

MAGNETIC STRUCTURE OF FRAMBOIDAL MAGNETITE IN RETURNED SAMPLE FROM ASTEROID RYUGU. Yuki Kimura¹, Takeharu Kato², Satoshi Anada², Ryuji Yoshida², Kazuo Yamamoto², Toshiaki Tanigaki³, Tetsuya Akashi³, Hiroto Kasai³, Tomoki Nakamura⁴, Masahiko Sato⁵, Tomoyo Morita⁴, Mizuha Kikuri⁴, Kana Amano⁴, Eiichi Kagawa⁴, Hisayoshi Yurimoto⁶, Takaaki Noguchi⁷, Ryuji Okazaki⁸, Hikaru Yabuta⁹, Hiroshi Naraoka⁸, Kanako Sakamoto¹⁰, Sei-ichiro Watanabe¹¹, Yuichi Tsuda¹⁰, and Shogo Tachibana^{5,10}. ¹Hokkaido University, Sapporo 060-0819, Japan (ykimura@lowtem.hokudai.ac.jp), ²Japan Fine Ceramics Center, Nagoya, 456-8587, Japan, ³Research & Development Group, Hitachi, Ltd., Hatoyama, Saitama, 350-0395, Japan, ⁴Tohoku University, Sendai 980-8578, Japan, ⁵The University of Tokyo, Tokyo 113-0033, Japan, ⁶Hokkaido University, Sapporo 060-0810, ⁷Japan, Kyoto University, Kyoto 606-8502, Japan, ⁸Kyushu University, Fukuoka 819-0395, Japan, ⁹Hiroshima University, Higashi-Hiroshima 739-8526, Japan, ¹⁰ISAS/JAXA, Sagami-hara 252-5210, Japan, ¹¹Nagoya University, Nagoya 464-8601, Japan.

Introduction: Magnetite is one of major aqueous alteration products and should be one of major reservoirs of magnetic remanence in carbonaceous chondrite. Their unique morphology such as framboid and plaquette, and characteristic magnetic property were results of their experienced processes and environments mainly in formation and possibly in oxidation/reduction, heating, shock and so on. Based on magnetic domain structure of framboidal magnetites in the Tagish Lake meteorite, we have proposed the formation process, place and timing in the early Solar System using electron holography [1,2]. To understand a part of the big picture in the early solar system, here, we applied electron holography to the returned sample from asteroid (162173) Ryugu and the nanoscale magnetic domain structures of the framboidal magnetites were observed.

Sample treatments: Ryugu samples were mounted on an indium plate in the glove box at Tohoku University. The indium plate was supported by silicon plate with less than $3 \times 3 \text{ mm}^2$ for easier handling. The samples were packed filled with N_2 gas and bring to a glove box at Japan Fine Ceramics Center for the analysis. The glove box has a capability to maintain the dew temperature of $\sim -80^\circ\text{C}$ in Ar gas. During the analysis, all the samples were stored in the glove box. A part of the sample was transferred to Hitachi Ltd. for electron holography using a transmission electron microscope (TEM) with the higher acceleration voltage.

Initially, we tried to work without carbon deposition in order to maintain the original condition as much as possible. However, the electrical charging was large and the first sample was flew off during the evacuation of a focused ion beam (FIB; Fig. 1). Therefore, all the samples on an indium plate was coated by carbon (30 nm in thickness) using a carbon coater (PECS II, Gatan Inc.). Specimen is not heated by the carbon coating, which does not affect to the magnetic domain structure and remanence of the magnetite.

All analysis was conducted in negligibly weak magnetic field conditions less than $50 \text{ } \mu\text{T}$ from receiv-

ing the sample on JAXA/ISAS curation facility to the electron holography. Later, scanning TEM-energy

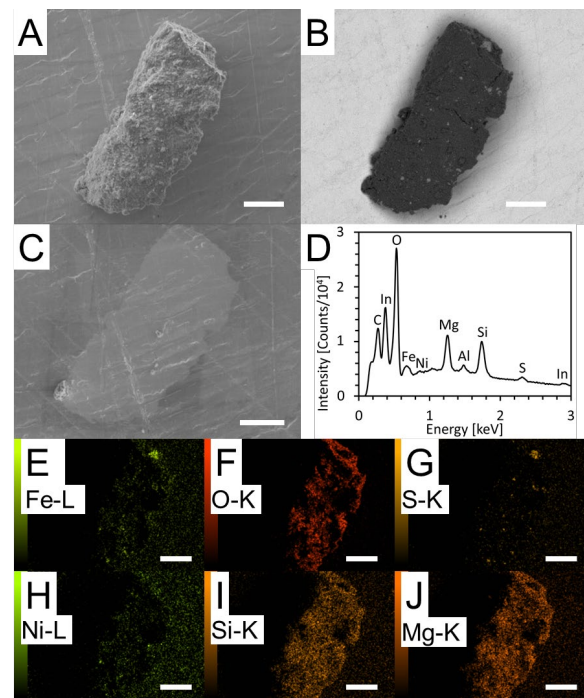


Fig. 1. An example of a fragment of the returned sample named A0064-FO010 from the asteroid Ryugu. A0064 is a grain collected at the first touchdown site. All data was collected by a SEM (JCM-7000, NeoScope, JEOL) with an acceleration voltage of 5 kV. Scale bars are $20 \text{ } \mu\text{m}$. **A.** Secondary electron image. **B.** Back scatter electron image. **C.** Secondary electron image after flew off the sample from the indium plate during the evacuation of a FIB. **D.** EDS spectrum of whole the view area in A using an EDS analysis system (JED-2300 Analysis Station Plus) installed in the SEM. The atomic ratio of iron, oxygen, sulfur, nickel, silicon, magnesium, aluminum and carbon are 2.9, 51.2, 3.4, 0.7, 15.9, 11.6, 2.1 and 12.2, respectively. **E-J.** SEM-EDS mapping data corresponding to iron, oxygen, sulfur, nickel, silicon and magnesium, respectively.

dispersive X-ray spectroscopy (EDS) elemental mappings, electron diffraction patterns, and electron energy-loss spectroscopy (EELS) were obtained in normal magnetic environments in these analytical methods.

All sample preparations and analyses were performed without exposure to air, except for sample loading and removal into the scanning electron microscope (SEM). The total duration for exposure to the air is less than 15 s. These samples were still stored in the glove box at this moment.

Sample selection and preparation of thin section for electron holography: The samples were observed using a SEM equipped with an EDS detector (JCM-7000, NeoScope, JEOL). To preserve a magnetic field in the environment of the sample stage as weak as possible, the acceleration voltage was maintained at 5 kV. In addition to magnetite framboid, individual magnetite particles with similar size and morphology with the

magnetite framboid were distributed on the surface of fragments of the returned sample in SEM images as shown in Fig. 1. Based on the SEM-EDS elemental mapping, framboids composed of spherical particles with iron and oxygen were selected and thin sections were prepared at -90°C and mounted on a standard TEM grid using cryo-FIB (NB5000, Hitachi High-Tech). The thin section was coated by carbon with 30 nm thickness in each face.

Figure 2A shows an example of a thin section of magnetites with diameters of $\sim 1\ \mu\text{m}$ in a magnetite framboid. SEM-EDS and STEM-EDS elemental mappings and electron diffraction patterns of the particles were obtained after observation by electron holography and confirmed that the spherical particles corresponded to magnetite [3]. Matrix surrounding the magnetites was phyllosilicate composed of mainly silicon and magnesium.

Electron holography: The thin section was loaded into the electron-holography TEM (HF3300-EH, Hitachi High-Tech) with an acceleration voltage of 300 kV. The magnetic field of the sample stage in the TEM is less than $\sim 17\ \mu\text{T}$.

Five images of holograms were taken with an exposure time of 20 s in each. Phase images reconstructed by the five images were averaged for precise visualization of the magnetic domains and flux distributions. The reconstructed image of electron wave passing through the sample has a both information of magnetic flux and inner potential of the sample. To subtract the internal potential, the TEM sample was rotated 180° and another series of holograms were recorded from the other side of the thin section [1].

Figure 2B is the resulting image showing nanometer-scale magnetic domain structure and magnetic flux distribution of the framboidal magnetites corresponding to Fig. 2A with a spatial resolution of 14 nm. All magnetites had vortex magnetic structures and a part of the magnetic flux leaked out of the particles. External magnetic flux is detectable as remanent magnetization in traditional macroscopic measurements. Therefore, the remanences of framboidal magnetites having vortex magnetic domain states contribute significantly to the natural remanent magnetization of asteroid Ryugu.

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References: [1] Kimura, Y. et al. (1997) *Nature Communications* 4, 2649. [2] Kimura, Y. et al. (2021) *The Astrophysical Journal Letters*, 917, L5. [3] Nakamura, T. et al. (2022) *Science*, Submitted.

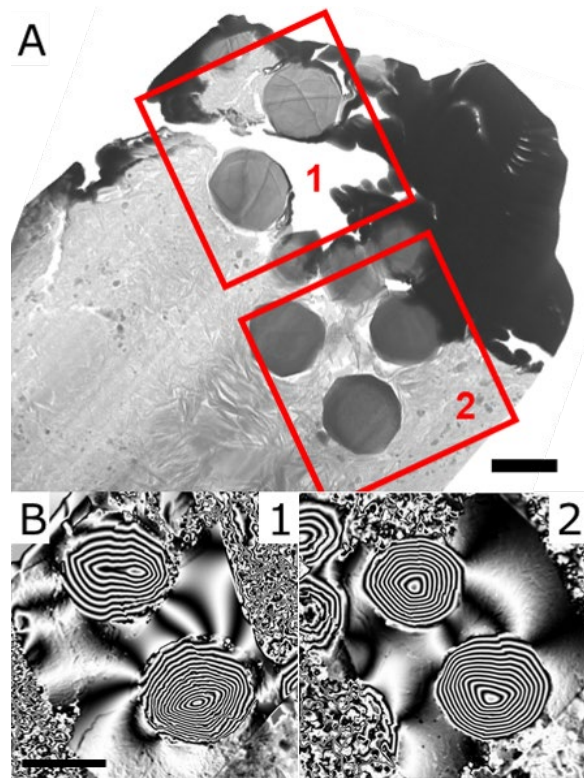


Fig. 2. Typical framboidal magnetite and its magnetic structure. A. TEM image of a thin section of a fragment from the sample A0064-FO007 prepared by FIB. Strong contrast (black) region is tungsten layer deposited to prevent beam damage during FIB thinning. B. Magnetic flux distribution image of remanent magnetic state of magnetites in A observed by electron holography. Half times phase-amplified for 2. Each particle has a concentric circular magnetic field (vortex structure). Scale bars are $1\ \mu\text{m}$.