Columbia University in the City of New York

Lamont Geological Observatory

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Six-month Status Report: March, 1965 - August, 1965

Grant NsG 232-62

Research on Quantities and Concentrations of Extraterrestrial Matter through Samplings of Ocean Bottoms.

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I. Work Accomplished

During the period March, 1965 - August, 1965 project research was carried out in the categories of magnetic and non-magnetic separations on ocean sediment flow-in samples, optical and electron-microprobe examinations of polished sections of spherules, magnetic separations on a trawl sample for argon-isotope studies, and model studies on underwater cratering. Work on the flow-in samples has strengthened the probability that the black, magnetic spherules we collect are indeed extra-terrestrial in origin. Electron microprobe results, in conjunction with optical photomicrographs, have provided further data on element distribution within spherules. These results are discussed in more detail below and in the following pages.

A. Magnetic and non-magnetic separations on flow-in samples.

A number of new flow-in samples became available during this report period, and we have been actively engaged in separation of microscopic spherules and other particles whose origin was not immediately explicable. In this work the samples have been sieved and only particles larger than 50 microns in diameter have been retained for magnetic separations. Magnetic and non-magnetic fractions of these residues have been searched optically. Further data on some of the recovered spherules are given in the section titled "Polished-section examinations".

One flow-in sample, V20-110, yielded 28 magnetic spherules larger than 50 microns from an initial, very rapid separation made on a sample weighing 6-8 kgm. This is considered to be rather good hunting and suggests that our further, more complete separations, will yield many more. It was noted that the core from which this material was taken contained extensive zones of volcanic ash in layers younger than our sampling level. The core itself had been taken from a point around 120 miles south of the Aleutians (49°14'N;180°00'W) and it was felt that this presented a good opportunity to look into the question of a volcanic vs extraterrestrial origin for microscopic spherules: if the spherules derive typically from volcanic outbursts, it would be expected that they will occur much more frequently in the volcanic ash layers than in layers of non-volcanic origin. Accordingly, one 51.5 gm composite sample from various ash horizons in this core, and one 51.5 gm flow-in sample with much lower ash content, were examined for spherules. Results of comparative searches in the two sample types were as follows:

(1) 51.5 gm volcanic ash horizon composite sample: 2 black, magnetic spherules, diameters 60 and 200 microns.

(2) 51.5 gm flow-in sample: 4 black, magnetic spherules, diameters 60, 60, 70, and 140 microns.

Since an inverse correlation was found between concentrations of spherules and volcanic ash horizons it is now felt that sources of terrestrial contamination are much less important than had been previously believed. At present, therefore, we are collecting spherules from ocean sediments predating the onset of industrial production of spherules, by using flow-in samples from the bottom of our ocean-bottom cores. This means the spherules are at least natural in origin. But the present work suggests that the only other probable natural terrestrial source of spherules, volcanism, is an unimportant contributor, therefore the vast majority of black, magnetic spherules we collect must be of extraterrestrial origin.

B. Polished-section examinations.

The previous report (September, 1964 - February, 1965) contained a summary of optical- and electron-microscope investigations made on a polished and etched section of a magnetic spherule of natural origin 250 microns in diameter. These showed a subparallel lathlike crystalline network imbedded in a fine-grained matrix material. Metallic inclusions were found at interfaces between these two phases.

During the current report period the principal investigator was privileged to use the electron microprobe facilities at the U. S. National Museum, Washington, D.C. This opportunity has provided further compositional data on the spherule mentioned above, as well as several others. Four spherules of undoubted natural origin and one from a trawl collection in recent red clay were examined with the electron microprobe, and derived compositional data are given in Table I. Percentage estimates of nickel and iron are given in Table II. To calculate these percentages, the raw data were corrected for background and compared to 100% standard compositions. Other corrections (for mass absorption, fluorescence, etc.) were not made because of the small-scale inhomogeneities being measured. From the photos of these spherules (Figs. 1, 5, and 7) it can be seen that the zones occupied by different chemical phases are very narrow - approaching the diameter of the electron beam - and rendering any calculated corrections virtually meaningless. It may be worthwhile to state qualitatively, however, that the true value for nickel may be as much as several percent higher than, and the true values for iron may be as much as several percent lower than, their listed estimates, due to mass absorption effects.

The greatest number of elements was detected in the 250 micron spherule (Fig. 1), which, besides nickel and iron, contains silicon, aluminum, magnesium, and calcium. It is considered significant, also, that all spherules were found to contain nickel.

Figures 2, 3, 4, 6, and 8 show electron-beam scanning photos for various elements in three different spherules. From these it can be seen that silicon tends to be segregated in patches or bands, following the structure of the spherule in various ways. Also as might be expected, nickel was found to be highly concentrated in the metallic inclusions. Nickel was found in all the spherules, but in those not containing metallic inclusions it seemed to be evenly distributed across the section. Apparent segregation of aluminum may be spurious because the final polishing medium was Al_2O_3, but a faint pattern is present in V16-30 (Fig. 3) that is similar to the silicon pattern for that spherule (Fig. 2). Magnesium was not found by scanning to be unevenly distributed in the one spherule where a trace was noted, but this may have been below the capabilities of the scanning method. It seems likely that V16-30 contains an interstitial siliceous phase that also contains other rock-forming cations. Whether this is similar or not to meteoritic silicate material is not known at present.

C. Trawling collections.

Very careful magnetic separations on a trawl sample of red clay from the mid-Pacific have been in progress for over one year. The goal has been to extract virtually all the magnetic fraction from a substantial sample of red clay, therefore no sieving process was carried out to reduce the volume of the original sample. Weight of the red clay sample was 80.92 gm, and the recovered crude magnetic fraction weighs 3.79 gm. Of this crude fraction, 15-25% consists of opaque magnetic minerals, while 25-20% appears to be quartz or feldspar and 60-55% apparently consists of a red, transparent, crystalline material. If the opaque magnetic minerals are the ones giving the argon-isotope anomalies reported by Merrihue and Tilles, the significant magnetic fraction of the original sample weighs 0.57-0.95 gm, equal to 0.70-1.17% of the total sample. The apparent presence of terrestrial quartz and feldspar in the samples run by Merrihue and Tilles means that their results would have been partially masked by this source of terrestrial argon. If this factor were allowed for, the result would be an enhancement of the observed anomalies.

Large magnetic spherules are not particularly common in the red clay mentioned above. One, 140 microns in diameter, yeilded a Stokes' Law density measurement of 4.58 $^{gm}/_{cm}3$ and a calculated mass of 4.14 X 10^{-6} gm. Compositional data on this spherule and others are reported in the section titled "Polished-section examinations".

D. Radioactivation analysis.

This remains a part of the research program but little was done on it during the period of this report. Studies are underway toward computer analysis of data but these will require more time before any results are obtained.

E. Model studies on underwater cratering.

A more elaborate experimental arrangement has been set up, with a pool of water five feet deep. We now have a "cratering platform" which can be lowered to have 0-4 feet of water overlying it. This allows us to make adjustments in depth of water, as well as distance between the bottom and the explosive charge. After each explosion the platform can be lifted out of the water for examination of the resulting bottom traces.

II. Projected Work

A. Magnetic and non-magnetic separations on flow-in samples.

Our program of supplying magnetic fractions of red clays to laboratories requiring them in their search for cosmogenic nuclides will continue. Searches for magnetic and non-magnetic spherules will continue in connection with our own compositional studies.

B. Polished-section examinations.

As a result of work reported here and in previous summaries, it is felt that the combined approach to polished-section studies is capable of great refinement and will be a very powerful tool in studying these microscopic objects. At present we are using optical photomicrography, electron photomicrography, and electron-microprobe analysis on the same polished section. Each method yields its own set of data which gains in value when combined with data from the other methods. For example, optical data on reflectivity indicate the presence of oxide and metallic phases and allow an estimate of their relative proportions. Peels from etched surfaces, photographed in the electron microscope, indicate differences in grain size between chemical phases and allow greater magnification than photos by the optical microscope. Subsequent electron-microprobe analyses give data on element distribution between phases that could not be obtained without the help of the photographs obtained by the other methods. This combined approach appears to be a unique one in this type of investigation, and it will be developed further in the future by refinement of techniques and extension to more individual spherules.

C. Trawling collections.

Examination of trawl samples for extraterrestrial material will continue as the samples become available.

D. Nondestructive radioactivation analysis.

Studies leading toward computer analysis of data will be continued. Analysis of spherules for rarer elements with lower concentrations than can be detected with the electron microprobe will be emphasized as a further amplification of the combined approach described in Section II. B.

E. Model Studies on underwater cratering.

Coarse parameters for the formation of impact traces on the ocean floor will be arrived at without taking account of scaling effects. These will be considered in the future. Results in the current series of experiments will be summarized in a succeeding report.

F. Phase-equilibrium studies.

High-temperature phase-equilibrium studies have been included in a pending proposal for extension of this grant. These will be conducted in the system Fe-FeO-MgO-SiO_2, a knowledge of which is basic to our understanding of processes involving the formation and chemical evolution of meteorites, as well as cosmic dust. This would be, in part, an extension of the principal investigator's doctoral research. It is hoped that an associate investigator will be found to undertake part of this work, and conversations with a suitable candidate are now taking place. The extent of the investigations proposed in this section will depend on finding the right person to carry them out.

III. Acknowledgment

The opportunity to conduct the research described in this report, made possible by NASA Grant 232-62, is gratefully acknowledged.

Respectfully submitted,

William A. Cassidy
(Research Scientist)

Table I. Raw data on element concentrations in five black, magnetic spherules. Ten-second counts corrected for background. Hyphenated values indicate limits of variation where a range of values was found across the section.

	Ni	Fe	Co	Si	Al	Mg	Ca	K	Na
V16-79-160μ	3659-6473	17201-17530	n.d.	0-22	tr?	0	0	0	tr?
V16-79-160μ (one spot: may be hole)	n.d.	n.d.	n.d.	3302	1059	n.d.	n.d.	n.d.	n.d.
BBD#1-140μ	0-172	16027-19432	n.d.	0-580	0	0	0	0	0
BBD#1-140μ (metallic inclusions)	14127-21670	17292-18111	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
V16-30-250μ	454-1286	13246-16423	tr?	513-5038	0-1466	36-390	0	0	0
V15-30-250μ (isolated spots)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	86-342	n.d.	n.d.
V16-79-180μ	1860-3281	16167-16739	tr?	0	0	0	tr	0	0
V20-110-90μ	228-645	19078-19489	n.d.	0-40	0	0	n.d.	n.d.	n.d.

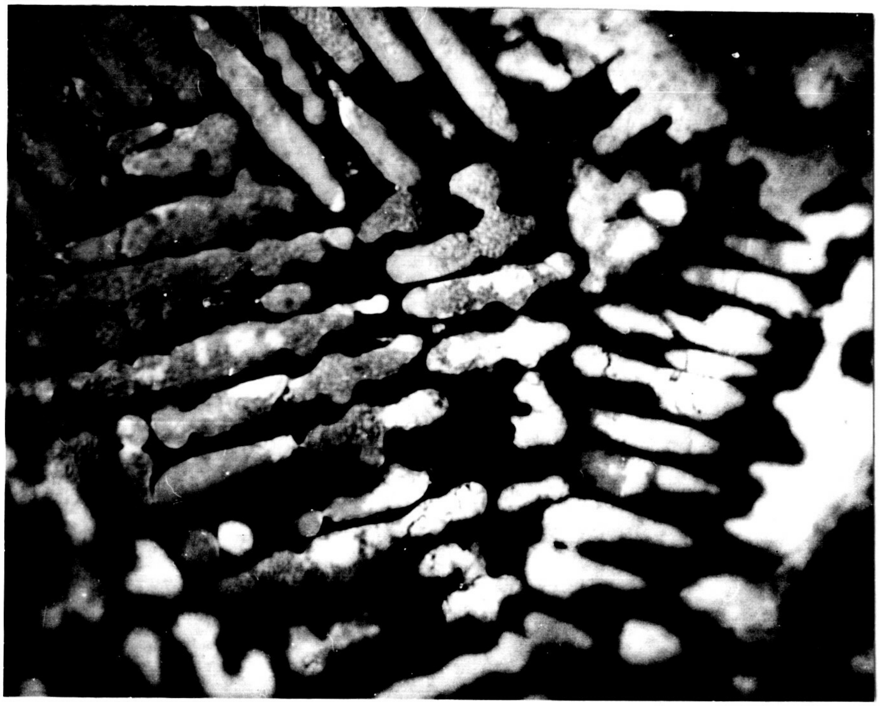
Table II. Percentage estimates of Ni and Fe in five black, magnetic spherules.

	Percent Ni	Percent Fe
V16-79-160μ	4.1 - 7.2	63.6 - 64.8
BBD#1-140μ	0.1 - 0.2	59.3 - 71.9
BBD#1-140μ (metallic inclusions)	15.8 - 24.3	63.9 - 67.0
V16-30-250μ	0.5 - 1.5	51.0 - 63.2
V16-79-180μ	2.2 - 3.8	62.3 - 64.5
V20-110-90μ	0.2 - 0.7	70.6 - 72.1

FIGURES

Fig. 1. Polished section of a black magnetic spherule 250 microns in diameter from V16-30. Note radiating, lathlike structure. This spherule was described in greater detail in the previous report.

Fig. 2. Electron-beam scanning photo of part of V16-30 (250 microns) in the light of $SiK\alpha$. Note that silicon concentrations appear to have the same pattern as the laths or as the interstices between the laths.



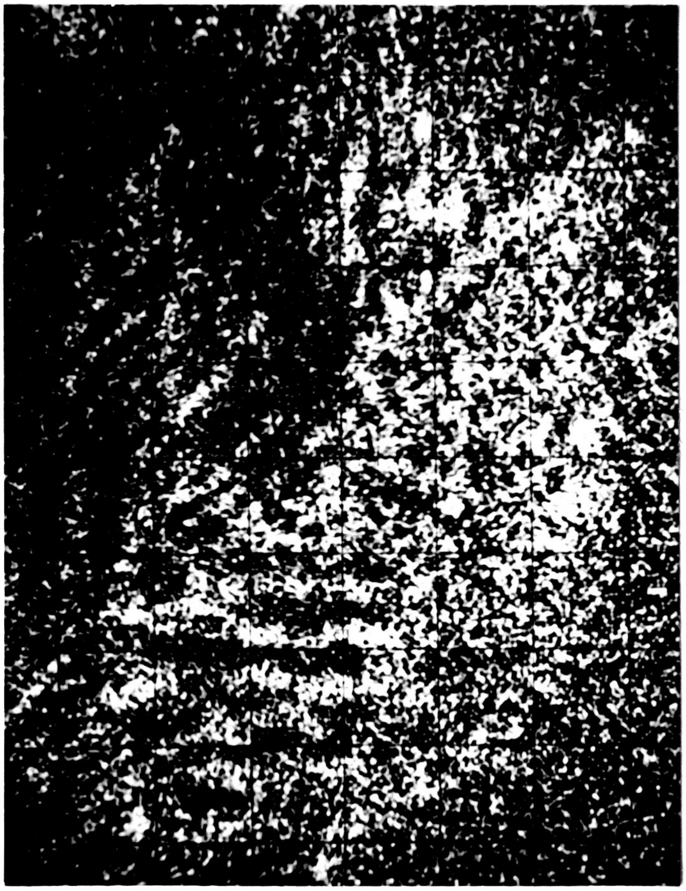
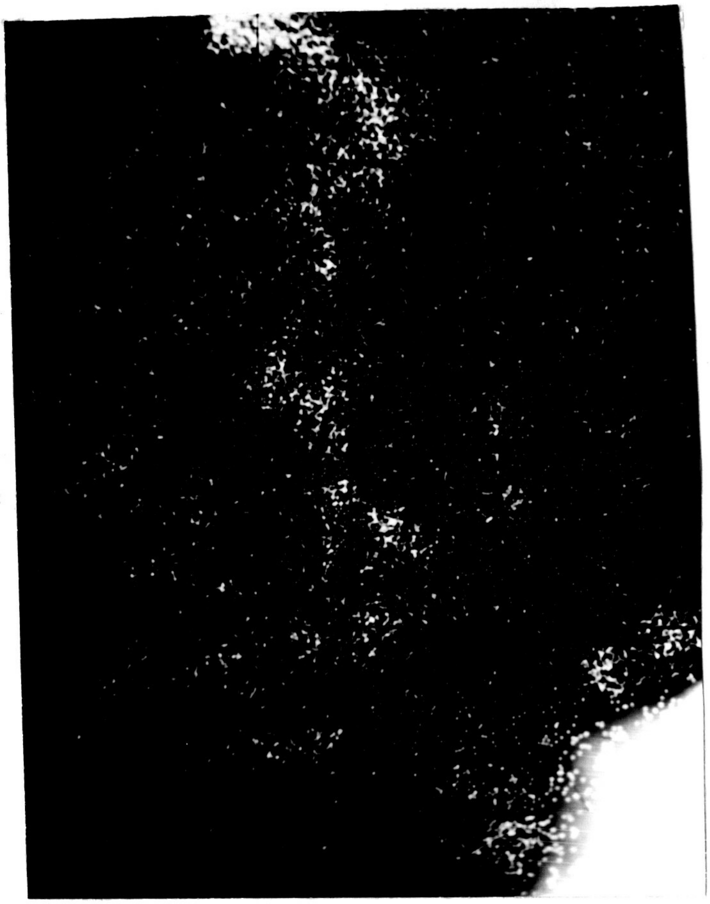


Fig. 3. Electron-beam scanning photo of part of V16-30 (250 microns) in the light of $AlK\alpha$. Apparent segregations of aluminum may be spurious, but the radiating lathlike pattern appears faintly.

Fig. 4. Electron-beam scanning photo of part of V16-30 (250 microns) in the light of $MgK\alpha$. Magnesium distribution appears even, but may be only background, due to low observed concentrations of that element.



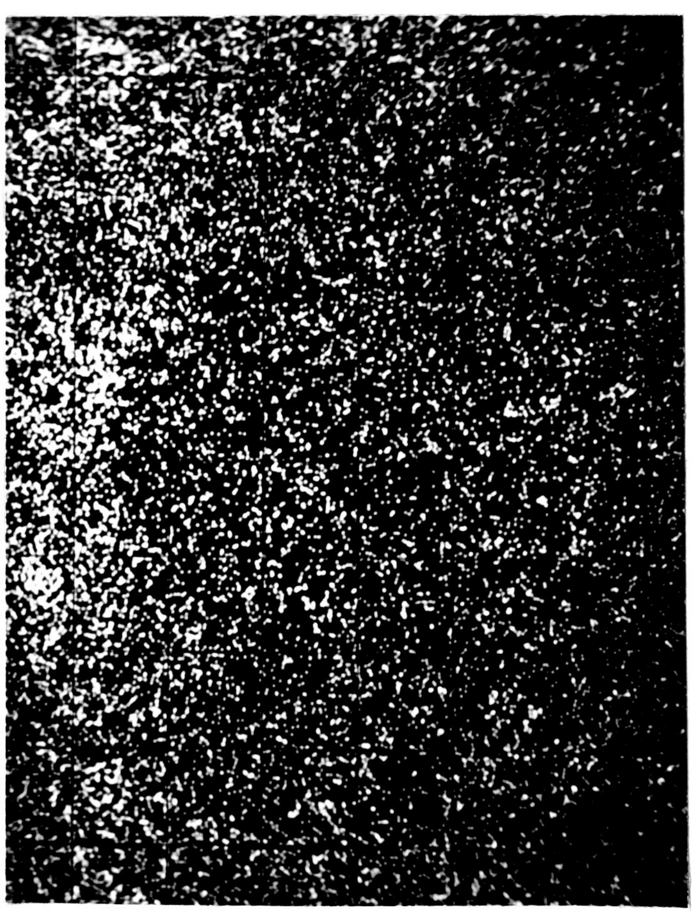


Fig. 5. Polished section of a black, magnetic spherule 160 microns in overall diameter from V16-79. Note black interstitial inclusions.

Fig. 6. Electron-beam scanning photo of part of V16-79 (160 microns) in the light of SiKα. These match the black interstitial inclusions of Figure 5, which therefore appear to be rich in silicon.

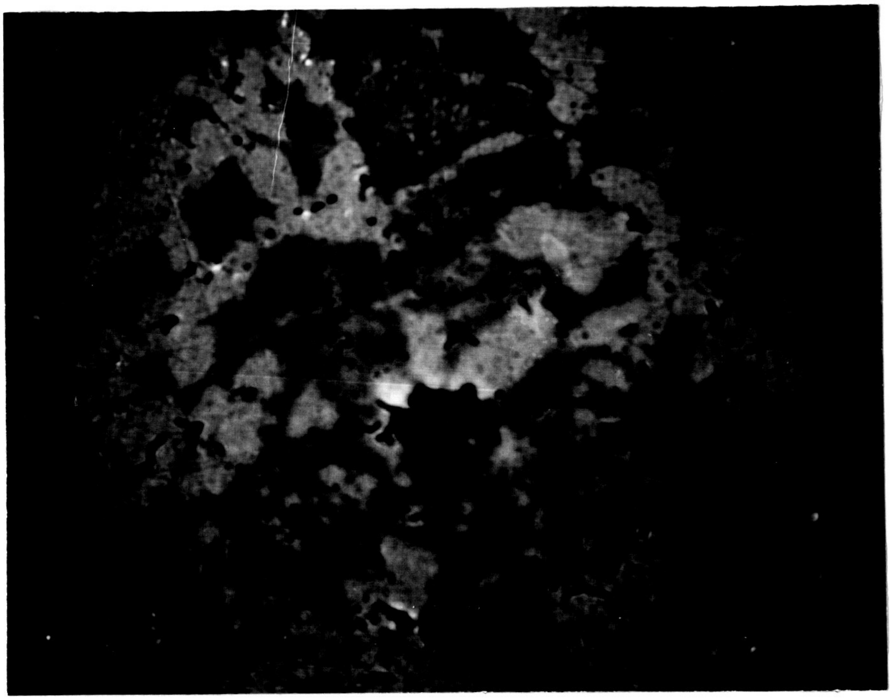


Fig. 7. Polished section of a black, magnetic spherule 140 microns in diameter from trawl sample BBD#1. Note highly reflecting metallic inclusions.

Fig. 8. Electron-beam scanning photo of part of BBD#1 in the light of NiKα. Note that areas of high nickel concentration match locations of metallic inclusions.