

# LABORATORY STUDIES OF EXTRATERRESTRIAL ICES – SAMPLE RETURN FROM ICY BODIES

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**Introduction:** The last decades of space exploration revealed the widespread occurrence and underlines the importance of Ices throughout our Solar System [e.g. 1, 2, 3]. Without a detailed know-ledge of Ice formation and evolution a comprehensive model for the development of habitable environments is impossible.

Ices are unique carriers of a number of different chemical tracers, which include isotopic signatures of hydrogen, carbon and oxygen, noble gases, trace element and REE patterns, to mention a few. Furthermore ice can capture, store, preserve and protect organics for extraordinary long time spans [4].

Whereas terrestrial ices are studied in great detail [e.g. 5], despite its importance, comparably little is known about extraterrestrial ice.

However, rapid technical developments in laboratories on Earth will enable a comprehensive analytical study applying traditional as well as novel techniques.

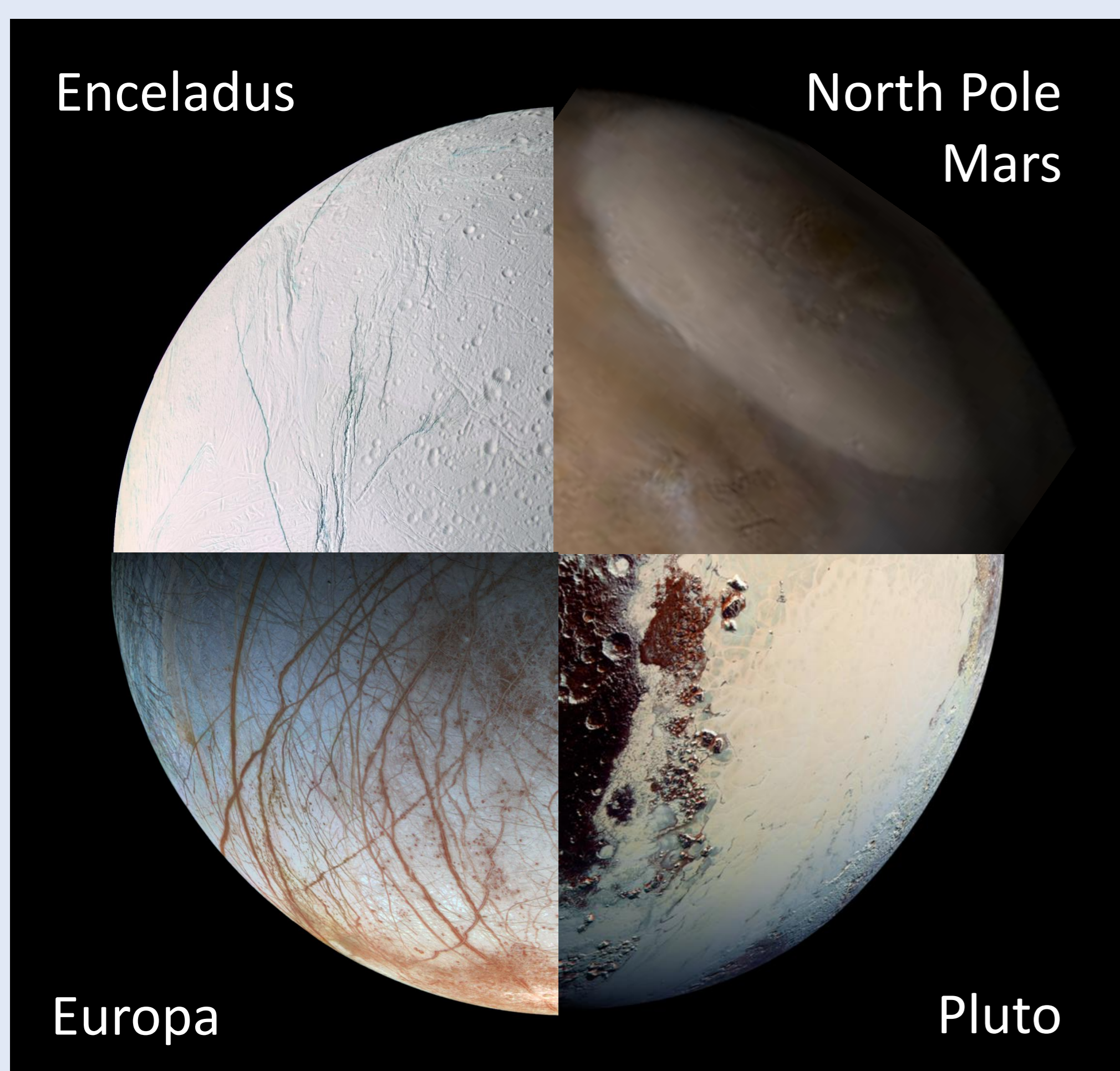


Figure 1: Examples for suitable mission targets, like the Icy Moons Enceladus and Europa, Planetary Ice shields on Pluto and Mars. Courtesy NASA/JPL-Caltech.

**Ice sample return:** Ice is one of the main components of comets. It also exists in dwarf planets [2], at the poles of Mars, in shadow regions of the Earth's moon [6], Mercury [1], it forms the crust of several moons of Jupiter, etc. (Figure 1). Beside the sampling procedure itself the main challenge in ice sample return studies is to keep the ices at very low temperature during capture, transport, re-entry, storage and analyses without ever interrupting the cooling chain.

**Analogue experiments:** Analogue studies on terrestrial ice (Figure 2) already overcome some of these challenges, which include ice core sampling in the Antarctica, transport, storage and electron back scatter diffraction (EBSD) studies using SEM [e.g. 7, 8, 9].

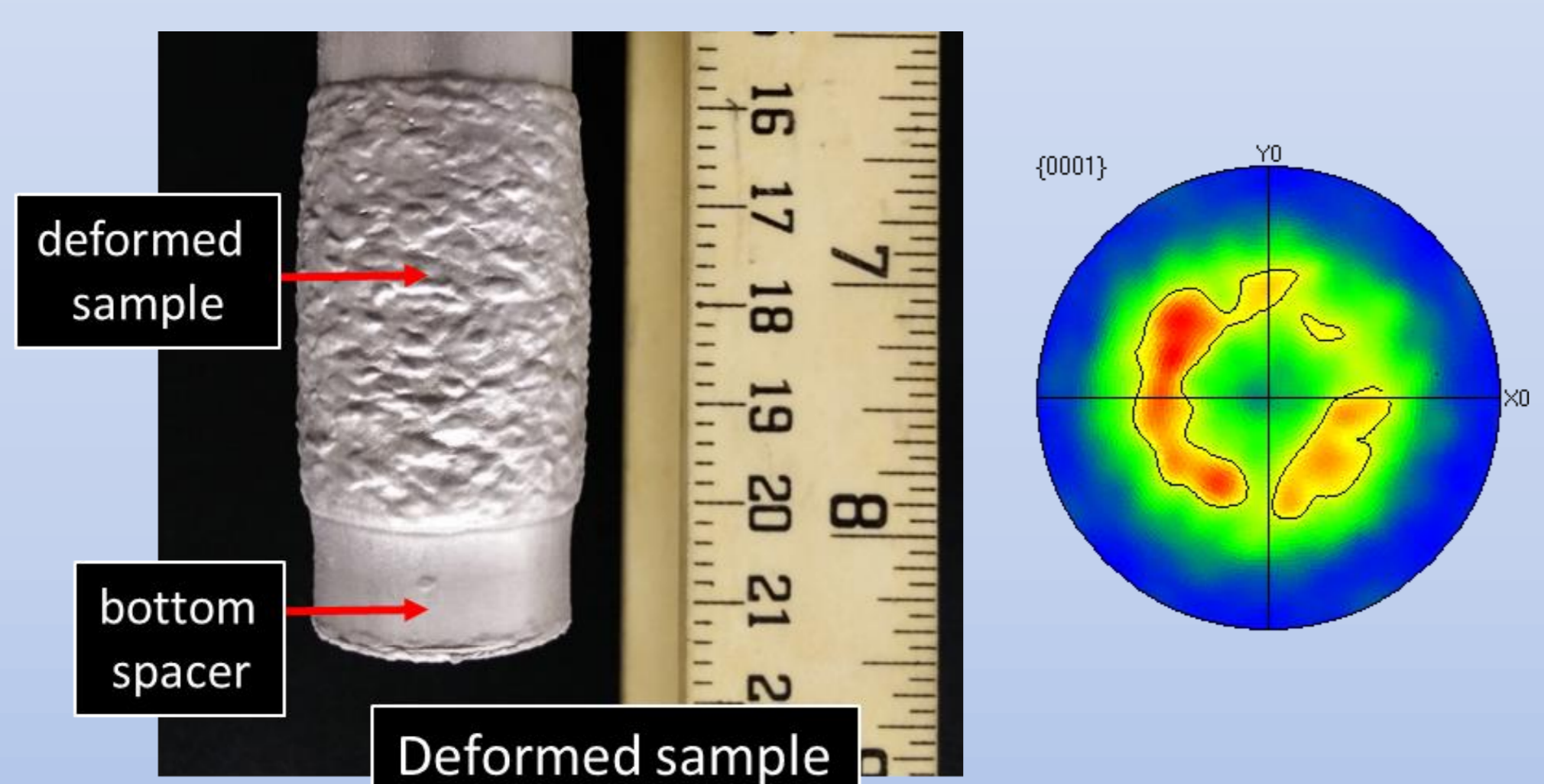


Figure 2: Analogue deformation experiment on synthetic ice samples. Similar natural ice cores can provide detailed information on deformation mechanism, strain and ice dynamics.

**Analytical Techniques:** Non-destructive measurement techniques, like low dose SEM, ESEM, synchrotron XRD and XRF, are scarce. Even if these techniques are applied under the most gentle conditions available, ices - beside organics - remain the most delicate samples due to their very unstable nature.

**Cryo Electron Backscatter Diffraction (cryo EBSD) of Ice:** EBSD turns out to be among the most powerful analytical techniques that SEM introduced to Earth and Planetary sciences about 20 years ago [10]. Just recently it became a routine technique to study water ice [7], (Figure 3) applying several appropriate strategies for reducing beam damage. EBSD allows to determine the structural state, identifying the crystal structure and its respective orientation (LPO) down to the nanometer scale. Based on these data, deformation mechanisms, which control the dynamics within glaciers, Ice shields and the icy crusts of planetary moons, can be identified.

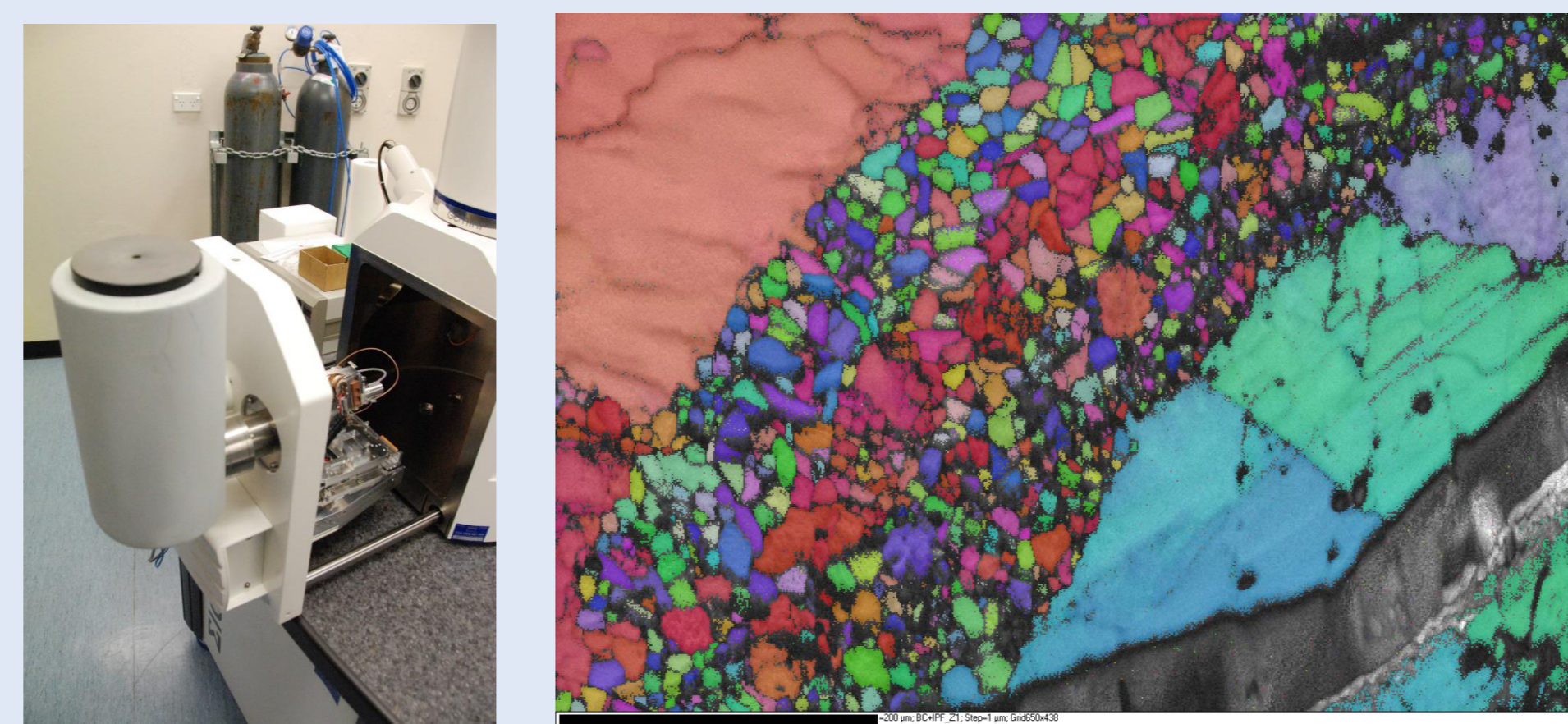


Figure 3: Orientation map of deformed ice in the SEM.

**Synchrotron Techniques:** Synchrotrons around the globe were used to study tiny particles of cometary [11, 12] (Figure 4) and interstellar sources [13, 14, 15] collected during NASA's Stardust mission. Although due to the sample collection procedure it did not include the study of ices yet, it was demonstrated that synchrotron sources are valuable tools to measure the main and trace element content of even the tiniest extraterrestrial particles. The use of synchrotron sources to study terrestrial ices is already established [e.g. 16]. It enables the study of very tiny inclusions of fluids and solids trapped within the ice.

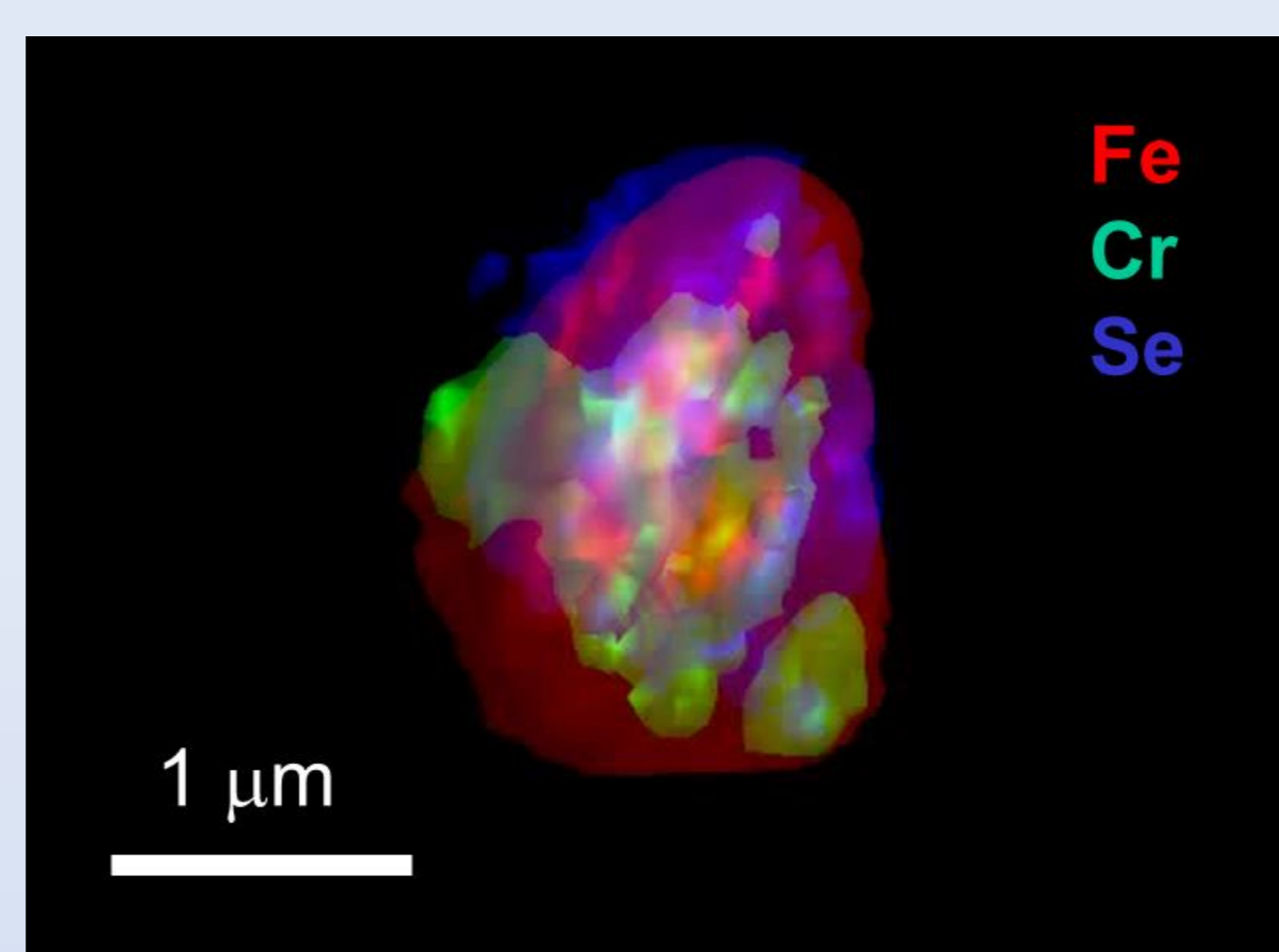


Figure 4: Synchrotron XRF trace element tomography of a cometary particle captured in Aerogel during NASA's Stardust Mission.

The development of new analytical approaches to measure REE-patterns in sub-micron inclusions applying confocal XRF set-ups and energy-dispersive X-ray imaging detectors [17] represent ongoing work in the framework of a long-term project of our group at the PETRA-III synchrotron facility (Hamburg, Germany) (Figure 5).

**References:** [1] Chabot et al. (2015) *Geology*, DOI: 10.1130/G35916.1. [2] Ruesch et al. (2016) *Science*, 353, DOI: 10.1126/science.aaf4286 [3] Stern, S. A. et al. (2015) *Science* 350, 292 [4] M. Becker M. et al. (1997) *Geochim. Cosmochim. Acta* 61, 475–481. [5] Tison et al. (2015) *The Cryosphere*, 9, 1633–1648, 2015. [6] Colaprete et al., (2010) *Science*, 330, 463. [7] Prior, D. J. et al. (2015) *Journal of Microscopy*, 259, 237–256. [8] Kidder, S., & Prior, D. (2014) *Journal of Microscopy*, 255, 89–93. [9] Cyprych, D. et al. (2016) *Earth and Planet. Sci. Lett.* 449, 272–281. [10] Prior D. J. et al. (1999) *American Mineralogist*, 84, 1741–1759. [11] Brownlee, D. et al. [2006] *Science*. [12] Flynn et al., (2006) *Science*. [13] Brenker et al. (2014) *Meteoritics & Planet. Sci.* 49, 1594–1611 [14] Westpfahl et al. (2014) *Science*, 345, 786–791. [15] Gainsforth, Z. et al. (2014) *Meteoritics & Planet. Sci.* 49, 1645–1665. [16] de Angelis et al. (2005) *Geophys. Res. Lett.*, 32. [17] Garrovet et al. (2014) *Analytical Chemistry*, 86, 11826–11832.

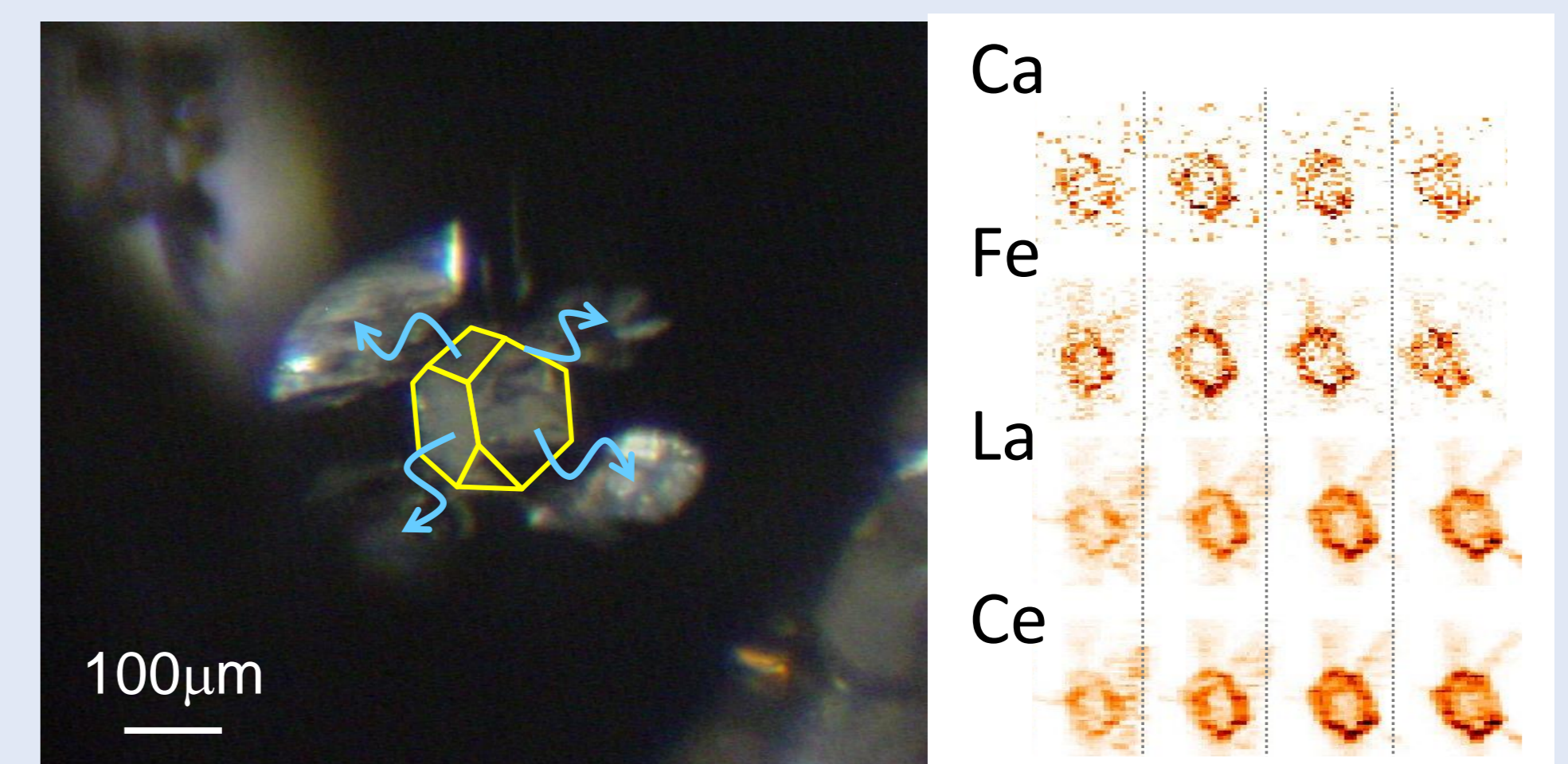


Figure 5: Synchrotron XRF trace element and REE tomography of fluid bearing inclusion in a low Z matrix (diamond). Spatial resolution down to 50nm can be obtained. Large scale routine runs collect data with sub-micron point resolution down to ppm concentrations.

**Neutron Techniques:** Pilot experiments to use neutron diffraction to constrain the kinetics of low temperature phase transformations in water ice, at ambient pressure is part of an ongoing project at the ANSTO Bragg Institute Neutron Beam Instrument to determine the proportions of amorphous, hexagonal and cubic ice that form on super-cooling from small water droplets.

**Secondary Ion Mass Spectroscopy - SIMS:** Isotopic analyses like the D/H-ratio, Oxygen and Carbon isotopic signatures are usually performed by mass spectroscopy. Depending on the required spatial and mass resolution different SIMS instruments including ToF-SIMS, Nano-SIMS and high mass resolution SIMS are available.

**Sample Curation:** The most crucial part in studying Solar System Ices in laboratories on Earth is the need to keep the samples at very low temperatures, safely store and securely send the samples for analyses, which will require a special cryo curation facility (Figure 6).

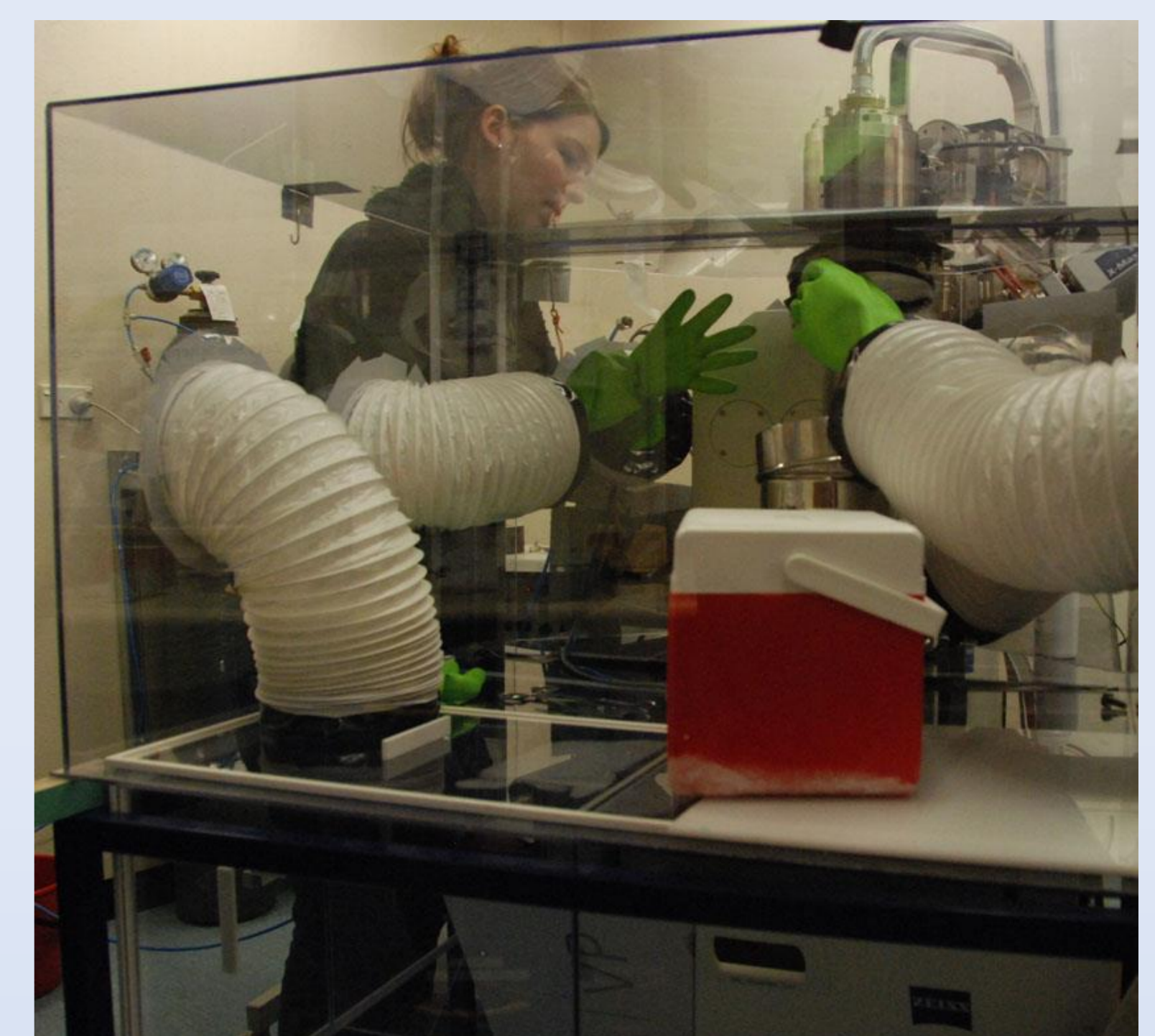


Figure 6: Ice sample curation and handling in nitrogen cooled environment. Shown is the process of loading samples into the Cryo-SEM at the University of Otago.

**Conclusion:** A comprehensive study of solar system Ices is fundamental for the understanding and reconstruction of processes which lead to the formation of our Solar System, its alteration history and finally the formation of habitable environments. Therefore, we predict that an ice sample return mission from either a comet or a moon will represent one of the main challenges within the next decades of solar system exploration.