Developments in microfabrication of mineral samples for simultaneous EBSD-EDS analysis utilizing an FIB-SEM instrument: study on an S-type cosmic spherule from Antarctica

Yu Kodama*, Naotaka Tomioka**, Motoo Ito** and Naoya Imae***

*Department of Marine & Earth Sciences, Marine Works Japan Ltd., Yokosuka 237-0063, Japan
**Kochi Institute for Core Sample Research, Japan Agency for Marine-Earth Science and Technology (JAMSTEC), Kochi 783-8502, Japan
***Antarctic Meteorite Research Center, National Institute of Polar Research, Tokyo 190-8518, Japan

A focused ion beam (FIB) scanning electron microscope (SEM) equipped with a low-energy Ar ion gun, an electron back-scattered diffraction (EBSD) detector, and an energy dispersive spectrometer (EDS) was newly developed for microfabrication, followed by submicroscopic chemical and crystallographic analyses in an identical sample chamber. The surface condition of mineral samples requires extreme care during FIB milling process, especially for EBSD measurement, as the EBSD pattern is greatly affected by a damaged amorphous surface caused by a high-energy Ga ion beam (30 keV), even when this damaged layer is only tens of nanometers in thickness. Low-energy broad Ar ion beam milling (1 keV) overcomes this problem and allows us to obtain sharp EBSD patterns. We applied the microfabrication-analysis protocol to a cosmic spherule. Comprehensive mineralogical datasets from SEM images, X-ray elemental maps, and EBSD patterns were successfully obtained from a minute mineral sample, where conventional cutting and polishing processes are not possible.

Keywords: Focused ion beam, Broad ion beam, Electron backscattered diffraction, Cosmic spherule

INTRODUCTION

The focused ion beam (FIB) in combination with a scanning electron microscope (SEM) is a promising tool for microfabrication and mineralogical analysis. The advantage of the FIB technique is the capability to extract micrometer-scale regions-of-interest in geomaterials (Wirth, 2009). In combination with the capability of an SEM, it further enables the observation of microtextural and elemental characterizations by secondary electron (SE) and back-scattered electron (BSE) images, as well as quantitative analysis if it has an energy dispersive X-ray spectrometer (EDS). In addition to elemental analysis with EDS, an FIB-SEM instrument equipped with an electron back-scattered diffraction (EBSD) detector facilitates the visualization of crystallographic orientations in polycrystalline mineral grains at micrometer-scale spatial resolution. Combined analysis with EDS and EBSD provides elemental compositions and phase identifications from an identical region-of-interest at sub micrometer to millimeter spatial scales.

However, the EBSD pattern is extremely sensitive to the surface condition of the sample, since it results from the topmost layer of the surface that is a few tens of nanometers in depth (Nowell et al., 2005). Conventional FIB fabrication using a high-energy focused Ga ion beam often causes the formation of an amorphous layer several tens of nanometer thick on the sample surface. In the case of microfabrication of a silicon single crystal, 30-keV Ga ion irradiation induced an amorphous layer of ~ 30 nm in thickness into the surface region, and a similar layer of ~ 10 nm was observed under irradiation by 10-keV Ga-ion irradiation (Kato, 2004). Moreover, ionic compounds (i.e., tausonite: SrTiO3, corundum: Al2O3) tend to be more sensitive to Ga-ion irradiation in com-
In order to reduce or remove the damage layer of the sample surface, sample preparation using low energy ion beam milling such as 1 to 5 keV FIB or broad Ar-ion beam has been demonstrated for various materials for high-quality TEM observation and EBSD analysis (Kato, 2004; Huang, 2004; Giannuzzi et al., 2005; Michael, 2007; Inoue and Kogure, 2012; Winiarski et al., 2017). Sample preparation combining focused Ga-ion beam and broad Ar-ion beam (Ar+ BIB) milling for silicon has been successfully demonstrated for high-quality EBSD patterns (Michael, 2007). Winiarski et al. (2017) utilized an Ar-ion milling to obtain 3D-EBSD tomography of WC-Co alloy and demonstrated large volumes (250 × 250 µm) of EBSD mappings. Inoue and Kogure (2012) applied surface preparation using 3 keV Ar-ion beam milling to phyllosilicates and successfully showed sharp EBSD patterns. Accordingly, low energy Ar-ion beam milling instrument is a better option in terms of a faster and easier sample surface treatment. In the above analysis methods, the samples are required to be transferred from an Ar milling instrument to an SEM-EBSD instrument. This transfer can cause a risk of sample loss, contaminations, and chemical reactions under atmospheric condition for sensitive materials.

The advantage of the FIB-SEM system equipped with a low energy Ar-ion gun, EDS and EBSD detectors is to perform ‘in-situ’ chemical and crystallographic analyses of a ‘fresh’ sample surface immediately after surface preparation without any sample transfer. Sample preparation of TEM samples using the FIB-SEM-Ar+ BIB system has been demonstrated, and improved images were successfully obtained (Stegmann et al., 2009; Kaur et al., 2016; Sato et al., 2017), therefore this system is also promising for EBSD analysis. While the milling rate of Ar-ion beam in this system is lower than that of standalone ion milling systems, optimization of FIB and Ar-ion milling conditions is necessary to push forward the advantage of this system.

In the present study, we developed microfabrication techniques for mineral samples utilizing combined EDS-EBSD analysis with an FIB-SEM instrument. The technique has been applied to an S-type cosmic spherule TT006b101 recovered from Antarctica that consists of silicate and oxide minerals for mineralogical and chemical analyses.

EXPERIMENTAL PROCEDURES

FIB-SEM instrument

The technical development in this study was demonstrated using the FIB-SEM instrument (Hitachi High-Technologies SMJ4000L) at the Kochi Institute of Core Sample Research, Japan Agency of Marine-Earth Science and Technology (Fig. 1). The SMJ4000L is equipped with three ion/electron guns—a focused Ga-ion beam, a field-emission electron beam, and a broad Ar-ion beam. The SEM column (ZEISS GEMINI column, thermal field emission type) and the FIB column (Ga liquid metal source) are orthogonally placed, and the Ar-ion and SEM columns are mutually inclined at approximately 60° (Fig. 2a). The system is equipped with an EDS detector (Oxford Instruments X-MAXN150, Fig. 2a) and an EBSD detector (Oxford Instruments NordlysNano, Fig. 2b).

We used Ga+ FIB and Ar+ BIB to prepare the sample surface for EDS and EBSD analysis. Ga+ FIB was performed in scanning mode with a spot size of ~40 nm (accelerating voltage: 30 kV, probe current: 300 pA) or ~5 nm (5 kV, 185 pA), and Ar+ BIB was performed in spot mode with a size of ~100 µm (1 kV, 14 nA). The beam direction of Ga+ FIB was nearly parallel to the surface of the sample, while that of Ar+ BIB was inclined at 17° to the surface (Fig. 2a). Fabrication by Ga+ FIB was carried out until the sample surface was completely flat. Subsequently, gentle milling by Ar+ BIB was conducted for 2-60 min.

Sample preparation

Single crystals of quartz and olivine. Natural quartz (SiO₂) from Mundo Nuevo mine, Peru, and natural olivine (forsterite: Fo₉₁) from San Carlos, Arizona, USA, were employed as representative samples of silicate minerals to evaluate ion-beam damage by Ga+ FIB and Ar+ BIB. These samples were embedded into acrylic resin, and the surfaces were made smooth and flat by a mechanical polishing process using diamond paste down to 1 µm. The polished samples were initially introduced to a standard single-beam FIB (Hitachi High-Technologies SMI4050) and fabricated to be 20 × 15 × 2 µm³ in dimension with Ga+ FIB at an acceleration voltage of 30
kV and a beam current of 240 pA. After the initial fabrication, the samples were mounted to a Cu–grid and transferred to the FIB–SEM SMJ4000L for further microfabrication for EBSD and EDS analyses.

**Cosmic spherules.** Due to limited working space in the FIB–SEM (SMJ4000L) chamber to transfer the extracted sample slab to a Cu–grid by a micro–manipulator, the initial preprocessing was performed using standard type single–beam FIB SMJ4050. In order to minimize contamination of organic materials from adhesive mate-

rials and physical instability caused by an electrostatic charging, the sample was mounted on an adhesive made of synthetic carbon nanotubes (Nitto Gecko®; Maeno and Nakayama, 2009). A portion of the sample was initially processed into a slab within 50 × 25 × 5 µm³ in dimension by 30–keV Ga⁺ FIB and mounted on a Cu–grid using carbon deposition (Figs. 3c and 3d). The Cu–grid with the sample was subsequently transferred into the FIB–SEM SMJ4000L. Then, the topmost surface of the sample was fabricated by 30–keV Ga⁺ FIB followed by low–energy ion milling with 1–keV Ar⁺ BIB for 60 min. Total fabrication time was approximately 10 h.

**EBSD analysis and EDS analysis**

SEM–EBSD analyses were carried out with a 20 kV acceleration voltage and an 8.0 nA electron probe current on a sample tilted by 50–70°. Simulated EBSD (Kikuchi) patterns of reference minerals for indexing were produced with lattice parameters atomic coordinates, and space groups in the following literatures: magnetite (cubic, Fd3m, a = 0.83958 nm; Wechsler et al., 1984); forsterite (orthorhombic, Pbnm, a = 0.4756 nm, b = 1.0207 nm, c = 0.5980 nm;
Smyth and Hazen, 1973); quartz (trigonal, \(P\overline{3}\alpha 21, \ a = 0.4916 \ \text{nm}, \ c = 0.54054 \ \text{nm}; \ Levien et al., 1980). \) EDS elemental analyses were performed at a 8 kV acceleration voltage and 3.0 nA of electron probe current without sample tilting. All EBSD and EDS analyses were operated using the AZtec software 3.1 (Oxford Instruments).

**Evaluation of EBSD pattern quality**

SEM–EBSD is a method to obtain qualitative and quantitative crystallographic information on materials of submicroscopic scale. Kikuchi bands in EBSD patterns show unique widths and directions corresponding to \(d\)-spacings and orientations of respective crystal planes (Schwarzer et al., 2009). For proper phase identification and crystal orientations, observed EBSD patterns should show the sharp edges and high contrast of the image to be automatically matched by an analytical software tool. We evaluated the values of ‘band contrast’ and ‘band slope’ among EBSD patterns depending on different Ga–ion and Ar–ion beam conditions. Both band contrast and band slope are the quality factors of the EBSD pattern derived from the Hough transformation (HF; Hough, 1972). Band contrast describes the average of intensity of the Kikuchi bands (intensity of the Kikuchi band corresponds to spots in HF images; Figs. 4e-4h) with respect to the overall intensity within the EBSD pattern, and band slope describes the maximum intensity gradient at the margins of the Kikuchi bands in the EBSD pattern (Maitland and Sitzman, 2007). These values range with 256 grades (0–255: low to high contrast) representing the sharpness of EBSD patterns.

**RESULTS AND DISCUSSION**

**Evaluation of EBSD patterns of silicate minerals at various ion-milling conditions**

FIB/BIB millings and EBSD analyses of silicate minerals were carried out in order to evaluate the quality of EBSD patterns at various FIB/BIB milling conditions: (1) sample surface finished at 30–keV Ga\(^+\) FIB, (2) 30–keV Ga\(^+\) FIB and 5–keV Ga\(^+\) FIB, (3) 30–keV Ga\(^+\) FIB and 1–keV Ar\(^+\) BIB, and (4) 30–keV Ga\(^+\) FIB, 5–keV Ga\(^+\) FIB, and 1–keV Ar\(^+\) BIB. In our case, 20 min was required to finish \(25 \times 5 \ \mu\text{m}^2\) on the sample surface of quartz, 15 min for \(8 \times 6 \ \mu\text{m}^2\) on the surface of forsterite, respectively; these process times are necessary to choose which condition is the most time-saving.

EBSD patterns obtained from the surface of quartz sample prepared by 30–keV Ga\(^+\) FIB show weak Kikuchi bands suggesting the surface mostly amorphized by ion beam damage (Fig. 4a). Kikuchi bands from the sample surface prepared by 5–keV Ga\(^+\) FIB are sharp (Fig. 4b), while those by 30–keV Ga\(^+\) FIB and Ar\(^+\) BIB are obscure (Fig. 4c). In most cases, the obscure band patterns can be indexed by manual matching, however, they cannot be accurately indexed by software–based automated pattern matching (Figs. 4a and 4c). In the sample surface finished by 5–keV Ga\(^+\) FIB and 5–keV Ga\(^+\) FIB with Ar\(^+\) BIB, EBSD patterns are obviously better indexed by the automated pattern matching (Figs. 4b and 4d). Comparing HF images (Figs. 4e-4h), those from the EBSD patterns prepared by 5–keV Ga\(^+\) FIB and 5–keV Ga\(^+\) FIB with Ar\(^+\) BIB gives 5 strong peaks (white triangles in Fig. 4h) and \(\sim 3\) additional peaks (Figs. 4f and 3h; additional peaks are indicated as black triangles in Fig. 4h). On the other hands, 30–keV Ga\(^+\) FIB and 30–keV Ga\(^+\) FIB with Ar\(^+\) BIB also gives 5 peaks as same as former conditions, however, additional peaks mentioned above are hard to be distinguished from the background (Figs. 4e and 4g).

The band contrast/slope values from samples prepared by 30–keV Ga\(^+\) FIB effectively increases with increasing the duration of Ar\(^+\) BIB up to 20 min, while those of the 30–min milling show lower values and do not show further improvement (Fig. 5). Meanwhile, the band contrast/slope values from samples prepared by 5–keV Ga\(^+\) FIB...
FIB do not show significant improvement, and these values are equivalent to the values obtained by 30-keV Ga⁺ FIB with enough Ar⁺ BIB. Therefore, appropriate process conditions of final polishing for natural quartz are 5–20 keV Ga⁺ FIB or ~20 min of Ar⁺ BIB. For natural quartz, improvement on EBSD patterns by 5–keV Ga⁺ FIB is equivalent to ~20 min of Ar⁺ BIB, because the required duration of FIB depends on the sample size, in our case, 30–keV Ga⁺ FIB with Ar⁺ BIB can be a reasonable option for samples larger than ~25 × 5 µm². Note that process time using Ar⁺ BIB is not affected by the sample size due to relatively large beam diameter of Ar⁺ BIB (~100 µm). In contrast, process time using Ga⁺ FIB operated in scanning mode is proportional to the sample size. EBSD patterns from the forsterite prepared by 30-keV Ga⁺ FIB show slightly obscure Kikuchi bands (Fig. 6a), while HF image has acceptable peaks for automated band detection (Fig. 6e). Other beam conditions obviously improved EBSD patterns, and peaks in the HF images are acceptable for indexing (Figs. 6b-6d and 6f-6h).

Comparing quality of EBSD patterns in respective beam conditions, band contrast shows slight improvement on EBSD patterns obtained by 30-keV Ga⁺ FIB with Ar⁺ BIB at the first 5 min, but it is not further improved in other conditions (Fig. 7a). On the other hand, Ar⁺ BIB improves band slope from the sample prepared by both of 30-keV Ga⁺ FIB and 5-keV Ga⁺ FIB (Fig. 7b). We have observed optimum durations of better surface preparation for natural forsterite; 6–8 min by 5-keV Ga⁺ FIB with Ar⁺ BIB and 10–15 min by 30-keV Ga⁺ FIB with Ar⁺ BIB. Even final polish of 30-keV FIB allows us to obtain acceptable EBSD patterns in our results. 30-keV Ga⁺ FIB with Ar⁺ BIB or 5-keV Ga⁺ FIB with Ar⁺ BIB are reasonable options for samples larger and smaller than ~5 × 4 µm², respectively.

A study on a cosmic spherule

Extraterrestrial materials are suitable samples for the micro sample preparation and analytical method since they are too small to be handled and processed by a conven-
tional cutting method (i.e., diamond wire or blade saws) and chemical/mechanical polishing processes. Interplanetary dust particles, micrometeorites, and asteroidal/cometary surface samples collected by the sample return missions, are important materials for elucidating the origin and nature of the early Solar System (e.g., Rietmeijer, 1998; Engrand et al., 1999; Brownlee et al. 2006; Noguchi et al., 2011). These samples are generally sub-millimeter in size and only available in a limited amount. Therefore, a microfabrication technique with an FIB is a central piece of sample preparation for minimizing material loss and/or mechanical/chemical damages.

We applied the method described in the previous section to an Antarctic micrometeorite particle of ~200 µm in diameter, TT006b101, collected from ice near the Syowa Station in the Antarctica (Iwata and Imae, 2002). The particle is classified into barred olivine S-type cosmic spherule (Genge et al., 2008), showing a spherical shape due to melting by heating upon atmospheric entry, and is mainly composed of silicates and Fe-oxide (Fig. 3a). The sample has been used as part of an ongoing project for comprehensive analysis which requires microfabrication into much smaller sizes for further TEM and NanoSIMS analyses.

Figure 8 shows comparison between EBSD phase maps by two different milling conditions. One was prepared by 30-keV Ga+ FIB (Fig. 8b), and the other was the same sample prepared by 1-keV Ar+ BIB (Fig. 8c). Since EBSD maps are obtained using software-based automated pattern matching, the indexing rate of each phase is strongly affected by quality of the EBSD pattern. The EBSD map without Ar+ BIB showed indexing rates of 9% (magnetite), 26% (forsterite), and 65% (non-indexed) of total pixels, whereas after Ar+ BIB showed improved indexing rates of 15% (magnetite), 62% (forsterite), and 23% (non-indexed). In terms of the indexing rate, Ar+ BIB for forsterite worked more effectively than for magnetite. Therefore, duration of Ar+ BIB should be optimized according to each material and balanced between all the materials involved, especially for the composite materials.

Figures 9 and 10 show the result of crystallographic maps by EBSD and X-ray elemental maps by EDS, respectively. The sample exhibits a petrographically uniform texture throughout the processed surface (Figs. 9a and 10). The elemental maps show that the cosmic spher-
odule is composed of three phases: euhedral to subhedral Mg–Fe–rich silicate and Fe-oxide, and anhedral Ca-Al–Fe–rich silicate (Figs. 9a and 10). Chemical composition of Mg–Fe–rich silicate (Fa22–33) were determined by EDS. The EBSD patterns found Mg–Fe–rich silicate grains (3.0–8.0 µm in size), and Fe-oxide grains (0.5–5.0 µm) to be forsterite and magnetite, respectively. Meanwhile, interstitial Ca-Al-Fe–rich silicate did not show any Kikuchi bands, suggesting its amorphous nature. Based on EBSD data, the directional basic lattice vectors of forsterite and magnetite were also compiled into stereographic projections (Fig. 11). As can be seen in figure 11, these forsterite and magnetite grains have the characteristic crystallographic relationship: [100]_Fo//<111>_Mag and [001]_Fo//<110>_Mag. This relationship is consistent with the coherent boundary of forsterite and magnetite sharing close-packed layers of oxygen for both crystal structures (Kohlstedt and Vander Sande, 1975; Puga et al., 1999).

The crystallographic orientations of forsterite and magnetite grains in combination with their grain sizes and morphologies may provide information about the crystallization processes of cosmic dust during rapid cooling after atmospheric entry heating.

Our study demonstrates the advantages of the FIB-SEM instrument equipped with low-energy Ar⁺ BIB allowing microtextural, chemical and crystallographic analysis of a ‘fresh’ surface of minute mineral samples immediately after microfabrication. The sample is further processed to be a 100 nm thin-film for TEM analysis to obtain detailed characterizations of defect structures and nanoscale inclusions. The thin-film samples can be inspected for trace element abundances and isotopic ratios with NanoSIMS. We are, therefore, currently working on a comprehensive analysis technique among FIB, SEM, NanoSIMS, and TEM for planetary materials (Ito et al., 2017).

**SUMMARY**

A comprehensive dataset including SE/BSE images, elemental compositions, mineral phases and crystallographic orientations was successfully obtained from a minute mineral sample after ion-processing in the identical in-

---

**Figure 9.** An EBSD map of the cosmic spherule TT006b101 consisting of forsterite, magnetite, and interstitial amorphous silicate. (a) Back-scattered electron image, (b) band contrast image, and (c) phase mapping image. White, gray, and black represent magnetite, forsterite, and non-indexed, respectively. (d)-(f) representative EBSD pattern of forsterite, magnetite, and amorphous silicate. The EBSD map was collected at following conditions: acceleration voltage: 20 kV, probe current: 8 nA.

**Figure 10.** X-ray elemental maps of the cosmic spherule TT006b101 by energy dispersive spectroscopy. Elemental maps were collected at following electron beam conditions: acceleration voltage, 8 kV; probe current, 3.0 nA.
strument of the FIB-SEM instrument. Combining phase maps by EBSD and X-ray elemental maps by EDS, all mineral phases including amorphous materials can be unequivocally identified. We emphasize that Ar⁺ BIB has great potential to remove the damaged layer formed during high-energy Ga⁺ FIB processing, and to improve the accuracy of EBSD analyses especially of ion-beam sensitive geological materials.

ACKNOWLEDGMENTS

This work was partially supported by the JSPS KAKENHI Grants No. 15H03750 to N. T. and No. 26287142, 18K18795, and 18H04468 to M. I. We thank J. Ando for providing natural quartz samples. The Antarctic cosmic spherules were provided by the Antarctic Meteorite Research Center of the National Institute of Polar Research. Constructive comments from T. Mikouchi and two anonymous reviewers are helpful to improve the manuscript.

REFERENCES

Brownlee, D., Tsou, P., Alén, J., Alexander, C.M.O.D., et al. (2006) Comet 81P/Wild 2 under a microscope. Science, 314, 1711–1716.
Engrand, C., DeLoule, E., Robert, F., Maurette, M. and Kurat, G. (1999) Extraterrestrial water in micrometeorites and cosmic spherules from Antarctica: An ion microprobe study. Meteoritics & Planetary Science, 34, 773–786.
Genge, M.J., Engrand, C., Gounelle, M. and Taylor, S. (2008) The classification of micrometeorites. Meteoritics & Planetary Science, 43, 497–515.
Giannuzzi, L.A., Geurts, R. and Ringnalda, J. (2005) 2 keV Ga⁺ FIB milling for reducing amorphous damage in silicon. Microscopy and Microanalysis, 11, S02, 828–829.
Hough, P.V.C. (1972) Method and means for recognizing complex patterns, U.S. Patent 3069654. 1962. or Richard O. Duda and Peter E. Hart. Use of the Hough transformation to detect lines and curves in pictures. Commun. ACM, 15:11–15, January.
Huang, Z. (2004) Combining Ar ion milling with FIB lift-out techniques to prepare high quality site-specific TEM samples. Journal of Microscopy, 215, 3, 219–223.
Huh, Y., Hong, K.J. and Shin, K.S. (2013) Amorphization induced by focused ion beam milling in metallic and electronic materials. Microscopy and Microanalysis, 19, S5, 33–37.
Inoue, S. and Kogure, T. (2012) Electron backscatter diffraction (EBSD) analyses of phyllosilicates in petrographic thin sections. American Mineralogist, 97, 4, 755–758.
Ito, M., Tomioka, N., Kodama, Y. and Imae, N. (2017) A FIB-NanoSIMS-TEM study of unmelted Antarctic micrometeorite TT006D107. Lunar and Planetary Science XLVIII, #1776.
Iwata, N. and Imae, N. (2002) Antarctic micrometeorite collection at a bare ice region near Syowa Station by JARE-41 in 2000. Antarctic Meteorite Research, 15, 25-37.

Kato, N.I. (2004) Reducing focused ion beam damage to transmission electron microscopy samples. Journal of Electron Microscopy, 53, 451-458.

Kaur, J., Kurimoto, N., Jamaludin, K.R., Mitsuhara, M., et al. (2016). Electron microscopy analysis of microstructure of postannealed aluminum nitride template. Applied Physics Express, 9, 6, 065502, 1-4.

Kohlstedt, D.L. and Vander Sande, J.B. (1975) An electron microscopy study of naturally occurring oxidation produced precipitates in iron-bearing olivines. Contributions to Mineralogy and Petrology, 53, 13-24.

Levien, L., Prewitt, C.T. and Weidner, D.J. (1980) Structure and elastic properties of quartz at pressure. American Mineralogist, 65, 920-930.

Maitland, T. and Sitzman, S. (2007) Electron backscatter diffraction (EBSD) technique and materials characterization examples. In Scanning Microscopy for Nanotechnology: Techniques and Applications (Zhou, W. and Wang, Z.L. Eds.). Springer, Boston, MA., 41-75.

Maeno, Y. and Nakayama, Y. (2009) Geckolike high shear strength by carbon nanotube fiber adhesives. Applied Physics Letters, 94, 012103 https://doi.org/10.1063/1.3050450.

Michael, J.R. (2007) Improved EBSD sample preparation via low energy Ga+ and Ar+ ion milling. Microscopy and Microanalysis, 13, 926-927.

Noguchi, T., Nakamura, T., Kimura, M., Zolensky, M.E., et al. (2011) Incipient space weathering observed on the surface of Itokawa dust particles. Science, 333, 1121-1125.

Nowell, M.M., Witt, R.A. and True, B. (2005) EBSD sample preparation: Techniques, tips, and tricks. Microscopy Today, 13, 44-49.

Puga, E., Ruiz Cruz, M.D. and Diaz de Federico, A. (1999) Magnetite-silicate inclusions in olivine of ophiolitic metabasalts from the Mulhacen Complex, Betic Cordillera, southeastern Spain. The Canadian Mineralogist, 37, 1191-1209.

Rietmeijer, F.J. (1998) Interplanetary dust particles. In Planetary Materials (Papike, J.J. Ed.). Reviews in Mineralogy, 36, Mineralogical Society of America, Washington, D.C., 1-95.

Sato, T., Nakano, K., Matsumoto, H., Torikawa, S., et al. (2017) High quality lamella preparation of gallium nitride compound semiconductor using Triple Beam system. In Journal of Physics: Conference Series, 902, 1, p. 012019, IOP Publishing, Bristol.

Schwarzer, R.A., Field, D.P., Adams, B.L., Kumar, M. and Schwartz, A.J. (2009) Present state of Electron Backscatter Diffraction and prospective developments. In Electron Backscatter Diffraction in Material Science (Schwartz, A.J., et al. Eds.). Springer, New York, 1-20.

Smyth, J.R. and Hazen, R.M. (1973) The crystal structures of forsterite and hortonolite at several temperatures up to 900 °C. American Mineralogist, 58, 588-593.

Stegmann, H., Ritz, Y., Utess, D., Engelmann, H.J. and Zschech, E. (2009) In-situ low energy argon ion milling of nanoelectronic structures using a triple beam system. Microscopy and Microanalysis, 15, 52, 170-171.

Wechsler, B.A., Lindsay, D.H. and Prewitt, C.T. (1984) Crystal structure and cation distribution in titanomagnetites (Fe3−x, Ti)xO4. American Mineralogist, 69, 754-770.

Winiarski, B., Gholinia, A., Mingard, K., Gee, M., Thompson, G.E. and Withers, P.J. (2017) Broad ion beam serial section tomography. Ultramicroscopy, 172, 52-64.

Wirth, R. (2009) Focused Ion Beam (FIB) combined with SEM and TEM: Advanced analytical tools for studies of chemical composition, microstructure and crystal structure in geomaterials on a nanometre scale. Chemical Geology, 261, 217-229.

Developments in microfabrication for EBSD-EDS analysis by FIB-SEM 415

Manuscript received December 27, 2018
Manuscript accepted July 10, 2020
Manuscript handled by Takashi Mikouchi