SC507.3AR

LUNAR SAMPLE ANALYSIS

Annual Report
as of 31 January 1972

GENERAL ORDER NO. 507

CONTRACT NO. NAS 9-11539

Prepared for:

NASA Manned Spacecraft Center
Science & Applications Directorate
Houston, Texas 77058

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805/498-4545

Study of excess Fe metal in the lunar fines by magnetic separation, Mössbauer spectroscopy, and microscopic examination

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ABSTRACT

A simple and convenient method of making quantitative magnetic separations of the lunar fines is described. The fractions obtained form groups containing distinctively different particle types; thus it appears that magnetic separation in itself may be a useful way of characterizing lunar fines. Mössbauer studies of fines 10084 show that the metal can not contain more than about 1.5% Ni implying that by far the bulk of it results from reduction rather than being a direct meteoritic addition. Mössbauer data also places an upper limit on the magnetite content of the fines at least an order of magnitude below that required to account for the characteristic ferromagnetic resonance observed. Microscopic examination of magnetic separates from the 15101 fines suggests that reduction of Fe accompanies every major impact event on the moon and also suggests that the bulk of the material at the collection site has at one time been in an impact plume.

INTRODUCTION

We have previously shown (Housley et al., 1970; 1971) that typical dark gray lunar fines at the Apollo 11 and 12 sites contain considerably more metallic Fe than the local igneous rocks, that most of this excess Fe metal is intimately associated with glassy material, and that a significant fraction of the metal grains are less than about 100 Å in diameter.

Table 1 summarizes all our absolute determinations of Fe metal content in lunar samples including new data on the Apollo 14 fines 14003,22 and 14163,52, Apollo 14 recrystallized breccia 14311,41 and Apollo 15 fines 15101,92. The second column in the table indicates the sieved size fraction which was studied. The third column gives the ferromagnetic Fe metal content determined from the area in the normal Mössbauer hyperfine pattern of Fe metal. The fourth column gives the Fe content corresponding to the excess area observed near zero relative velocity in the Mössbauer spectra. A sizable fraction of this has been shown (Housley et al., 1971) to be due to superparamagnetic Fe grains less than about 100 Å in diameter.

The widespread occurrence of excess Fe metal in the small size fractions of lunar fines suggested to us that further study of its composition, physical form, and association might yield valuable insights into the dynamical events associated with meteorite impacts on the moon and perhaps give information concerning the nature of the impacting bodies and/or the impacted lunar material.

Table 1. Metallic Fe in Lunar Fines and Rocks*

Sample	Size Range μm	Ferromagnetic Fe wt %	Excess Area as wt % Fe
77°K			
10084,85 (sp.g. < 3.3)	0-75	0.61 ± 0.04	0.29 ± 0.02
295°K			
10084,85 (sp.g. < 3.3)	0-75	0.51 ± 0.04	0.39 ± 0.02
12042,38	0-1000 ground	not determined	0.332± 0.004
12042,38 (sp.g. < 3.3)	45-150	0.34 ± 0.05	0.17 ± 0.02
12042,38 (sp.g. > 3.3)	45-150	0.04 ± 0.07	—
12025,15	0-150	0.38 ± 0.06	0.35 ± 0.02
12025,42	0-150	0.37 ± 0.06	0.35 ± 0.02
12025,88	0-150	0.39 ± 0.08	0.22 ± 0.02
12025,113	0-150	0.31 ± 0.05	0.43 ± 0.01
12038,47	ground	0.07 ± 0.03	0.04 ± 0.01
12052,16	ground	0.11 ± 0.06	0.03 ± 0.01
14003,22	0-45	0.40 ± 0.04	0.36 ± 0.01
14163,52	0-45	0.34 ± 0.03	0.28 ± 0.01
14311,41	ground	0.14 ± 0.04	0.06 ± 0.01
15101,92	0-75	0.20 ± 0.03	0.37 ± 0.02

*Error limits are one standard deviation due to counting statistics only.

All data were analyzed by the method described by Housley et al. (1971).

With the initial objective of facilitating this study we have developed a simple and effective method of magnetically separating small amounts of fine grained material quantitatively without loss or contamination. It now seems likely that this technique will be useful in a much wider range of studies.

Here we will describe the technique and report on studies of magnetic separates from the fines 10084,85 and 15101,92 by Mössbauer spectroscopy, optical microscopy, and scanning electron microscopy.

MAGNETIC SEPARATION

The magnetic separator as first assembled and used is shown in Fig. 1. Although refinements have been made since then the apparatus and procedure still remain extremely simple. The sample is spread over the bottom of a flat bottomed aluminum container which in turn sets on a flat supporting plate between the poles of a strong permanent horseshoe magnet. A thin flat soft Fe plate in contact with the upper magnet pole can be lowered to any desired height above the bottom of the sample container. To reduce the tendency of grains to stick to each other or the container, the latter is filled with pure ethanol to a level above the bottom of the soft Fe plate. A separation is made by gently sliding the sample dish around until all parts of the sample have been directly under the soft Fe plate. The Fe plate is then raised and removed from the magnet. During this operation the surface tension of the ethanol holds the collected material on the plate. After removal from the magnet, the plate and adhering

magnetic separate are held over a suitable container for a short time while the ethanol evaporates. When evaporation is complete, most of the collected separate simply falls off the plate and any which remains can be removed by gentle tapping or brushing. It is clearly important at this point that the Fe plate is sufficiently soft magnetically that it does not retain significant magnetism after removal from the magnet. Further fractions are obtained by repeating the procedure with the soft Fe plate at successively lower positions during the collections.

A significant amount of material ($\sim$ 5%) was collected from samples 10084,85 and 15101,92 at a separation of 6 mm allowing them to be quantitatively separated into a number of distinct magnetic fractions. Sieved size fractions 45-75 μm and 420-1000 μm were successfully separated. Repeat collection at a given separation yielded little additional material whereas a given fraction spread on a clean aluminum dish was almost entirely re-collected at the separation at which it was initially collected proving that the fractions obtained are indeed magnetically distinct.

Field and field gradient values at different separations h measured with a Hall effect gaussmeter are given in the second and third columns of Table II. The size of the probe prevented useful measurements for h values less than about 1 mm and perhaps introduces some uncertainty into the values at larger separations.

In a given magnetic field H and field gradient dH/dh, the magnetic force on a sample depends on its magnetization. For paramagnetic material, this is simply proportional to H. For ferromagnetic material the magnetization in a given field strongly depends on grain size and

shape. The limiting force for extremely small grains corresponds to that expected for free paramagnetic atoms. The force per unit mass at first increases linearly with particle size as mIH/3kT (Bean and Livingston, 1959) up to a diameter d of about 30 Å for Fe metal in about 1000 Oe where m is the mass, I the saturation moment per unit mass, and kT the temperature in energy units. Also for spherical Fe particles in H = 1000 Oe the magnetization approaches the saturation value of I as 1-(kT/mIH) (Bean and Livingston, 1959) for particles with $d \gtrsim 60$ Å. For a range of diameters greater than 60 Å, spherical Fe particles are expected to be fully magnetized along the field direction and hence to experience the maximum magnetic force. For d values somewhat greater than 200 Å, the uniform magnetization state will become unstable for low H values (Frei et al., 1957; Brown, 1963) and at 1000 Oe the force per unit mass will decrease with d. For $d >> 1000$ Å bulk behavior will be approached and the magnetization at low fields will depend on the demagnetization factor.

In column 4 of Table II we give the ratio of the magnetic force to the gravitational force for a hypothetical sample composed entirely of Fe^{++} ions. In column 5 we give the same ratio for fully aligned single domain Fe metal particles and in column 6 we give the result for spherical Fe metal particles large enough to approach bulk behavior. The magnetic forces expressed in columns 4, 5, and 6 of Table II can be used to place limits on the Fe metal content of any magnetic separate. For example, assuming a bulk density of 2.4 gm/cm^3 and an Fe^{++} content of 20% and correcting for the buoyancy of the alcohol one finds that the

fraction collected at h = 6 mm must contain at least 0.57% Fe metal whereas the fraction collected at h = 2 mm need contain only 0.17% If large grains behaving like bulk Fe metal are assumed, the limits respectively become 3.6% and 0.8%.

Table II. Characteristics of Magnetic Separator

h mm	H Oe	dH/dh Oe/cm	(F/mg) Fe^{++}	(F/mg) 60-200Å	(F/mg) bulk
2	1557	1525	0.421	340	73.2
3	1417	1270	0.334	284	55.6
4	1303	1015	0.245	226	40.8
5	1214	762	0.171	170	28.5
6	1151	508	0.108	113	18.0

MÖSSBAUER SPECTROSCOPY

Bulk samples of ferromagnetic material in zero applied field H generally assume a domain configuration such that there are no free poles at the surfaces and the demagnetization field is hence essentially zero. In this case the field at the atomic nuclei H_{int} is equal to the field H_{hf} produced by hyperfine interactions alone. For metallic Fe the width of a domain wall is about 1000 Å (Nagata, 1961) and particles must be much larger than this before bulk behavior can be assumed. There is good evidence (Runcorn et al., 1970; Nagata et al., 1970) that much of the Fe in the lunar fines is actually

present as particles considerably smaller than 1000 Å in diameter. There is no clear justification in this case for interpreting H_{int} values obtained at zero field in terms of the absence or presence of alloying elements. This ambiguity can be removed however by taking data in high applied fields H_o where the particles are all fully saturated and then extrapolating H_{int} vs H_o to zero H_o.

To obtain a sample as rich in metallic Fe as possible, we made a magnetic separation of the light fraction (sp.g. < 3.3) of the 0-75 μm fines 10084,85 which we had previously (Housley et al., 1970) shown to contain most of the Fe metal. A total of 80.3 mg of material was collected at separations ≥ 3 mm and was combined in the Mössbauer absorber. The remaining less magnetic material weighed 67.5 mg. The magnetic fraction subsequently proved to contain about 1% Fe metal which is consistent with the expected behavior of the separator for an average grain size between the limits corresponding to saturated single domain and bulk behavior.

The sample was mounted between Be windows in a superconducting magnet assembly and had a thickness of 41.4 mg/cm^2. The Mössbauer source was Co^{57} in Cu at 22°C and zero field. Spectra were obtained using an absolutely calibrated constant velocity spectrometer operating in an automated mode. At several fields and temperatures, complete spectra were recorded; additional data were recorded for velocity intervals which spanned the outer Fe metal lines. Typical data are shown in Fig. 2. In our complete series of runs data were collected at 295°K and H_o=0, 11°K and H_o=0, 11°K and H_o=55 kOe, and then a series

of decreasing field values ending with 11°K and H_o=0 again. Line positions and areas were determined by least squares fitting the data in the vicinity of the Fe lines to a Lorentz curve of unconstrained width, depth, and position plus a straight line of unconstrained height and slope to account for the overlapping area contributed by other phases. Areas were corrected for background which was measured for each run.

The H_{int} values obtained from the line positions are plotted against H_o in Fig. 3. The solid line is a least squares fit to the data for $H_o \geq 8$ kOe for which the grains are expected to be magnetically saturated. In calculating the demagnetization field we assume the Fe metal particles are spherical, which was the case for the vast majority of the hundreds of micron size metal grains that we have observed microscopically, and obtain a value of 7.4 kOe. Subracting this from the intercept of the solid line with the H_o=0 axis yields a value of H_{hf} of 340.6 ± 1.0 kOe. This corresponds with our value at 5°K 339.7 ± 0.2 for pure Fe metal. Using the data of Johnson et al. (1961) on the change in average hyperfine field produced by alloying Fe with Ni permits us to conclude that the metal grains cannot contain much more than 1.5% Ni on the average and need not contain any. This seems to be strong evidence that their origin is through a reduction process rather than by direct meteoritic addition.

The Mössbauer data also allow some rough conclusions to be drawn concerning the size distribution in the particles. When the particles are small enough that superparamagnetic relaxation becomes rapid compared to the Larmor frequency of the nucleus in the hyperfine field

then the hyperfine pattern is expected to collapse to a single line. The superparamagnetic relaxation time t for spherical Fe metal particles is expected to be given roughly by

$$\frac{1}{t} = \frac{2\gamma_0 K_1}{J_s} e^{-(K_1 V/4kT)}$$

where γ_0 is the gyromagnetic ratio, K_1 is the first order anisotropy constant, and J_s is the saturation magnetization. Using values of the constants appropriate for pure Fe the transition from ferromagnetic to superparamagnetic behavior in a Mössbauer experiment can be calculated to occur at a particle diameter of 134 Å at 295°K, 85 Å at 77°K, and 45 Å at 11°K. In addition, at 11°K and H_0=55 kOe grains down to 20 Å in diameter would be magnetically saturated and hence would appear in the ferromagnetic spectrum.

At 11°K the ratio of the area observed in the ferromagnetic component at H_0=55 kOe to that at H_0=0 is 1.53 ± 0.18 while the ratio expected just due to polarization of the originally unpolarized absorber is 1.5. The ratio between the areas obtained at 295°K and that obtained at 11°K in H_0=0 when corrected for the difference in Debye-Waller factors is 1.23 ± 0.16, which is only slightly greater than the ratio obtained previously (Housley et al., 1971) between 295°K and 77°K. These results imply that about 20% of the Fe metal is present as grains between about 134 and 85 Å in diameter and probably considerably less in the range 85 to 20 Å.

We did not see evidence for magnetite or a similar spinel phase in any of our spectra although weak Mössbauer evidence for it has been previously reported (Gay et al., 1970). In view of the current interest

this question has with regard to the interpretation of microwave resonance spectra of the lunar fines (Griscom and Marquardt, 1972; Weeks et al., 1972), we collected a set of data at 11°K and H_o=20 kOe spanning the expected positions of the outer magnetite lines at low temperature (Kundig and Hargrove, 1969). Any magnetite grain $\gtrsim$ 30 Å in diameter would be sufficiently near magnetic saturation to be observed under these conditions while any grain smaller would make only a weak contribution to the microwave resonance spectrum at room temperature and would have a temperature dependent intensity similar to paramagnetic materials. These data are shown in Fig. 4 and no magnetite lines are evident. The slight overall absorption in this region can probably be explained as the tails from resonance peaks at lower velocities. By summing all the area within ± 0.5 mm/sec of the expected magnetite line positions, we obtain an upper limit of 0.04% for the possible amount of Fe^{+++} in magnetite-like phases in this sample. Since this sample was composed largely of glass-welded aggregates this seems to be strong evidence against the interpretation offered by Griscom and Marquardt (1972) of their microwave resonance results and strong support for the earlier interpretation of Tsay et al. (1971).

CHARACTERIZATION OF FINES 15101,92

Two size fractions 45-75 μm and 420-1000 μm from the Apollo 15 fines 15101,92 which were collected in an area that appears to consist largely of ejecta from St. George crater (Swann et al., 1971) have been magnetically separated.

The coarse fraction material collected above 3 mm separation consisted entirely of glass welded aggregates. Those glass beads

present were collected between 1 and 3 mm. The nonmagnetic material which could not be picked up even on contact consisted largely of anorthite, but several dark fine grained rock fragments were also present.

The distribution of mass in the different magnetic separates of the 45-75 μm size fraction is shown in Table III. One striking fact immediately evident from this table is that 63% of the mass is accounted for by particles containing at least 0.17% Fe metal.

Examination of the different magnetic fractions by optical microscopy, Figs. 5-7, shows that they form easily recognizable distinct particle groups. The most magnetic particles are, except for a very rare pure metal grain, all dark, semi-opaque, vesicular, inhomogeneous glass-welded aggregates of very fine mineral fragments while no particles of this type are found in the least magnetic fraction. No optically resolvable metallic phase can be found in the majority of particles in the most magnetic group; these particles however all appear to contain regions of welding glass which has a milky appearance in reflected light as shown in Fig. 7b and which shows iridescence when viewed with an oil immersion lens. Fig. 7d shows a similar region which also contains some larger metal grains.

The least magnetic fraction consists largely of mineral grains containing about 50% plagioclase, about 35% mafic minerals, about 10% rock fragments all of which have the general appearance of the one shown in Fig. 7c, and about 5% homogeneous glass spheres and shards. An unusual feldspar crystal containing crystallographically oriented rod shaped inclusions is shown in Fig. 7c.

Intermediate magnetic fractions seem to consist largely of mineral grains or aggregates with varying amounts of attached welding glass.

Table III. Magnetic separation of 45-75 μm fraction of 15101,92 fines.

Designation	Height mm	Weight mg	% of Total
1	6	7.4	5.2
2	5	16.2	11.4
3	4	14.8	10.4
4	3	14.6	10.3
5	2	36.5	25.7
1-5	> 2	89.5	63.0
6	< 2	52.5	37.0
Total		142.0	100.0

For material collected at 3 mm separation welding glass is optically observable in all but about 10% of the grains in the polished grain mount.

Scanning electron micrographs at medium and high magnification of grains from the most and least magnetic fractions are shown in Figs. 8-10.

The numerous rounded surfaces indicating solidification of an unconstrained liquid in the glass-welded aggregates constituting the most magnetic fraction strongly suggests an origin in a hot impact plume (McKay et al., 1970). Some welding glass can be seen on the surface of particles in the least magnetic fraction.

Microscopic examination of material from the most magnetic fraction of the Apollo 11 fines 10084,85 shows that it is similar in all respects to the description given above for the most magnetic fraction of 15101,92.

Preliminary study of x-ray powder diffraction data on the most and least magnetic fractions from 15101,92 shows that these fractions contain the same crystalline phases in proportions which are similar.

CONCLUSIONS

The magnetic separator described allows clearly distinct groups of particles to be easily separated from the lunar fines. Since it is cheap and easily constructed and permits separation of small amounts of fine grain size material in a safe contamination free manner it appears suitable for use in a number of other studies of the chemical mineralogical and physical properties of lunar fines.

The Mössbauer data on fines 10084,85 lead to the conclusion that by far the bulk of the Fe metal present resulted from reduction rather than being a direct meteoritic addition. The physical characteristics of the particles from the most magnetic fractions of fines 10084,85 and 15101,92 are completely consistent with this conclusion. The absence of glass-welded aggregates in the less magnetic fractions of 15101,92 further suggests that Fe is reduced in every impact plume of sufficient size to produce the glass welded aggregates and hence does not require any special compositions or conditions, although this conclusion must be further confirmed by studying fines from other sites.

The facts that 63% of the material in 15101,92 was collected at separations that require at least 0.17% adhering Fe metal and that there seems to be a continuous gradation between glass welded aggregates which clearly formed in conditions similar to those expected in an impact

plume and minerals with small amounts of adhering welding glass suggest that the majority of fines at the collection site have been through an impact cloud rather than having gradually eroded out of rocks by some process such as thermal cycling.

The upper limit on the amount of magnetite or similar Fe^{+++} containing magnetic spinel present in the 10084,85 fines is an order of magnitude or more lower than that required to account for the characteristic ferromagnetic resonance observed in the fines.

ACKNOWLEDGEMENTS

We wish to express our deep gratitude to L. H. Hackett who performed the scanning electron microscope studies for us. The technical assistance provided by P. B. Crandall and K. G. Rasmussen is also gratefully acknowledged. This work was supported by NASA Contract NAS 9-11539.

FIGURE CAPTIONS

Fig. 1. Magnetic separator in simulated operating configuration. Soft Fe plate is lowered to desired height above sample which is in flat bottomed aluminum dish. Dish is then slid around slowly until all parts have been under the plate.

Fig. 2. Typical Mössbauer spectra taken at 11°K and the indicated magnetic fields plotted relative to Fe metal at 295°K as velocity zero. Displaced down 0.02 below each full spectrum the corresponding higher statistical accuracy data taken in the velocity range spanning the outer Fe metal lines are also shown.

Fig. 3. Internal fields H_{int} for the metallic phase in 10084,85 at 11°K obtained by analysis of the Mossbauer spectra plotted versus the applied field H_o. The solid line is a least squares fit to the data for $H_o \geq 8$ kOe. The dashed line would be followed by H_{int} for bulk Fe particles of spherical shape at low H_o.

Fig. 4. High statistical accuracy data at 11°K and H_o=20 kOe in the velocity intervals where magnetite lines would be found. Arrows indicate expected absorption centroids. No evidence for magnetite can be seen.

Fig. 5. Transmitted light micrographs of grains from the most magnetic (left), a medium magnetic (middle) and the least magnetic (right) fractions of the fines 15101,92 mounted in immersion oil. The top photo in each pair was taken with plane polarized light and the bottom photo was taken with partially crossed polarizers.

Fig. 6. Low magnification comparison of polished mounts of grains from the most magnetic and least magnetic fractions of the fines 15101,92. a) Least magnetic fraction: mineral grains and minor transparent glass, reflected light. b) Most magnetic fraction: semi-opaque glass-welded aggregates, reflected light. c) Least magnetic fraction, transmitted light. d) Most magnetic fraction, transmitted light.

Fig. 7. High magnification comparison of polished grains from most magnetic and least magnetic fractions of the fines 15101,92. a) Least magnetic, reflected light. b) Most magnetic, reflected light. c) Least magnetic transmitted light. d) Most magnetic, reflected light with surface relief emphasized by interference.

Fig. 8. Scanning electron micrographs of typical grains of glass-welded aggregates which make up the magnetic fraction of fines 15101,92 at medium magnification. Smooth rounded glass surfaces indicate unconstrained solidifcation as would be expected in an impact plume.

Fig. 9. Scanning electron micrographs of typical areas of surface on grains from the most magnetic fraction of fines 15101,92 at high magnfication.

Fig. 10. Scanning electron micrographs of typical grains from the least magnetic fraction of fines 15101,92 at medium magnification. The fraction consists mostly of crystalline mineral grains but contains minor amounts of homogeneous transparent glass.

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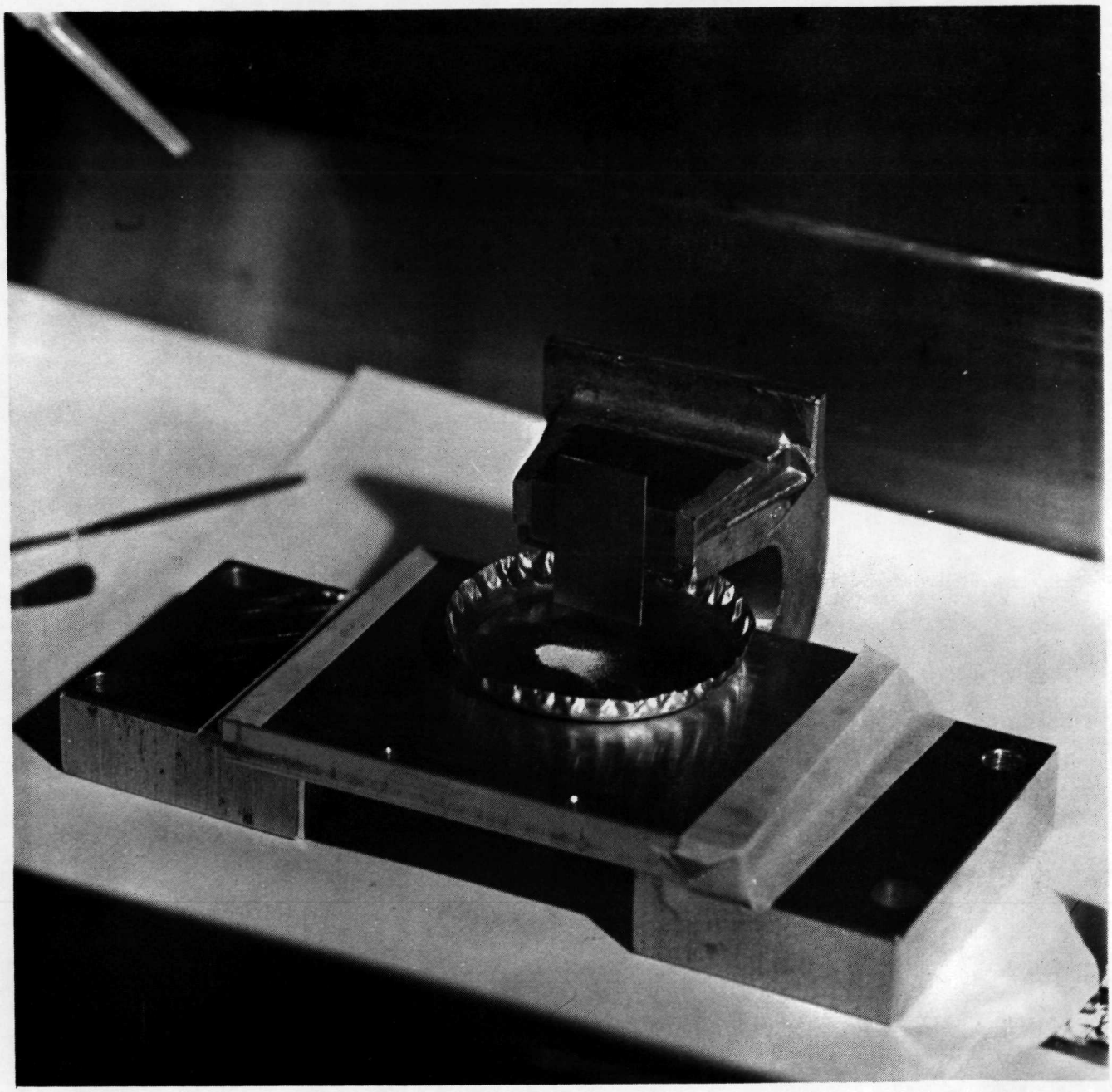

Figure 1

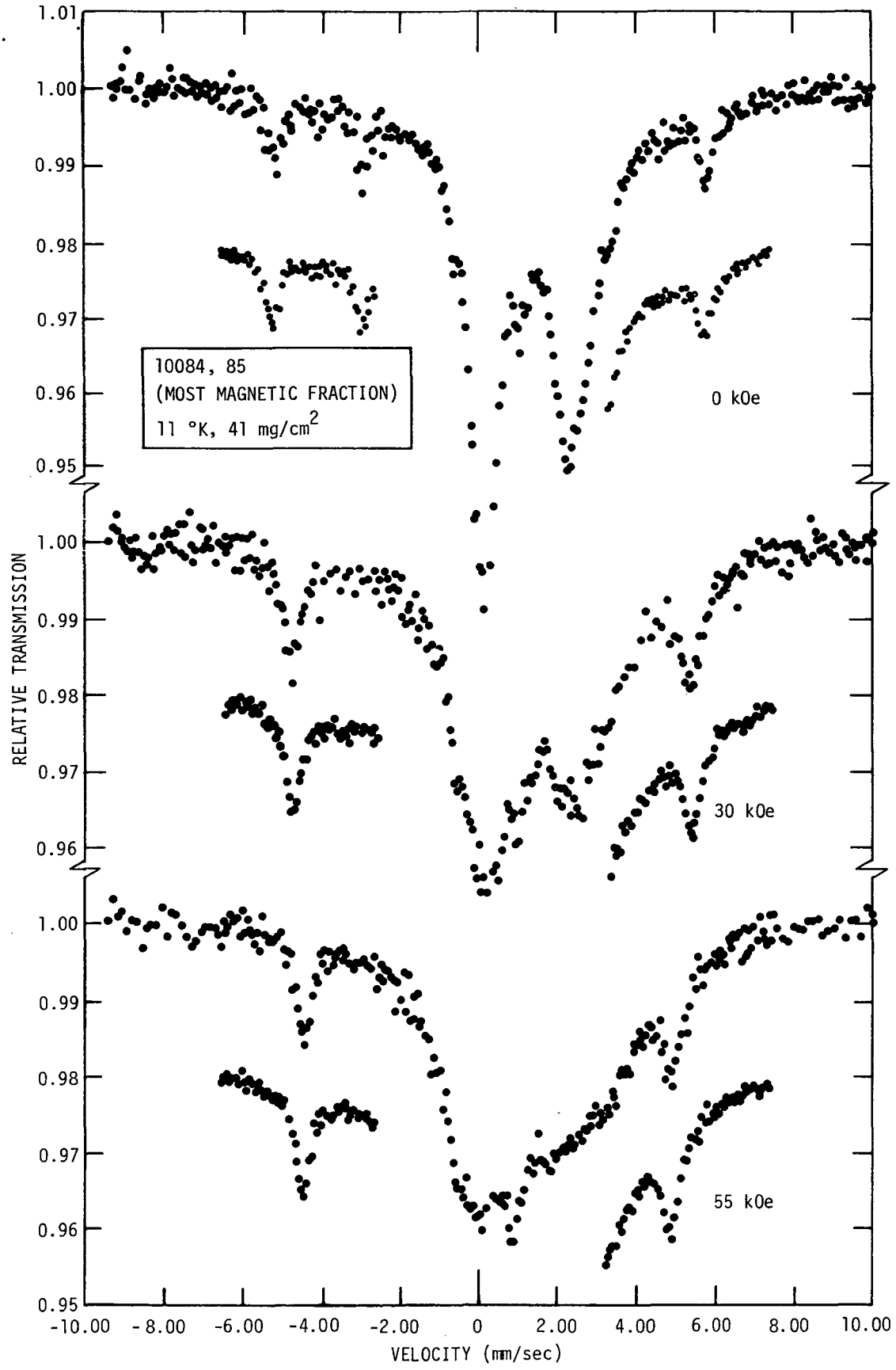
10084, 85
(MOST MAGNETIC FRACTION)
11 °K, 41 mg/cm2
0 kOe
30 kOe
55 kOe
RELATIVE TRANSMISSION
VELOCITY (mm/sec)
1.01
1.00
0.99
0.98
0.97
0.96
0.95
-10.00
-8.00
-6.00
-4.00
-2.00
0
2.00
4.00
6.00
8.00
10.00

Figure 2

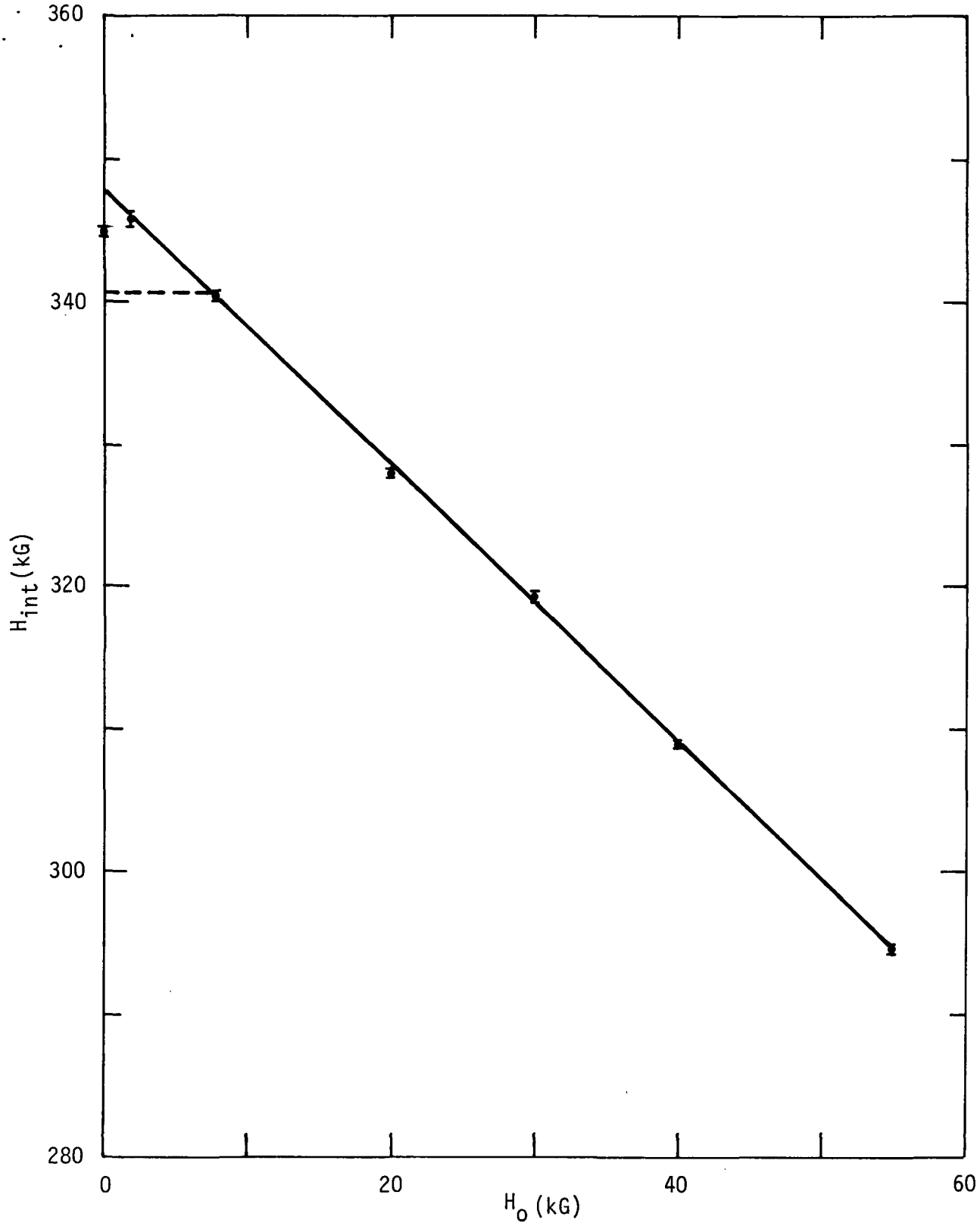

Figure 3

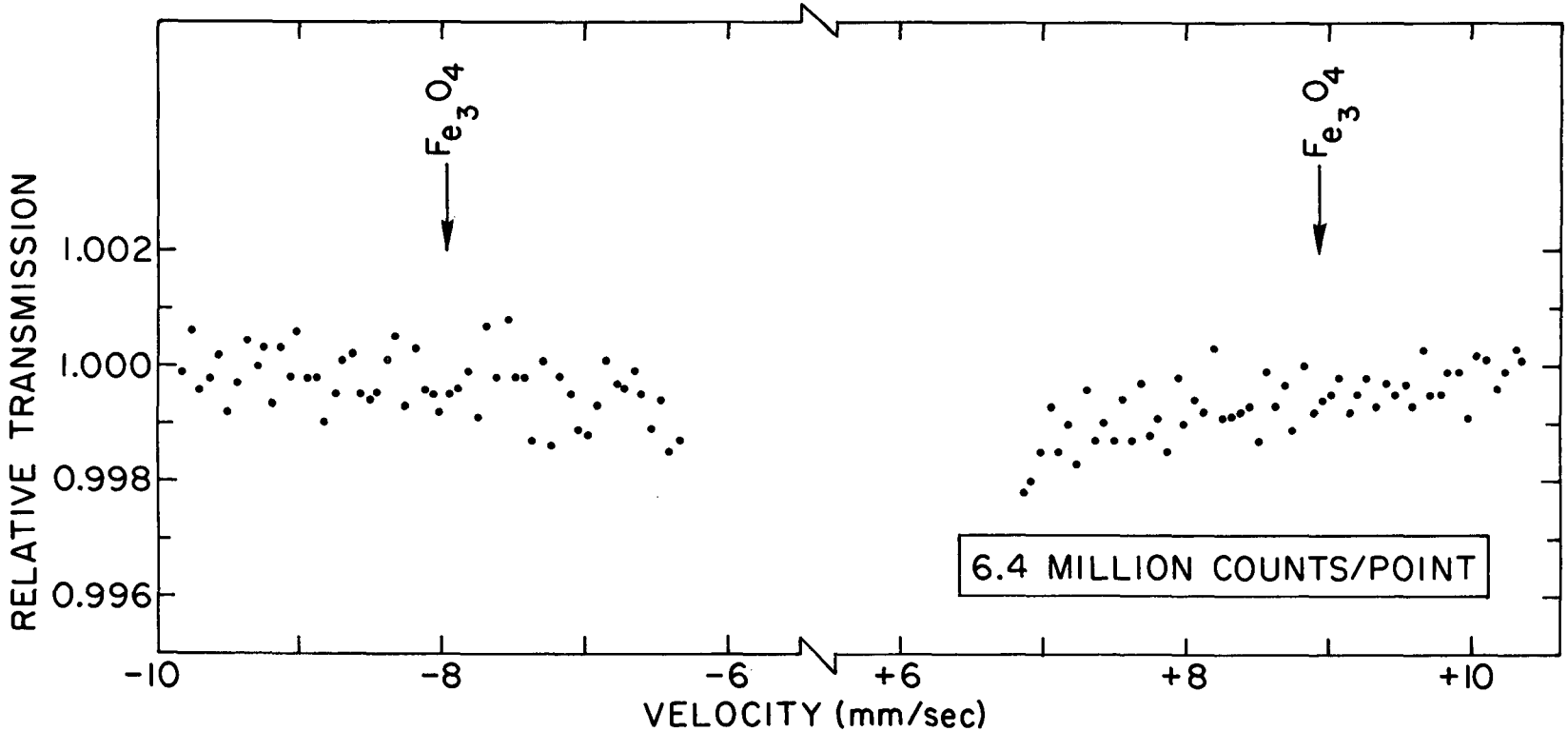
RELATIVE TRANSMISSION
1.002
1.000
0.998
0.996
Fe_3O_4
Fe_3O_4
6.4 MILLION COUNTS/POINT
-10
-8
-6
+6
+8
+10
VELOCITY (mm/sec)

Figure 4

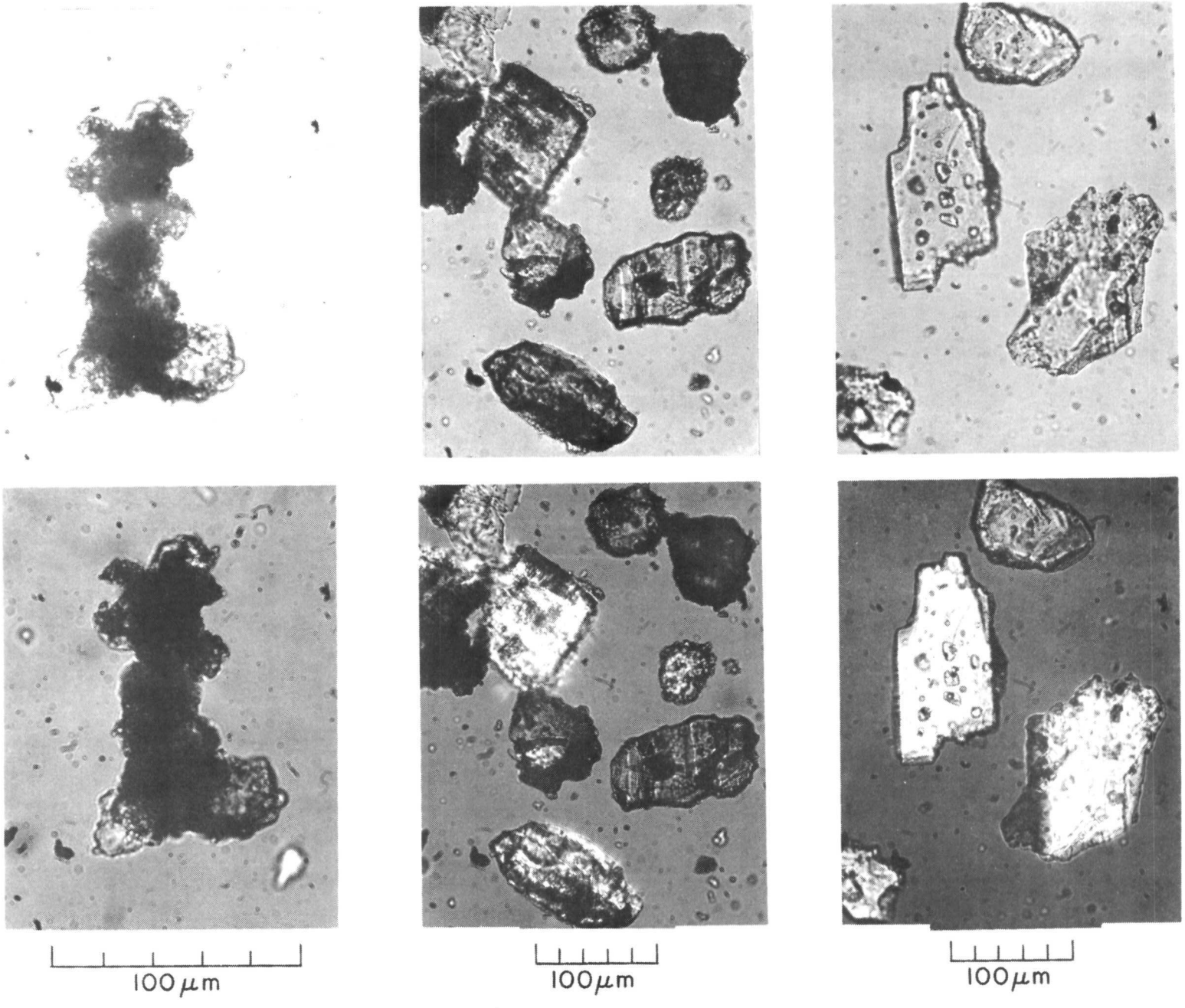

Figure 5

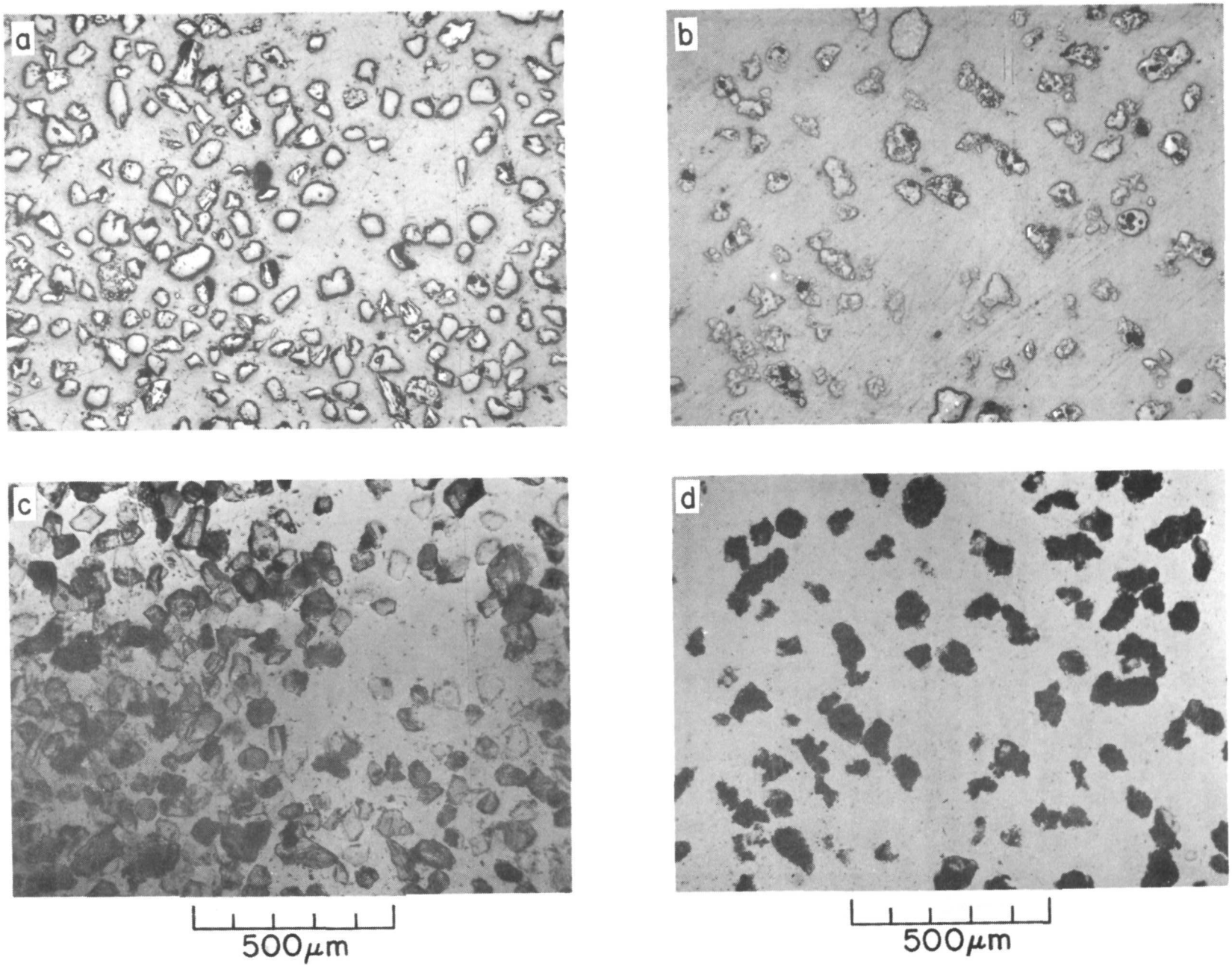

Figure 6

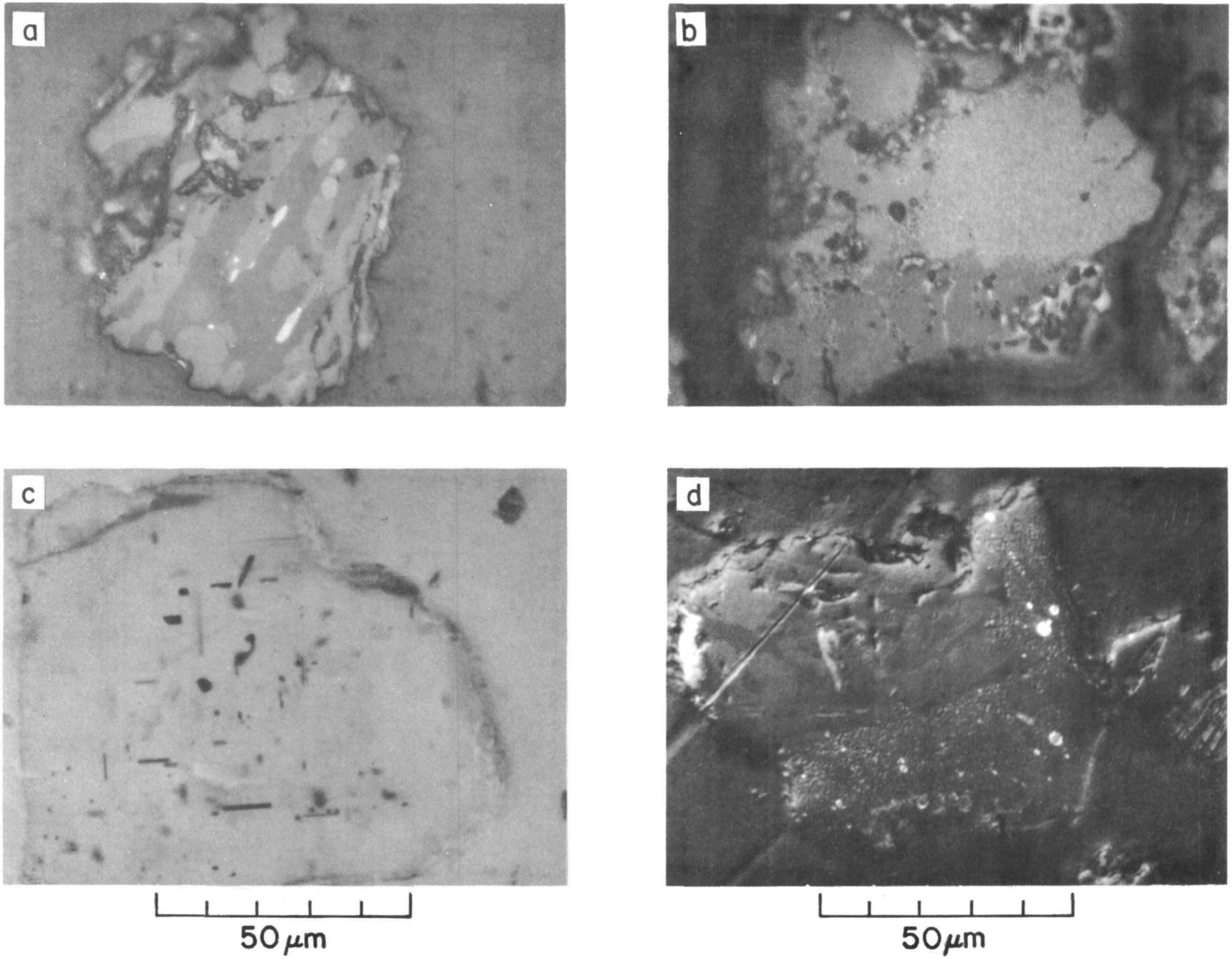

Figure 7

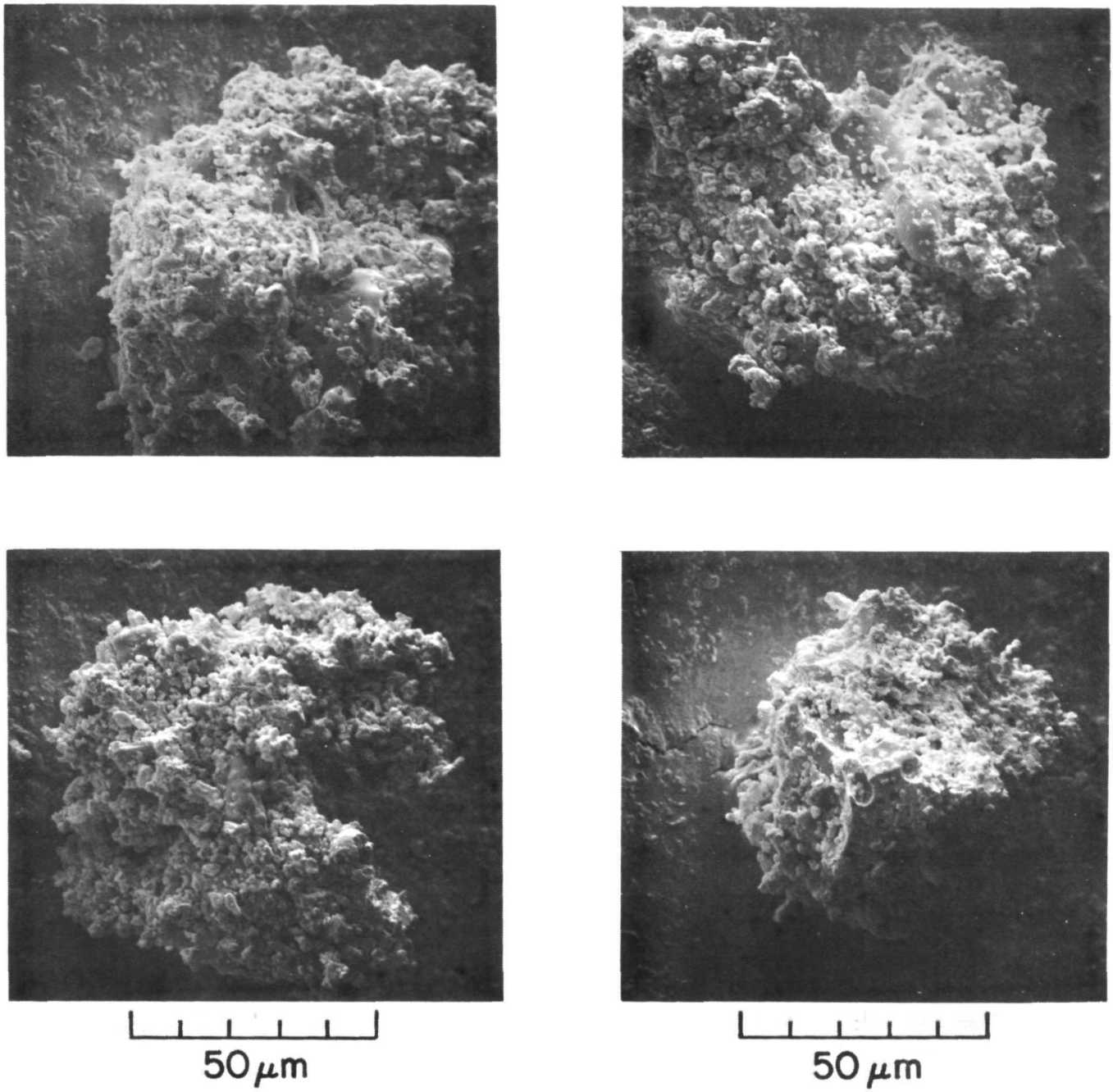

Figure 8

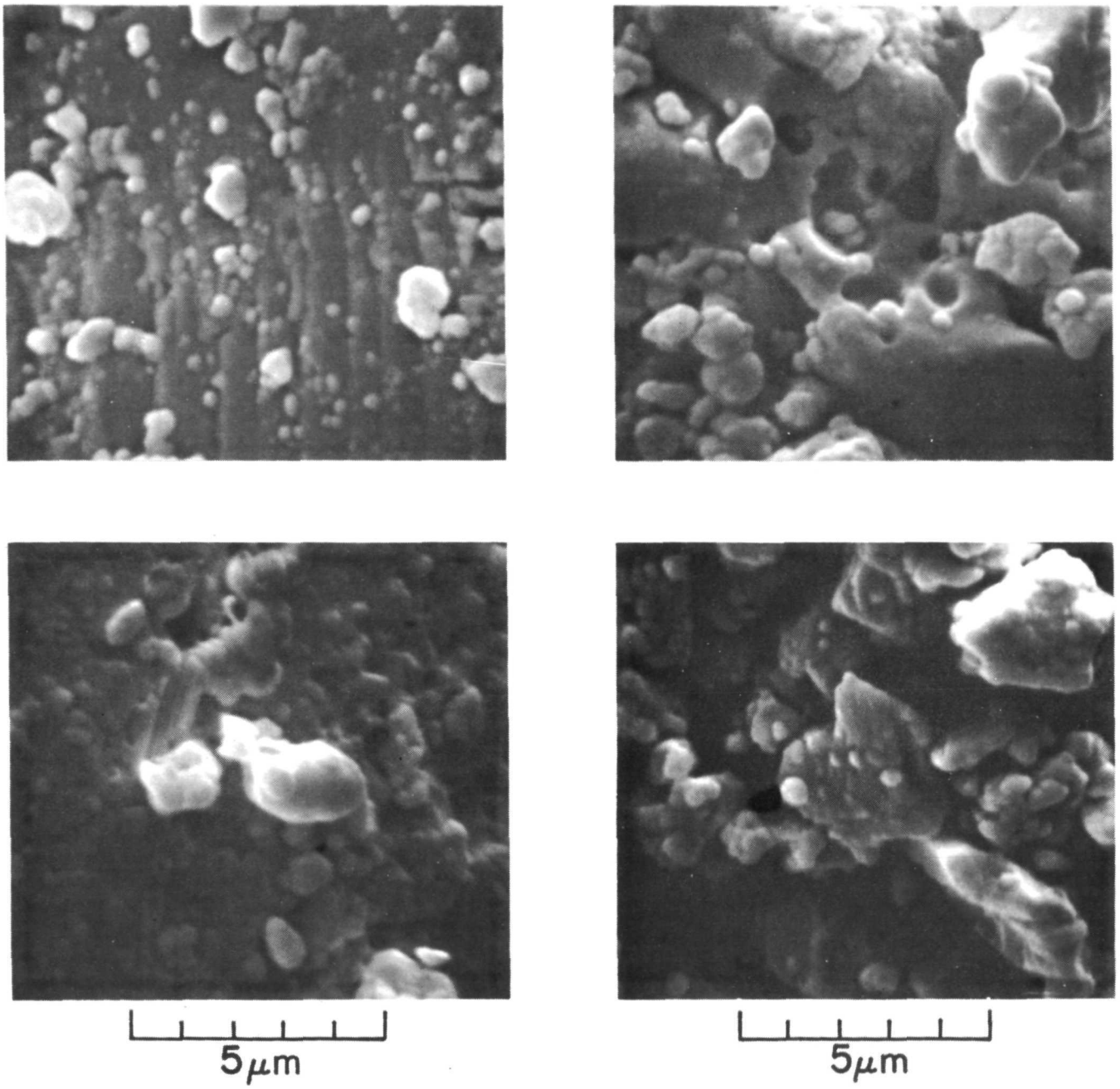

Figure 9

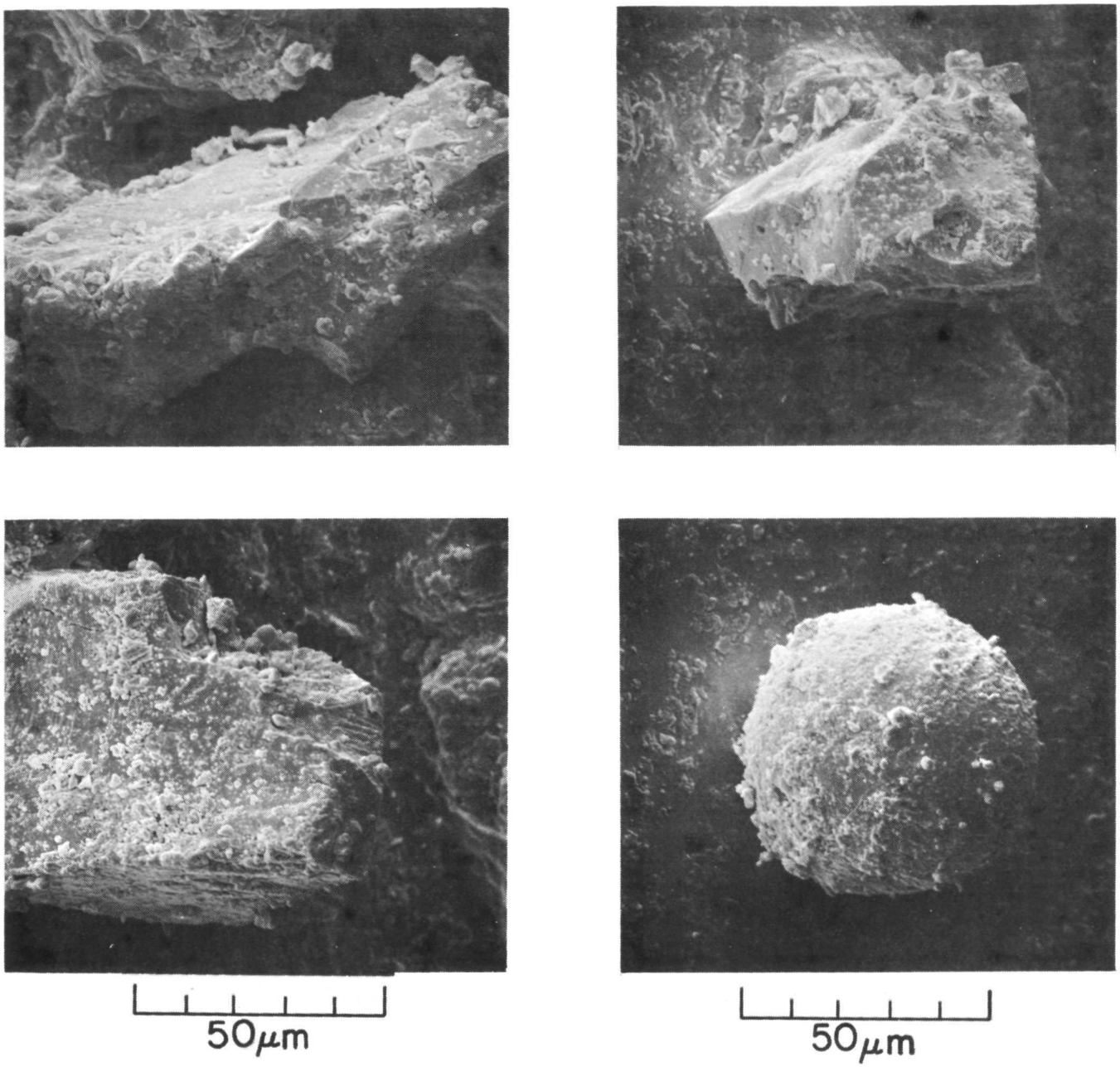

Figure 10