

**A CONSORTIUM STATUS REPORT: THE CHARACTERISATION OF THE ASTEROID ITOKAWA REGOLITH – A CORRELATED STUDY BY X-RAY TOMOGRAPHY, MICRO-RAMAN SPECTROSCOPY, AND HIGH-SENSITIVITY NOBLE GAS ANALYSIS.** H. Busemann<sup>1</sup>, C. Alwmark<sup>2</sup>, S. Bajt<sup>3</sup>, U. Böttger<sup>4</sup>, J.D. Gilmour<sup>1</sup>, U. Heitmann<sup>5</sup>, H.-W. Hübers<sup>4</sup>, M.M.M. Meier<sup>2</sup>, S. Pavlov<sup>4</sup>, U. Schade<sup>6</sup>, N.H. Spring<sup>1</sup>, I. Weber<sup>5</sup>. <sup>1</sup>SEAES, Univ. of Manchester, UK (henner.busemann@manchester.ac.uk), <sup>2</sup>Dept. of Geology, Univ. of Lund, Sweden, <sup>3</sup>Photon Sciences, DESY, Hamburg, Germany, <sup>4</sup>Institute of Planetary Research, DLR Berlin, Germany, <sup>5</sup>IfP, WWU Münster, Germany, <sup>6</sup>Helmholtz-Zentrum Berlin (HZB), Germany.

**Introduction:** Precious samples from S-type asteroid 25143 Itokawa have been sampled by the JAXA (Japanese Space Agency) Hayabusa mission in 2005 and returned to Earth in 2010. Itokawa is, succeeding the Moon and comet Wild 2, the third planetary body successfully probed by a sample return mission. The initial studies [e.g., 1-5] revealed that Itokawa consists mostly of type LL5-6 material. It experienced severe surface alteration due to space weathering, as documented by surficial, nanosize S-and Fe-bearing phases in some grains [3]. Noble gas studies [2] indicate that Itokawa experiences a surprisingly intense surface loss at a rate of tens of cm/Ma, implying that Itokawa (largest dimension ~540 m) will be destroyed quickly.

We received material through JAXA in Sept. 2012 and aim to analyze noble gases in Itokawa samples with high sensitivity, including Kr and Xe, which could not be studied previously, because of the low concentrations. We will combine the noble gas studies with scanning microRaman spectroscopy, Fourier-transform infrared spectroscopy (FTIR), and synchrotron radiation X-ray tomographic microscopy (SRXTM). These provide non-destructive characterizations of grain density, mineralogy, structure, and potential space weathering, which are essential to assess gas concentrations of potentially present cosmogenic, solar, trapped and radiogenic components. Here, we summarize the work of our consortium performed so far. Further studies will be presented at the meeting.

**Samples & Handling:** The allocated samples are listed in the table. Three particles have not been exposed to air. They have been delivered in pure N<sub>2</sub> in a special JAXA steel transport container [6]. The metal flange has been replaced, within a makeshift N<sub>2</sub>-filled glove box (HZB), with a transparent quartz window. This allowed us to study the samples, sandwiched between the two original synthesized quartz glass plates, by Raman spectroscopy without exposure to air.

Two samples were embedded in resin (potted buts (PB) = hemispherical remainders of micromilling), and two were allocated between microscope slides, see table (one cover slide was replaced by CaF). All four grains were examined by optical means and Raman spectroscopy. After subsequent FTIR examination of

#49-4 (the particle was placed on an IR-transparent CsI substrate disk with a Narishige micro-manipulator and W needle), the two embedded grains were cleaned mechanically from the original resin (“Embed 812”, acetone-insoluble) and fixed on glass slides with the acetone-soluble “UHU superglue EASY liquid”. Samples #35, #49-1 and -4 were manually transferred and fixed with spray glue onto commercial PVDF Fluorocarbon thread and then mounted in thin-walled (700 μm inner diameter) X-ray-suitable glass capillaries for SRXTM.

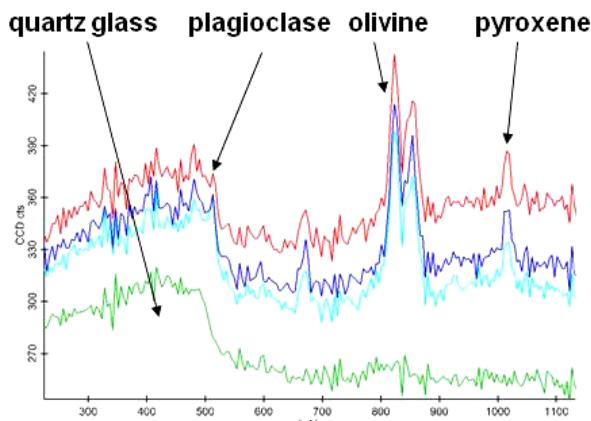
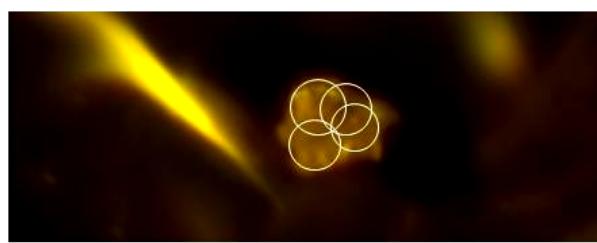
Sample #49 was fragmented during the previous study [3], the removal of the resin resulted in fragmentation of #51. Fragmentation is ideal for the final simultaneous high-sensitivity analysis of He-Ne (Zurich) and Kr-Xe (Manchester).

**Table.** Particles studied and techniques applied so far by this consortium.

Particle # RA-QD02-	notes	size / μm	Raman mode	FTIR	SRXTM
0035	embedded PB, space weathered [3]	75	spots/scan	x	
0049-1	NAA [5]	140	spots	x	
0049-4	NAA [5]	90	spots/scan	x	x
0051	embedded PB, not space weathered [3]	70	spots/scan		
0158	in N <sub>2</sub>	60	spots		
0187	in N <sub>2</sub>	60	spots		
0197	in N <sub>2</sub>	60	spots		

**Experimental: Raman Spectroscopy.** All samples were examined with DLR’s WiTec alpha300R confocal scanning Raman microscope [7,8], with a 532 nm laser excitation and a spectral resolution of ~4 cm<sup>-1</sup>. Samples #158, #187 and #197 were examined within the N<sub>2</sub>-filled port (see above) in spot mode for 120s per spot through a 10× objective with ~200 μW and <1 μm spot size. Samples #49-1 and -4 were investigated under similar conditions through cover slides.

A maximum power of 4 mW at a ~0.5-1  $\mu\text{m}$  spot size was applied for spot measurements on #51. Tests and calculations show that any significant heating during the measurements can be excluded. However, #51 was also scanned with up to 4 mW and 100 $\times$  objective for 10s/pxl, which caused heating and softening of the surrounding resin. Particles #35 and #51 have been previously examined by ion- and electron probe [4] as can be seen by various surficial Au and C coating remnants. We assume that some surface or resin C on the grains absorbed sufficient laser energy to heat the mount.



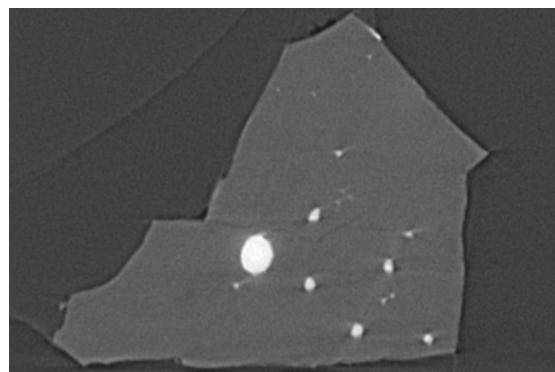
**Fig. 1** Raman spectra and major minerals identified in particle #197 within N<sub>2</sub>, detected through optical port and quartz glass (spots given above in optical image).

**FTIR.** Fragment 49-4 was examined in the mid-IR region in transmission. Although the CsI substrate is transparent down to 200  $\text{cm}^{-1}$ , we were limited by the DTGS detector to measure down to 400  $\text{cm}^{-1}$ . A transmission spectrum taken through the CsI substrate next to the particle served as our reference spectrum. The measurements have been performed with an “iN10” and a “Continuum” FTIR microscope (both Thermo Scientific Nicolet). The iN10 operates an internal globar source and requires a sufficiently large sample area ( $>50 \mu\text{m}^2$ ) to obtain spectra with good signal-to noise ratio.

**SRXTM.** Samples #49-1, -4 and #35 were examined at the “TOMCAT” beamline of the Swiss Light

Source (SLS), Villigen, Switzerland (see [9,10] for details).

**Results:** As expected [1,11], all particles analyzed here are either mono-mineralic, or mixtures of, predominantly olivine, pyroxene, and plagioclase, with the occasional occurrence of micrometer-sized metal-rich grains (Figs. 1 and 2). Even the three particles in N<sub>2</sub> could be Raman-examined at many spots through the sample port. They show indeed features of these three major minerals. Samples #158 and #187 show only olivine and #35 olivine and plagioclase signatures. Grain #197 has olivine but also shows pyroxene and plagioclase on top of broad quartz glass Raman features (Fig. 1). The Raman scan of #51 [8] reveals a mixture of minerals with sizes of a few to tens of  $\mu\text{m}$ . None of the grains, as anticipated, showed any feature that could hint at the presence of more pristine LL4 material. Masses and densities of particles #35, #49-1 and -4 and the mineralogy of #51 are discussed in more detail in the companion abstracts [8,9].



**Fig. 2** Cross section from 3D x-ray scan of #49-1 [9]. The brightness corresponds to the x-ray attenuation of the material. The bright spots are metal-rich grains in olivine. Longest dimension ~100  $\mu\text{m}$ .

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**References:** [1] Nakamura T. et al. (2011) *Science*, 333, 1113-1116. [2] Nagao K. et al. (2011) *Science*, 333, 1128-1131. [3] Noguchi T. et al. (2011) *Science*, 333, 1121-1125. [4] Yurimoto H. et al. (2011) *Science*, 333, 1116-1119. [5] Ebihara M. et al. (2011) *Science*, 333, 1119-1121. [6] Ishibashi Y. et al. (2012) *LPS XLIII*, Abstract #2887. [7] Böttger U. et al. (2011) *EPSC-DPS Joint Meeting, Nantes, France*. [8] Böttger U. et al. (2013) *LPS XLIV*, this meeting, Abstract #2092. [9] Meier M.M.M. et al. (2013) *LPS XLIV*, this meeting, Abstract #1937. [10] Alwmark C. et al. (2011) *Meteorit. & Planet. Sci.*, 46, 1071-1081. [11] Yada T. et al. (2012) *Meteorit. & Planet. Sci.*, 47, Abstract #5245.