

The Separation Particle Size and Strain by the
Scherrer Analysis Method

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Receiving Date: 2011/5/2 - Accept Date: 2011/6/7

Abstract

The Variation of crystallite size and strain of calcium oxide powder with the carbonate temperature of calcium carbonate is studied applying Scherrer analysis method for (111),(200) and (220) reflections .

The results show that size of the crystalline increases and strain decreases with increasing the rang 1100°C to 1200 °C , but the changes are dependent on the crystallographic direction .

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الفصل بين المقاس الحبيبي و الانفعال باستعمال طريقة شيرر في التحليل

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الخلاصة

تم دراسة مقاس الحبيبية والانفعال لمسحوق اوكسيد الكالسيوم المحضر بطريقة حرق كاربونات الكالسيوم بتطبيق طريقة تحليل شيرر لخطوط الانعكاس (111)، (200) و (220).
واظهرت النتائج انه بزيادة درجة حرارة الحرق من 1100°C الى 1200°C يزداد المقاس للحبيبية ويقل الانفعال ولكن التغيير يعتمد على تلك الاتجاهات.

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K.H.Harbbi

Introduction

Though the Scherrer method or half maximum breadth method is a frequent measure of mathematical distributions, its use has been limited in the field of X-ray diffraction. The applicability of this method to the analysis of the line broadening of Scherrer powder pattern peaks has been clearly demonstrated by Wilson [1] and developed by Klug and Alexander [2].

The oxide Mo, where M=Ca,Sr,Ba,.....,etc are best obtained by calcinating . The carbonates, dehydration of the hydroxide at red heat offers an altimate route [3, 4]. CaO is produced on an enormous scale in many countries. Its major end is used as a flux in steel manufacture, in the production of Ca chemicals, in the treatment of municipitl water supplies, in industrial wasts, in paper industry and in non-ferrous metal production[5]. It is very well know that the chemical reactivity of solids is strongly dependent on both crystallite size and construction of strains. These two facts explain the importance of the mechanical treatments of the materials in chemical technology in order to enhance the reactivity of solid. Thus , it has been report[6]that is possible to carryout, by grinding at room temperature, solid state reactions which to occur would ordinarily require much higher temperature.

Also the study of solid state reaction mechanism is usually approached either through kinetic investigations [7-9].

This paper present for the first time , according to our knowledge the effect of calcinations temperature on the crystallite size and crystal distortion (strain) in CaO powder derived from the thermal decomposition of **CaCo₃** powder for 30 minutes. The calcinations temperature is ranged from 1100°C to 1200°C .

Scherrer Analysis of Line β breadth

The first treatment of crystallite size broadening was due to Scherrer who investigated the case of crystals of regular cubic. The angular breadth (β) at half maximum intensity of a diffraction line produced by the powder method is given by [10]

$$\beta = k\lambda/L \cos \theta \quad \dots\dots\dots (1)$$

The Separation Particle Size and Strain by the Scherrer Analysis Method

K.H.Harbbi

Where L is the crystallite size, λ is the wave length of the radiation, β is the corrected breadth (in radians), and θ is the Bragg angle, k is the Scherrer constant whose value 0.94 [11]. This constant depends on the definition of breadth β [12].

Also the Scherrer constant depends on the crystal shape and the diffraction line indices, and in all cases to be not for from unity.

In a distorted crystal the interplaner spacing d will not be constant throughout and different parts of the crystal will reflect at different angles. The broadening of the lines in the powder pattern of in aggregate of such distorted crystals may be estimated from Bragg equation.

If the maximum strain in the crystal is ϵ the spacing will vary from $d(1 + \epsilon)$ to $d(1 - \epsilon)$, and θ will vary in a range numerically equal to $2\epsilon \tan\theta$. Since the deviation of the rays is 2θ , the range of angle over which the reflexion is appreciable will be [13]doubled:

$$\epsilon = \frac{\beta}{4 \tan\theta} \dots\dots\dots(2)$$

Where ϵ the crystal distortion (strain).

To find the correct crystallite size, correction for instrumental factor has to be applied on the breadth of the diffraction line. A number of methods have been proposed, some assume the shape of the line, while others donot.

The two most commonly assumed line shapes are the Cauchy and Gaussian functions. For Cauchy profile, originally made by Scherrer in his original analysis of crystallite size effects, the correct crystallite size breadth is given by [14]:

$$\beta = \beta_{meas} - \beta_{instr} \dots\dots\dots(3)$$

Where β_{meas} is the half width of the experimentally measured breadth and β_{instr} is the half width of the instrumental breadth.

The Separation Particle Size and Strain by the Scherrer Analysis Method

K.H.Harbbi

For Gaussian profile, which is more correctly represent the actual reflection profile and instrumental profile, and the relation for the correct size as [12]:

$$\beta^2 = \beta_{meas}^2 - \beta_{instr}^2 \quad \dots\dots\dots (4)$$

The relationship is holding for both – maximum and integral breadth. Since the actual profiles are never pure Gaussian or pure Cauchy [15]. These simple formulae have only limited practical value.

Since in the present case the full width at half the maximum, $\frac{W}{P} > 1$, the Gaussian assumption seems to be valid Decouvolution was accomplished with the relation [4].

Results and Discussion

Broadening of an X-ray diffraction profile is normally caused by small crystallite size, non-uniform strain within the crystallites and faulting on certain crystal planes.

The peak breadth of broadened line can be expressed interms of the crystallite size, hence measurement of the peak breadth gives method for determining crystallite size. An additional broadening termed "instrumental " arising from effects such as slit widths and wavelength widths must be corrected for in order obtain the correct size.

Figure (1) show X-ray diffraction spectra for CaO powder at 1100°C to 1150°C and 1200 °C [16]. Table (1) shows the half width β_{meas} for line profiles and β for corrections line profile with calcinations temperature for three reflections (111),(200), and 220 the half width decreases with temperatures increases from 1100°C to 1200°C . The crystallite size at different temperatures was determined from equation (1). The strain values are obtained from equation (2) and listed an lision Table (2). It is apparent that the temperature of calcinations has a significant effect on the half width specially above 1100°C when the conversion is at its beginning .the values of β for curves (111),(200),and (220) reflections in CaO obtained at 1100°C to 1150°C and 1200°C look different. This means that there is an amount of distortion in directions perpendicular to the (111),(200),and (220) planes. This indicates the anisotropic of the crystallites in there directions. Moreover as the temperature of calcinations increases from 1100°C to 1200°C , the crystallite size increases progressively for the above

**The Separation Particle Size and Strain by the
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K.H.Harbbi

reflections simultaneously and the broadening between the of (111) and other reflections becomes smaller which leads to larger crystallite size (94.677 nm).

The results in this table (2) show that as temperature increases the crystallite size perpendicular to (111),(200), and (220) planes increases when as, inverse behavior it observed for strains. The above behavior could be understood by assuming that the particles of the powder samples are formed by small crystallite welded in a mosaic structure and that the corresponding grain boundaries constitute the main contribution to the particle the lower the size of the crystallite.

Conclusion

- 1- The crystallites are highly an isotropic in nature and this an isotropic cannot be removed until temperature of 1200°C is reached
- 2- Temperature has a prominent effect on the size of the crystallites and the strain of sample
- 3- There is a considerable amount of distortion in directions perpendicular to the (111), (200) and (220) plans. This indicates the an isotropic nature of crystallites in these diractions.
- 4- However, increases in strain can explain this broadening of the profile.

The Separation Particle Size and Strain by the Scherrer Analysis Method

K.H.Harbbi

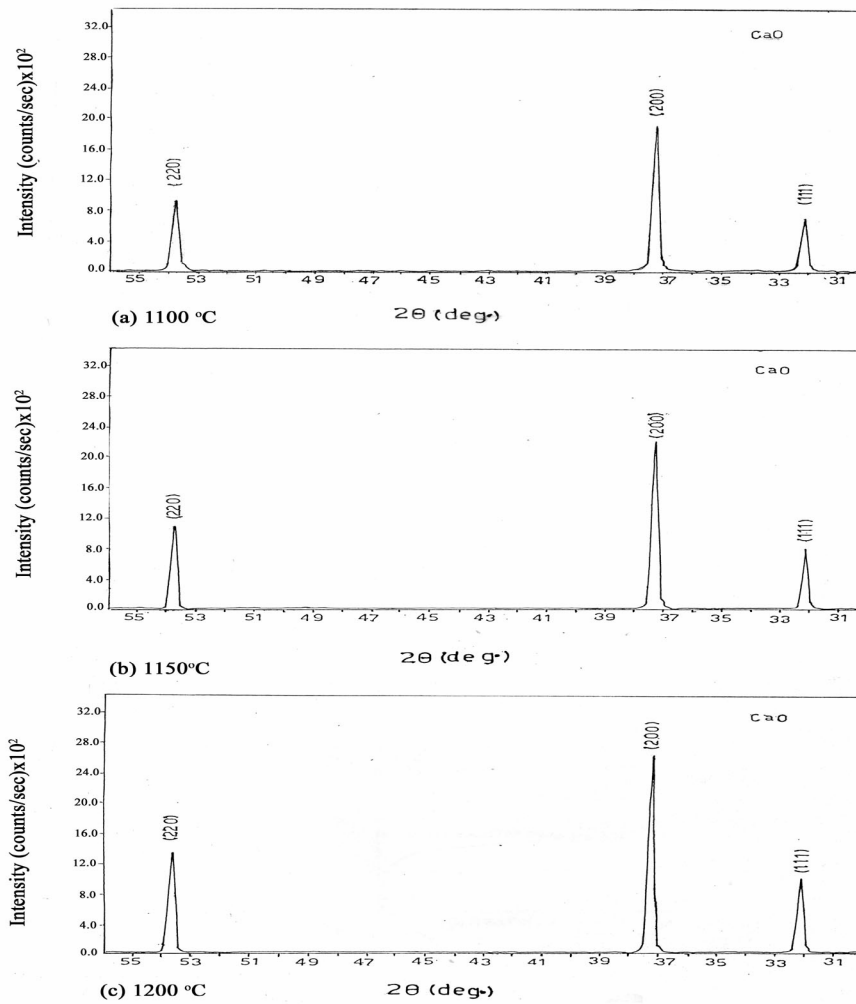


Figure (1): X-ray diffraction spectra for CaO powder at 1100°C to 1150°C and 1200°C

**The Separation Particle Size and Strain by the
Scherrer Analysis Method**

K.H.Harbbi

Table (1) Experimental measured breadth β_{meas} and crystallite breadth β for CaO obtained at 1100°C to 1150°C and 1200°C for (111), (200), and (220) planes

Reflection	Temperature(°C)	$\beta_{meas} \times 10^{-3}$ (rad)	$\beta \times 10^{-3}$ (rad)
111	1100	3.962	3.107
	1150	3.141	2.027
	1200	2.879	1.591
200	1100	3.455	2.486
	1150	2.879	1.591
	1200	2.617	1.045
220	1100	3.402	2.412
	1150	2.792	1.428
	1200	2.530	0.803

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

Reflection	Temperature(°C)	Crystallite size(nm)	Strain $\times 10^{-3}$
111	1100	48.481	2.699
	1150	74.312	1.761
	1200	94.677	1.382
200	1100	61.457	1.841
	1150	96.029	1.178
	1200	146.203	0.774
220	1100	67.268	1.191
	1150	113.621	0.705
	1200	202.057	0.396

The Separation Particle Size and Strain by the
Scherrer Analysis Method

K.H.Harbbi

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