

## Surface and internal structures of a space-weathered rim of an Itokawa regolith particle



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### ARTICLE INFO

#### Article history:

Received 1 September 2014

Revised 1 May 2015

Accepted 7 May 2015

Available online 15 May 2015

#### Keywords:

Regoliths  
Asteroid Itokawa  
Solar wind

### ABSTRACT

Surface morphologies of a regolith particle retrieved from Asteroid 25143 Itokawa were observed using field-emission scanning electron microscopy (FE-SEM). The images were compared with the internal structures of the space-weathered rim of the same particle observed by transmission electron and scanning transmission electron microscopies (TEM/STEM) to investigate whether there is a direct link between the surface morphology and internal structure. FE-SEM observation showed that most of the particle surface is covered by convex spots less than 100 nm in size. TEM/STEM observation revealed that this particle has a space-weathered rim composed of partially amorphous structures with nano-Fe particles and vesicles. The vesicles swell the surface and form blisters that correspond to the spotted structures observed by FE-SEM. These observations indicate that a space-weathered rim with blisters can be observed by FE-SEM without using destructive methods. The observation of the space-weathered rim by FE-SEM also enabled us to obtain the distribution of the space-weathered rim on the particle surfaces. The existence of space-weathered rims on the opposing surfaces of the particle shows that most of the surfaces were directly exposed to the space environment by movement on the Itokawa surface. The depths of the blister locations and the chemical composition of the space-weathered rim indicate that the observed space-weathered rim with blisters was formed mainly by solar wind irradiation. The space-weathered rim analyzed in this study is thicker than those of Itokawa particles previously examined, indicating that the rim may have experienced longer solar wind exposure than those previously observed.

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### 1. Introduction

Space weathering has been referred to describe the collective processes that alter the optical properties, chemical compositions, and structures of materials on the surfaces of airless Solar System bodies (Pieters et al., 2000). These processes include solar wind irradiation, implantation and sputtering, irradiation by galactic and solar cosmic rays, and micrometeorite bombardment (Pieters et al., 2000; Hapke, 2001; Clark, 2002). Space weathering is closely related to the alteration processes of the surfaces of atmosphere-free bodies. Therefore, the understanding of space weathering processes to interpret spectroscopic information of

airless celestial bodies obtained by ground-based and remotely sensed observations is important.

Several studies have examined space weathering on lunar surfaces. Single-domain metallic iron particles, commonly referred to as nanophase iron (npFe<sup>0</sup>) or submicroscopic iron, were observed in grain rims of lunar soils and in agglutinates, which are glassy products created by impact phenomena (Keller and McKay, 1993, 1997). These iron particles are believed to produce the general optical character of lunar soils (Pieters et al., 2000; Hapke, 2001; Clark, 2002; Chapman, 2004; Noble et al., 2007).

Some studies suggested that space weathering occurs on asteroidal surfaces (Sasaki et al., 2001; Clark, 2002; Vernazza et al., 2009). The Hayabusa spacecraft recovered regolith particles from MUSES-C Regio on the S-type Asteroid 25143 Itokawa (Yano et al., 2006; Nakamura et al., 2011; Tsuchiyama, 2014). Initial

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analyses of the Itokawa particles indicate that they are composed of minerals corresponding to LL5–6 chondrite (Nakamura et al., 2011; Tsuchiyama et al., 2011; Yurimoto et al., 2011; Nakashima et al., 2013). Noble gas analyses showed evidence of the implantation of solar wind and galactic cosmic rays (Nagao et al., 2011; Meier et al., 2014). Space-weathered rims, including the uppermost thin rims of vapor or sputtered deposition and thick rims formed mainly by solar wind irradiation, were observed (Noguchi et al., 2011, 2014; Keller and Berger, 2014; Thompson et al., 2014). It was found that the thick rims become partially amorphous, contain Fe-rich nanoparticles, and often contain vesicles (Noguchi et al., 2014). Unlike the lunar samples, solar wind irradiation is considered to be a major agent of the formation of space-weathered rims on the Itokawa particles (Noguchi et al., 2011, 2014). Thus, the analysis of the Itokawa particles is expected to provide important information about space weathering by solar wind irradiation, which is difficult to investigate in lunar regolith samples that have complicated rims, formed by both solar wind irradiation and micrometeoroid impact during longer residence time on the regolith than on Itokawa.

In previous studies, the structures of space-weathered rims were examined by observing cross sections produced by an ultra-microtome or focused ion beam (FIB) sampling using transmission electron and scanning transmission electron microscopies (TEM/STEM). Thus far, the surface morphologies of space-weathered rims have not been investigated. Some vesicle structures were observed in the space-weathered rims of both Itokawa and lunar samples by TEM/STEM that were believed to be closely related to the irradiation effects of solar wind (Keller and McKay, 1997; Noble et al., 2005; Noguchi et al., 2014). Some of the vesicles in the Itokawa particles swell the surface and form blisters (Noguchi et al., 2014). This finding suggests that the surface morphologies of the blisters of space-weathered rims can be identified by surface observation in the nano-scale using field-emission scanning electron microscopy (FE-SEM). If the surface morphologies of space-weathered rims can be specified, the distribution of space-weathered rims on the Itokawa particles can be easily and efficiently examined without sample destruction. Distribution of the space-weathered rims may provide detailed information about space weathering of regolith on Itokawa. In previous surface observations of lunar soils, blister-like objects on particle surfaces were reported (Assonov et al., 1998). However, a direct relation between the space-weathered rims and blister-like objects has not been examined.

In this study, we performed cross-sectional observation of space-weathered rims of an Itokawa particle using TEM/STEM after the observation of the particle surface by FE-SEM to determine whether there is a direct link between the surface morphologies and internal structures of the space-weathered rim of the Itokawa particle.

## 2. Samples and experimental methods

Thousands of very small particles were recovered in the two sample catcher rooms, A and B, in the sample container, which correspond to the second and first touchdowns to the surface of Itokawa, respectively (Nakamura et al., 2011; Yada et al., 2014). An Itokawa particle (RB-QD04-0043) that fell from catcher room B onto a quartz (silica glass) disk after physically tapping the container (list of distributed 1st AO Itokawa samples, <http://hayabusao.isas.jaxa.jp/catalog/dist/>) was analyzed. A schematic chart of the analysis procedure for the present sample is shown in Fig. 1. The particle was fixed with a 5- $\mu$ m-diameter carbon fiber using glycol phthalate, which partially covered the particle surface (Fig. 2A and B). Prior to FE-SEM observation, a three-dimensional

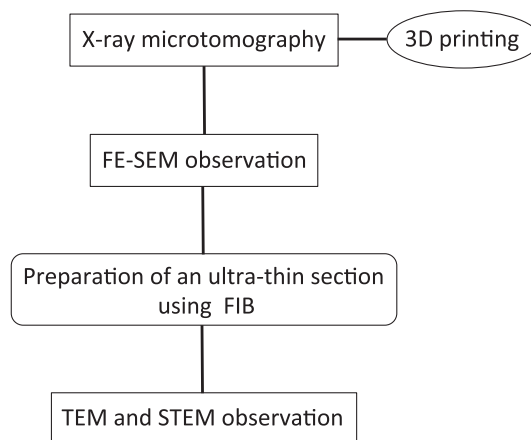


Fig. 1. A flow chart of the procedure about the present sample analyses.

(3D) structure of the sample was obtained using X-ray microtomography at beamline BL47XU of SPring-8, a synchrotron facility in Hyogo, Japan. We applied a dual-energy technique (Tsuchiyama et al., 2013) with X-ray energies of 7 keV and 8 keV, which can identify 3D distribution of minerals in Itokawa particles as well as their external shapes. The detailed imaging conditions were the same as those reported by Tsuchiyama et al. (2014).

The surface nano-morphology of the Itokawa particle was observed using an FE-SEM (JEOL JSM-7001F) equipped with an energy-dispersive X-ray (EDX) spectroscopic detector (Oxford Inca) at Kyoto University. To avoid possible nanometer-sized artificial decoration, the Itokawa particle was not coated with a conducting material, such as carbon. Moreover, to prevent the particle from being charged by electron irradiation, a secondary electron (SE) image observation was made at a low accelerating voltage of 2 kV in vacuum ( $9.6 \times 10^{-5}$  Pa) with an electron beam current of  $\sim 30$  pA. The observation was always made from two different angles with a  $5^\circ$  interval to obtain stereograms, which enabled us to evaluate the concavity or convexity of the surface morphologies. EDX analyses were performed at 15 keV in low vacuum with  $N_2$  (30 Pa) to identify minerals.

A region of interest (ROI) was selected based on the FE-SEM observation, and an electron-transparent section including the particle surface for TEM/STEM studies was prepared from the ROI using FIB-SEM (FEI Quanta 200 3DS) at Kyoto University. A plaster 3D model of the Itokawa particle was created using a 3D printer (ZPrinter 450, Z Corporation) at Tohoku University on the basis of the tomographic data (Fig. 2C). This step was performed to record the heterogeneous structure on the surface as recorded by the FE-SEM observation and to assist in the cutting process by FIB. To protect the particle surface from ion beam damage, the surface was coated with an approximately 20-nm-thick carbon. Then, the particle surface was coated by electron beam-deposited Pt followed by a Ga ion-deposited Pt layer. The section, approximately 1  $\mu$ m in thickness, was cut by a Ga ion operated at 30 kV with beam currents ranging from 3 nA to 30 nA, lifted from the particle, and mounted onto a TEM copper grid. The Ga ion source was operated at 30 kV with beam currents in the range of 100 pA to 1 nA to obtain a thickness of approximately 100 nm, and at 5 kV with a beam current of 48 pA for the final processing to remove the damaged surface layer.

The TEM imaging was performed using a field-emission transmission electron microscope (FE-TEM; JEM-2100F) equipped with an EDX system (JED-2300T). The accelerating voltage of the electron beam was 200 kV. High-angle annular dark-field (HAADF) imaging, which is sensitive to the atomic number of atoms (Z contrast imaging), was performed in the scanning transmission

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