

MÖSSBAUER SEARCH FOR FERRIC OXIDE PHASES IN LUNAR MATERIALS AND SIMULATED LUNAR MATERIALS, D.W. Forester, C.L. Marquardt, and D.L. Griscom, Naval Research Laboratory, Washington, D.C. 20390

Mössbauer (MB) studies have been carried out at room temperature on a $< 62\mu$ fraction of lunar soil 15021,118 and on a series of powdered simulated lunar glasses(1) which had been annealed at 650°C in various atmospheres.

Simulated Lunar Glasses. Mössbauer spectra of as-quenched simulated glass GS-64(1) displayed a strong asymmetric Fe^{2+} doublet due to the ~ 10 wt % FeO present. In addition there was a weak (~ 1 % of total iron) component near zero velocity, presumed to be Fe^{3+} .(1) The average quadrupole splitting (QS) of the Fe^{2+} doublet was 2.02 mm/sec and the average isomer shift (IS) with respect to Fe metal was + 1.06 mm/sec.

Upon heating to 650°C in air at ~ 0.5 Torr for 67 hours, the MB spectrum changed substantially. In addition to the original Fe^{2+} doublet, a distinct Fe^{3+} doublet was observed with $\text{QS} = .84$ mm/sec and $\text{IS} = + .58$ mm/sec, accounting for $\sim 40\%$ of the total iron. The strong ESR signal observed for this sample(1,2) may be due to the involvement of some of this Fe^{3+} in incipient superparamagnetic precipitates. This possibility is supported by a reduction of the Fe^{3+} area near zero velocity upon cooling to 3°K . However, any magnetically split spectral features were too weak or too broadened to be seen.

After a subsequent anneal at 650°C for ~ 2000 hours in an evacuated, sealed silica tube, the MB spectrum began to show evidence of Fe^{3+} in a "magnetite-like" phase (Fig. 1a). A spectrum of magnetite (Fe_3O_4) is included for comparison (Fig. 1b). The "magnetite-like" spectrum has a somewhat reduced splitting and is smeared out in comparison with Fe_3O_4 . The area of the "magnetite-like" phase is equivalent to ~ 2 wt % Fe_3O_4 , which is consistent with conclusions reached on the basis of ESR, X-ray diffraction, and susceptibility measurements.(2) This particular sample exhibited an ESR spectrum nearly identical in shape to the "characteristic" resonance(1) of typical sub-mm mare fines samples but 7 times more intense.(2) Thus, if the "characteristic" resonance should arise from "magnetite-like" phases, it is inferred that they are present in typical lunar soils in amounts ~ 0.3 wt %.

Lunar Fines. Fig. 1c shows the MB spectrum of a $< 62\mu$ fraction of 15021,118 which exhibited a "characteristic" resonance approximately twice as intense as typical unsorted, sub-mm fines from Apollos 11 and 12. The data represent more than 10^7 counts per channel. The iron metal lines indicate the presence of ~ 0.5 wt % ferromagnetic iron. Since this is a typical concentration(3) for mature lunar soils, the larger ESR signal apparently does not correlate with the amount of metallic

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$Fe \approx 160\text{\AA}$. The superparamagnetic fraction has not yet been estimated, however. The Ni content of the ferromagnetic iron in 15021,118 appears to be $< 3\%$ based on the room temperature data, but low-temperature, high-field runs will be required⁽³⁾ to confirm this estimate.

A careful comparison of Fig. 1a and Fig. 1c has revealed that the lunar spectrum is not inconsistent with the presence of up to 0.5 wt % "magnetite-like" phases. This upper limit is considerably higher than the one arrived at by Housley et al.⁽³⁾ (0.04% Fe^{3+}) for a highly magnetic fraction of 10084,85. Probably the single most important factor contributing to this discrepancy is the fact that Housley et al. considered only "pure" magnetite, rather than a range of "magnetite-like" phases, such as can be precipitated in simulated lunar glasses oxidized below their melting points.

Conclusions. If the lunar soils contain several tenths of a % "magnetite-like" phases of diverse stoichiometries, it will be difficult to verify unambiguously by the Mössbauer technique. On the other hand, previous observations of sharper peaks due to "iron spinel"⁽⁴⁾ may be attributable to samples with narrower ranges of stoichiometries. Based on the data obtained thus far, the suggested presence of Fe^{3+} in lunar fines cannot be rejected. It seems possible that the range of stoichiometries for any given sample may be narrowed by prolonged isothermal anneals in vacuo, if the behavior of the "characteristic" resonance under these treatments^(1,5) is any indication. MB studies of such "isothermally equilibrated" samples are projected.

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