## STUDY OF PARTICLES COLLECTED BY THE 1965 LUSTER ROCKET

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NOVEMBER 1966 DAC-59168

#### MISSILE & SPACE SYSTEMS DIVISION DOUGLAS AIRCRAFT COMPANY, INC. SANTA MONICA/CALIFORNIA



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# STUDY OF PARTICLES COLLECTED BY THE 1965 LUSTER ROCKET

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#### ABSTRACT

The NASA-Ames Research Center's Luster micrometeorite collecting rocket was successfully flown on 16 November 1965, during the Leonid meteor shower and collected for 206 seconds between altitudes of 63 and 144 km. Our collector surface consisted of titanium, tungsten, and molybdenum electron microscope screens each with a substrate of silicon monoxide and a shadow coat of nickel or indium; they were designed to withstand heating to 1,000°C. The prime objective was to test, by heat annealing, whether radiation damage distinguished extraterrestrial from terrestrial particles; the secondary objective was to obtain flux, composition, and other data about the micrometeorites.

Heating tests were completed on one group of 58 particles and on 2 individual particles. Of these, only one particle showed evidence of possible radiation damage annealing but it also showed some morphologic changes. It was concluded that all the studied particles could be contaminants and that more particles must be studied, together with composition and other data, before a conclusive answer can be given as to whether radiation damage is a criterion for micrometeorites. However, the heat-produced morphologic changes discovered in this study may also prove to be important in distinguishing micrometeorites from contaminants.

No chemical elements have yet been identified in preliminary electron probe analyses.

The flux observed, based on 8 individual particles and 2 groups of particles, is 0.3 particle/mm<sup>2</sup> of collector surface if each group is counted as a single particle, or 2 particles/mm<sup>2</sup> if every particle in the group is counted. These values do not include corrections for one-shadowed contaminants or for scanning efficiency.

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#### IN TRODUCTION

The importance of cosmic dust in general and micrometeorites in particular hardly needs to be pointed out today. These particles are of great interest to science and the space program because they are additions to, and erosional agents of, lunar and planetary surfaces; they are increments to the lithosphere and the atmosphere of the Earth; and they are potential erosional and destructive agents of man-made space hardware.

Although nearly 100 years have passed since micrometeoritic contributions to the sediments of terrestrial oceans were first recognized (Murray, 1876), an intensive study of these particles has commenced only with the advent of high-altitude aircraft, rockets, and Earth-orbiting satellites during the past decade. The studies have used two general approaches. The first is the indirect approach that deduced particle flux, velocity, and other characteristics from impacts on sensing devices usually flown on satellites. The second is the direct approach in which the particles themselves are collected and studied.

This report presents the results of a 4-month investigation of particles collected with the NASA-Ames Research Center's Luster rocket which was successfully flown on 16 November 1965. The study was performed under Contract NASw-1401, executed 7 July 1966 with the National Aeronautics and Space Administration Headquarters. Its primary purpose was to test whether radiation damage, as revealed by resumption of the crystalline state with heating, could provide a criterion for distinguishing extraterrestrial from terrestrial particles, a crucial problem in all direct particle studies. A secondary purpose was to determine size, shape, abundance, composition, and other important characteristics of micrometeorites.

We wish to thank P.A. Larssen and E.L. Miller for their valuable help with the electron probe analysis.

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#### PREVIOUS WORK LEADING TO CONTRACT STUDY

The first successful collection of particles from high altitudes for direct study was made with the Venus Flytrap rocket flown on 6 June 1961 by the Air Force Cambridge Research Laboratories (AFCRL). The AFCRL investigators, on the basis of controls built into the experiment, concluded that micrometeorites had been collected and were distinguishable from contaminant particles of terrestrial origin. They also found that micrometeorites displayed no diffraction patterns in the electron microscope, possibly because of radiation damage sustained in space (Soberman, <u>et al.</u>, 1961; Hemenway and Soberman, 1962). An alternative explanation, that the particles may have been crystalline but too dense to yield diffraction patterns, was ruled out by Greenman and Gilpin (in press), who showed that particles of the silicates, olivine and enstatite, in the same size range yielded distinct diffraction patterns.

AFCRL subsequently provided us with samples from this collection. We attempted to test for the presence of radiation damage by heating the particles to see if crystallinity, as evidenced by the appearance of electron diffraction patterns, could be restored at temperatures below the melting point. This test was important because no positive criteria are known by which micrometeorites can be recognized without ambiguity in any particle collection. If radiation damage were present, it would constitute such a criterion. The reason is that the bulk of the terrestrial contaminants in micrometeorite collections should be either crystalline or amorphous, not radiationdamaged to any significant degree. Particles of extraterrestrial origin, on the other hand, could suffer such damage to the crystalline lattice, according to the calculations of Ryan (1964). Moreover, the damage could survive in small particles that fall through the atmosphere too slowly to undergo appreciable frictional heating. Although several types of substrate were used as collectors on the Venus Flytrap flight, none were well suited for heating tests of this kind. The least faulty substrate, thin formvar double-shadowed with aluminum, had the disadvantage that the low melting point of aluminum, about 660°C, prevented the runs from being carried out above about 500°C. In addition, at temperatures below 500°C, heating caused enough damage to the substrate to make it almost impossible to follow the same particle through every heating stage. For this reason, a statistical approach was used in which as many particles as possible were examined after each stage.

The results of heating to 250°C, 400°C, and 500°C were that, in general, the percentage of particles showing evidence of crystallinity increased with increasing temperature. Statistical analysis indicated that the crystallinity was probably produced by the heating, although the differences in the percentages were not great enough to be certain on this point. The d-spacings of the diffraction spots were found to correspond rather well with those of olivine and enstatite, the major minerals of the most abundant meteorite type, indicating that annealing of radiation damage could have occurred. On the other hand, similar correspondence was found with other materials, such as mica and aluminum oxides and hydroxides, that could be contaminants or reaction products formed in the course of the heating tests. These studies are described by Greenman (1963, 1964).

The results of these first tests were thus suggestive, though inconclusive. As the difficulties were caused chiefly by the inadequate heat resistance of the available substrates, we developed a suitable substrate and supporting screen assembly for subsequent rocket collection flights. This consisted of a titanium supporting screen with a substrate of silicon monoxide shadowed with nickel. Tests showed that this assembly had good collection efficiency and was able to withstand heating to 950 °C with only a small amount of damage, although the nickel shadow was found to have disappeared. The disappearance of the nickel was considered an advantage in that it reduced the substrate thickness. For this reason, indium, which has a vapor pressure at 1,000 °C much greater than that of nickel, was also used as a shadowing metal on the Luster collecting substrates.

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#### LUSTER 1965 FLIGHT AND COLLECTOR DESCRIPTION

The Luster micrometeorite collecting rocket was successfully flown from the White Sands Proving Ground, New Mexico, on 16 November 1965 during the Leonid meteor shower. The nose cone carried particle collectors mounted on arms that extended and retracted during flight, and the entire assembly was parachuted to the ground and recovered. Collection was carried out for 206 sec between altitudes of 63 and 144 km (Farlow and Blanchard, 1966). The experiment was under the direction of the NASA-Ames Research Center and included collectors from guest scientists at Douglas and at a number of other organizations.

The Douglas collection surface was prepared by first casting a thin film of parlodion on water. The parlodion was then picked up on the titanium screens and dried. In addition to titanium, screens of tungsten and molybdenum (other high melting point metals) were also used to provide a variety of collector types for the heating experiments. The titanium and tungsten screens have keyed center holes and elongated openings which greatly facilitate the mapping of particles and finding them again after each stage of heating (Figure 1). The silicon monoxide substrate was next deposited on the parlodion, after which the parlodion was dissolved with amyl acetate. When dry, the screens and substrates were coated with approximately 25Å of nickel or indium deposited in a vacuum evaporator at an angle of 15° to the surface. After preparation, several screens were examined in the electron microscope to make sure that the substrates were in good condition and that contamination was slight.

The finished collector screens were mounted on a Lucite base,  $55 \times 50 \times 3$  mm, with Scotch brand "410" double-sided tape. An aluminum-coated shield was attached to one end of the Lucite plate to cover three rows of screens with an air gap of about 1.5 mm. This was for the purpose of contaminant control;

the covered screens were shielded from impact by particles during the collection phase of the flight but could accumulate contaminants carried in by air currents at other times. The completed collection surface (Figure 2) was shipped in a cleaned and sealed plastic box to NASA-Ames Research Center for incorporation into the Luster flight package. Also, an identical surface was shipped to be used as a spare, and a set of slides with 10 substrates each was shipped to be exposed in the Ames laboratory and in the White Sands portable clean room where the final assembly of the flight instrument took place. These were to be used as contaminant controls.

After the flight, the flight sample, spare, and controls were returned to Douglas where, in a Class 100 clean room, they were opened and given a second nickel or indium coat at 15° to the surface and at about 90° to the first coat. This double-shadowing technique served as a control to separate preflight contaminants, which should show two metal shadows, and postflight contaminants, which should show none, from particles collected in flight, which should show only one. Contaminants deposited during the time between shadowings should also show one shadow. Double-shadowing, in-flight shielding, and laboratory-exposed substrates thus were the three types of contaminant control used in this study.

#### STUDY PROCEDURES AND RESULTS

#### OBJECTIVES AND PROCEDURES

The prime objective of this study was to investigate, by means of heat annealing tests like those begun in the earlier Venus Flytrap work, the question of whether radiation damage can be used to distinguish extraterrestrial from terrestrial particles. Secondary objectives of the study were to obtain composition information on the particles from electron probe and electron diffraction analysis, to obtain data on abundance from particle counts, and to obtain data on size, shape, and other morphological properties from electron microscope observations.

To accomplish the primary objective, all particles with one shadow were initially considered to be of possible extraterrestrial origin and were mapped and counted. The only exception to this was in the few cases where particles of distinctive morphology were found with both one and two shadows; all of these were classed as contaminants. The procedure then was to heat the particles in stages of increasingly higher temperatures and after each stage to examine the electron diffraction patterns for evidence of newly produced crystallinity. Test temperatures began at 200°C and were increased in 200°C intervals to a maximum of 1,000°C or until diffraction effects were observed in the particles. Heating was done in a three-zone resistanceheated tube furnace (Figure 3). Temperatures were maintained within 10° of the nominal setting for 1 hour, and the furnace was then allowed to cool slowly to room temperature. In the early runs, the screens were placed in a tungsten container which, in turn, was placed in an Inconel tube that fit into the furnace. At higher temperatures, some sticking of the titanium screen to the tungsten container and some diffusion of one metal into the other made it necessary to replace the Inconel tube with one of quartz and the tungsten box with quartz boats to hold the screens.

To prevent chemical reactions of the particles with atmospheric gases, especially oxygen and water vapor, the heating was carried out in an inert gas atmosphere. The tube was first purged for 1/4 hour with high purity argon at a flow rate of 7 liters/min in the Inconel tube and 2.4 liters/min in the quartz tube. These were reduced to 2.4 and 1.4 liters/min, respectively, in the Inconel and quartz tubes during the heating and subsequent cooling.

#### STUDY RESULTS

#### Heat Annealing Experiments

Particles with one shadow were found on 4 of the 17 screens examined. These screens and the coordinate system by which they are designated are shown in Figure 2. All four of the screens with one-shadowed particles are titanium with nickel shadow coats.

The one-shadowed particles found consist of 8 individual ones (2 of which were found only after heat treatment), one group of 5, and one large group of 58 or more. Of these, the large group and 2 individual particles have gone completely through the heating tests, 2 individual ones are still being tested, 2 have been lost or are otherwise not capable of being studied further, and the remaining group and 2 individuals are available for additional investigations.

Figure 4 shows a particle from screen G-16, the only particle of those studied in which definite diffraction effects from heating have been found. The particle, about 0.9 micron in its longest dimension, had a dense nucleus surrounded by a less dense rim. Essentially no change occurred after heating to 400 °C. Neither were there any electron diffraction effects associated with the particle (the broad diffuse rings are attributable to the substrate). After heating to 600 °C, however, the nucleus became much less dense, and a dark, mottled area appeared around the particle, although its original outlines were not greatly modified.

The diffraction pattern after heating to  $600^{\circ}$ C shows a symmetrical line of spots, an inner pair corresponding to a d-spacing of about 2.8Å and an outer, second-order, pair corresponding to a d-spacing of about 1.4Å. A single

third-order spot of d-spacing 0.9Å is also faintly seen. These are associated with the particle because the diffraction pattern of the substrate is devoid of these spots, although the sharpening of the rings indicates that some recrystallization of the nickel shadow coat has taken place.

Because of the morphological change in the particle, it is not possible to conclude definitely that annealing of radiation damage occurred. In fact, these changes at 600°C make one suspicious as to whether it is of extraterrestrial origin at all, despite its single shadow. This point of the possible use of heating to distinguish extraterrestrial from terrestrial particles will be discussed in a later section. The 2.8Å d-spacing is close to that of prominent lines in both olivine and enstatite. However, definite identification cannot be made from only one d-spacing, because other substances may also have lines with this value. It is also possible that the mottled area is caused by recondensation of material volatilized from the particle in the course of heating and that this crystallized material has given rise to the diffraction pattern. Hemenway and Soberman (1962) were able to produce a similar effect by heating with the electron beam in the electron microscope. A third possibility is that the particle may have simply changed position by sliding, leaving the mottled material as a residue. This is suggested by examination of the stereo pair (Figure 5) taken with two different tilt positions of the particle on a tilting stage. This interpretation would lend support to the possibility that radiation damage annealing has occurred, because the mottled residue in this case could be the result of mechanical causes and not necessarily of chemical reactions or vaporization phenomena.

Figure 6 shows particles from screen N-16. These are a large swarm of the type termed "fluffy" by Soberman <u>et al.</u> (1961) and considered by them to be of extraterrestrial origin. The particles have highly irregular outlines and appear to be composed of a mosaic of smaller polygonal units. The largest is about 1/4 micron and they range in size down to less than 1/10 micron. The pictures before heating and after 600°C show the same particles; the one after 800°C is an adjoining group of the same type. No changes seem to have taken place in particle morphology and no diffraction effects were observed, even with a tilting stage. After the 800°C heating, it was found that some

rupture of the substrate near the particles had occurred, so it was decided to subject the particles at this point to electron probe analysis in an attempt to obtain composition information before possibly losing them with further heating.

If these particles are indeed extraterrestrial, their failure to yield diffraction patterns is because (1) they are amorphous, (2) they are radiation-damaged but must be heated above 800°C to be annealed, or (3) they are so intensely radiation-damaged that they are not capable of being annealed, as Primak (1960) found to be the case with quartz that was intensely irradiated with fast neutrons. If the first case is true, the radiation damage criterion for extraterrestrial origin would not be applicable universally. This is also the situation if the third case is true if heat annealing is used to detect radiation damage. If these are contaminants, on the other hand, they have no bearing on the validity of the radiation damage criterion. In this case, their diffraction behavior is explained by their amorphous nature.

A particle from screen P-15 is shown in Figure 7. It is about 0.8 micron in its longest dimension and has a three-lobed form suggesting a merger of three more or less spherical particles. It showed little, if any, change after 200°C (the apparent distortion in the picture is caused by its tilted position on the tilting stage). Beginning with the 400°C heating, however, it showed progressive disintegration until nothing but a faint residue was left after 1,000°C. No diffraction effects were observed. The sporadic spots appearing in the 800°C diffraction pattern are probably associated with the nucleation of the nickel shadow coat to a more coarsely crystalline state.

Screen A-16 is still undergoing heating tests. Figure 8 shows a particle with a dense, spherical core with a somewhat less dense rim providing a polygonal outline. It is about 1/2 micron in diameter. Figure 9 shows a large fluffy particle, about 1.5 micron in its longest dimension, that was only discovered after the 400°C heating. It yielded no diffraction pattern after being heated to this temperature.

An interesting instance of how spherical-looking, one-shadowed contaminants change with heating is afforded by the particles shown in Figure 10 from

screen C-16. These are a group of more or less spherical, in part polygonal, particles mostly between 1/10 and 1/5 micron. At every heating stage except that at 200°C, the particles pictured show only one shadow. The particles at the 200 °C stage, from another mesh opening on the same screen, have two shadows. Such a close association of similar particles showing both single and double shadows strongly suggests that all are contaminants that entered during the deposition of the first shadow coat. Possibly, they are incompletely vaporized droplets of nickel some of which arrived just before, others just after, the shadow coat. Comparison of the particles in successive heating stages shows that they have become circular and have expanded in size, although the dark crescent shadow marking the original particle boundary maintains its size more or less unchanged. This was a screen in which good evidence was observed of tungsten-titanium diffusion, which accounts for the highly crystalline substrate and the breaking up of the screen after 1,000°C. It also accounts for the diffraction patterns which show not only sharpening of the nickel rings but also additional rings and the spot pattern at 1,000 °C, probably caused by chemical reactions among the substrate materials.

To summarize the results of the heating tests, new diffraction spots were found in a particle that had also undergone morphologic change (screen G-16), but another particle that showed morphologic change (screen P-15) and others that did not (screen N-16) showed no new diffraction spots. Quite possibly, the two individual particles tested that changed in the course of heating are not of extraterrestrial origin because silicates and iron, of which, at first guess, micrometeorites may be largely composed, are stable at these temperatures. (It is assumed that they are also stable under these conditions in particles of submicron sizes, but this assumption should be tested.) The heating technique may thus have diagnostic value beyond that of radiation damage annealing alone. Contaminant particles of the type found with both one and two shadows on screen C-16, as well as organic substances and possibly hydrates and other materials, can easily be distinguished from stable materials by heat-produced morphologic changes even though all particles may have only one shadow.

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If the group of fluffy particles tested are of extraterrestrial origin, their failure to show either morphologic changes or diffraction patterns may indicate that the radiation damage criterion is not applicable to particles of this type. On the other hand, these, too, may be contaminants. To determine conclusively whether radiation damage is a criterion for extraterrestrial origin will, therefore, require tests on many more particles and the integration of data from these tests with composition information especially, as well as with morphologic and other information. Additionally, further investigation of heat-produced morphologic changes in silicates, iron, and other appropriate materials should be undertaken because these changes may well be of value in classifying certain particle types as contaminants.

#### Electron Probe Analysis

Electron probe analysis was carried out with an Applied Research Laboratories Model EMX electron microprobe X-ray analyzer. The particle on screen G-16 was subjected to analysis; the fluffy particles on screen N-16 are presently being examined.

Because little work has been done on submicron particle analysis with the electron probe, a large portion of the preliminary effort made in this study was necessarily devoted to developing successful techniques. One of the first difficulties encountered was the location of the particle with the probe, since the light microscope of the EMX does not give a high enough magnification to see it directly. With the use of the electron microscope to obtain a sequence of pictures of successively lower magnification, it was possible to localize the substrate area sufficiently for the particle to be picked up by the beam. Figure 11 shows the low-magnification electron microscope picture of the particle and of neighboring contaminants which assisted greatly in locating it. Figure 12 is the back-scattered electron display of the probe showing the particle and its neighbors.

X-ray scans showed only that two of the contaminant particles contained Na (Figure 13) and one contained Fe (Figure 14), neither of which was found in the particle of interest. The beam was next set on the particle and 10-sec counts were made to detect the elements from Mg to Ti (atomic numbers 12

through 22), as well as Cr, Mn, Fe, Ni, Zn, Au, and Pb. None were found, possibly because the particle may have become too thin in the course of the heating tests. It is likely that with longer counting periods and with other improvements in technique the major elements present in the particle will be detected.

#### Abundance and Size Data

A total of eight individual one-shadowed particles was found on the four screens indicated in Figure 2. In addition, groups of particles were found on two of these screens, the large swarm of at least 58 fluffy particles on N-16 and a group of 5 on P-15. Those not already pictured are shown in Figures 15 through 18. Figure 16 is interesting with regard to the source of fluffy particles. Although the large particle shown is itself dense at the center, it is surrounded by a dozen or more small fluffy particles that appear to have been thrown off from its edges. The particle in Figure 17 is also of interest because of its resemblance to the particle on screen G-16 (Figure 4) that yielded diffraction spots with heating.

To calculate the abundance data, we note that a central area of the screen of 2.1-mm diam is open to view in the electron microscope. The open mesh area makes up 56% of the titanium screen and 70% of the tungsten, giving 1.9 and 2.4 mm<sup>2</sup>, respectively, for the available scanning area of each screen. The total area of the 10 titanium and 7 tungsten screens scanned in this study is, then, 36 mm<sup>2</sup>. If the 2 groups are counted each as a single particle, the total of one-shadowed particles is 10, giving a value of 0.3 particle/mm<sup>2</sup> of collector area. If, on the other hand, each particle of the group is counted, the large fluffy group dominates the total, which now rises to 71. This gives a value of 2 particles/mm<sup>2</sup> of collector area.

These figures can be compared with the value of 7 particles/mm<sup>2</sup> given by Soberman <u>et al.</u> (1961) for the Venus Flytrap. It must be remembered, however, that the Venus Flytrap data are for particles estimated to have been extraterrestrial, the suspected one-shadowed contaminants having been eliminated from the count. In the case of this study, the count is for all oneshadowed particles, with the few exceptions mentioned earlier, so that the two flux values are not strictly comparable. However, a rough estimate of the scanning efficiency can be obtained from the fact that of the total of 8 particles and 2 groups, 2 particles were discovered during examination after the heating tests, not in the original preheating scans. This increase of 25%, taken as a measure of efficiency, gives maximum flux values of 0.4 and 2.5 instead of 0.3 and 2 particles/mm<sup>2</sup> in the two cases above. If the fluffy particles (one group of 58 and 2 individual) are taken to be the only extraterrestrial ones, the two flux values become 0.1 and 2 particles/mm<sup>2</sup>. Evidently, the flux on our Luster collector during the Leonid meteor shower is at most half the Venus Flytrap value but may be as much as one or two orders of magnitude less.

In size, the particles range from about 0.05 to 0.3 micron in the group of 58 fluffy particles and from 0.2 to 0.5 micron in the group of 5 particles on screen P-15. For the individual particles, the sizes are 1.5, 0.9, 0.8 (3 particles), 0.5, and 0.3. Data are not available for one particle.

#### Summary of Results

One group of 58 particles and 2 individual particles went completely through the heat annealing tests; 2 individual ones are still being tested. Of those for which the tests were completed, one individual showed new diffraction spots after 600°C but also showed morphologic changes, the second showed morphologic changes and no diffraction after 1,000°C, and the group of particles showed neither morphologic changes nor diffraction after 800°C. The possibility cannot be eliminated that all are contaminants and it is concluded that more particles must be studied together with composition and other data before a conclusive answer can be given as to whether radiation damage distinguishes extraterrestrial from terrestrial particles. However, the heat-produced morphologic changes may be important in making this distinction, so that heating could prove to be a valuable diagnostic technique for this as well as for radiation damage annealing.

No chemical composition data from the electron probe have yet been obtained from the particles except for the detection of Na and Fe in some of the contaminants. Success was achieved in the development of techniques for locating submicron particles in the probe.

Flux values for the Luster flight, based on 8 individual particles and 2 groups of particles, are 0.3 and 2 particles/ $mm^2$ , the first value applying if each group is counted as a single particle, the second if every particle in the group is counted. These values do not include corrections for one-shadowed contaminants or for scanning efficiency. Sizes are almost entirely in the range 0.1 to 1 micron, the particles in the groups being less than 0.5 micron, the individual particles being mostly greater than 0.5 micron.

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#### RECOMMENDED FUTURE STUDIES

Recommendations for future work are as follows:

- 1. Find and test more particles. Make greater use of the tilting stage in studying diffraction effects and particle morphology.
- 2. Continue and expand the composition studies with the electron probe.
- 3. Investigate heating inside the electron microscope with the heating stage or with the beam, if methods of calibration can be found.
- 4. Heat test submicron particles of olivine, enstatite, and other silicates and of meteoritic or similar iron to see whether or not they show morphologic changes at 1,000°C and below, and, if they do, what these changes are. Do the same with likely contaminant materials.
- 5. Irradiate olivine and enstatite particles to see whether electron diffraction can be caused to disappear and, if so, whether heat annealing can restore it.

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#### BIBLIOGRAPHY

N.H. Farlow and M.B. Blanchard. Examination of Extraterrestrial Particles Collected by the Luster Sounding-Rocket Experiment (abstract). Transactions of American Geophysical Union, Vol. 47, 1966, Pages 143-144.

N.N. Greenman. Tests for Radiation Damage as a Possible Criterion for Micrometeorites (abstract). Transactions of American Geophysical Union, Vol. 44, 1963, Page 877.

N.N. Greenman. Heat Annealing Experiments Performed upon Venus Flytrap Particles. Annals New York Academy Science, Vol. 119, Art. 1, 1964, Pages 298-317.

N.N. Greenman and C.B. Gilpin. Electron Diffraction Control Studies for Venus Flytrap Particles. Proceedings International Symposium on Meteor Orbits and Dust, 4th, Cambridge, 1965, Smithsonian Contributions to Astrophysics (in press).

C.L. Hemenway and R.K. Soberman. Studies of Micrometeorites Obtained from a Recoverable Sounding Rocket. The Astronomical Journal, Vol. 67, 1962, Pages 256-266.

J. Murray. On the Distribution of Volcanic Debris over the Floor of the Ocean. Proceedings Royal Society of Edinburgh, Vol. 9, 1876, Pages 247-261.

W. Primak. Review of the Gross Structural Effects of Energetic Atomic Particles on Vitreous and Crystalline Silica. Journal of Physics and Chemistry of Solids, Vol. 13, 1960, Page 279.

J.A. Ryan. Corpuscular Radiation Produced Crystalline Damage at the Lunar Surface. The Lunar Surface Layer. J.W. Salisbury and P.E. Glaser (editors), Academic Press, New York, 1964, Pages 265-312.

R.K. Soberman, C.L. Hemenway, T.G. Ryan, S.A. Chrest, J. Frissora, E.F. Fullam, J.J. Balsamo, J. Cole, D. Hallgren, P. Yedinak, A. Goodman, and G. Hoff. Micrometeorite Collection from a Recoverable Sounding Rocket. GRD Research Notes, No. 71, Air Force Cambridge Research Laboratories, Bedford, Massachusetts, 1961. PRECEDING PAGE BLANK NOT FILMED.











BEFORE HEATING

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 $600^{\circ}$ C







BEFORE HEATING









600°C





800°C



1,000°C Figure 7. (Continued)







400°C







Figure 10. (Continued)





Figure 14.



#### LIBRARY CARD ABSTRACT

One group of 58 particles, another of 5 particles, and 8 individual particles were found in a collection made with the NASA-Ames Research Center's 1965 Luster rocket. The large group and 2 individual particles were subjected to heat annealing tests to see if radiation damage is a criterion for micrometeorites. Only one showed evidence of radiation damage annealing but it also showed morphologic changes. More particles must be studied to establish or reject the radiation damage criterion, but the heat-produced morphologic changes observed may also prove important in distinguishing micrometeorites from contaminants. The flux observed, uncorrected for contaminants or scanning efficiency, is 0.3 particle/mm<sup>2</sup> of collector surface if the groups are counted as single particles, or 2 particles/mm<sup>2</sup> if every particle in the group is counted. Sizes are almost entirely in the range 0.1 to 1 micron.