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X-RAY DIFFRACTION BY ZINCIAN DOLOMITE

Abstract. Theoretical calculations of the intensities of X-ray reflections were carried out for different members of the $Ca(Mg, Zn)(CO_3)_2$ series. Some disturbing factors, i.e. Fe^{2+} substitution for Mg^{2+}, disorder of Ca and (Mg, Zn) over the cation positions and the possibility of disorder in zincian dolomite were taken into account. The experimental values of reflection intensities of zincian dolomite from the Waryński mine (Upper Silesia, Poland) point to Zn^{2+} for Mg^{2+} substitution close to 50%, which agrees fairly well with the results of chemical analysis.

INTRODUCTION

The problem of zincian dolomites, i.e. of the possibility of substitution of zinc for magnesium in the crystal structure of dolomite, arouses more and more interests among mineralogists and geologists concerned with ore deposits. The present state of knowledge of this problem has recently been summarized by Żabiński (1981).

Dolomites in which a considerable amount of Zn^{2+} substitutes for Mg^{2+} ions, were reported first time by Hurlbut (1957) from the Tsumeb mine, SW Africa. In subsequent years it was stated that zincian dolomites are widespread in the Silesian—Cracow Zn—Pb ore deposits. In a galmei sample from the Waryński mine (Upper Silesia) zincian dolomite with a high Zn content was found (Żabiński 1959). On the basis of "wet" chemical analysis of this sample the following formula of dolomite could be established: $Ca(Zn_{0.55}Mg_{0.45})(CO_3)_2$, minor elements being neglected, i.e. Zn^{2+} substitutes for Mg^{2+} up to more than 50 atomic per cent. This is all the more true since the electron microprobe investigations proved the non-uniform distribution of Zn in the individual dolomite crystals (Jasieńska, Żabiński 1972) whereas the sample subjected to "wet" analysis was an averaged one.

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On the basis of an X-ray powder pattern taken by film technique in a 114 mm camera, the following cell parameters of Zn-dolomite from the Waryński mine were calculated: $a = 4.815$, $c = 16.06$ Å (Żabiński 1981). It has been found, that the presence of Zn in the crystal structure of dolomite results only in small change of cell parameters. This influence is much more noticeable in the case of the c parameter (000l type reflections) than the a parameter (hki0-type reflections).

The subject of this paper is a study of the influence of Zn on the intensity of the X-ray reflections of dolomite. Theoretically calculated intensities for different members of a hypothetical $Ca(Mg, Zn)(CO_3)_2$ series were compared with the reflection intensities measured for Zn-dolomite from the Waryński mine.

X-RAY DIFFRACTION

The effect of zinc substitution for magnesium in dolomite on the X-ray diffraction pattern of the dolomite is surprisingly small. It is immediately comprehensible that the positions of the diffraction lines should be almost unchanged, since the ionic radii of Zn^{2+} and Mg^{2+} are virtually identical and the unit cell size is therefore only slightly affected. This is also true for Fe^{2+} substitution by Mg^{2+} (Howie, Broadhurst 1958) in spite of the much greater difference in size (Whittaker, Muntus 1970). However, it seems more surprising that the intensities of the lines are not very noticeably different, since Zn (atomic number 30) has a much greater X-ray scattering power than Mg (atomic number 12). In fact there are differences between the intensities of the lines of zincian dolomite and those given in the XRPDS Powder Diffraction File, but the significance is uncertain because different samples of dolomite of ordinary composition can give diffraction patterns whose lines differ substantially in intensity. This is believed to be due to varying degrees of order-disorder in the occupation of the two cation sites by Ca and Mg (Goldsmith, Heard 1961). In order to assess the significance of the intensities observed in the powder pattern of the zincian dolomite the following investigation was undertaken.

The structure factors for all observable reflections out to $2\theta = 60°$ were calculated using the atomic positional and vibrational parameters of dolomite given by Steinfink and Sans (1959). The structural details are given in Table 1. The structure factors were squared and corrected for the angular (L, P, arc length) factors appropriate to diffractometer recording with CuK_α radiation, and for multiplicity of the crystallographic forms. The intensities so derived were then scaled to $I_{10\bar{1}4} = 100$ in order to provide relative intensities that could be compared with the Powder Diffraction File and with the relative values obtained from experiment. Similar calculations were performed for hypothetical structures having 25, 50, 75 and 100% substitution of Mg^{2+} by Zn^{2+}, on the assumption that this would not affect the atomic parameters, and the results are listed in Table 2 and those for seven sensitive lines shown graphically in Fig. 1. The assumption is well founded as regards the positional parameters because of the close similarity in size of Mg^{2+} and Zn^{2+}, but their different masses would be expected to lead to some difference in their vibrational parameters. However, the effect of the temperature correction for Mg^{2+} is not very great, so that the expected slightly smaller effect of the true temperature correction for Zn^{2+} would not be expected to make very much difference, and would certainly be negligible at the lower 2θ values.

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In assessing the correspondence between the observed relative intensities and those calculated for various Zn contents it is necessary to take into account of other possible disturbing factors. One of these is Fe^{2+} substitution for Mg^{2+}.

Table 1

Atomic positions in dolomite from Steinfink and Sans (1959)

The vibration parameters have been modified, from the form in which they were given, for ease of computation such that the temperature factors is

$$\exp\{-A_1(h^2+hk+k^2)-B_2l^2\}$$

The space group is $R\bar{3}$

	Multiplicity of site	x	y	z	A_1	B_2
O	18	0.2374	−0.0347	0.2440	0.0085	0.00075
C	6	0	0	0.2435	0,0089	0.00035
Ca	3	0	0	0	0.0029	0.00020
Mg	3	0	0	0.5	0.0053	0.00029

The trigonometrical contributions of the sites to the structure factor are

$$A_0 = 6\{\cos 2\pi(hx+ky+lz) + \cos 2\pi(kx+iy+lz) + \cos 2\pi(ix+ky+lz)\}$$
$$A_c = 6\cos 2\pi lz$$
$$A_{Ca} = 3$$
$$A_{Mg} = 3\cos 2\pi lz$$

for $-h+k+l = 3n$, and zero otherwise

Table 2

Calculated relative intensities as a function of Zn substitution

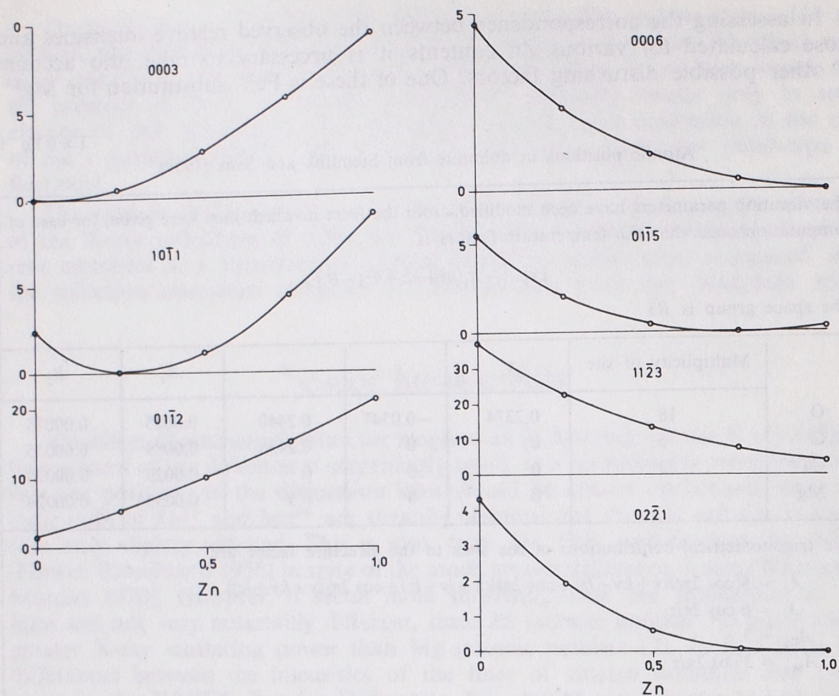| hkil	Mg	Mg.75Zn.25	Mg.5Zn.5	Mg.25Zn.75	Zn	Pure Mg dolomite with 35% Mg, Ca disorder
0003	0.1	0.5	2.6	5.6	9.1	0.6
10$\bar{1}$1	2.4	0.0	1.1	4.4	8.9	0.0
01$\bar{1}$2	1.6	5.3	10.1	15.5	21.0	1.6
10$\bar{1}$4	100	100	100	100	100	100
0006	4.7	2.3	1.0	0.3	0.0	4.7
01$\bar{1}$5	5.4	2.0	0.4	0.0	0.3	2.4
11$\bar{2}$0	11.3	14.0	16.4	18.6	20.7	11.3
11$\bar{2}$3	37	22.5	13.2	7.2	3.4	27.0
02$\bar{2}$1	4,3	1.9	0.6	0.1	0.0	2.3
20$\bar{2}$2	14.0	15.5	16.7	17.8	18.8	14.0
02$\bar{2}$4	4.4	5.6	6.6	7.6	8.5	4.4
01$\bar{1}$8	18.0	18.6	19.1	19.5	19.9	18.0
11$\bar{2}$6 } 0009 }	14.7	16.1	17.6	19.1	20.5	14.1
21$\bar{3}$1	3.6	4.8	5.9	7.0	8.0	5.8
12$\bar{3}$2	5.9	7.1	8.2	9.2	10.2	5.9

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Fig. 1. The first seven reflections from dolomite as a function of Zn substitution, omitting $10\bar{1}4$ (taken as standard = 100) and $11\bar{2}0$ which is insensitive. Intensities relative to $10\bar{1}4 = 100$

This is relatively simple. The effect on the intensities of a given Zn^{2+} substitution (x) is a function of $x(f_{Zn} - f_{Mg})$ where f_{Zn}, f_{Mg} are the atomic scattering factors of Zn^{2+} and Mg^{2+}. A substitution of Fe^{2+} to an extent x' would be the same function of $x'(f_{Fe} - f_{Mg})$. Thus the same effect on the intensities as is produced by $CaMg_{1-x}Zn_x(CO_3)_2$ would be produced by $CaMg_{1-x}Fe_x(CO_3)_2$ where

$$x'(f_{Fe} - f_{Mg}) = x(f_{Zn} - f_{Mg})$$

$$\text{i.e. } x' = \frac{x(f_{Zn} - f_{Mg})}{(f_{Fe} - f_{Mg})}$$

The expression involving the scattering factors varies only slightly with θ and is approximately 1.4. Thus virtually identical intensities would be given by dolomites of any composition $CaMg_{1-x}Zn_{x-r}Fe_r(CO_3)_2$, where $x - 0.3r$ is constant,

e.g. $x = 0.40$	$r = 0$	$CaMg_{0.60}Zn_{0.40}(CO_3)_2$
$x = 0.45$	$r = 0.17$	$CaMg_{0.55}Zn_{0.28}Fe_{0.17}(CO_3)_2$
$x = 0.49$	$r = 0.33$	$CaMg_{0.51}Zn_{0.16}Fe_{0.33}(CO_3)_2$
$x = 0.54$	$r = 0.50$	$CaMg_{0.46}Zn_{0.04}Fe_{0.50}(CO_3)_2$
$x = 0.56$	$r = 0.56$	$CaMg_{0.44}Fe_{0.56}(CO_3)_2$

and all these compositions would give the same calculated intensities.

The effect of disorder of Ca and (Mg, Zn) over the two cation positions is rather more complicated. For pure $CaMg(CO_3)_2$ it is fairly straightforward. Since

Ca^{2+} lies at the origin the trigonometric part of its structure factor contribution is constant for all reflections at +1. The Mg^{2+} lies at 0, 0, $\frac{1}{2}$, and its contribution is therefore also +1 for reflections with l even and −1 for reflections with l odd. Thus for l even it makes no difference whether the atoms are ordered or disordered over the two sites: the total contribution is $(f_{Ca} + f_{Mg})$. For l odd, if the occupancy of the site at the origin is $Ca_{1-y}Mg_y$, then the contribution is

$$(1-y)f_{Ca} + yf_{Mg} - yf_{Ca} - (1-y)f_{Mg} = (1-2y)(f_{Ca} - f_{Mg}).$$

The change in contribution as a result of the disorder is therefore $-2y(f_{Ca} - f_{Mg})$, and this can be compared with the corresponding effect of substitution of Zn^{2+} to the extent x, which is $-x(f_{Zn} - f_{Mg})$.

Thus a disorder of Ca and Mg to the extent y has the same effect on the absolute intensities of reflections with l odd as substitution of Zn^{2+} to the extent x where

$$y = \frac{x(f_{Zn} - f_{Mg})}{2(f_{Ca} - f_{Mg})}$$

Once again the coefficient of x can be taken as approximately independent of 2θ, with a value of approximately 1.4 (quite fortuitously the same as $\frac{(f_{Zn} - f_{Mg})}{(f_{Fe} - f_{Mg})}$).

The result is that, for example, the absolute intensities of reflections $hkil$ with l odd from $CaMg(CO_3)_2$ with a disorder parameter of 0.35 are the same as those from ordered $CaMg_{0.75}Zn_{0.25}(CO_3)_2$. However, this will not be quite true of their relative intensities, since the intensities of reflections of the latter compound are scaled to $I_{10\bar{1}4}$ (0.25 Zn) = 100 and the absolute value of this intensity is about 20% higher than $I_{10\bar{1}4}$ (pure dolomite). Nevertheless, since the reflections with l odd are substantially more sensitive to Zn content than those with l even, there is a closer correspondence between the intensities from disordered pure dolomite and ordered zincian dolomite than there is between those from ordered pure dolomite and ordered zincian dolomite, as may be seen from columns 2, 3 and 7 of Table 2.

When we consider the possibility of disorder in zincian dolomite a further complication arises. The effect already discussed for Ca, Mg disorder depends on its producing an increase in scattering power at the Mg site and a decrease at the Ca site. At a composition of around $CaMg_{0.65}Zn_{0.35}(CO_3)_2$ the scattering power at the Ca and (Mg, Zn) sites will be equal (strictly only at sin θ) $\lambda \sim 0.2$, but approximately so for a reasonable range of 2θ around 36° for $\lambda = 1.542$ Å); there will therefore be no effect on the intensities as a result of disorder. At higher Zn contents there will be a reversed effect. Thus, in general terms, at low Zn content disorder tends to simulate higher Zn content, and at high Zn contents it tends to simulate lower Zn content. Quantitatively the effect on the absolute intensities of the reflections with l odd will be

$$-2y(f_{Ca} - f_{Mg}) - z(1 - 2y)(f_{Zn} - f_{Mg})$$

where the disordered composition is $CaMg_{1-z}Zn_z(CO_3)_2$, and the corresponding effect of an ordered composition $CaMg_{1-x}Zn_x(CO_3)_2$ is $-x(f_{Zn} - f_{Mg})$. Hence it may be shown that the value of x that is simulated is given by

$$x = 0.7y + z(1 - 2y)$$

This function is tabulated in Table 3.

We therefore have a method which in principle should enable us to determine

Table 3

Values of x (the apparent Zn content deduced from Fig. 3) and y
as a function of z (the true Zn content deduced from Fig. 1) and y
(the disorder parameter)

y \ z	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
0	0	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90	1.00
0.1	0.07	0.15	0.23	0.31	0.39	0.47	0.55	0.63	0.71	0.79	0.87
0.2	0.14	0.20	0.26	0.32	0.38	0.44	0.50	0.56	0.62	0.68	0.74
0.3	0.21	0.25	0.29	0.33	0.37	0.41	0.45	0.49	0.53	0.57	0.61
0.4	0.28	0.30	0.32	0.34	0.36	0.38	0.40	0.42	0.44	0.46	0.48
0.5	0.35	0.35	0.35	0.35	0.35	0.35	0.35	0.35	0.35	0.35	0.35
0.6	0.42	0.40	0.38	0.36	0.34	0.32	0.30	0.28	0.26	0.24	0.22
0.7	0.49	0.45	0.41	0.37	0.33	0.29	0.25	0.21	0.17	0.13	0.09
0.8	0.56	0.50	0.44	0.38	0.32	0.26	0.20	0.14	0.08	0.02	−0.04
0.9	0.63	0.55	0.47	0.39	0.31	0.23	0.15	0.07	−0.01	−0.09	−0.17
1.0	0.70	0.60	0.50	0.40	0.30	0.20	0.10	0	−0.10	−0.20	−0.30

The negative values in the bottom right hand corner could of course only be obtained by extrapolating the curves in Fig. 2 to notional negative apparent Zn contents.

from the relative intensities of the reflections of a zincian dolomite both the zinc content and the degree of Ca, (Mg, Zn) disorder. From Fig. 1 we can take the variation of relative intensities of some reflections with l even as a function of Zn content. These are unaffected by disorder and therefore permit us to obtain independent estimates of Zn content from each reflection and to assess the best compromise solution, z. Reference to Fig. 2 then enable us to determine the appropriate ratio of the absolute value of $I_{10\bar{1}4}$ for this composition to that for pure dolomite. If the observed relative intensities of the reflections with l odd are multiplied by this factor one obtains their values on a scale for $I_{10\bar{1}4} = 100$ for pure dolomite. Reference to Fig. 3 then enables us to determine x, the apparent zinc content simulated by the odd reflections. By seeking this value of x in Table 3 in the column headed by the appropriate value of z then enables us to determine y.
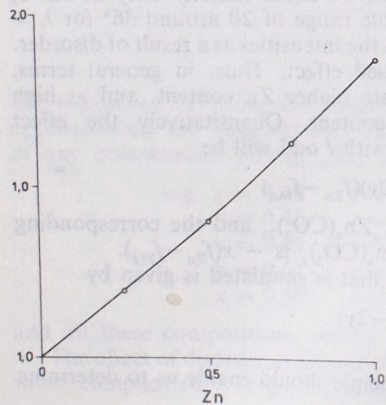
Fig. 2. Intensity of $10\bar{1}4$ of dolomite as a function of Zn substitution, relative to the intensity for pure $CaMg(CO_3)_2$
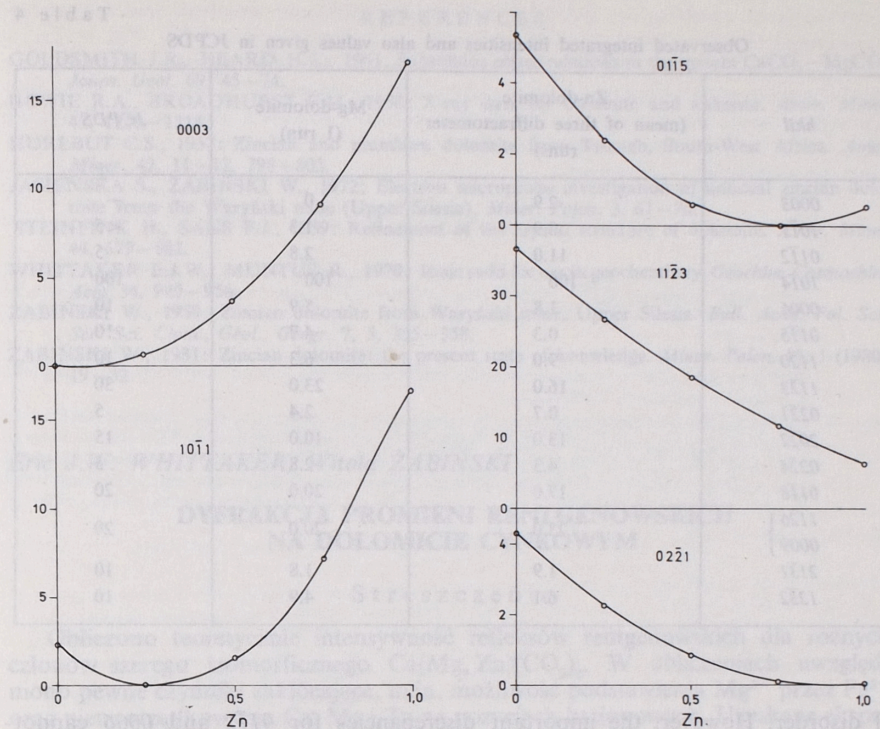

Fig. 3. Reflections from dolomite with l odd. Intensity relative to $10\bar{1}4$ from pure $CaMg(CO_3)_2$ in ordered conditions ($= 100$), as a function of Zn substitution. For a given material these values are obtained from intensities relative to their own $10\bar{1}4$ which are then multiplied by the appropriate value from Fig. 2

GENERAL DISCUSSION OF THE RESULTS

Integrated intensities for the zincian dolomite, a fairly pure dolomite, and the JCPDS data are presented in Table 4.

The match of the observed intensities from the zincian dolomite to the JCPDS is at first sight tolerable, especially as the latter are obviously only semi-quantitative, being rounded in general to the values 5, 10, 15, 20 and 30. The JCPDS value for $10\bar{1}1$ is of uncertain significance, and the omission of 0003 may be due to its low angle. One may discount systematic discrepancies at higher angles as due to incompatible applications of angular corrections. However, when all these allowances are made there are significant discrepancies for 0006, $01\bar{1}5$, $02\bar{2}1$. When the observed intensities are compared with the calculated ones for $CaMg(CO_3)_2$ these discrepancies remain, and others for 0003, $01\bar{1}2$ are emphasised. The discrepancies for the reflections with l odd are reduced when comparison is made with calculations for a disordered dolomite, and thus the abnormality of the zincian data would be less noticeable if the pattern were compared with one of a disordered dolomite. On an empirical basis it might then easily be discounted in view of the known variability of dolomite patterns as a result

Table 4

Observed integrated intensities and also values given in JCPDS

hkil	Zn-dolomite (mean of three diffractometer runs)	Mg-dolomite (1 run)	JCPDS
0003	2.9	0	—
10$\bar{1}$1	0.9	1.3	<5
01$\bar{1}$2	11.0	2.8	5
10$\bar{1}$4	100	100	100
0006	1.8	5.9	10
01$\bar{1}$5	0.3	4.7	10
11$\bar{2}$0	9.0	6.3	10
11$\bar{2}$3	16.0	23.0	30
02$\bar{2}$1	0.7	2.4	15
20$\bar{2}$2	13.0	10.0	5
02$\bar{2}$4	4.3	2.8	20
01$\bar{1}$8	17.0	20.0	20
11$\bar{2}$6 / 0009	23.0	20.0	20
21$\bar{3}$1	1.9	1.8	10
12$\bar{3}$2	6.1	4.9	10

of disorder. However, the important discrepancies for $01\bar{1}2$ and 0006 cannot be explained in this way.

Application of the theoretical methods developed above is not entirely straightforward however. Because of inevitable errors in intensity determination and the possible errors in the assumed structure, reflections can only be satisfactorily diagnostic if they vary by an order of magnitude as a function of Zn content. This is true for the first five reflections with l odd, but only for the first two with l even. With increasing angle the uncertainties become greater and greater, and observed values may even lie outside the range of calculated values at one end or other of the range. However if attention if confined to the even reflections $01\bar{1}2$ and 0006, and the odd reflections 0003, $10\bar{1}1$, $01\bar{1}5$, $11\bar{2}3$ and $02\bar{2}1$, tolerable results can be achieved.

The intensities for a fairly pure dolomite (containing 0.2% FeO by weight) are shown in Table 4 column 3. The even reflections give values of z 0.07 and -0.07 (extrapolated) with the mean of zero. The odd reflections give values of x of < 0.2, 0.07 (or 0.46), 0.05, 0.35, 0.23 (mean $\sim$ 0.15), which leads to a value of 0.2 for the disorder parameter y. The zincian dolomite (Table 4, column 2) gives values of z of 0.54 and 0.34 (mean 0.44) from the even reflections. At this value the method is relatively insensitive to disorder but the values of x are 0.52, 0.47, 0.52, 0.48, 0.48 (mean 0.49). The two values are not significantly different and since, at this value of z, disorder would lead to $x<y$. One can conclude that there is little if any disorder, any y must be taken as zero.

The data for dolomite presented by Howie and Broadhurst (1958) are not directly reconcilable with the present calculations but the relationship between their data for ankerite and dolomite is qualitatively similar to what is expected.

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DYFRAKCJA PROMIENI RENTGENOWSKICH NA DOLOMICIE CYNKOWYM

Streszczenie

Obliczono teoretycznie intensywność refleksów rentgenowskich dla różnych członów szeregu izomorficznego $Ca(Mg, Zn)(CO_3)_2$. W obliczeniach uwzględniono pewne czynniki zakłócające, m.in. możliwość podstawienia Mg^{2+} przez Fe^{2+} oraz nieuporządkowania Ca, Mg i Zn na pozycjach kationowych. Uzyskane eksperymentalnie wartości intensywności refleksów dolomitu cynkowego z kopalni Waryński (Górny Śląsk) wskazują na podstawienie Mg^{2+} przez Zn^{2+} zbliżone do 50%, co dość dobrze zgadza się z wynikami analizy chemicznej tego dolomitu.

OBJAŚNIENIA FIGUR

Fig. 1. Intensywność pierwszych siedmiu refleksów dolomitu jako funkcja stopnia podstawienia Mg^{2+} przez Zn^{2+}. Pominięto refleks $10\bar{1}4$ (przyjęty jako standard = 100) oraz refleks $11\bar{2}0$, który jest mało czuły. Intensywność podano względem $10\bar{1}4 = 100$
Fig. 2. Intensywność refleksu $10\bar{1}4$ dolomitu jako funkcja stopnia podstawienia Mg^{2+} przez Zn^{2+}, podana względem intensywności odpowiedniego refleksu czystego dolomitu $CaMg(CO_3)_2$
Fig. 3. Intensywność refleksów dolomitu o l nieparzystym, jako funkcja stopnia podstawienia Mg^{2+} przez Zn^{2+}. Intensywność podano względem $10\bar{1}4$ czystego dolomitu $CaMg(CO_3)_2$, o strukturze uporządkowanej (= 100). Dla danego ogniwa szeregu $Ca(Mg, Zn)(CO_3)_2$ wartości te otrzymano z intensywności względem ich własnego refleksu $10\bar{1}4$ mnożąc je następnie przez odpowiednie wartości wzięte z figury 2.

Эрик Д.У. УИТТАКЕР, Витольд ЖАБИНЬСКИ

ДИФРАКЦИЯ РЕНТГЕНОВСКИХ ЛУЧЕЙ НА ЦИНКОВОМ ДОЛОМИТЕ

Резюме

Проведено теоретическое вычисление интенсивности рентгеновских рефлексов разных членов изоморфного ряда $Ca(Mg, Zn)(CO_3)_2$. В расчерах

учитывались некоторые факторы, обусловливающие помехи, как например возможность замещения магния железом, неупорядочение Ca, Mg и Zn в катионных позициях. Полученные экспериментальным путем значения интенсивности рефлексов цинкового доломита из рудника Варыньски (Верхняя Силезия) свидетельствуют о замещении магния цинком в количестве близком 50%, что вполне удовлетворительно соответствует данным химического анализа указанного доломита.

ОБЪЯСНЕНИЯ К ФИГУРАМ

Фиг. 1. Интенсивность семи первых рефлексов доломита в зависимости от степени замещения магния (Mg^{2+}) цинком (Zn^{2+}). Пропущены рефлекс $10\bar{1}4$ (принятый в качестве стандарта = = 100) и рефлекс $11\bar{2}0$ слабо чувствительный. Интенсивность указана относительно $10\bar{1}4$ = 100

Фиг. 2. Интенсивность рефлекса $10\bar{1}4$ доломита в зависимости от степени замещения магния (Mg^{2+}) цинком (Zn^{2+}), указанная относительно интенсивности соответствующего рефлекса чистого доломита CaMg(CO$_3$)$_2$

Фиг. 3. Интенсивность рефлексов доломита с нечетным l в зависимости от степени замещения магния (Mg^{2+}) цинком (Zn^{2+}). Интенсивность показана относительно $10\bar{1}4$ чистого доломита CaMg(CO$_3$)$_2$ с упорядоченной структурой (= 100). В отношении данного члена ряда Ca(Mg, Zn)(CO$_3$)$_2$ указанные значения получены путем умножения интенсивностей по отношению к их собственному рефлексу $10\bar{1}4$ на соответствующие величины с фиг. 2.