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STUDY OF PARTICLES COLLECTED BY THE 1966 LUSTER ROCKET (LUSTER II)

FINAL REPORT

DOUGLAS MISSILE & SPACE SYSTEMS DIVISION

APRIL 1968

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FINAL REPORT

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PREPARED FOR THE NATIONAL AERONAUTICS
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ABSTRACT

The NASA-Ames Research Center's Luster II rocket for collecting cosmic dust was successfully flown on 22 October 1966 during the Orionid meteor shower and collected samples for 204 sec between the altitudes of 77 and 149 km. The Douglas collection surface, one of the guest experiments on board, consisted of titanium, tungsten, and molybdenum electron microscope screens, each with a silicon monoxide substrate and a nickel or indium shadow coat. The study objectives were threefold: (1) to test, by heating both the flight particles and control particles of known materials, whether radiation damage and heat-produced morphologic changes could distinguish extraterrestrial from contaminant particles; (2) to obtain flux, composition, and other data about cosmic dust; and (3) to test the validity of the radiation damage criterion by irradiation and heating tests on terrestrial silicate particles. (Work on this last objective was not accomplished because the irradiated samples have not yet been returned to Douglas.)

The conclusions reached are as follows:

1. A total of 91 one-shadowed particles was found (only one-shadowed particles are of possible extraterrestrial origin), but most were judged to be contaminants on the basis of morphologic changes with heating, absence of heavy elements in microprobe analyses, and greater numbers and larger sizes of particles than would be expected from reasonable flux and effective collection-time values.

2. No evidence of radiation damage annealing was found in any of 43 particles heat-tested. Since so many of the one-shadowed particles are contaminants, with the rest being suspect to varying degrees, it is concluded that more work is needed for final evaluation of the radiation damage criterion. However, the fact that none of the terrestrial particles showed evidence of radiation damage is in harmony with the idea that such damage is restricted to extraterrestrial particles.

3. Results of heating tests on control particles of known materials indicate that significant morphologic change at about 600°C or below or extensive destruction at temperatures of 1000°C or below mark a particle as a probable contaminant, if cosmic dust is assumed to be of meteorite composition. Particles that are stable with heat, on the other hand, may be either terrestrial or extraterrestrial in origin. Heat-produced morphologic changes, therefore, are a potentially valuable tool for identifying certain classes of contaminants.

4. Of five particles analyzed with the microprobe, two showed compositions consistent with extraterrestrial origin. One of these latter particles had iron, nickel, calcium, and possibly silicon. The other had iron, magnesium, and silicon with aluminum, phosphorous, and chlorine as possible minor constituents.

5. Silicate particles as small as 0.4 micron can be analyzed with the microprobe.

6. A formula, derived for the effective collection time, gives flux estimates for the Luster I and Venus Flytrap Flights which are one or two orders of magnitude lower than those previously published, and in better agreement with recent downward revisions in the estimated near-earth flux.

7. Calculations based on reasonable values of the flux and on the effective collection-time formula show that cosmic-dust particles can be collected in adequate numbers by rockets if sampling is begun at a low-enough altitude, particularly in the vicinity of the 63-km opening altitude of Luster I.

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Section 1
INTRODUCTION

Cosmic dust is of great scientific interest for the light it can shed on solar-system origins and processes; it is also of great engineering concern for the impact-and-erosion hazard it presents to men and equipment in space. The NASA-Ames Research Center's Luster rocket program, by returning samples to the laboratory for direct observation and measurement, can significantly advance our knowledge of these particles. However, a high "background" of terrestrial contaminants has been found in all Luster and other collections obtained to date, despite the use of the most advanced clean-room and handling techniques. The primary purpose of the Douglas studies, therefore, is to develop criteria for recognizing the extraterrestrial particles among the contaminants; the secondary purpose is to determine size, shape, abundance, composition, and other important characteristics of cosmic-dust particles in the vicinity of the earth.

This report presents the results of a six-month investigation of particles collected with Luster II, the second successful rocket in the Luster program, which was flown on 22 October 1966. The study was performed under Contract NASw-1635, executed 9 August 1967, with the National Aeronautics and Space Administration Headquarters. It is a continuation of studies, performed under Contract NASw-1401, of particles collected with Luster I, which was flown on 16 November 1965.

We wish to thank A. Phillips (of Douglas) for help with the heating-tilting stage experiments in the electron microscope and E. L. Miller (of Douglas) for help with the electron microprobe analysis.

Section 2
LUSTER II FLIGHT AND COLLECTOR DESCRIPTION

The Luster II rocket was successfully flown from the White Sands Proving Ground, New Mexico, on 22 October 1966 during the Orionid meteor shower. Identical in design to Luster I, it carried particle collectors mounted on arms that extended and retracted during flight, and the payload was parachuted to the ground for recovery. Collection began at an altitude of 77 km, continued to a peak altitude of 149 km, and ended when the payload had fallen to an altitude of 118 km, for a total collection time of 204 sec. The respective altitudes and collection time for Luster I were very similar--63, 144, and 116 km and 206 sec.

The Douglas collection surface (Figure 1), one of the guest experiments on board, was the same type as that flown on Luster I and details of its preparation and handling are given in the report of that flight (Greenman, et al., 1966). The substrates were of silicon monoxide, formed by depositing it on a parlodion film which was later dissolved with amyl acetate. The supporting screens, 3.05 mm in diameter, were of titanium, tungsten, and molybdenum. These were coated with nickel or indium and mounted on a Lucite base of 55 x 50 x 3 mm with strips of double-sided Scotch brand "410" 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 to shield the covered screens from impact during flight but to allow air currents carrying contaminants to reach them at other times, thereby providing one means of contaminant control.

Double-shadowing was another means of contaminant control. The samples, when returned to Douglas after the flight, were given a second nickel or indium coat in a Class-100, laminar-flow clean hood. (Preflight and postflight contaminants should show two or no shadows, respectively, whereas

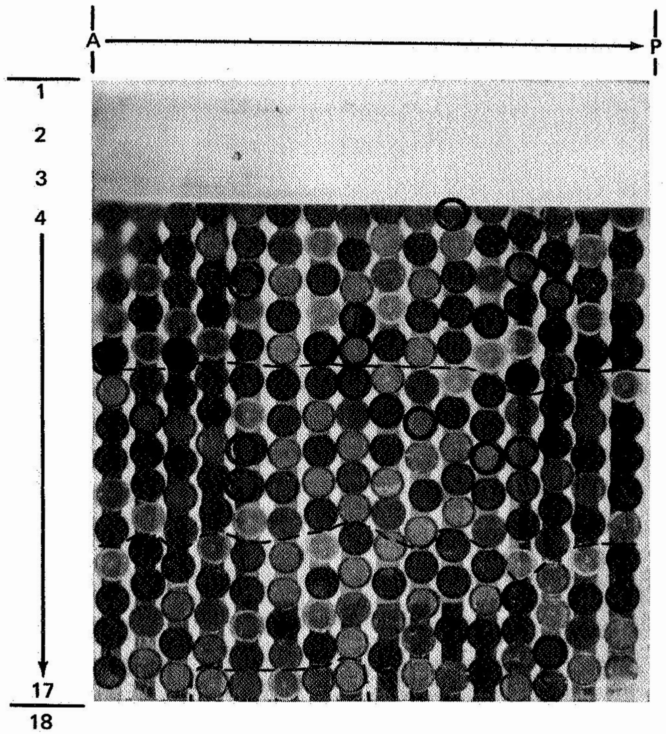


Figure 1. Collector Flown on Luster II

particles collected in flight as well as contaminants deposited during the time between coatings should show only one shadow.) A flight sample and spare sample were supplied to NASA-Ames Research Center. The spare sample went through the same preparation, handling, mounting, and other procedures as the flight sample, except that it was not flown. It therefore constituted a third means of contaminant control.

Section 3
STUDY PROCEDURES AND RESULTS

3.1 OBJECTIVES AND THEIR BASIS IN PREVIOUS WORK

The problem of recognizing particles of extraterrestrial origin in the Luster and other collections must be solved before reliable data from particle observations and measurements can be gathered. Significant contamination has been present in all collections made to date, and no criteria have yet been established that can distinguish unambiguously one type of particle from the other. Morphology, in particular, is not a reliable indicator of origin (Farlow, Blanchard, and Ferry, 1966). The prime objective of this study, therefore, was to investigate criteria that can be used to make this distinction. Emphasis was placed on testing two possible criteria--radiation-damage annealing and heat-produced morphologic changes.

The possibility that radiation damage may characterize extraterrestrial particles arose out of the early Venus Flytrap work (Soberman et al, 1961; Greenman, 1964). It is based on the idea that small extraterrestrial particles can suffer lattice damage from space radiation and that this damage can be annealed out at temperatures well below the melting point. In the electron microscope, these particles would show an electron-diffraction pattern only after being heated to moderate temperatures. Terrestrial contaminants, on the other hand, are either amorphous or crystalline. If amorphous, they should show no diffraction effects either before or after being heated; if crystalline, they should show diffraction patterns from the start. In past studies, one-shadowed particles have, indeed, generally been found to yield no diffraction patterns in the electron microscope. The Luster I study (Greenman et al, 1966) was devoted chiefly to testing this criterion; it was found that of eight single particles and two groups of particles, only one particle displayed a heat-produced diffraction pattern. However, this particle also showed changes in morphology, so the development of crystallinity could not be attributed definitely to radiation-damage annealing. Since all the particles found may have been contaminants, the search for evidence of radiation-damage annealing was continued in this study.

Another particle of the Luster I collection showed progressive destruction in the various stages of the heat treatment, but unlike the one just described, it showed no diffraction pattern. Still others, of the type termed "fluffy" by Soberman et al (1961) and considered by them to be of extraterrestrial origin, showed neither morphologic changes nor diffraction effects. This suggested that heat-produced morphologic changes could be useful in the determination of particle origin because silicates (the common constituents of meteorites and, hence, possibly of micrometeorites) were expected to be stable below 1000°C, whereas some types of contaminants, such as hydrates and perhaps organics, were expected to show distinct changes in this temperature range. The Luster I study was the first to include systematic heating experiments with cosmic dust so that little was known of what the effects might be. Also, it was not known whether stable materials, such as the silicates, would remain stable to as high a temperature in particles of micron and submicron size as in bulk. In this study, therefore, the heat-produced morphologic changes were investigated in particles of known composition, in the flight-collected particles, and in examples of contaminants on the flight screens (particles with no shadows or two shadows, for example). Materials selected for these experiments included (1) olivine and enstatite because they are the most common minerals of the stony meteorites, (2) canyon Diablo meteorite iron, (3) aluminum oxide grinding powder and (4) pollen grains and the powdery grains from moth wings to represent organics.

The secondary objectives of the study were, as before, to obtain information on the composition of the particles from electron-microprobe 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. The beam diameter of the electron microprobe is normally between 1/2 to 1 micron in diameter, so particles in the submicron-size range are near the probable limit of the usefulness of the instrument. This may well have affected the Luster I microprobe results. Therefore, in this study, particles of olivine and enstatite, deposited on substrates like those that were flown, were measured in the microprobe to obtain better control on the size limits at which reliable chemical measurements can be made.

A third objective was to test whether the process upon which the radiation-damage criterion is based can occur; that is, whether a submicron crystalline particle can be damaged by radiation to the extent that it does not yield an electron diffraction pattern, and, if it can, whether the damage can be annealed out by heating. Olivine and enstatite particles of submicron and micron size show electron-diffraction patterns (Greenman and Gilpin, 1967). Powdered samples of these minerals were sent to the NASA Plum Brook Reactor Facility to be irradiated by integrated fluxes of fast neutrons (greater than 1 MeV) of about 5×10^{19}, 2.5×10^{20}, and 5×10^{20} nvt. The irradiated particles were to be examined for changes in the diffraction patterns, and those in which the patterns had disappeared were to undergo heat-annealing tests. However, the samples have not yet been returned to Douglas, so this phase of the study is not included in this report.

3.2 HEATING EXPERIMENTS

3.2.1 Experimental Procedures

The procedures described in the Luster I study report were also followed in this study. The one-shadowed particles were mapped, and the screens were heated in an argon atmosphere with a three-zone, resistance-heated tube furnace. Tests began at a temperature of 400°C, and the temperature was increased in intervals of 200°C to a maximum of 1000°C. The particles were heated for one hour at each temperature. The 200°C stage of heating was omitted because no effects were observed at this temperature in the Luster I study.

Heating tests were also conducted in the electron microscope by means of a heating-tilting stage. This device allows the specimen to be tilted about a horizontal axis and rotated about a vertical axis, allowing it to be heated to almost 800°C. It also incorporates a liquid-nitrogen-cooled cold plate near the specimen to reduce contamination. With this device, the particles can be observed during the heating, although normally this is done only intermittently and the electron beam is turned down between times to prevent the buildup of contamination. Because particles do not have to be removed from the microscope, heating also can proceed directly from one temperature stage to the next without an intervening cool-down period, allowing the tests to be completed in much less time than with external heating.

There are several disadvantages of the device. First, oxidation reactions are somewhat more likely to occur in the normal microscope vacuum of the order of 10^{-5} mm Hg than when heating is done in an atmosphere of high-purity argon. Second, the operating temperature range does not extend to 1000°C. A third problem was discovered during the tests; the screens tended to stick to the molybdenum holder, probably because diffusion between the metals occurred at these temperatures. This was not as serious with tungsten screens as with those of titanium, which not only stuck to the holder but became brittle enough to break apart. Isolating the screen with thin mica spacers was found to prevent the sticking to the holder but was not completely satisfactory because the mica also tended to stick to the screen. Platinum spacers will probably provide a satisfactory solution to the problem in future work.

The internal heating tests were carried out at temperatures of 400°C, 600°C, and 750°C, and the particles were kept at each temperature for about one hour.

3.2.2 Flight-Particle Results

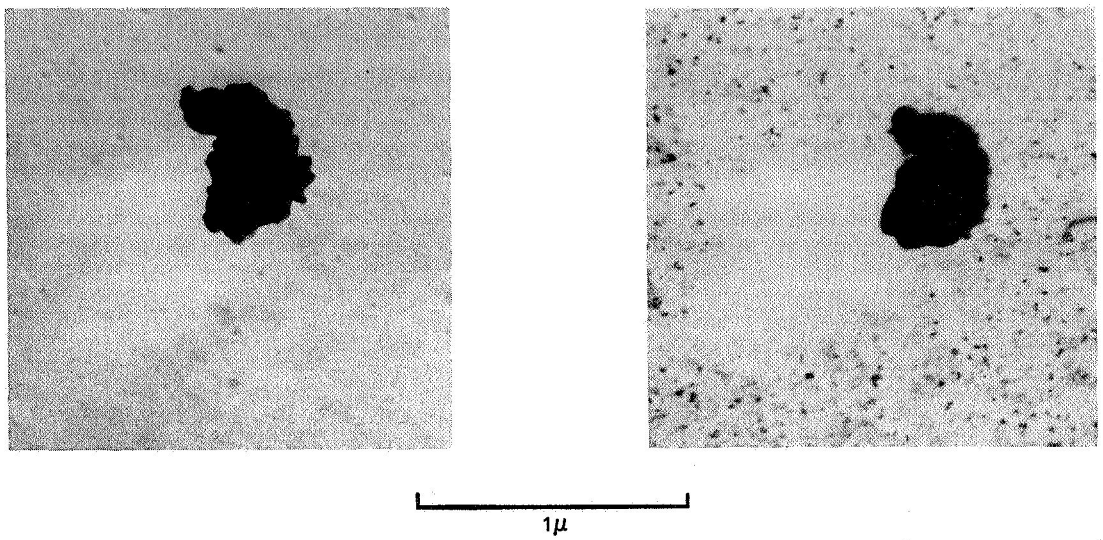
A higher contamination level was found on the Luster II samples than on those of Luster I, but it was comparable to the levels reported by other Luster guest experimenters. Many of the one-shadowed contaminant particles could be recognized by their flatness and angular shape as fragments of the silicon monoxide substrate film and were eliminated from the count. A total of 91 one-shadowed particles remained; these were found on 22 of the 23 screens examined. On the basis of the heating and microprobe results and of the size distribution, many of these, too, may be film fragments and other forms of contaminants as well. The screens examined and the coordinate system by which they are designated are shown in Figure 1.

About half the samples (11 screens and 43 particles) were heat-tested. No heat-produced diffraction patterns that could be attributed to radiation damage annealing were found in any of the tests. The only diffraction pattern changes observed were, as in the Luster I study, the sharpening of the nickel and indium rings and, in the case of nickel, the production of spotty ring patterns as the metals nucleated and became coarsely crystalline at elevated temperatures. Also, in no case were patterns observed to appear when the particle orientation was changed by means of the tilting stage. However, various types of morphologic change with heating were observed, although not all particles displayed such changes.

Figures 2 to 5 show some of the particles heated external to the electron microscope in an argon atmosphere. All these screens had nickel shadow coats. The particle in Figure 2, from screen C-8, shows a series of angular protuberances which has disappeared in the heating to 400°C; the entire particle outline has, in fact, become more rounded as if melted back. Olivine, enstatite, and meteorite iron, as will be shown in a later section, do not show such changes at this low a temperature so that this particle is probably a contaminant.

The particle shown in Figure 3, also from screen C-8, after being heated to 400°C has lost most of the lighter-toned periphery and also appears to have developed some structure in the center. As in the case of the preceding particle, these changes at this low a temperature indicate that this particle, too, is probably a contaminant.

In Figure 4, the particle from screen H-9 rests in the center of a dark circular area that indicates a reaction has taken place between particle and substrate. Heating to 400°C has caused extensive destruction to the particle and, in addition, has produced a second reaction ring within the original



a) UNHEATED

b) 400°C

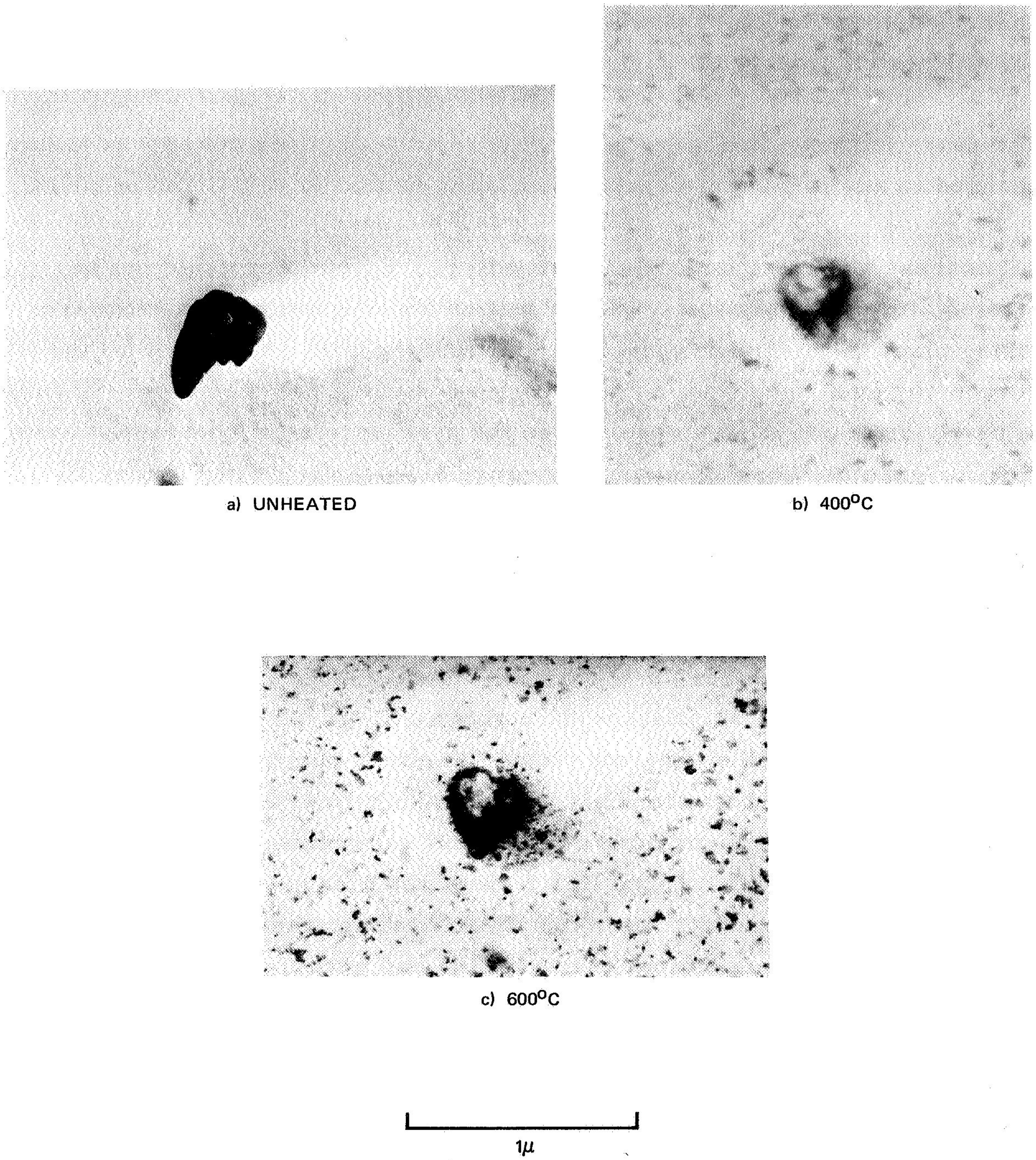
Figure 2. Particle on Screen C-8

a) UNHEATED

b) 400°C

1μ

Figure 3. Particle on Screen C-8



a) UNHEATED

b) 400°C

c) 600°C

Figure 4. Particle on Screen H-9

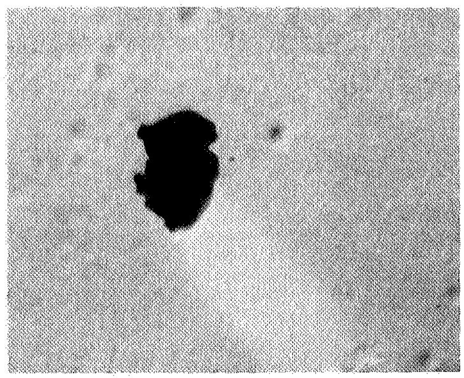
area. Heating to 600°C has not resulted in any significant further change. These extreme changes at relatively low temperature mark this particle as a likely contaminant also. It is particularly interesting that if the particles described above had not been heated, they may have been considered of possible extraterrestrial origin.

The particle shown in Figure 5, from screen N-16, does not appear to have changed to any important degree after being heated to 400°C and to 600°C.

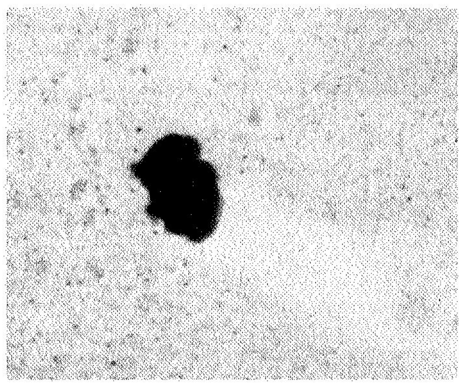
All of these particles were also heated to 800°C, but they could not be located afterward. Possibly the nucleation and crystallization processes taking place in the nickel caused the particles to shift about, fall off the screen, or even, perhaps, to be consumed in chemical reactions. Shifting of particles at 800°C was observed in the heating tests of control minerals (see for example the olivine and meteorite iron particles in Figures 10 and 12), but little or no particle loss occurred. This disappearance of the particles may be another indication that they are contaminants, at least in the case of those types that undergo changes in morphology at 400°C.

The results of the internal-heating tests, performed with the heating-tilting stage in the electron microscope, are shown in Figures 6 to 9. All screens in these tests had indium shadow coats. Figure 6 shows a particle from screen E-6 in which both transient and permanent morphologic changes were observed. A bulbous outgrowth formed after about 1 min of heating at 600°C; after an hour most of this feature had disappeared but the particle was otherwise unchanged. After the 750°C heating, the outgrowth was completely gone, and modification of the outline had occurred, indicating removal of additional material.

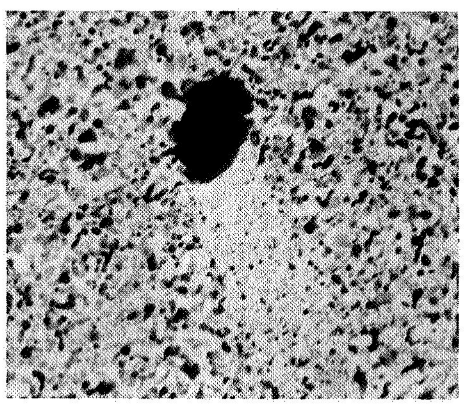
Two small particles from screen M-14 are shown in Figure 7. Both show distinct changes at 400°C but they are different for each particle. One appears to have spread to a spherical shape and displays a reaction rim. The other has maintained its general shape but has widened and has also grown a small protrusion.



a) UNHEATED



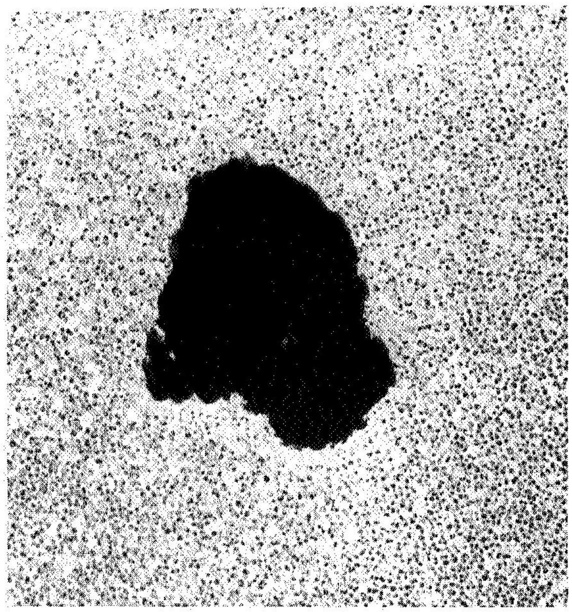
b) 400^{o}C



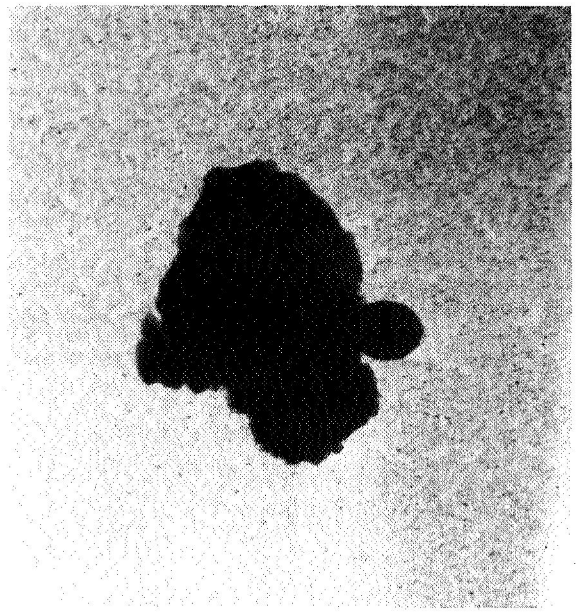
c) 600^{o}C

1μ

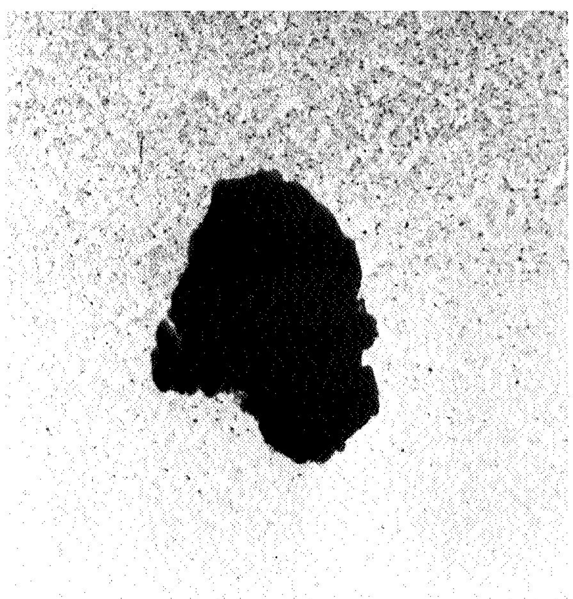
Figure 5. Particle on Screen N-16



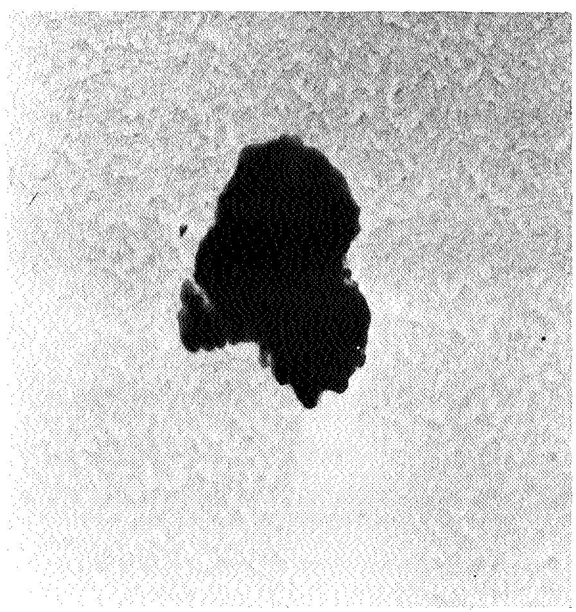
a) UNHEATED



b) 1 MINUTE AT 600^{o}C



c) 1 HOUR AT 600^{o}C



d) 750^{o}C

1μ

Figure 6. Particle on Screen E-6

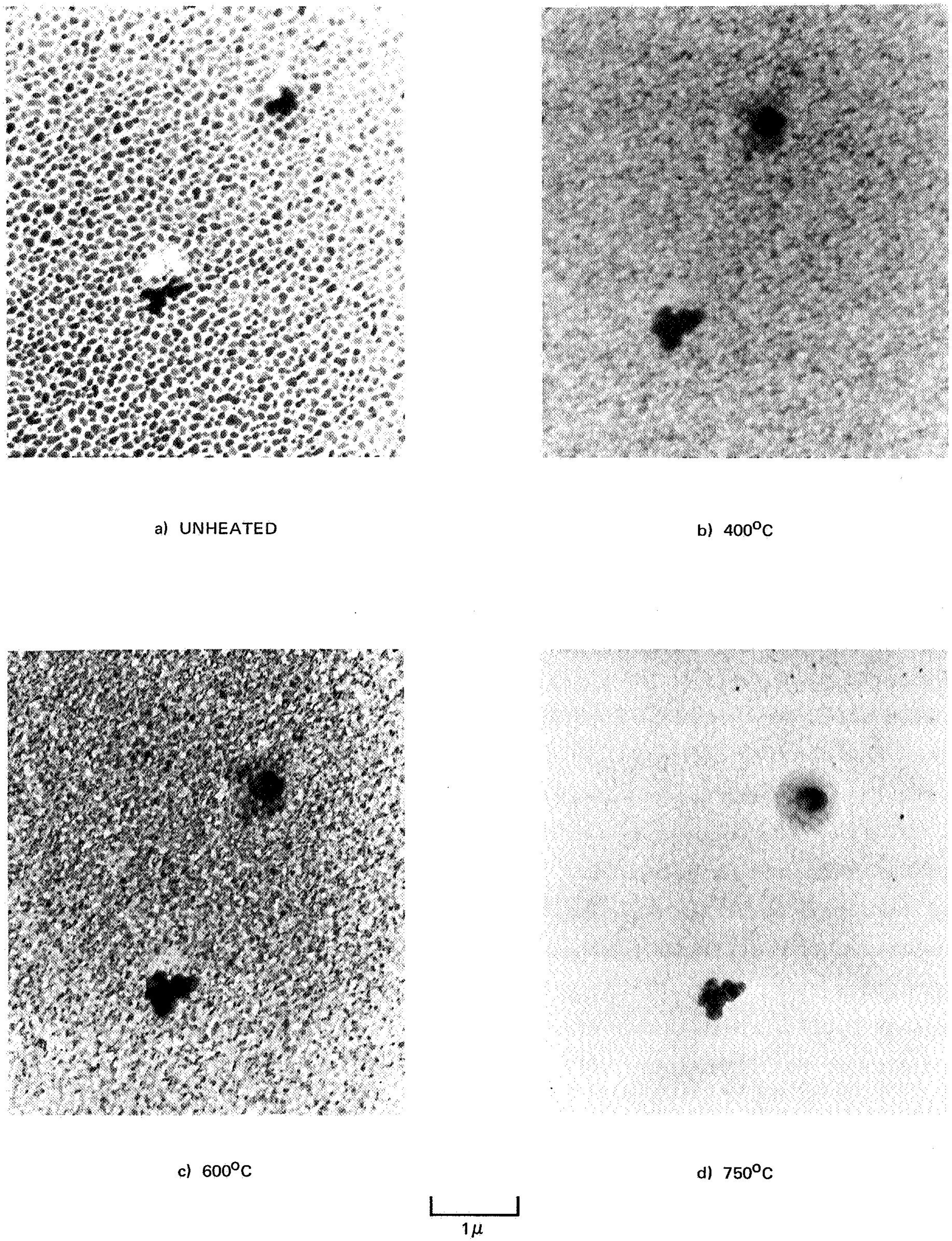


Figure 7. Particles on Screen M-14

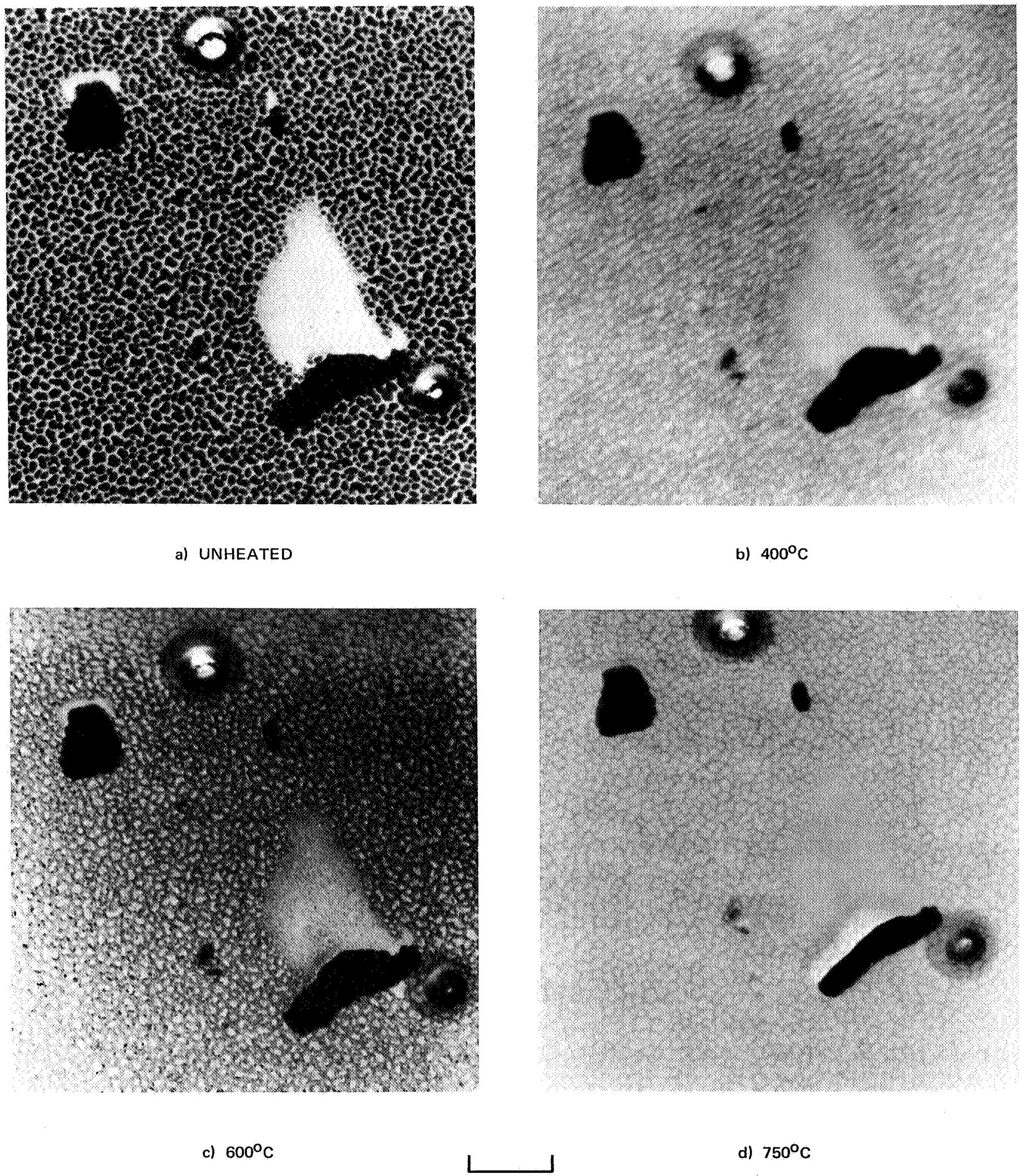
A group of three particles, also from screen M-14, is shown in Figure 8. Only the large, elongate particle seems to have undergone any change, but it is possible that the particle has merely rotated upward from its former position rather than that it has lost material in the heating. No elements heavier than silicon could be detected with the microprobe in this particle, despite its large size, and this, together with its angular shape and heat stability, points to its being a fragment of silicon monoxide film. The other two are likewise probably film fragments because of their angular shape, flatness, and heat stability.

The particle shown in Figure 9, from screen B-10, shows no essential change after the 400°C and 600°C heating stages; only slight modification is evident after the 750°C heating stage.

3.2.3 Control-Particle Results

Some results of the heating tests on particles of known materials are shown in Figures 10 to 19. The olivine particles shown in Figure 10 have remained unchanged through the 800°C heating stage. (At this stage, however, some additional particles can be observed in the photograph, probably having been shifted about as a result of the nickel mobility and of some substrate breakage. The sharply defined light and dark areas of the photograph are caused by the breaking and folding over of the substrate). At 1000°C, rounding and modification of outline appears to have occurred, although the particles seem to have remained largely intact. Particle size has not been greatly reduced, and the small particles have not disappeared. In the photograph of the unheated particles, many, especially the larger ones, can be seen to be aggregates rather than single particles, so that the apparent outline modifications at 1000°C are to some extent the result of shifting of the smaller particles of the aggregate. The rounding, however, indicates that some sintering of the particles may have accrued.

Figure 11 shows the enstatite particles; they behaved much like the olivine except that corresponding effects occurred at lower temperatures. At 600°C only minor changes are evident and these may be due to shifting of particles



a) UNHEATED

b) 400°C

c) 600°C

d) 750°C

1μ

Figure 8. Particles on Screen M-14

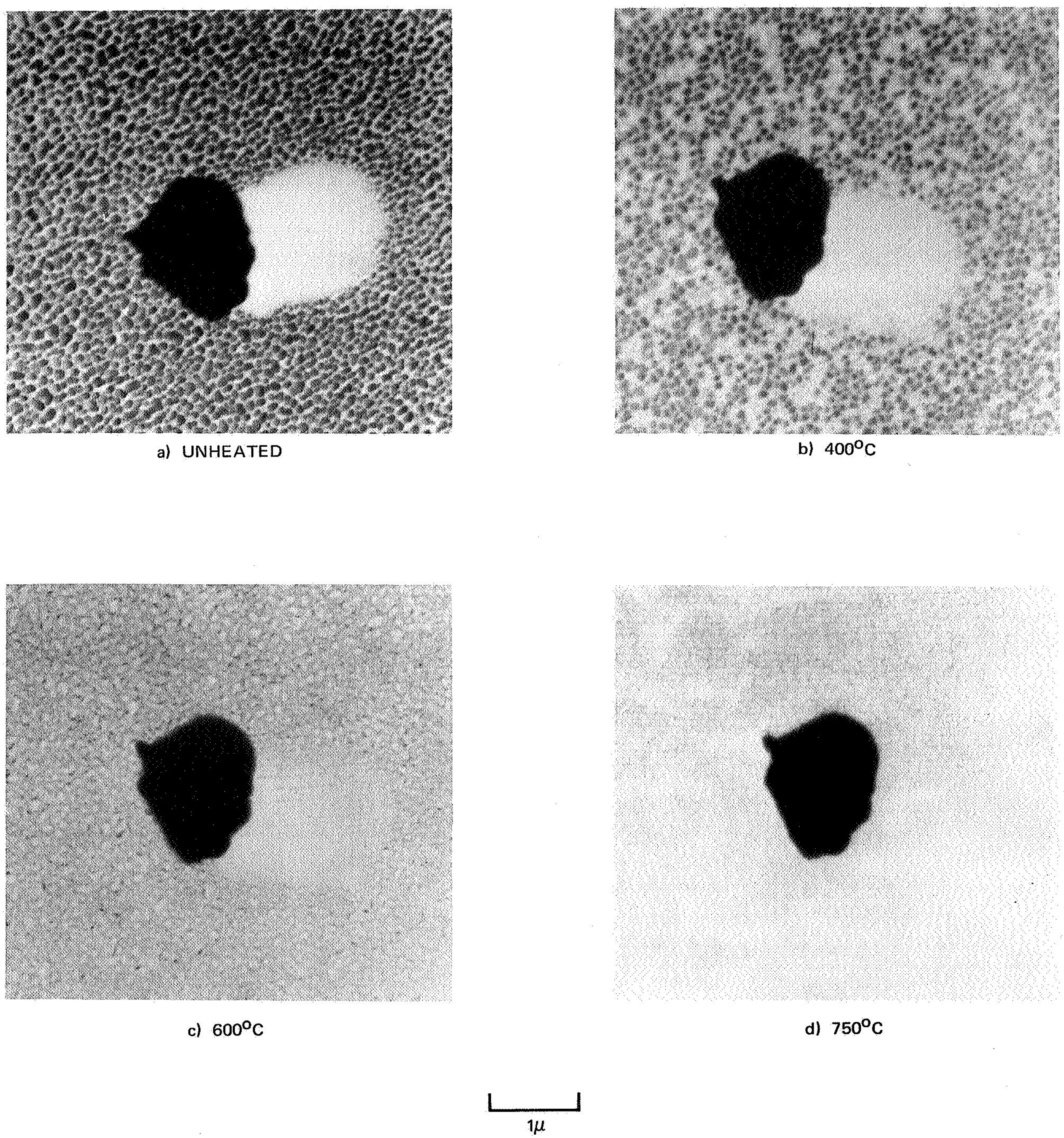
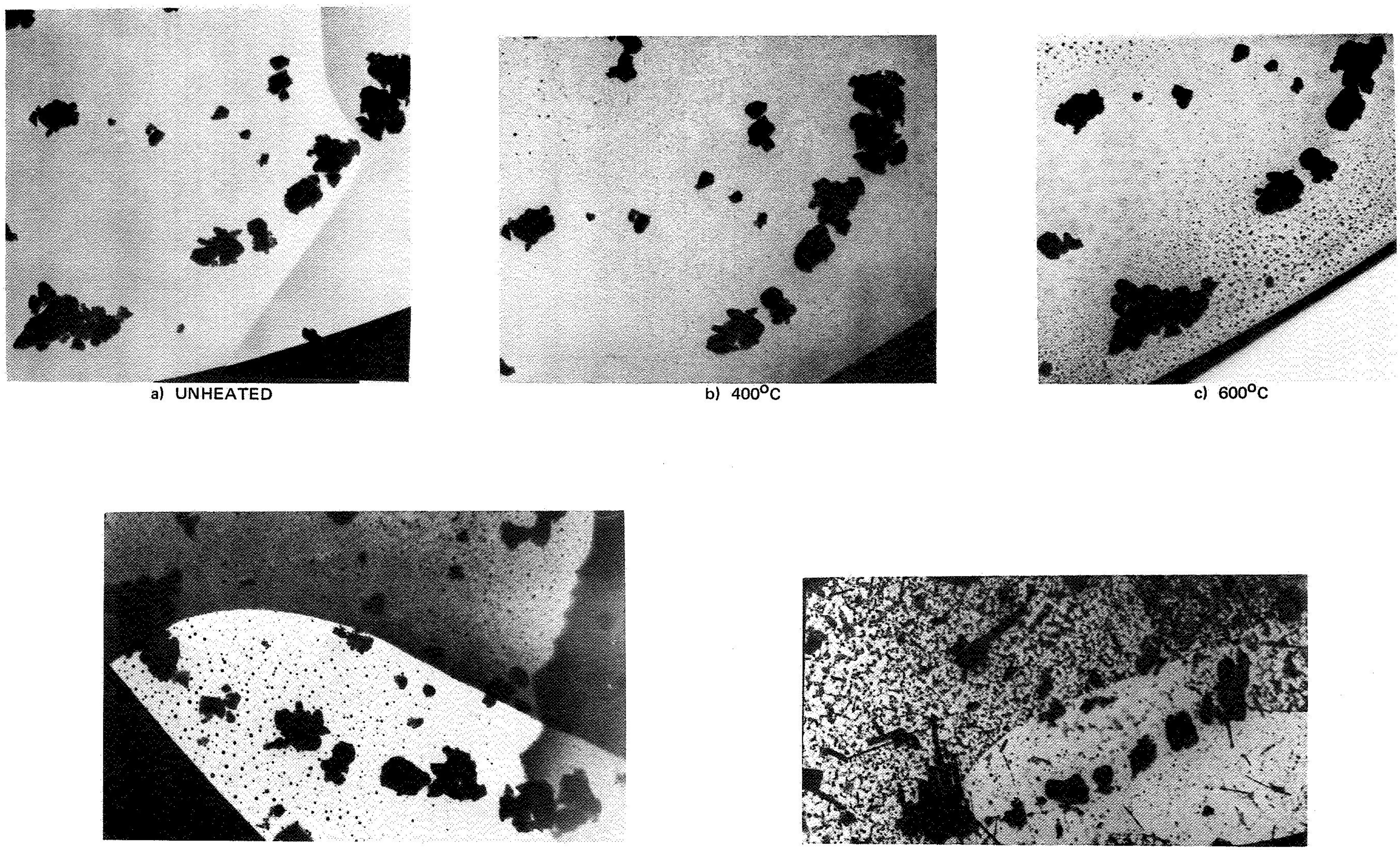


Figure 9. Particle on Screen B-10



a) UNHEATED

b) 400°C

c) 600°C

d) 800°C

e) 1,000°C

1μ

Figure 10. Olivine Particles

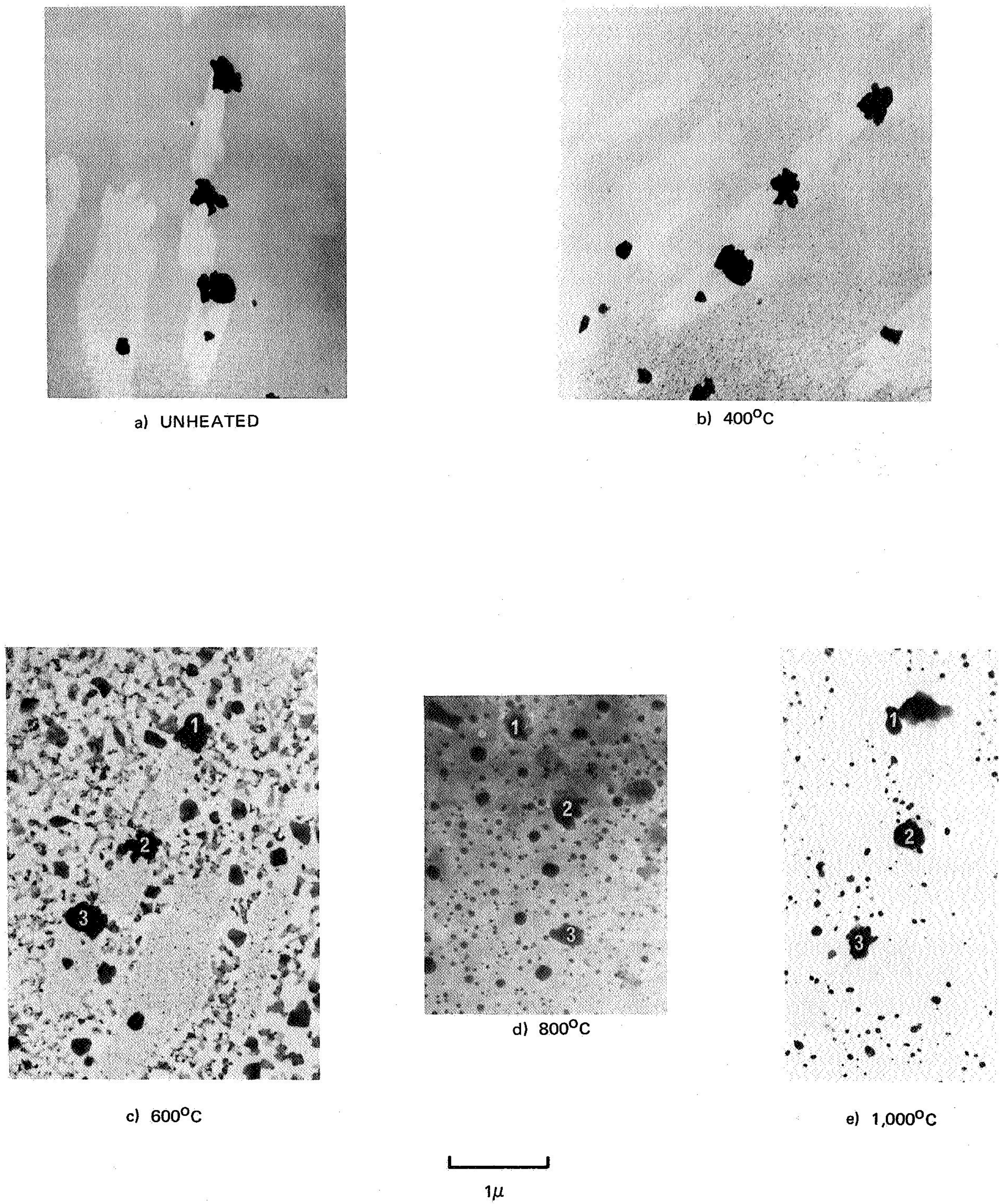


Figure 11. Enstatite Particles

making up the aggregate. At 800°C definite rounding and some size reduction have occurred, as well as some movement of the particles relative to one another. It is not certain whether or not the smaller particles may have survived because they cannot be distinguished in the photograph from the islands of nickel. At 1000°C the particles show little significant change from their appearance at 800°C.

In Figure 12, the two particles with single shadows, one about 0.8 micron and the other 0.2 micron, are Canyon Diablo meteorite iron. The larger of the two has come through the heating to 1000°C essentially unchanged. The smaller shows some rounding of outline at 800°C; it also appears to have survived the 1000°C heating. However, at this stage, because the size of the particle is comparable to that of the nickel nuclei, it is not certain whether the seeming change in outline and size is real or results merely from the formation of nickel nuclei adjacent to the particle. This series of photographs also shows some drastic changes in the unshadowed particles (which may not be iron) and examples of the appearance of new particles, some of which may be the result of crystal growth instead of or in addition to existing particles shifted about.

Figure 13 is an example of a meteorite-iron particle of about the same size as the larger one of Figure 12; it shows some rounding of outline at 800°C but is otherwise largely intact.

Particles of aluminum oxide polishing powder are shown in Figure 14. They show no changes at 800°C; at 1000°C, the largest particle (the only one discernible against the nucleated nickel background) shows some rounding and modification of outline but does not seem to be greatly affected otherwise. In this respect, it is similar to the olivine.

Some examples of the behavior of pollen grains with heating are shown in Figures 15 and 16. At 400°C, crystal-like growths have exuded from the grains. At 600°C, the opacity of one grain to the electron beam has been markedly reduced, revealing the porous and spiny structural features; at 800°C, the shape of the other grain has changed drastically. The extreme

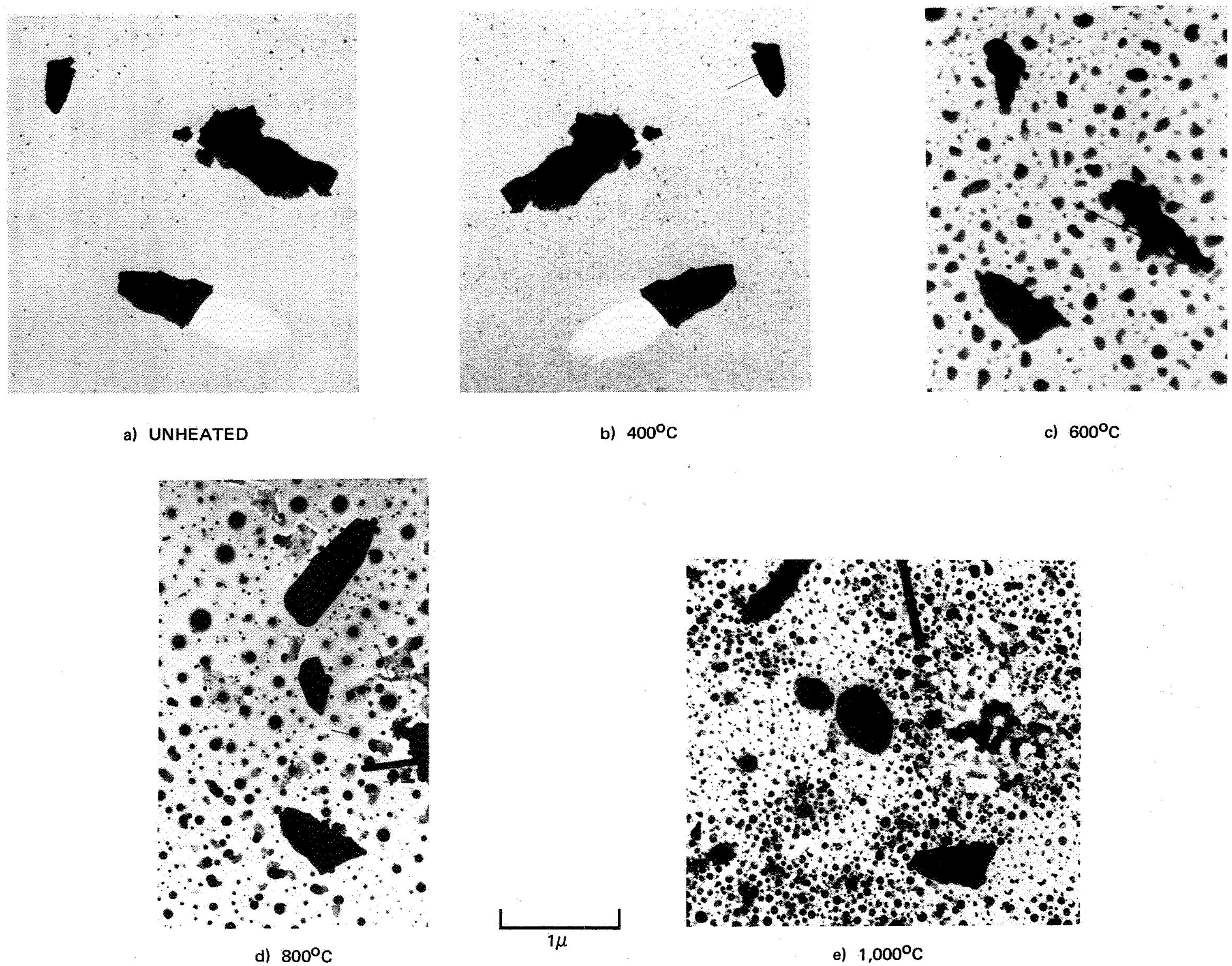


Figure 12. Canyon Diablo Meteorite Iron Particles

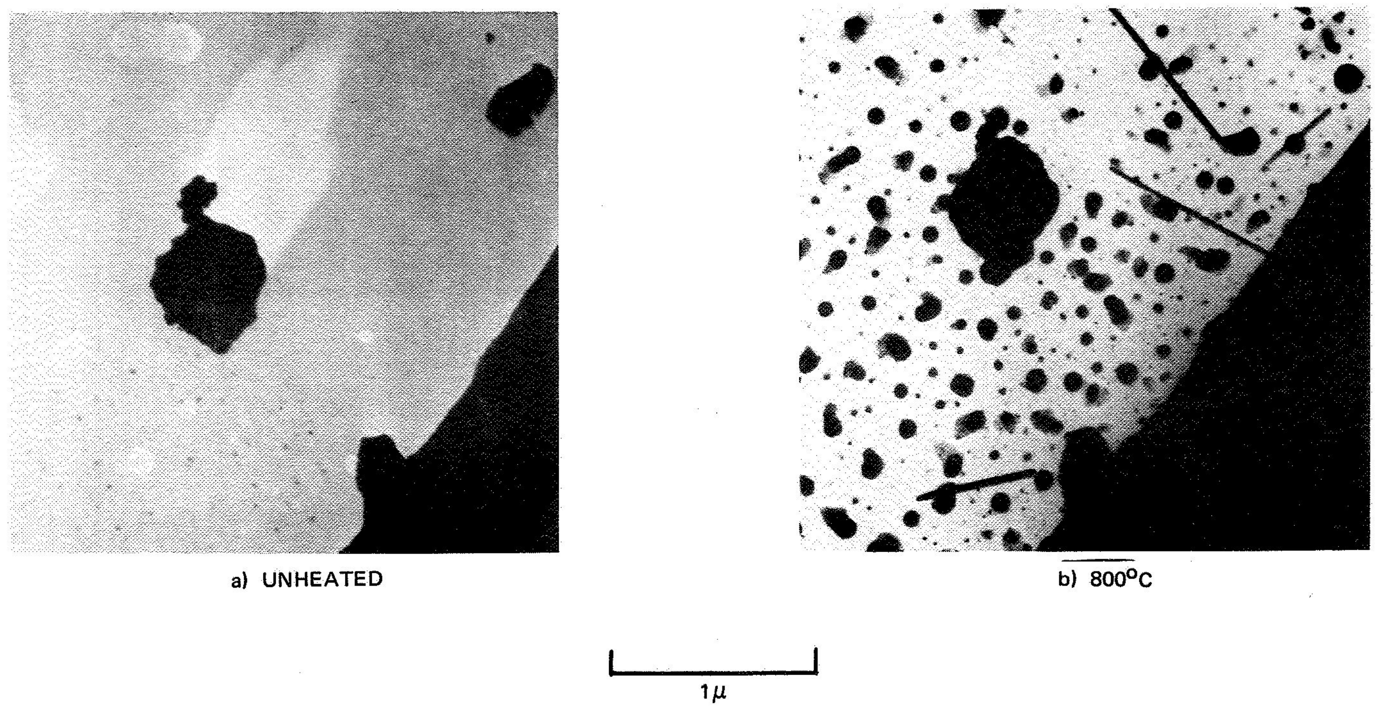


Figure 13. Canyon Diablo Meteorite Iron Particle

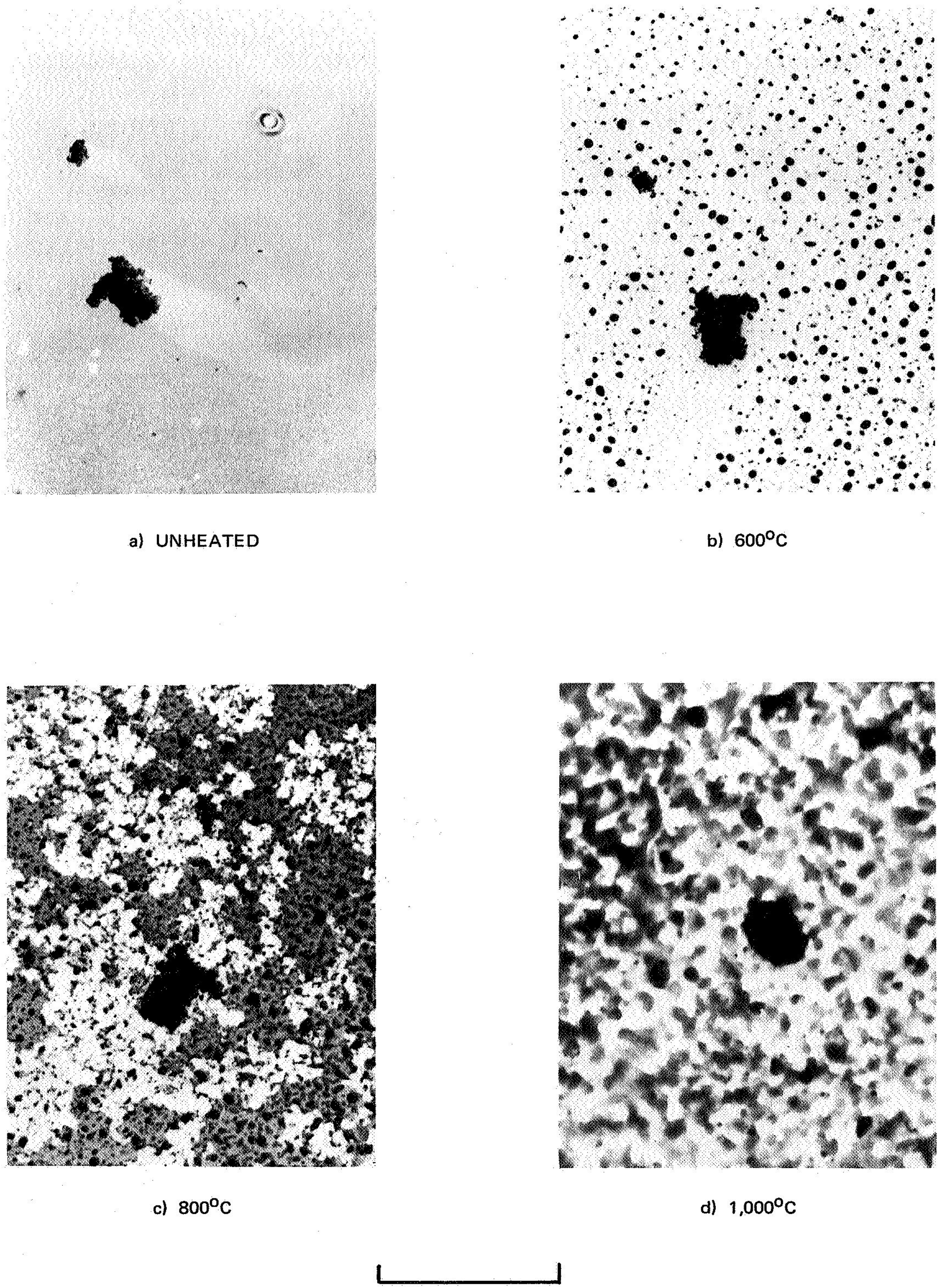


Figure 14. Particles of Aluminum Oxide Polishing Powder

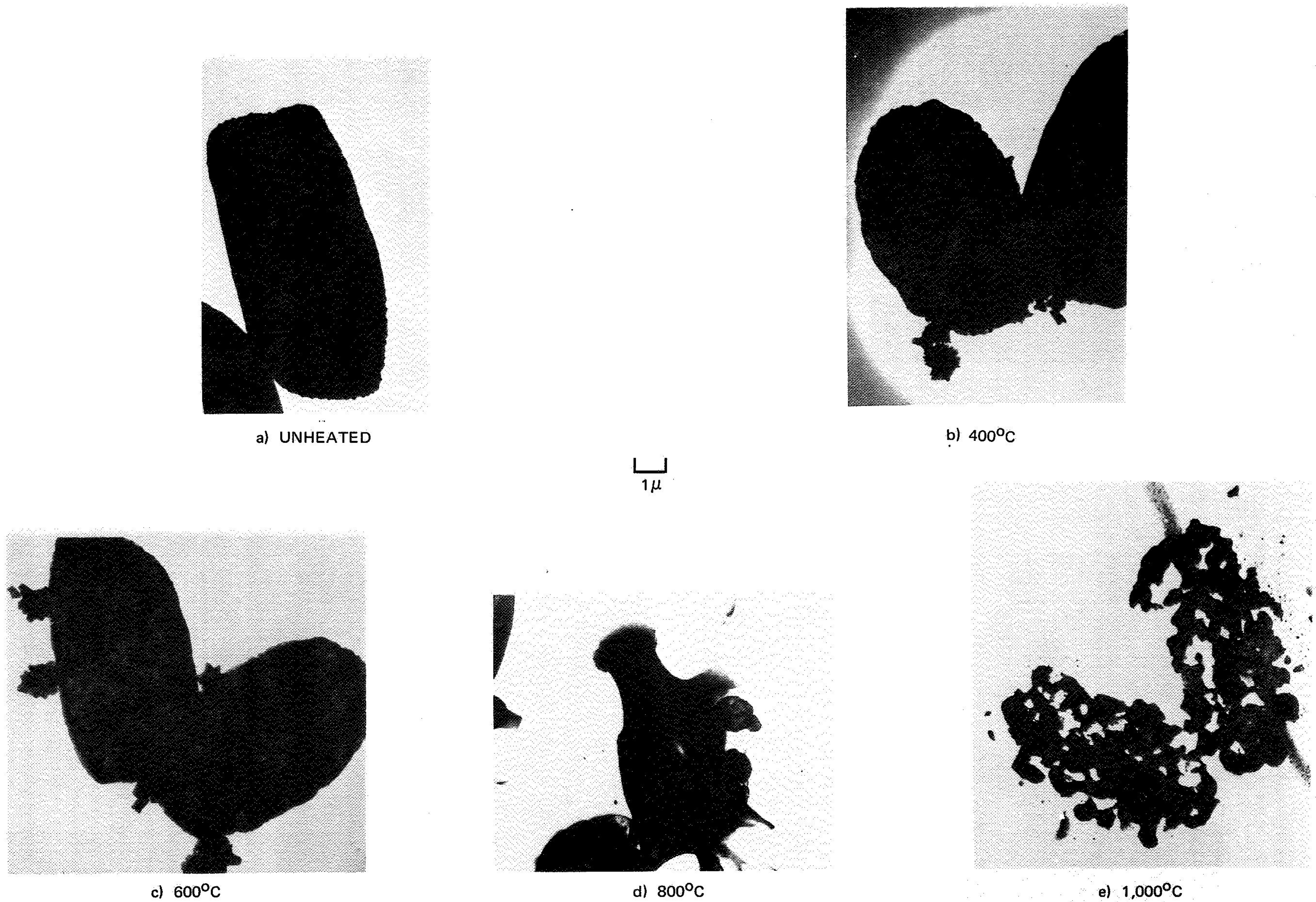


Figure 15. Pollen Grains

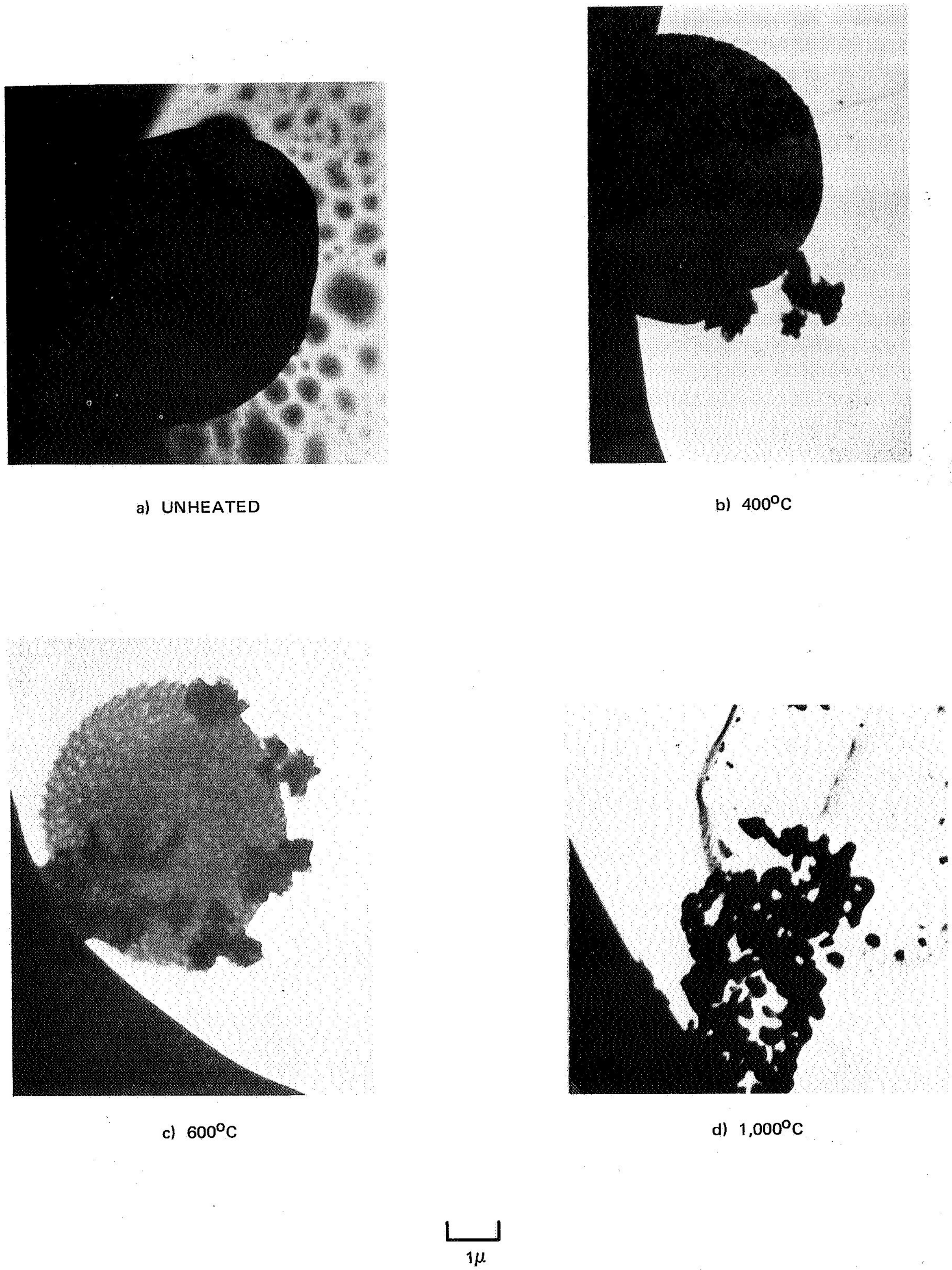


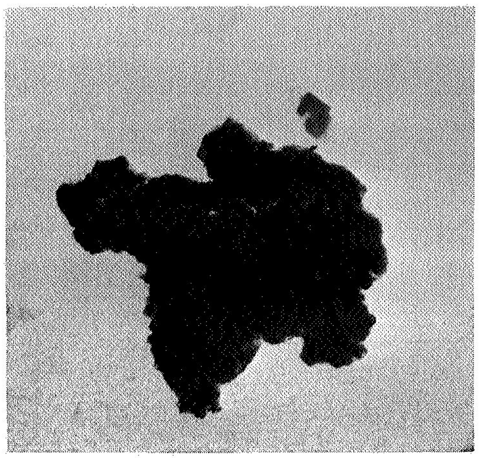
Figure 16. Pollen Grains

destruction resembling charring at 1000°C was observed in all pollen grains tested. Not all grains, however, showed the crystal-like exudations; some displayed only progressive decrease in opacity to the electron beam, or changes in shape, or both.

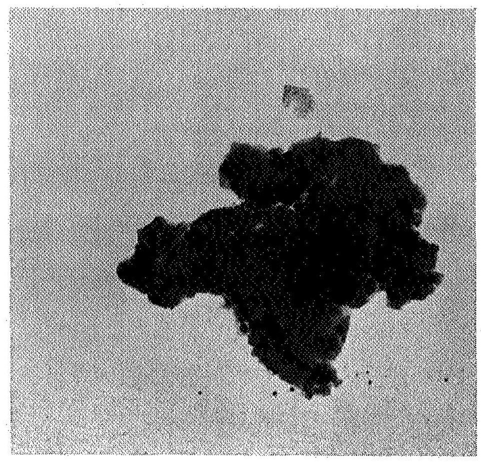
Fine powdery dust from the wings of moths was tested as another representative of organic materials. The particle shown in Figure 17 shows essentially no change up to 800°C, with the possible exception of some slight decrease in opacity and some variation in the shapes of the darker structures within the particle. The particle of Figure 18 differs in that it has a distinct biogenic structure; it shows virtually no change up to 600°C but at 800°C has acquired a "charred" appearance.

The particle shown in Figure 19 on screen J-10 is a two-shadowed contaminant heated in the electron microscope with the heating-tilting stage. Destruction has begun at the center of the particle at 400°C and has continued, until at 750°C most of the particle has disappeared.

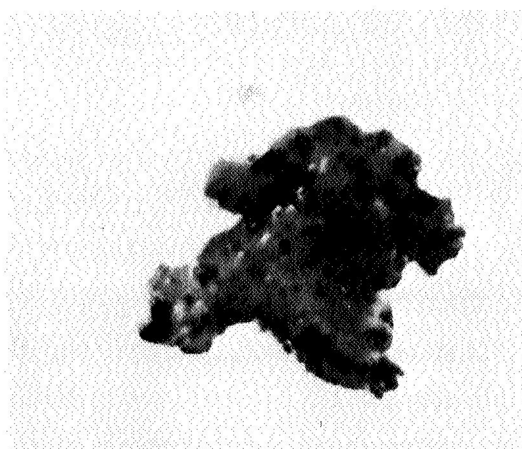
The heating experiments on the control particles show that under the test conditions (one-hour duration of each heating test, inert gas atmosphere, and about one-atmosphere pressure), submicron and micron particles of iron and the silicates olivine and enstatite have excellent heat stability. Iron is essentially inert at 1000°C; olivine shows some changes at 1000°C and enstatite at 800°C. The changes are mainly a rounding of the particle outline resulting from a moderate amount of sintering. However, with both silicates the bulk of the particle survives the 1000°C heating. These results make it highly probable that other high-melting-point silicates would show similar heat stability. If it is assumed, therefore, that extraterrestrial particles are likely to be composed of iron and silicates, heating tests can allow one to reject as a contaminant any particle that changes drastically at any temperature up to 1000°C or that shows significant change at temperatures of about 600°C and below. This criterion has been applied to some of the flight particles already described.



a) UNHEATED

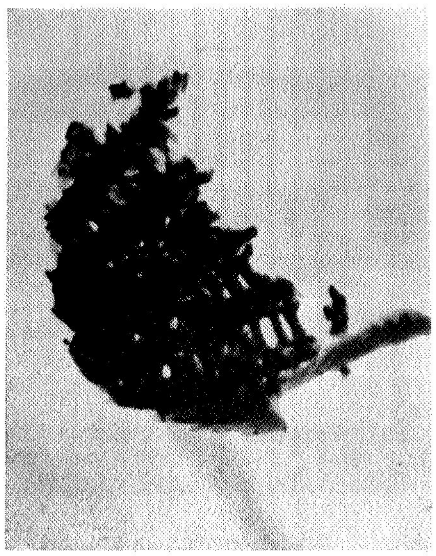


b) 600°C

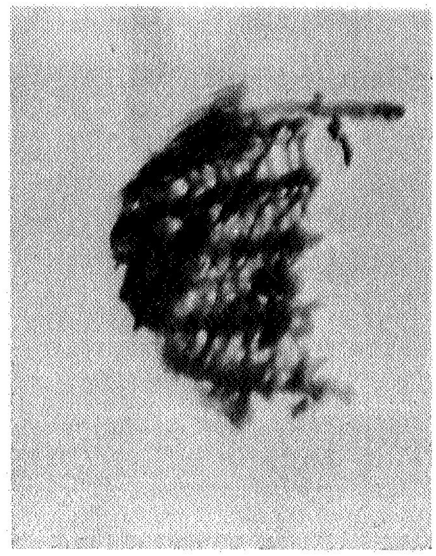


c) 800°C

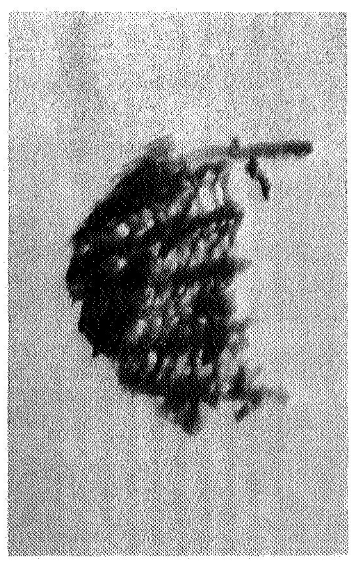
Figure 17. Dust from Moth Wings



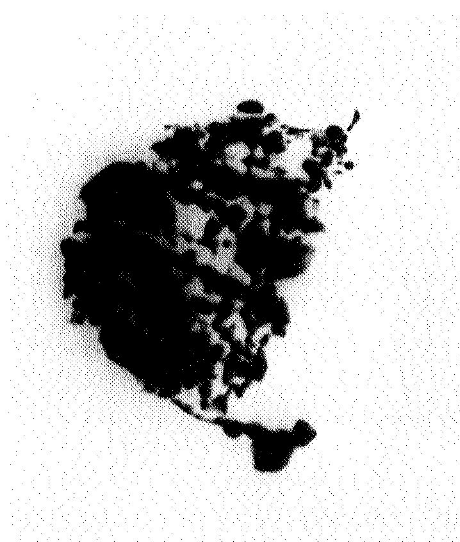
a) UNHEATED



b) 400°C



c) 600°C



d) 800°C

1μ

Figure 18. Dust from Moth Wings

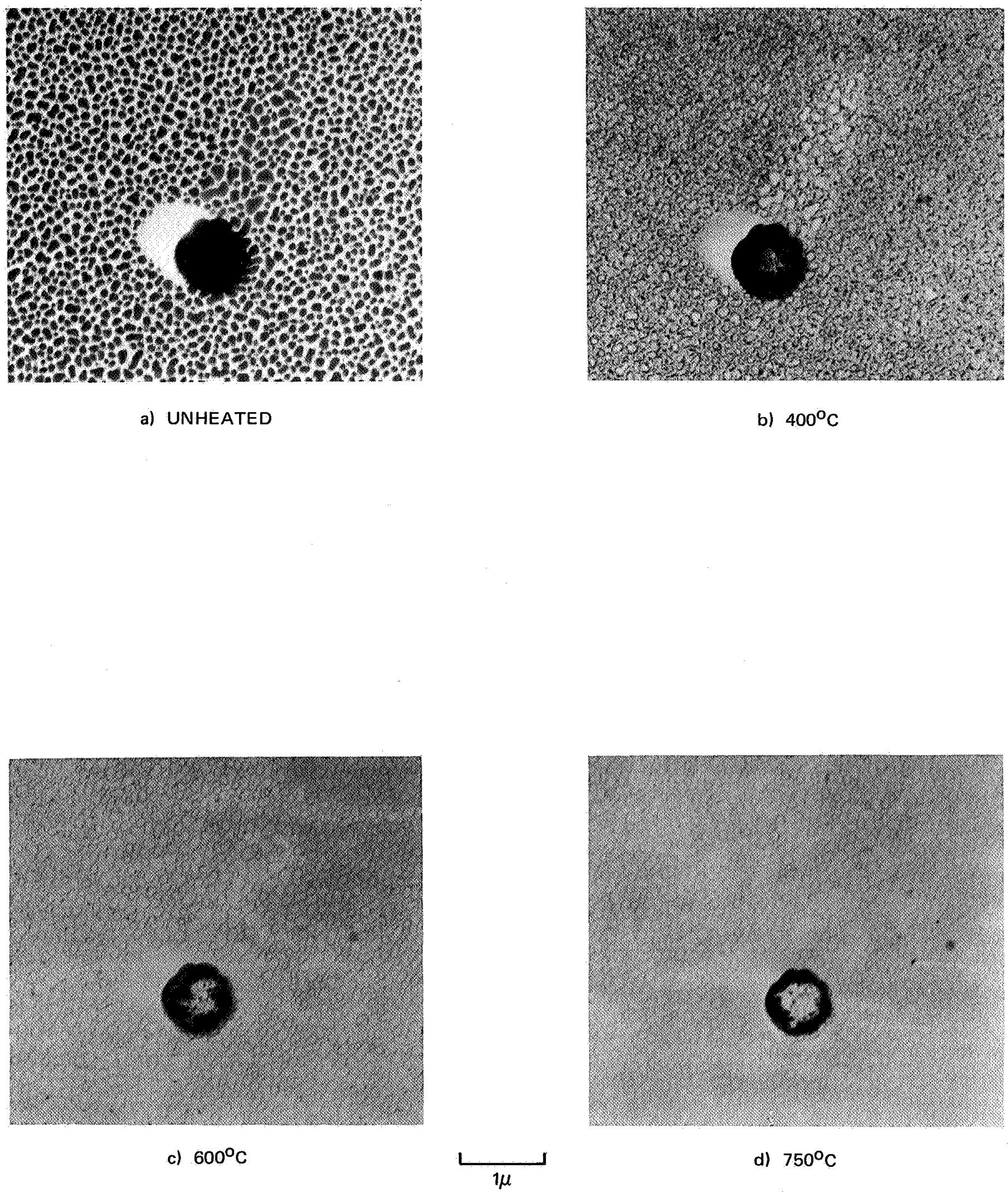


Figure 19. Two-Shadowed Particle on Screen J-10

Although heat stability may be considered a necessary condition for extraterrestrial origin, it is not a sufficient one. For example, aluminum oxide particles, one of the particles of moth-wing dust, and, of course, the silicon monoxide film fragments have excellent heat stability. However, with further experimentation, the heating technique may well prove capable of providing finer distinctions than are possible on the basis of the tests made to date. Heating in vacuum, as in the electron microscope, or even with oxygen and other gases to induce test reactions, may provide means of recognizing contaminants with greater assurance and greater specificity.

3.3 ELECTRON MICROPROBE ANALYSIS

3.3.1 Experimental Procedures

The electron microprobe analysis was carried out, as in the Luster I study, with an Applied Research Laboratories Model EMX microprobe. The particles were located by means of the same technique used in that study: a sequence of electron microscope pictures of successively lower magnification was taken to show the particles of interest and their location with respect to nearby recognizable features, such as distinctive screen imperfections or large particles that could be easily observed with back-scattered electron or nondispersive X-ray area scans. The screen was then placed in a standard holder for the Hitachi HU-11A electron microscope, and this assembly was mounted over a beryllium disk to minimize the background from electrons that penetrated the substrate.

The desired screen area was located by comparing the area scans in the microprobe with the photographs. This served to reveal the larger particles. The smaller ones not visible in the area scans, generally those of less than two or three microns in size, were located by determining from the photographs their approximate positions relative to the reference features. These positions were scanned manually, and the presence of the particle was indicated by an increase in the signal from the nondispersive X-ray detector.

The beam was then allowed to remain at this point, and the X-ray spectrometers were used to analyze the characteristic X-rays produced. Materials composed mainly of elements lighter than magnesium, such as organic materials, cannot be detected in this manner because the energies of the characteristic X-rays of these light elements are below the threshold of the nondispersive detector. For these materials the light-element X-ray spectrometer can be used, but the sensitivity is not as high as with the nondispersive detector.

Several combinations of beam voltage and current were used to determine the optimum operating conditions. In general, higher accelerating voltages give smaller beam diameters, but they also increase the X-ray background levels.

3.3.2 Flight-Particle Analysis

Five particles were examined--three from screen P-12 and one each from screens H-8 and M-14. Screens P-12 and H-8 had not undergone any heating tests but screen M-14 had been heated to 750°C in the electron microscope. Most of the effort was devoted to the unheated particles because thinning from material loss or contamination build-up from repeated electron microscope observations could have made it difficult to obtain good microprobe results from heat-tested specimens.

Positive results were obtained for two particles, both large. Figure 20 shows a particle more than 4 microns long from screen P-12, together with its microprobe area scans (the dark area in the scan photographs is a hole in the substrate). Its major constituents are iron, nickel, calcium, and possibly silicon. Silicon and nickel backgrounds are high because of the composition of the substrate and shadow materials, making it somewhat more difficult to detect these elements. The nickel intensity from the particle is probably too great, however, to be attributable to a buildup of shadow upon it and most likely results from the presence of nickel in the

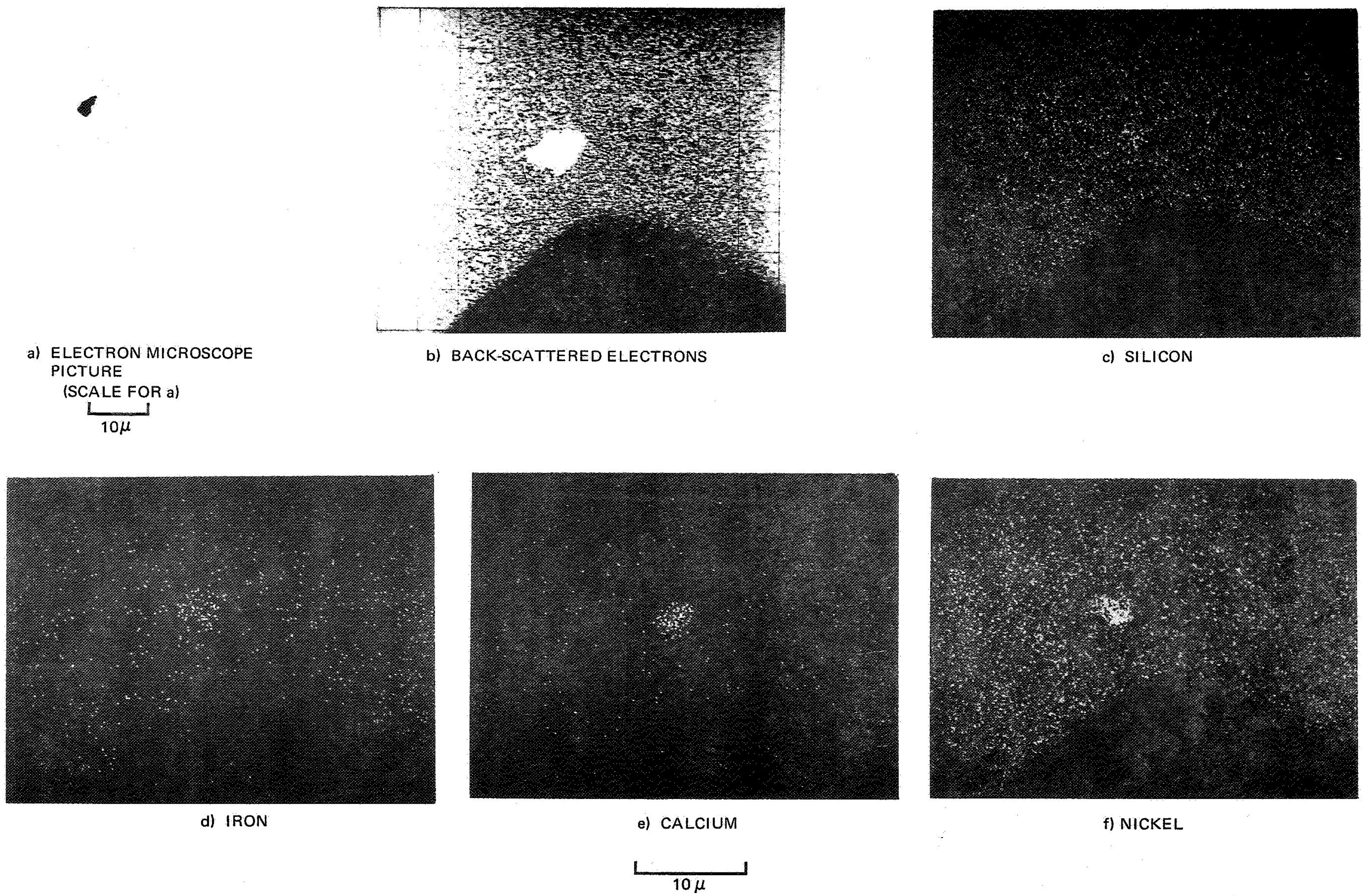


Figure 20. Particle on Screen P-12 and its Microprobe Area Scans

particle. In the case of silicon, on the other hand, the contrast is low, and the slight enhancement may be caused by silicon X-rays excited in the substrate by electrons scattered from the particle.

The particle shown in Figure 21 is from screen H-8 and is about 3 microns long by about 2 microns across. Its major constituents are iron, magnesium, and silicon. Possible minor constituents are aluminum, phosphorus, and chlorine.

The elements found in these two particles are not inconsistent with an extra-terrestrial origin. However, more data are required before such an origin can be considered. Heating tests for the existence of radiation damage have not been carried out because the contamination buildup from the microprobe is generally heavy enough to prevent electron-diffraction patterns from being obtained. Heat stability tests could be made; but since at least one of the particles is large enough that it might be manipulated, it may be better first to attempt to remove the carbonaceous contamination or even to perform other tests, such as X-ray diffraction.

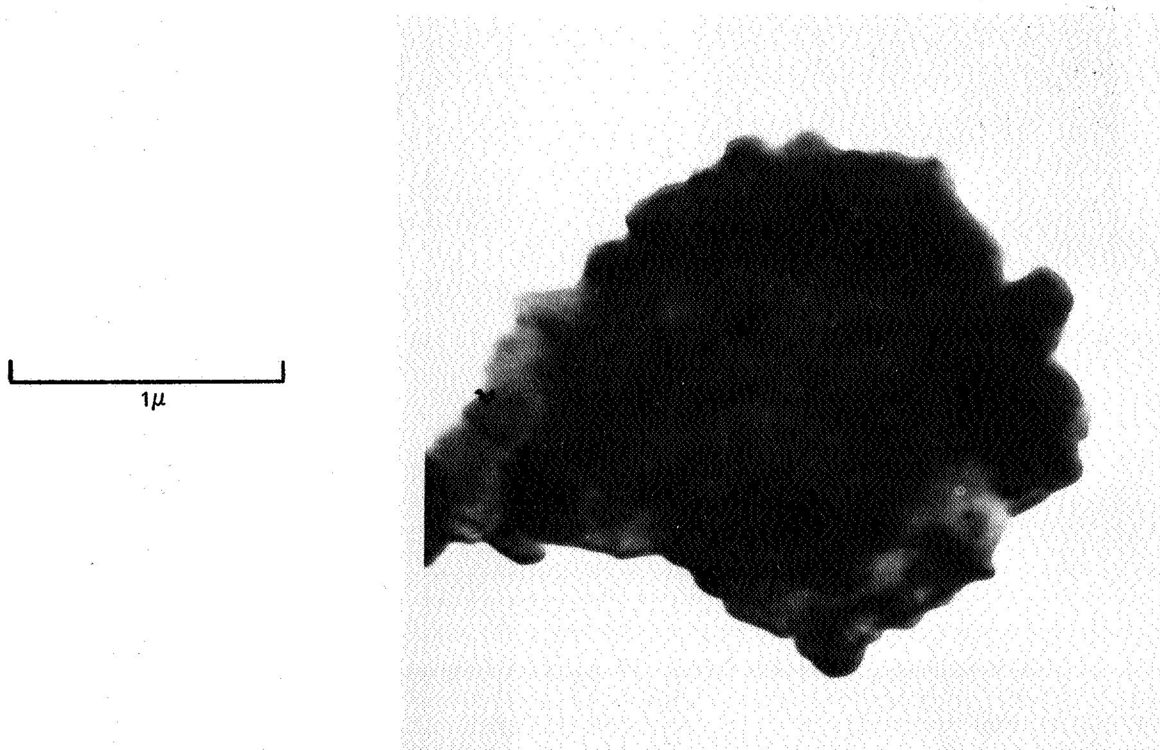


Figure 21. Particle on Screen H-8 Analyzed by Microprobe

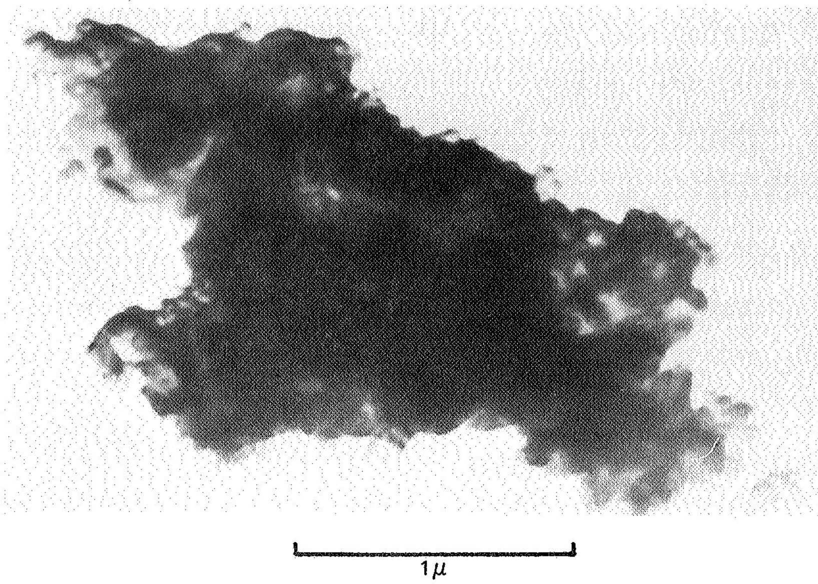
Another particle from screen P-12 is shown in Figure 22; it is about 3 microns in length. Carbon was the only element detected. (The irregular outline in the back-scattered electron photograph was caused by electrical-noise interference). A third particle from screen P-12 also gave a weak carbon signal and no indication of any other elements.

Microprobe analysis was also attempted on the large, elongated particle of Figure 8 from heated screen M-14. Although it is large enough to have been easily analyzed, neither area nor manual scanning of the part of the screen where it lay produced any detectable signal. It is very likely that this particle as well as the two above in which only carbon was detected are fragments of the silicon monoxide substrate film. The shapes of at least two of the three particles suggest this. So does the fact that no elements other than carbon were found, despite the large size, because large, flat fragments of silicon monoxide and nickel or indium would not give signals that rise above the background. The carbon may well be contamination buildup from the original scanning in the electron microscope. It is significant that no carbon was detected on the particle that had been heated in the electron microscope. The contamination in this case could have been burned off if a small amount of oxygen were present in the microscope column, as is possible at the operating vacuum of about 10^{-5} mm Hg.

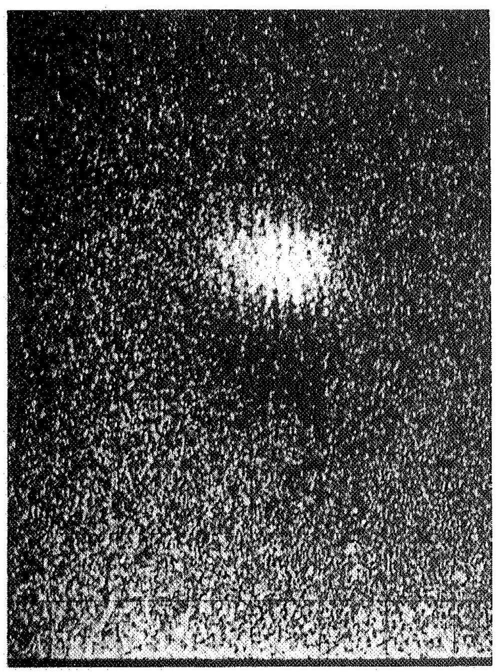
3.3.3 Control-Particle Analysis

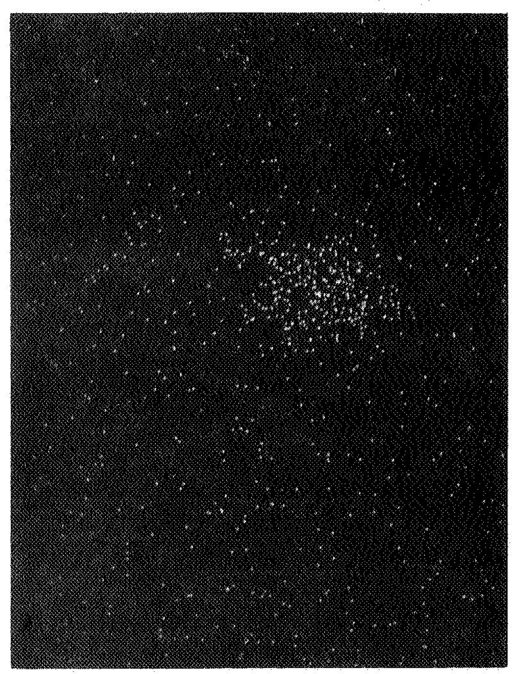
Figures 23 and 24 show the results of the microprobe scans of the silicate minerals olivine, $(Mg, Fe)_2SiO_4$, and enstatite, $(Mg, Fe)SiO_3$. In each figure "a" is the reverse print of the electron microscope photograph to show the size and shape of the particles, "b" is the numbering diagram for the particles, "c" and "d" are the nondispersive area scans (total X-ray output), the latter showing by the bright spots the fixed position of the beam when the 10-sec counts were made, and "e", "f", and "g" are, respectively, the silicon, iron, and magnesium area scans.

Only particles larger than about 2 or 3 microns are visible on the nondispersive area scans. The same limits hold for the element scans, except that the display for enstatite is less clear. Possibly the reason is that more enstatite particles are in the field of view than olivine particles.



a) ELECTRON MICROSCOPE PICTURE



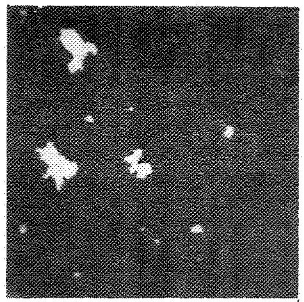


1μ

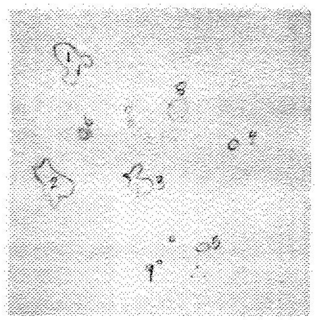
b) BACK-SCATTERED ELECTRONS

c) CARBON

Figure 22. Particle on Screen P-12 and its Microprobe Area Scans

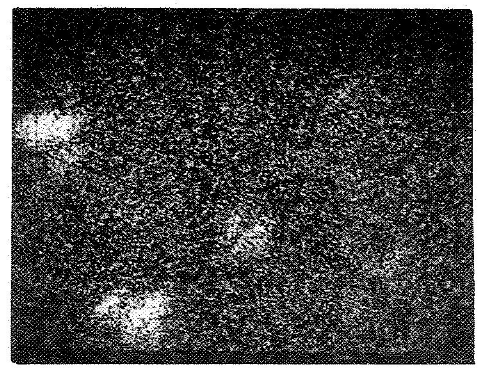


a) ELECTRON MICROSCOPE PICTURE, REVERSE PRINT



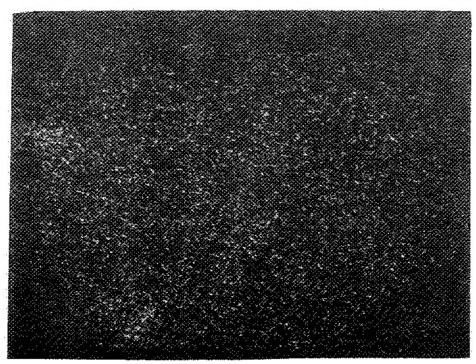
b) PARTICLE NUMBERING DIAGRAM

10 μ
(SCALE FOR a AND b)

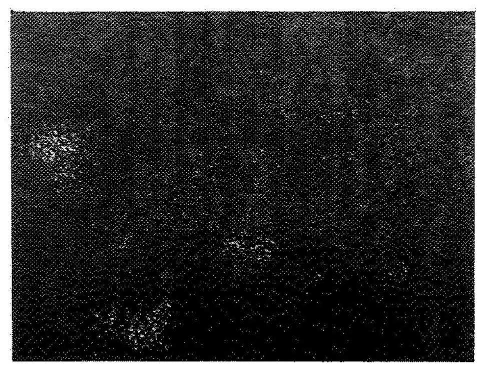


c) TOTAL X-RAYS, NONDISPERSIVE DETECTOR

d) TOTAL X-RAYS, NONDISPERSIVE DETECTOR; BRIGHT SPOTS SHOW BEAM POSITION FOR 10-SECOND COUNTS



e) SILICON

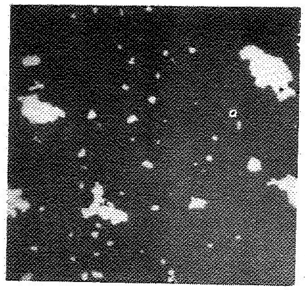


f) IRON

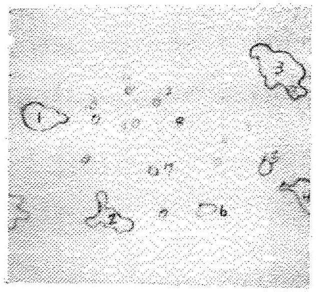
g) MAGNESIUM

10μ
(SCALE c, d, e, f AND g)

Figure 23. Olivine Particles and Their Microprobe Area Scans

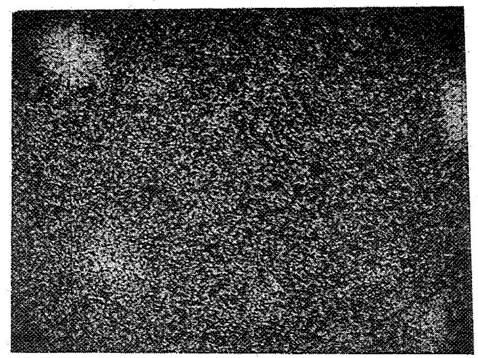


a) ELECTRON MICROSCOPE PICTURE, REVERSE PRINT

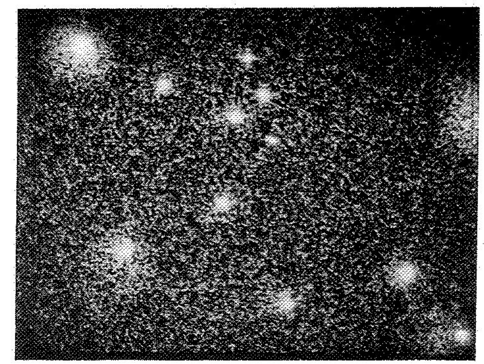


b) PARTICLE NUMBERING DIAGRAM

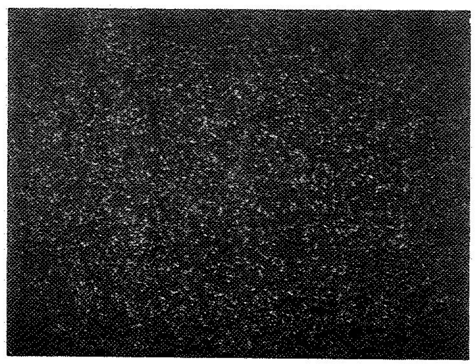
5 μ
(SCALE FOR a AND b)



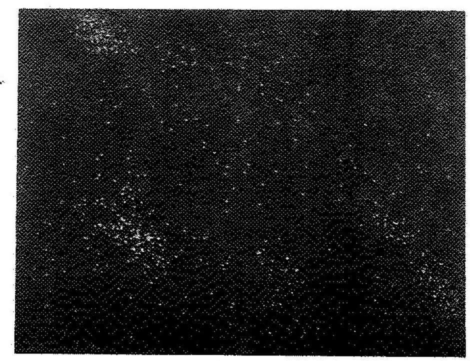
c) TOTAL X-RAYS, NON DISPERSIVE DETECTOR



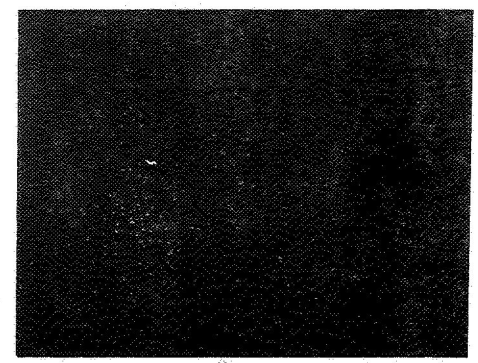
d) TOTAL X-RAYS, NONDISPERSIVE DETECTOR (BRIGHT SPOTS SHOW BEAM POSITION FOR 10-SECOND COUNTS



e) SILICON



f) IRON



g) MAGNESIUM

5 μ
(SCALE c, d, e, f, AND g)

Figure 24. Enstatite Particles and their Microprobe Area Scans

Table 1 gives the results of 10-sec counts with the dispersive detectors to analyze for silicon, iron, and magnesium. "Particle" 8 is the background count taken at beam position 8 of Figures 23b and 24b. The data show that iron and magnesium can be detected in particles as small as 0.4 micron, the size of olivine particle 7 and enstatite particle 11. Presumably other elements also can be detected in particles this small if the background is comparably low. Silicon, on the other hand, is more difficult to detect because of the high background from the substrate. However, a rough estimate from the data is that a silicon count of 10% above background may be obtained from particles of about 3/4 to 1 micron in size. In general, it appears that particles of 0.4 micron and larger on substrates of the type

Table 1

ELECTRON MICROPROBE X-RAY COUNTS FROM PARTICLES
(30 kV, 0.05 μA, 10-sec counting period)

Particle	Number of Counts		
	Silicon	Iron	Magnesium
OLIVINE			
8 (Background)	1575	24	21
1	2151	1323	716
2	3352	2516	2137
3	2742	1602	1465
4	2349	897	700
5	2074	771	635
6	1744	221	204
7	1552	65	57
9	1635	127	112
ENSTATITE			
8 (Background)	1686	48	31
1	5067	3168	1687
2	2564	813	447
3	5920	4050	1822
4	4132	2208	1165
5	2493	815	504
6	2139	373	255
7	1960	213	142
9	2035	251	150
10	1977	224	136
11	1675	77	54
12	1748	138	88

flown on Luster II can be analyzed successfully with the electron microprobe. Particles with only silicon as the chief constituent, such as quartz particles for example, would have to be larger to be analyzed with success; but particles of this composition may be rare or absent in cosmic dust if cosmic dust is assumed to be of meteoritic composition.

3.4 ABUNDANCE AND SIZE DATA

The calculation of the extraterrestrial particle flux at rocket altitudes from the particle abundance on the collectors has generally followed the method of Soberman et al (1961). The particles are assumed to have a constant ambient flux, f_a, and both they and the collector are assumed to be moving vertically. The number, N, of gathered particles of a given size per unit area of the collector is then written as

$$N = \int n_a (v_p \pm v_c)\, dt = f_a t \pm f_a \int \frac{v_c}{v_p}\, dt = f_a \left(t \pm \int \frac{dh}{v_p}\right)$$

where n_a is the ambient particle density, v_p and v_c are the particle and collector velocities, respectively, h is the altitude, and t is the time interval from the opening of the arms to the time when $v_p = v_c$ on the downward leg of the flight. For all practical purposes, it can be taken as the open time of the collector. The plus sign is used when the collector is rising and the minus sign when it is falling, so that from the symmetry of the trajectory those parts of the integral above the closing altitude cancel out and the equation becomes

$$N = f_a \left(t + \int_{h_o}^{h_c} \frac{dh}{v_p}\right)$$

where h_o and h_c are, respectively, the altitudes at which the arms open and close. (More accurately, h_c is the altitude at which $v_p = v_c$, but again, it can be considered with insignificant error as the closing altitude).

The expression in parentheses, t_e, is the effective collection time and has been evaluated by Carr and Gabe (1967) for Luster I, by Shafrir (1967) for Lusters I and II, and by Soberman et al (1961) for the Venus Flytrap. Customarily, v_p is assumed to be the terminal velocity. Shafrir, however, also considered the effect of a braking altitude above which the particles travel with cosmic velocity and below which they fall with terminal velocity. This altitude is well within the range of the Luster flights for the particle sizes of greatest interest, but Shafrir's values differed only slightly from comparison values he calculated on the assumption of terminal velocity only. This is probably to be expected because the second term in the expression for t_e, above, is the time it takes for a particle to fall from the closing to the opening altitude; this time is greatest, and hence its effect on t_e is greatest, for the lowest altitude.

No unanimity appears to exist as to the correct formula to use for the terminal velocity. For this calculation the Stokes equation with the Cunningham correction (Green and Lane, 1957) has been selected as the most appropriate for particles in the range of 0.1 to 10 microns at the low pressures existing at the Luster altitudes. This results in

$$v_p = \frac{\rho_p g D^2}{18\,\eta}\left(1 + \frac{2B\lambda}{D}\right)$$

where ρ_p is the particle density, taken to be 3 g cm^{-3}, g is the gravitational acceleration (cm sec^{-2}), D is the particle diameter (cm), η is the air viscosity (g cm^{-1} sec^{-1}), λ is the mean free path of the air molecules (cm), and B is a numerical factor which is ≈ 1. With D less than 10 microns and with $\lambda > 0.04$ cm at Luster altitudes, $2B\lambda/D >> 1$, so that

$$v_p \approx \frac{\rho_p g D^2}{18\eta}\left(\frac{2B\lambda}{D}\right) = \frac{gD\lambda}{3\eta}$$

With this expression, we have

$$t_e = t + \int_{h_o}^{h_c} \frac{dh}{v_p} = t + \int_{h_o}^{h_c} \frac{3\eta}{gD\lambda}\,dh$$

The integral can be evaluated numerically, but the variation with altitude of all the quantities is so small compared to the large variation in λ that all except λ can be treated as constants with negligible error. The expression can then be written

$$t_e = t + \frac{3\eta}{gD}\int_{h_o}^{h_c} \frac{dh}{\lambda} = t + \frac{3\eta}{gD}\int_{h_o}^{h_c} \frac{e^{-K(h-h_o)}}{\lambda_o}\,dh$$

$$= t + \frac{3\eta}{gD\lambda_o K}\left(1 - e^{-K(h_c-h_o)}\right) = t + \frac{3\eta}{gD\lambda_o K}$$

where $\lambda = \lambda_o e^{K(h-h_o)}$ closely approximates the altitude dependence of the mean free path over the Luster altitude range, and $e^{-K(h_c-h_o)} \ll 1$. This is of the form $t_e = t + \frac{k}{D}$. With t in sec and D in microns, k can be calculated from values at $h = h_o$ of η, g, and λ_o taken from the US Standard Atmosphere (1962). K is determined from the equation for λ by using values of λ_c at $h = h_c$ taken from the same reference. This gives the following:

Luster II	$k = 9.5 \times 10^3$ sec μ
Luster I	$k = 7.0 \times 10^4$ sec μ
Venus Flytrap	$k = 1.3 \times 10^3$ sec μ

These values of t_e give a flux of particles greater than 0.1 micron about an order of magnitude less than that reported by Soberman et al (1961) for the Venus Flytrap and about 1 to 2 orders of magnitude less than that calculated by Farlow, Blanchard, and Ferry (1967) for Luster I from the Douglas and other guest experimenter data. (Soberman and Hemenway [1965] later used a terminal-velocity formula based on gas kinetic relationships to recalculate t_e and the flux for the Venus Flytrap and arrived at about the same values as this study.) Since most if not all of the 91 particles found in this Luster II study are probably contaminants, a meaningful flux value cannot be given. If all these particles were extraterrestrial the flux would be 580 particles m^{-2} sec^{-1} greater than 0.1 micron; although this is about a third of the Venus Flytrap flux of 1800 calculated from the t_e above, it is probably too high to be of value even as an upper limit.

Table 2 gives the size and abundance data on the particles. The numbers are recalculated on a per m^2 basis in the third column, and in the fourth column they are recalculated to an abundance for the total area of the Douglas collector open to observation, 3.6×10^{-4} m^2 on 229 screens. (Titanium screens have 52% open mesh area and 1.8 mm^2 available for scanning, tungsten screens 40% and 1.4 mm^2, to correct the figures given in the Luster I report.)

Table 2

SIZE AND ABUNDANCE OF ONE-SHADOWED PARTICLES ON LUSTER II COLLECTOR

Diameter (μ)	Number Found	Normalized per m^2	Normalized per Entire Collector
0.1-0.2	4	2.8×10^5	100
0.2-0.4	22	1.6×10^6	580
0.4-0.8	34	2.4×10^6	860
0.8-1.6	20	1.4×10^6	500
1.6-3.2	10	7.1×10^5	260
3.2-6.4	1	7.1×10^4	26
Total	91	6.5×10^6	2300

These numbers can be compared with the number expected. Let N_c and f_c be, respectively, the cumulative number of particles per m^2 of collector surface and the cumulative flux of particles per m^2 per sec with diameters greater than D. Then

$$N_c = \int_D^\infty t_e \, df_c$$

The well-known cumulative flux curve is of the form

$$f_c = aD^{-b}$$

where a and b are constants. Taking df_c to be positive, this gives

$$N_c = \int_D^\infty (t + \frac{k}{D}) \, d(aD^{-b}) = \int_D^\infty tabD^{-(b+1)} \, dD + \int_D^\infty kabD^{-(b+2)} dD = taD^{-b} + \frac{kabD^{-(b+1)}}{b+1}$$

The value of N_c thus depends on the values chosen for the constants a and b of the flux equation. Data from the Gemini S-10 and S-12 experiments (Hemenway, Hallgren, and Kerridge, 1967) as well as other recent data indicate f_c does not exceed about 1 particle m^{-2} sec^{-1} at D = 0.1 micron. Quantity b is the negative slope of the flux curve; it is about 3 in the large-size region of the curve but appears to be very close to 1 in the region of 0.1 to 10 micron. Naumann's (1966) analysis of data from Pegasus and Explorers 16 and 23 indicates slopes of about 1/3 and less in this size range. Therefore, the calculations shown in Table 3 were made using one particle m^{-2} sec^{-1} for f_c at D = 0.1 micron, making $a = 10^{-b}$, and using both 1 and 1/3 as values for b. The table gives the expected number of particles per m^2 and per total area of the Douglas collector for the Luster I, Luster II, and Venus Flytrap flight parameters.

The particle abundances shown in the table depend strongly on the altitude at which the collector opens. As between the Venus Flytrap and Luster I abundances, for example, there is an increase of almost two orders of

Table 3

EXPECTED NUMBER OF EXTRATERRESTRIAL PARTICLES FOR VARIOUS ROCKET-COLLECTION FLIGHTS

Diameter (μ)	Luster II				Luster I				Venus Flytrap			
	b = 1		b = 1/3		b = 1		b = 1/3		b = 1		b = 1/3	
	m^{-2}	$Collector^{-1}$	m^{-2}	$Collector^{-1}$	m^{-2}	$Collector^{-1}$	m^{-2}	$Collector^{-1}$	m^{-2}	$Collector^{-1}$	m^{-2}	$Collector^{-1}$
0.1-0.2	36,000	13	14,000	6	260,000	94	110,000	39	5,000	1	2,000	1
0.2-0.4	9,000	3	5,800	2	66,000	24	43,000	16	1,200	1	800	
0.4-0.8	2,300	1	2,300	1	17,000	6	17,000	6	340			
0.8-1.6	560		900		4,100	2	7,000	2				
1.6-3.2					1,100		2,900	1				
3.2-6.4							1,100	1				

magnitude in going from an opening altitude of 88 km to one of 63 km. These higher Luster I numbers represent one particle for every two screens of the Douglas collector, indicating that adequate quantities of extraterrestrial particles can be collected with rockets if the assumptions on which the calculations are based are correct.

The problem lies not so much with the abundance of cosmic-dust particles as with the abundance of contaminant background. The 9 to 17 particles expected on the Luster II collector are less than 1% of the normalized number of 2300 actually found, almost all of which can be considered background. Even the much higher numbers for Luster I are only about 5% of this background. Two approaches can be followed to make the extraterrestrial component recognizable and available for study. One is to search for methods of reducing the background by at least several orders of magnitude. This probably cannot be done by merely improving present clean room and handling techniques but may be feasible with collectors of new design. The second approach is to seek distinguishing "fingerprints" by which an extraterrestrial particle can be recognized. These would include evidence of high-velocity penetration for particles collected above the braking altitude, where they are traveling with cosmic velocity. They would also include radiation damage and characteristic morphologic changes with heat.

The fact that significant advances along the lines of the first approach still lie in the future points up the importance of continuing the effort to develop criteria for recognition. Heat-induced morphologic changes have been shown to have some diagnostic value even though they are not likely to provide a simple "go/no-go" determination. Although the radiation damage criterion has yet to be proven, it cannot be rejected on the basis of this study because the morphologic changes, the microprobe results, the size distribution, and the small number of extraterrestrial particles expected for the Luster II flight conditions make it probable that all of the heated particles are contaminants. The failure to find evidence of radiation-damage annealing, therefore, is consistent with the idea that such damage is confined to extraterrestrial particles. This justifies continuing the effort to evaluate this criterion by studying the Luster III and IV collections now on hand, by studying the irradiated olivine and enstatite particles for control, and, if feasible, by heat-testing the two microprobe-analyzed particles of suggestive composition (Figures 20 and 21).

3.5 CONCLUSIONS

1. No heat-produced diffraction patterns that could be attributed to radiation damage were found in any of 43 particles tested. This does not rule out the validity of the radiation-damage criterion for extraterrestrial origin because indications are that these are contaminants. Rather, it can be taken as negative evidence, at least, that radiation damage is restricted to extraterrestrial particles.
2. Tests with control particles of known materials show that morphologic changes with heating help to identify some contaminants. If cosmic-dust particles are assumed to consist of meteoritic materials, particles that show significant change at temperatures of about 600°C and below or that show extensive destruction at these or higher temperatures up to 1000°C can be rejected as contaminants. Particles that are stable with heat, however, may be either terrestrial or extraterrestrial in origin.
3. Of the five particles from the flight samples analyzed with the electron microprobe, two particles showed compositions consistent with an extraterrestrial origin. More tests are required, however, before such an origin can be considered. At least one particle may be large enough to be manipulated so that additional tests not possible with submicron particles, such as X-ray diffraction, may be made.
4. Tests with control particles of olivine and enstatite show that silicate particles as small as 0.4 micron can be analyzed with the electron microprobe.
5. A formula was derived for the effective collection time based on the Stokes equation with the Cunningham correction; it gives flux estimates for the Luster I and Venus Flytrap Flights which are 1 to 2 orders of magnitude lower than those published previously. These are in better agreement with recent downward revisions in the estimated near-earth flux.

6. Calculations based on the estimated flux and on the effective collection-time formula show that reasonable numbers of cosmic-dust particles can be collected by rockets if sampling is begun at sufficiently low altitudes, particularly in the vicinity of the 63-km opening altitude of Luster I. The problem is to recognize these particles among a background several orders of magnitude more abundant. Techniques to do this by drastically reducing the background may be feasible but they are still in the future. On the other hand, finding diagnostic criteria of extraterrestrial origin, such as high-velocity impact, radiation damage, and heat-produced morphologic changes, is an effort in which progress has been made in this and other Douglas studies, and such effort should be continued.

7. The conclusion that most of the one-shadowed particles are contaminants is based on (a) the morphologic changes found in many after they were heated to 600°C and less, (b) the failure of the microprobe to find any element except carbon in some, and (c) the greater numbers and larger particle sizes than would be expected for this flight from the effective collection-time formula and a reasonable estimate for the near-earth flux.

Section 4

RECOMMENDED FUTURE STUDIES

1. Study the irradiated olivine and enstatite particles to see whether the electron-diffraction patterns have disappeared and, if they have, whether heat annealing can restore them. Two of the three samples making up the experiment have already been irradiated and are now being shipped back to Douglas; the third is still undergoing irradiation in the reactor. This control study is to determine whether the principle upon which the radiation-damage criterion is based is valid.

2. Study the flight samples of the 1967 Luster III and IV by means of heating tests and microprobe analyses. These collectors carried some aluminum oxide substrates (in addition to the customary ones of silicon monoxide), the low silicon background of which will improve the results of the microprobe analyses. They also carried sets of two parlodion-covered screens, one above the other, designed to collect and identify extraterrestrial particles by their high velocity signature. A particle with appropriate velocity would penetrate the top layer and come to rest on the bottom one, where it could be located in association with the overlying hole.

3. Make electron-diffraction, heating, and other appropriate tests on the two microprobe-analyzed particles of suggestive composition (Figures 20 and 21).

4. Heat particles of olivine, enstatite, meteorite iron, and other control materials of this study in the heating stage of the electron microscope to compare their stability and behavior in vacuum with their already-observed stability and behavior in an argon atmosphere. In addition, study the effect of atmospheres other than argon (such as oxygen), on heat-produced morphologic changes.

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