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MINERALOGICAL ANALYSIS OF LUNAR ROCK PARTICLES

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Résumé. — On présente les résultats de spectroscopie Mössbauer du ^{57}Fe obtenus pour une série de fragments à gros grains (contenant pour la plupart deux minéraux discernables) ou à grains fins des roches recueillies au cours des expériences LUNA 16 et LUNA 20. Le diamètre des particules varie de 0,2 à 0,5 mm. Un dispositif pour les centrer et les présenter de manière reproductible a été mis au point. Dans le spectre Mössbauer on a séparé jusqu'à 3 composantes (magnétiques ou quadrupolaires). La comparaison avec les spectres de standards terrestres révèle la présence d'ilménite, d'olivine, de pyroxène, une phase métallique et une spinelle. L'évaluation des surfaces dans les spectres rend possible l'analyse minéralogique semi-quantitative de l'abondance du fer dans les échantillons.

Abstract. — Mössbauer measurements on ^{57}Fe in a series of LUNA-16 and LUNA-20 coarse-grain (mostly containing two distinct minerals) as well as fine-grain rock fragments have been performed. The diameter of the particles ranges from 0.2 to 0.5 mm; a device for their reproducible adjustment and centering has been developed. In the Mössbauer spectra up to 3 split (magnetic or quadrupole) components were resolved, the comparison of whose splittings with the terrestrial standards revealed the presence of ilmenite, olivine, pyroxene, metallic phase and a spinel. The gained area distribution in the spectra made it possible to perform a semi-quantitative mineralogical analysis of the iron abundances in the samples.

1. Introduction. — The cosmochemical importance of iron and the convenience of Mössbauer experiment on ^{57}Fe made the Mössbauer spectroscopy a widely applied method for mineralogical studies of lunar returned materials. The sensitivity of this method made it possible to perform the measurements on fine isolated particles of lunar minerals and rocks of the size down to tenths of mm [1]. Based on this, we started a more systematic study of the selected fine particles from the Soviet LUNA-16 and LUNA-20 samples [2].

In this work, by selecting particles containing optically distinguishable grains of minerals and by using a special centering device we succeeded in some cases to isolate contributions of these distinct minerals and in other cases to perform a semiquantitative mineralogical analysis concerning the iron distribution among different phases in accordance with microscopic appearance of the tiny rock fragments.

2. Experimental arrangement. — Similarly as in our previous works, all spectra were obtained with a double-parabolic-time-mode Mössbauer spectrometer NP-255 (KFKI, Budapest, Hungary) in transmission geometry at room temperature. High density ^{57}Co sources of 1 mm diameter were used, at first ca 5 mCi in Pd matrix, later ca 20 mCi in Rh (The Radiochemical Centre, Amersham, England). Calibration of the velocity scale was performed with sodium nitroprusside or iron standards in cases of metallic phase

present using identical diaphragm diameter as for the respective sample. The measured spectra were least-squares fitted by a set of Lorentzian lines with physically justified constraints of parameters.

To avoid difficulties with the adjustment of the measurement geometry in a series of numerous measurements, a special device for reproducible mounting and centering of samples has been developed (Fig. 1).

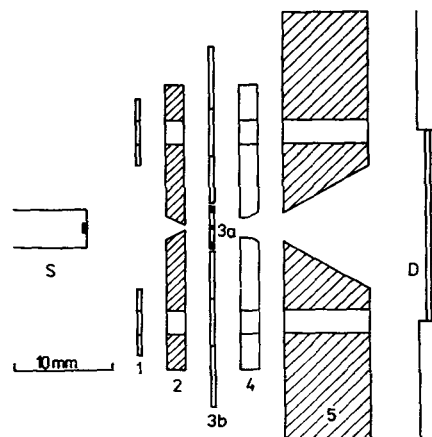


FIG. 1. — Experimental arrangement for measuring small particles (blown-up cross-section). S : source. 1. holder ring (brass). 2. entrance diaphragm (lead). 3a. capsule with sample (polyethylene). 3b. centering ring (brass). 4. holder ring (brass). 5. detector shield (lead). D : detector-entrance window (beryllium). Parts 1 to 4 are assembled together and mounted on 5.

All samples are separately mounted into small polyethylene capsules 3a, suitable also for microscopic observations. Under the microscope, the encapsulated particle is centered in the brass holder 1 + 4 by means of the centering ring 3b using a lead diaphragm 2 with suitably chosen diameter. This holder can be reproducibly mounted on the source-detector arrangement. Just a bit of carefulness enables us to work in such a way with these small particles and to reach acceptable counting rates as in any other transmission experiment.

3. Measurements and results. — From the particles available from LUNA-16 and LUNA-20 material we succeeded in measurements both of some coarse-grain ones, containing optically distinguishable mineral grains, and of some fine-grain rock fragments.

Spectra of the samples of the former type are shown in figure 2. Besides the grain containing mostly pyro-

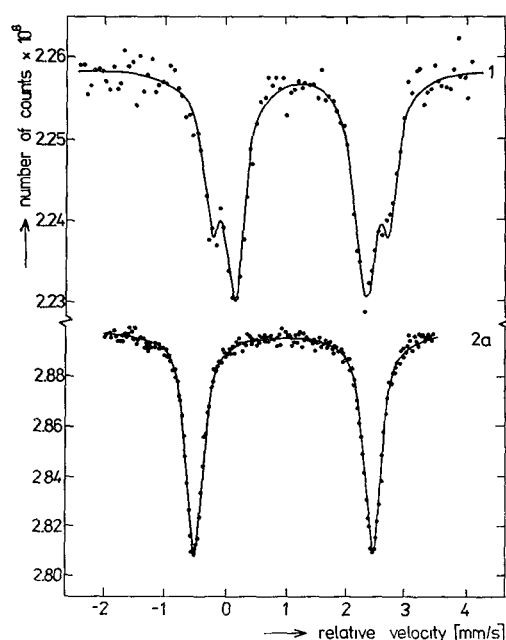


FIG. 2. — Mössbauer spectra of lunar minerals: 1. pyroxene (run B). 2a. olivine. Numeration of the spectra agrees with Table I.

xene (top spectrum), the pure olivine grains with one small ilmenite inclusion were measured (at the bottom is one of these spectra). In figure 3 two of the spectra of the latter type of samples are shown. At the top is a spectrum of basalt-like fine-grain gabbro of Mare origin. At the bottom a spectrum of iron containing minerals of anorthositic rock from lunar highlands is exhibited. One sample of metal-containing breccia was also remeasured. Such type of spectra has already been shown in [1].

Least-squares analysis of all the measured spectra (on the assumption of asymmetric doublets with equal line widths) led to the decomposing into 2-3 quadrupole

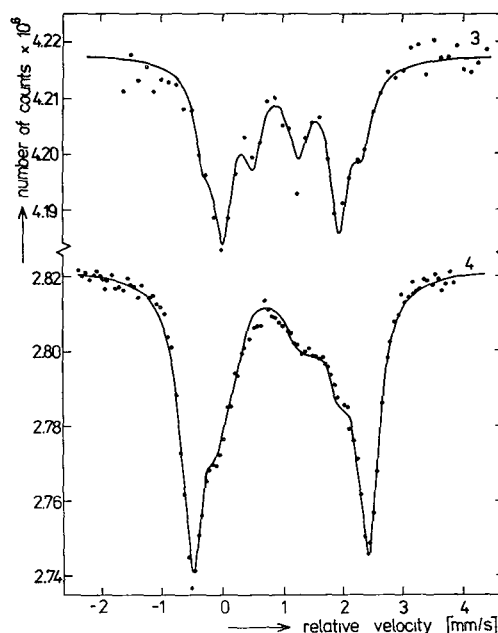


FIG. 3. — Mössbauer spectra of lunar rocks: 3. gabbro (run B). 4. anorthosite (run B). Numeration of the spectra agrees with Table I.

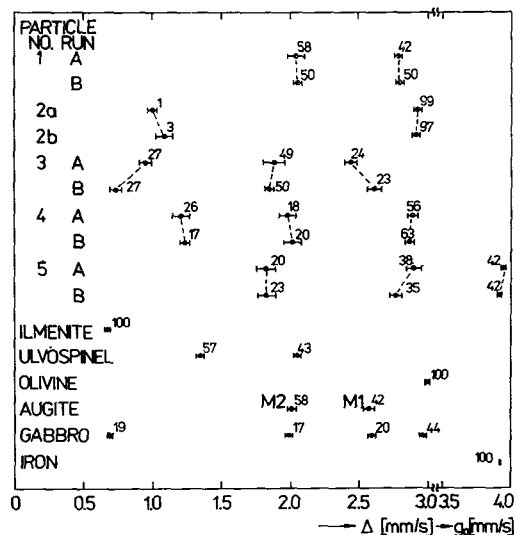


FIG. 4. — Quadrupole splittings Δ and ground level magnetic splittings g_0 of spectra components of lunar particles and of some terrestrial materials. Figures denote intensity of components, letters A and B repeated runs of measurements. Numeration of the spectra agrees with Table I.

components (and the magnetic one in the last mentioned case) and yielded their quadrupole (or magnetic, respectively) splittings and relative areas in the spectra. Splittings of individual components are displayed in figure 4, where the reproducibility of the process can be seen. Averaged results are summarized in table I.

TABLE I

Description and composition of the LUNA-16 and LUNA-20 particles

Particle No.	Origin	Description	Size [mm]	Components [at. %]		Olivine	Metal
				Ilmenite	Spinel		
1	L 20	Pyroxene	0.4×0.4				
2a	L 20	Olivine (+ ilmenite)	0.3×0.4	0.6 ± 0.4		99.4 ± 0.4	
2b	L 16	Olivine + ilmenite	0.2×0.3	3.2 ± 0.5		96.8 ± 0.5	
3	L 16	Gabbro	0.2×0.3	27 ± 2			
4	L 20	Anorthosite	0.5×0.7		18 ± 4	62 ± 3	
5	L 16	Breccia with metal	0.2×0.3			36 ± 2	42 ± 5

4. **Discussion.** — As expected, in the mineralogically well defined particles 1, 2, 3, individual quadrupole components are clearly resolved and corresponding minerals can be unambiguously identified. In the particle 1, only pyroxene was found with possible slight admixture of olivine, as it follows from the increased quadrupole splitting of the M1 iron site in comparison with the terrestrial augite and clinopyroxene in gabbro. Similarly, samples 2a and 2b are showing almost exclusively the olivine lines, well matched to these of the terrestrial samples. The ilmenite contribution is almost indistinguishable; very small intensity of its lines probably causes the bias of their quadrupole splittings.

This experience has been used in analyzing spectra of rock fragments. In the gabbroidic particle 3, about 1/4 of iron has been found in ilmenite structure and the rest in pyroxene with well resolved iron sites. The spectra of particles 4 and 5, when looking at their decomposition, seem to be more complicated. Though in both of them the contributions of the two iron sites in pyroxenes were found, the increased quadrupole splittings and the intensities of the M1 lines witness the unresolved contribution of olivine.

In the sample 4 the third quadrupole component cannot be reasonably explained by ilmenite. In accordance with the microscopic appearance, its explanation by the presence of a member of spinel group, binding about 20 % of the entire iron, seems to be most likely. Similarly, in the sample 5, about 49 % of iron is present in the metallic form and causes a six-line component in its spectrum.

Summarizing the results for the lunar pyroxenes, considerable variability in the quadrupole splittings and relative site abundances has been observed. This, in principle, is in accordance with the measurements for pure lunar clinopyroxenes [3], but still the increased occupancy of one M1 site of the samples 4 and 5 and a distinct shift of their quadrupole splittings towards the value of olivine, compared with particle No. 1 or even the terrestrial standard, leaves the space for the mentioned unresolved contribution of olivine. In the remaining two cases, the M1 intensity and splitting for particle 1 agrees well with the mentioned value for the clinopyroxenes [3], whereas those of sample 3 resemble the terrestrial augite.

There is unambiguous correspondence between the

quadrupole splitting and FeO/(FeO + MgO) ratio in olivines [4]. According to this, our quadrupole splittings correspond to the values of 0.55 and 0.60 of this ratio for samples 2a and 2b, respectively. This is exactly what we assumed for the olivine in the average sample of LUNA-16 and LUNA-20 regolith [5].

The gained ^{57}Fe ground state splittings of the metallic phase $g_0 = 3.930$ mm/s is about 0.4 % higher than that of metallic iron 3.916 mm/s [6]. Supposing that this is due to the iron alloying with nickel and taking into account the hyperfine field increase of 0.67 % per wt. % Ni [7], it is reasonable to admit the presence of ca 0.5 wt % Ni in the metallic phase.

5. **Conclusion.** — Analogically to the common studies of the average powder or fine-grain lunar regolith, by overcoming experimental difficulties the possibility was achieved to perform Mössbauer measurements of individual small particles. In comparison with the former ones, the spectra of these particles are less complicated, their 2 to 3 components corresponding to distinct minerals in coarse grained particles or to the constituents of a well defined rock fragment. As it has been shown, qualitative and semiquantitative mineralogical analysis can be performed also for these samples. Nevertheless, for a more complete phase analysis (as e. g. in [5]) the contributions of rather near pyroxene M1 and olivine doublets remain to be distinguished.

In case of fully resolved components, as e. g. here the ilmenite, metal and in some cases also olivine, conclusions can be drawn about the composition of the respective phase. It is our aim, by choosing suitable samples and increasing the resolution, to study in detail the types of lunar pyroxenes (with the hope to gain valuable thermodynamic data), metallic inclusions and to learn more about spinels not fully explored yet.

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