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W. F. Müller, H. R. Wenk and G. Thomas

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STRUCTURAL VARIATIONS IN ANORTHITES

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ABSTRACT

Variations of structure and optical properties in anorthites (An 93-97%) of different origin are analyzed with the petrographic microscope, U-stage methods, X-ray single crystal analysis and high voltage electron microscopy. No significant variation has been found in the orientation of the indicatrix and of the lattice constants. But c -type reflections ($h + k$ even, l odd) are strong and sharp in anorthites from slowly cooled rocks and diffuse in anorthites of identical chemical composition from quenched volcanic rocks. Large type c -antiphase domains (5000-10000 \AA) are found in the slowly cooled rocks, c -domains in volcanic rocks are small (100 \AA) or could not be imaged. The presence of only b-domains in lunar basalt 14310 indicates quenching of this rock. Large c -domains in the Apollo 15 genesis rock (15415, Lally et al. 1972) indicate slow cooling similar to terrestrial metamorphic rocks.

INTRODUCTION

Of all feldspars calcic plagioclase has been least studied and many of its structural properties are still unclear. Smith and Ribbe (1969) give a summary of the present knowledge on the plagioclase structures and for all details we refer to that paper. In pure anorthite the Al-Si distribution in the tetrahedral framework is essentially ordered (Kempster et al. 1962, McGaw et al. 1962) in agreement with the

aluminum-avoidance principle (Loewenstein, 1954). This order persists up to high temperatures (Laves and Goldsmith, 1955). The ordering of Al-Si causes a doubling of the c-axis which is expressed in the appearance of additional reflections in the diffraction pattern.

There are four classes of reflections (Fig. 2)

Type-a - reflections ($h + k$ even, l even) are due to the basic feldspar structure. They are present in anorthite and become increasingly diffuse with higher Al-content and disappear around An 70%. Bytownites show only these a- and b- reflections. The corresponding structure is named body-centered-anorthite.

Type-c - reflections ($h + k$ even, l odd) and d - reflections ($h + k$ odd, l even) occur in anorthite only. They are sharp in slowly cooled crystals with An 95% (primitive anorthite) and diffuse with streaking in quenched and Na-rich anorthite (transitional anorthite). Upon heating the c - reflections of primitive anorthite become more diffuse and disappear below 350°C. These changes occur immediately and are reversible (Brown et al. 1963, Bruno and Gazzoni 1967, Foit and Peacor 1967, Laves, Czank and Schulz 1969). Therefore they are interpreted as caused by a displacive phase change and not a diffusive transformation (Laves and Goldsmith 1961, Megaw 1962). The structure determination of primitive anorthite (Kempster et al. 1962) in fact suggests that there are two statistically overlapping Ca-positions or that there is strong anisotropic thermal vibration of the Ca-atom in the tetrahedral framework which produces the primitive $c = 14\text{\AA}$ unit cell. At lower anorthite content order/disorder of Si/Al causes distortions of the lattice and small amounts of disorder may be important in the

nucleation of primitive anorthite domains (Smith and Ribbe 1969). The Si-content (and not the Ca-content as has been suggested by Lally et al. 1972) appears to have the most important influence on the structure in sodium bearing anorthite. The purpose of this study was to investigate structural variations in anorthites of different origin. Crystals of similar chemical composition and a different thermal history are characterized by their optical properties, their crystal structure and their microstructure. Since the pioneering work of McConnell and Fleet (1963), Nissen and Bollmann (1966), Ribbe (1962), electron microscopy has become a very powerful tool in the study of feldspars. We applied this method to anorthites (with the special aim to make transmission electron microscopy more popular in mineralogy and petrology). The present contribution is only concerned with natural crystals. In a second stage we will try to reproduce the observed structures in the laboratory.

PETROGRAPHY

The only thing which the four specimens chosen for this analysis have in common is that they all contain plagioclase with an anorthite content ranging between 93 and 97%. Locations, textures, origin, age and mineralogical composition are about as different as can be (Fig. 2).

The first specimen coming from the Fra Mauro area on the moon (Apollo 14, specimen 14310) is an ilmenite bearing anorthite rich ortho-clinopyroxene basalt of subophite texture (Fig. 2a, cf. Wenk et al. 1972).

Another volcanic rock is an anorthite-pigeonite tuff, very rich in euhedral anorthite crystals and two glass phases from Miyake Island,

Tokyo Bay, Honshu, Japan (Fig. 2b 16707 cf. Leisen 1934, Kôzu 1914).

302 is a eucrite meteorite from Serra Mage, Brasil (Ulbrich, 1971). The mineralogical composition is granular anorthite and pigeonite with "myrmekitic" exsolutions of augite (Fig. 2c).

Sci 59 is a metamorphic calcsilicate rock of miocene age from V. Schiesone (Bergell Alps, N. Italy, cf. Wenk 1970). The slightly foliated specimen consisting of diopside, hornblende, anorthite, calcite, sphene and ore bearing shows typical annealing textures (Fig. 2d) and is one of the slowly cooled amphibolite facies rocks of the leontine zone (E. Wenk, 1962).

The optical properties of plagioclase, especially the orientation and shape of the indicatrix in the triclinic crystal, vary greatly with the chemical composition and thermal history. Euler I angles are used to describe the orientation of the indicatrix. For sodium rich plagioclase data points of these angles fall on two distinctly different curves for volcanic and plutonic plagioclase (Burri, Parker, Wenk, 1968). These curves join at An 90% and from there on to pure anorthite a broad band of irregularly scattering points characterize the orientation (Fig. 1). The band for ψ and ϕ is about 18 degrees wide which is well beyond statistical scatter due to errors in the measurements. Euler I angles for the four specimens studies in this paper are listed in Table 1. There is no significant difference between the two quenched volcanic anorthites and the slowly cooled meteoritic and metamorphic crystals. Thus the orientation of the indicatrix, which is a convenient parameter to describe sodium rich plagioclase,

is unsuited in the anorthite range and other parameters have to be found. Such a parameter as has been mentioned in the introduction is the crystalstructure expressed in the absence, presence or diffuseness of b and c - reflections. X-ray precession photographs (Fig. 2) show for the volcanic samples (Fig. 2e,g) a transitional anorthite pattern with sharp b and c reflections which are streaking approximately parallel to b^* in $Ok1$ photographs and for the slowly cooled samples (Fig. 2g,h) they show a primitive anorthite structure with strong and sharp c - reflections. There is a clear variation in diffuseness of c - reflections of chemically identical anorthites and comparison of the lunar 14310 crystal with the anorthite in the Japanese volcanic tuff indicates that the crystalstructure of both crystals is very similar. Lattice parameters, taken from precession photographs do not show significant variation (Table 2).

TRANSMISSION ELECTRON MICROSCOPY

A Hitachi HU650 electron microscope with 650 kV acceleration voltage was used for the electron microscopic studies. Thinned specimens suitable for electron transmission were obtained from standard petrographic thin sections by ion-bombardment (Castaing, 1955; for experimental details see Barber, 1970, and Radcliffe et al., 1970). Due to the increased penetrating power of the high voltage electron microscope compared to a 100 kV electron microscope and to the preparation method large areas could be examined.

It may be useful to discuss briefly some basic principles of order-disorder transformations, antiphase domain boundaries and transmission

electron microscopy. For details the reader is referred to review articles by Marcincowski (1963), Cohen (1970) with a discussion by Warlimont (1970), and to textbooks on transmission electron microscopy (Thomas 1962; Amelinckx, 1964; Hirsch et al., 1965). We consider an alloy with 50% atoms A and 50% atoms B crystallizing in a structure in which the atomic positions are randomly occupied by the atoms A and B. If the alloy cools below a critical temperature, ordering of the atoms may take place. In the ordered structure, called a superstructure of the disordered one, all atoms A have atoms B as their closest neighbors and vice versa. Ordered domains may nucleate and begin to grow at different places within the same disordered crystal. When these ordered domains impinge on each other they may either fit perfectly together forming a single crystal or they may meet each other "out of phase" or "out of step" (Fig. 4). In this case a boundary called an antiphase domain boundary (APB) is produced between the domains. The region enclosed by an APB is called an antiphase domain. The antiphase vector \bar{p} describes the displacement of a particular atom species between two adjacent domains. Thus \bar{p} is the displacement necessary to produce the two domains from a single crystal. Depending on crystal structure there are many possibilities for such vectors. When electrons characterized by a distinct wavelength λ encounter a crystalline specimen, part of them will be diffracted on suitable oriented lattice planes under the corresponding Bragg angles ϕ_i . Electron diffraction patterns (3a-d) contain the primary beam and diffracted beams indexed h_i, k_i, ℓ_i corresponding to the reciprocal lattice vectors \bar{g}_i . Image contrast is obtained using an

an aperture by which a distinct beam can be selected to pass through to the final image. The other beams are withheld. If only the primary beam is allowed to pass a so-called bright field image results. If a diffracted beam is selected for image formation a dark field image is obtained. In dark field images the parts of the specimen will shine up from which (sufficient) electrons have been scattered into the corresponding Bragg reflection. Therefore, taking a superstructure reflection for imaging the regions of the specimen will appear bright which contain the superstructure. If the crystal contains APB's they may be resolved under certain conditions. Image contrast from APB's depends on several parameters the most important being the phase change associated with the displacement across the boundary. This phase change is given by the phase angle $\alpha = 2\pi \bar{g} \cdot \bar{p}$. A phase jump in the amplitude occurs if $\alpha \neq 0 \text{ mod } 2\pi$, i.e. for $\bar{g} \cdot \bar{p}$ non-integral. If $\bar{g} \cdot \bar{p}$ is integral, i.e. when \bar{p} equals a lattice translation vector, α is zero (no contrast). Thus, in general, superlattice diffraction vectors are necessary to provide contrast. If $\alpha = \pi \text{ mod } 2\pi$, symmetrical fringes occur about the center of the fault in dark field whereas when $\alpha = 2\pi/3 \text{ mod } 2\pi$ symmetrical fringes occur in bright field. However, other factors affect the fringe symmetry, e.g. when the diffraction vectors differ in magnitude but are parallel across the domain wall symmetry properties disappear due to the "excitation error" $\Delta \bar{g}$. This means that the direction of the \bar{p} vector is not easily determined from fringe symmetry but requires careful tilting experiments in order to investigate the same boundary under different reflecting conditions $\{\bar{g}_i \cdot \bar{p}_i\}$.

Keeping these principles in mind the anorthite specimens have been analyzed and the following features have been observed:

Anorthite from Lunar Basalt 14310: A typical selected area electron diffraction pattern containing sharp a- and b- reflections and diffuse reflections is shown in Fig. 3a. Very weak and diffuse d- reflections were observed. The c- reflections were streaked in directions perpendicular to about $(2\bar{3}1)$ in a selected area diffractogram normal $[211]$. This is in agreement with observations by Ribbe and Colville (1968), Appleman et al. (1971), and Christie et al. (1971).

Dark field images using b- reflections revealed smoothly curved antiphase domain boundaries (Fig. 3e). The size of these domains (500-1000Å) is distinctly smaller than those found by Christie et al. (1971) in lunar rock 10029. Christie et al. (1971) proposed a displacement vector $\bar{p} = \frac{c}{2}[001]$. So far, no contrast experiments and calculations have been done to determine the actual displacement vector among the many possibilities.

Anorthite from Miyake Islands, Japan: Selected area electron diffraction pattern show sharp a- and b and slightly diffuse c- reflections. Small domains in the order of 70 - 100Å could be imaged in dark field using type c- reflections (Fig. 3f). The c- domains in the Japanese anorthite may be ordered domains in a disordered matrix. The type c- reflections are less diffuse than those of the lunar anorthite. So we conclude that the c- domains in the lunar anorthite were not resolved because they were smaller and probably less ordered than in this anorthite. The occurrence of such small type c- domains in lunar rocks was reported

by Appleman et al. (1971) and Christie et al. (1971). Type b- domains were not observed in the anorthite from Japan.

Anorthite from the Eucrite Meteorite Serra de Maga: Selected area diffraction patterns show strong a- and b- and weaker c- reflections. The type c- reflections are sometimes slightly diffuse. APB's were visualized in bright field and dark field images using c- reflections (Fig. 3g). The width of mesh of the network formed by the APB's was varying from about 500 to 5000 Å. The domains frequently appear to be elongated parallel to c^* . During the work at the microscope an oscillation of the domain walls has been observed on the screen.

Type b- domains were not observed.

Anorthite from Val Schiesone, Alps: Selected area electron diffraction patterns showed strong a- reflections, weaker b- and c- reflections, very weak d- reflections. All reflections were sharp (Fig. 3d).

Bright field and dark field images with c- reflections operating revealed large antiphase domains separated by APB's (Fig. 3h). The areas surrounded by APB's as measured in the electron micrographs had a diameter up to several microns. The APB's have apparently a preferential orientation parallel to c^* . Ribbe and Colville (1968) assume an antiphase vector $\bar{p} = \frac{1}{2}[\bar{a} + \bar{b} - \bar{c}]$ for the c- domains. For this \bar{p} the phase shift α equals $\pi \text{ mod } 2\pi$ if the diffracted beam \bar{g} is a c- reflection. We observed symmetrical fringe contrast about the center of the APB-fault in dark field. This result would be consistent with the case for the condition $\alpha = \pi$, e.g. it would be consistent with a displacement vector $\bar{p} = \frac{1}{2}[\bar{a} + \bar{b} - \bar{c}]$. But, as

noted before for the b- domains, there are also other possibilities which have to be examined by contrast experiments and calculations.

CONCLUSIONS

Presence and size of b- and c- antiphase domains in anorthite is a sensitive parameter to characterize calcic plagioclase. The variations in size are influenced by the chemical composition and the thermal and mechanical history. If the An content is known then it is possible to derive the cooling history as is demonstrated by the comparison of the four samples. The size of the domains is a function of nucleation and growth rate. During quenching the domains cannot grow and a pattern of very fine c- domains results which may be partially disordered, causing the diffuse c- reflections. During annealing slightly below the disordering temperature the domains grow. In slowly cooled anorthites (An > 95%) no b- domains were present. It is uncertain if the intensity of c- reflections and shape and size of c- domains is only due to the Ca- position or if Si/Al disorder produced at very high temperatures in the lava has a secondary influence. The c- domains (probably caused by a displacive transformation around 300°C) may reflect only the very late cooling history whereas b- domains (an indication for Al/Si disorder) form already at high temperatures above ~800°C therefore influenced during all stages of the cooling. Heating experiments and microscopy will solve some of these questions. So far we can only empirically use the size of c- domains to obtain information about the cooling history. Lally et al. (1972) recently found large c- domains in an An 93-95 anorthite from the Apollo 15 genesis rock (15415)

and attribute it to the high Ca- content. From our evidence we conclude that the genesis rock has to be a slowly cooled rock, similar to terrestrial metamorphic rocks or meteorites. This is very different from most other lunar rocks, such as 14310-basalt where the antiphase domains indicate rapidly cooling of a volcanic rock. The differences between the two volcanic and the two plutonic specimens (missing of b- domains in the Japanese tuff compared with missing c- domains in the lunar basalt and differences in size in meteoritic and metamorphic anorthites) may be partly caused by small variations in chemical composition.

The presence of submicroscopic structures in all plagioclase crystals raises the intriguing question how these features which are invisible in the light microscope are expressed in the optical properties. Antiphase domains, submicroscopical twins, exsolutions are likely to have some imprint on the orientation of the indicatrix and the fact that the optical state and the structural state not always conform (Wenk, 1968, Wenk and Nord 1971) can possibly be explained as an influence of the microstructure.

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TABLE I

Euler I angles relating the optical indicatrix and crystal coordinates.

Accuracy of Euler angles is $\pm \frac{1}{2}^\circ$.

specimen	twin-laws	$2V_\gamma$	ϕ	ψ	ϕ	An	Or	Ab
			in degrees			in mole percent		
14310 lunar plag. pyrox. basalt	Ab,Ca,Ab-Ca Pe (rare) Baveno right		$25-\frac{1}{2}$	-2	38	85.3 ₀	1.72	12.9 ₈
			25	-4	38	87.6 ₉	1.22	11.0 ₉
		81	$23\frac{3}{4}$	$-5\frac{1}{2}$	38	87.6 ₀	1.38	11.0 ₂
		77	22	$-5\frac{1}{2}$	37	97.0 ₉	0.48	6.4 ₃
		78	24	-6	34	94.3 ₃	0.39	5.2 ₈
			$23\frac{1}{2}$	-6	38	93.4 ₁	0.45	6.1 ₄
volcanic tuff, Miyake Is. Japan	Ab,Ca Ab-Ca		$20\frac{1}{4}$	-10	34	94.3 ₀	.06	5.1 ₀
			21	-9	34	95.6 ₄	.07	4.2 ₉
			16	$-11\frac{1}{2}$	34	94.5 ₂	.01	5.4 ₇
			20	$-8\frac{1}{2}$	$36\frac{1}{2}$	94.9 ₉	.09	4.9 ₂
			$20\frac{1}{2}$	-8	37	93.9 ₉	.06	5.9 ₅
			23	-6	35	---	---	---
Serra de Magè eucrite meteorite	Ab,Ca, Ab-Ca Pe		20	$-8\frac{1}{2}$	$37\frac{1}{2}$	94.8 ₃	.09	5.0 ₈
		77	23	-6	38	95.3 ₇	.04	4.5 ₉
Sci. 59 diops. calc. anorth hbl. fels (rare) V. Schiesone, (Alps)	An,Pe, Ab-Ca	77	15	-8	38	97.4	.01	2.5 ₉

Ab. albite
Ca Carlsbad
Ab-Ca albite-Carlsbad
Pe pericline

TABLE 2 Lattice Constants and Structure of Anorthites (from Precession Photographs).

specimen	$\frac{a_0}{\text{in } \text{Å}}$	$\frac{b_0}{\text{error is } \pm .01 \text{Å}}$	$\frac{c_0}{\text{in degrees, error is } \pm 0.1^\circ}$	α	β	γ	structure	An	Or	Ab
								in mole percent		
14310 lunar plag. pyrox. basalt	8.18	12.87	14.19	93.3	116.2	91.0	transitional	93.4 ₄	0.16	6.4 ₀
volcanic tuff Miyake Isl. Japan	8.17	12.87	14.17	93.2	115.8	91.2	transitional	95.4 ₄	.02	4.5 ₄
Serra de Magè Eucrite Meteorite	8.19	12.88	14.18	92.9	116.1	91.4	primitive	95.5 ₇	.05	4.3 ₉
Sci 59 diops. hbl. calc. anorth. fels. V. Schiesone, (Alps.)	8.18	12.89	14.19	93.3	116.0	91.1	primitive	97.1 ₆	.25	2.5 ₉

FIGURE CAPTIONS

Fig. 1. Variation of Euler angles ϕ and ψ which characterize the orientation of the optical indicatrix in the crystal for calcic anorthites (data are from the literature).

Fig. 2. (a) -- (d) Photomicrographs using a petrographic microscope with crossed nicols.

(c) - (h) X-ray single-crystal precession -a photographs (Mo radiation, Zr filter).

Fig. 3. (a) - (d): Selected area electron diffractograms. (a) - (c) 650 kV, (d) 500 kV acceleration voltage.

(e) - (h): Transmission electron micrographs. (e) Type b, (f) - (h) type c- domains. Dark field. The operating diffracted beam \bar{g} is inserted. 650 kV acceleration voltage.

Fig. 4. Schematic representation of an antiphase domain boundary (APB) in an hypothetical alloy with atoms A (circles) and B (dots). \bar{p} is called the displacement vector or antiphase vector.

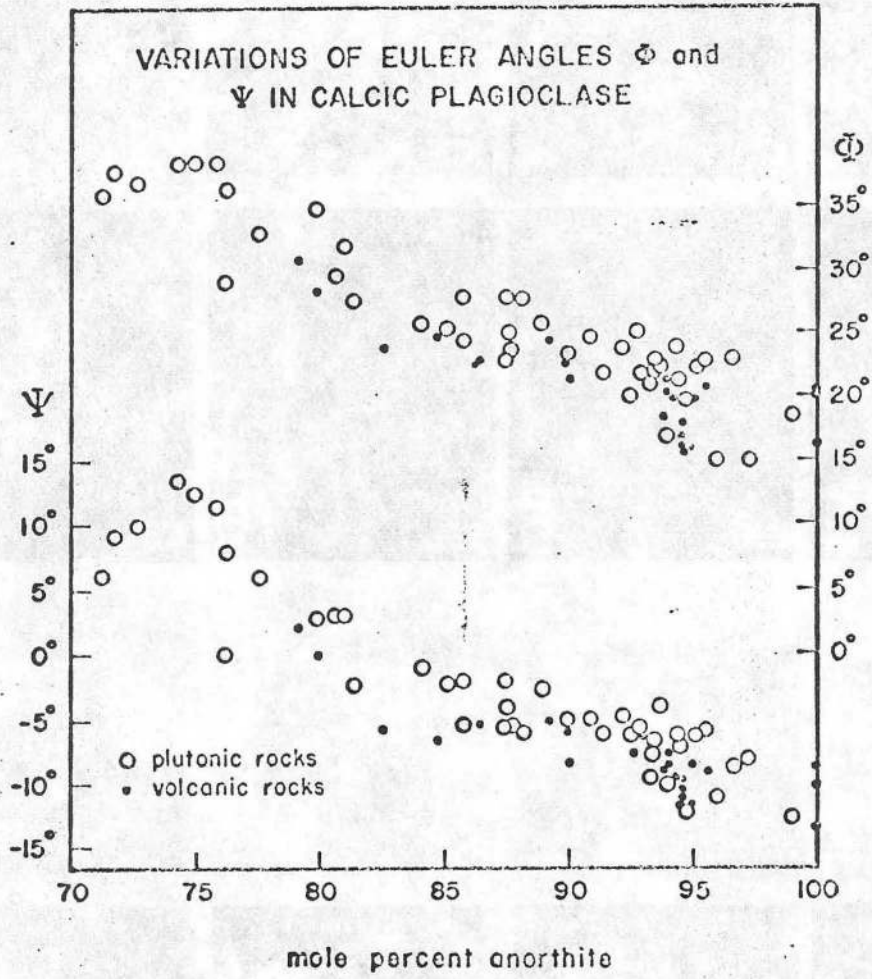


Fig. 1

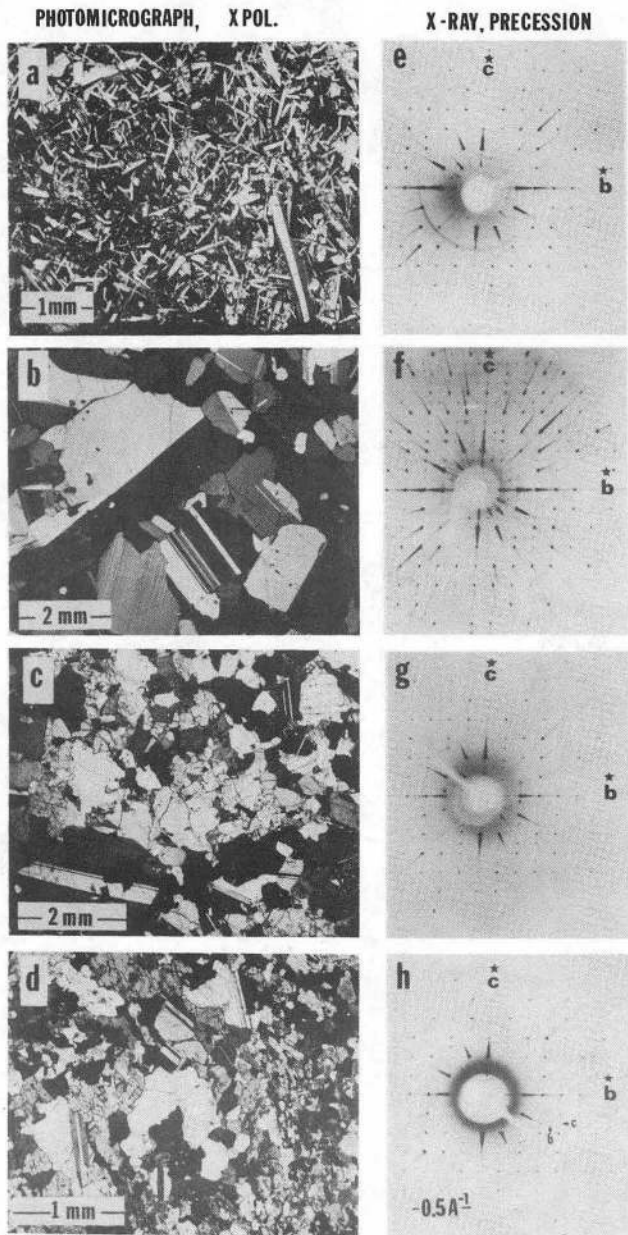


Fig. 2

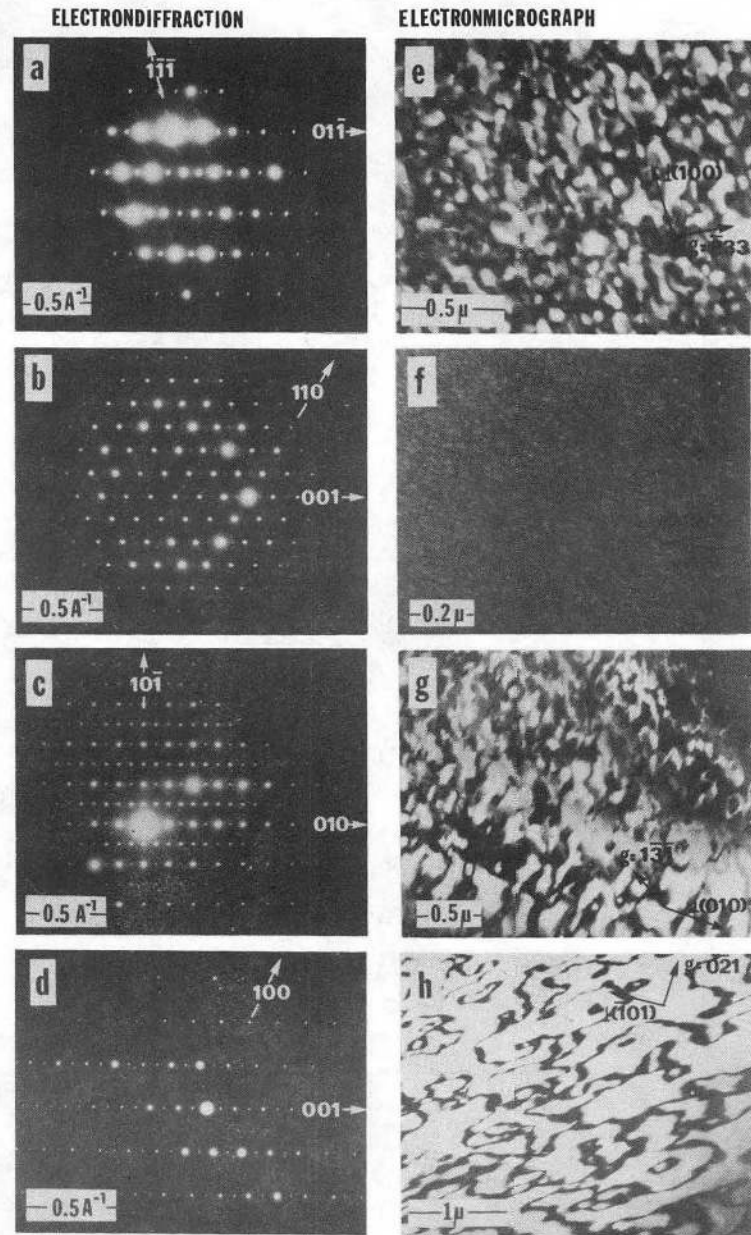


Fig. 3

XBB 7112-6108

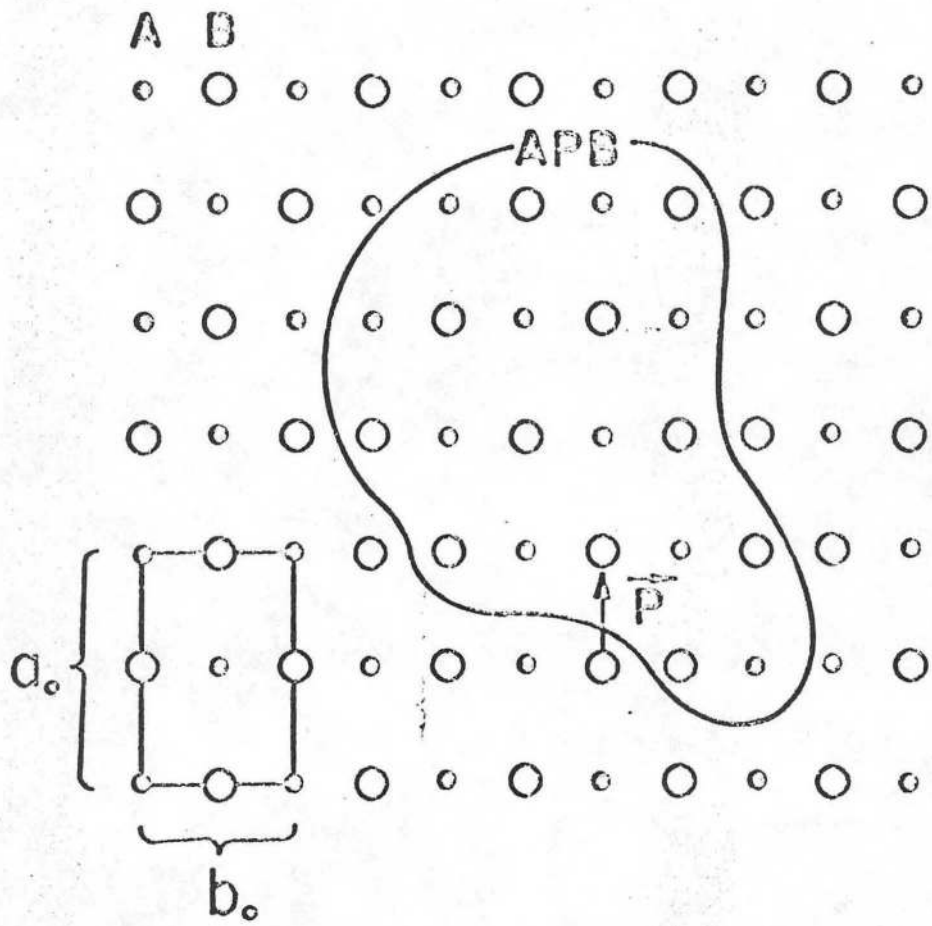


Fig. 4

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