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Development the Fourier analysis method of x-ray diffraction to calculate variables in crystal structure as well as calculation of some lattice parameters

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Abstract: The Fourier method was developed to calculate other important variables in the crystalline structure, such a strain, which is equal to 7.4828×10^{-3} instead of the mean square strain and the energy density of the strain, which is equal to 2799614.7 dyne / cm² and the stress equal to 7.4828×10^8 dyne/cm². The results obtained from the Fourier method for calculating other parameters of the manganese oxide lattice for each peak of x-ray diffraction peaks such as the texture coefficient, its mean value equal to 0.99999 and the micro strains, which its mean value equal to 4.47×10^{-3} dislocation density, its mean value are equal to 37.3 (lines .m⁻²) and the specific surface area its mean value is equal to 19,58432 of crystalline volume. A comparison was also made between the values of the square of strain and the apparent strain.

Keywords: Development Fourier analysis, X-ray Diffraction Lines, Lattice parameters .

1. Introduction

X-ray diffraction peak profile analysis is a powerful method for determining the microstructural properties of ultrafine-grained materials. The effects of crystallite size and lattice strain on peak broadening can be separated on the basis of their different diffraction order dependence. The standard methods of x-ray diffraction profile analysis based on the full width at half-maximum (FWHM), the integral breadths and on the Fourier coefficients of the profiles provide the apparent crystallite size and the mean-square of lattice strains [1]. X-ray micro-diffraction semblance using Fourier method at single crystal Alumina, Cu samples. The asymmetric and widened diffraction semblance recorded territory were analyzed by the Fourier analysis method and the average [2]. There are two basic techniques of x-ray line profile analysis: Fourier space technique under which Fourier analysis [3] also forms a part, and real space technique like ; integral breadth variance analysis and peak- fitting methods [4]. It has been shown that each of the above techniques leads to similar results for domain size and dislocation density [5].



Scherrer equation explains peak broadening in terms of incident ray divergence which makes it possible to satisfy the Bragg condition for non-adjacent diffraction planes. Once method effects have been excluded, the crystallite size is easily calculated as a function of peak width (full width at half maximum peak intensity (FWHM), peak position and wavelength. Warren and Averbach's way takes not only the peak width into account but also the shape of the peak. This method is based on a Fourier deconvolution of the measured peaks and the instrument broadening to obtain the true diffraction profile. This process is capable yielding both the crystallite size distribution and lattice micro strain [6]. In the case of sampled diffraction patterns, as in crystallography, Fourier refinement methods generally require a good approximation to the real solution before refinement is at all meaningful. The continuity of the observed Fourier transform partially removes this condition. One further piece of information, namely knowledge that the structure is of finite rather than infinite thickness, is sufficient to restrict the number of possible solutions to a very small number, and often to just one [7].

2. Theory

2.1 Fourier Analysis Method

The shape of the x-ray diffraction of a crystal can be determination in terms of scattering intensity as function of scattering direction defined by the scattering parameter, or by the scattering angle 2θ from equation $n\lambda = 2d \sin\theta$

Empirically the value of $h(2\theta)$ of intensity of the sample MnO for the sample, and the function $f(2\theta)$ can be calculate by the volume of $h(2\theta)$ and $g(2\theta)$ intensity of the stander sample[8]. In addition to the line broadening due to the particle size and strain ,there is a source of broadening due to the equipment itself (slit size, penetration in the sample, imperfect focusing). The source of broadening is called instrumental broadening. A Modification for the contribution of the contributory broadening can be made considering that the experimental profile $h(x)$ is a convolution of the sample profile $f(x)$ and the instrumental contribution $g(x)$.

$$h(x) = f(x) \times g(x) \dots\dots\dots(1)$$

Demonstrated that $F(x)$ could be obtained the Fourier coefficient of $g(x)$ and $h(x)$. The $g(x)$ profile is obtained through the acquisition in the same conditions as the experimental profile $h(x)$, of a standard sample. The observed x-ray diffraction lien profile, $h(\epsilon)$ is the convolution of the instrumental profile, $g(\epsilon)$ and pure diffraction profile $f(\epsilon)$ [9]. Simplified integral-breadth technique that depends on some assumed analytical forms of the peak profiles. The iterative technique of consecutive folding's. Deconvolution technique of Stokes), it follows that deconvolution can be performed easily in Fourier transforms of respective functions [10]:

$$F(t) = H(t)G(t) \dots\dots\dots (2)$$

Where F(t),H(t) and G(t) are Fourier transforms used to calculate the crystallite size and lattice strain by use in also the equation (3) and (4).

$$\text{Function of size (F.S)} = \exp -L/D \dots\dots\dots (3)$$

$$\text{Function of strain (F.}\epsilon) = \exp -2\pi^2 \langle \epsilon L^2 \rangle h^{\circ 2} L^2/a^2 \dots\dots\dots (4)$$

Where D:the effective size

$$\langle \epsilon^2 \rangle_{hk} : \text{the micro strain of the lattice } h^{\circ 2} = h^2 + k^2 + l^2, c^2 = 2\pi^2 h^{\circ 2} / a^2 \text{ [11].}$$

X-ray diffraction from crystal planes happens at well defined angles that satisfy Bragg Numerically, intensity diffracted from an unlimited crystal should contain of diffraction lines without width at some discrete diffraction angles. However, both method and specimen broaden the diffraction lines, and the observed line profile is a convolution of three functions [12]. L is the Fourier length from :

$$L = \lambda / 2 (\sin \theta_2 - \sin \theta_1) \dots\dots\dots (5)$$

Where the peak profile is calculated from θ_1 to θ_2 and λ is the x-rays' wavelength [13]. It was development the Fourier analysis method of x-ray diffraction to calculate variables in crystal structure such as:

A. Calculation between lattice strain and root- mean strain of Fourier method. From the relation between strain and local root mean- square strain which is :

$$\langle \epsilon^2 \rangle^{1/2} = (2/\pi)^{1/2} \epsilon \dots\dots\dots (6)$$

To calculate the lattice structure for the case of a Gaussian micro strain distribution it is possible to calculate the local root-mean-square strain by using root-mean square strain [14,15].

B. Through uniform deformation energy density model (UDEDM) can extract energy density of crystal. In equation (7) :

$$u = (\epsilon^2 / 2) Y(hkl) \dots\dots\dots (7)$$

when the strain energy density u is considered. According to Hooke's law, the constants of proportionality related with the stress and strain relation are not all autonomous, the energy density u (energy per unit volume) is as a function of strain [16-18].

C. From uniform stress deformation model (USDMD) strain is computed of the Hook's Law preserving line a proportionality between stress and strain by:

$$\sigma = Y \epsilon \dots\dots\dots (8)$$

Where σ is the stress and Y is the Young's modulus [19].

Also we can calculate four lattice parameter such as :

2.1.1. Texture Coefficient (TC)

computation of constructing variables to explain the special favored, the texture coefficient $Tc(hkl)$ It is computed using the following formul [20,21]:

$$TC(hkl) = \left[\frac{I(hkl)/I^\circ(hkl)}{\sum_{hkl} I(hkl)/I^\circ(hkl)} \right] \times 100 \% \dots\dots\dots(9)$$

Where $I(hkl)$ is the measured relative intensity of a plane hkl , and $I^\circ(hkl)$ the standard intensity of the plane taking from the JCPDS data [22, 23]. Where is the calculated intensity is I , standard intensity is I_0 , the number of diffraction peaks Nr and miller indices are hkl .

2.1.2 Micro Strains

The micro strains are investigated through the growth of thin films, and will be raised from stretching or compression in the lattice to make a deviation in the a lattice constant so the strain broadening is caused by varying displacements of the atoms with respect to their reference lattice position [24]. This strain can be computation from the formula,

$$\langle \varepsilon \rangle = \beta \cos \theta / 4 \dots\dots\dots(10)$$

Where β is FWHM (radian), θ is Bragg diffraction angle of the xrd peak (degree)[25].

2.1.3. Dislocation Density

A dislocation is an flaw in a crystal related in the lattice in one part of the crystal with that in another fraction. Different vacancies and interstitial atoms, dislocations are not equipoise flaw. Thermodynamic thoughts are respect to calculation far their existence in the observed densities. The growth method involving dislocation is a matter of importance [26]. The intrinsic stress and dislocation density are determined by using the X-ray line profile analysis [27,28]. By using equation (11) the dislocation density (δ) in the sample has been determined and results from both the formulas are approximately same .

$$\delta = 1/D^2 \dots\dots\dots(11)$$

Where δ is dislocation density, and D is crystallite size (nm). The dislocation density of the sample is inversely proportional to crystallite size[29,30].

2.1.4. The Area of the Particle

The stage of the area portray of the nano particles case by the effect of the size and low of this size And the ship between the area and the size is high the size is low Also the specific surface area (SA) can be calculate from :

$$SA = 6 \times 10^3 / Dp \rho \dots\dots\dots(12)$$

Where A is the area of the particle and DP is calculate from manner of method [31-33] and ρ is take of the MnO (5.39 g.cm) [34].

3. Results and Discussion

A . Calculation between lattice strain and root mean strain of Fourier method have been using equation (6) to find the value (ε)

$$\epsilon = 7.4828 \times 10^{-3}$$

The result was lasted in table (1). The equation (6) a good result to calculate the local root-mean-square strain by using root-mean square strain [14,15]

B. The equation has been used (7) to find a value of strain energy (u).

$$u = 2799614.7 \text{ dyne / cm}^2$$

The value energy is lasted also in table (1) . The the strain energy density u is considered and according to Hooke’s law, the constants of proportionality related with the stress and strain relation are no tall autonomous, the energy density u (energy per unit volume) is as a function of strain [16-18].

C. The equation (8) was used to find the stress which was based on the

$$\text{Young’s modulus } \sigma = 7.4828 \times 10^8 \text{ dyne / cm}^2$$

The result is also shown in table (1) . These result is the stress and Y is the Young’s modulus [19] and from The equation (8) we can see the positive relationship between σ and ε and the constant proportional is Y.

Table (1) : Three lattice parameters are calculated from the Fourier method.

Strain (ε)	Stress (σ) dyne/ cm ²	Energy (u) dyne/ cm ²
7.4828 × 10 ⁻³	7.4828× 10 ⁸	2799614.7

It is possible to calculate lattice parameters by the calculated particle size [35].

3.1 Texture Coefficient (TC)

Equation (9) was used to calculate the texture coefficient (TC) for all lines of diffraction pattern and the results are listed in table (2). Shows the high the (TC) value of the (200) peak, meaning that level (200) has the best preferred orientation .

Table (2) : The values of texture coefficient (TC) for lines of diffraction peaks .

Peak (hkl)	TC
111	4.4834
200	29.7288
220	13.3672
311	2.5280
222	1.8507

The Texture Coefficient is used to describe the prevailing trend and represents the shape of the specified level, which includes a deviation from one. This confirms that the direction of the crystal growth of the preferred levels is within this direction. When $T_c \leq 1$ is multi-crystallization, but in non uniform directions, the improvement of crystalline growth of the material is associated with the value of this factor. If $T_c = 1$ is the ideal case for surface growth.

3.2 Micro Strain (S)

The micro strain was calculated using equation (10) then the results are listed in table (9). The micro strain depends directly on the (2θ) and Bragg diffraction angle of the XRD peak . So the strain broadening is caused by varying displacement of the atoms with respect to their reference lattice position, recorded on the micro strain table (3).

Table (3) : The values of Micro strain for all the lines of diffraction pattern .

Peak (hkl)	θ degree	$\beta(\theta)$ radian	$\beta(\theta)\cos(\theta)$	Micro strain (S) ($Lines^{-2}.m^{-4}$)
111	17.75	0.021282	0.020268	5.067×10^{-3}
200	20.3	0.01788	0.016769	4.192×10^{-3}
220	29.5	0.018089	0.015743	3.935×10^{-3}
311	35.375	0.022895	0.018668	4.667×10^{-3}
222	37.125	0.022531	0.017964	4.491×10^{-3}

3.3. Dislocation density

To determine the dislocation density equation (11) was used and the values of dislocation density for all diffraction lines are listed in table (4) .calculate for each line of x-ray diffracted pattern where the crystallites size here represents crystallites calculated in the Fourier analysis method.

Table (4) : The values of dislocation density for lines of diffraction peaks .

Peak (hkl)	D nm	Dislocation density ($lines/m^2$)
111	5.3885	34.4×10^{15}
200	16.087	3.8×10^{15}
220	9.0594	12.1×10^{15}
311	3.9999	62.5×10^{15}
222	3.6828	73.7×10^{15}

3.4 The Area of the Particle

Equation (12) we can calculate the specific surface area of all diffraction peaks and the results listed in the table (5) shows the different result D and the Area of the Particle. The value of the density ρ in equation (12) of MnO nano particle is equal to 5.39 gm/cm^3 [67] .

Table (5) The values of The Area of the particle for lines of diffraction peaks:

Peak (hkl)	D nm	The Area of the particle (m^2/g)
111	5.3885	20.6583×10^4
200	16.087	6.9197×10^4
220	9.0594	12.2874×10^4
311	3.9999	27.8300×10^4
222	3.6828	30.2262×10^4

4. Conclusions

Now that the Fourier method has been developed, it is possible to calculate the new properties of the crystalline structure such as the emotion plus the rate of the emotion box as well as the stress and energy. It is possible to develop this method to calculate other characteristics but they will be left at present as future work. When comparing the Fourier method in the analysis of these methods which are used in this work. It was concluded that the results of the Fourier method are the most accurate results because this method is based mainly on the analysis of the X-ray diffraction line, starting from the line tails up to the top for the intensity and diffraction angle. Fourier methods are the most general method for extracting volume and intensity but require high accuracy in diffraction line analysis as well as in calculating granular size and emotion separately from each other. The calculation of most of the crystalline parameters is linked to the particle size and the strain of the calculation. Therefore, the particle size must be calculated precisely because it gives accurate information about the crystalline structure .

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