



## Oxygen Isotopic Compositions of Asteroidal Materials Returned from Itokawa by the Hayabusa Mission

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greatly between particles (fig. S7), which is typical of moderately shocked astromaterial corresponding shock stages up to S4 (6, 26).

MUSES-C Regio probably formed by segregation and accumulation of fine gravel into areas close to the gravitational center of Itokawa due to global-scale electrostatic grain levitation, vibration-induced granular migration, and deposition of slow moving ejecta launched from surface impacts (27–29). Therefore, particles in MUSES-C Regio originally derived from diverse regions of Itokawa. Fortunately, despite the small mass of the recovered Itokawa samples, they record the critical steps in the history of this asteroid. Itokawa was classified as an S-type asteroid from terrestrial remote sensing, and it has been commonly suggested that S-type asteroids, the most abundant asteroids in the inner asteroid belt, are the parent bodies of ordinary chondrites. Our petrologic data from MUSES-C Regio confirm that Itokawa is indeed an ordinary chondrite (LL4 to LL6), thereby finally linking these asteroids and meteorites.

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#### Supporting Online Material

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Figs. S1 to S8  
Tables S1 to S5  
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## Oxygen Isotopic Compositions of Asteroidal Materials Returned from Itokawa by the Hayabusa Mission

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Meteorite studies suggest that each solar system object has a unique oxygen isotopic composition. Chondrites, the most primitive of meteorites, have been believed to be derived from asteroids, but oxygen isotopic compositions of asteroids themselves have not been established. We measured, using secondary ion mass spectrometry, oxygen isotopic compositions of rock particles from asteroid 25143 Itokawa returned by the Hayabusa spacecraft. Compositions of the particles are depleted in <sup>16</sup>O relative to terrestrial materials and indicate that Itokawa, an S-type asteroid, is one of the sources of the LL or L group of equilibrated ordinary chondrites. This is a direct oxygen-isotope link between chondrites and their parent asteroid.

**M**ineral compositions of asteroids are inferred from visible and near-infrared reflectance spectroscopy. The spectroscopic similarity between some asteroids and meteorites suggests that meteorites come from asteroids and allows indirect assessments of asteroid-meteorite connections and inferences regarding chemical compositions of asteroids (1). Of the ~40,000 meteorites we know of, only 14 have had their pre-impact orbits ascertained (2). The aphelia of these 14 orbits are located within the Main Asteroid Belt between Martian and Jovian orbits, which is consistent with an asteroidal origin. However, even the parent

asteroids of these 14 meteorites have not been identified.

The taxonomy of meteorites largely has been based on the whole-rock chemical and oxygen isotopic compositions. Each meteorite group, and probably each planet, has a characteristic chemical composition and a unique oxygen isotopic composition (3, 4). The origin of oxygen isotopic variations in the solar system is thought to be an isotope-selective photodissociation of carbon monoxide that occurred before planet formation (5–7). The unique oxygen isotopic composition of a planet is thought to be produced by a combination of gas-dust chemistry and accretion

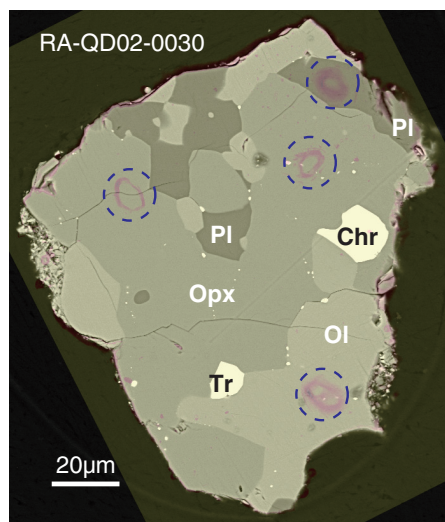
physics in the solar nebula (6, 8). The Earth and the Moon—the only bodies for which we have measurements—have similar oxygen isotopic compositions within an uncertainty of ±0.016 per mil (‰) [2 SD (2σ)] (9, 10). The determination of an oxygen isotopic composition of an asteroid or a planet therefore would provide an indisputable means to clarify mechanisms of planet formation in the solar nebula and to connect an asteroid or a planet to a specific meteorite group.

The Hayabusa spacecraft made two touchdowns on the surface of asteroid 25143 Itokawa on 20 and 26 November 2005 JST and successfully collected grain particles from the surface of the asteroid. Itokawa is classified as an S-type asteroid. As inferred from reflectance spectrometry, it consists of materials similar to primitive

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achondrites or ordinary chondrites (11), which can be distinguished by their oxygen isotopic compositions (4). Previous near-infrared reflectance



**Fig. 1.** Measurement spots for oxygen isotope analysis. An optical microscope image after measurements is superimposed on the backscattered electron image before measurements. Primary ion beam craters are indicated with dashed circles. The spatial resolution ( $\sim 10 \mu\text{m}$ ) was sufficient to measure an object with coexisting minerals and to allow analyses free of contamination from the respective minerals. Ol, olivine; Opx, orthopyroxene; Pl, plagioclase; Chr, chromite; Tr, troilite.

spectroscopy by Hayabusa suggests that the asteroid's surface has an olivine-rich mineral assemblage that is potentially similar to that of LL5 or LL6 chondrites, with different degrees of space weathering (12). The major mineral assemblage of the sample grains collected by Hayabusa is olivine, pyroxene, plagioclase, iron sulfide, and iron-nickel metal (13). The grain sizes are less than  $150 \mu\text{m}$  (mostly less than several tens of micrometers), and crystal sizes in the grain are less than  $80 \mu\text{m}$  (mostly less than  $20 \mu\text{m}$ ) (14).

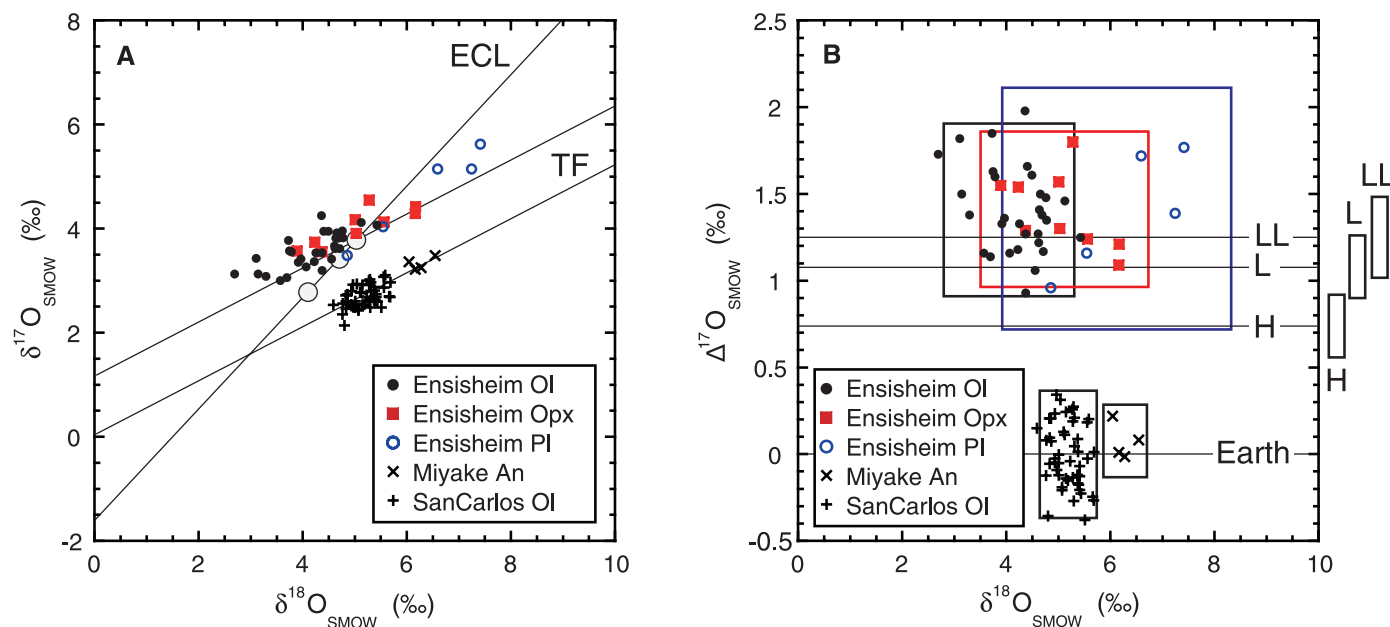
We used the Hokudai isotope microscope system (15) to determine oxygen isotopic compositions of minerals in 28 of these grains, corresponding to measurements of 19 olivine crystals, 7 orthopyroxene crystals, and 7 plagioclase crystals (table S4). The results include multiphase measurements within a grain: analysis for the coexisting olivine-orthopyroxene-plagioclase system in grains RA-QD02-0010 and RA-QD02-0030 and analysis for the coexisting olivine-plagioclase system in grain RA-QD02-0031 (Fig. 1).

The analytical uncertainty was determined from oxygen isotope measurements of an ordinary chondrite, Ensisheim LL6. It is  $\pm 0.7\text{‰}$  ( $2\sigma$ ) for  $\delta^{17}\text{O}_{\text{SMOW}}$ ,  $\pm 1.5\text{‰}$  ( $2\sigma$ ) for  $\delta^{18}\text{O}_{\text{SMOW}}$  for olivine and orthopyroxene, and twice that for plagioclase, where SMOW is standard mean ocean water. The precision of  $\Delta^{17}\text{O}_{\text{SMOW}}$  is  $\sim \pm 0.5\text{‰}$  ( $2\sigma$ ) for all analyses [Fig. 2 and supporting online material (SOM) text]. This precision is sufficient to distinguish most meteoritic

materials known to date from terrestrial materials. However, uncertainties of  $\delta^{17}\text{O}_{\text{SMOW}}$  and  $\delta^{18}\text{O}_{\text{SMOW}}$  are too high to allow a precise determination of metamorphic temperatures by means of the isotopic fractionation among the minerals. Nevertheless, the mineralogical order of isotopic equilibration by thermal metamorphism on the parent body could be recognized within the measurement uncertainties. Thus, the analytical uncertainties could be applied to the Itokawa grains.

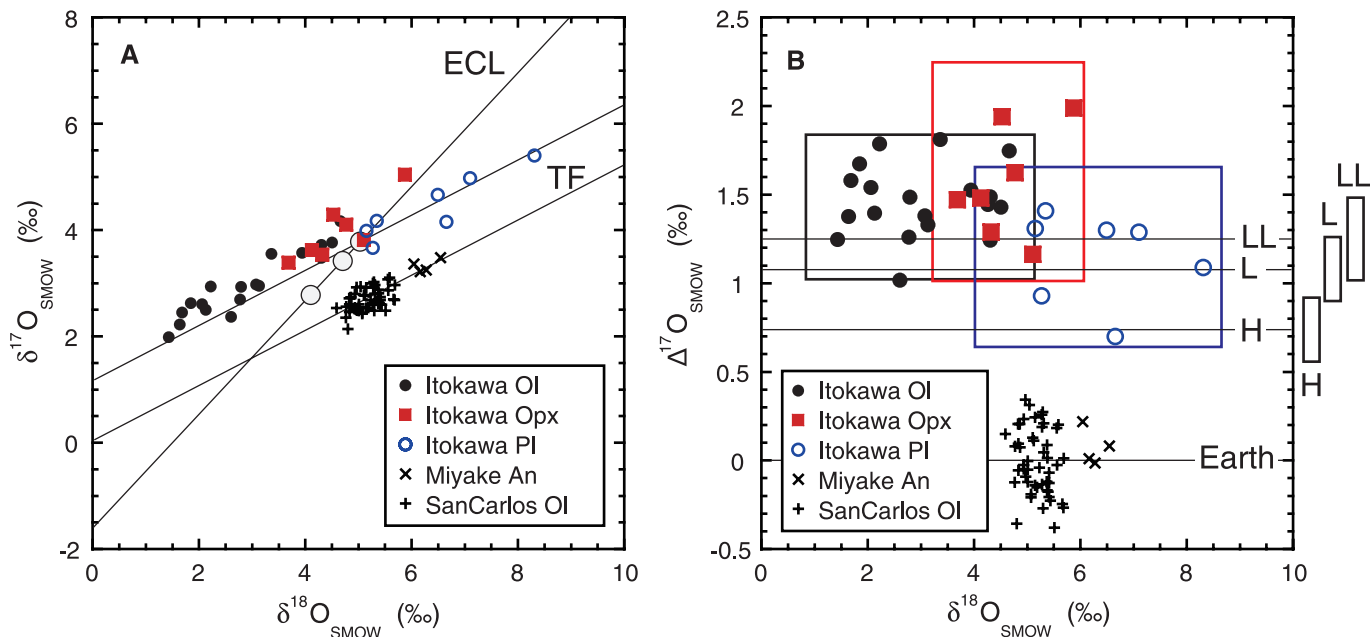
The variations of  $\Delta^{17}\text{O}_{\text{SMOW}}$  for Itokawa minerals are about  $\pm 0.5\text{‰}$  ( $2\sigma$ ) (table S4), which are equivalent to the dispersion expected from measurement uncertainties. All oxygen isotopic compositions of the minerals from Hayabusa sample return capsule plot on the upper side of terrestrial standards on a three-isotope oxygen diagram and are distributed parallel to the terrestrial mass fractionation line (Fig. 3). This indicates that the grains returned by Hayabusa are not terrestrial materials and further demonstrates that the spacecraft retrieved asteroid Itokawa's surface materials during touchdown.

Isotopic compositions of meteorites occupy distinct regions of the oxygen three-isotope diagram according to meteorite group. The region of the Itokawa grains overlaps with those of the ordinary chondrites (16). Ordinary chondrites are subdivided into H, L, and LL chondrites. These groups also have distinct ranges of whole-rock oxygen isotopic compositions, with magnitudes of departure from the terrestrial fractionation



**Fig. 2.** Oxygen isotopic compositions of Ensisheim minerals (A and B) compared with those of a forsterite crystal from San Carlos, Arizona, USA and an anorthite crystal from Miyake-jima, Japan. Instrumental mass fractionation for each mineral is corrected by use of the reference value shown in table S3. Isotope variation defined by  $2\sigma$  for each mineral phase is shown by a rectangle with a color of the corresponding symbol. Open circles on the ECL (equilibrated chondrite line) correspond to average O isotopic compositions

of ordinary chondrites, LL, L, H, from top to bottom. TF, terrestrial fractionation line; Ol, olivine; Opx, orthopyroxene; Pl, plagioclase; An, anorthite; Miyake, Miyake-jima. Data are from tables S2 and S3.  $\Delta^{17}\text{O}_{\text{SMOW}} = \delta^{17}\text{O}_{\text{SMOW}} - 0.52 \delta^{18}\text{O}_{\text{SMOW}}$ . A mass fractionation line of the average O isotopic composition of LL chondrite group is shown as a reference. Variations ( $2\sigma$ ) of whole-rock  $\Delta^{17}\text{O}_{\text{SMOW}}$  values for H, L, and LL chondrite groups are shown to the right of (B).



**Fig. 3.** Oxygen isotopic compositions of Itokawa minerals (**A** and **B**) compared to those of a forsterite crystal from San Carlos, Arizona, USA and an anorthite crystal from Miyake-jima, Japan. Isotope variation defined by  $2\sigma$  for each mineral is shown by a rectangle with a color of the corresponding symbol. Open circles on the ECL correspond to average O isotopic compositions of ordinary chondrites, LL,

L, H, from top to bottom. TF, terrestrial fractionation line; Ol, olivine; Opx, orthopyroxene; Pl, plagioclase; An, anorthite; Miyake, Miyake-jima. Data are from tables S2 and S4. A mass fractionation line of the average O isotopic composition of LL chondrite group is shown as a reference. Variations ( $2\sigma$ ) of whole-rock  $\Delta^{17}\text{O}_{\text{SMOW}}$  values for H, L, and LL chondrite groups are shown to the right of (**B**).

line,  $\Delta^{17}\text{O}_{\text{SMOW}}$ , being  $0.73 \pm 0.18\text{‰}$  ( $2\sigma$ ) for H-chondrite group,  $1.07 \pm 0.18\text{‰}$  ( $2\sigma$ ) for L-chondrite group, and  $1.26 \pm 0.24\text{‰}$  ( $2\sigma$ ) for LL-chondrite group (17). The ranges of  $\Delta^{17}\text{O}_{\text{SMOW}}$  of L and LL group overlap each other, but compositions from the H group are distinct from the other groups.

Unequilibrated chondrites consist of minerals having highly variable  $\Delta^{17}\text{O}_{\text{SMOW}}$  values. Two mechanisms can homogenize the  $\Delta^{17}\text{O}_{\text{SMOW}}$  among minerals: metamorphism and melting (18). Minerals of equilibrated chondrites become homogenized to a  $\Delta^{17}\text{O}_{\text{SMOW}}$  value by metamorphism toward the whole-rock oxygen isotopic composition, with variability decreasing in the order of metamorphic grades from type 4 to 6.

The  $\Delta^{17}\text{O}_{\text{SMOW}}$  values for Itokawa are  $1.46 \pm 0.41\text{‰}$  ( $2\sigma$ ) for olivine,  $1.57 \pm 0.62\text{‰}$  ( $2\sigma$ ) for orthopyroxene, and  $1.15 \pm 0.51\text{‰}$  ( $2\sigma$ ) for plagioclase (table S4). The observed variations among the minerals are within analytical uncertainties of our measurements. The mean  $\Delta^{17}\text{O}_{\text{SMOW}}$  for minerals from Itokawa,  $1.39 \pm 0.36\text{‰}$  ( $2\sigma$ ), coincides with that of LL or L chondrite groups but is clearly distinguished from H chondrites (Fig. 3B). The small variation of  $\Delta^{17}\text{O}_{\text{SMOW}}$  demonstrates that the Itokawa minerals were equilibrated during metamorphism.

The variations of  $\delta^{18}\text{O}_{\text{SMOW}}$  of orthopyroxene and plagioclase from Itokawa are similar to those measured from the Ensisheim LL6 chondrite. The range of variation in  $\delta^{18}\text{O}_{\text{SMOW}}$  of Itokawa olivine is greater than that of Ensisheim

olivine and is as large as those of Itokawa plagioclase. The large variation for Itokawa olivine could be attributed to instrumental mass fractionation relating to irregularities of the sample surface owing to the small size of the grains. Nevertheless, the isotopic relationship among olivine, orthopyroxene, and plagioclase shows that the oxygen isotopes fractionated under equilibrium between coexisting phases. Degrees of the isotopic fractionation among minerals are slightly larger in Itokawa materials than in Ensisheim. The larger isotopic fractionation among the minerals may indicate that the metamorphic temperature was lower in Itokawa material than in Ensisheim.

The metamorphic temperature would be determined by means of the oxygen isotopic fractionation among minerals. The plagioclase-olivine, orthopyroxene-olivine, and plagioclase-orthopyroxene temperatures are calculated to be 600, 650, and 720°C, respectively, through application of an oxygen isotope thermometer (19). The estimated temperatures from 600 to 720°C for Itokawa are lower than those for LL6, L6, and L5 chondrites and higher than for a L4 chondrite (16).

On the basis of this equilibration and the small variation of  $\Delta^{17}\text{O}_{\text{SMOW}}$ , the petrographic type of Itokawa is equivalent to type 4–6 in the LL or L chondrite group. The Itokawa material is compatible with an LL4–6 chondrite classification if we combine the oxygen isotope data with the results of the chemical compositions of minerals (13).

The oxygen isotopic composition of asteroid Itokawa thus provides unequivocal evidence that ordinary chondrites come from S-type asteroids.

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14. All samples collected by the Hayabusa spacecraft and analyzed here have been characterized by means of x-ray microtomography, x-ray diffraction analysis, x-ray fluorescence analysis, scanning electron microscopy, and electron probe microanalysis before isotope measurements were made (13). For our analysis, chemically equilibrated grains were prepared. Chemically less equilibrated grains described in (13) have not been included because a precise chemical characterization was still in process, and they were not ready for this study. The less equilibrated grains were not common in



- the Hayabusa sample return capsule (23). We mounted each grain at the center of an epoxy disk and polished the surface according to the processes established for the preliminary examination. We coated a thin layer of gold with a thickness of 60 nm on the samples for secondary ion mass spectrometry.
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### Supporting Online Material

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Materials and Methods  
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Tables S1 to S4  
Reference (20)

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## Neutron Activation Analysis of a Particle Returned from Asteroid Itokawa

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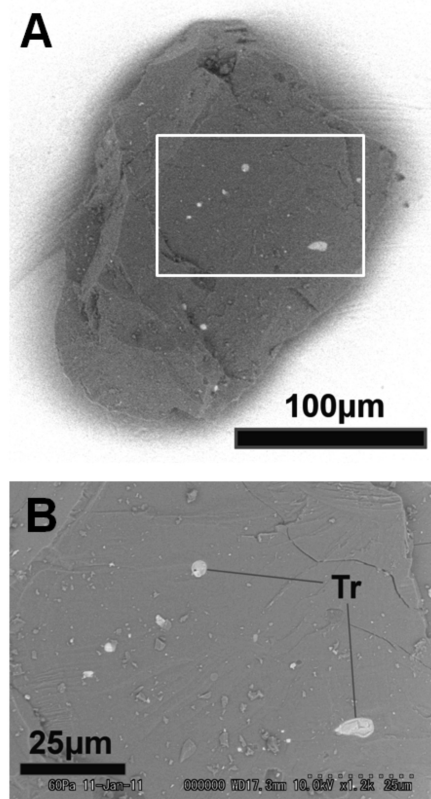
A single grain (~3 micrograms) returned by the Hayabusa spacecraft was analyzed by neutron activation analysis. This grain is mainly composed of olivine with minor amounts of plagioclase, troilite, and metal. Our results establish that the Itokawa sample has similar chemical characteristics (iron/scandium and nickel/cobalt ratios) to chondrites, confirming that this grain is extraterrestrial in origin and has primitive chemical compositions. Estimated iridium/nickel and iridium/cobalt ratios for metal in the Itokawa samples are about five times lower than CI carbonaceous chondrite values. A similar depletion of iridium was observed in chondrule metals of ordinary chondrites. These metals must have condensed from the nebular where refractory siderophile elements already condensed and were segregated.

The Hayabusa spacecraft was launched on 9 May 2003 and reached asteroid 25143 Itokawa in September 2005 (1). After accomplishing numerous scientific observations (2, 3), the spacecraft tried to collect surface material from Itokawa by touching down to the asteroid in November 2005 (4). The spacecraft then navigated back to Earth. Despite encountering several difficulties, Hayabusa finally returned to Earth on 12 June 2010, and its entry capsule was successfully recovered. Although the sample collection was not nominally performed, it was hoped that some extraterrestrial material was stored in the capsule. After careful and extensive examination, more than 1500 particles were recognized by microscopes, most of which were eventually judged to be extraterrestrial, probably originating from Itokawa (5).

We analyzed one of the largest grains returned by Hayabusa (RA-QD02-0049) through instrumental neutron activation analysis (INAA). We used a scanning electron microprobe (SEM) to perform the initial characterization of this grain at the receiving room at Institute of Space and Astronautical Science, Japan Aerospace Exploration Agency. The SEM results indicate that the particle is a large crystal of olivine (Fig. 1A), and small inclusions of troilite are contained in this olivine (Fig. 1B). In addition, small pieces of silicates are attached on the surface of olivine (Fig. 1B). Before assaying to INAA, the sample was rinsed with ethanol for the inspection of organic materials at Kyushu University, Fukuoka, Japan, where the rinsed sample was also analyzed by a Raman spectrometer for the characterization of carbonaceous compounds. The sample was then carefully placed into a quartz sample holder for neutron irradiation at Kyushu and brought to the Kyoto University Research Reactor Institute (KURRI), Kumatori, Osaka, Japan. Along with reference standards, the Itokawa sample was irradiated with neutrons at a thermal neutron flux of  $8.2 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$  for 19 hours. After irradiation, the quartz holder was replaced with a new (nonirradiated) one to reduce the background radioactivity during gamma-ray counting. During this procedure, the sample was split into five small grains, the largest of which was named RA-QD02-0049-1. The remaining four smaller grains were placed together into one sample holder and named RA-QD02-0049-2. Together with reference standards, both samples were

measured for their radioactivity at KURRI for the first three weeks and then at the Low Level Radioactivity Laboratory of Kanazawa University, Tatsunokuchi, Kanazawa, Japan. The detailed procedure for INAA is described in the supporting online material (SOM).

Both samples have similar chemical compositions, suggesting that the grain is fairly homogeneous in its chemical composition (Table 1). According to the surface observation by SEM, grain RA-QD02-0049 is composed mainly of olivine, with plagioclase and troilite as trace components. A slightly high Na content for RA-QD02-0049-1 suggests that it is somewhat plagioclase-rich relative to RA-QD02-0049-2.



**Fig. 1.** Back-scattered electron image of a whole view (A) and a partial view (B) of Itokawa particle RA-QD02-0049. (A) The particle is almost exclusively made of olivine with minor amounts of what are probably plagioclase and opaque inclusions mostly of troilite. (B) Enlarged view of the boxed area in (A) where some troilite (Tr) inclusions are observed. Many small pieces of silicates deposit on the surface.

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