Micromorphology and Surface Characteristics of Lunar Dust and Breccia

Abstract. Although nothing of direct biologic interest was observed in the sample studied, small shaped glass particles and glazed pits resemble objects which elsewhere have been described as fossils. These features, although nonbiological, do bear on processes of lunar weathering and outgassing. The glazed pits are impact features. Fusion of their surfaces released gases. Electron microscopy of the glasses, pits, and angular microfractured mineral grains indicates a prevalence of destructive weathering processes—thermal expansion and contraction, abrasion by by-passing particles, and, of course, impact.

Our sample consisted of 7.4 g of fine black surface dust (sample 10086,15), 0.92 g of fragments from the interior of a breccia block (10091,4), a single 1.22-g fragment showing an external surface of the breccia (10091,5), four thin sections (10019,15; 10046,56; 10061,27; 10065,25), and three probe mounts (10059,32; 10059,37; 10067,28).

Both breccia and fines consist of a matrix of mostly very fine grained, mainly angular particles of mineral, rock, and glass, within which are scattered larger mineral and rock fragments as well as spheroids and fragments of glass. The main difference is the induration of the breccia, which shows a strong (shock?) coherence between grains and probably some welding between glass particles. Point counts show the order of abundance to be glass, rock fragments, plagioclase, pyroxene, ilmenite, olivine; but identification of minerals and estimates of their abundance and chemistry were not the goal of this study.

Our mission was to search for evidences of present or former life on the moon and, failing that, to consider aspects of the lunar sample that might



Fig. 1. Size distribution of glass spheroids from Apollo 11 thin sections 10059,37; 10061,15; 10061,27; and 10067,28.

bear indirectly on the question of life processes.

Toward this end a variety of sample preparations was employed, resulting in end-products that were examined under varied modes of light microscopy, by scanning and transmission electron microscopy, and in the mass spectrometer. In addition to the study of optical thin sections prepared in Houston, our procedures involved the preparation of strewn slides of the dust, both before and after HCl-HF maceration; the preparation and vacuum-coating of standard plug mounts, grids, and replications of dust and rock fragments for electron microscopy; and the fusion (under vacuum) of dust at temperatures above 1200°C, followed by studies similar to the above and by mass spectrometry of volatiles obtained. Techniques employed are familiar or obvious ones.

As it became evident that our sample contained neither life nor fossils, interest focused on the abundant tiny glass spheroids and ovoids which, if carbonaceous, could be mistaken for microfossils. We also studied features indicative of outgassing and weathering processes (by techniques of reference (1) which might suggest the former presence of a lunar atmosphere in which biogenic or prebiogenic processes could have occurred.

Although glass appears to be the most abundant component of our sample, it is hard to estimate just how much there is. The minute size of many of the matrix grains, the degree of overlap in thin section, and the abundance of opaque minerals make it difficult to employ standard point-count procedures with confidence. Nevertheless, it seems that from 20 to 50 percent or more of the total dust and breccia is glass. This glass has an index of refraction mostly greater than 1.64, implying a density greater than 2.94 (2). Its color ranges from dark amber through yellow to clear, presumably varying with iron content. Small glass particles adhere to a weakly magnetized knife blade. Some are embayed, fractured, crazed, cracked, and locally devitrified, and they adhere to surrounding mineral particles as if hot at the time of deposition. One dumbbell-shaped fragment consists of two fused glass spheres, implying the possible significance of fusion of hot glass as a cementation mechanism.

What is of interest here, however, is that between 1 and 5 percent of the total rock consists of mostly tiny spheroids, ovoids, and other shaped particles of glass (Figs. 1-3). The abundant spheroids and ovoids are similar in shape to some algal and bacterial unicells, and the smaller ones are comparable in size. Indeed, were such glassy particles to be encased in carbon and then dissolved or altered (as might happen in a parent body such as that of the carbonaceous chondrites), they would make impressive pseudomicrofossils.

The size distribution of the glass spheroids in fact resembles the mortality curves of some microorganisms, ranging from diameters of well under 1 micron to 392 microns in populations counted and skewing so sharply toward smaller sizes (Fig. 1) as to suggest that, with better statistics in the smaller grain sizes, the curve would approach both x and y axes asymptotically. Indeed, examination of the lunar dust under the scanning and transmission electron microscopes shows a multitude of submicron sized spheroids going right down to a few tens of angstroms. Such a size distribution, however, is not like that usual for the morphologically simple procaryotic unicells which the glass spheres resemble in a crude way, but rather like the mortality curves of sexually reproducing microorganisms. This is not to propose that there are or were solid glass Protozoa on the moon but to add one more warning to the many that have already been given about a too-ready interpreta-



Fig. 2. Size distribution of glass ovoids from Apollo 11 thin sections 10059,37; 10061,15; 10061,27; and 10067,28.

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tion of exotic objects as of vital origin on the basis of gross morphology alone.

The lunar glasses also contain many internal structures of interest bubbles, "organelle"-like mineral inclusions, fine tubular structures, and microlites such as are well known in natural glasses on earth and some of which make rather spectacular pseudofossils (3). Some of the glasses involve complex composite structures.

The warning deserves emphasis. Elsewhere on the lunar or Martian surface may be lifelike artifacts that will be harder to discriminate from the real thing.

In addition to the glass observed as shaped particles and fragments, crusts of glass partly envelop rock or mineral particles, completely envelop dust aggregates of different shapes (including spheres), and glaze the surfaces of small rimmed pits or microcraters (Fig. 3, i–j). Such glazed surfaces, moreover, show bubble aggregates indicative of outgassing as well as local microfracture patterns consistent with origin of the microcraters by impact.

Although less than 1 square centimeter of external surface (10091,5) was available for study, examination of this and of smaller fragments from a dust sample (10086,15) revealed a number of roughly circular depressions with raised rims and glazed surfaces (Fig. 3, i-j). Pits observed range from 5 mm to 50 microns in diameter, average 0.5 mm, and occur with a frequency of about 10 per square centimeter. Curiously, no overlapping of such pits was observed. Examination by scanning electron microscopy shows these glazed surfaces to be pitted with innumerable smaller pits and bubblelike features, many of which contain still smaller "bubbles." The bubbles, both large and small, usually also have slightly raised margins, but they also commonly show their largest diameter beneath an overhang, are preferentially located on elevated surfaces, and are clearly burst bubbles and evidence of outgassing.

The glazed surfaces of the larger pits within which the bubbles are located are only a few microns thick, and the glazing extends the full width of the rim, sometimes bubbling over slightly onto the unglazed surrounding rock. Subconcentric microfractures may parallel the margins between glazed pit and unglazed rock, and microfractures within the glazed areas run between the bubbles with no apparent displacement. The sides of the pits are occasionally



Fig. 3. Shaped glass particles and glazed microcraters from Apollo 11 dust and breccia. (Bar scales give magnification as indicated. Photographs b-g with optical microscope, a and h-j with scanning electron microscope). (a) Glass ellipsoid with terminal nipples from dust sample 10086,15. (b) Large composite glassy ovoid including matrix material, circular voids (where spheres ripped out during preparation) and glassy spheroids within a framework of wispy glass. Thin section 10019,15. (c) Section of a spheroid showing glassy rind around matrix and mineral core. Thin section 10019,15. (d) Slightly oblate and fractured glass spheroid with nipple on upper end. Thin section 10019,15. (e) Glass spheroids surrounded by fine grained matrix. Thin section 10019,15. (f) Dumbbell-shaped glass object with bubbles and mineral inclusions. From strewn slide of dust sample 10086,15b. (g) Glass spheroid with textured surface. Slide 10019,15. (h) Glass spheroid with protuberances and concentric flanges. Grain in dust sample 10086,15. (i) Margin between glazed pit (at right) and unglazed portion of microbreccia (at left) showing burst bubbles with overhanging rims. Surface of chip picked from dust sample 10086.15. (j) Crater-like glazed pit showing terracing and bubbles. External surface of breccia sample 10091,5.

terraced (Fig. 3, j). Depth is roughly proportional to pit diameter. The bubbles within the pits tend to cluster and have rounded bottoms as well as (commonly) overhanging edges. They are comparable to the bubbles of scoriaceous terrestrial basalts.

We interpret the glazed pits as impact craterlets. The bubbles are just that —the product of outgassing resulting from high-velocity impact and local fusion of the lunar surface. The gas may have come from the projectile itself (for example, cometary particles) or, less likely, from fusion of the lunar breccia on impact. The subconcentric fractures at the margins, as well as the terraces within some of the glazed pits, were probably caused by the initial impact.

On the "biological" side these glazed pits are comparable to some impressions that have been described as pre-Paleozoic fossils. Of more interest, however, are their implications for a lunar atmosphere.

We wanted to know the source of the gas evaded during surface fusion and whether the glass spheroids and other shaped particles observed could be derived from fusion of local materials. With the help of Mark Stein, therefore, a small quantity of the lunar dust was fused under vacuum in a tantalum crucible after outgassing to 10⁻⁷ torr. Fusion took place at 1200° to 1400°C, producing glass spheroids having identical appearance and physical properties to those of the lunar dust and breccia. Vigorous bubbling during the fusion process indicates a sufficient quantity of indigenous volatilizable materials to account for the observed vesiculation of the glassy crusts, although very little of this remained gaseous at room temperature.

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Analysis of Lunar Material for Organic Compounds

Abstract. A sample of lunar material from Apollo 11 was subjected to analysis by several techniques, which included mass spectrometry, gas chromatography, liquid chromatography, and nuclear magnetic resonance and their variations, in an effort to detect the presence of organic compounds. None were found. On the basis of the sensitivity ascribed to certain of the methods employed, it is assumed that if organic matter were present it would exist in concentrations less than 1 part per million.

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Three sample-handling procedures were utilized during efforts to determine the existence of organic compounds in the Apollo 11 lunar sample 10086.5 (class D). First, an aliquot of untreated sample was directly analyzed by gas chromatography and mass spectrometry. Second, the sample was subjected to extraction by certain organic solvents (1), the solvents were removed, and portions of the residue, if any, were analyzed by gas chromatography, mass spectrometry, and nuclear magnetic resonance. Third, an aliquot of the sample was acid-hydrolyzed, and the hydrolyzate was extracted and subjected to analysis by high-performance liquid chromatography on pellicular resins, by gas chromatography, and by mass spectrometry.

All solvents were of spectroscopic grade and were redistilled when necessary. A series of blanks was run prior to assay of the material in all instances.

For the nuclear-magnetic-resonance analysis a 23-g sample of lunar material was placed in an extraction thimble and extracted with a solution containing 80 ml of a 4:1 benzene-methanol (spectrograde, redistilled solvents) mixture in a Soxhlet extraction apparatus for 24 hours and then was stirred as a slurry for 24 hours. The contents were decanted into four test tubes and centrifuged for 2 hours. The supernatants were combined, and an aliquot was evaporated to dryness in a stream of pure nitrogen. The contents were triturated 18 hours with 0.8 ml of spectrograde carbon tetrachloride, and the carbon tetrachloride solution was added to a Wilmad Imperial 507PP sample tube (5 mm). A small amount of Matheson tetramethylsilane was added as a reference signal for the field-frequency lock. A blank was determined with identical materials and apparatus.

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The nuclear-magnetic-resonance study was conducted with a Bruker HFX-3 spectrometer operating at 90 Mhz (H¹). The sensitivity was increased by use of a 4096 channel computer of average transients (CAT). The spectrum was determined over a sweep range from 30 to 1166 hz downfield with tetramethylsilane. The sweep speed, determined by the CAT, was 80 msec per channel, and the signal was filtered with an input integrator (time constant = 80 msec per channel).

After 27 hours, the spectrum showed peaks at 133 hz and a multiplet at 305 hz. The multiplet was also present in the blank and is a solvent impurity. No other peaks were present. The peak at 133 hz was probably due to the intense tetramethylsilane peak due to repeated scans by the signal-averaging device as evidenced by the spectrum of the blank.

The sensitivity of the Bruker HFX-3 spectrometer for a 1-percent (by volume) ethylbenzene solution, using a 5mm sample tube, is given as a 50-to-1 signal-to-noise ratio. The CAT further increases the signal-to-noise ratio by a factor of 40. Therefore the sensitivity of a sample with the same molecular weight and number of hydrogen atoms as ethylbenzene will be approximately 5×10^{-4} . Any material present in a concentration of less than 1×10^{-4} will be difficult to detect.

An analysis of the sample extract was made with a high-performance liquid chromatography system (2) equipped with an ultraviolet detector. With this technique, minute quantities of substances, such as nucleic acid constituents, which have absorbtivity at 250 nm, can be separated and determined.

Ten grams of the finely ground lunar sample were digested with 50 ml of 2.5N hydrochloric acid at 50°C for 2 days. After centrifugation the extract was lyophilized. Then 1 ml of distilled water was added to the vessel in order to prepare the sample solution for the chromatographic investigation. Another portion of this sample solution was then processed for analysis by mass spectrometry and gas chromatography.

The LCS 1000 liquid chromatograph (Varian Aerograph) with 1-mm inside-diameter columns packed with pellicular cation exchange or anion exchange resins (particle diameter 50 μ m) was used in a fashion described earlier (2). The length of both columns was 150 cm. Dilute potassium hydrogen phosphate solutions having different pHvalues were used as eluents. In some experiments gradient elution (concentrated KH_2PO_4 served as strong eluent) was employed. The flow rate was 10 ml/hour, and the column temperature was 60°C in all experiments. A 10- or 20- μ l sample solution was injected onto the chromatographic columns with a Hamilton No. 701 microsyringe.

Chromatographic runs were made on both cation exchanger and anion exchanger columns with eluents of various pH values in the range 2 to 7. No peaks attributable to retarded solutes have been found. In gradient elution with both columns small peaks appeared regularly at a certain point of the chromatogram, but these peaks were also obtained when a solvent blank was analyzed. Thus it is concluded that under the experimental conditions described no nucleic acid constituents or ultraviolet-absorbing materials of similar chromatographic behavior have been extracted from the lunar sample in an appreciable quantity. Considering the sensitivity of the technique, therefore, the amount of individual nucleic acid constituents, if such were present, must be less than 10⁻¹⁰ mole per gram of sample.

For the gas-chromatography analysis a 25-mg sample of the lunar material without prior treatment was placed in a small quartz tube containing quartz wool and attached to a supported coated open tubular (SCOT) column, 15.1 m long with a 0.058-cm inside diameter, containing silicone fluid (SF96, 1000 cs) and Igepal CO-880 (nonylphenoxy-polyethyleneoxy-ethanol). The column was connected to a flame ionization detector (sensitivity 10⁻¹² g/sec), and the entire system was