

## Studies of lunar plagioclases, tridymite, and cristobalite\*

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**Abstract**—Plagioclase feldspar single crystals and powders from eight Apollo 11 and six Apollo 12 igneous rocks have been studied by X-ray and electron diffraction, by transmission and scanning electron microscopy, and by Mössbauer spectroscopy. Effects of sample inhomogeneity were avoided by using a variety of techniques on the same single crystal. X-ray and electron-diffraction results are in good agreement. All samples show *a*- and *b*-reflections; those with  $\gamma > 90.5^\circ$  show diffuse *c*-reflections. Diffuse *d*- and *e*-reflections were observed in a few samples. Correlation with chemical composition obtained by microprobe and bulk chemical analyses shows a strong dependence of  $\gamma$  on Ca content. However, formulation of an X-ray diffraction method for determining composition requires further chemical and crystal-structure analyses, because of the small range of cell parameters and their dependence on interrelated effects of Al/Si order and composition. Iron occurs in both the large-cation and tetrahedral sites. Preliminary crystallographic data were obtained for cristobalite and tridymite from four Apollo 12 rocks.

### INTRODUCTION

IN THIS WORK we have attempted to make detailed comparisons between several kinds of precise X-ray and electron-diffraction data from lunar plagioclases, and to correlate the results with chemical data obtained independently for the same specimens. Our aim has been to devise simple methods for determining lunar plagioclase compositions, and to compare the details of the ordering processes in lunar and terrestrial plagioclases. We also wished to determine whether a greater degree of similarity existed among plagioclases from the Bench Crater locality than for all Apollo 12 samples.

In as many cases as possible, we have examined an individual single crystal of plagioclase by X-ray diffraction, electron-microprobe and electron-diffraction analysis. This was done both to evaluate the relative usefulness of different techniques, and to correlate precisely the various physical and chemical properties even though the bulk feldspar samples might be inhomogeneous. A Mössbauer study was performed on one sample to determine the correct partitioning of ferrous iron between the calcium-sodium and the tetrahedral sites.

We have also obtained crystallographic data on several samples of lunar cristobalite and tridymite.

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## EXPERIMENTAL METHODS

*Samples studied*

We have examined crystals from Apollo 12 rocks 12021, 12038, 12040, 12051, 12052, and 12063, and have re-examined crystals from Apollo 11 rocks 10003, 10020, 10047, 10049, 10050, 10057, 10071, and 10072. There is a very limited range in chemistry and cell parameters among the plagioclases from the different rocks. Fine, colorless needles found in vugs in sample 12038 (one piercing an ilmenite crystal) were identified by EVANS (1971) as plagioclase crystals in an unusual habit elongated parallel to the  $c$  axis, and bounded by (100) and (010) faces. Cell parameters were obtained for a single crystal and the value of  $\gamma$  implies a composition of  $Ab_{17}An_{83}$ , essentially the same as that found for the groundmass crystals in this sample.

*Mössbauer spectra*

Distribution of iron in the crystal structure was studied by Mössbauer absorption experiments. A sample of approximately 530 mg of crushed and purified feldspar from rock 12038 was used; the sample contained a minor amount of ilmenite. A 200-channel "flyback" constant-acceleration spectrometer, supplied by courtesy of the National Bureau of Standards, recorded the spectrum at room temperature (Table 1, Fig. 1). The source was  $Co^{57}$  in palladium. In computer fitting of the spectra, no constraints were applied to any of the feldspar peaks. However, the locations of the ilmenite impurity peaks were held constant to agree with the known peak locations of lunar ilmenite (HIRZINBERG and RILEY, 1970), and the heights and widths of the two ilmenite peaks were constrained to be equal.

*Chemical studies*

Bulk samples were prepared from mineral grains that passed through ninety mesh bolting cloth and then were centrifuged in bromoform-acetone mixtures ( $G = 2.87$  and  $2.65$ , respectively) to obtain concentrates of plagioclase and of silica. Further concentration was performed magnetically. Recovery was greater than 90% in all cases. The percentages by weight of plagioclase and silica minerals are as follows. 12021,29: sample weight 2.15 g, plagioclase 19.1%, tridymite 2.0%; 12038,72: 3.90 g, plagioclase 17.0%, cristobalite 1.8%; 12051,36: 2.99 g, plagioclase 16.8%, cristobalite 1.2%; 12052,57: 2.51 g, plagioclase 3.0%, cristobalite 0.4%; 12063,122: 0.47 g, plagioclase 10.2%. These percentages are somewhat lower than those estimated during the preliminary examinations (WARNER, 1970). The differences probably reflect sample inhomogeneities in small samples of relatively coarse-grained rocks. We have no explanation for the abundance of tridymite in rock 12021.

Two of the feldspar separates (12038, 12051, Table 2) were chemically analyzed to correlate the chemistry with the X-ray data and to verify that the single crystals were representative of the bulk feldspar.

Some individual single crystals that had been examined by X-ray diffraction techniques were analyzed for us by charitable experts (A. E. BENCE, State University of New York, Stony Brook; F. R. BOYD, JR., Geophysical Laboratory, Washington, D.C.) using electron-microprobe methods,

Table 1. Mössbauer hyperfine parameters and areas for lunar plagioclase from rock 12038.

	Area*	Isomer shift, mm/sec†	Quadrupole splitting, mm/sec	Widths	
				Low velocity peak, mm/sec	High velocity peak, mm/sec
Doublet 1:					
tetrahedral sites	58%	0.92	1.55	0.54	0.58
Doublet 2:					
Ca Na sites	42%	1.14	2.13	0.45	0.58

\* Ilmenite peaks, which accounted for 3% of the original area, were subtracted to arrive at these values.

† Isomer shifts are given with respect to the centroid of the metallic iron spectrum.

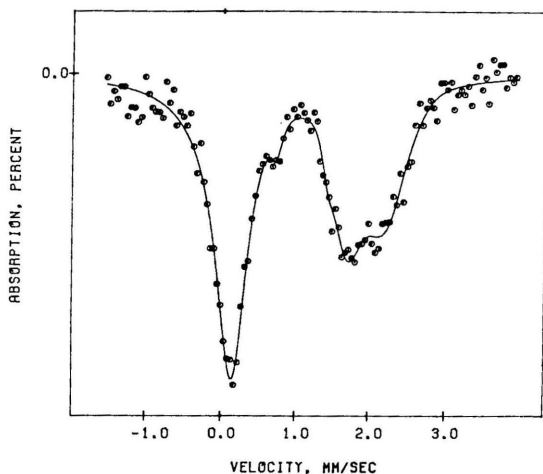


Fig. 1. Mössbauer spectrum of feldspar concentrate from rock 12038,72. There are two major doublets present; the left-hand peaks are superposed, but the right-hand peaks are partially resolved. These doublets are due to ferrous iron in the Ca/Na and tetrahedral sites. A much less intense doublet due to ilmenite impurity can also be seen between the major peaks.

and the results are reported in Table 2. Additional plagioclase analyses became available at the Second Lunar Science Conference. These results are included in Table 2, together with values for plagioclase from rock 12063, communicated to us by W. E. TRZCIENSKI, Jr., Princeton University.

#### *X-ray diffraction studies*

Powdered samples were prepared with internal standards  $\text{BaF}_2$  ( $a = 6.1971 \text{ \AA}$ ) or  $\text{CaF}_2$  ( $a = 5.4620 \text{ \AA}$ ). Diffractometer patterns were obtained using Ni-filtered Cu radiation ( $\text{Cu K}\alpha_1$ ,  $\lambda = 1.5405 \text{ \AA}$ ); the methods and data processing were the same as those described by STEWART *et al.* (1970).

Single-crystal examinations of selected crystals from rocks 12021, 12038, 12040, and 12051 were made both with oscillation and Weissenberg methods using Ni-filtered Cu radiation and with Buerger precession methods (crystal-to-film distance 60 mm) using Zr-filtered Mo radiation. In order to check for the presence of weak reflections, exposures were made up to 300 hours at 50 kV, 20 mA (Mo radiation). All crystals studied were examined with the polarizing microscope and chosen to be as free as possible from twinning. Cell parameters were obtained from measurements of the single-crystal patterns and agree within the errors of measurement with those obtained from the powder refinements.

#### *Electron microscopic studies*

Six plagioclases from Apollo 11 rocks (10003, 10020, 10050, 10057, 10071, and 10072) and five from Apollo 12 rocks (12021, 12038, 12040, 12051, and 12052) that had previously been studied by X-ray diffraction were investigated with the following instruments: 80–100 keV (Siemens), 200 keV (JEOL), and 1000 keV (Hitachi and AEI).

Two methods of sample preparation were used as follows. (1) A small number of grains from the heavy-liquid grain separates were ground in a small agate mortar, mixed with a few drops of alcohol, the suspension sucked into a fine-tipped glass pipette and floated onto a carbon foil or carbon net foil. (2) A method was developed with the assistance of Dr. C. WILLAIME, University of Paris, to convert the small original crystal fragments used for the X-ray single-crystal investigation (equant, smallest 0.03 mm) into a tiny amount of powder to be used as a mount for electron-diffraction and

Table 2. Chemical data for Apollo

Rock number	10050	12051	12051	10047	12021	12021
Analysis number	1	2	3	4	5	6
Method	Wet chemical	X-ray fluor.	Electron microprobe	Electron microprobe	Electron microprobe	Electron microprobe
Sample	0.36 g	0.15 g	20 grains, averaged	Strongly zoned crystals	Single crystal F4, 3 points averaged	Single crystal F6, 3 points averaged
Oxide				Weight %		
SiO <sub>2</sub>	46.3	45.8	46.4	49.26	46.78	46.99
TiO <sub>2</sub>	0.05	0.09		0.09		
Al <sub>2</sub> O <sub>3</sub>	34.0	33.6	33.6	31.19	33.46	32.58
FeO	0.99	0.87	0.83	0.65	0.52	0.53
MnO	0.03	0.02				
MgO	0.3	0.24		0.14		
CaO	17.1	18.2	17.9	17.11	18.28	17.71
Na <sub>2</sub> O	1.05	0.85	0.83	1.61	0.80	1.02
K <sub>2</sub> O	0.09	0.04	0.06	0.09	0.04	0.05
TOTAL	99.91	99.71	99.62	100.14	99.88	98.88
					Number of atoms on the	
Si	2.135	2.100	2.147	2.258	2.157	2.186
Ti	0.002	0.003		0.003		
Al	1.848	1.857	1.832	1.685	1.817	1.785
Σ Si, Ti, Al	3.99	3.96	3.98	3.95	3.97	3.97
Fe	0.038	0.034	0.032	0.025	0.020	0.021
Mn	0.001	0.001				
Mg	0.021	0.017		0.010		
Ca	0.845	0.914	0.887	0.840	0.903	0.883
Na	0.094	0.077	0.074	0.143	0.071	0.092
K	0.005	0.002	0.004	0.005	0.002	0.003
Σ Ca, Na, K, Mn, Mg	0.97	1.01	0.97	1.00	0.98	0.98

(1) Includes 0.01 Cr<sub>2</sub>O<sub>3</sub>, 0.01 SrO, 0.01 BaO. Analyst, J. ITO (FRONDEL *et al.*, 1970). (2) MgO, Na<sub>2</sub>O, 0.02 P<sub>2</sub>O<sub>5</sub> determined by chemical methods. Microscopic count for 507 grains showed 1.0% cristobalite, which was corrected for as SiO<sub>2</sub> before recalculating to 100%. Analysts R. P. CHRISTIAN, H. J. ROSE, JR., F. CUTTITA, U.S. Geological Survey. (3) KEIL *et al.* (1971). (4) WARE *et al.* (1970). Crystals zoned An<sub>90</sub> cores to An<sub>75</sub> rims. (5) Analyst F. R. BOYD, JR., Geophysical Laboratory. Fe<sup>IV</sup> 0.002, Fe<sup>VII</sup> 0.018, from Mössbauer studies (VIRGO *et al.*, 1971). (6) Analyst F. R. BOYD, JR., Geophysical Laboratory. Fe<sup>IV</sup> 0.003, Fe<sup>VII</sup> 0.018, from Mössbauer studies (VIRGO *et al.*, 1971).

electron-transmission studies. The crystallite was removed from its glass rod mounting, washed in acetone and/or alcohol, placed between two very clean glass slides, and powdered by pressing the glass between two fingers. The area on the glass containing the powder was marked on the back side of both glasses and the sides with the powder on them were evaporated with carbon. By dipping the end of the glass into a basin with distilled water, the carbon layer can be made to float on the surface, and the area containing part of the powder can then be transferred onto an electron-microscopic copper grid.

At 100 keV, relatively long exposure times (up to 12 minutes) could be used to produce enhanced satellite intensities. Such exposures were possible because of the high mechanical stability of the beam for the 100 keV microscope compared with that of the 1000 keV instruments. In the long exposures no effects due to deterioration of the crystals during electron irradiation could be detected, i.e., all satellites and other additional intensities observed could be attributed to structural features of the crystallites (Figs. 2, 3). For some diffractograms, especially those showing diffuse reflections,



## 11 and Apollo 12 plagioclases.

12063 7	12063 8	12038 9	12038 10	12038 11	12038 12	12038 13	10057 14
Electron microprobe	Electron microprobe Zoned crystal, 3 points averaged	X-ray fluor. 0.15 g	Electron microprobe Single crystal F1, 5 points averaged	Electron microprobe Single crystal F6, 5 points averaged	Electron microprobe Single crystal T4, 5 points averaged	Electron microprobe 6 grains averaged	Electron microprobe
Weight %							
48.9 0.08 32.1 0.70 0.24 17.4 1.14 0.05 100.61	47.78 32.37 0.81 17.23 1.11 0.69 99.99	48.8 0.08 32.0 0.70 0.01 0.30 16.4 1.75 0.07 100.11	47.43 32.50 0.43 0.23 17.18 1.65 0.02 99.44	47.27 32.69 0.43 0.23 17.09 1.66 0.03 99.40	47.61 32.58 0.46 0.22 16.92 1.81 0.04 99.64	48.3 31.7 0.49 16.5 1.81 0.02 99.13	50.04 0.19 30.49 0.82 0.38 15.89 1.98 0.24 100.03
basis of 8 oxygen atoms							
2.230 0.003 1.726 3.96 0.027 0.016 0.850 0.101 0.003 0.97	2.204 1.760 3.96 0.031 0.852 0.099 0.040 0.99	2.176 0.003 1.784 3.96 0.027 0.0003 0.021 0.834 0.161 0.004 1.02	2.193 1.771 3.96 0.017 0.016 0.851 0.148 0.001 1.02	2.186 1.782 3.97 0.017 0.016 0.847 0.148 0.002 1.01	2.196 1.771 3.97 0.018 0.015 0.836 0.162 0.004 1.02	2.234 1.728 3.96 0.019 0.021 0.818 0.162 0.001 1.00	2.292 0.007 1.646 3.95 0.031 0.026 0.780 0.176 0.014 1.00

(7) TAYLOR *et al.* (1971), unshocked feldspar. (8) Includes 0.02 BaO. W. E. TRZCIENSKI, Jr., Princeton University (written communication, 1971). (9) Includes 0.01 P<sub>2</sub>O<sub>5</sub>, MgO, Na<sub>2</sub>O and P<sub>2</sub>O<sub>5</sub> determined by chemical methods. Analysts R. P. CHRISTIAN, H. J. ROSE, Jr., and F. CUTTITA, U.S. Geological Survey. Microscopic count of 425 grains showed 2.8% cristobalite which was corrected for as SiO<sub>2</sub> before recalculation to 100%. Fe<sup>IV</sup> 0.011, Fe<sup>VII</sup> 0.016, from Mössbauer studies. (10) Analyst A. E. BENCE, State University of New York, Stony Brook. Fe<sup>IV</sup> 0.007, Fe<sup>VII</sup> 0.010, from Mössbauer studies. (11) Analyst A. E. BENCE, State University of New York, Stony Brook. Fe<sup>IV</sup> 0.007, Fe<sup>VII</sup> 0.010, from Mössbauer studies. (12) Analyst A. E. BENCE, State University of New York, Stony Brook. Fe<sup>IV</sup> 0.008, Fe<sup>VII</sup> 0.010, from Mössbauer studies. (13) KEIL *et al.* (1971). (14) WARE *et al.* (1970). Zoning observed, An<sub>76-82</sub>.

the lamellar contrast was increased before reproduction by enhancement with a LogEtronic D-54 enlarger with electronic contrast control.

High voltages were applied mainly to thicker specimens less disturbed by the grinding process, in order to investigate whether the thickness of the sample has an influence on the appearance in electron diffraction of those types of reflections which were not found by X-rays in the first stage of this work (STEWART *et al.*, 1970). The crystallite size in the high-energy work was between that for the X-ray work (0.1 mm) and the 100 keV study (about 1000 Å). The appearance of the satellite reflections seems to be totally independent of specimen thickness.

In addition to *h0l* diffractograms, those showing the  $\bar{h}kh$  reciprocal plane were found convenient for checking the different classes of reflections in feldspars. The angle between the two reciprocal axes,  $[\bar{h}0h]^*$  and  $[0k0]^*$ , is approximately 90 deg, and the electron beam has an inclination of only about 12 deg to the *c*\* axis, the normal to the (001) plane which is the main surface of many cleavage (powder) particles.

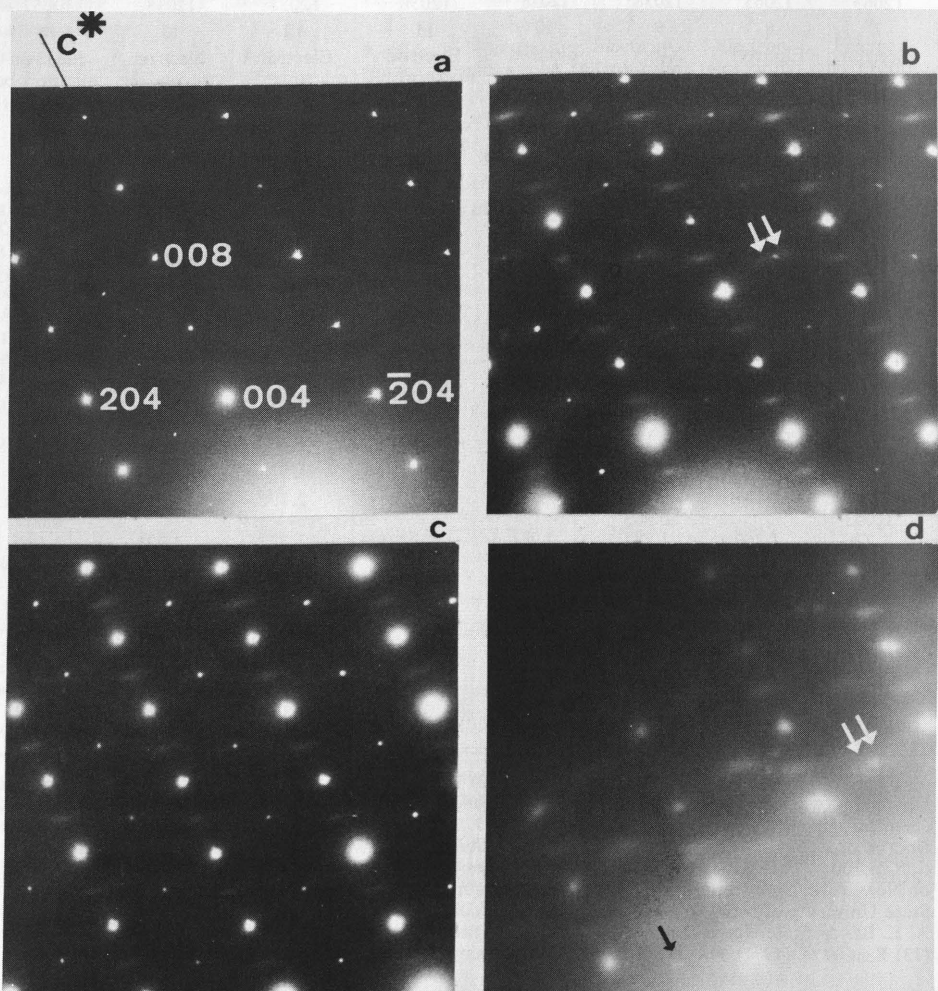


Fig. 2. (a)–(c): Three selected-area  $h0l$  diffractograms taken at 100 keV of the same bytownite particle from rock 10050. Lens currents, tilt orientation, and apertures were kept constant; exposure times were increased. (a) 1.5 minutes;  $a$ -reflections strong,  $b$ -reflections very weak. (b) 3 minutes; sharp  $b$ - and diffuse elongated  $c$ -reflections can be seen. One pair of diffuse satellites surrounding a  $b$ -reflection is marked by arrows. (c) 6 minutes;  $c$ -reflections are more pronounced. (b) and (c) are contrast-enhanced prints. (d)  $h0l$  diffractogram taken at 1000 keV of a bytownite particle from rock 12038 with one pair of satellites marked by double arrows, and one  $d$ -reflection marked by a single arrow.

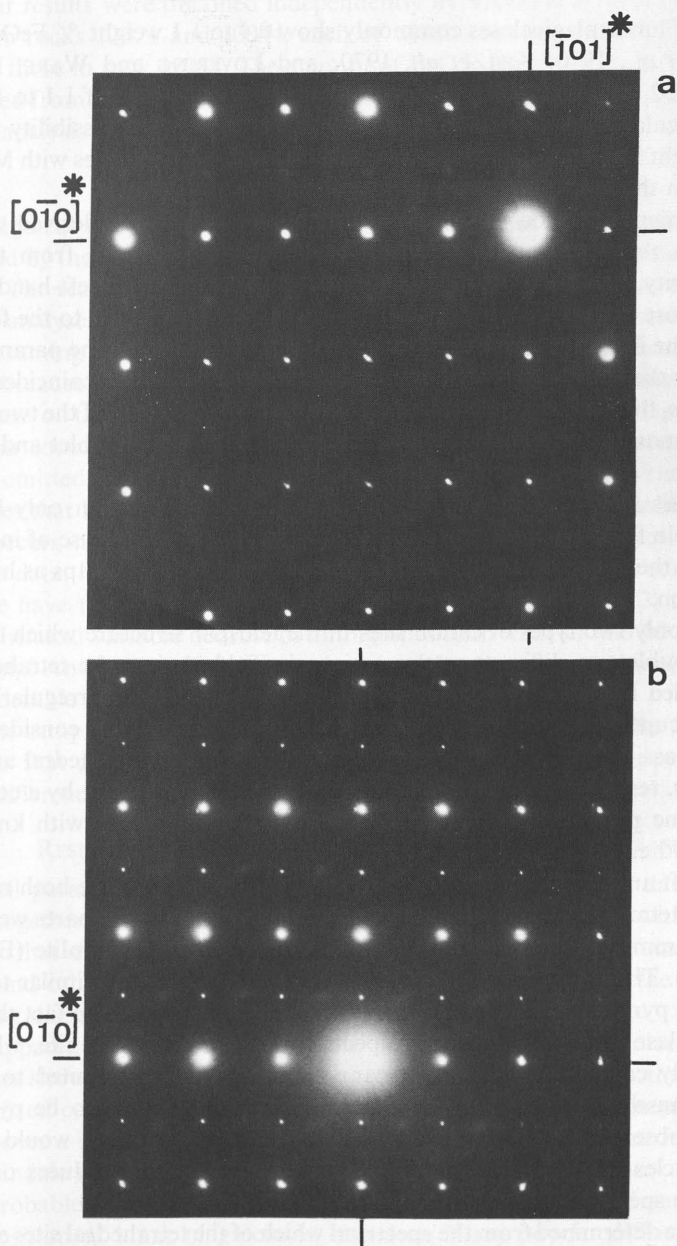


Fig. 3. Selected-area diffractograms showing the  $\bar{h}kh$  net of bytownite. The relative intensities and degrees of diffuseness of *b*- and *c*-reflections can be compared. (a) Rock 12052; 200 keV diagram. *b*-reflections diffuse; *c*-reflections too weak to be detected in this diagram. (b) Rock 10050; 100 keV diagram. *b*-reflections sharp, *c*-reflections diffuse and elongated.

## FERROUS IRON IN LUNAR PLAGIOCLASES

Analyses of lunar plagioclases commonly show 0.4 to 1.1 weight % FeO (Table 2; cf. FRONDEL *et al.*, 1970; KEIL *et al.*, 1970; and LOVERING and WARE, 1970). An analysis of 12052 plagioclase by BENCE *et al.* (1970) gives a range of 1.1 to 1.6 weight % FeO. Recalculations of these analytical figures suggested the possibility that some of this iron might be in tetrahedral coordination. The results of studies with Mössbauer spectra confirm this suggestion.

Site assignments for iron in feldspar from rock 12038 can be derived with some certainty from the Mössbauer spectrum shown in Fig. 1. Aside from the minor ilmenite impurity, there are apparently two doublets present, the left-hand peaks of which are almost coincident. We have fitted Lorentzian line shapes to the four peaks (as well as to the ilmenite impurity peaks), and the resultant hyperfine parameters and area ratios for the feldspar are given in Table 1. Due to the near coincidence of the left-hand peaks, there may be some uncertainty about the matching of the two doublets. We judged it most likely that the outer peaks constituted one doublet and the inner peaks, another.

On the basis of their hyperfine parameters, these doublets can only be due to ferrous iron. No ferric iron was detected in the spectrum, but because of interference from ilmenite, the limit of detection is rather high in this case, perhaps as high as 5% of the total iron.

There are only two types of cation sites in the feldspar structure which could give rise to two doublets as different as those given in Table 1, i.e., the tetrahedral sites usually occupied by aluminum or silicon cations, and the larger, irregularly coordinated sites occupied by calcium or sodium cations. Solely on this consideration we can therefore assign the two ferrous doublets (Fig. 1) to the tetrahedral and Ca/Na groups of sites, respectively, and in fact this assignment is borne out by a comparison of the hyperfine parameters (Table 1) with those of iron in sites with known configuration in other minerals.

Isomer shift and quadrupole splitting for doublet 1 (Table 1) are both rather low, as is usual for tetrahedral sites. The isomer shift of 0.92 mm/sec compares well with the value of 0.95 mm/sec for the tetrahedral ferrous iron site in staurolite (E. DOWTY, unpubl. work). The parameters for doublet 2 (Table 1) are rather similar to those of the *M2* site in pyroxenes. This result is not surprising in view of the fact that the Ca site in plagioclase is similar in some respects to the pyroxene *M2* site, which often contains chiefly calcium. This doublet cannot, however, be attributed to pyroxene impurity, because approximately 2% of the sample would have to be pyroxene to produce the observed absorption, and this amount of pyroxene would be easily visible in the clear feldspar concentrate. The ilmenite, which produces only minor features of the spectrum, is easily visible optically.

It cannot be determined from the spectrum which of the tetrahedral sites and Ca/Na sites are occupied by iron, but with respect to the tetrahedral sites it is probably a safe assumption that iron preferentially occupies those which are filled in ordered plagioclase by aluminum. The rather large line-widths measured for both doublets suggest that iron occupies more than one site in each case, but this point is not certain because there are other factors which can cause line broadening.

Similar results were obtained independently by VIRGO *et al.* (1971) for lunar feldspars from rocks 12021 and 10044, each of which contains about 0.2 weight % less FeO than those in rock 12038. The amount of FeO in the plagioclase from rock 12038 is estimated from the total absorption to be 0.8 weight %, in good agreement with the chemical analysis (No. 9, Table 2).

#### COMPOSITION OF LUNAR PLAGIOCLASES

The physical properties and chemical composition of lunar plagioclase must be determined by independent methods for a meaningful correlation between them to be achieved. The results of chemical analyses are given in Table 2. The values obtained from the analyses were recalculated to 100%, and the formula contents were calculated assuming eight oxygen atoms. Summations of the formula contents are problematical because of the analytical errors associated with methods using small samples, and because of the uncertainties introduced by the relatively high ferrous iron and magnesium contents and low alkali and alkaline-earth contents of lunar feldspars. We have omitted iron from the summations; its partitioning is unknown except for the samples that have been studied by Mössbauer methods. Presumably in every case less iron occurs in the tetrahedral sites, so that no deficiency in large cations is apparent to us. The data, however, are not sufficiently accurate for any final conclusions to be made. We have taken the calcium content of the formula (Table 3) to be the independent chemical variable because of ambiguities as to what should be included in the conventional An-content. Calcium is the most abundant large cation, and therefore has considerable influence on the cell parameters.

For the samples we studied, the range of composition of Apollo 12 plagioclases is less than that of the Apollo 11 plagioclases, and this difference appears to reflect a geologic difference at the two landing sites.

#### RESULTS OF X-RAY DIFFRACTION STUDIES ON PLAGIOCLASES

The cell parameters obtained from the refinements of powder data are given in Table 3, together with the number of Ca atoms per formula unit with eight oxygen atoms, so that correlation can be made with the chemistry. Estimates by optical methods and other reported compositional data are separated in the tabulation for reasons just discussed.

It is apparent from Table 3 that the cell parameters (particularly  $\gamma$ ) of lunar plagioclases show a dependence on Ca content. NISSEN (1969) and others have shown that at a fixed composition, the cell parameters of terrestrial calcic plagioclase may also vary over a small range with the degree of Al/Si order. The maximum degree of order attainable is, of course, a function of plagioclase composition.

It is probable that eventually a method can be devised for estimating accurately the composition of lunar plagioclases, based on X-ray powder diffraction methods. However, it is first necessary to untangle the interrelated effects of composition and thermal history on the degree of Al/Si order, and of composition and Al/Si order on the cell parameters. At the present time the available data are insufficient to accomplish this, especially in view of the very restricted total range of variation in the cell parameters of lunar plagioclases. We need, first, good chemical analyses of many more of

Table 3. Crystallographic data correlated with calcium

Rock number	ANS	10003	12040	10050	12051	10047	12021
Chip number	305	38	20	32	36	27	29
Space group	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$
Reflection types obs.	all						
<i>b</i>		m, sharp	m, sharp	m, sharp	m, sl. diffuse	w, sharp	m, sharp
<i>c</i>		w, diffuse	w, diffuse	w, diffuse	vw, diffuse	vwv <sup>2</sup>	w, diffuse
<i>d</i>		not obs.	not obs.	vw?	not obs.	not obs.	not obs.
Cell parameters							
<i>a</i> (Å)	8.179(2)	8.192(3)	8.181(2)	8.178(2)	8.186(2)	8.187(3)	8.183(2)
<i>b</i> (Å)	12.869(2)	12.892(3)	12.875(2)	12.872(2)	12.873(3)	12.882(3)	12.871(2)
<i>c</i> (Å)	14.174(2)	14.186(4)	14.180(2)	14.184(2)	14.184(3)	14.196(2)	14.180(4)
$\alpha$ (°)	93.11(2)	93.24(3)	93.35(2)	93.28(2)	93.31(3)	93.37(3)	93.43(2)
$\beta$ (°)	115.89(2)	116.03(2)	115.93(2)	115.95(2)	115.94(2)	116.05(2)	115.94(2)
$\gamma$ (°)	91.23(2)	91.02(2)	90.99(2)	90.95(3)	90.91(3)	90.81(3)	90.79(2)
Cell volume (Å <sup>3</sup> )	1338.6(3)	1342.6(4)	1339.4(4)	1339.0(4)	1341.0(4)	1341.4(4)	1339.2(4)
Number of lines refined	33	24	37	31	29	22	29
Std. error <sup>2</sup> $\theta$ , CuK $\alpha_1$	0.013	0.019	0.016	0.018	0.020	0.016	0.016
Calculated $\Delta 2\theta$ , <sup>2</sup> $\theta$ , (132)-(132), CuK $\alpha_1$	2.26	2.22	2.25	2.20	2.16	2.18	2.20
$\Gamma$ , <sup>2</sup> $\theta$ , CuK $\alpha_1$ (SMITH and GAY, 1958)	1.47	1.37	1.43	1.35	1.27	1.28	1.31
B, <sup>2</sup> $\theta$ , CuK $\alpha_1$ (SMITH and GAY, 1958)	0.72	0.77	0.73	0.74	0.75	0.78	0.75
Ca atoms per eight oxygen atoms	1.00			0.85	0.91	0.84	0.89
Notes on chemistry	synthetic, STEWART (1967)	optical est. An 85-87 STEWART <i>et al.</i> (1970)	microprobe analyses: An 88-96 ANDERSON <i>et al.</i> (1971); An 92-95 WALTER <i>et al.</i> (1971)	Table 2; optical est. An 83-85 STEWART <i>et al.</i> (1970)	Table 2; also microprobe analyses, An 92-94 WALTER <i>et al.</i> (1971)	Table 2; optical est. An 81-83 STEWART <i>et al.</i> (1970)	Table 2; other microprobe analyses: An 90-97 DENCE <i>et al.</i> (1971); An 89-93.7 KLEIN <i>et al.</i> (1971); An 91-96 WALTER <i>et al.</i> (1971)

Errors in parentheses are one standard deviation; for 8.169(4) read  $8.169 \pm 0.004$  Å, etc.

these plagioclases, particularly of the less Ca-rich Apollo 11 samples. Second, we must complete several critical crystal-structure refinements in order to determine the actual degree of Al/Si order at several compositions. These studies are now in progress.

The types of reflections observed for single crystals from each of the rocks are listed in Table 3. Within each rock, the diffraction effects and cell parameters were found to be uniform from crystal to crystal, and there are few differences in diffraction effects among the plagioclases from the four different rocks. All the crystals examined exhibit strong, sharp *a*-reflections ( $h + k$  even,  $l$  even). The *b*-reflections ( $h + k$  odd,  $l$  odd) are always present and moderately strong, but vary in character, being most diffuse (but not elongated) in crystals from rocks 12038 and 12051. In the 12038 crystals, although almost all the *b*-reflections are symmetrical and centered precisely on lattice points, a few seem to be replaced by a pair of very diffuse spots, of unequal intensity, on either side of the *b*-position on the  $0kl$  and  $h0l$  nets. The separation seems to be approximately normal to (011). The *b*-reflections are fairly sharp for the crystals from rock 12021 and sharp for those from rock 12040.

Weak *c*-reflections ( $h + k$  even,  $l$  odd) are present in all Apollo 12 crystals, but are most diffuse for those from rock 12038, where streaks that are almost (but not quite) continuous can be observed on the  $0kl$  net elongated approximately parallel to  $b^*$ . In crystals from rock 12021, the *c*-reflections are also weak, diffuse, elongate on the  $h0l$  net in a direction between  $a^*$  and the projection of *a*. These observations are consistent with the direction of elongation found for the diffuse *c*-reflections of



contents for Apollo 11 and Apollo 12 plagioclases.

12063 122	12053 89	12038 72 <i>P</i> $\bar{1}$	12052 57 <i>P</i> $\bar{1}$	10020 41 <i>P</i> $\bar{1}$	10057 36 <i>I</i> $\bar{1}$	10072 33 <i>I</i> $\bar{1}$	10071 29 <i>I</i> $\bar{1}$	10049 22
		m, diffuse vw, diffuse vw, diffuse	m, diffuse* vw, diffuse not obs.	w, diffuse vw, diffuse not obs.	vw, diffuse not obs. not obs.	vw, diffuse* not obs. not obs.	vw, diffuse* not obs. not obs.	
8.185(3)	8.169(4)	8.180(3)	8.180(3)	8.167(3)	8.174(3)	8.178(2)	8.175(3)	8.176(2)
12.879(3)	12.877(3)	12.874(3)	12.875(5)	12.861(3)	12.876(3)	12.871(3)	12.860(4)	12.864(3)
14.194(4)	14.186(3)	14.196(3)	14.190(2)	14.186(2)	14.192(4)	14.194(4)	14.190(4)	14.192(2)
93.33(3)	93.39(3)	93.45(2)	93.36(3)	93.46(2)	93.34(3)	93.39(3)	93.25(4)	93.40(2)
116.01(2)	115.97(2)	116.06(2)	116.06(3)	115.92(2)	116.06(2)	116.09(2)	116.04(3)	116.07(2)
90.74(3)	90.65(3)	90.63(2)	90.59(3)	90.54(3)	90.46(3)	90.45(3)	90.41(4)	90.33(3)
1339.2(5)	1338.2(5)	1338.2(5)	1339.1(4)	1336.6(6)	1338.6(4)	1338.6(4)	1337.2(6)	1337.6(4)
27	27	24	24	22	30	31	24	25
0.019	0.019	0.019	0.018	0.017	0.023	0.022	0.023	0.016
2.17	2.12	2.14	2.07	2.09	2.00	2.01	1.92	1.97
1.18	1.17	1.14	1.07	1.11	0.94	0.95	0.79	0.85
0.78	0.75	0.79	0.78	0.74	0.81	0.82	0.82	0.82
0.85		0.84	0.82		0.78			

Table 2	microprobe analysis; An 70-78 BANCROFT <i>et al.</i> (1971); optical est. An 78-95 CHRISTIE <i>et al.</i> (1971)	Table 2; optical est. An 75-80 CHRISTIE <i>et al.</i> (1971)	microprobe analysis, BENCE (writt. comm. 1970); BENCE <i>et al.</i> (1970)	optical est. An 78-80 STEWART <i>et al.</i> (1970)	Table 2; optical est. An 75-76 STEWART <i>et al.</i> (1970)	optical est. An 75-76 STEWART <i>et al.</i> (1970)	optical est. An 74-75 STEWART <i>et al.</i> (1970)	optical est. An 73-74 STEWART <i>et al.</i> (1970)
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\* Some material shows diffuse *e*-reflections and no *b*-reflections in electron diffractograms.

transitional anorthite by RIBBE and COLVILLE (1968). No *d*-reflections ( $h + k$  odd,  $l$  even) were discovered despite the long exposures and careful searching. Both *b*- and *c*-reflections become stronger and less diffuse in the order 12038, 12051, 12021, 12040.

Twinning was found to be common except for crystals from rock 12038 where only a few albite-law twins were noted. Most of the crystals from the other three rocks were twinned by the albite law, but a few from rock 12051 exhibited twinning by the Carlsbad law. CHRISTIE *et al.* (1971) observed Carlsbad and albite twin laws for plagioclase crystals from sample 12038,32, and Carlsbad and complex Carlsbad-albite twinning for strongly zoned plagioclases in the composition range An<sub>78-95</sub> from rock 12053,66. BANCROFT *et al.* (1971) observed the presence of twinning on the albite, Carlsbad, albite-Carlsbad and pericline laws for plagioclases in the range An<sub>82-87</sub> for 12051 and An<sub>70-78</sub> for 12053.

## RESULTS OF ELECTRON MICROSCOPIC STUDIES ON PLAGIOCLASES

The electron-diffraction method is sensitive to very weak reflections, and the white radiation which complicates the interpretation of X-ray diffractograms is absent. The corresponding electron-transmission micrographs show directly the presence of domains, stacking faults, and dislocations, and also give information about the degree of homogeneity in the arrangement of such features within very small areas. These studies therefore supplement those carried out by the X-ray diffraction methods.

All plagioclase electron diffractograms show the presence of both *a*- and *b*-reflections, provided the diffractograms are made with sufficient irradiation time. No evidence for lunar plagioclases with a *C*-centered lattice has been found. The *b*-reflections observed have various degrees of diffuseness relative to the *a*-reflections (Figs. 2, 3). For example, in the bytownite crystals from rock 10050, the *b*-reflections are very sharp, although much less intense than are the nearest *a*-reflections, whereas in crystals from rock 10020 the *b*-reflections are diffuse. Similarly, the *b*-reflections are sharp in bytownite from rock 12021 and diffuse in bytownite from rock 12052. Details on the degree of diffuseness of *b*-reflections are given in Table 3.

The plagioclases with  $\gamma$ -angles higher than about 90.5 deg show additional very diffuse *c*-reflections strongly elongated approximately parallel to  $[80\bar{2}]^*$  on the *h*0*l* nets and to  $[\bar{2}32]^*$  on the  $\bar{h}kh$  nets, in agreement with the conclusion by RIBBE and COLVILLE (1968) that the direction of elongation is near  $[\bar{2}31]^*$ . The character of these reflections is illustrated in Fig. 2 by *h*0*l* diffractograms of bytownite from rock 10050, and in Fig. 3 by  $\bar{h}kh$  diffractograms.

Diffuse *c*-reflections are also found in bytownite from rock 10020, together with the presence in transmission electron micrographs of a fine lamellar structure of alternating dark and white bands of strongly varying thicknesses (Fig. 4). These bands were also observed in dark field using a single *c*-reflection. The mean periodicity of this structure is about 50 to 70 Å, and the lamellae are irregular enough to cause diffuse reflections in electron diffraction patterns. The lamellae are probably nearly parallel to (231). We propose the term *c*-domains for them. Examples of similar fine lamellae in lunar bytownite from rock 12038 were reported simultaneously by CHRISTIE *et al.* (1971). They may be out-of-step domains of primitive anorthite structure related by body centering vectors  $(\mathbf{a} + \mathbf{b} - \mathbf{c})/2$  across the domain boundaries (RIBBE and COLVILLE, 1968).

The high-energy electron-microscope study of specimens from rocks 10050, 12021, and 12038 confirms the presence of reflections of type *a* and *b*, and of diffuse and elongated *c*-reflections. Several examples of bytownite subgrain boundaries showing a regular dislocation network were observed (Fig. 5).

Some of the selected area electron diffractograms for bytownite particles from rocks 12052, 10071 and 10072 show diffuse *e*-reflections of the first order. These were also found in X-ray precession photographs by BANCROFT *et al.* (1971) in rock 12052. The separation of the *e*-reflections and their relative positions in reciprocal space are similar to those observed for calcic terrestrial labradorite that displays *e*-reflections.

Very diffuse pairs of satellite reflections near the *b*-reflections in patterns from rocks 10050 and 12038 are illustrated in Fig. 2. The line connecting these diffuse intensities is approximately parallel to the elongation of the diffuse *c*-reflections. These satellites have not yet been observed in any other plagioclases, and their implications for the domain structure of these bytownites are being investigated. The same contrast-enhanced diffractograms may show some extremely weak diffuse scattering near the positions of the *d*-reflections.

Scanning electron-micrographs of bytownites grown in vugs show smooth surfaces and only minor indications of impact or deformation. In some cases the boundaries between plagioclases and pyroxenes may have a peculiar vermicular pattern, as



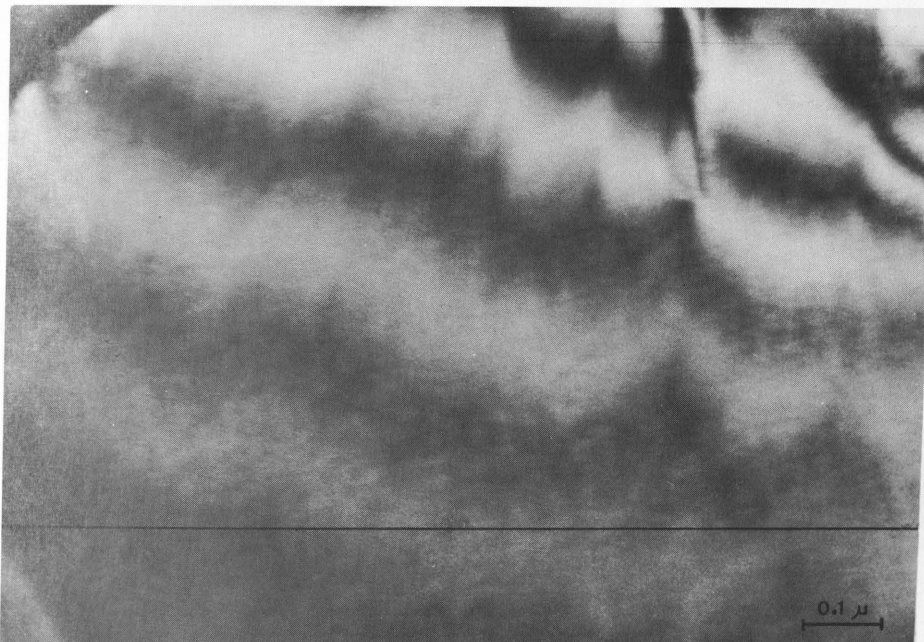


Fig. 4. Lamellar  $c$ -domains in the margin of a cleavage flake of bytownite from rock 10020. The domain boundaries are approximately parallel to the horizontal black line; they are visible as diffraction contrast in the dark thickness fringes. Bright field image; contrast enhanced.

reported by BAILEY *et al.* (1970, Fig. 11). This effect may be due to the presence of the glassy films described by WEILL *et al.* (1970).

#### RESULTS FOR TRIDYMITE AND CRISTOBALITE

The cell parameters obtained by X-ray diffraction powder methods for four silica samples are given in Table 4. A single crystal from rock 12021, when studied by the precession X-ray diffraction method, proved to be tridymite with an apparently orthorhombic sub-cell having space group  $C222_1$ ,  $a = 8.65 \pm 0.02 \text{ \AA}$ ,  $b = 5.01 \pm 0.01 \text{ \AA}$ ,  $c = 8.21 \pm 0.02 \text{ \AA}$ . These parameters agree well with those given by DOLLASE (1967) for orthorhombic material obtained by heating monoclinic, meteoritic tridymite, which transformed reversibly to the orthorhombic phase on heating to  $180^\circ\text{C}$ .

Data have been collected for a structural refinement of the lunar tridymite, and preliminary results, starting with the coordinates given by DOLLASE (1967) and refining only on the orthorhombic subcell reflections yield the same anomalously high temperature factors, short Si-O distances, and large Si-O-Si angles found by him, despite convergence of our data to a weighted  $R$  (HAMILTON, 1965) of 0.073. However, following a suggestion by W. DOLLASE, University of California, Los Angeles (oral communication, 1971), we have found superlattice reflections on the  $h0l$  precession photographs which indicate that the true cell is much larger and of lower symmetry.

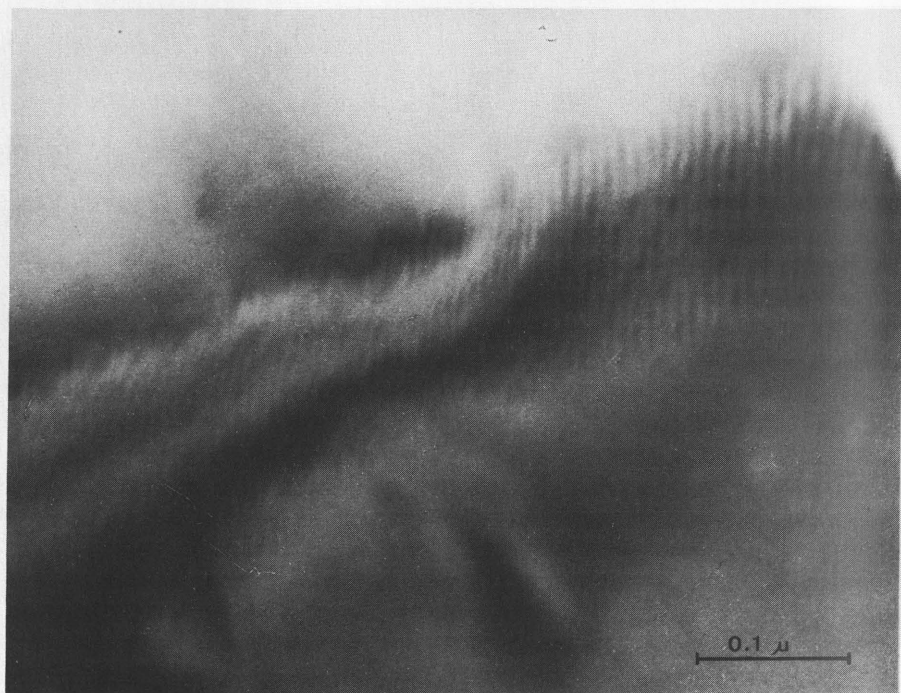


Fig. 5. Contrast possibly caused by dislocation network on subgrain boundary in bytownite from rock 10050; 1000 keV diagram.

Positional disorder in the structure is indicated by the presence on  $h0l$  electron diffractograms of a continuous band of intensity along  $c^*$  (Fig. 6). Furthermore, electron-transmission micrographs show the presence of fine, sharp lamellae of varying thicknesses (Fig. 6). Investigation of the structural problem is continuing, and the final results will be reported elsewhere.

#### DISCUSSION

The data in Table 3 show that the  $c$ -reflections first appear in lunar compositions near  $An_{80}$ , a point near which such reflections also first appear in terrestrial feldspars.

Table 4. Crystallographic data for four Apollo 12 silica minerals.

Rock number, chip number	12051,36	12038,72	12052,57	12021,29
Mineral	Cristobalite	Cristobalite	Cristobalite	Tridymite
Symmetry	Tetragonal	Tetragonal	Tetragonal	Orthorhombic(?)
Cell parameters				
$a$ (Å)	4.9784(4)	4.9793(4)	4.9811(9)	8.660(2)
$b$ (Å)				4.999(2)
$c$ (Å)	6.9312(9)	6.9372(9)	6.936(2)	8.205(3)
Cell volume (Å <sup>3</sup> )	171.79(3)	171.99(2)	172.10(6)	355.2(2)
Number of lines refined	26	26	8	11
Std. error, $^{\circ}2\theta$ , CuK $\alpha_1$	0.018	0.017	0.013	0.023

Error in parentheses is one standard deviation; for 4.9784(4) read  $4.9784 \pm 0.0004$  Å, etc.

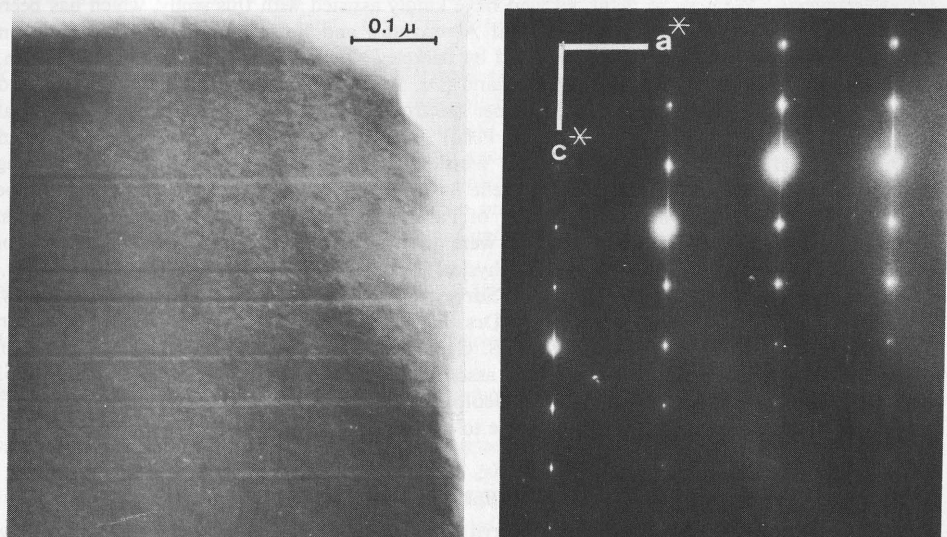


Fig. 6. Lamellae in tridymite from rock 12021 and a corresponding  $h0l$  diffractogram (80 keV) showing diffuse streaks along  $c^*$ .

Apparently, differences in ferrous iron content have no effect on the first appearance of  $c$ -reflections.

The exact nature of the Al/Si ordering is not known in any lunar plagioclase. Simple estimates, based on methods proposed by SMITH and GAY (1958), suggest that many Apollo 11 and Apollo 12 plagioclases are neither as highly disordered as has been observed in some heated terrestrial samples of the nearly equivalent composition, nor as highly ordered as has been observed in plagioclase with nearly the same composition from terrestrial intrusive rocks. Not all lunar samples studied have the same structural state, judged by the SMITH and GAY parameters and the quality of the reflections on X-ray and electron diffractograms. Heating experiments are planned utilizing some of our carefully characterized feldspar separates to elucidate these matters. The lunar plagioclases may be uniquely informative of the ordering process in terrestrial feldspars because of their crystalline perfection and freedom from chemical alteration.

The feldspars from rocks collected at Bench Crater (12038, 12040, 12053, 12063) show as great a range of composition and diffraction effects as do the feldspars from three other widely separated Apollo 12 locations. There is no reason to assume, therefore that the rocks from Bench Crater represent a highly variable local bedrock penetrated at this crater, rather than the typical suite of rocks present in the regolith and rearranged by the cratering event. Similar speculation has been expressed by BROWN *et al.* (1971) and by several groups attempting to use chemical and petrographic data to classify the rocks collected (BIGGAR *et al.*, 1971; WARNER and ANDERSON, 1971; O. B. JAMES and T. L. WRIGHT, U. S. Geological Survey, oral communication, 1971).

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