General Mineralogy

Mineralogy and Petrology of Some Lunar Samples

Abstract. Chemical analyses and norms of four samples are presented which confirm original estimates of low silica, unusual abundance of titania, and low oxidation state of the rocks. Accounts are given of mineralogy and petrology of fine- and coarse-grained igneous rocks and microbreccias with emphasis on chemical composition of individual minerals and glasses. The glasses are either spheres that scatter widely around the composition of lunar basalts or coating glasses that approximate basalts and microbreccias in composition.

Rocks returned by the Apollo 11 mission are all part of the lunar regolith; it may be inferred that some are volcanic rocks originating at no great depth, while others, the microbreccias, form part of a surface layer possibly differing from the dusty maria surface only in degree of compaction. Some components of the regolith show evidence of shock metamorphism and melting; many may have originated at a considerable distance from the point at which they were collected.

The compositions of individual phases within this material have been determined with a Cambridge "Geoscan" electron probe operating at an accelerating potential of 20 kv. The experimental procedure and treatment of data have been described (1) and all analyses have been referred, either directly or indirectly, to the primary standards listed by these authors. Bulk rock compositions have been determined with classical wet chemical techniques.

Chemical analyses (2) reveal low silica values, comparatively low alumina values, and unusual abundance of titania (Table 1); iron exists in a very low state of oxidation. This may be explained either by the presence of some of the titanium in the titanous state or by interference in the ferrous iron titration, caused by the presence of sulfides. The metallic iron values accepted are based on values obtained by Bancroft from Mössbauer spectra on samples 1, 2, and 3; it is assumed at this stage that sample 4 has the same metallic iron content as analysis 1.

An attempt was made to determine the metallic iron content of these samples by subjecting them to attack by a

30 JANUARY 1970

stream of dry chlorine gas (free of HCl) in a tube furnace heated to 350°C, following the method of Moss and Hey (3) for the treatment of meteorites. The volatilized FeCl₃ was then determined by means of a silver reductor and standard ceric sulfate solution. A correction was made for the presence of FeS which is also attacked under these conditions (Table 1). These figures appear to be distinctly too high, especially so in analysis 2. The high results are thought to be due to attack of some other mineral phase present, probably an iron-titanium oxide. Tests were made on a Norwegian ilmenite which proved to be completely decomposed by the chlorine treatment. It is unlikely that the ilmenite present in the lunar samples could have been so completely broken down, for much higher results for Fe would have been obtained. It is more likely that a spinel is involved. Investigation of this matter is proceeding.

The SiO₂ was determined by dehydration with HCl and volatilization with HF. Any SiO_2 in solution after this stage was recovered from the pyrosulfate fusion of the ammonia precipitate. The Al₂O₃ was obtained by difference from the ammonia precipitate. Total iron was determined by titration with ceric sulfate solution after treatment in a silver reductor. The FeO was determined by the modified Pratt method. The MnO was obtained colorimetrically after oxidation with KIO₄. The MgO was precipitated as magnesium ammonium phosphate and ignited to $Mg_2P_2O_7$. The CaO was precipitated as oxalate and ignited to oxide. The Na₂O and K₂O were determined with an EEL flame photometer. Total H₂O was determined by the

Penfield method with $PbCrO_4$ as a flux. The almost constant value of H_2O^+ is significant; it must be held in the glass and would appear to be original water, since the samples have never been subjected to a damp atmosphere for any significant length of time. The H₂Owas obtained from the loss in weight at 105°C. The TiO₂ was estimated colorimetrically with H_2O_2 . The P_2O_5 was determined colorimetrically by the vanadomolybdate method. The Cr₂O₃ was determined by the diphenyl carbazide method (4). Sulfur was precipitated as BaSO₄ after Na₂CO₃ fusion and extraction of the cake with water, allowance being made for reagent blank. Fluorine was determined by the Sen Gupta modification of the method of Huang and Johns (5). Trace elements on these rocks are reported by Brown (6).

Detailed examinations were made of thin sections from four igneous rocks: 03/44 (type B); 17/50 (type B, but transitional to type A); 45/34 (type A); and 58/23 (type A), probably the coarsest grained rock of the entire Apollo 11 suite. In addition, grain mounts of the ferromagnesian mineral fractions from 44/43 were examined, and small xenoliths of type A rocks, some hardly altered, others thoroughly recrystallized, were studied in the breccia 65/15.

The grain size of the igneous types ranges from that of a fairly coarsegrained basalt to that of the coarsest dolerite with plagioclase crystals up to 3 mm in length. Type B rocks are holocrystalline; there is a little acid glass in the residuum of 17/50 (Table 2, analysis 13), and rather more in 45/34. Three minerals, clinopyroxene, bytownite, and ilmenite (in decreasing order of abundance) are prominent phases in all the rocks; of these, clinopyroxene predominates absolutely. In 45/34 there are up to 5 percent phenocrysts and microphenocrysts of olivine (Fa29), which are enclosed by clinopyroxene and sometimes sharply jacketed by it, suggesting a reaction relation similar to that seen occasionally in terrestrial basanitoids. In

03/44 and 58/23, coarse patches of cristobalite are found in the residuum, sometimes intergrown with a more sodic plagioclase or with little patches of an alkali feldspar. In 03/44, 45/34, and 58/23 the pyroxene is intergrown with or mantled by an iron-rich pyroxenoid. This is very unevenly distributed and has continued to crystallize until a very late stage, for it is sometimes intergrown with the cristobalite. This mineral is also described (7). Accessories are a brown spinel (picotite) that occurs as tiny octahedra enclosed in the olivine as in alkali basalts, an opaque chrome titanospinel (Table 2, analysis 7) associated with the ilmenite (Table 2, analysis 8), a few needles of rutile in the residuum, and slender prisms of apatite. Other accessories are troilite, chalcopyrite, and metallic iron, and a late spinel, probably ulvöspinel, though the composition of this phase has not been confirmed. In

the heavy residue of 44/43, one grain of an alloy of composition roughly $Cu_{70}Zn_{30}$ (brass) was found. Contamination was suspected but an additional grain of apparently the same material which has not yet been analyzed had troilite and feldspar firmly attached to it.

Textures range from subpoikilitic in 03/44 and 58/23 to intergranular in 17/50 and 45/34 where lath-shaped plagioclases separate areas composed mainly of overlapping equant pyroxene grains, pyroxenoid, and ilmenite. The texture of 17/50 recalls that of the perpendicular feldspar rock of the Skaer-gaard intrusion. In 17/50 and 58/23 Universal stage studies suggest some degree of preferred orientation in the plagioclase.

In general a single clinopyroxene phase is present, which in the coarse dolerites display remarkably little variation in 2V (46° to 42°), but probe

Table 1. Analyses of lunar rocks and C.I.P.W. norms. 1. Apollo 11, 84/14 fines; Fe 1.22 by Cl method. 2. Apollo 11, 45/24 type A; Fe 1.78 by Cl method. 3. Apollo 11, 44/39 type B; Fe 1.23 by Cl method, revised estimate of metallic Fe 0.20 percent. 4. Apollo 11, 60/25 type C; Fe 1.18 by Cl method.

	1	2	3	4
		Analyses of lunar	rocks	
SiO ₂	42.16	39.60	42.46	41.96
$Al_2 \tilde{O}_3$	13.60	9.51	10.21	11.85
Fe_2O_3	nil	0.06	nil	nil
FeO	15.34	19.12	17.60	16.51
MnO	0.20	0.28	0.28	0.23
MgO	7.76	8.10	5.96	7.63
CaO	11.94	11.07	12.25	11.38
Na ₂ O	0.47	0.36	0.48	0.49
K ₂ O	0.16	0.05	0.11	0.20
H ₂ O+	0.05	0.15	0.10	0.11
$H_2O \rightarrow$	nil	nil	0.01	0.04
TiO	7.75	11.10	9.18	9.02
P ₀ O _F	0.05	0.04	0.04	0.07
CroOo	0.30	0.47	0.21	0.31
S	0.12	0.14	0.18	0.15
F	nil	nil	nil	nil
Fe	0.60	0.20	0.60	0.60
Total	100.50	100.25	99.67	100.55
Less S -	0 0.03	0.04	0.05	0.04
Total	100.47	100.21	99.62	100.51
Total Fe	17.56	21.59	20.85	18.90
as Fe ₂	O ₃			
		C.I.P.W. norm	S	
Q	0.46	0.78	4.34	1.87
Ör	0.95)	0.301	0.65	1.18
Ab	3.98 39.46	3.05 27.54	4.06 30.09	4.15 34.88
An	34.53	24.19	25.38	29.55
Wo	10.18)	12 72)	14.67)	11.05
En	5 60 19.98	6.81 >25.04	6.89 29.17	6.03 21.70
Fs	4.21	5.51	7.61	4.62
Hy (En	13.73 24.04	13.36 10.81 24.18	7.96 16.76	12.97 9.94 22.91
Cr	0.44	0.69	0.31	0.46
TI	14.70	21.09	17 44	177 13
11	14.72	21.06	1/.44	17.15
Mt	nil	0.09	nıı	nii
Ар	0.12	0.09	0.09	0.17
Ру	0.22	0.26	0.34	0.28
Fe	0.60	0.20	0.60	0.60
Total	100.04	00 01	99.13	99 98
	100.04	77.74	77.13	

data indicate zoning from about $Ca_{35}Mg_{45}Fe_{20}$ to a subcalcic ferroaugite near Ca₂₈Mg₁₃Fe₆₀. A remarkable feature in these coarse-grained rocks is the prominence of subcalcic augite. In the basalt 45/34 and transitional rock 17/23 a different trend emerges characterized by a large variation in 2V; the early pyroxene, $2V = 48^{\circ}$, $Ca_{44}Mg_{41}Fe_{15}$ is more lime-rich and is zoned continuously from $2V = 42^{\circ}$ to 20°, OAP / / (010). From probe data the final pyroxene of 45/34 has a composition Ca₁₅Mg₅₄Fe₃₁ which is remarkably magnesian. In this rock the pyroxene fractionation appears to be following the tholeiitic quench trend (8). Analyses of pyroxenes (Table 2, analyses 1 and 2) indicate a striking reduction in the alumina and titania contents and a slight increase in manganese with fractionation. At a moderately late stage pyroxene ceased to crystallize, hence iron-rich pigeonite and ferroaugite are absent.

Exsolution lamellae were not observed optically but x-ray studies (9) indicate that they are present on a submicroscopic scale. A possible explanation of the abundance of subcalcic augite and of the absence of visible exsolution lamellae in such coarse-grained rocks may lie in the titania content of the pyroxenes depressing the solvus. Orthopyroxene is absent except possibly in a very fine-grained granulitic xenolith in one of the breccias where the original pyroxene has unmixed into two phases, one of which has low birefringence.

The pyroxenoid has the fractured appearance in thin section typical of minerals of the wollastonite group; it has also their fibrous character and a marked tendency to grow as curved crystals. Mosaic textures of interlocking grains and undulose extinction, even where the crystals are not bent, may indicate a monoclinic parent. A deeper yellow color peripherally and an associated variation in birefringence are evidence of zoning. The mineral is triclinic optically, has perfect (110) and $(1\overline{10})$ cleavages and a poorer one on (100). Twins on (100) are as common as in clinopyroxenes; in such cases the optic axial planes of the two subindividuals intersect at an angle of 5°. The angle $2V_z$ ranges from 42° to 47°, and $Z \wedge$ $[001] = 43^{\circ}$ to 45° . The optic axial plane passes within 5° of [001]. An apparently single crystal from 58/23 was found to be zoned continuously from Wo₂₉En₄₂ Fs_{29} to $Wo_{14}En_5Fs_{81}$ (Fig. 1A). The

explanation of the appearance of the pyroxenoid is not obvious, but extremely low pO_2 might suppress the formation of fayalitic olivine. The appearance of the pyroxenoid in place of iron-rich pyroxenes must imply that here we have in some way passed beyond the stability field of pyroxenes, possibly by way of a monoclinic-triclinic inversion associated with increasing iron and decreasing calcium and magnesium contents. Titania and manganese contents are significant and must have a bearing on the genesis of the mineral which seems to be related to ferrosilite III. Crystals from 44/43 examined by electron diffraction prove to be unmixed into approximately equal amounts of two phases with similar structures. These may represent an iron wollastonite and ferrosilite III.

Plagioclase crystals are flattened on (010); twinning is mostly on the albite, carlsbad, and albite-carlsbad laws; some pericline twinning was observed and an acline A twin determined in 17/50. In 58/23 a banate twin was observed enclosed in the pyroxenoid. Euler angles determined on 17/50 demonstrate it to be virtually unzoned plagioclase An₇₈, in 58/23 the composition is An₈₈, and in 45/34 where the feldspar is zoned compositions ranging from An₇₀ to An₅₉ were encountered. In 58/23 the late plagioclase intergrown with cristobalite has a composition of An₇₆; it is associated with little rectangular patches of an optically negative alkali feldspar.

In the coarser rocks two iron-titanium oxide phases are present, and these are always closely associated; in 45/34 ilmenite only was observed. Analyses of two ilmenites and a spinel from 85/48 are presented in Table 2 (analyses 8, 9, and 7). These ilmenites carry distinctly more magnesium than do those from 45/35but are otherwise similar, and all are notable for titanium being consistently



Fig. 1. (A) Atomic percentages of Ca, Mg, and FeMn in pyroxene and pyroxenoid. Zoning of pyroxene-pyroxenoid grain indicated by dashed line. $\times 2$, $\times 3$, Normative pyroxenes from analyses 2 and 3 of Table 1. (B) Plot of normative constituents of glasses. Squares represent lunar rocks 84/14, 60/25, 44/39, 45/24; dotted lines represent area enclosed by welding or coating glasses; circles represent individual glass spheres; +Ais olivine tholeiite, Mid-Atlantic Ridge; and +B is olivine plus clinopyroxene-enriched dolerite.

in excess of its stoichiometric proportion; it is likely that some is in the titanous state. The spinel is rich in chromium and titanium and carries considerable magnesium and aluminum; again some titanium may be trivalent. Exsolution of a chromium-rich phase is present in some of the coarser ilmenites.

As the norms of the analyzed samples must correspond closely with the modes it is remarkable that all show the presence of normative quartz as the silica values are so very low. Other features are the exceptionally low soda, and very high chromium in relation to the high Fe/Mg ratios. The intimate association of metal and sulfide phases indicates liquid immiscibility in a magma which must already have undergone considerable differentiation. The remarkable chemical features are clearly portrayed in Fig. 1B where comparisons are made with terrestrial basaltic types. In these surface or near-surface lunar rocks it is likely that soda, alumina, and silica may have escaped with the volatile phase; the reduced effect of gravity may account for the extreme fractionation *in situ* observed in the ferromagnesian silicates of the coarser rocks. The interstitial glass (Table 2, analysis 13) indicates the possibility of a high silica, high potassium fractionation trend.

Microbreccias 46/48, 48/42, 60/28, 65/15, and parts of 85/4 and 85/5 contain four principal components—angular fragments of basaltic igneous rocks or their individual minerals, glassy spheres, meteoritic material, and a cementing glass.

The igneous fragments show a wide variety of quench textures; individual minerals may be normal or may display shock effects ranging from the granulitization of ferromagnesian minerals and twinning of ilmenite to maskelynitization and ultimately fusion of plagioclase. In some cases the latter has recrystallized anew to feathery, twisted aggregates, in others to panidiomorphic crystals set in glass.

Some small fragments occur with

Table 2. Analyses of lunar samples. 1, Augite, 45/35/1; 2, pigeonite, 45/35/1; 3, pyroxenoid from microbreccia, 65/15.1; 4 and 5, plagioclase, 60/28; 6, maskelynite, 85/4/10.2; 7, spinel, 85/4/14.2; 8, ilmenite, 17/12; 9, geikielo-ilmenite, 85/4/10.3; 10, tachylitic glass, 85/4/5; 11, glass sphere, 85/30/4; 12, coating glass, 85/4/17a; 13, interstitial glass, 17/12 (high total due to loss of alkalis during measurement of silica).

	1	2	3	4	5	6	7	8	9	10	11	12	13
SiO ₂	48.00	52.06	46.09	50.06	43.59	44.90	.40	.05	05	40.81	41.61	41.08	78.02
TiO₂	5.91	1.35	1.22	—	_	.02	25.12	54.20	56.30	10.73	7 21	8 34	76.92
Al ₂ O ₃	4.93	.85	.75	30.60	35.38	34.78	6.23	.05	1.64	10.75	8 95	13.07	11.88
Cr_2O_3	.70	.25	.13				21.40	.60	.34	.32	48	30	11.00
FeO	8.49	19.15	37.53	.61	.32	.21	40.71	43.94	32.39	19.51	21 74	17.65	2.88
MnO	.09	.35	.61	—			.34	.43	.34	23	24	23	2.00
MgO	13.27	18.61	1.66	.26	.07	.05	5.72	.71	9 63	6 11	8 50	8 11	
CaO	19.88	6.89	10.40	15.05	19.44	18.92	.01	.10	.44	11.92	9 97	0.11	2 10
Na2O			.08	2.14	.38	.60				34	24	9.93 40	2.10
K2O				.20	.06	.08				17	.24	.40	6.86
Total	101.27	99.51	98.47	98.92	99.24	99.56	99.93	99.98	101.08	100.34	99.05	.10 99.27	103.28

30 JANUARY 1970

microporphyritic plagioclase (An₉₅) in a granulitic mosaic of plagioclase, olivine with or without clinopyroxene, and geikielo-ilmenite (Table 2, analysis 9) plus rutile, suggesting remelted rocks but not necessarily resulting from shock.

The glassy spheres range from 2 mm to $\sim 1 \ \mu m$ in diameter, the larger ones being found in 85/4 and 85/5. They are predominantly spherical but ellipsoidal and dumbbell-shaped forms also occur, suggesting oscillation of liquid drops in flight. Some are colorless, others pale green, yellow, or brown. The smaller ones are nonvesicular but, with increase in size, vesicles become more common; in one example a centrally disposed vesicle was seen to occupy some 25 percent of the total volume. Some spheres are wholly glassy apart from very occasional droplets of metallic iron which sometimes project from, or are concentrated at, the surface and may possibly have been incorporated during flight. A number contain partially resorbed relicts of any of the minerals of the igneous rocks. Others have devitrified or partially crystallized and occasionally resemble chondrules in texture but differ in their mineralogy (ilmenite with or without rutile, olivine, plagioclase).

In some specimens metallic iron droplets (with 1 to 3 percent nickel) with or without troilite are more common and occur in a cellular or contorted flow pattern similar to that found in the glass which welds together some fragments of microbreccia in the lunar fines. The presence of minute droplets at the surface of one sphere was also revealed by the use of the scanning electron microscope which showed many particles 0.1 to 0.5 μ m in diameter with an atomic number considerably greater than that of the matrix. Further examination in the electron microscopemicroanalyzer (10) indicated that the Ni, S, and Ti contents of these droplets were low and that the iron content was at least 70 percent. Two small spheres ~ 60 μm in diameter contained within the microbreccia (60/28) show a "plumpudding" structure of uniformly sized and distributed droplets and crystallites, possibly of ilmenite, which suggests a cooling rate intermediate between that which produces the very fine droplets and that which gives the chondritic texture.

The composition of some glassy spheres is plotted in Fig. 1B; measurements at different points on the same sphere vary only slightly and though the



Fig. 2. Fragment of iron meteorite from 85/4 showing kamacite with Neumann bands and α - ε iron transformation structure (\times 110).

analyses concentrate around the composition of lunar basalt and microbreccia it is much more variable than that of the coating glasses described below. This variation is probably best explained as the result of impact fusion of basaltic rocks. Alternatively, those close to lunar basalts in composition may have arisen by lava fountaining in the same way as the spheres found associated with Peles hair, or any forced discharge of hot, dust-laden, incandescent gas such as occurs in the furnaces of coal-burning power stations.

A second type of glass in the microbreccias is found molded around preexisting rock, mineral, or glassy fragments as irregular seams from 3 to 200 μ m in thickness. This glass is probably of the same origin as that which coats and welds together 1- to 5-mm fragments of microbreccia occurring in the lunar fines (85/4). It is vesicular and with a complex flow pattern marked by slight differences in color and refractive index and by trails of immiscible droplets of metallic iron (Ni < 3 percent) and subordinate troilite. The maximum size of these droplets is of the order of 10 μ m, but, in the transmission electron microscope, particles down to 20 Å diameter are visible. In total, the droplets probably amount to less than 0.2 percent of the glass and the ratio of iron to troilite is higher than in the igneous rocks, suggesting that sulfur vapor from the dissociation of troilite is responsible in part for the vesicular nature of this glass. The finely divided metal probably accounts for the dark body color of the glass which may be olive, pale brown, or pale yellow in thin section.

The composition of a typical glass is given (Table 2, analysis 12), and in Fig. 1B it is seen that these coating or welding glasses are close in composition to the microbreccias and basalts. They may be therefore considered as their direct fusion products or the introduced quench products of a basaltic magma.

In 85/4, 85/30 some glass coatings are up to 2 mm thick differing from those above only in the absence of a complex flow pattern. Their contact with basaltic rocks simulates trachylitic chilled edges but the discordant nature of the glass-ilmenite phenocryst contacts precludes such an origin. Their composition is close to that of basalt and again they must be regarded as direct fusion or introduced quench products of a basaltic magma. One fragment of which the outer surface was preserved showed several microimpact craters: these were studied with the scanning electron microscope and, in general, consisted of a central smooth crater surrounded by features controlled by steep radial and shallow concentric fractures.

Finally, occasional angular or wirelike fragments of meteoritic iron are observed in both microbreccias and fines. These are dominantly kamacite (about 7 percent Ni: 0.5 percent Co) although one fragment (Ni 7 percent) with well-developed Neumann bands and weakly developed α - ε transformation structure, includes small drops of zoned taenite (core Ni 9 percent, rim Ni 14 percent) (Fig 2).

S. O. Agrell, J. H. Scoon

I. D. MUIR, J. V. P. LONG J. D. C. MCCONNELL, A. PECKETT Department of Mineralogy and Petrology,

University of Cambridge, England

References and Notes

- 1. T. R. Sweatman, J. V. P. Long, J. Petrol. 10,
- 332 (1969). 2. J. H. Scoon (Cambridge Univ.) performed the
- J. H. Scool (Cambridge Univ.) performed the chemical analyses of the rocks.
 A. A. Moss, M. H. Hey, D. I. Bothwell. Mineral Mag. 32, 802 (1961); A. A. Moss, M. H. Hey, C. J. Elliot, A. J. Easton, *ibid.* 36, 101 (1977) (1967)
- 4. E. B. Sandell, Colorimetric Determination of Traces of Metals (Interscience, New York, 1944), p. 196
- 5. J. G. Sen Gupta, Anal. Chim. Acta 42, 119 (1968)
- (1968).
 G. M. Brown, C. H. Emeleus, J. G. Holland, R. Phillips, *Science*, this issue.
 A. T. Anderson, Jr., A. V. Crewe, J. R. Gold-smith, P. B. Moore, R. C. Newton, J. E. Olsen, J. V. Smith, P. J. Wyllie, *ibid.*, this issue.
 I. D. Muir and C. E. Tilley, *Geol. J.* 4, 143, (1964)
- P. Gay, G. M. Bancroft, M. G. Bown, Science, 9.1
- 10. P. Duncumb, The Electron Microprobe (Wiley,
- New York, 1966), pp. 490–499. 11. I. D. Muir is responsible for the petrography
- of the igneous rocks We thank the Cambridge Scientific Instrument 12. Company for use of the scanning electron microscope, P. Duncumb (Tube Investments Research Laboratory) for use of the electron microprobe microanalyzer, and our colleagues in this department for assistance
- 4 January 1970

SCIENCE, VOL. 167