which confirms the textural relations.

The density and logarithm of viscosity of silicate melts are approximately linear functions of composition, and published density and viscosity data for simple silicate systems allow one to calculate these properties for natural lavas with accuracies comparable to those of current measurements (*7*). We calculate the density of sample 10022 liquid to be 2.93 to 2.96 g/cm^3 between 1400° and 1250°C. The viscosity varies from 6 to 27 poises over the same temperature range, which is comparable to the viscosity of glycerin at room temperature. The low viscosity [the viscosities of olivine basalts (*8*) range at least an order of magnitude higher] is related to low concentrations of Si and Al and makes it likely that, for comparable thermal histories, lunar lavas, in spite of lower gravity, are capable of faster and more extensive flow than their terrestrial counterparts. In view of the large areal extent of plateau-type lavas on earth, it is not difficult to imagine lunar lavas flowing, topography permitting, over areas comparable to mare basins. The pronounced fluidity might also result in the formation of long lava tubes which could form sinuous rills when the upper crusts are breached. The large density contrast between ilmenite and the liquid, the long ilmenite crystallization interval, and the low viscosity of the liquid would favor ilmenite-liquid separation wherever lavas were ponded to any depth. The igneous rocks of the Sea of Tranquillity may owe their high ilmenite content to such a mechanism, and mascons may be related to concentrations of dense, early-crystallizing solids in the central portions of the basins of the deeper circular maria. In any case, such reasoning leads one to expect that the ilmenite (and therefore Fe and Ti) content may be quite variable over the lunar surface.

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1. Standard analytical conditions: Applied Research Labs electron microprobe x-ray analyzer; 15 kv; sample current, 30 to 50 × 10^{-9} amperes; 1- to 2-μm beam; 10-second counting interval. When convenient, Ti through Ni were analyzed at 20 kv. The counting interval was increased for minor elements, and a larger beam was used in alkali-rich areas. Drift, dead time, background, absorption, generation, and fluorescence corrections were applied to all analyses. Details of all analyses are available on request.
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4 January 1970

High-Voltage Transmission Electron Microscopy Study of Lunar Surface Material

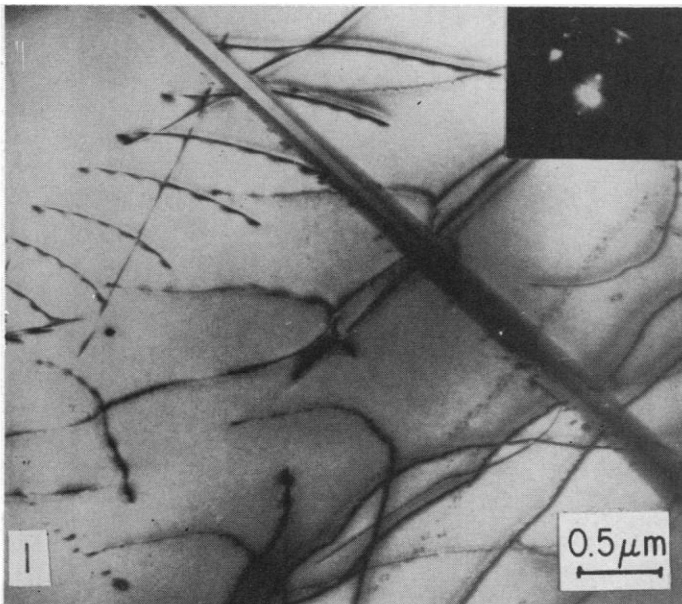
Abstract. *The internal substructures of a type B sample have been examined at high magnification and compared with terrestrial rocks. Selected ultrathin sections were prepared from these multiphase materials by an ion-thinning technique and examined in a 1-Mev electron microscope, with complementary optical analyses. The structures in the ilmenite and plagioclase indicate that the lunar material has undergone plastic deformation by dislocation movement and possibly microtwinning, with subsequent recovery. The pyroxene exhibits complex lamellar structures of submicron spacing. These various observations are consistent with the optical microscopy evidence for distortion and recovery and identify the processes involved.*

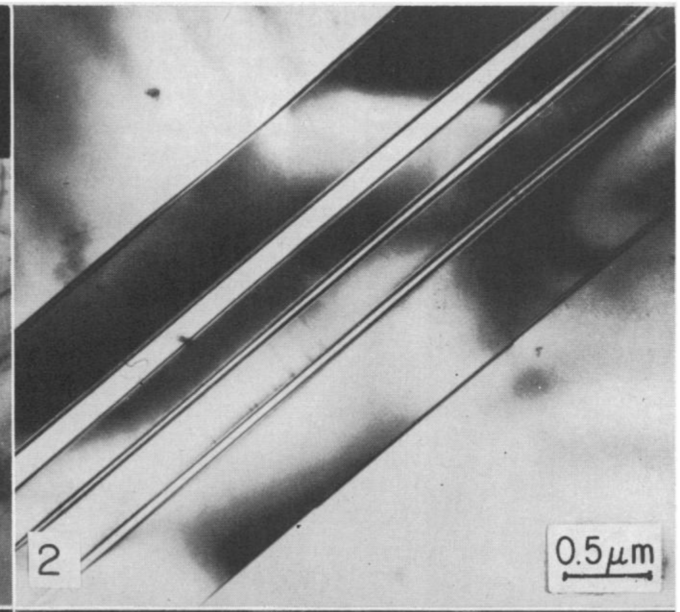
The use of transmission electron microscopy to study the fine-scale substructure in thin foils of metals and some ceramic materials during the past decade has been of considerable value in elucidating various structure-history and structure-property relationships (*1*). However, with few exceptions, this technique has not been applied to geological materials because of the difficulty of preparing suitable electron-transparent foils from multiphase nonmetallic solids. Previous electron microscopy studies of geological importance appear to have been restricted to a few single-phase rock-forming minerals (*2*, *3*), with thin foils usually prepared by mechanical cleavage. The latter method has the major disadvantages of uncertainty as to the location of the fragment in the original specimen and the possibility of introducing artifacts.

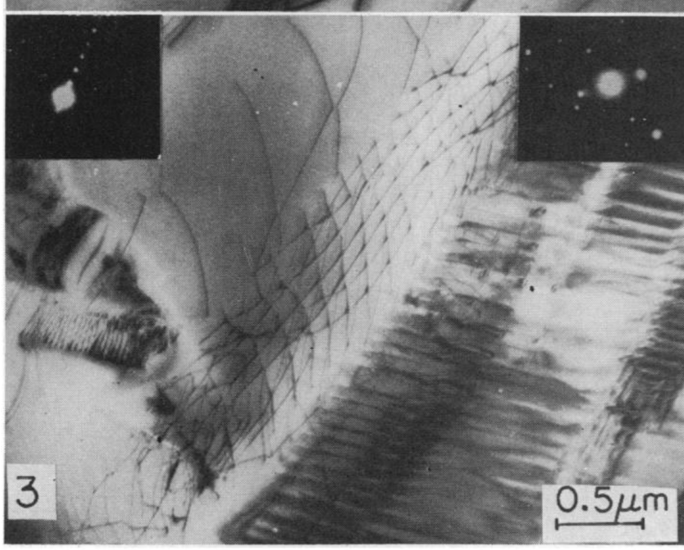
Although the potential of ion-bombardment thinning to prepare foils for electron microscopy was recognized some 20 years ago (*4*), only recently has apparatus capable of producing glow-discharge plasma beams that are stable over long periods of time been perfected (*5*). Such apparatus can successfully thin metals and ceramics and has now been shown to be suitable for a number of geologically important materials (*6*). However, the foil thickness of a few thousand angstroms required for conventional (100-kv) transmission microscopy results in a delicate and friable specimen which is easily broken and destroyed in handling. Consequently, the recent advent of high-voltage microscopy offers a major advantage for the study of geological materials because the specimen thickness can be increased to the order of 1 μm, which facilitates both preparation and handling. Furthermore, the higher voltage permits increased resolution by reduction of chromatic aberration, increased precision of selected-area electron diffraction, and reduction of the possibility of specimen damage in the microscope from ionization and beam heating effects.

The combination of ion thinning and high-voltage microscopy has particular value for the study of the substructure of lunar material, in that wastage and loss during specimen preparation of the limited amount of available material is minimized. The present paper describes the application of these new techniques to the study of the substructure of a sample of type B lunar rock (Preliminary Examination Team Report sample 10029-1) with the objectives of comparing the principal characteristics with those of analogous terrestrial material to assist in elucidating the mechanical and thermal history of the lunar material. This study is the first in a program of electron microscopy observations of the internal structure of lunar surface material brought back from the Apollo 11 and 12 missions.

The specific techniques used for preparing and examining the 10029-1 sample were developed on specimens of terrestrial rocks chosen to simulate the lunar material as closely as possible. The initial series of simulation specimens included several Hawaiian basalts, together with pyroxenite and anorthosite. After the release of chemical analyses and







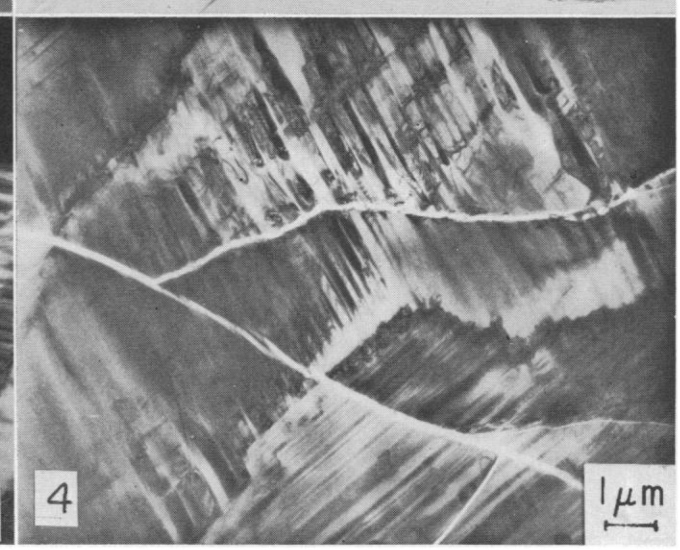


Fig. 1. Transmission electron micrograph of ilmenite, showing deformation twin and slip dislocations. Apollo 11 sample 10029-1; 800 kv; × 19,600. Fig. 2. Transmission electron micrograph, showing deformation twins in plagioclase. Apollo 11 sample 10029-1; 800 kv; × 19,600. Fig. 3. Transmission electron micrograph, showing interface between pyroxene and plagioclase phases. Apollo 11 sample 10029-1; 800 kv; × 19,600. Fig. 4. Transmission electron micrograph of pyroxene, showing characteristic substructure and cracking. Exsolution lamellae occur along several intersecting crystallographic orientations. Apollo 11, sample 10029-1; 800 kv; × 5880.

petrographic information in the Apollo 11 Preliminary Examination Team report, specimens of diabase (Ironwood, Michigan, and Beaver Bay, Minnesota) were examined as more closely resembling the lunar material. The examination sequence finally adopted to correlate large-area studies at low magnification by optical petrography with high magnification electron petrography is the following. Standard thin sections of a given specimen are examined in the optical microscope to identify the phases present, their textural relationships, and evidence for deformation or chemical zoning. Areas of interest for more detailed observation are then selected and 3-mm diameter discs cored out (with the features of most interest in their central region) ultrasonically. Each disc is then mounted on an alumina ring (approximately 0.25 mm thick) to provide mechanical support for direct insertion in the electron microscope and other handling. The disc is next reduced in its central region to a thickness of about 1 μm by bombardment with argon-ion plasma beams from both sides. In this procedure, the specimen is positioned in a high-vacuum chamber between two plasma guns and rotated about an axis at an angle of 75° to the plasma beams. With the guns operated at 6 kv and a beam current of 50 μA, a suitably thin section (indicated by the development of perforations) is prepared in 15 to 20 hours. Although the resulting specimens are too thin to show interference colors between crossed polarizers, a careful photographic comparison is made before and after thinning (with a large-scale montage or map of the complete specimen made at the latter stage) to permit exact identification in the electron microscope of specific features examined optically. The specimen is then transferred to a specially designed holder and examined by transmission electron microscopy. The high-voltage machine used is that at the 1-Mev facility completed recently at the U.S. Steel Research Center (*7*). Experiments with the various geological materials mentioned earlier have shown that no radiation damage under the high-voltage electron beam is apparent in any of the minerals observed. The only mineral in which such damage has been found to occur to date is quartz (*3*). On the basis of these experiments, an optimum operating voltage of 800 kv was selected for the examination of the lunar material, with a specimen beam current of 1 μA. Apertures to permit the selection of areas down to 0.1 μm in diameter at the specimen for electron diffraction were used in conjunction with a two-circle goniometer stage fitted with an anticontamination device to permit prolonged observation for diffraction-contrast experiments.

The three major minerals in the medium-grain-sized 10029-1 rock sample are clinopyroxene, calcic-plagioclase, and ilmenite, in order of the volume fraction present and in agreement with the preliminary analysis for type B rocks given in the Preliminary Examination Team Report. Optically, the major features of the lunar samples are (i) a more general cracking (irregular and curved cracks and cleavage) of all minerals than is normally found in terrestrial rocks (this is consistent with mild shock damage from meteorite impact), and (ii) a wide variety of features in the pyroxenes, including growth twins with [001] axis and planar composition surfaces parallel to (100) planes (common in terrestrial rocks), marked misorientations within many of the single grains (subgrains and kinking, with kink boundaries nearly perpendicular to [001]), and lamellar structures or striations defined by zones of different interference colors. The latter features are of variable orientation but appear close to the (001) plane and parallel to the *a*-axis—they are not fully resolvable optically but could be interpreted as deformation bands, twins, or arrays of slip dislocations from pencil glide. Despite the optical evidence of deformation, the general absence of visible "damage" in the subgrains suggests that some recovery occurred subsequent to or coincidental with the deformation.

The rates of ion thinning of these major minerals were found to differ sufficiently (decreasing in the order plagioclase, pyroxene, ilmenite) that the specimens were best examined at successive stages of thinning for large-area observation of desired phases in the electron microscope. These differences exit in both lunar and terrestrial materials, but the controllability of the thinning process appears to be easier for the lunar specimens (this may be due to the absence of hydrated phases—a particularly prominent structural difference between the lunar rock and the diabase). The optically opaque ilmenite was observed in electron transmission to contain substantial arrays of slip dislocations and some microtwinning (Fig. 1), together with an occasional subgrain. The latter probably originated in the original crystal formation, since the dislocation arrays show little evidence of rearrangement other than the existence of small loops (Fig. 1). In contrast to the ilmenite, the principal feature of the plagioclase is extensive microtwinning (Fig. 2). The twins, at least some of which are of the deformation type, appear in groups or bundles and may correspond to the "lamellar twinning" parallel to the long dimension of the lath and plate-shaped crystals observed optically. The less extensive arrays of dislocations in the plagioclase exhibit rearrangements, including the development of networks corresponding to low-angle boundaries (Fig. 3), indicative of recovery. None of the occasional compositional zoning observed optically was found, nor any fine-scale exsolution structures, such as have been noted in recent electron microscopy studies of K-rich feldspars (*8*).

The clinopyroxene differed from both the other minerals in that no evidence of slip dislocations was found in electron transmission. The principal structural feature observed was an extensive and complex pattern of parallel striations on a submicron scale (Fig. 3). A more illustrative example of this structure in a single pyroxene grain is shown in a low-magnification composite electron micrograph (Fig. 4). Selected-area electron diffraction measurements failed to detect any misorientation between individual striations or across the "domain" boundaries shown in the micrograph (*9*). The appearance of the striations changes markedly as the diffraction contrast conditions are altered (compare Figs. 3 and 4), and any dislocations present may be obscured by this structure. The domains are on a much smaller scale than the subgrains observed optically, and none of the latter have yet been observed in the areas examined. The terrestrial pyroxene in the diabase contained more complex and irregular striated structures. The optical measurements made on the lunar pyroxene are consistent with the composition of pigeonite or its extension into the augite field (subcalcic augite). In terrestrial volcanic rocks, pigeonite is commonly zoned between Ca-rich and Ca-poor regions; some evidence for such zoning was observed in the lunar sample. Electron-microprobe evidence for a fine-scale Ca-zoning in bronzite was recently reported (*10*). In view of these indications, it appears likely that the striations in the lunar rock arise from a submicron scale exsolution and that these fine striations are responsible for the "lamellar structures" observed optically. Additional features observed were precipitate particles in both the plagioclase and pyroxene (of the order of a micron in size and as yet unidentified), and some evidence of highly localized "black-spot" radiation damage in the plagioclase.

It is concluded from the above results that the lunar sample 10029-1 has been lightly deformed and simultaneously or subsequently heated sufficiently to permit recovery. This electron microscopy evidence is consistent with the optical observations and provides more specific knowledge of the structural features. This preliminary study also shows that high-voltage microscopy provides a significant addition to the field of petrography.

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8 January 1970