ELECTRON MICROPROBE STUDY OF SPHERULES FROM ATLANTIC OCEAN SEDIMENTS

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Abstract - Spherules recovered by K. Utech from Atlantic Ocean sediments (Albatross expedition, lat. 23°58'N, long. 38°56'W) were studied by means of electron microprobe techniques. Unlike the analyses of spherule surfaces reported by WRIGHT, HODGE and LANGWAY (1963), LANGWAY (1963), SCHMIDT (1964), HODGE, WRIGHT and LANGWAY (1964), MUTCH (1964), and LANGWAY and MARVIN (1964), those in the present study were performed on polished sections of the spherules. Apparent terrestrial alteration of particle surfaces and the introduction of contaminants suggest that analyses limited to surfaces cannot be regarded as representative of entire spherules. Thirteen spherules ranging from 60 to 450 μ in diameter were classed into four groups based on their textures and compositions; Group 1: magnetite laths containing Ni and Co, surrounded by Si-rich films at grain boundaries. Occasionally, an off-center, circular area of metallic Ni-Fe-Co and/or trevorite ($NiFe_2O_4$) is present. Group 2: homogeneous magnetite, with small amounts of Ni and Co. Group 3: magnetite with Si-rich lamellae in a dendritic intergrowth. Group 4: irregular particles commonly with low Fe content, but occasionally with minute Ni-rich flakes embedded in a granular matrix. Each particle shows effects of alteration, ranging from a narrow rim at the circumference on most to essentially complete corrosion of others. Lesser

amounts of major constituents were present in alteration zones; in addition, minor amounts of Si, Al, P, Ca, Mg, Mn, Cr and Ti were noted there. Where metallic Ni-Fe-Co areas were altered, trevorite with traces of V was formed.

The data suggest that spherules in Groups 1 and 2 are of meteoritic origin, representing ablation droplets swept from meteorites during passage through the earth's atmosphere, as first proposed by MURRAY and RENARD (1884). No definite conclusions were derived as to the origin of particles in Groups 3 and 4.

INTRODUCTION

MURRAY and RENARD (1884) were the first to investigate the chemical composition of micron-sized spherules which they recovered from sediments of the deep ocean basins. At the time of their study, the methods of analysis were inadequate for examining individual particles, and only the average abundances of major constituents in bulk samples could be determined. After MURRAY, several other workers studied similar materials, but were able to obtain only the most general chemical data (see compilation, SCHMIDT (1965)). In recent years, the development of the electron microprobe permitted the examination of individual spherules. Several workers employed this instrument in the study of particulate matter suspected to be of extraterrestrial origin; for example, FREDRIKSSON (1956); CASTAING and FREDRIKSSON (1958); FECHTIG and UTECH (1964); WRIGHT, HODGE and LANGWAY (1963); LANGWAY (1963); SCHMIDT (1964); HODGE, WRIGHT and LANGWAY (1964); MUTCH (1964); LANGWAY and MARVIN (1964). However, the analyses reported in the latter six papers were conducted only on the

surfaces of spherules. This practice suffers from several limitations. First, the curved surfaces of spherules produce variations in the electron incidence and X-ray take-off angles, leading to results which are difficult to interpret (BIRKS, 1963). Second, it was found by SCHMIDT et al. (1963) that the surfaces of most spherules are textured, showing considerable microtopography. This would also contribute to variation in emergent path length of X-rays from the sample. Third, the electron beam penetrates only a few microns into the sample, and the composition of internal components of spherules was beyond the reach of the surface analyses. Fourth, it was found that spherule surfaces are altered terrestrially, and measurements of their compositions are not representative of entire spherules (Fig. 1, Table 1). Therefore, in the present study, polished sections of spherules were prepared to overcome these limitations.

The samples investigated were kindly provided by Dr. Karl Utech of the Technische Hochschule, Braunschweig, Germany. As part of a study of microscopic particulate matter, Dr. Utech magnetically recovered black spherules from dispersed sediment samples of the mid-Atlantic Ocean (lat. 23°58'N, long. 38°56'W, Albatross expedition). Following microscopic examination, spherules were individually selected at random and mailed to our laboratory for analysis.

METHOD

Spherules were embedded in clear plastic hardened at 60°C, and polished with successively finer abrasives to yield a flat, smooth surface. The polished section of each spherule was photographed at high

magnification to provide a map for guiding analyses, and the samples were then vacuum coated with a layer of carbon a few hundred angstroms thick in order to make them conductive. The analyses were performed with an Applied Research Laboratories electron microprobe X-ray analyzer.

The chemical analyses of the spherules were performed in the following manner. Spherules were optically positioned and viewed while being bombarded by the electron beam. The ability to view a spherule during analysis permits an accurate correlation of chemical composition with the position of the beam on the sample. The analyses were conducted in three stages. First, each component was analyzed qualitatively by means of a stationary electron beam and by running the spectrometers through the complete wavelength ranges which correspond to elements of atomic numbers Z = 12 through 92 (magnesium through uranium). Second, scanning-beam photographs of the spherules were taken by sweeping the electron beam over a selected area and displaying the characteristic X-ray intensities detected by the spectrometers on an oscilloscope screen; polaroid photographs of these images are shown in Figs. 2 and 3. Third, quantitative analyses of the elements known to be present were carried out.

In the quantitative analyses, pure elements were used as standards for the determination of Mn, Cr, Ti and Co. To determine Ni both pure Ni and an analyzed steel of known composition (Ni = 5.01%) were used. For Fe, both pure Fe and a chemically analyzed magnetite (Fe_3O_4, Fe = 72.4%) were used as standards. For S, the standard was chemically analyzed pyrite (FeS_2, S = 53.60%), while for P, the standard was chemically analyzed apatite (P = 18.2%). The standard for Si and Mg was chemically analyzed olivine (19.0 and 29.2%, respectively). Ca and Al

values were based on chemically analyzed andesine (6.9 and 14.4%, respectively). Corrections were made for wavelength shift, detector dead time, background, mass absorption (using HEINRICH'S (1965) mass absorption coefficients and ADLER'S (1964) tables), secondary fluorescence (using WITTRY'S 1962 formulas) and atomic number. The atomic number corrections were made with experimentally determined factors (compare KEIL and ANDERSEN, 1965). The measurements were made with 20 kV electrons and up to 40 seconds of integration time.

RESULTS

Analyses of the 13 spherules examined are listed in Table 1. On the basis of their chemical compositions and internal structure, these spherules can be classified into four groups as decribed below.

Group 1: Spherules 1, 5 and 6

As illustrated by Fig. 1, Spherule 1 has three internal zones.

a. A circular "inner core" of approximately 50 μ diameter is acentrically located. It consists of two parts. The inner portion is similar in appearance to "cores" or "nucleii" pictured by FREDRIKSSON (1956) and by FECHTIG and UTECH (1964) but of different composition. The previous workers found cores to contain about 80% Fe, 20% Ni. However, the inner core of Spherule 1 has 77.5% Ni, 21.6% Fe and 1.19% Co (Table 1). The distribution of Ni and Fe in the inner core is demonstrated by electron scanning-beam pictures (Fig. 2) in which image intensity is roughly proportional to the amounts present. Only trace amounts of Si, Mn and Ti were detected in the inner core (Table 1). The outer zone, on the other hand, is comprised of a homogeneous oxide

of nickel and iron (Fe = 41.0%, Ni = 20.2%; Table 1). The boundary between the inner and outer zone is irregular. RAMDOHR (1964) suggested that this phase is trevorite, the nickel spinel ($NiFe_2O_4$). The chemical composition obtained by microprobe analysis is consistent with RAMDOHR'S suggestion. Also present, but in minor amounts, are Si, Ca, Al, Co, Ti, Mg, S and Mn; traces of V were also detected in this phase (Table 1). Figure 2 shows the Ni, Fe and Si distributions in part of the outer core.

b. The main portion of Spherule 1 is an aggregate of magnetite grains of about 5 μ × 15 μ in size. The optical identification was confirmed by the chemical data, since the Fe content of this material agrees well with that for the standard magnetite (Fe = 71.0% for the spherule vs. Fe = 72.4% for the standard). These results are consistent with the work of MUTCH (1964) who employed X-ray photographs of individual spherules recovered from Paleozoic salts to show the presence of magnetite as aggregates of variously oriented crystals. The magnetite of Spherule 1 is rich in Ni (1.42%) and in Co (0.25%) (Table 1). Minor amounts of Si, Al, Ca and Cr are also present. Figure 1 shows that darker-colored films about 1-5 μ across are present at the magnetite grain boundaries. These were investigated by scanning-beam techniques (Fig. 3) which show the films to be richer in Si. In order to evaluate the Si contents of this material, the sample was moved in 1 μ steps under the static electron beam and the readings were recorded after each step. Highest values for Si obtained in this manner are about 3.4% Si.

c. A lighter gray rim approximately 20 μ wide occurs at the circumference of Spherule 1 (Fig. 1). The boundary of this rim toward the inner magnetite main mass is diffuse, and the outer surface of the

spherule is irregular. The outer rim contains less iron than the magnetite of the spherule (Fe = 64.2% in the rim vs. Fe = 71.0% in the main magnetite, Table 1), representing a greater degree of oxidation. While Ni (1.93%) and Co (0.27%) are present in amounts comparable to the magnetite, Mg, Ca, Al and Si were found to be somewhat more abundant (Table 1).

Spherules 5 and 6 are similar to Spherule 1 in both texture and composition (Fig. 4). Spherule 5, which measures 182 μ × 164 μ, is the larger, slightly elliptical particle at the left of the figure. It consists of two textural and compositional regions, a central zone and an outer rim. The central zone is an aggregate of magnetite grains (Fe = 70.6; Table 1), differing from Spherule 1 primarily in the higher Ni and Co contents (Ni = 3.87%; Co = 0.56%). No metallic core is present, although it is possible that one is to be found beneath the level of the section examined. As in Spherule 1, thin films richer in Si occur at the boundaries of the magnetite grains, and minor amounts of other elements are present (Table 1). The lighter, outer rim of Spherule 5 is pitted, and the surface of the particle is irregular and uneven. Less Fe is present in the outer rim than in the center, another similarity to Spherule 1 (Table 1).

Spherule 6 is the smaller (70 μ diameter) particle at the right of Fig. 4. It contains an acentrically located circular core as does Spherule 1. This core is trevorite (Table 1), as found in Spherule 1, but metallic nickel-iron is not present. In addition, the central region and outer rim of Spherule 6 are also similar in composition to comparable parts of Spherule 1 (Table 1). In view of the close similarities in

texture and composition, it appears likely that Spherules 1, 5 and 6 were formed by the same mechanism and from similar parent materials.

Group 2: Spherules 2, 3 and 12

Spherules 2, 3 and 12 are 160 μ, 60 μ and 50 μ in diameter, respectively. Each is a homogeneous body, and lacks the variations in internal texture which characterized the spherules just described. Table 1 shows that Spherules 2, 3 and 12 are quite similar in composition, and that the amounts of principal constituents vary over small ranges: Fe from 64.0% (Spherule 12) to 68.2% (Spherule 3); Ni from 0.41% (Spherule 12) to 1.62% (Spherule 2); Co from 0.20% (Spherule 12) to 0.38% (Spherule 2). Chemically, these spherules are similar to the outer rims of the spherules of Group 1.

Group 3: Spherules 7 and 8

Spherules 7 and 8 (200 μ and 450 μ in diameter, respectively) are texturally and compositionally distinct from those of Groups 1 and 2. A dendritic texture of light lamellae in darker matrix is the most striking feature of these spherules (Figs. 5 and 6). Both spherules are fragile and soft which somewhat hindered sample preparation; relief produced during polishing caused the central portion of Spherule 7 to be out of focus (Fig. 5). In addition, Spherule 8 appears to have suffered severe surface corrosion (Fig. 6).

Results of analyses are given in Table 1. The light lamellae contain considerably more Si than the gray matrix; therefore, attempts were made to measure the composition of both light lamellae and gray matrix. The narrow light lamellae (about 1 μ wide) in Spherule 7 caused the electron beam to overlap the adjacent material and made it impossible

to determine the compositions of the two phases individually. Consequently, the Fe contents of these components in Table 1 are probably averaged and are not representative. Fortunately, the lamallae in Spherule 8 are somewhat wider (on the order of 10 μ across) and permitted approximate analyses of each phase. The analyses were carried out by moving the sample in 1 μ steps under the static electron beam. Highest Fe with lowest Si values were measured in the gray matrix, while highest Si and lowest Fe values were measured in the light lamellae (Table 1). The gray matrix contains 63.6% Fe, 1.70% Si and 3.0% Mg, but only barely detectable amounts of Ni (0.05%) and Co (0.08%). The light lamellae contain less Fe (36.7%) but are much richer in Si (14.0%) and Mg (5.7%).

Spherules similar to numbers 7 and 8 were recovered from marine sediments and studied by PARK and REID (1964). They also found low Fe contents, and very little Ni, with small amounts of Cu, Mg, Si, Ca, Cr and Mn. CHAO, DWORNIK and LITTLER (1964) pictured an iron spherule from a slag specimen with an intergrowth of phases remarkably like that found in Spherules 7 and 8. They found, however, that lamellae of α-iron alternated with those of cementite (Fe_3C), unlike the composition of spherules from ocean sediments.

Group 4: Particles 4, 9-11 and 13

Particles in this group are actually subrounded to irregular fragments and not spherules (Figs. 7, 8 and 9). The Fe contents of these particles are much less than for spherules of other groups, and their irregular shape also sets them apart. However, combined

presentation of the results for these particles is merely a convenience, and does not imply any genetic similarity. The dark, rounded areas in Particles 9 and 10 are holes produced by plucking of the soft material of the particles during polishing. Particles 9 and 10 are similar in texture with (1-2 μ across) light gray flakes embedded in a darker gray matrix. Data for Particle 9 suggest that these flakes are richer in Ni (0.47% in the flakes vs. <0.02% in the matrix). Analyses of the matrix material in Particles 9 and 10 show that it is rich in Fe (42.2% and 36.6%, respectively) and also contains Si (1.8% and 2.0%) with traces of other elements (Table 1). Both particles are poor in Co (0.08% and 0.05%).

Particles 4, 11 and 13 have a granular texture. Table 1 shows that the particles contain 37.1%, 30.3% and 44.4% Fe, respectively. The Ni and Co contents of Particles 4 and 11 are 0.06% or less. Particle 13 (Fig. 6) contains a greater amount of Ni (0.45%). Only minor amounts of other elements are present.

DISCUSSION

The present investigation demonstrates that the limitations of examination of particle surfaces can be over come by study of polished sections, which affords the opportunity to examine all parts of spherules. This is especially important because knowledge of the chemical composition of all phases present is essential if the origin of the spherules is to be properly interpreted. There is at present no parameter that can be used with absolute certainty to determine whether or not an

individual spherule or particle in the micron-size range is of extra-terrestrial origin.* However, a combination of certain chemical and textural characteristics allows, at least in some cases, more definite conclusions to be drawn to the origin of these objects. The presence of nickel and cobalt can be used with some confidence to establish a meteoritic origin. Only when a relationship to meteorites can be demonstrated can one, at this stage, be sure of the extraterrestrial nature of the material. The absence of nickel and cobalt, on the other hand, does not necessarily imply a nonmeteoritic origin, since these elements may have been lost in the process of spherule formation (FECHTIG and UTECH, 1964; SCHMIDT et al., 1964). Other parameters can be used with even less confidence. The presence of appreciable amounts of manganese, for instance, makes a meteoritic origin unlikely, although the object may still be extraterrestrial. High concentrations of elements known to be enriched in common terrestrial compounds suggest terrestrial origin (e.g. Zn). The drop-like shape and internal structure of many spherules are frequently attributed to formation during entry through the earth's atmosphere, although spherules can also be formed in terrestrial processes, such as welding, vulcanism, etc. In the discussion which follows, the data are evaluated on the basis of the above listed parameters in an attempt to indicate the origin of each group of particles.

Group 1

Spherules 1, 5 and 6 each contain Ni and Co. Moreover, their form and texture suggest that they were rapidly cooled from a melt. The data

*Measurements of cosmic-ray produced isotopes or primordial noble gas isotopes are restricted to bulk samples of larger mass.

suggest, therefore, that this group of spherules was produced during ablation of the surfaces of larger meteorites upon entry into the earth's atmosphere, as first proposed by MURRAY and RENARD (1884). The Ni- and Co-rich cores in Spherules 1 and 6 imply that ablation temperatures were sufficiently high to cause fractionation of Ni and Co from Fe. Simultaneous oxidation of Fe to magnetite was apparently followed by rapid cooling, preventing the growth of large magnetite grains. The accentrically located, dense Ni-rich cores may have been near the spherules' leading edges as they fell toward earth. The iron-poor (oxygen-rich) rim around the spherules probably originated by terrestrial weathering after the spherules were embedded in the sediment. The observed alterations may represent the attempt of the spherules to approach an equilibrium with the sedimentary environment. This mechanism may also account for the presence in the spherule outer rims of trace elements common to sediments, such as Al, Ca and Mg. It seems likely that the trevorite, which also contains Al, Mg and Ca, in addition V, originated in a similar way by terrestrial alteration of the metallic nickel-iron. Another, but less likely alternative, is that the trevorite may have formed from the nickel-iron during the latter part of the passage of the spherule through the earth's atmosphere.

Group 2

Spherules 2, 3 and 12 are homogeneous, with compositions similar to the outer rims of spherules in Group 1. They contain >0.4% Ni and >0.20% Co, and also appear to be of meteoritic origin. They may have merely been more weathered and oxidized than those in Group 1. Any internal texture which may have been present was destroyed.

Group 3

The origin of Spherules 7 and 8 is uncertain. Their form suggests that they were once molten. The compositions and textures of these spherules are distinct from the previous groups. They have also suffered severe alteration and corrosion.

Group 4

Particles 9 and 10 are apparently composed of different materials. Flakes with 0.4% Ni were found embedded in a Ni-poor (<0.05% Ni) matrix in these particles. Particles 4, 11 and 13 are homogeneous. No definite conclusions as to the origin of these particles were derived. Although Group 4 particles are irregular rather than spherical, their low Ni and Co contents are noteworthy.

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Table 1. Chemical composition of spherules by electron microprobe techniques (wt. percent)

Spherule	Fe	Ni	Co	Mn	Al	Si	P	S	Ca	Ti	Cr	Mg	Other
Group 1													
1 Rim	64.2	1.93	0.27	0.03	0.33	0.20	0.08	<0.02	0.89	<0.02	0.07	0.89	
Trevorite	41.0	20.2	0.38	0.09	0.77	2.70	0.36	0.13	0.87	0.30	<0.03	0.48	tr. V
Ni-Fe	21.6	77.5	1.19	0.08	<0.02	0.05	n.d.	<0.02	0.03	0.04	<0.02	n.d.	
Magnetite	71.0	1.42	0.25	<0.02	0.23	0.14	0.07	<0.02	0.06	<0.02	0.06	<0.02	
Si rich	56.0	n.d.	n.d.	n.d.	n.d.	3.4	n.d.	n.d.	0.60	n.d.	n.d.	n.d.	
5 Rim	64.3	4.02	0.59	0.04	0.09	0.08	<0.02	0.05	<0.02	<0.02	0.13	n.d.	
Magnetite	70.6	3.87	0.56	0.05	0.13	0.05	<0.02	0.04	<0.02	<0.02	0.12	0.13	
6 Rim	67.7	1.08	0.30	n.d.	0.06	0.06	n.d.	n.d.	<0.02	<0.02	n.d.	n.d.	
Trevorite	43.9	18.4	0.53	0.10	n.d.	2.47	0.36	n.d.	0.69	0.05	0.14	0.42	tr. V
Magnetite	71.7	1.13	0.39	0.03	<0.02	<0.02	<0.02	n.d.	<0.02	<0.02	0.18	<0.02	
Group 2													
2	64.6	1.62	0.38	<0.02	<0.02	<0.03	n.d.	<0.02	<0.02	<0.02	<0.02	<0.02	
3	68.2	0.72	0.25	<0.02	<0.02	<0.02	n.d.	<0.02	<0.02	0.09	0.20	<0.02	
12	64.0	0.41	0.20	<0.02	0.06	0.06	<0.02	0.05	<0.02	<0.02	<0.02	<0.02	
Group 3													
7 Lamellae	46.0	n.d.	n.d.	n.d.	n.d.	3.9	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	
Matrix	53.3	0.20	0.15	0.07	1.1	1.7	0.35	0.15	0.40	0.06	0.82	3.60	
8 Lamellae	36.7	<0.02	n.d.	<0.02	0.07	14.0	n.d.	<0.02	1.1	<0.02	<0.02	5.7	
Matrix	63.6	0.05	0.08	<0.02	<0.02	1.70	0.09	<0.02	<0.02	<0.02	0.03	3.0	
Group 4													
4	37.1	0.06	0.04	0.20	1.7	0.78	n.d.	<0.03	0.29	0.26	0.56	0.89	
9 Matrix	42.2	<0.02	0.08	0.32	2.0	1.8	1.33	0.17	1.00	0.90	0.34	1.48	
Flakes	48.0	0.47	n.d.	n.d.	2.0	1.5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	
10	36.6	<0.02	0.05	<0.02	2.9	2.0	0.69	0.14	0.91	0.70	0.48	1.56	
11	30.3	0.03	0.04	0.23	3.5	0.65	0.91	0.12	0.50	0.42	0.05	1.52	
13	44.4	0.45	0.06	0.16	3.0	0.6	0.04	0.10	0.35	0.28	0.40	1.65	

n.d. = not determined.

FIGURE LEGENDS

Fig. 1. Polished section of Spherule 1. a. Metallic nickel-iron. b. Trevorite. c. Magnetite. d. Oxidation rim. e. Si-rich rims.

Fig. 2. Electron beam scanning photographs of the core portion of Spherule 1. Right, metallic nickel-iron. Left, trevorite.

Fig. 3. Electron beam scanning photographs of the magnetite portion of Spherule 1 showing magnetite grains and Si-rich films at their boundaries.

Fig. 4. Polished sections of Spherules 5 and 6. a. Trevorite. b. Magnetite. c. Oxidation zone.

Fig. 5. Polished section of Spherule 7.

Fig. 6. Polished sections of Spherules 8 and 12 and Particles 9 and 13.

Fig. 7. Polished section of Particle 9. Note highly reflecting Ni-rich flakes embedded in the gray matrix.

Fig. 8. Polished section of Particle 10.

Fig. 9. Polished section of Particle 11. Dark area is a cavity in the center of the particle.

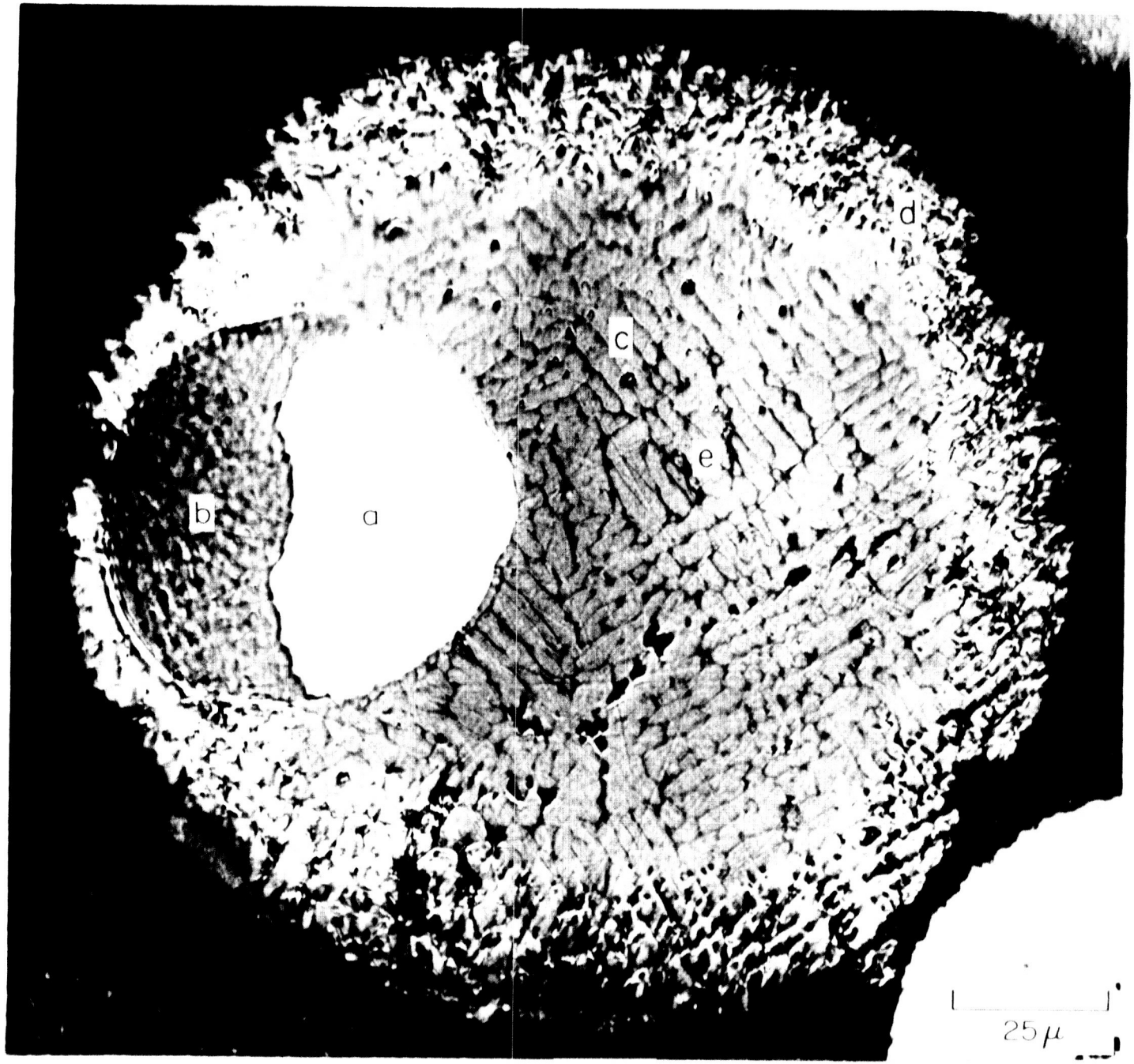
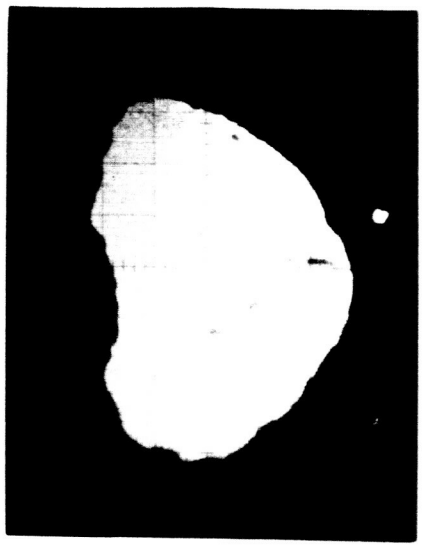
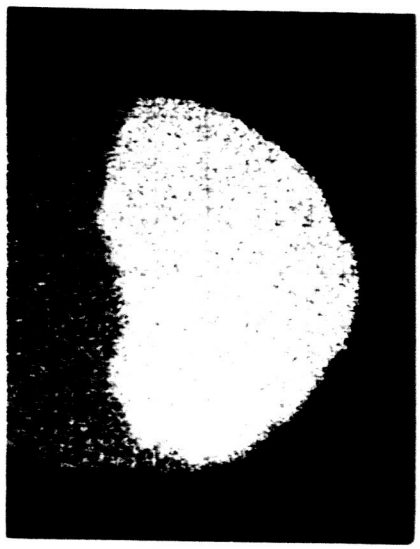


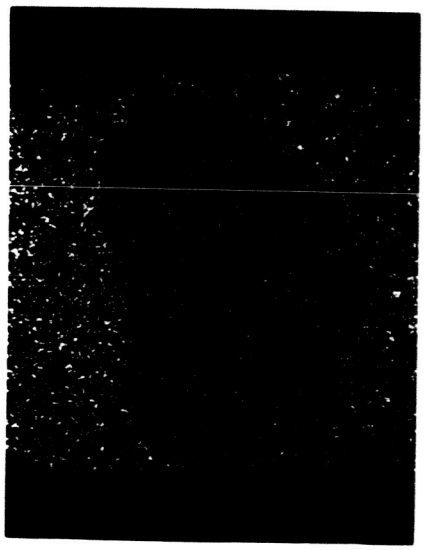
Fig. 1



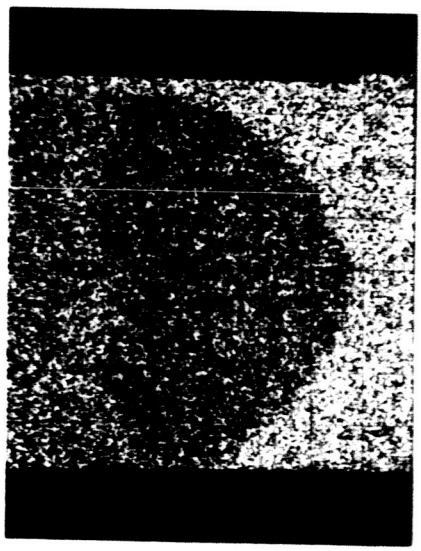
BSE



$Ni_{K\alpha}$



$Si_{K\alpha}$



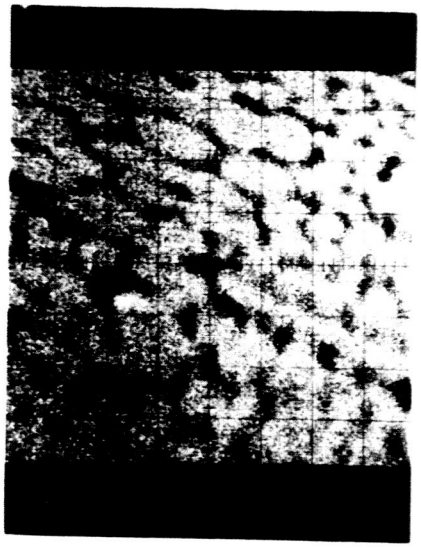
$Fe_{K\alpha}$

25μ

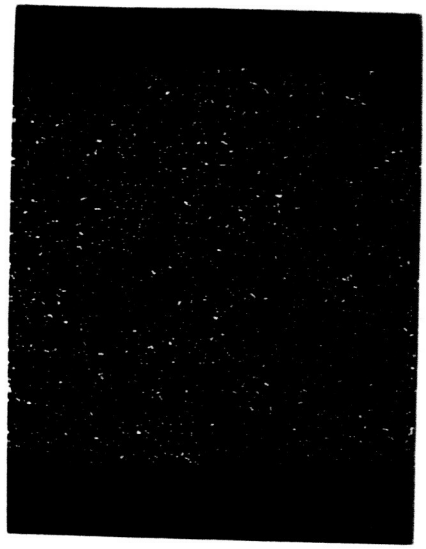
Fig. 2

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER MOFFETT FIELD CALIFORNIA

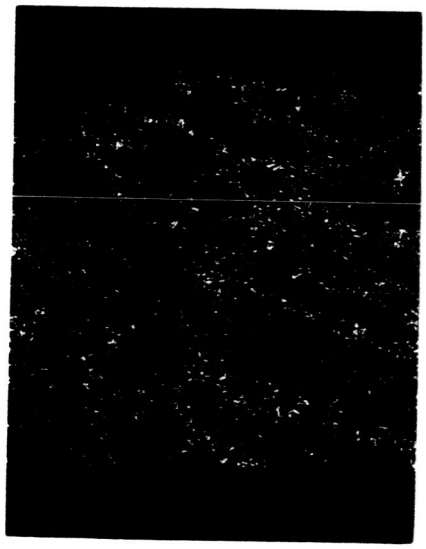
A-33341-4



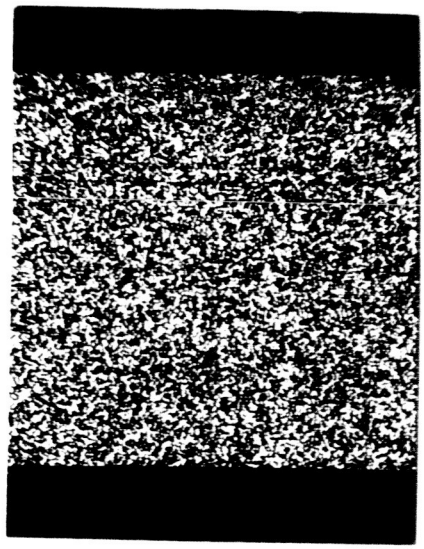
BSE



$Ni_{K\alpha}$



$Si_{K\alpha}$



$Fe_{K\alpha}$

25μ

Fig. 3

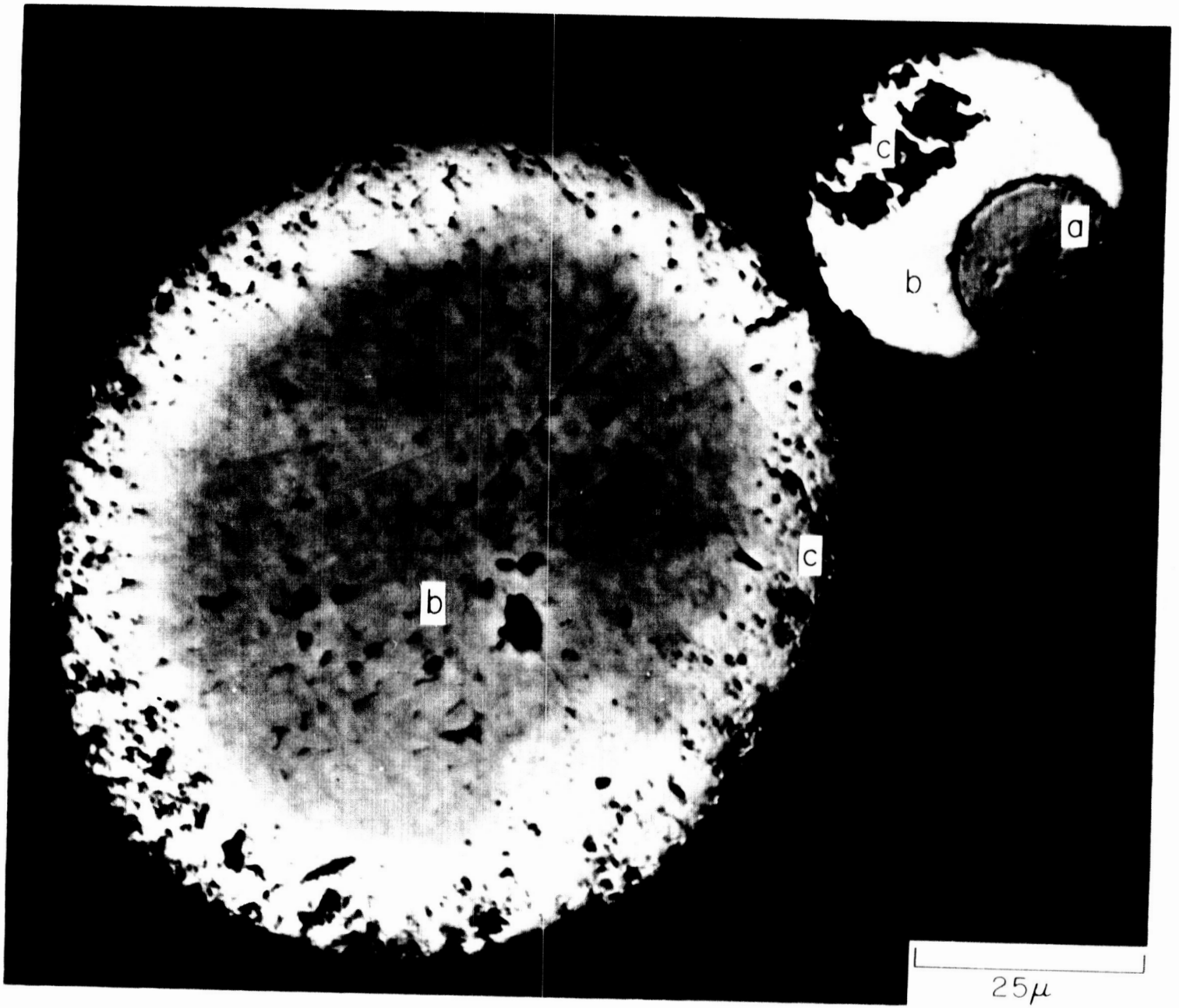


Fig. 4

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER · MOFFETT FIELD, CALIFORNIA

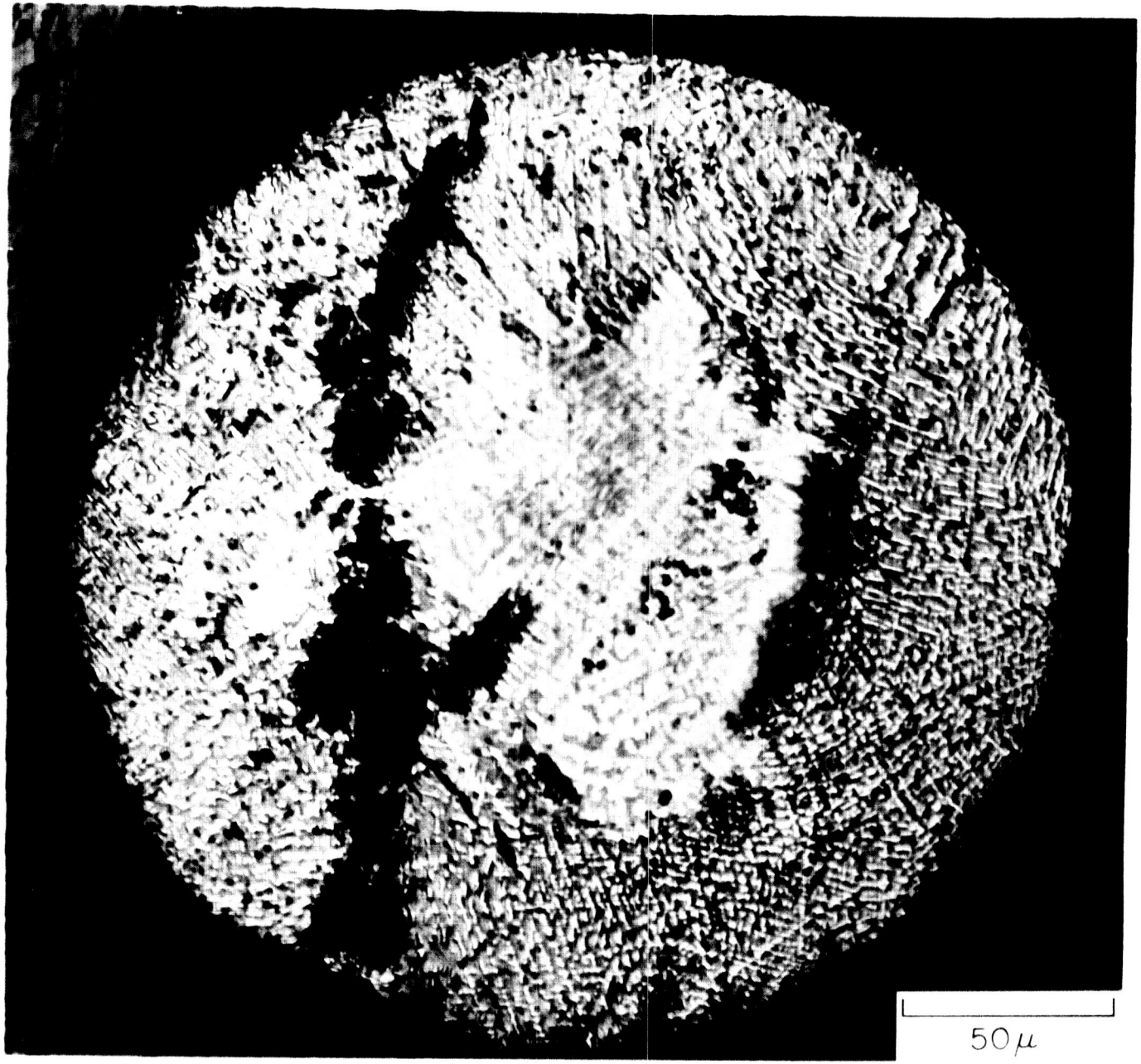


Fig. 5

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER MOFFETT FIELD CALIFORNIA

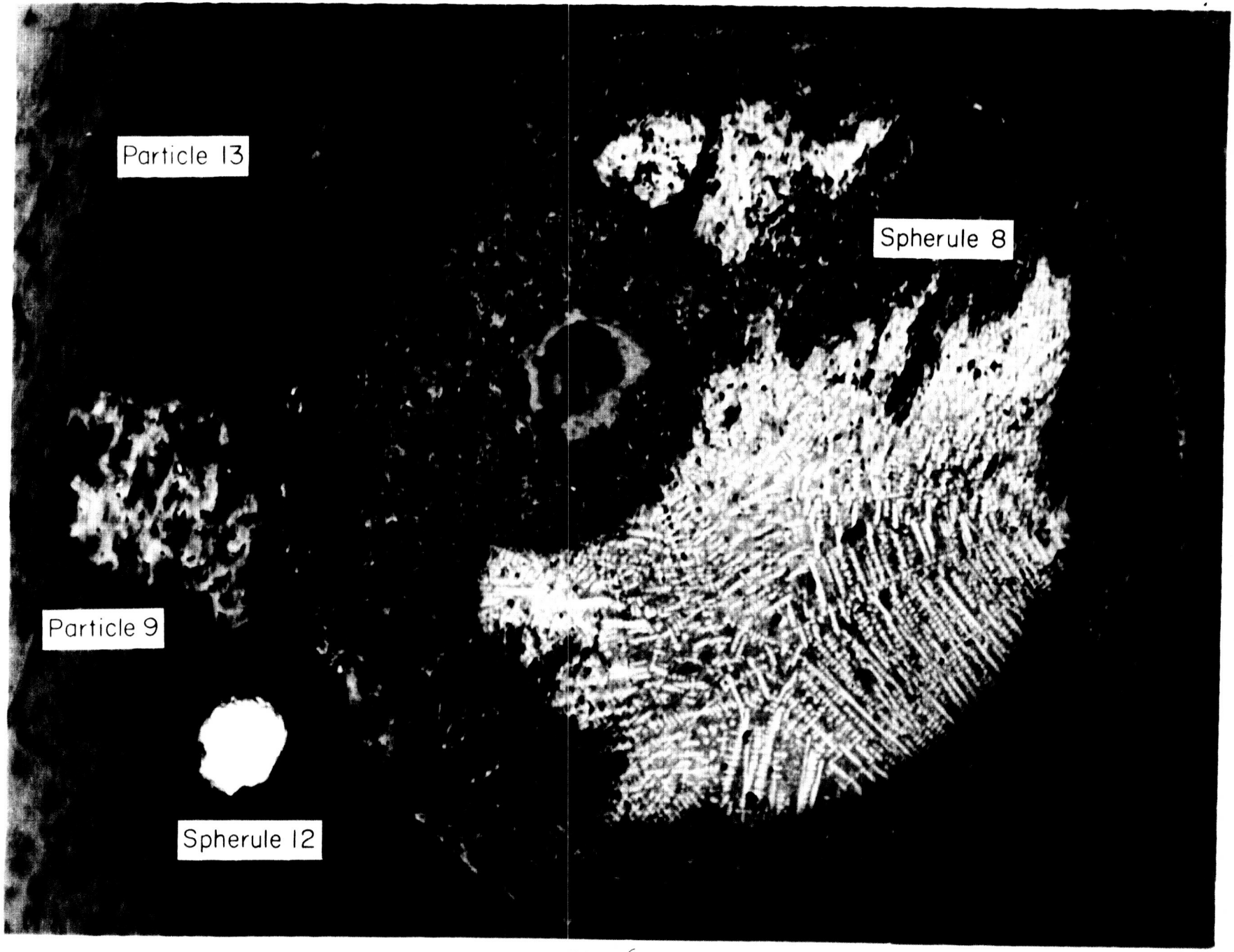


Fig. 6

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER, MOFFETT FIELD, CALIFORNIA

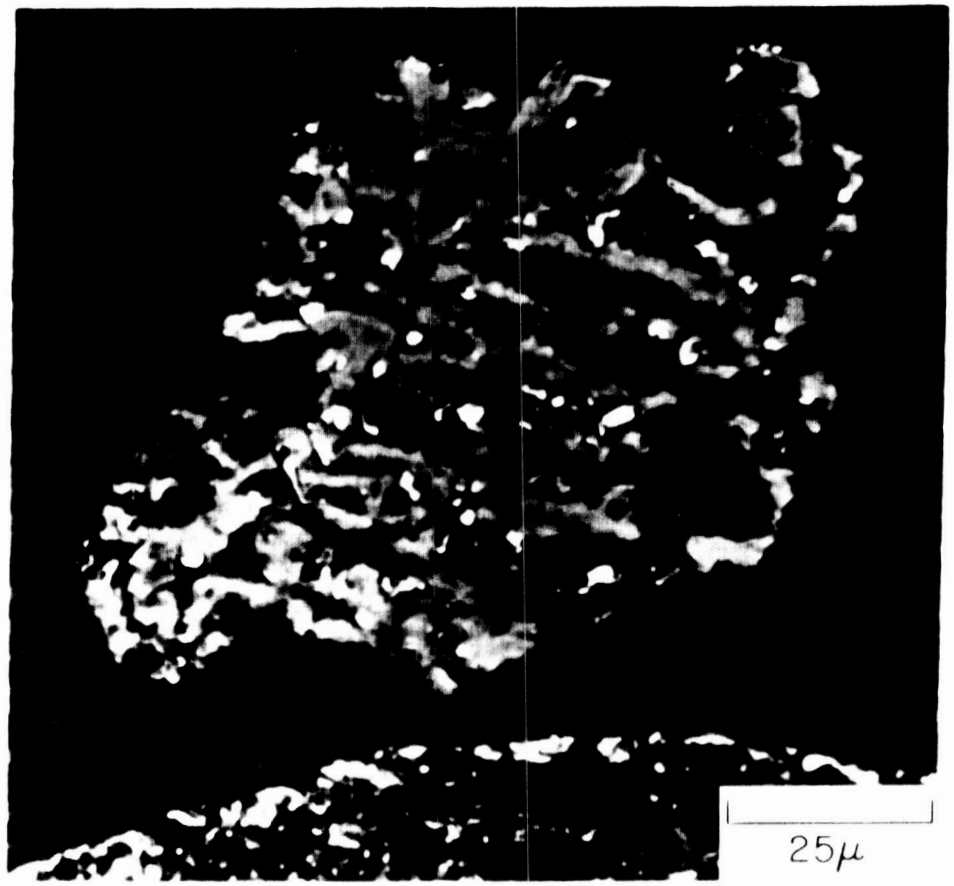


Fig. 7

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER MOFFETT FIELD CALIFORNIA

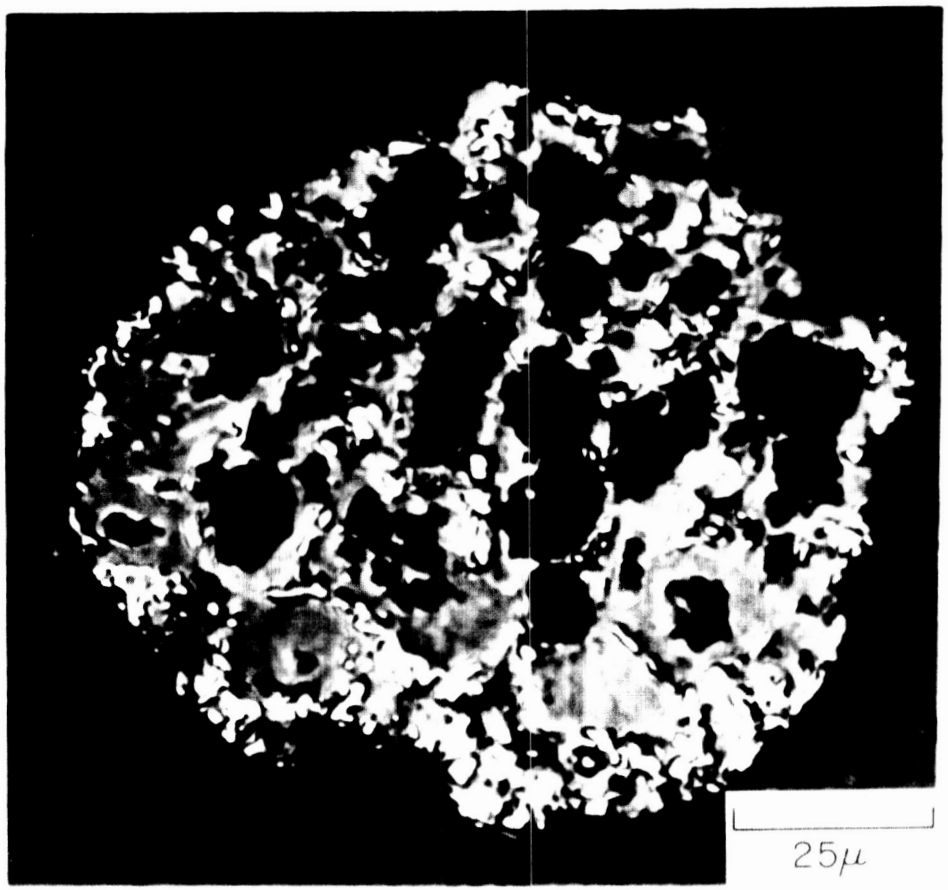


Fig. 8

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER, MOFFETT FIELD, CALIFORNIA

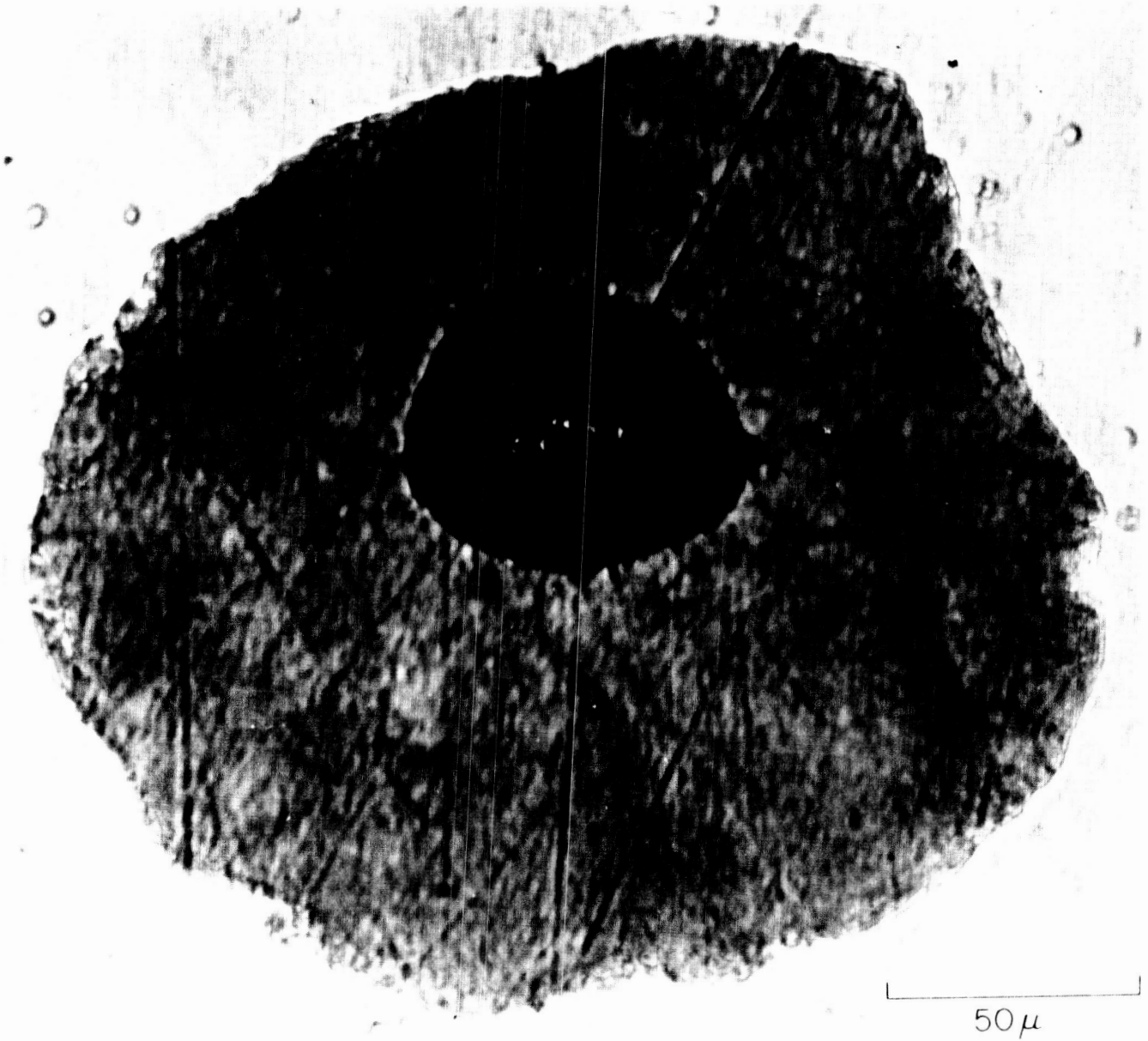


Fig. 9