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Using the Size Strain Plot Method to Specity Lattice Parameters

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Abstract

X-ray diffractometers deliver the best quality diffraction data while being easy to use and adaptable to various applications. When X-ray photons strike electrons in materials, the incident photons scatter in a direction different from the incident beam; if the scattered beams do not change in wavelength, this is known as elastic scattering, which causes amplitude and intensity diffraction, leading to constructive interference. When the incident beam gives some of its energy to the electrons, the scattered beam's wavelength differs from the incident beam's wavelength, causing inelastic scattering, which leads to destructive interference and zero-intensity diffraction. In this study, The modified size-strain plot method was used to examine the pattern of x-ray diffraction lines (101),(121),(202),(042), and (242) for calcium titanate(CaTiO3) nanoparticles in this study. To calculate the new variables, the size strain plot method was created., X-ray line analysis and calculation of crystal size and lattice tension of calcium titanate oxide nanoparticles. It is used to calculate the crystal volume (44.7 nm) and to calculate the determination of network parameters such as the texture modulus (Tc), macro stress (MS), specific surface area (SSA), and dislocation density(η), respectively.

Keywords: Calcium titanate,size strain plot,Texture Coefficient

1. Introduction

The most important method for determining crystal structure is X-ray diffraction (XRD). The size of crystalline blocks and the degree of crystalline structural deformation in nanocrystals can be determined using this method. Methods for analyzing nanocrystalline materials using X-rays are currently being developed. [1-2]. X-ray diffraction is the most important method for determining crystal structure (XRD). This method may determine the size of crystalline blocks and the degree of crystalline structural deformation in nanocrystals. X-ray analysis methods for nanocrystalline

materials are currently being developed. The X-ray profile analysis is an average method widely used in determining crystalline size. In the Size-strain plot technique, which assumes that the Gaussian function governs the (strain profile), the significant angular vertices are less critical. [3].

Calcium titanate ($CaTiO_3$) is a material of Perovskites and one of the most prevalent structural families. It is present in various compounds with diverse properties, uses, and significance. Paul Scherer developed Schererer's Formula in 1918, which computes the crystal size (D) of nanomaterials using the entire width of the X-ray diffraction pattern at half is the most significant value of the peaks [4]. The diffraction pattern's maxima are widened by a factor which is inversely proportional to crystallite size, and the extra broadening is measured. The formula can be used to estimate the size of a powder specimen if the crystallites are small enough [5].

Calcium Titanate is a colorless, diamagnetic solid, often coloured owing to impurities. It is a chemical compound of titanium, oxygen, and calciumPerovskite is a mineral with the formula $CaTiO_3$.

Calcium titanate ($CaTiO_3$) is semi-conductive, photorefractive, and ferroelectric. It is reduced to give ferrotitanium alloys or titanium metal [6]. Multiple methods can prepare calcium titanate. $CaTiO_3$ has been synthesized at high temperatures using mixtures of calcium carbonate ($CaCO_3$), titanium dioxide (TiO_2), and calcium oxide (CaO). It also uses forgiving chemistry methods such as sol-gel or solvothermal methods, hydrothermal or organic-inorganic solution, or coprecipitation [7].

2. size strain plot

Data from high-angle reflections are given less weight in this strategy. Because XRD data is of lower quality and peaks overlap at greater angles with higher diffracting, the isotropic broadening is improved. The Gaussian function depicts the strain profile, while the Lorentzian function depicts crystallite size [8]., according to this assumption. The equation was also used to express the real broadening of this method.

$$\beta_{hkl} = \beta_L + \beta_G \qquad (1)$$

L and G Peak broadening is represented by Lorentz and Gaussian functions, respectively.

size-strain plot method can be determined by the following equation:

$$(d_{hkl}\beta_{hkl}cos\theta)^2 = \frac{k}{D}(d^2{}_{hkl}\beta_{hkl}cos\theta) + (\frac{\varepsilon}{2})^2 \qquad (2)$$

For spherical particles, the form is expressed as 3/4, where K is a constant

In comparison to other methods, the Size-Strain plot method has a significant benefit, because it gives the great angle peaks are given less weight, [9]. As a result, despite the fact that the Gaussian function is believed to control (the strain profile), the great angle peaks are given less weight. [3].

If ($\varepsilon = 0$)

$$\mathrm{D} = \frac{\mathrm{k}(d^2{}_{hkl}\beta_{hkl}cos\theta)}{(d_{hkl}\beta_{hkl}cos\theta)^2} \qquad (3)$$

If(D=∞)

$$(\frac{\varepsilon}{2})^2 = (d_{hkl}\beta_{hkl}cos\theta)^2 = \mathrm{y\_intercept} \qquad (4)$$

Herein case, Equation (2) was used to calculate the crystallite size and lattice strain. As indicated in the table, ($d_{hkl}{}^2\beta_{hkl}\cos\theta$) on X-axis and ($d_{hkl}\beta_{hkl}\cos\theta)^2$ on the y- axis is calculated for each line shape as shown in **Table (1)**. Using the marks of plotting the relationship, the crystallite size and lattice strain were calculated. between ($d_{hkl}{}^2\beta_{hkl}\cos\theta$) and ($d_{hkl}\beta_{hkl}\cos\theta)^2$.

Table 1. The results the Size Strain Plot method of ($d_{hkl}^2\beta_{hkl}\cos\theta$) and ($d_{hkl}\,\beta_{hkl}\cos\theta)^2$ for all the profile lines

Peaks	**2θ(degree)**	**θ(degree)**	**$cos\theta$**	**β(deg)**	**$\beta\ (rad)$**	**d (nm)**	**(dβcosθ)^2 X10^{-6}**
(101)	23.26247	11.63123	0.979465	0.219002	0.00382	0.38207	2.04398
(121)	33.25642	16.62821	0.958181	0.392082	0.00683	0.26918	3.11219
(202)	47.48688	23.74344	0.915357	0.292729	0.00510	0.19131	0.79966
(042)	59.30423	29.65211	0.869045	0.731695	0.01276	0.15570	2.98290
(242)	69.53593	34.76796	0.821468	0.310271	0.00541	0.13508	0.36071

The slope and intercept of the plot were calculated in **Figure (1)**, using equation (2) to calculate the framework strain from the y-intercept and the crystallite size of the slope (2).

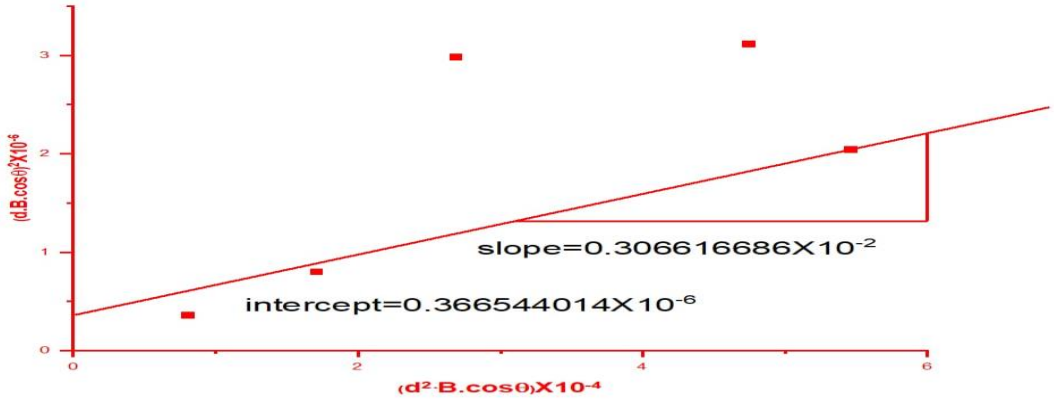

Figure 1. Plot of Size-Strain plot method

Table 2. Using Size –Strain plot to calculate crystallite size and lattice strain

K	λ (nm)	Y(intercept)	X_1	X_2
0.89	0.15406	0.366544014X10^{-6}	3.0936432X10 $^{-4}$	5.95625427X10^{-4}
slope	D(nm)=K/slope	$\mathcal{E}$=2(intercept)$^{1/2}$	Y_1	Y_2
0.306616686X10^{-2}	44.71	1.21085X10^{-3}	1.31262383X10^{-6}	2.19034815X10^{-6}

Table (3) Calculation of the lattice strain, stress, the crystallite size, and the energy after modifying

$\mathcal{E}$=2(intercept)$^{1/2}$	U (KJ/m^3)	σ (G Pa)	D(nm)
1.2108X10^{-3}	0.1158X10^{-3}	0.18430463	44.71

3. Determination of the lattice parameters:

3.1 Texture Coefficient (Tc):

The texture coefficient Tc was used to quantify the XRD results (hkl). The following equation can be used to compute this factor for each direction.

$$T_{c(hkl)} = \frac{I_{(hkl)}/I_{0(hkl)}}{(1/N)[\sum_N I_{(hkl)}/I_{0(hkl)}]} \qquad (5)$$

The obtained and standard intensities of the (hkl) plane are represented by I(hkl) and I0(hkl), respectively, and N denotes the number of diffraction peaks [10].

To determine whether one orientation stands out from the others, the T_C formulation compares the power ratios of surfaced coatings to non-textured models. The link is familiarized by comparing each ratio's average value of all replication intensity ratios. The total number of reflections should be equal to the sum of the T_C values, so the T_C per reflection cannot exceed n. A T_C value is greater than the one indicating a texture [11].

3.2 Micro strains (MS):

During thin film growth, microstrains form as a result of pressure or stretching in the lattice, causing it to deviate from the lattice constant. As a result, strain stretching is caused by changing the displacement of the atoms in relation to their reference lattice position. The strain was calculated using the equation:

$$< MS >= \frac{\beta\ cos\theta}{4} \qquad (6)$$

Where: β=FWHM of the intensity of the peak in radian;θ= Bragg angle [11].

3.3 Specific Surface Area (SSA)

Because of the large surface-to-volume ratio with decreasing particle size, Surface states will be crucial in nanoparticles [12]. A crucial characteristic is specific surface area.

It is a quantitative value derived from information that can be used to identify the kind and characteristics of a material. It is especially important in adsorption, heterogeneous catalysis, and surface reactions. SSA denotes the Surface Area (SA) per mass according to [13] (Zhang et al., 2016). The specific surface area and the surface-to-volume ratio of materials increase dramatically as their size decreases [14].

$$SSA = \frac{SA_{part}}{V_{part} * densty} \qquad (7)$$

$$S = 6 * \frac{10^3}{D_p} \qquad (8)$$

Where (S) is the specific outward area, (D) is the sizespherical shaped; and (P) is the density of $CaTiO_3$

3.4 Dislocation density (η)

The Dislocation density (η) can be calculated from equation [15].

$$\eta = \frac{1}{D^2} \quad (lines/nm^2) \qquad (9)$$

4. Result and Discussion

Using equation (5), we calculated the outline lines in the $CaTiO_3$ powder x-ray diffraction design (5). When TC >1, it is confirmed that the selected levels' crystal development will proceed in this direction. The improvement of the material's crystal formation is correlated with the value of this factor when TC < 1 is polycrystalline but in a non-uniform direction. The best situation for surface expansion is if TC=1.
Table (4) shows the Texture Coefficient results for all outline lines.

Table 4. The results of (Tc) for all outline lines.

Peaks	I	I (h kl)	I (h kl)/I_0(h kl)	Tc
(1 01)	10	400.6596	40.06596	1.341837
(1 2 1)	100	1802.3441	18.0234	0.6036
(2 0 2)	40	916.6432	22.9168	0.7675
(0 42)	15	608.5128	40.5675	1.3586
(2 42)	20	554.4279	27.7213	0.9284
Sum			149.2951376	AV: 1

Micro strains were premeditated using equation (6) for the outline lines in the $CaTiO_3$ powder x-ray diffraction design. **Table (5)** shows the Micro strains results for the outline lines .

Table 5. The results of Micro strains all peaks of $CaTiO_3$ nanoparticles

(h k l)	2θ(degree)	θ(degree)	Cos θ	β (rad)	<M s>$X10^{-3}$
(1 01)	23.26247	11.631235	0.979465485	0.003820379439	0.93548245
(1 2 1)	33.25642	16.62821	0.958181797	0.006839663761	1.638410328
(2 02)	47.48688	23.74344	0.915357585	0.005106502732	1.168569002
(0 42)	59.3042323	29.65211615	0.869045281	0.012764026	2.77312914
(2 42)	69.5359388	34.7679694	0.821468132	0.005412508205	1.111550751
Sum					7.626141671
AV.					1.52

For all of the outline lines in the x-ray deflection pattern of CaTiO3 powder, the Specific Surface Area was calculated using equation (7). Table 1 shows the results of the Specific Surface Area for all of the outline lines.

Table 6. The results of specific surface Area (SSA)

D (nm)	V part= 4/3D	SSA(m^2/g)
32.29477611	43.05	35.36
SA	Intensity	
$6X10^3$	3.94	

It can be calculated Specific Surface Area from equation (9) for all the outline lines in the x-ray diffraction pattern of $CaTiO_3$ powder. The results of Dislocation density (η) for all the outline lines are registered in **Table (7).**

Table 7. The results of dislocation density (η)

(h kl)	D (nm)	η=$1/D^2$ (lines/nm^2)

(1 01)	36.6424298	0.744785942
(1 21)	20.92171259	0.002284575613
(2 02)	29.33361225	0.001162167982
(04 2)	12.36089207	0.006544860152
(2 42)	30.83831302	0.001051523997

5. Conclusions

In this study, all the profile lines in the $CaTiO_3$ powder's x-ray diffraction pattern had their micro strains calculated. A specific surface area was calculated for all the profile lines in the $CaTiO_3$ powder x-ray diffraction pattern. For all profile lines in the x-ray diffraction, the dislocation density (h) can be calculated in the x-ray diffraction pattern of $CaTiO_3$ powder. It can be used to determine the kind and characteristics of a material. All the profile lines in the $CaTiO_3$ powder's x-ray diffraction pattern had their texture coefficient calculated. When TC>1, for(101),(042), it is confirmed that the preferred levels' crystal growth direction is in that direction. The improvement of the material's crystal growth is correlated with the value of TC, but TC <1 for (121), (202), and (242) is polycrystalline, albeit in a non-uniform direction.

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