattice parameter and reveal the increasing sensitivity of widths and intensities to microstructural and instrumental factors. This interpretation is directly connected to the previous XRD results: the q -position analysis reinforces the lattice parameter of approximately 2.859 Å, the q -broadening analysis supports the apparent coherent-domain and Williamson–Hall discussion, and the q -dependent intensity behavior explains why the structural assignment is robust, even though the measured intensities do not perfectly follow the simplified calculated model. Complementary microscopic data such as SEM or AFM, if available, would be required to compare the coherent-domain length with particle morphology. In this context, reciprocal-space analysis strengthens the manuscript by connecting peak position, peak width, and intensity into a single physically interpretable diffraction framework.

4.6. Peak-Profile Functions and Fitted Parameters

The final local peak analysis employed pseudo-Voigt rather than purely Gaussian profiles. This choice is physically more appropriate for metallic powder diffraction because the observed line shape may contain both Gaussian and Lorentzian contributions arising from instrumental resolution, finite coherent-domain extent, strain distributions, axial divergence, and other profile effects. The pseudo-Voigt function provides a stable analytical approximation of a Voigt-type profile and is therefore suitable for extracting peak centers, FWHM values, and integrated areas from the present laboratory dataset. Pearson VII is also a valid empirical alternative; however, no single profile function can uniquely separate physical broadening mechanisms without an independently measured instrumental-resolution function. The (110) and (200) peaks were adequately represented by single pseudo-Voigt functions, whereas the visibly asymmetric high-angle (211) reflection required a two-component pseudo-Voigt treatment to represent the partially resolved Cu $K_{\alpha 1}/K_{\alpha 2}$ doublet. Accordingly, the fitted peak centers are used for indexing and lattice-parameter determination, while the fitted widths are reported only as instrument-uncorrected apparent line-broadening indicators and not as quantitative coherent-domain-size or microstrain values.

The (110) reflection at $2\theta = 44.850^\circ$ is the sharpest and most intense peak and therefore provides the most stable peak-center estimate and the most favorable counting statistics. The (200) reflection at $2\theta = 65.218^\circ$ is weaker and broader, so its fitted FWHM and integrated area are more sensitive to the selected background interval and fitting window. The high-angle (211) region is the most profile-sensitive part of the pattern because the Cu $K_{\alpha 1}/K_{\alpha 2}$ separation becomes more pronounced with increasing diffraction angle and produces a visible shoulder. The two-component pseudo-Voigt representation describes this asymmetric envelope more realistically than a single symmetric function. The representative reflection position reported for indexing is retained as the effective peak position of the (211) feature; the component decomposition is used only to describe the line shape and does not indicate the presence of an additional crystalline phase.

Peak-profile selection affects the reliability of all downstream quantities but not to the same degree. Small changes in the fitted center can influence the calculated d -spacing and lattice parameter, whereas changes in FWHM directly affect the formal Scherrer indicators and the qualitative Williamson–Hall coordinates. Variations in the baseline and profile tails can also alter the integrated areas used for relative-intensity comparison. Therefore, the pseudo-Voigt treatment improves the flexibility and physical plausibility of the local fits compared with a pure Gaussian model, but it does not remove the need for instrumental-profile calibration. Quantitative line-profile analysis would require a

strain-free reference standard measured under identical conditions, explicit instrumental deconvolution, and preferably a unified whole-pattern Voigt or pseudo-Voigt refinement. In the present work, the fitted centers support the robust bcc α -Fe indexing and lattice-parameter analysis, whereas the fitted widths and areas are interpreted with the stated model-dependent limitations [40–46].

4.7. Reflection-by-Reflection Interpretation

The (110) reflection is the first and strongest peak in the α -Fe pattern. It appears at $2\theta = 44.850^\circ$ and gives $d = 2.020 \text{ \AA}$. Because $N = 2$ for this reflection, the lattice constant extracted from it is $2.853 \text{ \AA}$. The strength of the (110) peak is consistent with its relatively large Lorentz polarization factor, high Fe form factor at lower scattering vectors, and multiplicity of 12. Its large integrated area also makes it the most stable reflection for intensity normalization.

The (200) reflection appears at $2\theta = 65.218^\circ$ and gives $d = 1.430 \text{ \AA}$. The extracted lattice parameter is $2.860 \text{ \AA}$, which is close to the average value. Its experimental intensity is much weaker than that of (110), consistent with the decrease in atomic form factor and Lorentz polarization factor at higher angles and with its lower multiplicity of 6. The fitted FWHM gives an apparent Scherrer size of 40.9 nm , which is close to the average coherent-domain size. This reflection is therefore useful for confirming both the bcc indexing and the consistency of the lattice parameter.

The (211) reflection appears at $2\theta = 82.540^\circ$ and gives $d = 1.170 \text{ \AA}$. It yields the largest individual lattice parameter of $2.865 \text{ \AA}$, which is very close to the accepted value. However, it also shows the largest FWHM and therefore the smallest Scherrer size. This could indicate stronger high-angle broadening due to microstrain or profile effects. The experimental intensity of this reflection is higher than the simplified calculated value, which may reflect preferred orientation, peak asymmetry, or fitting of a partially split high-angle peak. Thus, the (211) reflection is structurally useful but microstructurally more sensitive than the first two peaks.

Considering the three peaks together provides a balanced interpretation. The peak positions form a highly consistent bcc sequence, while the peak widths and intensities reveal the limitations of simplified analysis. This is typical of laboratory powder diffraction: phase identification is strong, and lattice-parameter extraction is reliable, but detailed microstructure and intensity modeling requires additional corrections.

4.8. Error Budget and Uncertainty Hierarchy

A careful diffraction manuscript should distinguish among different sources of error. The first category is angular-position error, which affects d -spacing and the lattice parameter. This error may arise from zero shift, sample displacement, peak asymmetry, or imperfect wavelength calibration. The second category is width error, which affects Scherrer size and Williamson–Hall analysis. Width error may arise from instrumental broadening, peak-profile choice, K_α doublet effects, microstrain, and counting statistics. The third category is intensity error, which affects structure-factor comparison. Intensity error is usually the largest because it depends strongly on sample preparation, preferred orientation, absorption, surface roughness, background subtraction, and the fitted peak area [40–49].

In the iron analysis, the lattice parameter is the most reliable quantitative result. The three values differ by only about $0.012 \text{ \AA}$, and the average agrees well with the accepted value. The Scherrer size is less reliable because no instrumental correction was applied. Therefore, the average of 38.4 nm should be considered an apparent coherence length. The Williamson–Hall analysis is even more limited because it uses only three reflections. A linear fit through three points can illustrate the size-strain concept, but it cannot provide a robust statistical separation of size and strain broadening. A larger number of reflections

and instrumental-standard correction would be required for quantitative confidence [48,49].

The intensity comparison carries the largest uncertainty. The discrepancy between calculated and experimental relative intensities is expected because the powder was deposited on tape rather than prepared as an ideal randomly oriented powder layer. The (211) intensity enhancement may result from texture or high-angle profile effects. Therefore, the intensity analysis should be used qualitatively to support the bcc model, not as a strict refinement [13–16,50].

4.9. Toward a Full Rietveld Refinement of the Iron Dataset

The present analysis uses a classical peak-by-peak workflow, but the same dataset could be further developed using Rietveld refinement. In a Rietveld approach, the entire observed diffraction profile is fitted by a calculated pattern derived from a structural model. For α -Fe, the structural model would be simple: a bcc cell with space group 1m-3m and Fe occupying the body-centered positions. The refinable parameters would include the scale factor, background coefficients, zero shift, lattice parameter, peak-shape parameters, preferred orientation, and possibly isotropic displacement parameters. The refinement would provide a more rigorous test of the phase assignment and a more reliable estimate of profile residuals [47].

A full refinement would also treat the $K\alpha$ doublet, peak asymmetry, and instrumental resolution more accurately. The present analysis uses average wavelength and simplified peak fitting, which are adequate for identifying α -Fe but less ideal for high-precision microstructure. If an instrumental standard such as LaB6 or silicon were measured under the same conditions, instrumental broadening could be subtracted from the iron peak widths. This would improve the Scherrer and Williamson–Hall estimates and allow for more meaningful separation of crystallite size and microstrain [49].

Another advantage of Rietveld refinement is that it can explicitly model preferred orientation. Powder prepared on adhesive tape may not be randomly oriented. If specific crystallographic planes are preferentially aligned parallel to the sample holder, some reflections can be enhanced or suppressed. This effect directly affects relative intensities but has only a minor effect on peak positions. Including a preferred-orientation correction would likely improve the intensity agreement and clarify whether the strong measured (211) reflection reflects texture, profile fitting, or another experimental factor. Therefore, the present work can be viewed as a foundation for a more advanced refinement workflow rather than as a replacement for it [50].

4.10. Novelty and Contribution of This Work

Overall, this work uses a laboratory XRD pattern of iron powder as a model dataset to demonstrate a transparent crystallographic workflow. The analysis integrates Bragg-law conversion of peak positions, cubic indexing, lattice-parameter calculation, apparent Scherrer coherent-domain estimation, Williamson–Hall size-strain assessment, and simplified relative-intensity interpretation. The localized peak fits confirm the three principal α -Fe reflections at approximately 44.85°, 65.21°, and 82.54° in 2θ , assigned to the (110), (200), and (211) planes, respectively. While the peak positions provide robust confirmation of the bcc α -Fe phase, the peak widths and integrated intensities require more cautious interpretation because they are sensitive to instrumental broadening, $K\alpha$ doublet effects, preferred orientation, sample packing, background subtraction, and profile asymmetry. Therefore, the Scherrer and Williamson–Hall results are best regarded as apparent microstructural indicators, and the relative-intensity comparison should be used as qualitative support for the structural assignment rather than as a full quantitative refinement.

5. Limitations and Future Perspectives

The main limitation of the present analysis is the small number of observable reflections. Only three strong α -Fe peaks are available in the measured angular range. This is sufficient for bcc indexing and lattice-parameter determination, but it limits statistical confidence in Williamson–Hall analysis and intensity refinement. A broader angular range, higher counting statistics, and better peak resolution would improve the reliability of microstructural parameters [39].

A second limitation is the absence of instrumental broadening correction. The observed peak width is a convolution of sample broadening and instrument broadening. Without a standard sample measured under identical conditions, the instrumental contribution cannot be separated from crystallite-size and strain contributions. Therefore, the Scherrer and Williamson–Hall sizes should be described as apparent coherent-domain estimates. Future work should include an instrumental standard such as silicon or LaB6 and perform deconvolution before reporting quantitative crystallite size and microstrain [46–49].

A third limitation is sample preparation. Mounting powder on double-sided tape is practical but can introduce preferred orientation, uneven sample height, and background scattering from the substrate. Future experiments should use a low-background sample holder, minimize texture by gentle powder randomization, and control sample thickness. Repeated measurements with sample spinning would also improve grain statistics and reduce preferred-orientation effects [4–9,25,50].

Future analysis could also include full Rietveld refinement, Pawley or Le Bail fitting, and whole-pattern residual evaluation. These approaches would provide a more rigorous assessment of the lattice parameter, peak profile, background, zero shift, and preferred orientation. If the sample contains surface oxide or minor impurity phases, whole-pattern refinement would also help detect weak secondary peaks that may not be obvious in a simple three-peak analysis. Combining XRD with scanning electron microscopy, energy-dispersive spectroscopy, or magnetic characterization would further strengthen the relationship between structure, microstructure, composition, and properties [47,50].

Despite these limitations, the current results are sufficient to validate the α -Fe structure and demonstrate the principles of powder XRD analysis. The measured peak positions are consistent with bcc iron, the average lattice constant is close to the accepted value, and the peak-width analysis provides a reasonable apparent coherence length. Therefore, this work offers a strong foundation for a more advanced structural refinement of iron powder and a useful model for analyzing other metallic powders [26–36].

6. Conclusions

The XRD pattern of iron metal powder was analyzed using a comprehensive structural workflow based on Bragg’s law, cubic indexing, structure-factor selection rules, peak-width analysis, and relative-intensity comparison. The three observed reflections at 44.850° , 65.218° , and 82.540° were assigned to the (110), (200), and (211) planes of body-centered cubic α -Fe. The squared-sine ratios and bcc selection rule provide strong evidence for the phase assignment. The average lattice parameter of $2.859 \text{ \AA}$ agrees closely with the accepted value for α -iron, confirming the accuracy of the structural interpretation [45–47].

The indexed peak positions and the derived average lattice parameter provide robust confirmation of body-centered cubic α -Fe. By contrast, the peak-width calculations are based on observed FWHM values that were not corrected using an external instrumental standard. The Scherrer outputs are therefore reported only as uncorrected apparent co-

herence-length indicators. The Williamson–Hall coordinates are retained only as a qualitative illustration of the observed angular broadening trend. Because only three reflections were available and instrumental broadening was not independently determined, no regression-derived coherent-domain size or microstrain is reported. Quantitative determination of coherent-domain size and microstrain would require a strain-free standard, such as silicon SRM 640f or LaB₆, measured under identical instrumental conditions, together with Voigt or pseudo-Voigt whole-profile modeling and explicit instrumental deconvolution.

The calculated relative intensities reproduced the principal qualitative trend but differed from the experimental values, particularly for the high-angle (211) reflection. This mismatch is attributed mainly to preferred orientation, powder packing, grain statistics, background treatment, Cu $K_{\alpha 1}/K_{\alpha 2}$ -related profile effects, and the simplified calculated-intensity model. Overall, the manuscript demonstrates how a routine iron-powder XRD dataset can be converted into a transparent structural-validation study. The analysis confirms the bcc α -Fe phase and quantifies the lattice parameter while explicitly distinguishing these robust peak-position results from the more model-dependent peak-width and intensity interpretations [50].

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