

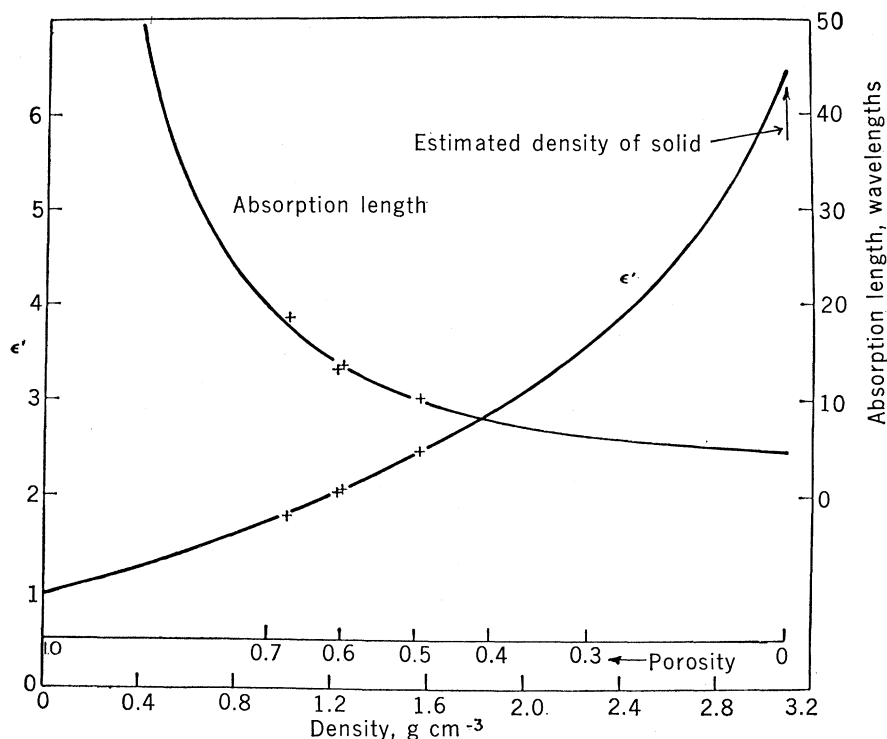


Fig. 4. The dielectric constant and absorption length of the bulk sample at 450 Mhz as a function of the powder density. The solid curves are the Rayleigh formula (7). The estimated density of solid is from Lunar Sample Preliminary Examination Team (6).

electron micrographs referred to below. The three methods give consistent results (Fig. 3).

The detailed shapes of particles can be seen to a resolution of 300 Å in numerous scanning electron microscope pictures that were taken to see whether the origin of the material was revealed by the particle shapes.

Our studies indicate that various different effects have been active in producing the fine material. Some particles are spherical and rounded, suggesting condensation from a vapor or freezing of a liquid in free fall. Others are sharp-edged and angular, undoubtedly the result of fracture. They lack, in general, any obvious indication of a crystalline structure, as neither cleavage planes nor preferred angles are seen. It would appear that most of the fractured material is amorphous, or, if any of it is crystalline, that the size of the crystals is below the limit of resolution.

The spherical or compact round particles seen are less frequent but may form a continuous sequence from the 100-μm range down to very small sizes. The great majority of particles in the 10- to 1-μm size range have, however, more intricate shapes that are not readily understood. There are many rounded surfaces, and yet the particle as a whole is not compact. Elongated objects with

rounded ends, surfaces where the sense of the curvature often changes, rough spots occurring in smooth surfaces, and various other features argue against any single explanation—liquid droplets, condensation, or fracturing. Additional processes such as erosion by sputtering, partial melting, and partial evaporation must be considered, and scanning electron microscope study of these mechanisms is needed before all the responsible processes can be identified.

Measurements were made by means of the technique used for determining the electrical properties of terrestrial

rock powders (7). The dielectric constant (ϵ') and loss tangent of lunar dust at several stages of compaction were measured at 450 Mhz. The measurement in each case included a measurement of the density of the sample, and the porosity was calculated from the quoted specific gravity of the rock of which the powder is composed (6). The dielectric constant and the absorption length (Fig. 4) are consistent with the values deduced from ground-based radar and radiometric observations, respectively. As with terrestrial rock powders, the dielectric constant and loss tangent as a function of porosity follow the Rayleigh mixing formula and, by extrapolation, suggest a permittivity for the solid rock of the same composition as the lunar dust which is near the average for dense terrestrial rocks (about 7). The permittivity is about 3 for the dust at a typical "loose packing" porosity of 0.6. The absorption length at the same porosity, in this sample, is about 10 wavelengths.

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8. This work was done under NASA contract NAS9-8018. We are grateful to Corning Glass Works for the assistance given with the electron micrography. We thank Paul Shapshak, F. Briggs, and J. Winters.

5 January 1970

Magnetic Resonance Studies of Lunar Samples

Abstract. *Electron spin resonance searches at 9.5 gigahertz on several fines samples and portions of several rocks have yielded signals whose lineshapes and temperature dependences show that the samples are principally ferromagnetic in nature. Proton magnetic resonance searches at 60 megahertz of these samples have not revealed any signals ascribable to water or any other types of hydrogen in concentrations greater than 0.0001 percent by weight contained in narrow lines (5 oersteds wide or less) and 0.01 percent by weight in wide lines (as wide as 100 oersteds).*

We are conducting magnetic resonance studies of the lunar samples returned by the Apollo 11 mission in order to (i) determine by nuclear

magnetic resonance (NMR) the total proton concentration along with the distribution of any protons detected between water, hydrated minerals, organic

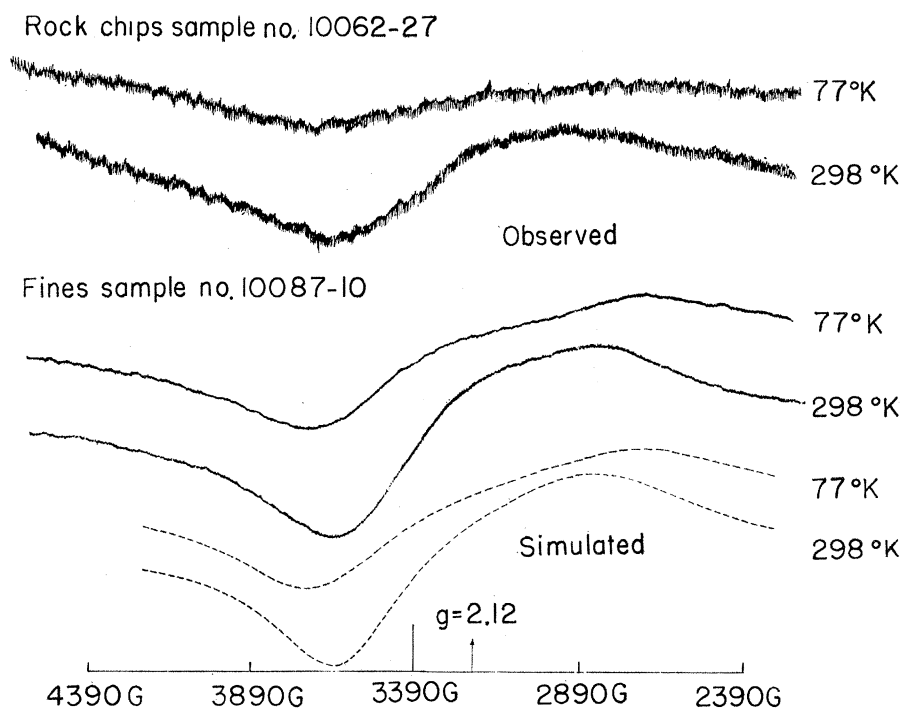


Fig. 1. Observed and computer-simulated ferromagnetic resonance spectra for the lunar samples.

protons, and solar wind protons; (ii) study by NMR any other magnetic nuclei present at significant concentrations to be satisfactorily examined by NMR; and (iii) search for electron spin resonance (ESR) signals which might arise from radiation damage to inorganic or organic components, or both, of lunar rock and soil. We describe here our initial ESR searches for species with unpaired electrons and our NMR studies directed towards the detection of protons in the lunar samples.

The ESR studies were carried out with a conventional 9.5 Ghz Varian V-4500 spectrometer fitted with 100 khz modulation, a rectangular cavity, and variable temperature provisions (down to 4°K). The NMR spectrometer consisted of a Varian 30-cm magnetic system (14 kilooersteds) whose power supply was swept by a digitally generated voltage ramp. The latter was either provided by frequency-to-voltage conversion from a digitally swept HP-5100 Frequency Synthesizer or from a HP-5480 Signal Averager. In all cases the sweep ramp was advanced synchronous with the HP-5480 address advance by an HP-2115 Controller. Repetitive linear sweeps up to 200-oersted wide could be generated. The detection system consisted of a 60 Mhz Varian V-4311 rf unit and cross coil probe [constructed of low proton materials and possessing a 15-mm (inner diameter) low proton-containing insert] whose out-

put was passed to a PAR Model HR-8 Lock-in Amplifier. The modulation frequency was 205 hz; modulation levels between 1 millioersted and 50 oersteds were employed.

Our samples consisted of several portions of fines (sample 10087) and portions of rock 10017 (external and internal sample), rock 10046 (external and internal sample), and rock 10062 (external and internal sample). For ESR, samples in quartz tubes (2 to 3 mm outer diameter) were employed. The NMR samples were studied in quartz tubes with outer diameter of 15 mm. Samples not sealed at the Lunar Receiving Laboratory were handled and stored under an atmosphere of dry nitrogen.

In the ESR experiments, a rather broad signal centered at $g = 2.12 \pm 0.05$ was detected in both the rock chip sample 10062-27 and in the fines sample 10087-10 (Fig. 1). For the latter, the signal intensities were roughly an order of magnitude stronger than those observed for the rock chip sample. In both cases, no temperature dependence of the intensities was noted. The line-shapes of these signals were studied over the range of 77° to 298°K. Computer simulation of the line-shapes (1) employing a model consisting of ferromagnetic centers of the order of $1 \mu\text{m}$ in diameter distributed throughout the samples appears to fit the observed signals. The g value of 2.12, as well as

the first order anisotropy constant ($2k_i/M_z$) of 400 oersteds obtained at room temperature and 500 oersteds at 78°K, suggest that these ferromagnetic centers are probably metallic iron.

Our magnetic resonance searches at 60 Mhz on all our lunar samples have thus far failed to detect any signals that we would attribute to protons. The instrumentation and the low proton probe we are employing is capable of detecting and has been calibrated to measure proton contents down to the 0.0001 weight percent (1 ppm) contained in resonance lines 5 oersteds wide and the 0.01 percent (100 ppm) contained in resonance lines as wide as 100 oersteds. We have studied a variety of model samples containing protons. They consisted of H_2 gas at various pressures between 760 mm and 7.6 mm, H_2 gas admixed with paramagnetic NO, H_2 gas in silica (2), terrestrial rocks and soils, and samples of various terrestrial materials admixed with known amounts of protons. In every case, proton NMR signals were easily detected.

In the initial rare gas analyses of a number of Apollo 11 samples (3), seven samples exhibited ^4He contents in the range of 0.01 to 0.8 scm^3/g (volume of 1 cm^3 of gas at 1 atm and 273°K per gram of sample). For a different fines sample than we have, the preliminary gas analysis (3) indicated possible H_2 and ^4He contents of 0.8 and 0.2 scm^3/g , respectively. In view of the ratio of ^1H to ^4He in the solar wind (4) and if these resident gas contents arise from the solar wind, it is not unreasonable to expect the occurrence of proton contents of the order of 1 to 3 scm^3/g in some lunar surface rock and soil samples. As discussed above, our instrumentation can readily detect gaseous H_2 equivalent to 0.01 scm^3/g of rock or soil if it gives rise to a resonance line 10 oersteds wide or less. The linewidths of gaseous H_2 samples are 0.6 oersted wide or less. Gaseous H_2 trapped in silica (2) exhibits a linewidth of 1.4 oersteds. Based on our experiences with terrestrial rock and soil samples, we expected that any protons in water molecules or on carbon atoms in the lunar samples might exhibit linewidths of 1 to 10 oersteds. We have detected such protons in terrestrial samples down to the 10 ppm level. In any case, we did not expect to detect the residual proton content of sample 10017 because of its low He content (3×10^{-4} to $11 \times 10^{-4} \text{ scm}^3/\text{g}$) (3).

Because aluminum was found to the extent of 4.1 to 6.9 weight percent in

all Apollo 11 samples thus far analyzed (3), we searched for ^{27}Al resonances in our samples but our results were ambiguous. All the terrestrial samples containing Al examined exhibit strong ^{27}Al signals.

Our failures to detect proton magnetic resonance signals could indicate that the proton contents of our samples are very low or that the resonances of any resident protons are too broad to be detected under the conditions employed thus far. This latter situation could very well prevail based on the ESR experimental detection of ferro-

magnetic centers discussed above and the large magnetic susceptibilities of the Apollo 11 samples found in the initial magnetic measurements (3).

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4. The ratio of ^1H to ^4He in the solar wind is of the order of 20; for example, see M. Neugebauer and C. W. Snyder, *J. Geophys. Res.* **71**, 4469 (1966).
5. We thank D. B. Nash, M. M. Neugebauer, E. M. Bollin, P. C. Lauterbur for their many helpful discussions. This paper is contribution No. 3998 from the Division of Chemistry and Chemical Engineering, California Institute of Technology. Sponsored under NASA contract No. NAS 7-100.

4 January 1970