

$$A_{12} = (L_{11}L_{12} + L_{21}L_{22})/3, \\ A_{11} = (L_{11}^2 + L_{21}^2)/3, A_{22} = (L_{12}^2 + L_{22}^2)/3.$$

Dabei ist z. B. L_{12} die Komponente von L_1 in Richtung x_2 . Liegen die Hauptachsen des Gestalttensors parallel zu x_1, x_2 , so ist $A_{12} = 0$, also

$$L_{11}L_{12} + L_{21}L_{22} = 0.$$

Führt man als neue Variable die Koordinaten ξ_1, ξ_2 eines Punktes ξ und η_1, η_2 eines Punktes η ein:

$$\xi_1 = L_{11}/\sqrt{3A_{11}}; \xi_2 = L_{12}/\sqrt{3A_{22}}; \eta_1 = L_{21}/\sqrt{3A_{11}}; \\ \eta_2 = L_{22}/\sqrt{3A_{22}},$$

so gelten für die vier Unbekannten $\xi_1, \xi_2, \eta_1, \eta_2$ die drei Gleichungen

$$\xi_1^2 + \eta_1^2 = 1; \xi_2^2 + \eta_2^2 = 1; \xi_1\xi_2 + \eta_1\eta_2 = 0.$$

Offensichtlich führt hier der Ansatz

$$\xi_1^2 + \xi_2^2 = 1; \eta_1^2 + \eta_2^2 = 1$$

zum Ziel, wobei die auf dem Einheitskreis liegenden Punkte ξ und η durch die Nebenbedingung $\xi_1\xi_2 + \eta_1\eta_2 = 0$ an einander gekoppelt sind. In Fig. 1 sind derartige zusammengehörige Punktpaare durch ●●

bezw. ○○ bzw. □□ gekennzeichnet. Sie entsprechen den auf einer punktiert eingetragenen Ellipse liegenden Endpunkten der bei $x = 0$ beginnenden Kantenvektoren L_1, L_2 und sind in Fig. 1 gleichfalls als Pfeile eingezeichnet, wobei die Pfeilspitzen durch punktierte Linien mit den entsprechenden Punkten ξ, η verbunden sind. Es ergibt sich hieraus das schon im Text erwähnte Resultat, dass alle Parallelogramme, die den gleichen Gestalttensor haben, mit allen vier Seiten eine Ellipse von aussen berühren müssen, deren Hauptachsendurchmesser gegeben sind durch $2/\sqrt{3}|T_1|$ und $2/\sqrt{3}|T_2|$.

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Acta Cryst. (1953). **6**, 413

The Structures of the Plagioclase Felspars. II

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(Received 27 August 1952 and in revised form 19 February 1953)

The atomic coordinates for body-centred anorthite (72%) have been refined by the method of least squares. The interatomic distances are recalculated and discussed. The results confirm that the fine-structure of body-centred anorthite is mainly due to a segregation of aluminium and silicon atoms into definite positions in the crystal lattice.

1. Introduction

The structure of albite ($\text{NaAlSi}_3\text{O}_8$), according to Taylor, Darbyshire & Strunz (1934), is essentially similar to that of orthoclase. The other component of the plagioclase series, anorthite ($\text{CaAl}_2\text{Si}_2\text{O}_8$), has also a similar structure, but weak subsidiary layer lines in X-ray oscillation photographs around the c axis indicate a duplication of the c axis length for that component. Chao & Taylor (1940) reported that X-ray oscillation photographs around the c axis of a plagioclase crystal of intermediate composition (60% An) show *pairs* of weak subsidiary layer lines, located symmetrically about the positions of the single ones in a corresponding anorthite pattern. Cole, Sörum & Taylor (1951, here referred to as paper I) have de-

scribed these subsidiary spectra in more detail. From oscillation photographs of a large number of plagioclase crystals of known composition, varying from nearly pure albite to nearly pure anorthite, they concluded that the ranges of true isomorphism at either end of the series extend to about 15% An and 85% An respectively, that an intermediate superstructure range exists between the limits 30% An to 72% An, whereas the remaining ranges from 15% to 30% An and from 72% to 85% An can be interpreted as disorder ranges. The results of preliminary heat-treatment experiments were discussed and related to recent optical investigations on high-temperature and low-temperature plagioclases (see for example Köhler, 1949).

A detailed investigation on the structure of the

intermediate plagioclases was carried out by Sörum (1951). First, an extension and adaptation of the optical models, originally suggested by Chao & Taylor (1940), led to a preliminary interpretation of the subsidiary spectra and the variation of their separations with chemical composition in terms of a structure corresponding to a regular intergrowth of domains with albite-like structure and domains with anorthite-like structure. Secondly, a Patterson synthesis, mainly based on the intensities of the subsidiary reflexions, led to a fairly detailed description of the superstructure for a plagioclase specimen of composition near 50% An, and to a determination of the structure for body-centred anorthite, i.e. for a specimen of composition near 72% An.

A refinement of the atomic coordinates obtained in this preliminary structure determination seemed highly desirable, as important and interesting conclusions are drawn on the basis of small differences in the bond-lengths for this latter structure. The present paper reports the results of a least-squares refinement of the atomic coordinates for 72% An.

2. Experimental

The previous works on the plagioclase structures have supported the point of view that the superstructure of the intermediate plagioclases consists of a regular intergrowth of domains with the albite structure and domains with the anorthite structure. The subsidiary reflexions in the X-ray photographs originate, therefore, mainly from differences in the atomic coordinates for the albite and the anorthite component. The lattice geometry of these superstructures, for varying compositions of the specimen, was outlined in Paper I, and it was shown (Sörum, 1951) that certain Patterson sections, calculated from the intensities of the subsidiary reflexions alone, are especially useful for the study of the details of the structure. Furthermore, it was shown that special interpretations can be ascribed to the different sections. Thus, one section may be attributed to the 'fine-structure' of the anorthite component, another section mainly to the albite component, while other sections have maxima corresponding to vectors between atoms of the albite and the anorthite component. The finer details of the structure of body-centred anorthite, with a com-

position corresponding to the end of the 'isomorphous' range (72% An), could thus be derived from the appropriate Patterson section calculated for a plagioclase specimen of composition near 50% An. The success of this structure determination provided final evidence for the existence of domains with anorthite-like structure in the superlattice of the intermediate plagioclases. This structure derivation was checked and to some extent improved by comparison of calculated and observed structure factors for a specimen of composition near 72% An. The structure obtained was of moderate accuracy, giving a value of the reliability index of 0.21 for the $hk0$ reflexions and 0.31 for some hundred subsidiary reflexions. The detailed procedure for the derivation of this structure has been given in the original paper (Sörum, 1951).

Complete intensity data for 72% An were not recorded, and the further refinement has, therefore, been carried out by the method of least squares (Hughes, 1941).

The structure of body-centred anorthite can be conceived as consisting of groups, slightly different, around $\{0, 0, 0\}$ and $\{0, 0, \frac{1}{2}\}$; each group comprising 8 silicon-oxygen tetrahedra and two cations. The structure factor can then be expressed as

$$F(hkl) = \sum_i 8f_i \cdot \cos 2\pi(hx_m + ky_m + lz_m) \cdot \cos 2\pi(hx_d + ky_d + lz_d),$$

where x_m etc. are the average parameters for the two groups, i.e. $x_m = \frac{1}{2}(x_2 + x_1)$ etc., and x_d etc. are the differences, $x_d = \frac{1}{2}(x_2 - x_1)$ etc., subscript 1 referring to the group around $\{0, 0, 0\}$ and subscript 2 to the group around $\{0, 0, \frac{1}{2}\}$. The quantities x_d and y_d are of the order of magnitude of a few degrees, whereas z_d differs a few degrees from 90° . The least-squares refinement could be carried out in successive stages. First, the x_m and the y_m coordinates could be refined separately from the $hk0$ data, as the $F(hk0)$'s depend very little, except for high orders, on the x_d 's and the y_d 's. Secondly, the structure factors for the subsidiary reflexions depend mainly on the coordinates x_d, y_d and z_d . For these coordinates, however, only a partial refinement was carried out for the y_d 's, together with some small adjustments in the x_d 's and the z_d 's.

The least-squares refinement improved the agreement between observed and calculated structure fac-

Table 1. Atomic coordinates of the body-centred anorthite structure for a specimen of composition near 72% An

These values are used for calculation of the structure factors in Table 2. Subscript 1 refers to the group around $\{0, 0, 0\}$ and subscript 2 refers to the group around $\{0, 0, \frac{1}{2}\}$. (All coordinates are given in degrees.)

	Ca	Si ₁	Si' ₁	Si ₂	Si' ₂	O _{A1}	O _{A2}	O _{B1}	O _{B2}	O _{C1}	O _{C2}	O _{D1}	O _{D2}
x_1	92	3.5	-5	-112	111.5	8	112.5	-69	63	9	-10	65.5	-50.3
y_1	1.9	60	62.5	39.8	44.2	42.8	2	45.8	49.5	110	112	34.5	41
z_1	21	36	-43	60	-64	-4	-45	36	-46	34	-50	67	-82
x_2	98	-0.5	1	-118	111.5	8	120.5	-69	73	3	-10	55.5	-46.3
y_2	-3.1	65	60.5	34.8	48.2	45.8	12	48.8	51.5	114	112	35.5	38
z_2	195	222	141	236	112	182	137	224	136	222	142	251	98

Table 2. *Observed and calculated structure factors for a plagioclase specimen of composition near 72% An (body-centred anorthite)*

The atomic coordinates quoted in Table 1 have been used for these calculations.

hkl	$2 \sin \theta$	$ F_o $	F_c	hkl	$2 \sin \theta$	$ F_o $	F_c	hkl	$2 \sin \theta$	$ F_o $	F_c
110	0.235	80	+96	660	1.470	50	+46	181	0.966	25	+31
110	0.240	—	+50	4,10,0	1.470	—	-17	181	0.970	—	-10
020	0.240	—	+22	2,12,0	1.473	89	+89	181	1.012	40	-41
130	0.400	157	-149	590	1.530	142	-122	251	0.708	31	-28
130	0.425	197	-168	680	1.542	26	-54	251	0.683	33	-32
200	0.430	—	-38	750	1.560	—	+15	251	0.736	24	+22
220	0.462	284	-302	1,13,0	1.570	—	-24	251	0.780	10	+14
040	0.473	296	-296	1,13,0	1.573	68	+76	271	0.922	21	-15
220	0.483	288	-297	680	1.590	—	-18	271	0.868	48	-47
150	0.616	65	-67	750	1.608	103	+73	271	0.950	18	+41
240	0.620	85	-43	5,11,0	1.645	87	+30	271	0.960	—	-5
310	0.628	87	+100	3,13,0	1.650	62	+13	291	1.140	35	-45
150	0.638	70	-43	770	1.660	56	+55	291	1.120	32	-33
240	0.650	130	-71	800	1.680	—	+29	291	1.150	21	-21
310	0.650	128	+134	0,14,0	1.680	74	-75	291	1.200	29	-28
060	0.711	195	+220	820	1.700	—	-3	361	0.942	38	+45
330	0.730	49	-45	6,10,0	1.710	—	-12	361	0.900	38	-60
170	0.752	153	+129	2,14,0	1.710	35	-58	361	0.980	—	+1
170	0.760	—	+38	820	1.715	94	-64	361	0.980	—	+10
260	0.808	124	+112	840	1.720	112	+97	381	1.135	20	-14
400	0.840	250	+237	770	1.722	69	+69	381	1.095	29	+21
260	0.850	141	+103	6,10,0	1.770	50	-23	381	1.143	75	-62
350	0.850	—	+36	840	1.770	105	+79	381	1.200	37	+67
420	0.876	67	-68	790	1.780	115	+76	3,10,1	1.340	25	-17
350	0.880	—	+17	860	1.788	116	+96	3,10,1	1.270	—	+3
440	0.950	—	+9	1,15,0	1.797	32	-36	3,10,1	1.400	37	+46
080	0.955	117	+126	1,15,0	1.822	38	-65	471	1.168	10	+32
370	1.024	63	+76	4,14,0	1.837	56	-72	471	1.140	—	-2
280	1.030	292	-318	790	1.854	78	-52	471	1.178	21	+14
280	1.062	195	-212	860	1.855	99	+132	471	1.250	33	-30
370	1.072	107	+102	910	1.875	49	+46	491	1.340	35	+35
460	1.075	189	+212	910	1.880	—	+23	491	1.305	46	+56
190	1.085	53	-27	4,14,0	1.880	—	+50	491	1.424	13	-15
530	1.090	95	-95	880	1.890	87	-100	561	1.240	33	-40
190	1.100	172	-148	930	1.892	113	-93	561	1.200	24	-23
460	1.122	241	+253	5,13,0	1.900	—	+39	561	1.280	29	+40
530	1.123	123	-124	5,13,0	1.910	40	+30	561	1.310	—	-1
0,10,0	1.195	107	+113	0,16,0	1.912	31	+78	581	1.400	25	-21
390	1.220	42	-30	930	1.930	98	-99	581	1.350	19	+25
480	1.242	42	+32	6,12,0	1.942	32	-31	581	1.445	17	+23
390	1.255	—	-27					581	1.460	21	-25
2,10,0	1.256	77	-108	011	0.130	—	+2	5,10,1	1.578	19	-23
600	1.258	226	+234	011	0.130	—	-7	5,10,1	1.542	—	+7
2,10,0	1.280	—	-16	031	0.353	32	-24	5,10,1	1.611	37	+48
620	1.285	148	-141	031	0.361	25	+20	5,10,1	1.660	24	-24
480	1.306	114	-116	051	0.582	27	-23	651	1.350	—	+15
570	1.310	80	-53	051	0.582	—	-6	651	1.465	27	-28
640	1.320	155	-170	071	0.830	23	+22	671	1.490	—	+2
1,11,0	1.332	—	+10	071	0.845	34	+35	671	1.450	18	-21
1,11,0	1.335	46	+54	141	0.500	26	-32	671	1.520	—	+12
640	1.360	218	-225	141	0.492	37	+37	671	1.565	29	+38
570	1.375	48	+55	141	0.518	23	+21	691	1.642	21	-22
660	1.430	—	+13	141	0.550	—	-11	691	1.605	—	-8
4,10,0	1.430	49	+84	161	0.722	21	-28	691	1.654	46	-42
0,12,0	1.430	41	+43	161	0.722	—	+4	691	1.734	45	+60
3,11,0	1.432	31	+31	161	0.745	42	+27	761	1.606	—	-3
710	1.460	104	+109	161	0.776	29	-28	761	1.555	26	-34
590	1.465	126	-140	181	0.960	50	+35	761	1.638	36	-39
710	1.470	85	+76								

tors considerably. Thus, the index of reliability comes out as 0.15 for 78 observed $hk0$ reflexions, 0.22 for 57 observed subsidiary reflexions and 0.16 for all these reflexions together. At the same time the agreement for 21 $hk0$ reflexions and 18 subsidiary reflexions, being too weak to be observed, was appreciably improved.

The atomic coordinates obtained are set out in Table 1, while observed and calculated structure factors for 99 $hk0$ reflexions and 75 subsidiary reflexions are listed in Table 2. The details of the structure for body-centred anorthite is discussed in the section below.

Table 3. *Interatomic distances for the body-centred anorthite phase (72% An, 28% Ab)*

The data in this table are calculated from the atomic coordinates quoted in Table 1, and by using the lattice constants of pure anorthite. (All distances are given in Ångström units.)

Group around	Central atom	O _{A1}	O _{A2}	O _{B1}	O _{B2}	O _{C1}	O _{C2}	O _{D1}	O _{D2}	Average values
{0, 0, 0}	Si ₁	1.70	—	1.71	—	1.80	—	1.68	—	1.72 ₂
	Si ₁ '	1.65	—	—	1.75	—	1.77	—	1.66	1.70 ₇
	Si ₂	—	1.56	1.64	—	—	1.63	—	1.60	1.60 ₇
	Si ₂ '	—	1.69	—	1.62	1.54	—	1.67	—	1.63 ₀
{0, 0, ½}	Si ₁	1.76	—	1.68	—	1.75	—	1.72	—	1.72 ₇
	Si ₁ '	1.67	—	—	1.72	—	1.82	—	1.70	1.72 ₇
	Si ₂	—	1.68	1.54	—	—	1.65	—	1.68	1.63 ₇
	Si ₂ '	—	1.65	—	1.62	1.55	—	1.67	—	1.62 ₂
{0, 0, 0}	Ca	{ 2.34 2.56 }	2.65	2.58	(3.06)	2.98	(3.03)	2.41	(3.39)	2.59
{0, 0, ½}	Ca	{ 2.61 2.63 }	2.63	2.59	(2.99)	2.88	2.91	(3.05)	(3.59)	2.70

3. Discussion of the structures

Most of the information obtained in the present work is concerned with the structure of the body-centred anorthite phase, and the discussion of this structure is, therefore, of primary interest here. It was pointed out in the original paper, that there is a difference in the average bond lengths from the Si₁, Si₁' positions and the Si₂, Si₂' positions. Thus, for the former an average bond length of 1.70 Å and for the latter of 1.60 Å were found. It was also suggested that this difference in bond lengths may be due to segregation of Al atoms into the Si₁, Si₁' positions and Si atoms into the Si₂, Si₂' positions. Changes in the positions of the cations and in lattice constants were also shown to be consistent with this point of view, though the chains of tetrahedra in the direction of the *c* axis are considerably zigzagged in anorthite, while practically straight in the albite structure.

The interatomic distances have been recalculated with the new atomic coordinates obtained by least-squares refinement and listed in Table 1 above. The new values for the distances are quoted in Table 3. It may be seen from this table that the refinement has not led to any great alterations in these values, though a slight increase in the average values may be noted. Thus for the Si₁, Si₁' positions the mean values for four different tetrahedra are 1.72₂, 1.70₇, 1.72₇, and 1.72₇ Å, and for sixteen bonds from these positions 1.72₁ Å. The corresponding values for the Si₂, Si₂' positions are 1.60₇, 1.63₀, 1.63₇, 1.62₂ and 1.62₄ Å respectively. These average values for the bond-lengths are in good agreement with earlier observations. Thus, Hey & Taylor (1931) found 1.72 Å for the Al-O distance in sillimanite, and a survey of the literature indicates that 1.62 Å would be the best value for the Si-O distance in silicates (see, for example, Cole, Sörum & Kennard, 1949). The limits of error in the values, obtained here, should not exceed 0.02 Å, and it appears safe to conclude that

the difference in the bond lengths for the Si₁ and the Si₂ positions is real.

Thus it is found that the results of the least-squares refinement confirm the earlier findings, and add certainty to the conclusion that the 'fine-structure' of the body-centred anorthite is mainly due to a segregation of the aluminium atoms and the silicon atoms into definite positions of the crystal lattice.

The distances from the cation to the nine nearest oxygen atoms for the two groups are given in Table 3. For the group around {0, 0, 0} the values are spread from 2.34 to 3.39 Å, with an average value of 2.59 Å for the six nearest neighbours, and three more neighbours at 3.03, 3.06 and 3.39 Å. For the group around {0, 0, ½} the values are more nearly equal, and slightly longer, though not significantly, having an average value for the six nearest neighbours of 2.70 Å, and three other oxygens at distances 2.99, 3.05 and 3.59 Å. With the exception of the Ca-O_{A1} and the Ca-O_{D1} distances (2.34 and 2.41 Å), all these distances are considerably longer than the sum of the Goldschmidt radii (2.40 Å) and also much larger than the Ca-O distances (2.38 Å) found, for example, in awillite (Megaw, 1952). This may be partly due to the coordination for the oxygens and partly to the somewhat irregular shape of the oxygen polyhedra around the cations.

The details of the structure for body-centred anorthite (72% An) must also be valid for the anorthite component in the superlattice of the intermediate plagioclases, since these details were actually derived from a Patterson section for a specimen of composition near 50% An. It is not unlikely that a similar sort of segregation exists in the albite structure, but the information obtained on this component in the present work is not accurate enough to permit any conclusions on this point. Such an accurate knowledge of the albite structure would be required for a detailed discussion of the intermediate type of structure.

The writer wishes to express his most sincere thanks to Prof. Sir W. L. Bragg and Dr W. H. Taylor for suggestions and interest in this work, which was begun in the Crystallographic Laboratory, Cavendish Laboratory, Cambridge, while he had the benefit of a British Council Scholarship. He is indebted to Norges Tekniske Högskoles Fond and to Norges Almenvitenskapelige Forskningsråd for financial aid.

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Acta Cryst. (1953). **6**, 417

On the X-ray Diffraction Patterns of Polymer Films

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(Received 23 August 1952)

Equations are derived which relate the angular spread of arcs in X-ray diagrams of oriented polymer films to the dispersion of the crystallites. A simple graphical method of measuring the dispersion is indicated, and the use of the equations in indexing reflexions and in checking the self-consistency of the interpretation of the X-ray diagram is pointed out.

1. Introduction

Polymer films usually show some degree of uniplanar orientation, i.e. the molecules tend to lie with their fibre axes in the planes of the films. In some cases stretched films have high uniplanar orientation, and also considerable orientation about the direction of stretching; such a film, when examined by X-rays with the beam parallel to the plane of the film, frequently gives a diffraction photograph in which the apparent orientation is better than can be readily obtained with fibre specimens of the same polymer. It is clear, however, that these X-ray photographs differ in several respects from rotation or fibre photographs, and a geometrical investigation is necessary before they can be fully interpreted. Such an investigation is attempted here. The angular position of a reflexion with reference to the equator of the diagram is first calculated for a single crystallite in terms of parameters which define the orientation of the crystallite and the polymer film. The reflexions in an X-ray photograph of a film or fibre are in general not spots, but are drawn out into arcs as a result of the imperfect orientation of the crystallites. We derive equations connecting the angular spread of these arcs with the dispersion of orientation.

The equations obtained may be used to calculate the dispersion of the crystallites from measurements of the lengths of equatorial arcs, and also to deduce

ξ and ζ values from the reflexions. Comparison of the measured lengths of the various arcs with the values calculated from the dispersion gives a useful check on the self-consistency of the interpretation of the X-ray diagram, and may enable a decision to be made as to whether all the reflexions originate from the same crystalline phase.

Throughout this paper we shall assume that a flat photographic plate, perpendicular to the primary beam is used. The results could easily be extended to cylindrical or conical films.

2. Specification of orientation

Consider a plane through the 'fibre axis' of any crystallite, perpendicular to the polymer film. The angle φ between this plane and the direction of orientation (i.e. of rolling or stretching) is defined as the 'in plane' disorientation of the particular crystallite. The 'out of plane' disorientation, ψ , is the angle between the plane of the film, and a plane through the fibre axis which intersects the film in a straight line perpendicular to the direction of orientation. In this way φ , ψ are defined symmetrically, and equations derived for the diagram obtained with the beam passing through the edge of the film may be transformed into relations which hold for the beam normal to the film by interchanging φ and ψ (with an appropriate change of sign). To define the signs of φ and ψ we consider the