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ANTARCTIC MICROMETEORITES COLLECTED AT THE DOME FUJI STATION

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Abstract: Antarctic micrometeorites (AMMs) were found among the precipitated fine particles recovered from a water tank in the Dome Fuji Station. These AMMs had been contained in the recent fallen snow around the station. Initial processing of the precipitated particles revealed that they were dominated by natural and artificial terrestrial materials, thus a series of processes were developed to separate AMMs from terrestrial particles. The recovery rate of AMMs by the processes was approximately 45% in weight, which was determined from a weight ratio of recovered/accreted AMMs. The micro-morphology and major-element concentration of the recovered AMMs were characterized. They appear to have been heated upon atmospheric entry to varying temperatures and can be classified into two major types based on the degree of heating: (1) fine-grained, irregular-shaped, partial-melted micrometeorites with chondritic composition, and (2) total-melted spherical micrometeorites with chondritic composition except for volatile elements. A digital catalog for the AMMs identified in this study was established on the web site [URL: *http://dust.cc.gakushuin.ac.jp/*], in which optical characteristics, high-resolution images, and chemical compositions of individual AMMs are presented. The AMMs listed in the catalog are the first Japanese collection of extraterrestrial dust. The criterion and techniques developed for the selection and initial analysis of AMMs are applicable for the dust samples that are being collected by the 39th Japanese Antarctic Research Expedition team.

1. Introduction

Micrometeorites are extraterrestrial particles smaller than approximately 1 mm in size and have been collected from deep-sea sediments (*e.g.*, MURRAY and RENARD, 1881; BRUUN *et al.*, 1955), the stratosphere (*e.g.*, CDPET, 1981–94; WARREN and ZOLENSKY, 1994), and the South and North Pole regions (*e.g.*, MAURETTE *et al.*, 1986, 1991, 1992a, 1994) where accumulation rates of terrestrial particulate are very low. They have a wide range of parental sources such as asteroids, comets, Mars, and Moon, as well as interstellar streams of dust (*e.g.*, TAYLOR *et al.*, 1996), and dominate the mass being accreted to the Earth (LOVE and BROWNLEE, 1993). It is believed that micrometeorites are a relatively unbiased sampling of the millimeter-sized meteoroids that traverse the inner solar system. Thus they should provide valuable information that cannot be derived from meteorites.

Micrometeorites usually have to be separated from a set of particles containing many terrestrial particles and a very small amounts of micrometeorites, such as precipitated fine particles of ice melt water from Antarctica and fine-grained muddy sediments from deep-sea bottoms. In the case of the deep-sea sediments it is very difficult to separate non-spherical micrometeorites due to extremely high abundance of terrestrial fine particles. Most previous studies on the micrometeorites from deep-sea sediments were, therefore, focused on the "spherules" (*e.g.*, YADA *et al.*, 1996). Compared with the deep-sea sediments, particles recovered from Antarctic ice fields showed a higher abundance of extraterrestrial material. YANO and NOGUCHI (1998) have carried out a systematic selection and an initial examination of AMMs contained in the 50–100 μm-sized particles sieved from 1 t of ice melt water and succeeded in separating unmelted AMMs. The sample particles they used were collected by MAURETTE *et al.* (1992a, b) at the blue ice field near Cap Prudhomme.

In the present study we have performed selection and an initial examination of AMMs in precipitated fine particles collected from approximately 100 t of snow melt water in Antarctica, in order to establish selection methods and criteria for identification of AMMs from among abundant terrestrial contaminants. The criteria and techniques developed in this study can be applied to the selection and classification of AMMs recovered from dust samples being collected by the 39th Japanese Antarctic Research Expedition (JARE-39) team in 1998–1999.

2. Collection Site and General Description of the Precipitated Fine Particles

The Antarctic Dome Fuji Station is located on the top of a moraine in Queen Maud Land at 77°19′ degrees south latitude, 39°42′ degrees east longitude, and is 3810 m above sea level. An average temperature in this region is −53.9°C and an average snow accumulation rate is approximately 10 cm per year, as determined from the spacing of tritium-rich ice layers generated by nuclear experiments (FUJII, 1998, personal communication). Four hundred liters of water that are used for the daily life of the residents are prepared every day from accumulated snow around the station. The snow is collected under the ground in the depth from 2.5 to 5.0 m. These layers of snow are estimated to have fallen during 1950's and 1970's based on the rates of snow accumulation. The precipitated material was pumped out from the bottom of a water tank made of stainless steel and was dried in the air in the station. Dr. KOJIMA at National Institute of Polar Research (NIPR) requested Prof. FUJII and JARE-37 team to collect the precipitated material three times in 1996 and to bring it back to NIPR, Japan. The precipitated material are residues of melting of approximately 100 t of snow. The material was kept frozen at −20°C in the deep-freezer room in NIPR until the initial investigation.

The total mass of the precipitated material is approximately 50 g. The material collected on April 23rd 1996 has been preserved in two bottles and named hereafter 960423-1 and -2 and that collected on June 3rd and September 1st 1996 was named 960603 and 960901, respectively. In the present study parts of 960423-1,-2, and 960901 were processed. A whole view of 960423-1 is shown in Fig. 1. Observation by a stereo optical microscope showed that the material consists mainly of artificial contamination such as pieces of wood and paint, fibers of clothes, and even human hairs. These contaminants were apparently incorporated during transfer of the snow to the water tank. The high abundance of contamination made it difficult to select AMMs directly from the precipitated material, thus for an initial processing we have mechanically separated the AMM-enriched fraction as described below.

Fig. 1. A complete view of the precipitated fine particles 960423-1. The dominant fraction consists of pieces of wood and fibers of clothes that were efficiently removed by the initial processing. A ruler on the right side measures 1 mm per one unit.

3. Initial Processing of the Precipitated Fine Particles

The precipitated fine particles were poured into pure water that contains little surfactant. Then the material was separated into eight fractions by grain size, magnetism, and sedimentation rates in the water by the following steps. (1) Separation of floating material on the water, which removed more than half of the precipitated material. The floating material was mostly pieces of wood and fibers of clothes. (2) Separation of particles with diameters more than 300μm by sieving. According to MAURETTE *et al.* (1987, 1994), AMMs with diameters more than 300μm are very rare, while our observation of the precipitated material showed that terrestrial material larger than 300μm was very abundant. This process removed approximately 20% of the precipitated material. (3) Separation by magnetism. (4) Separation of the magnetic particles by sedimentation rates in water. Water containing the magnetic particles was stirred well and left static. Water with precipitating material was removed 10 s later

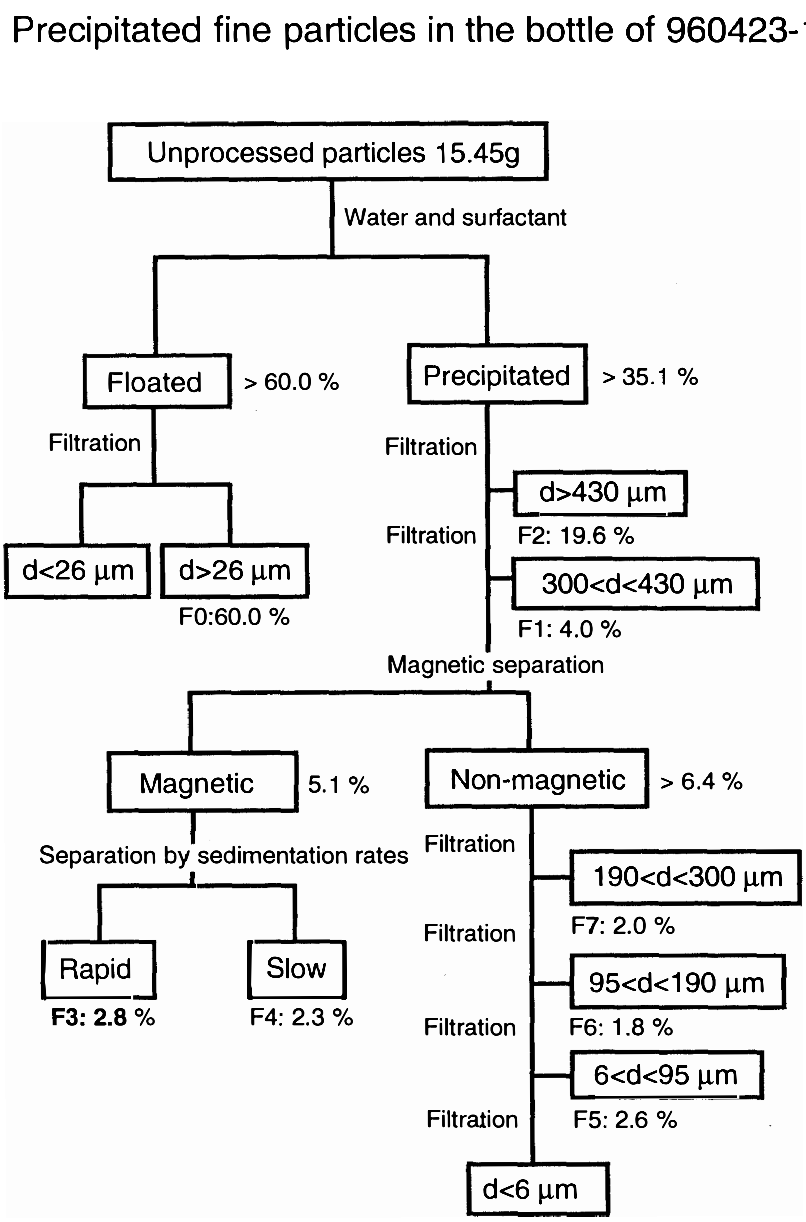

Fig. 2. A flow chart showing the whole process of initial processing to obtain an AMM-enriched fraction. Total recovery of the process is 95.1% and remainder is present in d<6μm and d<26μm fractions that were kept in the water.

and precipitated material was left in the bottom. Most flaky-shaped iron rust was removed due to high drag in the water. The whole separation process is summarized in Fig. 2.

We have carefully observed particles in all fractions by high magnification stereo microscopy and found that particles showing features of AMMs were preferentially present in the fraction of magnetic particles less than 300μm diameter with high sedimentation rates. Hereafter we denote this fraction as fraction F3 (Fig. 2). 433, 1662, and 436 mg of fraction F3 were separated from 960423-1, -2, and 960901, respectively. We cannot exclude the possibility of the existence of AMMs in fractions other than F3. In fact, one partially melted AMM was found in a non-magnetic fraction. But abundance of AMMs in fractions other than F3 is very low, thus we have intensively investigated the fraction F3.

4. Initial Characterization of the Fraction F3

Particles in the fraction F3 (Fig. 3) were characterized under stereo microscopes and classified into 7 types (Table 1). The main population of particles is irregularly or spherical shaped iron metals and oxides. The appearance and gloss of the spherical iron-oxides are very similar to those of iron-type cosmic spherules. Quantitative analyses of cross sections of the spherules were performed using an electron microprobe analyzer at Kyushu University (Jeol JX-733 superprobe), operated at accelerating voltage 15 kV and emission current 10 nA, showed that most of the iron-oxide spherules contain a trace amount of Ni (<0.1 wt%), Si (<1.0 wt%), and Mn (1.0 wt%), which differs from the typical compositional features of iron-type cosmic spherules that contain more Ni and less Si and Mn (Table 2). To determine whether these iron-oxide spherules are extraterrestrial or not, we have carried out gamma-ray measurement of the

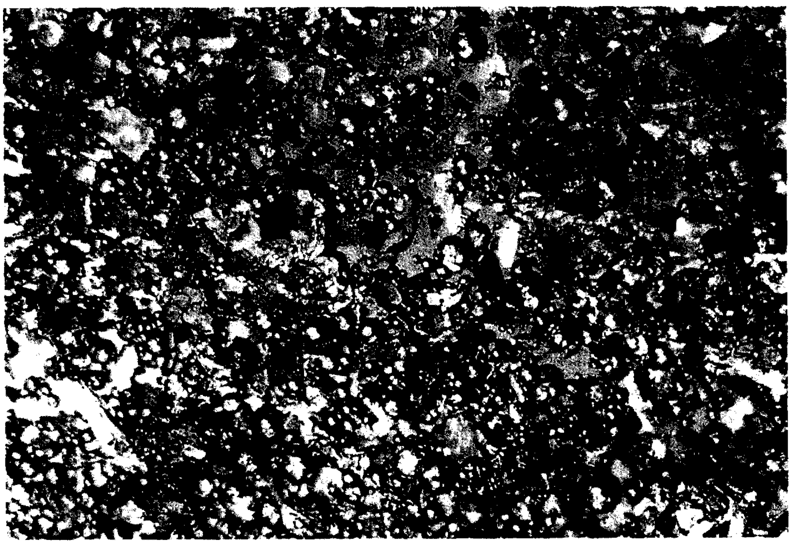

Fig. 3. An enlarged view of the fraction F3. Many dark gray spherules with variable diameter are present along with minor amounts of dark brown or transparent ones. Dark colored irregular-shaped particles, similar to the appearance of micrometeorites, are also abundant. Width of the image is 1.3 mm.

fraction F3. If the spherules are really extraterrestrial, gamma-rays from the nuclides produced by an interaction between cosmic rays and elements in spherules should be detected. Approximately 1 g of the fraction F3 from 960423-2 was investigated for an ^{26}Al-induced gamma-ray with a counting time 51 days at Tanashi low-background laboratory in Institute for Cosmic Ray Research, Univ. of Tokyo. No detectable ^{26}Al gamma-ray at 1809 keV was observed (Fig. 4), suggesting a low concentration of extraterrestrial material in the fraction F3. The absence of ^{26}Al and presence of trace Si and Mn indicate that most iron-oxide spherules in the fraction F3 are not extraterres-

Table 1. Optical characteristics and proportion of particles in fraction F3.

Type	Proportion in weight	Shape	Transparency	Color	Luster	Type
A	7%	**Irregular**	**Opaque**	**Dark**	**Dull to subvitreous**	**AMMs, TCA*, TCN#**
B	4%	Irregular	Opaque	Dark	Metallic to vitreous	TCA
C	16%	**Spherical to spheroidal**	**Opaque**	**Gray to dark**	**Dull to vitreous**	**AMMs, TCA, TCN**
D	5%	Spherical to spheroidal	Transparent to translucent	Colorless	Vitreous	TCA, TCN
E	16%	Irregular to cubic	Transparent to translucent	Yellow to colorless	Dull to vitreous	TCA, TCN
F	4%	Irregular	Opaque	Brown to dark green	Dull to vitreous	TCA, TCN
The others	48%	Irregular	Opaque	Dark to silver	Diverse	TCA

* TCA: terrestrial contamination artificial.
# TCN: terrestrial contamination natural.

Table 2. Major element abundance in Fe-rich spherules.

	Dome-Fuji spherules*	Deep-sea spherules#
Na_2O	0.00	0.02
MgO	0.02	0.08
Al_2O_3	0.06	0.02
SiO_2	0.16	0.04
SO_3	0.00	0.00
K_2O	0.00	0.00
CaO	0.01	0.02
TiO_2	0.00	0.01
Cr_2O_3	0.00	0.04
MnO	0.26	0.00
FeO	90.36	95.47
NiO	0.00	3.50
Total (wt%)	90.87	99.20

* An average value of analyses of 28 spherules.
# An average value of analyses of 12 spherules.

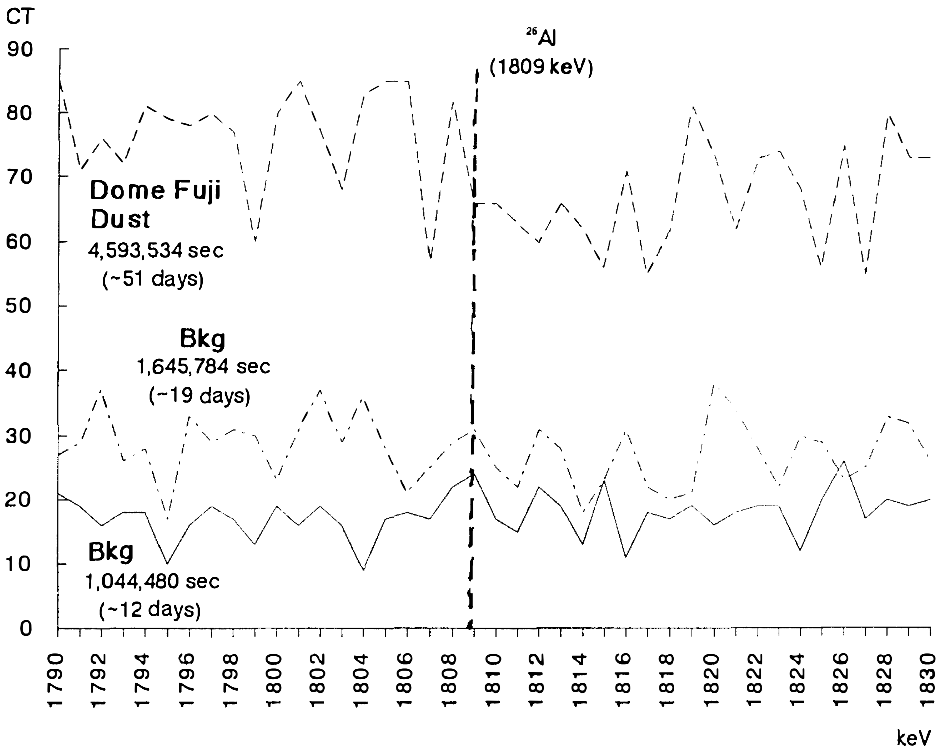

Fig. 4. An energy spectrum of gamma-rays in the range from 1790 to 1830 keV, where gamma-rays measured with the faction F3 is shown as "Dome Fuji Dust" and those without the faction F3 as "Bkg". No peak from ^{26}Al at 1809 keV is observed. The gamma-ray intensity of "Dome Fuji Dust" with a counting time 51 days is 2.5 times as high as that of background with counting time 19 days, indicating that gamma-rays from the fraction F3 are very weak.

trial and they were probably welding evaporates of iron material that were produced by some activities around the Dome Fuji Station.

5. Identification and Characterization of AMMs from the Fraction F3

In the next step we have done a direct hand picking of micrometeorites from the fraction F3 under high-magnification microscopes using a micromaniplator or a wet thread of brush for water color painting. Based on the morphology, surface color, luster, and transparency, particles of the fraction F3 were classified from type A to F as shown in Table 1. We selected AMM-candidates mainly from types A and C particles. The candidates are divided into two types: (1) irregularly shaped black to dark brown particles that were selected from type-A particles and are candidates for unmelted to partially melted micrometeorites, and (2) gray to black spherules showing lineation on the surfaces, which were selected from type-C particles and are candidates for totally melted micrometeorites, so called stony-type (S-type) cosmic spherules. The major element concentration and detailed morphology of the candidates were investigated by following instruments: (1) low-vacuum scanning electron microscopes with energy dispersive spectrometers (LV-SEM/EDS) at Kyuhsu University (Jeol JSM-5800LV) and Tokyo Institute of Technology (Jeol JSM-5300LV), which were operated at

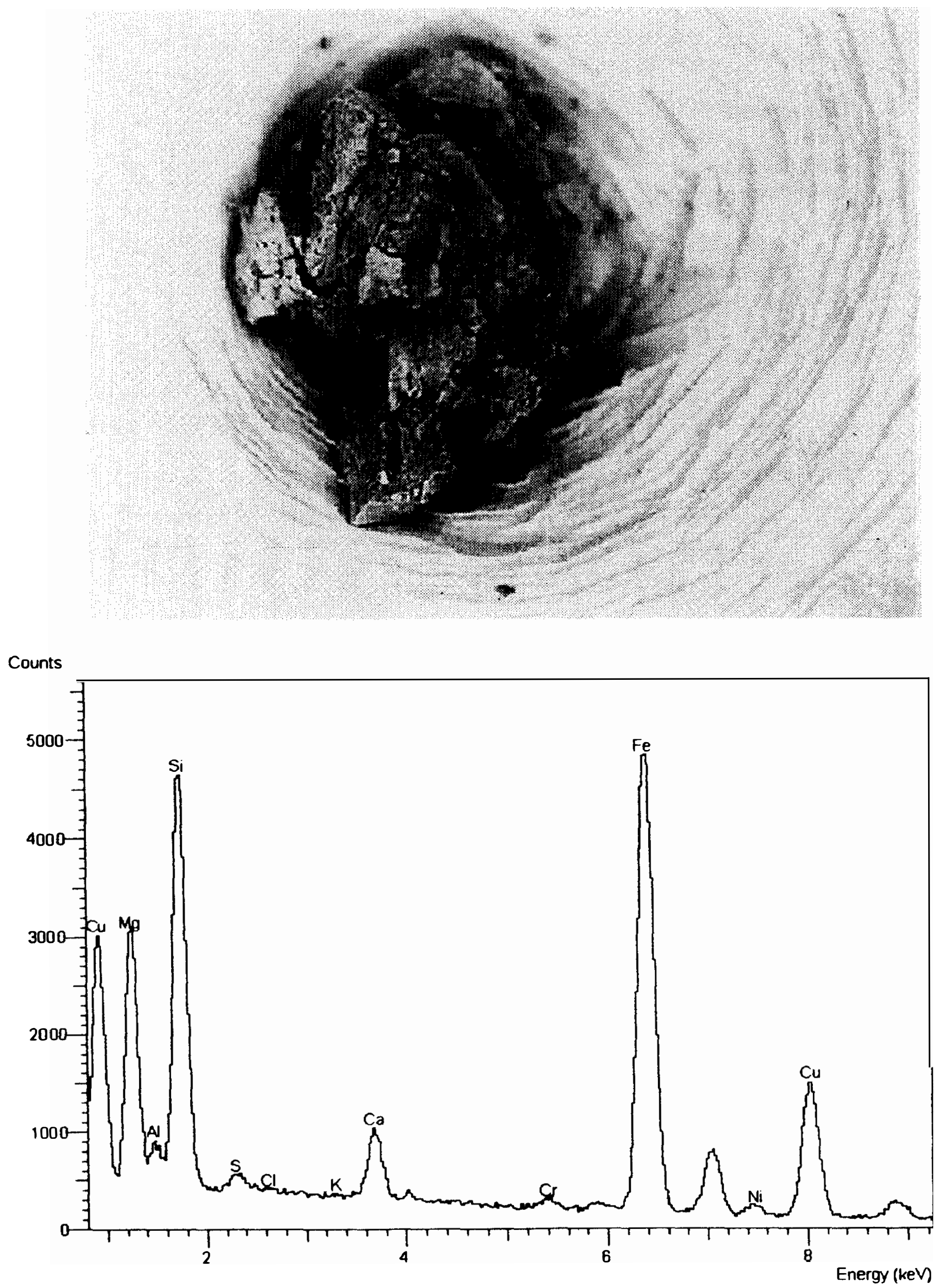

Fig. 5. An SEM image and an X-ray energy spectrum of an AMM with 250μm diameter. All AMMs identified in this study were numbered like "F96AK017" to indicate that the collection site is Dome Fuji Station ("F"), the year of collection is 1996 ("96"), the name of a fraction of particles is a fraction F3 of 960423-1 ("A"), the facility processed the particle is Kyushu University ("K"), and the particle identification number is 17 ("017"). Note that Cu in the energy spectrum is not indigeneous to the particle. It is from a Cu plate that holds the particle in the SEM chamber.

electron accelerating voltage 20 kV and emission current 1 nA, (2) a field-emission SEM with energy dispersive spectrometers at Dokkyo University (Jeol JSM-6301F) operated at accelerating voltage 20 kV and emission current 0.5 nA , and (3) an electron probe microanalyzer at NIPR (Jeol JXA-8800) with a wavelength dispersive spectrometer, operated at accelerating voltage 15 kV and emission current 11 nA. Bulk elemental analyses of candidates were obtained by using a defocused electron beam whose diameter is close to that of a particle.

Particles were identified as micrometeorites when they showed the following compositional features in the X-ray energy spectra (Fig. 5): (1) large peaks at Mg, Si, and Fe, (2) small peaks at Al and Ca, (3) traces of Cr and Ni, (3) variable height of a peak at S, and (4) absence of other elements, especially K. These are general characteristics of a chondritic composition, but sometimes terrestrial volcanic grains can have these characteristics. We analyzed several aggregates of fine-grained matrix material of the Allende CV3 and the Murchison CM2 chondrites for comparison. The X-ray energy peak heights of AMMs were consistent with those of Allende and Murchison by factors of three in most cases. The AMMs identified in this study are, therefore, real AMMs for the most parts, but we cannot rule out the possibility that some of them are terrestrial. For the further investigation a high-sensitive detector for cosmogenic nuclides is required to establish an extraterrestrial origin for these particles, as described in NISHIIZUMI *et al.* (1995) and NAKAMURA *et al.* (1999a, b).

Semi-quantitative SEM/EDS analyses using a defocused electron beam revealed that major element composition of external (surface) parts of AMMs was quite different from that of internal parts. The composition of external parts was obtained by analyses

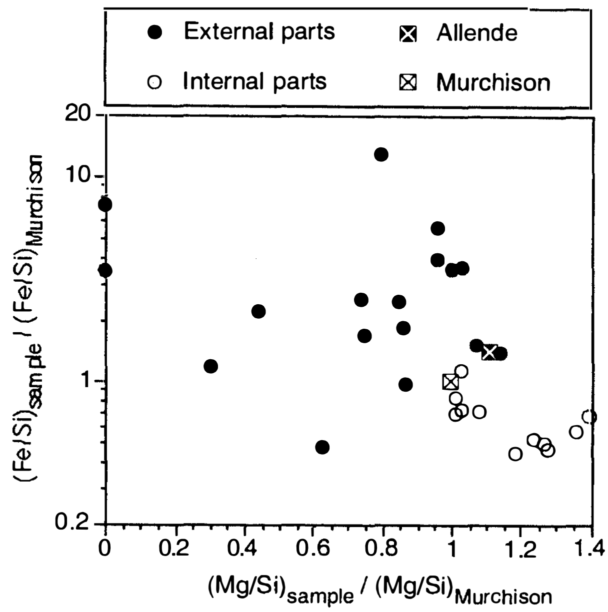

Fig. 6. A diagram showing Fe and Mg contents of the interior and exterior parts of AMMs. The Fe and Mg concentrations are normalized to Si and a composition of matrix of Murchison. Murchison and Allende data are shown for comparison. It is evident that interior parts are richer in Fe than the surface.

of exterior surfaces of AMMs, whereas that of internal parts was obtained by analyses of cross sections of AMMs (Fig. 6). There is a clear compositional trend: internal parts are rich in Mg and have compositions close to those of Allende and Murchison, whereas external parts are rich in Fe occasionally up to a factor of 10 when normalized to Si and a Murchison composition (Fig. 6). This confirmed the Fe enrichment on the surfaces of cosmic dust reported in previous studies (*e.g.*, BROWNLEE *et al.*, 1975; KELLER *et al.*, 1992).

Up to now we have identified 230 AMMs and selection of AMMs is still ongoing. The AMM catalog showing optical characteristics, images, and compositional data of individual AMMs is available to the public on the web site <*http://dust.cc.gakushuin.ac.jp/*> and detailed information of the catalog is reported in MURAKAMI *et al.* (1998). Optical characteristics of AMMs identified in this study showed some diversity in morphology and surface color. For semi-quantitative determination of the surface color of AMMs by stereo-microscope observations, a color index chart was prepared (Fig. 7), where colors were sequentially numbered from dark gray to black. Small amounts of red was added to all indices to reproduce AMM surface color more correctly. Thirteen AMMs were classified by the surface color. When AMMs do not show an exact color on the chart, the closest color was chosen for the classification. Figures 8a–d show a relationship between surface color and compositional features of AMMs. Most data plots seem to scatter in all diagrams and any correlation cannot be observed, suggesting that surface color of AMMs appears to be independent on the compositions (Figs. 8a–d).

In order to see the effects of heating and terrestrial alteration on the surface color of AMMs, thermally metamorphosed CM meteorites and S-type cosmic spherules collected from deep-sea sediments were investigated using the color chart. For the heating effects, AMMs were subjects to heating during residence in space and during passage of terrestrial atmosphere. To know the heating effects in space we compared AMMs with thermally metamorphosed CM chondrites, because it is known that CM chondrites are similar to AMMs in mineralogy (*e.g.*, BECKERLING and BISCHOFF, 1995;

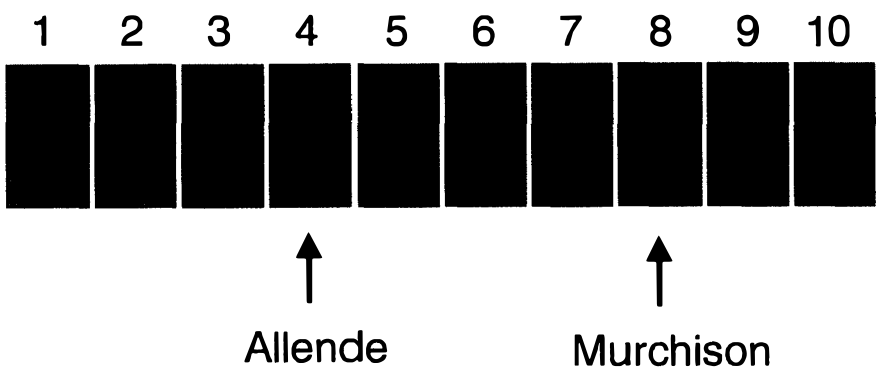

Fig. 7. A color index chart for the classification of surface color of micrometeorites and meteorites. For color standards, small chips of matrices of the Allende CV3 and the Murchison CM2 chondrites were measured for their surface colors and were classified to be index 4 and 8, respectively.

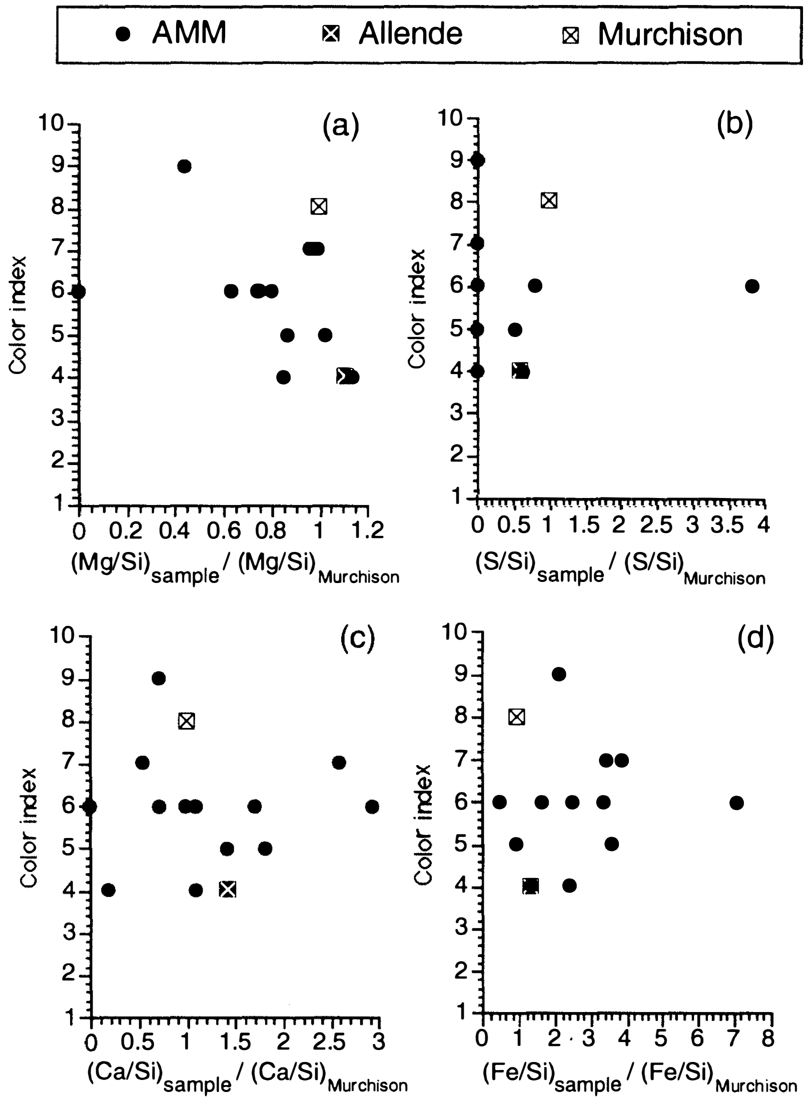

Fig. 8. A relationship between surface color and compositional features of AMMs. There seem to be no correlation between color index and Mg (a), S (b), Ca (c), and Fe (d) content.

GENGE *et al.*, 1997). Small chips with diameters up to 500μm of matrices of strongly heated CM chondrites Yamato-86789 and Belgica-7904, mildly heated Yamato-793321 (*e.g.*, AKAI, 1988; MATSUOKA *et al.*, 1996), and non-heated Murchison, Murray, Cold Bokkevelt, and Yamato-791198 were classified by the surface color (Fig. 9a). The diagram shows that CM chondrites having been more thermally metamorphosed exhibit lighter colors from index 5 to 7 (Fig. 9a). On the other hand, strongly heated AMMs which are nearly spherical due to melting generally showed darker color from index 7 to 9. For the terrestrial alteration effects, surface colors of the S-type spherules from deep-sea sediments, which have been experienced severe alteration, were investigated. They show variable surface colors ranging from index 1 to 9 (Fig. 9b), whereas S-type spherules from the Dome Fuji Station show dark colors from index 7 to 9.

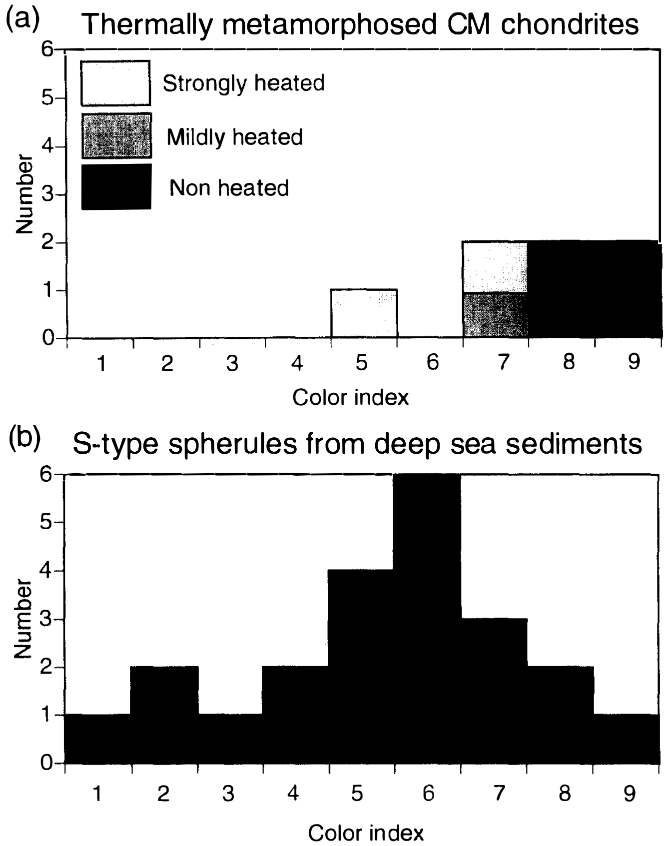

Fig. 9. (a) The colors of matrix of naturally heated CM chondrites. The more highly heated the meteorite, the lighter the colors that are exhibited. (b) Surface colors of S-type cosmic spherules recovered from deep-sea sediments, showing a wide range of color index.

6. Discussion

6.1. Implication from surface color of AMMs

In this section we will discuss how optical characteristics of AMMs such as surface color and morphology relate to the formation of AMMs. AMMs showed some diversity of surface colors, from color index 4 to 9 (Figs. 8a–d) and morphology, from irregular to spherical, suggesting a variety of origin and formation processes. EDS analyses indicate that colors of AMM surfaces seem to be independent of major-element composition (Figs. 8a–d). Many AMMs showed Fe enrichment on the surfaces (Fig. 6), indicating magnetite-rim formation around AMMs (*e.g.*, BROWNLEE *et al.*, 1975). Magnetite is black material and thus formation of magnetite rims reinforces blackening of AMM surfaces. But there seems no correlation between Fe content and color of AMM surfaces. Many physical properties such as grain size of magnetite, mixing ratios of magnetite to Fe-rich olivine, and the degree of smoothness of the surfaces would be responsible for the darkening of AMMs.

Formation of the magnetite rims takes place during passage of atmosphere (*e.g.*, BROWNLEE *et al.*, 1975) and requires heating at temperatures over 1100°C (FLYNN *et al.*, 1993) under a limited range of oxygen fugacity. Thus AMMs having magnetite rims were heated at least to 1100°C in the atmosphere. On the contrary, heating of CM chondrites in space does not accompany magnetite formation (*e.g.*, MATSUOKA *et al.*, 1996) and has changed the color of fine-grained matrix from dark to light (Fig. 9a). Therefore the presence of magnetite rims and dark surface color of many AMMs suggests that they have experienced stronger heating in the atmosphere than in space.

On the other hand, the terrestrial alteration causes erosion of the surfaces of micrometeorites (*e.g.*, CHEVALLIER *et al.*, 1987) and internal parts of micrometeorites must be exposed during the erosion. The color of the internal parts is variable depending on mineralogy and chemical compositions, which would explain why S-type spherules from deep-sea sediments, having been much more altered than AMMs, exhibit a variety of colors on the surfaces (Fig. 9b). The other alternative is that they were less likely to form magnetite rims in the first place.

6.2. *Calculated recovery rates of AMMs by the method developed in this study*

Next we calculated a recovery rate of AMMs by the process developed in this study. Terrestrial accretion rates of AMMs were estimated in various ways (*e.g.*, PEUCKER-EHRENBRINK, 1996) and the newest value is reported to be 2700±1400 tones per year for the AMMs in the diameter ranging from 50 to 700 μm (TAYLOR *et al.*, 1998). Assuming a homogeneous accretion of micrometeorites in any region of the Earth, the accretion rates in Antarctica is 5.3 μg per square meters per year, which corresponds to 1 particle of AMMs in typical size range. The rate of snowfall around the Dome Fuji Station is estimated to be 35 *l* in water per square meters per year (FUJII, 1998, personal communication). Based on the rates of AMM accretion and snow fall, 35 *l* water should contain 5.3 μg of AMMs. In the Dome Fuji Station 400 *l* snow-melt water was used every day. Thus, 960423-1 and -2 are precipitated material from 40000 *l* of water (100 days) and 960901 is from 35600 *l* (89 days) and, according to the AMM abundance in the water, the former should contain 6057 μg and the latter contains 5391 μg of AMMs. On the other hand, we have separated 2095 mg of fraction F3 from 960423-1 and -2 and separated 436 mg of F3 from 960901. Assuming that all AMMs in precipitated material are contained in fraction F3, 100 mg of F3 from 960423-1 and -2 should contain 289.1 μg of AMMs, whereas 100 mg of F3 from 960901 contains 1236.5 μg of AMMs.

In order to obtain the recovery rates of AMMs we have investigated 100 mg of F3 from 960423-1 and 100 mg of F3 from 960901. Seventeen and 56 AMMs were recovered from the F3 of 960423-1 and from that of 960901, corresponding to 149 and 488 μg of AMMs, respectively. By comparing these actual recovered mass with the expected ones, we can estimate recovery rates of AMMs to be 51.5 and 39.4% for 960423-1, -2 and 960901, respectively. The average rate is 45%. This rate appears to be fairly good considering the abundant terrestrial contamination in the unprocessed precipitated material (Fig. 1).

Compared with the recovery of AMMs by the EUROMET group, concentration of AMMs in unprocessed precipitated material from the Dome Fuji Station (~5 AMMs/1 g precipitated material) is better than the sediment in blue ice lake in Greenland (~1

AMM/1 g; MAURETTE *et al.*, 1987), but much worse than the blue ice field in Antarctica (~1000 AMMs/1 g; MAURETTE *et al.*, 1991). The ratio of unmelted and partially melted micrometeorites to totally melted ones is 1.1, which is higher than the two other collection sites: 0.4 and 0.7 for the blue ice lake in Greenland and the blue ice field in Antarctica, respectively. This is mainly due to the difficulty in distinguishing between spherical AMMs and welding contaminants, which resulted in limited recovery of totally melted AMMs in this study.

7. Concluding Remarks

Precipitated material in the water tank in the Dome Fuji Station was investigated. A series of processes for selection and initial examination of AMMs were developed. Approximately 45% of AMMs in the precipitated material was recovered by the processes. Based on the snow accumulation rates micrometeorites found in this study are supposed to have fallen to Antarctica recently, thus further investigations of individual AMMs might provide us valuable information on the nature of modern interplanetary dust.

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