

**NEW NOBLE GAS DATA AND FURTHER EXAMINATIONS OF DUST FROM ASTEROID ITOKAWA.**

H. Busemann<sup>1</sup>, M.M.M. Meier<sup>2</sup>, F. Altmann<sup>3</sup>, C. Alwmark<sup>4</sup>, S. Bajt<sup>5</sup>, J. Beyersdorfer<sup>3</sup>, U. Böttger<sup>6</sup>, S.A. Crowther<sup>7</sup>, J.D. Gilmour<sup>7</sup>, U. Heitmann<sup>8</sup>, H.-W. Hübers<sup>6</sup>, C. Maden<sup>1</sup>, F. Marone<sup>9</sup>, S.G. Pavlov<sup>6</sup>, U. Schade<sup>10</sup>, N.H. Spring<sup>7</sup>, M. Stamparoni<sup>9</sup>, and I. Weber<sup>8</sup>. <sup>1</sup>IGP, ETH Zürich, Switzerland (henner.busemann@erdw.ethz.ch), <sup>2</sup>CRPG-CNRS Nancy, France, <sup>3</sup>Center for Appl. Microstructure Diagnostics (CAM), Fraunhofer Inst. for Mechanics of Materials, Halle, Germany, <sup>4</sup>Dept. of Geology, Univ. of Lund, Sweden, <sup>5</sup>Photon Sciences, DESY, Hamburg, Germany, <sup>6</sup>Inst. of Optical Sensor Systems, DLR Berlin, Germany, <sup>7</sup>SEAES, Univ. of Manchester, UK, <sup>8</sup>IfP, WWU Münster, Germany, <sup>9</sup>Swiss Light Source (SLS), PSI Villigen, Switzerland, <sup>10</sup>Helmholtz-Zentrum Berlin, Germany.

**Introduction:** JAXA's Hayabusa sample return mission to near-Earth asteroid 25173 Itokawa [1] provides an invaluable record of the surface and history of a metamorphosed (type 4-6) LL chondritic parent body. The analysis of these surface grains allows us to match remote observations and modeling of an asteroid's history and current properties with ground truth laboratory-based examinations; mass spectrometry and electron microscopy of the dust enable us to study in detail the history of asteroidal regolith and compare the results with theoretical considerations of the evolution of rubble piles in the inner solar system [2-5].

In order to determine the cosmic ray exposure (CRE) history and noble gas contents of the Itokawa surface dust, JAXA allocated several particles to our consortium. While most particles from the 1<sup>st</sup> AO (announcement of opportunity) have been examined [e.g. 6-12], those from the 2<sup>nd</sup> AO are still being analyzed.

Here, we present new results for the latter batch including micro-Raman spectroscopy to establish the mineralogy of the grains, and synchrotron radiation X-ray tomographic microscopy (SRXTM) to precisely determine their volumes and masses. Furthermore we discuss an attempt to remove outer layers of extraterrestrial grains by Focused Ion Beam (FIB) technique, to minimize the interference of abundantly incorporated solar wind (SW, found within the uppermost 100s of nm of regolith grains) on the volume-correlated cosmogenic noble gases. We also discuss the He and Ne content and CRE age of two newly studied grains from the first batch.

**Experimental:** All grains discussed here with the techniques applied so far are listed in table 1. The mineralogy obtained by Raman spectroscopy [6], volumes determined by SRXTM [9] and resulting masses are given in table 2. Particles 194, 235 and 241 were delivered in N<sub>2</sub> atmosphere to avoid contamination with terrestrial noble gases. They were transferred inside a N<sub>2</sub>-filled glove box into a sealed container equipped with a BaF<sub>2</sub> window, suitable for infrared and Raman spectroscopy. During transport to the Berlin research facilities the container broke and all particles were exposed to air. The particles were recovered, however grain 194 fragmented severely. Together with the in-

tact grains 235 and 241, sufficiently large fragments of grain 194 (typically >25 μm) were transferred by micromanipulator to fluorocarbon threads and individually analyzed by SRXTM at the Swiss Light Source [9].

**SRXTM and volume determination:** From the reconstructed X-ray images, volumes were determined for grains 241 and 235 (Table 2), which agree with estimates based on an empirical relationship ( $V = A^{1.375}$  within a factor of 2.2 at 95 % level) between the grain cross-sectional area A (SEM imaging by JAXA) and volume V (SRXTM of 39 Itokawa grains [13]). This correlation allows us also to estimate the volume of grains where SRXTM data are not available. Precise volumes are essential to obtain useful CRE ages [9].

**Table 1.** Particles studied and techniques applied.

Particle # RA-QD02-x	notes	opt. estim. size / μm	Raman mode	Infrared	SRXTM	He/Ne
<i>1<sup>st</sup> AO particles</i>						
0051	PB	70 × 40	spots/scan		x	x
0158	originally in N <sub>2</sub>	60 × 30	spots		x	x
<i>2<sup>nd</sup> AO particles</i>						
0039	PB, C-coated	45 × 25	spots			
0068	PB, C-coated	70 × 30	spots			
0194*	originally in N <sub>2</sub>	90 × 80	spots	x	x	
0235	originally in N <sub>2</sub>	70 × 35	spots	x	x	
0241	originally in N <sub>2</sub>	80 × 40	spots	x	x	

PB = "potted butt", remainders in epoxy after sectioning, used for SIMS analysis; \*split into several fragments.

A summed volume of all recovered fragments of grain 194 of ~110000 μm<sup>3</sup> was provided by SRXTM, however the empirical relationship suggests a total volume of ~290000 μm<sup>3</sup>, suggesting significant loss during fragmentation or subsequent recovery. We hope to convert the accident into our advantage: some of the recovered fragments may originate from the interior of the original grain, shielded from SW implantation. This could help to reach our goal of analyzing the cosmogenic component of a Hayabusa grain without interference by superficially incorporated SW. We aim

to locate the position of each fragment in the original grain using phases with slightly distinct X-ray absorption determined by SRXTM, once 3D models of the fragments are available. MicroRaman spectroscopy [6] was performed on all non-embedded grains. A Fo# of  $70 \pm 10$  was used to determine all masses in table 2.

**Table 2.** Volumes, masses and mineralogy.

Particle #	SRXTM vol / $\mu\text{m}^3$	SEM vol / $\mu\text{m}^3$	mineralogy	mass / ng
0051	not detected	~5000	Ol, Px, Pl	~19
0158	23500±1000	~25000	Ol	84±4
0039	15300 [13]*	~18000	Ol, Px	tbd
0068	55000 [13]*	~58000	Ol, Pl	tbd
0194	~110000	~290000	Ol, Tr	~397
0235	97700±400	~87000	Ol, Pl, Tr	352±11
0241	33900±600	~37000	Px, Tr, Fe	122±4

\*before sectioning.

**Plasma-FIB erosion of grain surfaces:** In order to minimize the impact of the SW, we studied ways to remove the grain surface. We cut two fossil micrometeoritic chromite grains [14] in two ~equal pieces using a Plasma (Xe) FIB instrument at the Fraunhofer CAM (Halle, Germany). One half of each grain was left as a reference while the second half was turned onto the cut surface. The exposed surface was then eroded by at least a few  $\mu\text{m}$  using a high-current ion beam. The grains were then analyzed for He and Ne. For both grains, the technique was successful in reducing the SW concentration of the eroded piece with respect to the pristine one by factors of 3 and 100 for Ne and 6 and 165 for He, respectively. The large difference in reduction efficiency may suggest that the SW is unevenly distributed over the grain surfaces or, perhaps, shielding effects. Surprisingly, the erosion of the surface did not result in a better resolution of the cosmogenic component: for the chromite grain with a factor of 100 SW-Ne reduction, the pristine half has a  $^{21}\text{Ne}$ -CRE age of ~50 Ma, while the eroded half has an age of only ~1 Ma. This suggests that the cosmogenic Ne was reduced by a similar large factor as the SW-Ne, contrary to our expectations. For the chromite grain with a factor of 3 reduction in SW-Ne, the CRE age was reduced from 3 to 2 Ma. At present we do not have an explanation for these observations. We consider that the cosmogenic Ne in these chromite grains is inhomogeneously distributed – or that bombardment in the Plasma-FIB heated the grains sufficiently to lose a large fraction of the cosmogenic component. Further

investigations are clearly needed before applying this technique on Hayabusa grains.

**New noble gas data and CRE ages:** Due to a long down time of the high-sensitivity mass spectrometer used for this project, only two further 1<sup>st</sup> AO grains, 051 and 158, could be measured for their He and Ne contents and CRE ages. Grain 051 was a “potted butt” (table 1). The thin grain was successfully recovered from the resin, but it broke upon transfer onto the fluorocarbon thread into several fragments. These fragments were too small to have their volume reliably determined using SRXTM due to artefacts related to sharp edges. Its mass was estimated using the cross-sectional area and assuming a thickness of 4  $\mu\text{m}$ , as suggested by the thickness of other fragments on the fluorocarbon thread. Combining its mass with the measured cosmogenic  $^{21}\text{Ne}$  (with large errors due to very low gas amounts), results in a CRE age of  $9 \pm 7$  Ma, consistent with all previously determined ages with similarly large uncertainties [7-12,15]. A  $^3\text{He}/^4\text{He}$  ratio of 0.03 and a  $^3\text{He}/^{21}\text{Ne}$  ratio of ~30 (similar to other grains) was determined for grain 051. Combining the well-measured volume of grain 158 with Fo# of  $72 \pm 8$  [6] leads to a mass of  $84 \pm 4$  ng. Its Ne isotopic composition ( $^{20}\text{Ne}/^{22}\text{Ne} = 12.3 \pm 1.0$ ;  $^{21}\text{Ne}/^{22}\text{Ne} = 0.0307 \pm 0.0045$ ) falls, within uncertainties, onto the SW fractionation line, corresponding to a maximum CRE age of 0.63 Ma ( $2\sigma$ ). The  $^3\text{He}/^4\text{He}$  and  $^4\text{He}/^{20}\text{Ne}$  ratios of  $4.4 \times 10^{-4}$  and 417, respectively, confirm that He and Ne in grain 158 are dominated by the SW.

**Conclusions:** Our new data support the uniformly short exposure to cosmic rays of the Itokawa regolith of <8 Ma [10-12] suggesting a freshly rejuvenated regolith. Further noble gas data for 2<sup>nd</sup> AO grains are expected to be presented at the meeting.

**References:** [1] Nakamura T. et al. (2011) *Science*, 333, 1113-1116. [2] Noguchi T. et al. (2011) *Science*, 333, 1121-1125. [3] Keller L.P. and Berger E.L. (2014) *Earth Planets & Space*, 66, 71. [4] Michel P. (2014) *Elements*, 10, 19-24. [5] Fujiwara A. et al. (2006) *Science*, 312, 1330-1334. [6] Böttger U. et al. (2014) *LPSC*, 45, #1411. [7] Busemann H. et al. (2013) *LPSC*, 44, #2243. [8] Meier M.M.M. et al. (2014) *LPSC*, 45, #1247. [9] Meier M.M.M. et al. (2013) *LPSC*, 44, #1937. [10] Busemann H. et al. (2014) *Meteorit. Planet. Sci. Suppl.*, 49, #5362. [11] Busemann H. et al. (2014) *Hayabusa 2014 Symp. Solar System Materials*, Sagamihara, Japan, 1204-1600. [12] Meier M.M.M. et al. (2014) *Hayabusa 2014 – Symp. Solar System Materials*, Sagamihara, Japan, 1204-1545. [13] Tsuchiyama A. et al. (2014) *Meteorit. Planet. Sci.*, 49, 172-187. [14] Schmitz B. et al. (2003) *Science*, 300, 961-964. [15] Nagao K. et al. (2011) *Science*, 333, 1128-1131.