Mineralogy of 100-μg Test Samples for Muses-C Mission

By

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Abstract: We have characterized mineralogy of individual mineral grains and small mineral aggregates picked out from 1H and 2H powder test samples for Muses-C mission. Total amounts we used for mineralogical characterization are approximately 100 μg. Fourteen individual mineral grains, less than 150 μm in diameter, and six small mineral aggregates, less than 200 μm in diameter, were exposed one by one to synchrotron X-ray radiation in order to determine bulk mineralogy based on X-ray diffraction characteristics. The samples of individual mineral grains were polished and analyzed by an electron microprobe analyzer (EPMA) for quantitative analysis of major-element concentration. The samples of mineral aggregates were microtomed or crushed for detailed mineralogical observation and quantitative chemical analysis by transmission electron microscopy (TEM). All mineralogical information, including bulk mineralogy, chemical composition, and nano-scale textures, were obtained from very small quantity of test samples owing to efficient combination of the mineralogical analysis we developed.

Our mineralogical analysis showed that both 1H and 2H samples are silicate-rich material with minor amounts of FeNi-metal and FeS- and FeNiS-sulfide. The silicates are mostly olivine and pyroxene and rarely plagioclase. The mineral combination of the 1H and 2H samples suggests that they are similar to stony meteorites, especially chondrites. The presence of abundant coarse olivine and low-Ca pyroxene with homogeneous composition F077 and F078, respectively, suggests that the coarse silicate grains in the 1H sample are similar to those in equilibrated L chondrites. But there are some mineralogical features in the 1H sample inconsistent with the idea that 1H sample is powdered material of a single equilibrated L chondrite. On the other hand, there are abundant mineralogical characteristics found in the 2H test sample, indicating that 2H sample is similar to anhydrous carbonaceous chondrite with low petrologic type.

1. INTRODUCTION

We have received 1H and 2H test samples from ISAS on October 17th, 2000. The amounts of Mineralogy of 100-μg test samples samples allocated to us were so huge and such amounts

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cannot be expected for the initial characterization of the true sample recovered from an asteroid. The most important point we think for the initial characterization is to characterize mineralogy using minimum quantity of the samples, because total amounts of the true sample is limited but the number of scientific projects for the sample will be unlimited. From this stand of view, we focused to characterize mineralogy of individual mineral grains and some mineral aggregates selected from the 1H and 2H test samples and most parts of the samples were returned to ISAS without any processes.

2. EXPERIMENTAL PROCEDURES

2.1 Sample selection

Both 1H and 2H samples are powders and consist of relatively coarse single mineral grains more than 50 μm in diameter and fine-grained mineral aggregates. Thus, samples we analyzed are divided into two types: single mineral grains less than 150 μm in diameter and small aggregates with diameter less than 200 μm consisting of very fine minerals. We have picked out single mineral grains showing different optical characteristics under a high-resolution stereoscope that represent coarse mineral grains in the 1H and 2H samples. Seven mineral grains numbered from 1HS1 to 1HS7 and three mineral aggregates from 1HF1 to 1HF3, and seven mineral grains from 2HS1 to 2HS7 and three aggregates from 2HF1 to 2HF3 were selected from the 1H and 2H test samples, respectively.

2.2 X-ray diffraction analysis

We have utilized extremely strong X-rays generated from synchrotron radiation for X-ray diffraction analysis, because strong synchrotron X-rays, whose intensity is higher than that of normal X-ray generator at least by 103, induce strong diffraction of individual small mineral grains. X-ray diffraction analysis was performed at the beam line 3A in the photon factory institute of material science, high energy accelerator research organization, using a Gandolfi camera that can obtain a powder X-ray pattern from an individual mineral particle. The X-ray was monochromated to 2.161 +/− 0.002 Å. The wavelength of X-ray was precisely determined using diffraction patterns of a standard Si particle. The monochromated X-ray gives very sharp X-ray reflection peaks with very low background level (Fig. 1), compared with normal X-ray generators whose X-rays are combination of Kα1 and Kα2 lines from particular elements such

![X-ray diffraction pattern of Si crystal exposed to monochromated synchrotron X-ray radiation.](image-url)
as Cr and Cu.

Individual samples (single mineral grains and mineral aggregates) were mounted on a thin glass fiber with 5 μm in diameter with a very small amount of glue and exposed to synchrotron X-ray for 10 to 30 minutes. The exposure duration depends on the diameter of the samples. In the present investigation, the minimum size of the sample is approximately 25 μm (2HS3), but we can obtain a clear X-ray diffraction pattern from a single interplanetary dust particle less than 10 μm (Nakamura et al., 1999). The X-ray photographs were scanned by a high-resolution scanner (Canon FB:200) and the read data were analyzed for integrated intensity and peak center positions by X-ray reflections by the G antic2 software that we have developed.

2.3 EPMA analysis

After X-ray diffraction measurements, the samples of single mineral grains were polished using the micro-diamond paste, and analyzed by an EPMA (JEOL JXA-733 superprobe) equipped with an x-ray length-dispersive X-ray spectrometer (WDS). WDS quantitative analyses were performed at 15 kV accelerating voltage and 10 nA beam current with a focused beam 2 μm in diameter. Quantitative chemical compositions were obtained via the ZAF correction method. Standard minerals and conditions Mineralogy of 100-μg test samples of X-ray acquisition for the analysis of silicate and oxide minerals are summarized in Table 1.

Table 1: Standard minerals and conditions for the analysis of silicate and oxide minerals

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* List of analyzing crystal

2.4 TEM observation

Subsequent to X-ray diffraction analysis, the samples of mineral aggregates were embedded in epoxy resin and microtomed by Leitz-Reichert Super Nova ultramicrotome for TEM.
Table 2: Chemical composition of the individual mineral grains in the 1H test sample determined by EPMA analysis.

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(JEOL JEM-2000FX II) observation. Each sample of the mineral aggregates was composed of fine-grained fragments stuck together by acetic-acid-soluble glue used for X-ray diffraction analysis. Four aggregates, two from 1H and two from 2H test sample, were investigated by TEM. Ultramicrotomy and crushing were applied for TEM sample preparation.

Two mineral aggregates, one from 1H (1HF2) and the other from 2H (2HF3), were processed by ultramicrotomy. They were dipped into a small drop of acetone on a slide glass for disaggregation. The disaggregated grains were then stuck together by epoxy resin, EMbed 812. The gathered grains were located at a corner of a flat micromold in which epoxy resin had been half-filled and that had been cured at 75°C for 6 hrs. Then the micromold was filled by the epoxy resin and cured at 75°C for 48 hrs. The cured micromolds were hand-trimmed and sliced by Leitz Reichert Super Nova ultramicrotome. The thickness of each section was 100 nm.

Another two aggregates, one from each test sample (1HF3 and 2HF1), were processed by crushing. They were dipped into a small drop of acetone on a clean slide glass for disaggregation. Then another clean slide glass was covered and pressed to crush the disaggregated grains. Crushed fragments were sticking on both slide glasses. A small drop of ethyl alcohol was dropped on the crushed fragments. By attaching TEM microgrids to the drops of ethyl alcohol, fragments suspended in the drops were gathered on the microgrids.

Along with high-resolution imaging and electron diffraction analysis, quantitative elemental analysis was performed by Philips DX4 energy-dispersive X-ray spectrometer. Although the analysis was based on Cliff-Lorimer approximation, the effects by absorption were considered by regarding k factors as the function of total counts of X-rays. K factors have been already determined by one of the authors (T. Noguchi) using many mineral standards.
Fig. 2 a and b: A BSE image and X-ray diffraction pattern of 1HS2 (a) and 1HS3 (b). The portions analyzed by an EPMA are shown in the BSE image and the results are listed in Table 2. (a) 1HS2 is forsterite. (b) 1HS3 consists of olivine (ol), glass (gl), and low-Ca pyroxene (px).

3. RESULTS OF ANALYSIS

3.1 1H test sample

Single mineral grains

Back-scattered electron (BSE) images and X-ray diffraction pattern indicated that 1HS1 is an olivine particle with uniform chemical composition at Fo76.5. EPMA analysis showed that it contains 0.5 wt% MnO and less than 0.1 wt% CaO (spot 1–3 in Table 2). 1HS2 is also olivine with a Mg/Fe ratio, MnO and CaO contents similar to 1HS1 (Fig. 2a and spot 4–6 in Table 2). X-ray diffraction data indicated that 1HS3 consists mainly of low-Ca pyroxene and olivine (Fig. 2b). EPMA analysis showed that, along with olivine (spot 13–15 in Table
Fig. 2 c and d: A BSE image and X-ray diffraction pattern of 1HS4 (c) and 1HS7 (d). The portions analyzed by an EPMA are shown in the BSE image and the results are listed in Table 2. (c) 1HS4 is troilite. (d) 1HS7 consists of taenite (tae) and kamacite (kam).

2) and pyroxene (spot 7–9 in Table 2), it contains small amounts of glass that is rich in SiO₂, FeO, and MgO and with appreciable amounts of Al₂O₃, MnO, CaO, Na₂O, Cr₂O₃, and NiO (spot 10–12 in Table 2). The glass has an intermediate concentration of SiO₂, MgO, and FeO between coexisting olivine and low-Ca pyroxene, this may be a melt having been produced at the grain boundary between olivine and pyroxene during shock compression. The glass is not a mesostasis of chondrules, because it is depleted in Al₂O₃ and CaO. Composition of olivine in 1HS3 is almost identical to that of 1HS1 and 1HS2. Low-Ca pyroxene has a relatively uniform composition from En₇₈.₃Wo₂.₀ to En₇₈.₇Wo₃.₆ (spot 7–9 in Table 2). 1HS₄ is a troilite (FeS) particle judging from X-ray diffraction pattern and chemical composition (Fig. 2c and spot 16–17 in Table 2). 1HS₅ is low-Ca pyroxene with compositions ranging Mineralogy of 100-µg
Fig. 3: (a) X-ray diffraction pattern of 1HF1 mineral aggregate. It is composed of olivine (ol), low-Ca pyroxene (px), and troilite (tr). (b) X-ray diffraction pattern of 2HS3, showing that olivine (ol) is dominant and small amounts of Ca-rich pyroxene (px) and troilite (tr) are present.

test samples from En_{78.1}Wo_{17} to En_{78.3}Wo_{20}, which is similar to those of low-Ca pyroxene in 1HS3 (spot 18–20 in Table 2). 1HS6 is an olivine particle having similar composition to 1HS1 (spot 21–22 in Table 2). 1HS7 consists of kamacite with 4–5 wt% Ni and taenite with 34–41 wt% Ni (spot 23–27 in Table 2). X-ray diffraction and EPMA observation confirm the coexistence of kamacite and taenite (Fig. 2d).

**Mineral aggregates**

X-ray diffraction and TEM analysis were performed on the mineral aggregates, but no EPMA analysis was done because size of each mineral in the aggregates is too small. X-ray diffraction analysis of the mineral aggregates 1HF1, 1HF2, and 1HF3 showed that they are virtually similar in mineralogy. They show strong olivine and low-Ca pyroxene reflections and variable intensity of reflections of troilite (Fig. 3a). No reflections from other minerals are recognized, which indicates that the abundance of minerals other than olivine, pyroxene, and troilite are very low if they are present. The relative abundance of olivine, low-Ca pyroxene, and troilite was obtained to be 67, 25, and 8 wt% for 1HF1, 78, 19, and 3 wt% for 1HF2, 61, 36, and 3 wt% for 1HF3, respectively, and the average abundances are 26, 69, and 5 wt%, respectively. Principles and procedures for determination of the relative minerals abundance were given in Nakamura et al. (2001) and also summarized in appendix 1.
Fig. 4a and b: (a) Low-magnification bright field (BF) photomicrograph of an aggregate of olivine and an aggregate of olivine and pentlandite. Holey material shown in this photomicrograph is thin plastic film supporting ultrathin sections. Selected area electron diffraction (SAED) patterns of olivine and coexisting pentlandite are also shown in the insets. (b) BF photomicrograph of clinopyroxene and SAED pattern of the crystal. Crushed sample.

TEM observation revealed that olivine, low-Ca pyroxene, Ca-rich pyroxene, troilite, and pentlandite were constituent of the mineral aggregates of 1H test sample. Olivine was the most abundant phase and low-Ca pyroxene in the form of both orthopyroxene (opx) and clinopyroxene (cpx) was next abundant. Pentlandite is rare. Most olivine grains appear as individual grains or aggregates of olivine. Grain size of olivine ranges from several μm to a few hundred nm. As a rare case, pentlandite coexists with olivine (Fig. 4a). TEM observation of the
boundary between the olivine and pentlandite clearly shows that they are one grain made of two minerals. Figure 4b is a bright field (BF) TEM image of low-Ca clinopyroxene. Subgrain boundaries of polycrystalline twins are shown in the crystal. Figure 4c is a BF image of Ca-rich pyroxene. It does not show characteristic texture such as exsolution lamellae. Ca-rich pyroxene was relatively rare. Figure 4d is a BF image of troilite.

Mg/(Mg+Fe) ratio of fine-grained olivine in the mineral aggregates has relatively wide variation, although there is a peak between 0.5 and 0.6 (Fig. 5a). However, olivine with less
Fig. 5: Distribution of Mg/(Mg+Fe) ratio of olivine in the 1H (a) and 2H (b) test samples. (a) Coarse olivine in 1H is homogeneous, but fine olivine is heterogeneous. (b) Both coarse and fine olivines are heterogeneous in composition.

than 0.45 Mg/(Mg + Fe) ratio contains higher iron and lower silicon than stoichiometry of olivine. Therefore, there is a possibility that highly ferroan olivine in this sample may contain a Fe-bearing phase as fine inclusions. Different from the case of olivine, fine-grained low-Ca pyroxene in the 1H test sample has a narrow range of Mg/(Mg+Fe) ratio between 0.75 0.8 (Fig. 6a). Ca-Mg-Fe atomic ratios indicate that Ca-rich pyroxene is augite (Fig. 6a).

3.2 2H test sample

Single mineral grains

EPMA analysis indicated that 2HS1 is an olivine particle showing a wide range of Fe-Mg zoning from Fo52.2 at the rim to Fo95.8 at the core (Fig. 7a and spot 1–4 in Table 3). MnO concentration is richer at the rim than at the core, while CaO concentration is richer at the core than at the rim. X-ray diffraction pattern indicated that reflections from olivine are tailed to the lower diffraction angle, suggesting that small amounts of FeO-rich olivine are present together with large amounts of Mg-rich olivine. This is consistent with the results of EPMA analysis. 2HS2 is a particle consisting of olivine and low-Ca pyroxene judging from the X-ray
diffraction pattern (Fig. 7b). But EPMA analysis indicated that it consists only of olivine with composition ranging from Fo94.7 to Fo97.2 (spot 5–7 in Table 3). This discrepancy suggests that pyroxene does not come out on the polished surface and must be present somewhere in the 2HS2. 2HS3 is olivine with Fo94.4 (spot 8 in Table 3), which is consistent with the results of X-ray diffraction analysis. Mineralogy of 100-μg test samples 2H84 is a mixture of almost pure anorthite, forsterite, and spinel (Fig. 7c). Chemical compositions of anorthite (spot 9–11 in Table 3) and spinel (spot 15–16 in Table 3) are similar to those found in CAI in carbonaceous chondrites (e.g., El Goresy et al., 1985; McGuire and Hashimoto, 1989). X-ray diffraction analysis showed that forsterite and anorthite are the major constituent and spinel is minor one in 2HS4 (Fig. 7c). X-ray diffraction data showed that 2HS5 consists of olivine and low-Ca pyroxene, but only olivine was found by EPMA analysis (spot 17–19 in Table 3). Pyroxene does not appear on the polished surface, like that in 2HS2. Olivine and pyrrhotite are present in 2HS6 based on the results of EPMA and X-ray diffraction analysis (spot 20–22 in Table 3), whereas pentlandite is dominant in 2HS7 (Fig. 7d and spot 23–24 in Table 3).

**Mineral aggregates**

Like mineral aggregates in the 1H sample, those of the 2H sample were also investigated by X-ray diffraction and TEM. X-ray diffraction patterns of 2HF1, 2HF2, and 2HF3 are basically similar to one another. They showed that olivine is the dominant mineral in the mineral aggregates in the 2H test sample (Fig. 3b). Minor amounts of Ca-rich pyroxene are also recognized (Fig. 3b). Troilitle rarely occurs. The relative abundance of olivine and Ca-rich pyroxene is 92 and 8 wt% for 2HF1, 97 and 3 wt% for 2HF2, and 91 and 9 wt% for 2HF3, respectively. This differs from the 1H sample in lower abundance of pyroxene and troilitle. Reflections from
olivine 2HF1 are often doublet, indicating that olivine consists of two populations with different Mg/Fe ratio.

TEM investigation of the mineral aggregates having been analyzed by X-ray diffraction showed that they contain olivine, low-Ca opx and cpx, Ca-rich pyroxene, plagioclase, chromite, and troilite. In the two mineralogical aggregates studied, the most abundant phase was olivine. Chromite and plagioclase were very rare. Figure 8a is a BF image of olivine. Grain size of olivine ranges from several μm to several hundred nm in diameters. Low-Ca pyroxene in this specimen was rarer than that in the 1H specimen. Figure 8b is a low-magnification BF image of sodic plagioclase (Ab82) contacting with Ca-rich pyroxene. Plagioclase showed twinning. Ca-rich pyroxene in this specimen usually appeared as discrete grains. Only one euhedral chromite grain was found (Fig. 8c). It is chromite with composition YCr=0.88 and Mg/(Mg+Fe)=0.13. Troilite was found, but pentlandite was not (Fig. 8d).

Mg/(Mg+Fe) ratio of fine-grained olivine has a sharp peak around 0.5 (Fig. 5b). In addition to this peak, there is another small peak around 0.7 0.8. Ca-Mg-Fe atomic ratios of pyroxenes (Fig. 6b) indicates that Ca-rich pyroxene was sarite. Different from the case of olivine, fine-grained low-Ca has a narrow range of Mg/(Mg+Fe) ratio between 0.75 0.8 (Fig. 6b).
A series of mineralogical analysis of the test samples showed that both 1H and 2H samples are silicate-rich material with minor amounts of FeNi-metal, FeS- and FeNiS-sulfide. The silicates are mostly olivine and pyroxene and rarely plagioclase. The mineral combination of the 1H and 2H samples suggests that they are similar to stony meteorites, especially chondrites. The presence of abundant coarse olivine and low-Ca pyroxene with homogeneous composition at Fe$_{77}$ and En$_{78}$, respectively, suggests that the coarse silicate grains in the 1H sample are similar to those in equilibrated L chondrites. But heterogeneous Mg/Fe ratio in fine-grained olivine and the presence of pentlandite coexisted with Fe-rich olivine was observed. This sig-
nature cannot be observed in equilibrated chondrites, because fine olivine grains are the first to reach equilibrium in Fe-Mg interdiffusion. Therefore, mineralogy of the 1H sample appears to be inconsistent with those of pulverized material of a single equilibrated L chondrite. On the other hand, the presence of Mg-rich coarse olivine grains with composition of almost pure forsterite, Fe-Mg zoning in the coarse olivine, fine-grained Fe-rich olivine that are unequilibrated with the coarse olivine, Ca-rich pyroxene with sarite composition, and a parts of CAI containing anorthite and spinel suggests that mineralogy of the 2H test sample is similar to that of anhydrous carbonaceous chondrite with low petrologic type. Mineralogy of 100-μg test samples.

Fig. 7 c and d: A BSE image and X-ray diffraction pattern of 2HS4 (c) and 2HS7 (d). The portions analyzed by an EPMA are shown in the BSE image and the results are listed in Table 3. (c) 2HS4 consists of olivine (ol), anorthite (pl) and spinel (sp). In both BSE image and diffraction pattern, the three minerals are observed. (d) 2HS7 is pentlandite.
Fig. 8 a and b: (a) BF photomicrograph of olivine and SAED pattern of the crystal. Ultramicrotomed sample. (b) BF photomicrograph of orthopyroxene and SAED pattern of the crystal. Ultramicrotomed sample.
Fig. 8 c and d: (c) Low-magnification BF photomicrograph of sodic plagioclase and coexisting Ca-rich pyroxene. SAED patterns of these crystals are also shown in the insets. Ultramicrotomed sample. (d) BF photomicrograph of chromite and SAED pattern of the crystal. Ultramicrotomed sample.
REFERENCES


Mineralogy of 100-μg test samples

APPENDIX

Determination of the relative mineral abundance from X-ray diffraction data

Our X-ray diffraction analysis showed that fine-grained mineral aggregates in the 1H test sample consist mainly of olivine, low-Ca pyroxene, and troilite. Relative abundance of the three minerals was determined from the integrated intensities of particular X-ray reflections: olivine (130) reflection at about 2.77 Å, a reflection at about 2.88 Å for low-Ca pyroxene, and a reflection at 2.65 Å for troilite. The integrated intensity of 2.65 Å reflection is defined as that from troilite. The olivine (130) reflection at about 2.77 Å is a single reflection with no overlaps from other two minerals, thus we define the integrated intensity of the reflection at 2.77 Å as that from olivine. Low-Ca pyroxene occurs as opx and cpx. Both opx and cpx give strong reflections at 2.88 and 3.16 Å, but they differ in an intensity ratio between the two reflections. Thus, an opx/cpx ratio in individual micrometeorites can be estimated from the intensity ratio of reflections at 2.88 and 3.16 Å. The reflections at 2.88 Å include opx (610), cpx (310), and cpx (211), while the reflections at 3.16 Å include opx (420), opx (221), and cpx (220). Relative intensities of these reflections from the same weight of low-Ca opx and cpx are calculated, using the structural parameters of opx (Morimoto and Koto, 1969) and cpx (Morimoto et al., 1960), where Fe in both pyroxenes is distributed between M1 and M2 sites with the site preference K = 0.1 (Hafner and Warburton, 1971; Virgo and Hafner, 1969). Integrated intensities of low-Ca opx and cpx in individual micrometeorites are obtained by partitioning intensity of reflection
at 2.88 Å using the relative abundance of opx and opx.

Through the process stated above, the integrated intensity of the particular X-ray reflections from olivine, low-Ca pyroxene, and troilite are determined for individual mineral aggregates. To convert the integrated X-ray intensity into relative mineral abundance, the relative intensity of the three minerals at a constant weight needs to be known. For this purpose, three particles with approximately 100 μm in diameter that are mixtures of equal weights of fine-grained olivine (Fo100), low-Ca opx (En100), and troilite are prepared as standard samples. The X-ray integrated intensities were determined in the same way as applied for mineral aggregate samples and the relative X-ray intensity of the three minerals at a constant weight is obtained by averaging the intensities derived from the three standard particles.

Between the standard particles and the mineral aggregate samples, there are two mineralogical and compositional differences that need to be corrected: (1) the standard particles contain only low-Ca opx, while the mineral aggregate samples contain both opx and cpx, and (2) the average Fo# and En# are variable in the mineral aggregate samples, whereas those of the standard particles are Fo100 and En100, respectively. For the difference (1), intensity of cpx was converted to that of opx with the same weight and En#, using an intensity ratio of opx to cpx at 2.88 Å and then the total intensity of low-Ca pyroxene in the mineral aggregates samples was obtained. For the difference (2), in the case of olivine and low-Ca pyroxene the X-ray intensity at a constant weight changes with Mg/Fe ratios. Therefore in order to compare X-ray intensities between the mineral aggregate samples and the standard particles, the X-ray intensities of olivine and low-Ca pyroxene in the mineral aggregate samples, having various Fo# and En#, need to be converted to the intensities of Fo100 and En100, respectively, with the same weights. For this reason, the relationship between X-ray intensity and Fe/Mg ratio at a constant weight was calculated for olivine and low-Ca opx, using the structural parameters given in Brown (1982) for olivine and those in Morimoto and Koto (1969) for low-Ca opx. In the calculation, Fe in olivine prefers M2 site and that in low-Ca opx is distributed between M1 and M2 sites with the site preference K = 0.1 (e.g., Virgo and Hafner, 1969).

On the other hand, average Fo# and En# in the mineral aggregate samples need to be determined. It is known that the cell dimension of olivine and pyroxene increases with increasing site occupancy of large cations such as Fe²⁺ (e.g., Yoder and Sahama, 1957). For olivine, the (130) spacing at about 2.77 Å can be converted to Fo# using an equation given in Shinno (1980). For low-Ca pyroxene, a relationship between En# and the (610) spacing of low-Ca opx was calculated using the cell parameter given in Brown (1967). In calculation two assumptions were made: (1) low-Ca opx contains little amounts of Al, and (2) Mineralogy of 100-μg test samples the interlayer spacing of low-Ca opx (610) is identical with that of low-Ca cpx (310). From the estimated Fo# and En# in the mineral aggregate samples, the intensity of olivine and low-Ca pyroxene converted to those of Fo100 and En100, respectively, was obtained. Then, the relative mineral abundance of olivine, low-Ca pyroxene, and troilite was determined by comparing X-ray intensity of each mineral in the mineral aggregates of the 1H test samples with that of the standard particles. Uncertainty of the relative abundance of each mineral is estimated to be less than 15%. The uncertainty is mostly come from that of pyroxene abundance due to some assumptions we made, as stated above.

For the mineral aggregates of the 2H test sample, we used standard particles containing equal amounts of olivine and Ca-rich cpx. The way of obtaining the relative abundance between olivine and Ca-rich pyroxene in the mineral aggregates was the same as that applied for the 1H test sample. Mineralogy of 100-μg test samples.