

References and Notes

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Electron Microprobe Analysis of Lunar Samples

Abstract. *Plagioclase feldspar, clinopyroxene, and ilmenite in a polished thin section of a type A crystalline rock were analyzed. The clinopyroxene grains are compositionally variable, and both high Ca and low Ca phases are present. The plagioclase is compositionally homogeneous. The ilmenite is chemically homogeneous except for occasional, small areas of high local chromium concentration. Accessory minerals are: apatite (containing Cl, F, Y, and Ce), troilite, and metallic iron. Glassy spherules from the lunar soil are for the most part similar in composition to the crystalline rocks; however, some appear to have been monomineralic. The crystalline rock has apparently formed by relatively rapid cooling of a silicate melt under conditions of low oxygen partial pressure. Many components of the soil appear to have formed by meteoritic impact.*

We present here electron microprobe and petrographic analysis of Apollo 11 samples of a crystalline rock and separates from a soil sample. A polished thin section of sample 10017-16 was examined microscopically. The sample is an equigranular, crystalline rock in which most crystals are about 0.5 to 1.0 mm in largest dimension. In transmitted light, four major phases can be recognized: (i) clinopyroxene, which occurs as short subhedral crystals; (ii) plagioclase, occurring as anhedral lath-shaped crystals; (iii) opaque minerals, dominantly euhedral ilmenite; and (iv) a partly crystalline, very fine-grained intergranular mesostasis containing several phases. A few vesicles, generally

circular and about 1 mm in diameter, constitute about 5 percent by volume of the sample.

The modal mineral composition of the sample, determined by point counting (816 points, 200 mm² area), was (in percent by volume): clinopyroxene, 49.7; plagioclase, 18.0; opaque minerals, 23.9; and mesostasis, 8.3. The subophitic texture of the sample and the modal mineral composition are typical of the type A crystalline rocks described by the Preliminary Examination Team (PET) (1).

The clinopyroxene is colorless to very pale reddish-brown. The crystals do not exhibit definite pleochroism or color-zoning, and no reaction rims were ob-

served at the contacts with other minerals. There is an irregular cleavage in the clinopyroxene grains. The extinction angle $Z \wedge c$ is about 45°, thus indicating an augite. The plagioclase is apparently unzoned. The pyroxene grains are slightly fractured, but neither pyroxene nor plagioclase shows any evidence of shock metamorphism. Distinctive shock features such as intense fracturing, "planar features," or isotropization of plagioclase were not observed. In view of this lack of evidence for shock metamorphism of the major minerals, it seems clear that the mesostasis cannot be shock-melted glass and that it probably represents residual crystallization of the melt from which the sample formed.

Although sample 10017-16 does not contain olivine, it has a mineral composition consistent with the chemistry of other samples (1) and is representative of the material at the Apollo 11 landing site.

The plagioclase phase of sample 10017-16 appears generally homogeneous both between grains and within individual grains. The average composition of the plagioclase is SiO₂, 49 percent; CaO, 16 percent; Al₂O₃, 33 percent; total iron as FeO, 1 percent; TiO₂, 0.2 percent; K₂O, 0.2 percent; Na₂O, 2 percent; and anorthite, 76 percent.

In contrast, the clinopyroxenes in the sample appear heterogeneous both between grains and within grains. Most of the measured compositions center around 30 atom percent FeSiO₃, 30 atom percent CaSiO₃, and 40 atom percent MgSiO₃. Some pyroxene crystals show bands and regions of higher atomic number due to higher iron concentrations (see Fig. 1). These areas appear to be a second pyroxene phase with low calcium concentration (pigeonite?) at the boundaries of the prevalent pyroxene phase (Table 1).

Table 1. Representative composition of various phases in crystalline rock 10017 (type A).

Sample	Weight percentages as oxide									Sum
	Na	Mg	K	Al	Si	Ca	Ti	Mn	Fe as FeO	
Pyroxene		14.6		3.3	49.6	15.8	2.3		15.1	100.7
Pyroxene		15.9		3.6	49.6	17.0	2.7		9.9	98.7
Pyroxene		12.4		2.2	49.4	14.0	1.7		19.5	99.4
Pyroxene		20.8		0.9	52.0	4.1	0.8		21.5	100.2
Ilmenite		0.91		0.63	0.11		52.8	0.34	46.1	100.9
Ilmenite		0.88		0.71	0.12		51.4	0.33	46.5	100.0
Spherule (130-3)	0.2	5.0	0.1	12.5	42	14	7.4	0.18	16.5	98.4
Spherule (L-8)	0.3	11	0.1	13	46	12	2.5	0.14	15	100
Spherule (122)	0.5	13	0.16	4.5	37	8	10	0.24	24.5	97.9
Spherule (128)	3.0	13.5	0.06	7.4	43	10	0.5	0.19	21.2	96.2
Spherule (129)	0.7	5.4	0.1	24.5	47	16	0.5	0.07	5.7	100

Several apatite grains were identified. The mineral was found to contain small concentrations of Cl, F, Y, and Ce.

One part of the section contained a number of very silica-rich areas mixed with the plagioclase. The distribution of these areas is shown in the electron microprobe scanning pictures (Fig. 2).

Ilmenite is the dominant opaque phase. The grains, except for small areas of increased Cr content, are homogeneous within grains and between grains. Representative compositions of the ilmenite are shown in Table 1. The ilmenite is virtually stoichiometric in Fe and Ti, thus indicating little or no solid solution of Fe_2O_3 or Ti_2O_3 . Chromium is present but has not yet been determined quantitatively. Very small areas of high local Cr concentration occur within the ilmenite grains and near their edges. As the Cr content increases, the Ti concentration decreases markedly while the Fe level is relatively unchanged.

Small grains of troilite were occasionally observed. The troilite contained no detectable impurities, and the composition is apparently stoichiometric FeS. Metallic Fe particles 5 mm or smaller in diameter are either associated with or are included in the troilite grains (Fig. 3). The two particles shown, an iron grain and a troilite grain, occur in the area of interstitial mesostasis. Around these grains K, Al, and Si show high concentration, but Na and Ca are much lower than in the plagioclase in this rock section.

The lunar soil contains numerous distinctive glass particles, generally spherules believed to represent droplets of shock-melted material (1, p. 1219). These particles were concentrated from 4 g of lunar soil by mineral separation

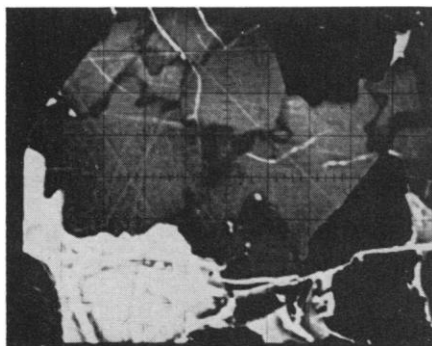


Fig. 1. Electron microprobe scan pictures of sample 10017 showing areas of higher iron concentrations in the pyroxenes. The very bright area is plagioclase, and the larger light-gray area consists of pyroxene. The darker gray bands and regions show substantial increases in iron concentration.



Fig. 2. Electron microprobe scan pictures of sample 10017, type A, showing silica-rich areas mixed with the plagioclase. (A) Target current scan: The darkest areas are ilmenite, the middle-gray areas are pyroxene, and the light-gray areas are plagioclase. (B) Fe K x-ray scan: The brightest areas are the ilmenite, and the next brightest areas are the pyroxene; (C) Si K x-ray scan: The bright areas in the lower right are due to SiO_2 .

with heavy liquid techniques, a procedure which allowed almost complete recovery of glassy spherules greater than 150 μm in diameter. A total of 107 spherules were recovered. These ranged in size from 35 μm in diameter up to an oval-shaped body 1280 μm long by 1041 μm wide. Approximately 50 percent of the spherules are 125 to 500 μm in diameter.

Nearly 95 percent of the glassy particles are spherical or oval in shape. The remainder are dumbbell-, teardrop-, or rod-shaped. Most (~80 percent) of the spherules are opaque black or translucent wine-red, and most of the remainder are brown or green. The surfaces of the spherules range from smooth to rough and pitted.

Many spherules have what appears to be a metallic coating or beads on their surface, or both. Scanning electron microscope photographs of one such spherule (320 μm in diameter) show that portions of the surface are covered with small flattened beads up to approximately 25 μm in width. Electron probe microanalysis shows that the beads are predominantly iron (95 ± 2 percent), with 3.5 to 4 percent approximately 0.3 percent Co and 0.15 percent P homogeneously distributed.

Ten spherules were analyzed by electron probe microanalysis. The compositions of some of the spherules included in Table 1 are similar to those reported for lunar rocks and soil. These spherules were mainly deep red or brown in color. One of these spherules contained over 15 metallic inclusions less than 10 μm in diameter consisting of iron containing 1 to 2 percent Ni. The amount of TiO_2 in the host glass was anomalously low (2.5 percent).

The remaining three spherules had different composition from that of the

majority (Table 1). One spherule, No. 122, contained anomalously low concentrations of Al_2O_3 and high concentrations of FeO (24.5 percent) and TiO_2 (10 percent). Another, No. 129, has a composition close to that of plagioclase. The third, No. 128, is characterized by low amounts of Al_2O_3 and high concentrations of FeO and MgO.

Four angular glass particles were analyzed. These were for the most part isotropic, ranged from translucent to opaque, and contained weakly, birefringent lath-like inclusions. All four angular fragments were heterogeneous, with compositions similar to that of the lunar rocks and soil. The presence of Ni-Fe spherules in the glassy spherules is strong evidence for impact origin.

Two types of metals were investigated: (i) metallic areas associated with particles separated magnetically from soil sample 10084 and (ii) a metal fragment extracted from a breccia rock 10046-18a.

Several particles were extracted from

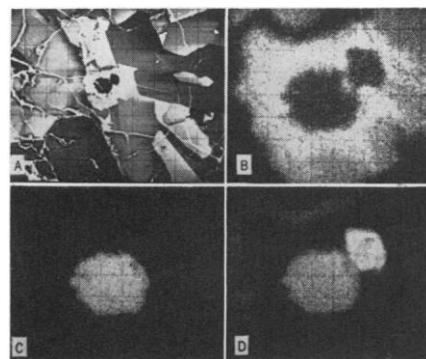


Fig. 3. Electron microprobe scan pictures of sample 10017 showing a troilite and an iron bleb and the distribution of K around these blebs. (A) Sample current scan; (B) K x-ray scan; (C) S x-ray scan; and (D) Fe x-ray scan.

the soil with a hand magnet. Some are melted, spherical in shape, and composed mainly of glass. Others are vesicular and complex in shape and contain large cavities and fragments of crystalline rock.

Scanning electron microscopic examination showed that metallic areas occur both within and on the surface of the soil particles. The metal content varied greatly from one particle to another. The metal inclusions in the soil particle are small ($< 30 \mu\text{m}$) and often rounded. They are swathed in troilite, and their microstructure indicates shock-hardening.

Two metal areas, less than $10 \mu\text{m}$ in the largest dimension, were analyzed. One contained 13 percent Ni, 85 percent Fe, approximately 0.9 percent Co, and 0.4 percent P. The other contained 10 percent Ni, 85 ± 5 percent Fe, approximately 0.6 percent Co, and 0.35 percent P.

Another rounded metallic phase in a frothy complex soil particle is composed of essentially Fe and 0.4 percent Co. Nickel and P were not detected.

The metallic sample from the breccia is irregularly shaped and about 0.7 mm in its longest dimension. In a polished section, it exhibits a eutectic-like structure consisting of a very fine dispersion of phosphide and metal with lesser amounts of sulfide and carbide.

Extensive electron microprobe analysis of this particle shows the following. The metallic area surrounding the eutectic structure consists mainly of Fe with about 2 percent Ni, 1 ± 0.2 percent P,

and no Co. The eutectic-like area contains on the average 6 ± 1 percent Ni and 1 to 1.5 percent P with small amounts of S and C. Electron microprobe scans show a strong correlation between Ni and P in the eutectic with the highest Ni region adjacent to the eutectic area. The carbides contain about 6.5 percent Ni and up to 8 percent P. The sulfides also contain Ni in solution. The amount of Ni in the phosphides $[(\text{Fe-Ni})_3\text{P}]$ was 10 percent as contrasted to that of the adjacent metal with 6 ± 1 percent Ni and less than 1 percent P.

Our findings bear out the suggestion made by PET (1) that the terrain from which these samples were collected is internally generated igneous rock overlain by an impact-formed breccia with some meteoritic material.

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Mineralogical and Petrological Investigations of Lunar Samples

Abstract. *Fragments of igneous rocks and breccias, and one coarse-grained rock with thin sections, have been studied. Minerals found include pyroxene, plagioclase, olivine, ilmenite, troilite, ulvöspinel, native iron, cristobalite, tridymite, alkali feldspar, apatite, and quartz. Textures are described and interpreted. Among features revealed by optical, microprobe, x-ray diffraction, and electron microscope methods are extreme zoning and unmixing in pyroxene grains, compositional variations in ilmenites, and effects of shock metamorphism. Some trace elements were determined by x-ray fluorescence analysis.*

Work we have carried out so far has been primarily on particles and dust (10085,2) from the 1- to 10-mm fraction of the bulk sample, and on rock 10044 (rock specimen 10044/43, covered section 10044/42, polished section 10044/41). In this report specimens from 10085,2 are given the prefix B followed by our own laboratory serial number.

In the igneous rocks grain size has

been found to vary from one rock fragment to another, and associated with varying grain size there tend to be characteristic textures. No phenocrysts have been observed in any rocks. The coarsest rock examined (10044/43) has equant pyroxenes up to 2 mm across, skeletal-to-acicular ilmenites up to 2 mm, and plagioclase laths up to 1.5 mm in length. With decreasing grain size, the ilmenites change from

skeletal (up to 0.5 mm across) through acicular grains and a dendritic variety to equant ilmenites ($\sim 0.1 \text{ mm}$) in the finest rock. These changes are paralleled by the plagioclases. The pyroxenes, on the other hand, are anhedral and subophitic. Within the range outlined, six distinct textural types have been seen: (i) medium-grained equant (up to 2 mm): 10044/41, B4; (ii) acicular plagioclase and ilmenite with anhedral pyroxene of medium to fine grain size (up to 0.5 mm): B3, B8, B9; (iii) fine-grained rock (up to 0.15 mm) with characteristic sets of thin subparallel ilmenite lamellae: B1; (iv) granular fine-grained rock (up to 0.1 mm): B5; (v) rock characterized by plumose intergrowths of plagioclase and pyroxene (0.25 mm) resembling chondritic texture: B18a; (vi) vesicular glass with rounded ilmenites: B18b. The principal minerals in types i to v are the same, although the proportions vary (see Table 1).

The modal analyses of the different igneous rock types (Table 1) show a remarkable consistency in pyroxene content, an inverse relation between plagioclase and ilmenite, and an increase in ilmenite content with decreasing grain size. A very high content of ilmenite is notable in all of the rocks.

Among breccias two distinct and several intermediate types can be distinguished. The first consists of fragments of igneous rocks, single mineral grains, and a matrix of fine-grained dust. Ilmenites in these breccias frequently show evidence of shock. The second type of breccia is predominantly glassy, containing angular fragments of low-reflectivity glass in a glassy matrix of slightly higher reflectivity. Specks of nickel-iron up to $40 \mu\text{m}$ in diameter have been seen. This breccia may be the product of more severe shock metamorphism. A spherule from within it has the composition $\text{Fe}_{94.0} \text{Ni}_{4.8} \text{Co}_{0.9}$, which is similar to that of a spherule from impactite glass from Lake Bosumtwi Crater, Ghana (1). No oxides or sulfides are present in the breccia.

Intermediate between these two types are breccias consisting of varying proportions of glass as well as mineral and rock fragments. Vesicles have been observed in many of these breccias. Flow textures occur in some of the glassy portions. Native iron occurs both as minute spherules and as larger (up to $50 \mu\text{m}$) fragments with serrated rims.

Ilmenites in various rock types were surveyed. They display minor variations in $\text{TiO}_2:\text{FeO}$ ratios within single rock