Studying the Connection Between Partical Size and Lattice Distortions Through X – Ray Diffraction Line Profile Analysis for CaO Powder

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Abstract

The integral breadth method has been utilized to analyse line profiles broadening and lattice strain of CaO at different temperatures

The effect of temperature on crystallite size and strain has also been investigated . The crystallites are found to be highly anisotropic even at high temperatures .

Introduction

A detailed knowledge of crystallite size and strain in a finely divided powder often helps to correlate many physical properties of a system under going transformation and solid – state reactions . X – Ray line broadening analysis provides a method of finding crystallite size and strain integral breadth methods (1 – 4) .

Calcium oxides have been important applications in manufacuturing (5 – 8) . The present investigation aims were to find the variation of crystallite size and strain with temperature after correction line profile .

Experimental

In this work, calcium carbonate was used to produce calcium oxide by treatment of $CaCO_3$ at 1100 °C , 1150 °C and 1200°C . The calcination time is chosen as 30 minutes for each run under static air , heating rate of 10°C /min and α- alumina as reference material . Diffraction pattren for the three samples were obtained using Philips automatic powder diffractometer (pw1820) with flat monochromator , step rate 0.02/1.0sec. Specimens were prepared by mounting

amono – layer of each sample on glass slide that had been coated with silicon gerase . Estimates of line position , breadth were taken from chart recording over the angular range 31 to 55 (2θ) . The peak of each line was taken as its position and width at half the maximum height ,2w , was used as a measure of breadth .Three peaks (111, 200 and 220) were used for this study and the peak of standard specimen SiO_2 as the instrumental correction .

Analysis of line breadth :

A preliminary investigation on the calcium oxide powder shows it to be cubic with the lines of the JCPDS card no.4 – 0777 . Three lines resolved were taken for analysis . No multiple - order reflections were observed .In order to correct the FWHM of the broaded profile (h) for the contribution from the instrumental function (g) , both functions were assumed to be gaussian . This was chosen in accordance with the condition given by (9) :

$2w / B2\theta = 2JI = 0.63662$ (Cauchy) $= 2(\log c^2)^{1/2}/JI^{1/2} =$ 0.93949(Gaussian) ----------[1]

Where 2w is the FWHM and B2 θ is the integral breadth . Since in the present case $2w / B2\theta > 1$, the Gaussian assumption seems to be valid Dcconvolution was accomplished with the relation given by(10)

$2w\ f = [(2w\ h)^2 - (2wg)^2]^{1/2}$------[2]

where 2wf, 2wh, 2wg are the FWHM for f , h and g profiles respectively .

The breadth in (2 θ) is related to breadth in S by

$B\ 2\theta = Bs\ d\ (2\theta)/ds$ ----------[3]

Where Bs is fiend as the ratio of the total intensity of the line to its peak intensity .

Equation (3) which becomes , since

$S = (2d \sin\theta / \lambda\kappa) - n$ ----------[4]

$B2\theta = Bs\ \lambda\kappa/d\cos\theta$ ----------[5]

Where K is the Scherres constant whose value (O. 94).

Then the quantity

$E = \lambda\kappa\ /\ B2\ \theta \cos\theta = d/Bs$----------[6]

has the dimensions of length , and E called crystallite size ,

Wilson (11) has shown that the integral profile breadth in (S) units generated by distortions (strains) alone may be expressed:

$Bs = 4e \sin\theta / \lambda = 2es = 2e/d_{hkl}$------[7]

Equation [7]may be expressed on the 2 θ scale in radians as :

$B2\theta = 4e \tan\theta$ ----------[8]
Stokes and Wilson (12) have defined an " apparent strain " η by
$\eta = B2\ 0 \cot\theta$ ----------[9]
Which may be written in the manner of equation [8] as :
$B2\theta = \eta \tan\theta$ ----------[10]

Results and Discuion

Figures (1a – c) shows powder x- Ray diftraction of CaO at 1100 °C ,1150 °C and 1200 °C, respectively . Table (1) shows the variation 2wh for line profiles and 2wf for correction line profile with calcinations temperature for the three reflections 111 , 200 , and 220 the integral breadth decreases with temperature decreases from 1100 °C to 1200 °C . the crystallite sizes at different temperatures were determined from equation [6] . The results are shown in table (2) the curves for different reflections are quite different which means that there is a considerable amount of lattice distortion (apparent strains) η

In directions perpendicular to the 111 , 200 , and 220 planes . This indicates the anisotropic nature of the crystallites in this direction This results in crystallite size and strain broadening of the profiles that have been observed in (13) which used Fourier analysis method and with ratio error depended on the method analysis .In general method, the crystallite size in the direction considered increases and lattice strain decreases with increasing the calcinations temperature in the range 1100°C to 1200°C .

From the above observations , the following can be concluded :

- The crystallite sizes found in (111) , (200) and (220) planes for CaO at 1100 °C , 1150 °C , and 1200°C , are of the same order of magnitude .
- The size of crystallites perpendicular of the (111) plane is smaller than those perpendicular to the (200) and (220) plane
- The growth of crystallites is developed for the three crystallo graphic directions simultaneously and the differences between the broadening of these reflections decreases with increasing the calcinations temperature up to 1200 °C , i.e. , the anisotropy can be reduced by high temperatures of calcinations.
- An inverse relationship is observed between the crystallite and apparent strain for CaO at different temperatures .

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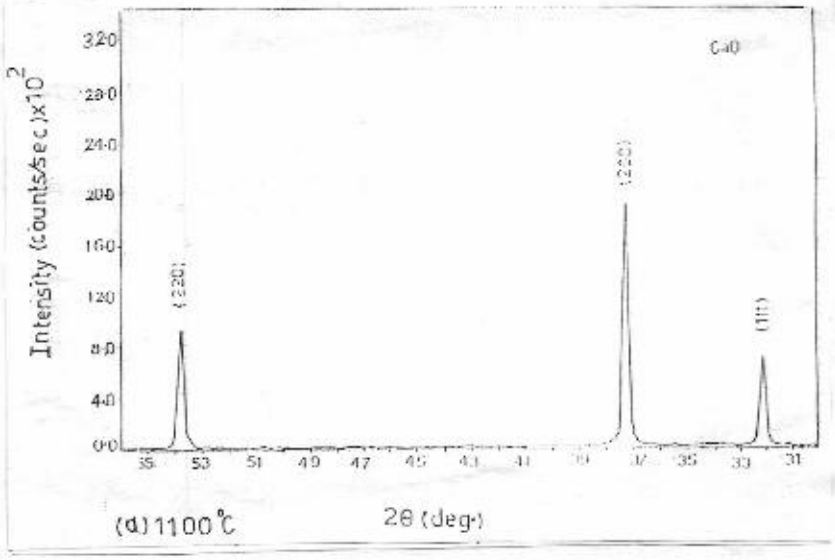
Table (1) Experimental FWHM (2Wh) pure diffraction FWHM (2Wf) and integral breadth (B2O)for CaO obtained at 1100°C , 1150°C and 1200°C for (111) ,(200) , and (220) planes

Reflection	Temp (°C)	2 W h(deg)	2W f(deg)	B2 θ X10^{-3}(rad)
111	1100	0.314	0.298	5.367
	1150	0.264	0.234	4.346
	1200	0.228	0.193	3.584
200	1100	0.292	0.265	4.922
	1150	0.257	0.226	4.197
	1200	0.214	0.176	3.269
220	1100	0.285	0.258	4.792
	1150	0.242	0.209	3.881
	1200	0.207	0.167	3.101

Table (2) Crystallite size and strain for CaO for three planes at different tempertures

Reflection	Temp (°C)	Crystallite size(nm)	Strain $X10^{-3}$
111	1100	28.06	18.64
	1150	34.65	15.08
	1200	42.02	12.44
200	1100	31.04	14.56
	1150	36.40	12.04
	1200	46.73	9.68
220	1100	33.85	9.46
	1150	41.80	7.64
	1200	52.32	6.12



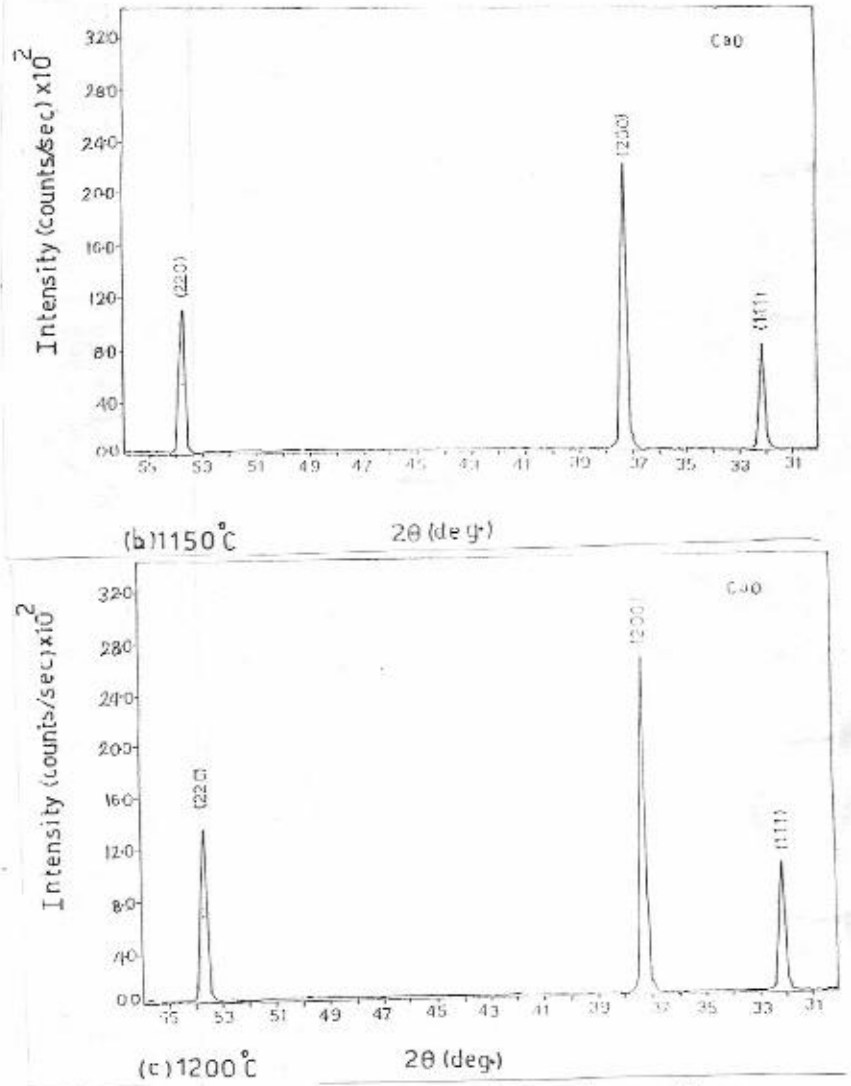


Fig (1 a- c) X – Ray diffraction patterus of calcium carbonate after thermal decomposition at 1100 °C (a) ,1150°C (b)and 1200°C (c)

دراسة الربط بين المقاس الحبيبي وتشوه الشبيكة من الاشكال الجانبيه لحيود الاشعه السينيه لمسحوق CaO

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الخلاصة

تم استخدام طريقه العرض المتكامل لتحليل الخطوط الجانبيه لحيود الاشعه السينيه وأنفعال الشبيكة لمسحوق اوكسيد الكالسيوم . وتم دراسه تأثير درجه الحراره على المقاس الحبيبى والأنفعال . وأظهرت النتائج ان درجه التبلور غير متشابهه حتى عند درجات الحراره العاليه .