EURO-CARES

A PLAN FOR EUROPEAN CURATION OF RETURNED EXTRATERRESTRIAL MATERIALS

WORK PACKAGE 4

INDUSTRY VISITS

(DELIVERABLE D4.4)

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1. INTRODUCTION

1.1 General considerations

Many companies propose generic products that are relevant of Euro-CARES activities. However, a few of them have specific cutting edge products that represent significant improvements and anticipate future developments. The visited companies were selected based on such products for the instruments that were identified as priority for an Extraterrestrial Sample Curation Facility (ESCF) defined by the WP4 based on D4.2 and D4.3. This includes notably sample handling tools, optical and infra-red microscopy as well as methods for chemical analysis. More specific products of potential use as secondary priority have also been selected again based on novelty and potential future developments.

Company	**Technique/Product**	**Priority**
Leica	Sample preparation ; Optical microscopy	1 ; 1
Agilent	Infra-red microspectroscopy with focal plane array detector	1
Dynamic Imaging Analytics	3D optical microscopy	1
FEI	Xe-focused ion beam sample preparation	2
Bruker	X-ray fluorescence spectroscopy (XRF) and energy dispersive X-ray spectroscopy (EDS)	1

Table 1. List of visited companies

1.2 Roadmap

Although all companies obey to specific demands, a list of questions that could/should be addressed during the visits has been established.

- What is the main purpose of the instrument? What is flexibility with sampling introduction system?
- What would be the involvement of the company in developing new technology in the framework of H2020 program (including if no funding is available)?
- Are adjustments specific to EURO-CARES possible?
- Can the instrument be located/operated in a clean room?
- Could the instrument be outside and the sample inside the clean room?
- How destructive is the technique?
- What are the maintenance/staff/volume/fluid requirements? Any special needs?
- What is the cost?
- What are the power supply requirements?
- Does the instrument present any risk of contamination?
- Is the company familiar with installing instruments in clean environments?
- How easy is it to fix the instrument or to solve a contamination problem in such a clean environment?

2. AGILENT

Agilent is a company selling mostly conventional gas/liquid chromatography and mass spectrometry products. However, Agilent also has a spectroscopy branch with IR spectroscopy products relevant of EURO-CARES and sample return early characterization. The IR branch development is based in Australia.

FTIR

http://www.agilent.com/en-us/products/ftir/ftir-microscopes-imaging-systems

Contacts France[1]:

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Luc Lebreton, sales representative [luc.lebreton@non.agilent.com]

Agilent France is located in the Courtaboeuf commercial area near Paris, Les Ulis 91. An onsite demo laboratory is in development.

Because the Agilent IR engineer was not available during the period of time convenient for EURO-CARES, a web-conference was organized. As an expert in IR spectroscopy and upon request fromWP4, Rosario Brunetto (CNRS Researcher at the Institut d'Astrophysique Spatiale, IAS) participated at the web-conference. He develops studies of natural/analog extraterrestrial materials using the new Agilent FTIR microscope equipped with a FPA detector and studies the surface of asteroids by IR. He provided valuable experience with the FPA detector and comments from a trained user.

Two Agilent IR products are of interest for EURO-CARES:

Product 1: Topscan 4300

The Topscan 4300 instrument provides handheld ATR IR spectroscopy for macroscopic samples. It is used in geology for rocky samples and can potentially be useful for very rapid characterization of large samples or labware in a contamination control approach. It however requires contact with the sample on ~ mm^2 areas.

[1] All the contact information and prices obtained following these visits are given as of March 2017.

Fig. 1 Example of Handheld Topscan 4300 in the field (left) or for control of material quality (right). (c) Agilent.

Price ~ 35 000 €

Product 2: FTIR Microscope Cary series with Focal Plane Array Detector (FPA)

The Cary FTIR microscope is a rather conventional IR microscope but it is provided with a highly efficient new type of detector, the Focal Plane Array (FPA) detector.

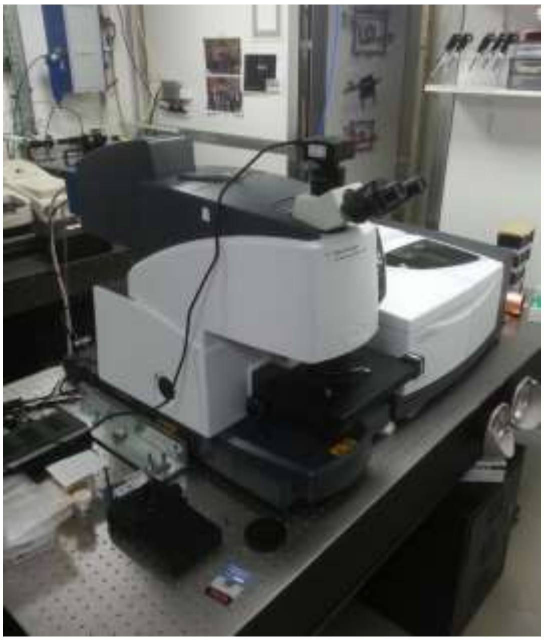

Fig. 2. Cary IR microscope with FPA detector installed on the SMIS beamline at the Soleil synchrotron in partnership with IAS. (c) courtesy of R. Brunetto.

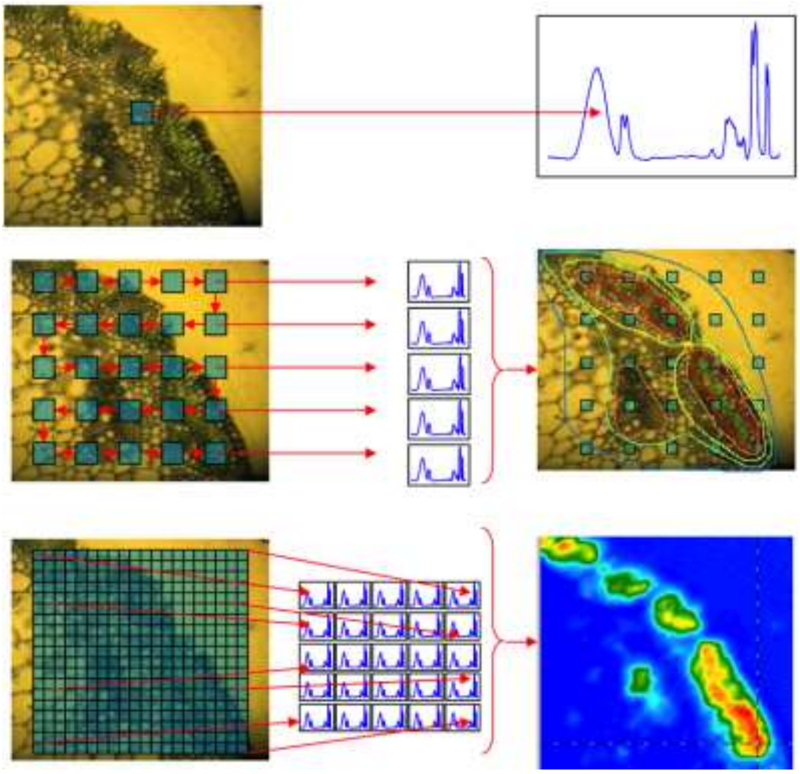

Fig. 3 Principle of the FPA detector. (c) Agilent.

The strength of the FPA detector is to allow the simultaneous analysis of a sample surface by direct imaging using a defocused laser beam. Three analytical modes are thus available instead of two: (1) spot mode analysis, (2) discrete surface analysis by scanning spot analyses as in conventional IR microscopes plus (3) direct imaging analysis (Fig. 3). The strong advantage of the latter technique is the possibility to acquire data very rapidly on a surface by multispectral analysis. Each pixel of the image contains a full spectrum, with the possibility to map only one given spectral region or peak. The pixel size depends on the objective lens used for acquisition (Fig. 4). Three objective lenses are available with different magnification and working distance. At the highest magnification (×25) the pixel size is decreased to 0.66 µm. Conversely, at the lowest magnification (×4) the pixel size is 19 µm but the working distance of 3.8 cm allows micromanipulation and introduction of thick samples. The standard magnification (×15) is considered to be a good compromise with pixel size ~1 µm and a working distance of ~ 2 cm. Note that these objective lenses are quite standard so that they can be replaced by other conventional optical microscopes objective lenses (e.g. Olympus, Leica, Zeiss...). The lateral resolution, apparently below the diffraction limit, is obtained by the projection of the size of individual detector cells by the objective lens on the sample. The FPA detector is currently available with three configurations: 32 × 32; 64 × 64 and 128 × 128 pixels. The possibility to acquire simultaneously a large number of spectra implies a larger counting time for an individual spectrum, hence a much higher sensitivity for a total acquisition time that is significantly reduced.

Fig. 4. Objective lenses available on the Cary IR microscope ((c) Agilent)

The FPA detector has been developed by the US army. Only two companies have agreement to sell it and Agilent has the benefit of a special agreement, which allows fast delivery and improved service.

The whole Cary microscope uses regular laboratory power supply and occupies less than 2 m on bench. Besides this, it requires dry air or dry N_2 to purge the interferometer from atmospheric H_2O, CO_2 etc. and a liquid N_2 tank to refrigerate the FPA detector. Two small liquid N_2 dewars are sufficient to cool down the instrument and operate during 8 hours when started warm. Otherwise 1 small dewar from time to time is enough.

Price ~ 200 000 euros

The FTIR microscope is suitable for a clean room. Agilent has experience of installing materials in clean room and has maintenance engineers with clean room experience. Although potential modifications on the instrument to fit custom requirements in the ESCF is limited to the framework of commercial applications, such modifications may be possible on a case by case basis and need discussions and agreement with the head of the IR microscopy production.

We note that an additional new FTIR development will be released by Agilent for the pharmaceutical industry in 2017, but is still under commercial embargo as the present report is written. Given the effort put on IR microscopy in the past few years by Agilent, upcoming developments will be focused on other methods such as UV microscopy in the next few years. By the time a space mission could be in progress, the interest could have returned to IR so that it is difficult to foresee future developments more than a few years ahead.

3. LEICA

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3.1. Optical microscopy

The optical microscopy branch of Leica proposes a number of highly modular microscopes starting from simple stereomicroscopes. Because individual microscopes can be assembled from a number of catalog parts, every single microscope can be adapted to the user needs. Because of this specificity of optical microscopy, highly customized combined systems involving optical microscopy and other technology such as automated workflows or various types of spectroscopies (UV, IR, Raman...) can be implemented. Examples of such combinations include for instance the Renishaw Raman micro-spectrometers routinely equipped with DM 2500 optical microscopes. Note that complex systems can include optical parts bought independently. In this respect a new product released in 2017 is a combination of optical microscope and LIBS (Laser Induced Breakdown Spectrometry), where samples are shot with a laser under the microscope and the laser plume is analyzed by X-ray spectroscopy. While this was designed for automotive industry and cannot be applied to precious fragile samples not to be vaporized at the ~50 µm scale in a pre-characterization step, this may be useful for contamination control where optical microscopy allows particle counting and size distribution in the size range > 1 µm and LIBS gives the chemistry of the largest contaminant particles. Such developments have been possible because Leica is head of a group that includes other technological companies, which allows easy interactions and development of new products, for instance for particle counting and contamination control.

Another important category of products related to optical microscopy is software development for automated workflows with integrated sample handling, observation and image analysis. Such softwares allow handling data from several microscopes and can be used for correlative imaging. This is already done in a quite routinely manner in biology institutes (e.g. Curie or Pasteur institutes in France) although it is not yet common in material science. Owing to a partnership with National Instruments numerous interfaces are written in Labview. An interesting option for an ESCF is the possibility to have motorized/automated microscopes equipped with HDMI ports and Wifi connection allowing a control via an application. This has for instance been done for smartphones for a student demonstration in a microscopy class but can be developed to perform optical microscopy without touching the samples. This may allow the user to stay outside a cleanroom or outside containment in the case of biosafety.

Further customization and specific designs can be studied and implemented in several different manners: (1) using an integrated approach with outside resources via partnerships with other companies outside the Leica group; examples of such approaches include protocols with Thales or Thorlabs, (2) using a facilitated integrated approach with companies inside the Leica group, (3) by interactions with various Leica branches and (4) eventually by the development of new products. Successful examples of specific designs achieved in various ways include:

- optical microsocpy in a rheology experiment at UPMC, Paris

- a custom optical microscope onboard the International Space Station (ISS)

- Leica has been involved in developing super-resolved fluorescence microscopy beyond the optical diffraction limit that resulted in Chemistry Nobel Prize 2014 to Drs Eric Betzig, Stefan W. Hell and William E. Moerner.

A list of microscopes of possible interest for the ESCF is briefly examined below. All microscopes work with regular laboratory power supply and can be installed on a bench. For contamination issues it is possibility to remove some components (e.g in the ISS microscope), to change, if possible, some mechanical part or the material in which they are made, eventually upon interaction with other Leica branches to find replacement materials or a solution for a specific problem. Leica has an internal process called "special part request" to investigate such special requests not initially planned in the catalog. It is also possible to establish a "blacklist" of unwanted materials in advance and to evaluate whether they can be skipped or replaced.

Size and prices of optical microscopes are variable depending on modularity as numerous options are already commercially available.

- *Stereomicroscopes*

M50-M60-M80 series from 3000 to 50 000 € (depending on software, motorization, light configuration etc.)

- *Optical microscopes*

DM 750-2500 M series: routine optical microscope from completely manual to completely motorized (XYZ + camera)

DM 2700 M: includes all contrast methods (bright and dark field, phase-contrast, differential interference contrast, qualitative polarization, fluorescence). DM 2700 P has a rotative stage. DM 2700 is not motorized

DM6 (Fig 5): 100% motorized microscope (aperture, light distribution, condenser, revolver); all positions and setting can be saved, so that it is possible to repeat an observation in identical conditions even after a long time. This may be an interesting option in an ESCF, where repeatability may be important in a chain of analyses.

DM4: intermediate between DM 2700 and DM6

DM2700 12 000 - 20 000 € (+10 000 to have it motorized)

DM6M 50 000 to 60 000 €

DM4P in between

The price depends also on the quality of the chosen objective lenses

Fig. 5. DM6 fully automated polarized light microscope. © Leica

- *Large sample microscopes*

DM 8000/12000 for wafer inspection in clean room (Fig. 6). This type of microscope could be useful for very large samples (8 or 12 inches diameter, respectively), e.g. for contamination control or size distribution of a sample dispersed on a plate. An elevated version exists for samples thicker than wafers. All contrast methods are implemented, as well as UV light and and transmitted IR light.

Price : 50 000 to 70 000 € with UV and IR light

Fig. 6 DM 12000 for large sample control in clean room. © Leica

DM 2500 MH (Fig 7) combines the advantages of a microscope and binocular stereomicroscope: it has the column and stage of a binocular stereomicroscope with optics and light of a microscope. This design allows the possibility to work on large samples and to increase the working distance, which could potentially be useful for non flat pebble-sized samples or for observation of handling tools.

Price : 15 000 - 25 000 €

Fig. 7. DM 2500 MH. (c) Leica.

Both DM 2500 MH and DM 8000/12000 may be useful instruments for the optical investigation of sample return capsules before opening and sample handling or to observe any sealed container.

- *Inverted microscopes*

DMi8 (Fig 8): An inverted microscope may also be useful to handle and observe samples of any size and shape (reflected light can be used with any sample; transmitted light can be used only for the smallest samples, possibility of UV fluorescence). The arm length can be increased so that the surface of even very thick samples can be examined.

Fig8. DMi8 inverted microscope. (c) Leica

Price: 15 000 to 60 000 €

- *3D optical microscopes*

Two microscopes have been designed to perform 3D topography and rugosity imaging (in association with other microscopy techniques).

DVM 6: Automated digital microscope (only one optical column) useful for instance for quality control. DVM 6 has a hybrid stage partly manual partly motorized. It is sold with image analysis software. It is very stable but not modular. The 3D information is obtained by focus variation.

Price: 30 000 to 60 000 €

DCM 8: Optical roughness meter with bright field microscopy and confocal microscopy. The 3D information is obtained by interferometry and focus variation. The precision on Z is sub-mm and depends on stage size and movement resolution in XY.

Price: 80 000 - 170 000 €

Detailed sample brochures can be downloaded at the following URLs.

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20DCM8/Brochures/Leica_DCM8_Brochure_EN.pdf

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20DVM6/Brochures/Leica_DVM6_brochure_EN.pdf

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20M125%20C/Brochures/Leica_M205A_C_M165C_M125_brochure_EN.pdf

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20DMi8%20ID/Brochures/Leica_DMi8_Ind-Brochure_en.pdf

http://www.leica-microsystems.com/products/light-microscopes/life-science-research/inverted-microscopes/the-leica-dmi8/

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20DMi8%20for%20Documentation/Brochures/Leica_DMi8-Brochure_en.pdf http://www.leica-microsystems.com/fileadmin/downloads/Leica%20DM12000%20M/Brochures/Leica_DM8000_M_DM12000_M-Brochure_en.pdf

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20DM4%20P/Brochures/Leica_POL_Microscopes-Brochure_en.pdf

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20DM6%20M/Brochures/Leica_DM4_DM6_M-Brochure_en.pdf

http://www.leica-microsystems.com/products/light-microscopes/industrial-materials/upright-microscopes/details/product/leica-dm2500-mh/

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20DM2700%20M/Brochures/Leica_DM2700_M-Brochure_en.pdf

http://www.leica-microsystems.com/fileadmin/downloads/Leica%20A60%20S/Brochures/Leica_A60S-A60F_Brochure_EN.pdf

3.2. Sample preparation

Most sample preparation tools developed by Leica concern more specifically advanced specific techniques that are not of first order priority for the ESCF such as preparation of electron transparent sections for Transmission Electron Microscopy (TEM) or extra-high quality surfaces for Electron BackScattered Diffraction (EBSD), Atom Probe Tomography or any of other new analytical methods that requires extremely flat and smooth surfaces. This includes notably ion milling or polishing instruments. Still two sample preparation tools were considered to be of interest for Euro-Cares and are described here. The EM TXP, a sample cutting and trimming system under stereomicroscope allowing high precision cutting and milling and the VCT 500 cryogenic vacuum transfer system.

Product 1: EM TXP

The EM TXP system is a cutting and trimming system installed under a binocular microscope that allows precise sample preparation. Different cutting and trimming tools can be introduced to handle the sample, for instance diamond saw, trimmer, small 25 mm in diameter polishing cloths, microdrill etc. The sample can be rotated 360° and inclined by 30°. A multi-direction stage allows free positioning of the sample. The rotation speed can be adapted from rapid to slow to achieve cutting, trimming or just polishing. The EM TXP is installed at IMPMC as a demo product (Fig. 9).

Fig. 9 EM TXP installed at IMPMC (top two images). Details of the sample chamber (bottom left) and subset of tools (bottom right, (c) Leica).

In the framework of the ESCF, the advantages of such a system are (1) precise cutting of precious samples, (2) handling of small samples, and (3) production of dust mostly within the boxed area for a limited dissemination in the sample handling room. A specially designed vacuum cleaner with HEPA filters can be used for aspiration during cutting if dust needs being removed in the case of dry cutting. Alternatively, any vacuum aspiration system can be installed. For humid cutting or trimming a peristaltic pump allows dust removal. In the end, the production of dust is limited compared to regular sawing or polishing systems. It still produces too much dust for a clean room but is suitable for a "grey" room. It must be noted that it can also be customized using small tools from other companies if necessary. The possibility to change and clean instruments and cloths between each samples limits cross contamination

It costs ~ 40 000 euros total (including diamond, tungsten carbide or boron nitride tools) and works with regular lab power supply. Its size occupancy on the bench is ~30 cm by 30 cm and its height is comparable to that of an optical microscope.

Product 2: VCT 500

The vacuum sample transfer system VCT 500 (Fig. 10) proposed by Leica allows sample transfer under vacuum at cryogenic temperatures from one instrument to another. The VCT 500 model derives from an older version updated in 2015. It is based on a shuttle that holds the sample under vacuum and controlled temperature owing to a cold trap (Cu, Au ring around the sample). The vacuum is achieved in the shuttle once installed on its sample introduction and pumping base (Fig. 11). Custom docks are installed on the instruments of interest to allow transfer from the shuttle to the instruments. The strength of this device is that most of it can be customized based on the user's needs. Custom designs can be done in interaction between the user and Leica.

Three shuttle models are commercially available with different arm length. The pumping system yields a primary vacuum in the VCT 500 with a rotary pump, but once connected to a high vacuum instrument it can hold a secondary vacuum down to 10^{-7} mbar. If necessary, it is possible to measure the vacuum inside the shuttle. The transfer dock can be installed on various instruments (e.g. SEM, electron probe, atom probe, glove boxes etc.) with a custom design basically for any instrument (Fig. 12). 10 different sample holders are available commercially but custom sample holders can also be designed (Fig. 13). A lot of options can be customized for any application, from the arm length to the material in which the sample holder is made.

As of 2017, examples of this transfer system include for instance the CEA (Commissariat à l'Energie Atomique), Saclay, France, where the VCT 500 was installed to transfer radioactive samples between different glove boxes. A primary vacuum was sufficient in this case to maintain a negative pressure gradient so that radioactive samples do not contaminate the ambient air. It has also been installed in biology laboratories to transfer samples between 5 different SEMs. Compared to other companies, the design of the VCT 500 allowed in this case to have a single transfer system connected to 5 docks, one for each SEM, instead of having 5 sample transfer systems. A highly custom installation was the design of an introduction system in partnership with CAMECA-US for the highly specialized Atom Probe instrument. A final illustrating example is the installation of a VCT 500 with enhanced arm length and modified sample holders in stainless steel instead of aluminium for a synchrotron beam line at the ESRF (European Synchrotron Radiation Facility) in Grenoble, France.

In the framework of an ESCF the VCT 500 could easily be installed in a clean room to transfer samples between different instruments or from a clean room to another. Custom design may even allow the direct transfer of samples from the return canister to ESCF instruments. As space and infrastructure are concerned it is necessary to anticipate ~ 1m around each instrument to move and handle the shuttle and its long arm (typically 50 to 70 cm) and enough space in corridors between various instruments. The shuttle itself is only ~ 25 x 15 x 15 cm. Motorized stages may be necessary to move the samples from the dock to the instrument or glove box.

Fig. 10. Sample transfer system VCT 500. (c) Leica.

Fig. 11. VCT 500 connected to the sample introduction and pumping system. (c) Leica.

Fig. 12. Connection of the VCT to the dock installed on an instrument. (c) Leica.

Fig. 13. Example of commercially available sample holders. (c) Leica.

The VCT 500 transfer system cost between ~100 000 euros and ~ 200 000 euros depending on the level of customization. Its pumping system base holds on a bench (~50 cm by 40 cm) and only requires regular laboratory power supply. It is adapted to handling within a cleanroom facility. Its rather smooth surface allows cleaning of its outside parts. If any contamination issue rises, the possibilities of custom design are so that it is possible to have special designs to overcome the contamination problem. Because it is designed to hold a secondary vacuum, contamination issues due to degassing problems are probably very limited. Individual sample holders cost between 1000-2000 euros and 4000-5000 euros depending on complexity and customization level. For instance it is possible to have sample holders with additional protection of the samples.

A general observation with the Leica company and products is that contact is easy and Leica is used to a lot of custom installations and requirements, so that a workflow specific to the ESCF is most likely easy to design. In general, they are used to work with cleanroom facilities and have qualified technicians and engineers. Although they also have commercial obligations they are ready to study new designs and adaptations of their instruments in partnership with scientists, with the idea that even one shot installations could result in future commercial instruments.

4. FEI COMPANY

Xenon ion Plasma Focused Ion Beam instrument (variously called with Xe-FIB or PFIB) is an emerging tool that can be used for cutting of samples hundreds of microns in dimension (Burnett et al., 2015). The Helios PFIB is produced by the company FEI, but our visit was to the University of Manchester where researchers have installed one of the first instruments. This enabled us to view the instrument in routine use. In Manchester, it is situated in the Materials Science department and is mainly utilised for metal milling.

It is similar in concept to a more conventional Ga liquid metal FIB milling system, but it can excavate larger volumes. This is because it can provide a maximum current of 1.□A compared with 65 nA for Ga-FIB. In addition, the use of Xe as a primary beam reduces contamination that is a potential side effect of using Ga FIB and reduces the amount of irradiation damage to the sample from the beam (Burnett et al., in press).

The main function of this instrument is to prepare thin sections for further study, for example for TEM analysis or synchrotron analysis. However, it is also of interest to a curation facility because it has the potential to be used for precise cutting of dust grains and rocklets returned from space in preparation for distribution to external scientists.

According to their website, the Helios PFIB DualBeam enables the user to:

•Achieve Up to 50X higher throughput milling with Xenon Plasma FIB and dedicated recipes and chemistries

•Gain the highest resolution imaging with unique monochromator technology

•Acquire the most reliable and repeatable long term data acquisition with unique 5 axis piezo stage

•Prepare highest quality 3D results with proven and optimized 3D acquisition packages (AutoSlice and View, EBS3)

•Perform the easiest 3D data processing with Avizo software

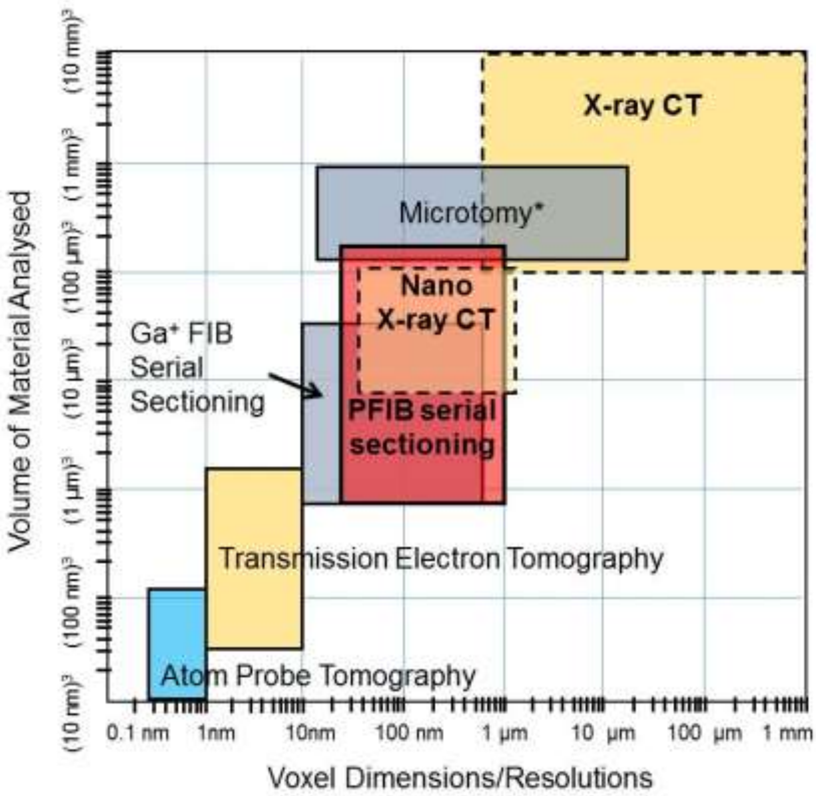

Figure 14. 3D imaging methods for materials science. Non-destructive methods are represented by dashed lines. Microtomy is typically only for soft material [from Burnett et al., 2015].

The superior milling of the XeFIB compared to other focused ion beam instruments enables it to cut samples that are of the order of up to a few hundred microns (Fig. 14). These samples are around 1□m thick and can be used for x-ray beam instrumentation such as XANES.

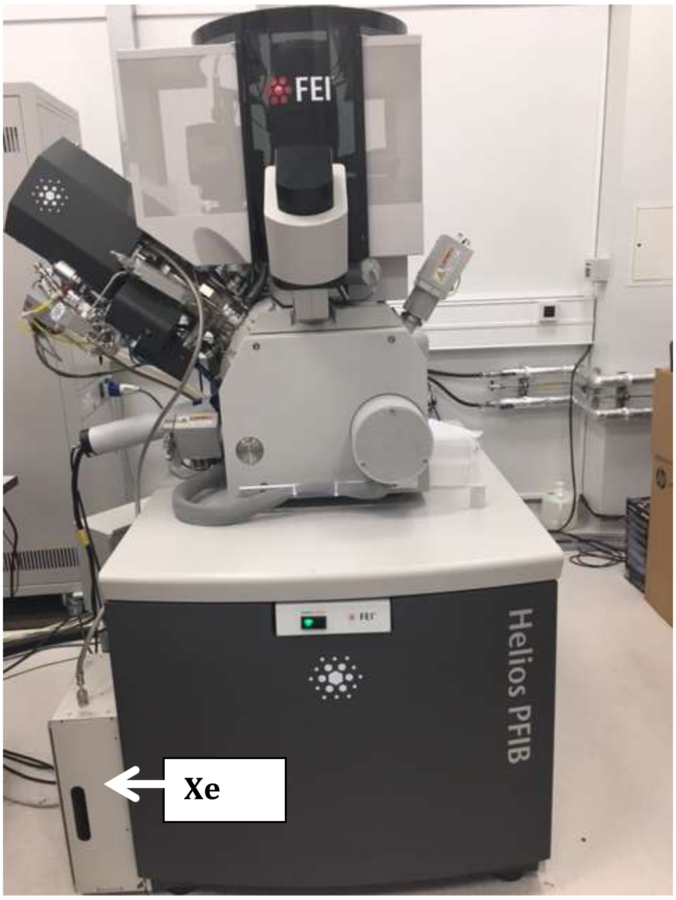

Figure 15. The Helios PFIB.

The instrument is about the same size as a scanning electron microscope, being approximately 2m high (Fig. 15).

Figure 16. The sample chamber of the PFIB.

The sample is introduced to the instrument through a sample chamber (Fig. 16) that is also similar to that of an SEM. There is the potential to incorporate samples with a range of sizes and shapes into the sample chamber.

Figure 17. The computer monitor associated with the PFIB, showing the progress of the milling.

The imaging of the sample is of a very good quality and the progress of the milling is monitored using a screen (Fig. 17).

Requirements: The PFIB requires a Xenon source, which is the form of canisters of Xe gas (Fig. 15). It has no other significant requirements for gas or for excessive power. It operates under a vacuum, therefore it requires pumps. These may be located in another room and the instrument could be located within a clean room environment.

Conclusions: This instrument is potentially useful for an ESCF, performing the role of rock cutting regolith samples, and would be a suitable addition since it can be operated under clean conditions.

References:

TL Burnett, R Kelley, B Winiarski, M Daly, K Mani, PJ Withers, Large volume 3D characterization by plasma FIB DualBeam microscopy, Microscopy and Microanalysis (2015) 21, S3 pp. 2003-2004

T L Burnett, R Kelley, B Winiarski, L Contreras, M Daly, A Gholinia, M G Burke, P J Withers, Large Volume Serial Sectioning Tomography by Xe Plasma FIB Dual Beam Microscopy, Ultramicroscopy (2015) In Press, (doi:10.1016/j.ultramic.2015.11.001)

5. Dynamic Imaging Analytics

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Dynamic Imaging Analytics is a small company that specialises in the development and delivery of high performance imaging solutions. Their approach is built upon a bottom-up understanding of imaging detectors, founded upon the fundamental physics of devices and their main activities include optimizing bespoke camera systems and finding new applications for existing and new technologies. While microscopes camera systems are relatively mature technologies, considerable challenges exist in the photo cataloguing of complex samples that may be returned by future missions, especially fine grained regolith materials where recent advances in light field cameras offer considerable potential benefit to an advanced sample curation facility.

Two key areas of technology development have been identified that have high potential application to future sample curation facilities – light field cameras and Structure from Motion techniques. Indeed, the application of both these technologies combined offer considerable possibilities for advanced 3D documentation of complex samples.

Dynamic Imaging Analytics have expertise in both these areas, and have developed and/or are developing systems incorporating aspects of this technology in a wide range of specialist, very demanding applications from spaceflight instrumentation funded by the UKSA to high speed geometry reconstruction for topflight motorsport companies.

CCD camera systems now provide rapid, high quality data capture at low cost, and can be provided in a wide range of formats and married to an even wider range of optical systems to record excellent images of most materials, samples, object, etc. Control of optical design, reduction of aperture, etc can provide for good resolution images with good depth of field.

However, in microscope systems at higher magnification the depth of field becomes increasingly narrower. The result of this is that when imaging fine grained materials only a very narrow slice of the volume of the material being imaged is in focus (or the image is captured with low resolution). One solution that has been developed in recent

years is to obtain a stack of images for each scene that can then be combined into a single image with suitable software. This can be time consuming and result in errors in the reconstructed image. The light field camera systems now being developed, together with the necessary image processing software are now providing an alternative solution to this problem, and with added 3D information.

Light Field Camera Systems

A light field camera captures information about the light field emanating from a scene; that is, the intensity of light in a scene, and also the direction that the light rays are traveling in space. This contrasts with a conventional camera, which records only light intensity.

New, compact light field cameras use an array of micro-lenses placed in front of an otherwise conventional image sensor to sense intensity, color, and directional information (Fig 18; 19). Each microlens produces an image of part of the main image, the sum of the many sub-images can then be re-constructed to produce a complete image with a much higher depth of field (> 4 times conventional system). In addition to the well-focused image, detailed 3D information can also be extracted from a single image such that the shape of the scene can be determined.

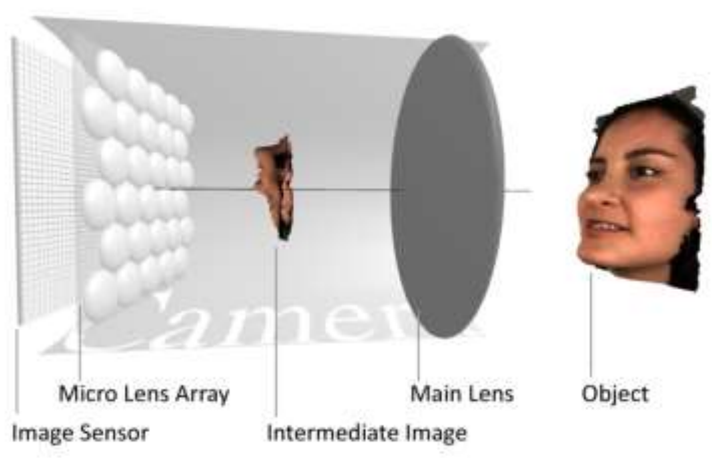

Figure 18. A light field camera creates an intermediate image using a conventional main lens in front of a micro lens array. This image then passes through the micro lens array using a conventional CCD. Image courtesy of Raytrix GmbH.

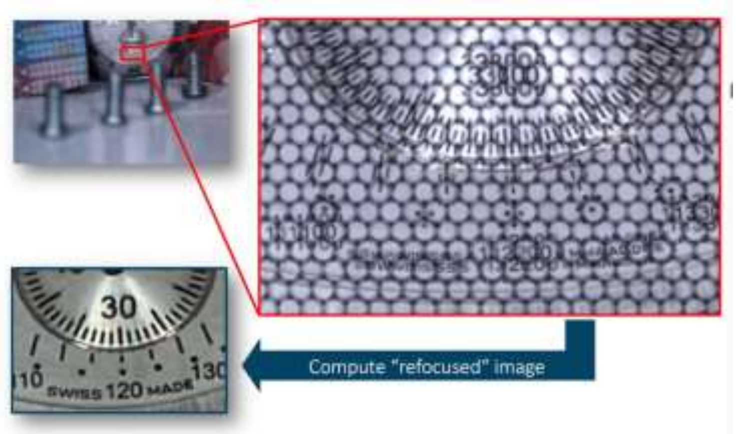

Figure 19. Demonstrates the sub-images created by the micro-lens array, that can then be re-constructed into a single, well-focused image. Courtesy of Raytrix GmbH.

Current Instruments

The existing prototype microscope system employs a Raytrix light field camera (10 Megapixel (2.5 megapixel effective) and provides approx. x100 magnification. The quality of the images has been demonstrated with images of fine grained regolith simulant and chips of martian meteorite (Fig 20).

Figure 20. Total focus images captured with a microscope equipped with a Raytrix light field camera of a chip of a martian meteorite (left) and a lunar regolith simulant (middle). For comparison, the lunar regolith simulant was imaged with a conventional microscope(right) at the same magnification, demonstrating a very narrow depth of field. The field of view for each image is approx. 1.8mm across

The single image files collected can also be used to provide 3D images, although at the present time 3D shape models cannot yet be constructed (indeed, multiple viewing angles would be required for complete coverage).

These images demonstrate that when characterising a sample of regolith with particle size ≤ 1mm it will be possible to rapidly and accurately photo document a large number of grains at suitable resolution to see fine grain textures and structural features (Fig. 21).

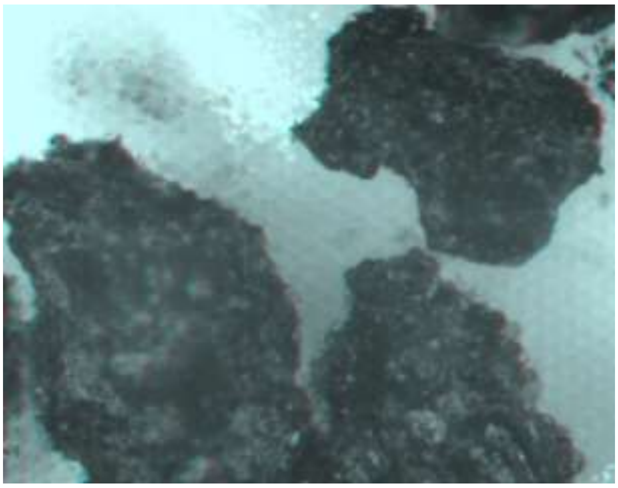

Figure 21. 3D image (view with red-blue 3D glasses) constructed from same image capture that was used to generate the Figure 3 (middle) image.

The current cameras have image capture rates up to 60 fps, but limitations on the computing power to process the images limits real time display to only 8 fps. with conventional microscopes.

The current microscope and camera systems have no moving parts over and above conventional microscope/camera systems and therefore are fully clean room compatible.

The current system, including all the necessary image processing software will cost in the region of €20k each.

Planned Future Technology Development

Progress is being made to improve several aspects of the camera and microscope systems:

1) Improved spatial resolution/depth of field through next generation light field camera optics.

2) Improved processing to provide faster real time video
3) Colour images as opposed to current monochromatic images
4) Multi-spectral version to provide diagnostic information.

The advent of colour images and/or multi-spectral imaging would add much more information to the very early characterization of the samples. This could be useful for screening for certain grain types with specific mineralogy for instance.

The increase in real time video performance could make such equipment be of considerable assistance in the use of such cameras for the manipulation of fine material in high quality clean cabinets or bio-containment boxes where it is necessary for remote viewing handling of the samples. When viewed under the microscope the operator would have a deeper volume to work in without having to continually re-focus the microscope.

L*ong Term Possible Future Development*

The company was very interested in the possibility of developing the existing standalone microscope systems for more complete, high throughput photo documentation.

Building upon existing large scale systems currently being developed for motorsport applications possible systems for a sample curation facility were considered.

To provide full coverage of each particle multiple images would be required. This may be best achieved with multiple cameras positioned in a ring around the sample(s), probably with a minimum spacing of 90˚. In principle this would also allow a 3D shape model with high resolution images to be constructed. Such information could have high value for the curation process, providing mass estimates more accurately and quickly, and providing additional information that may permit details of the internal structure to be more accurately inferred – critical for sample sub-division.

Key developments towards a tool for sample curation would include:

1) Optimising camera/microscope design and positioning

2) Optimising nature of transparent material for mounting the samples for highest quality images as some images obtained through the sample base (low refractive index clear glass mount may be ok).
3) Optimising image registration for regolith-like materials
4) Developing 3D shape models of irregular-shaped samples from the light field camera images
5) Optimising system flow for high sample throughput – e.g. imaging multiple grains on a single mount or developing a mechanism to move samples through camera system with high levels of automation.

6. BRUKER NANO GMBH BERLIN

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For precious samples returned from Solar System exploration missions to asteroids, Mars, the Moon, and comets the relevant microanalytical instruments were discussed focusing on:

(1) TXRF: Bruker S4 T-STAR
(2) Benchtop µXRF: Bruker M4 TORNADO
(3) FEG SEM-EDS: Bruker XFlash FlatQUAD
(4) STEM-EDS: FEI ChemiSTEM

These Bruker instruments are all suitable for use in a clean room environment. They have been developed to be used without the use of liquid nitrogen or other coolants, and they utilise an ordinary laboratory power supply.

The table overleaf summarises instruments for non-destructive, non-invasive analysis and/ or chemical analysis at high spatial resolution.

Instrument	Bruker S4 T-STAR	Bruker M4 Tornado	Bruker XFlash FlatQUAD SDD	FEI ChemiSTEM
Method	Benchtop TXRF	Benchtop µXRF	SEM-EDS	STEM-EDS
Excitation	X-ray source	X-ray source	e^- source (FE-SEM)	e^- source (STEM)
Sample type	solids e.g single grains (liquids, suspension)	bulk samples (drill cores)	bulk samples (drill cores)	electron transparent samples
Preparation	preparation on 30 mm quartz discs	no preparation required	conductive coating, working in low vacuum is not required using ultra low beam currents (< 10 pA)	focused ion beam, ion milling
Spatial Resolution	-	x,y: ~20-50 µm z: >20 µm*	~20 nm to 3 µm*	<1 nm
Elements	Z≥10 (Na)	Z≥10 (Na)	Z≥4 (Be)	Z≥4 (Be)
Detection Limit	ppb – mass%	>10 ppm*	>0.08 mass% (Z≥10)	>0.02 mass% (Z>10)

* Depending on the matrix and analytical conditions

TXRF: Bruker S4 T-STAR

Ultra trace element analysis can be performed on samples e.g. single grains can be analysed non-destructively on quartz discs (Figs. 22-24). No further preparation is required.

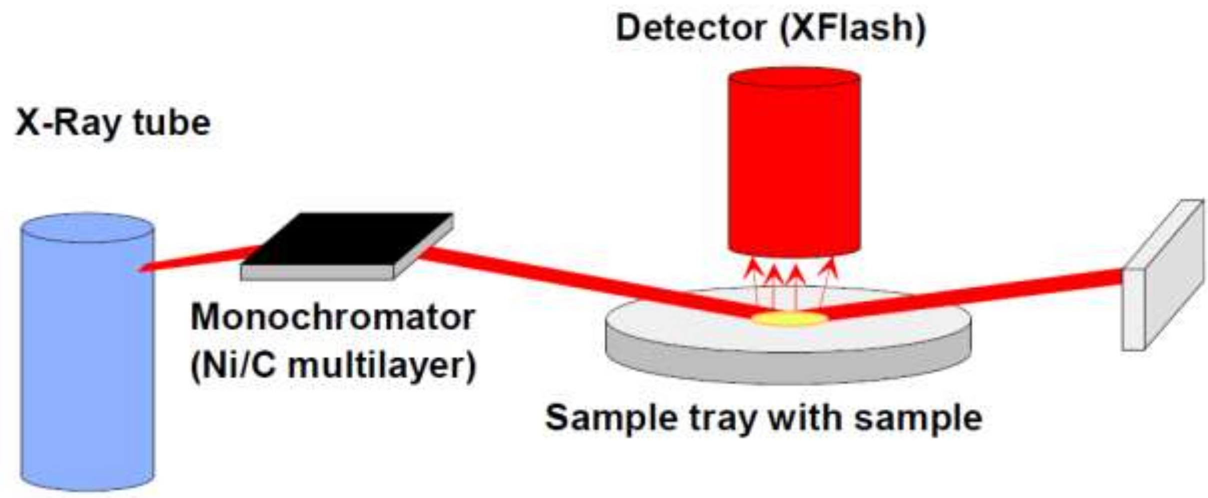


Figure 22. Schematic working principle of TXRF

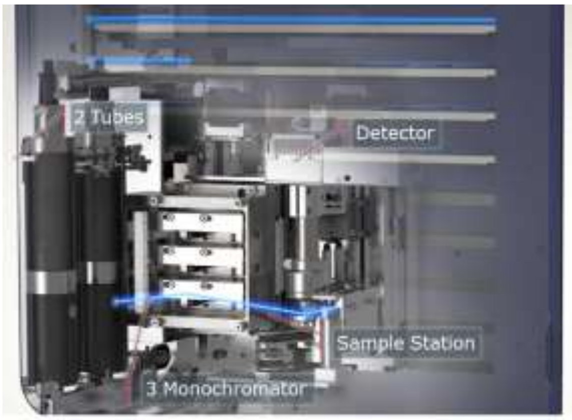


Figure 23 a) Instrument *(b) Setup*

Fig. 24. Sample holder

Relevant references

T. Yu. Cherkashina, S. V. Panteeva A. L. Finkelshtein, V. M. Makagon (2013) *Determination of Rb, Sr, Cs, Ba, and Pb in K-feldspars in small sample amounts by total reflection X-ray fluorescence* X-ray spectrometry 42, 207-212

Wafaa Zaki (2013) Classification of an Unidentified Meteorite Through TXRF Technique and the Chemical Comparison with a Known Meteorite, AIP Conference Proceedings;Dec2013, Vol. 1569, p492

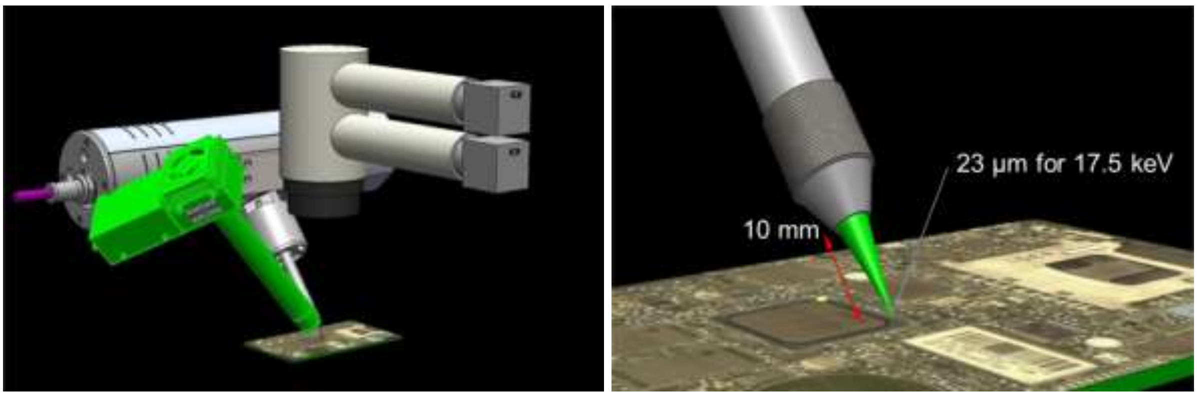
Klockenkämper, R. (1997): Total-Reflection X-Ray Fluorescence Analysis, Wiley & Sons.

Micro-XRF: Bruker M4 Tornado

Using this instrument (Fig 25), non-destructive chemical analysis (mapping, main to trace elements) of samples with rough surfaces is possible in a vacuum chamber. Due to the higher penetration depths of X-ray (compared to SEM-EDX), phases which are located below the sample surface can be identified.

Cost: Approximately €60,000

Figure 25. M4 Tornado (above) and Schematic working principle of TXRF (below)



Application examples

Below is an example spectra of NIST 612 with approx. 500 ppm of more than 20 elements, EPMA (blue) and μ-XRF (red) (Fig 26) . Elements have different excitation probability, and higher sensitivity for heavy elements.

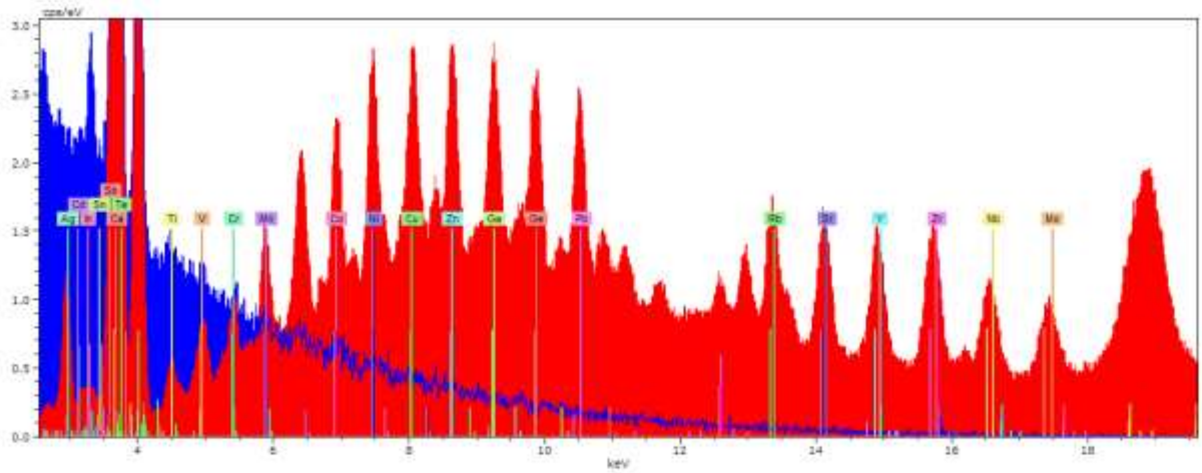


Figure 26. Spectra of multi-element standard.

An example analysis of a BARB5 drill core is given below, showing the heterogeneous distribution of siderophile elements (Figure 27).

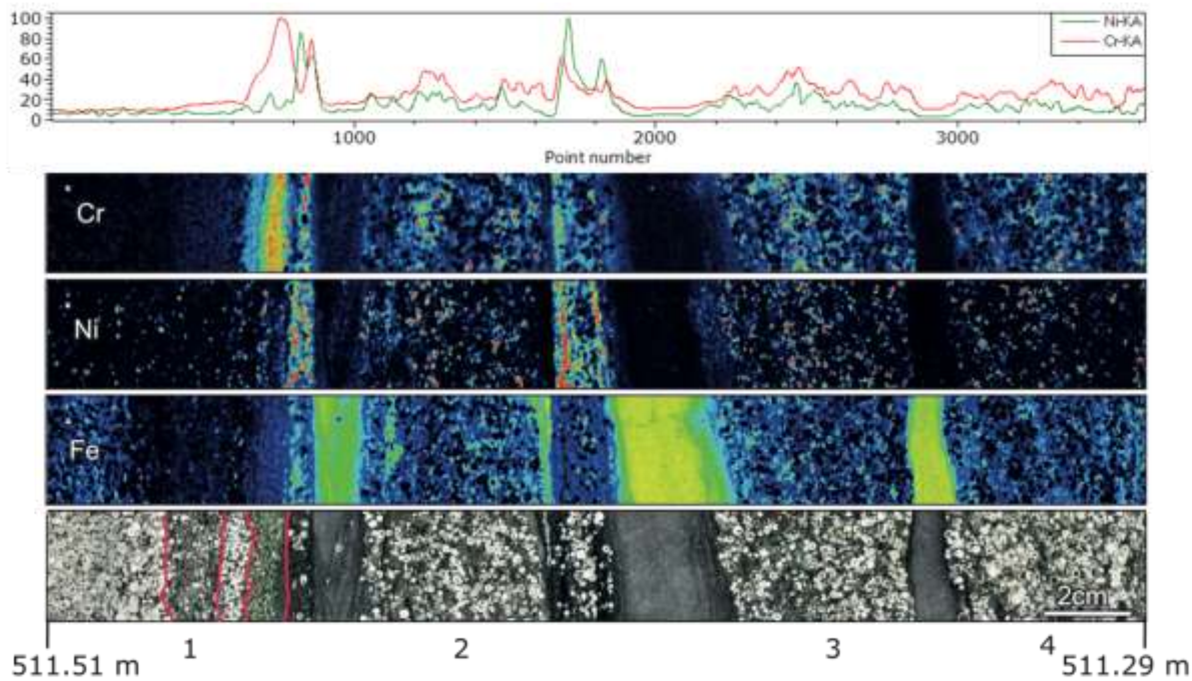


Figure 27. Element images in Cr, Ni and Fe of BARB5 drill core.

FEG SEM-EDS: Bruker XFlash FlatQUAD

Cost: Approximately €100,000

Conventional solid state detectors (SSD) are at an angle to the incident electron beam, which makes them inefficient. The annular BRUKER SDD is inserted between the pole piece of the SEM and the sample (Fig. 28). This geometry allows sufficient data collection on uncoated, beam sensitive and non conductive samples with substantial surface topography using ultra low beam currents under high vacuum. Compared to low vacuum analysis, this approach avoids beam skirting effects. In addition, hydrocarbon contamination is reduced. The possibility to analyse beam sensitive or precious specimens in a close to natural state with little preparation and to study fine scale structures and surface layers allows to preselect samples for further TEM investigations.

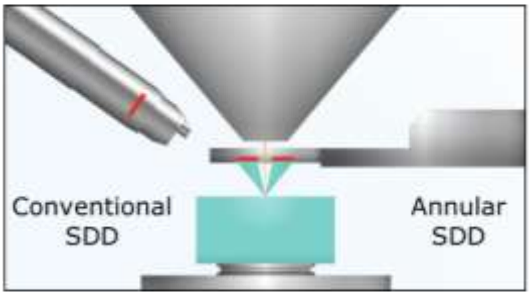


Figure 28. Geometry of the annular SDD compared to conventional SDD

References

Ralf Terborg, Andi Kaeppel, Baojun Yu, Max Patzschke Tobias Salge, Meiken Falke
Advanced Chemical Analysis Using an Annular Four-Channel Silicon Drift Detector
Microsc. Today 25: 30–35.

STEM-EDS

FEI ChemiSTEM (Fig 29) integrates 4 Silicon Drift Detectors (SDDs) very close to the sample area. These detectors are windowless to boost collection efficiency and light element detection capability.

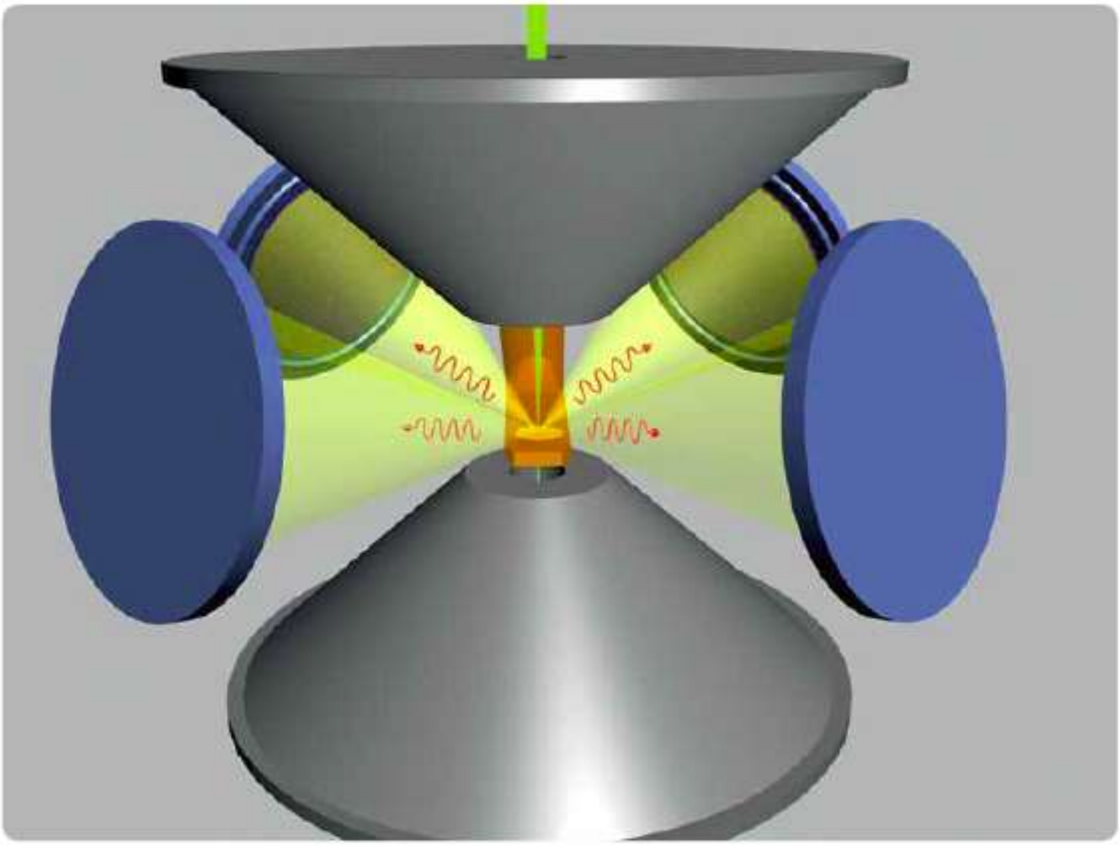


Figure 29. FEI ChemiSTEM Schematic of the ChemiSTEM design, showing the X-FEG high-brightness, Schottky electron source, and the Super-X™ geometry including 4 SDD detectors arranged symmetrically around the sample and the objective lens pole pieces.

Conclusions

Our industry visits enabled us to network with scientists and engineers who are designing the next generation of analytical instrumentation, and we have identified several instruments that would be useful or essential to our ESCF, in enabling the initial characterization of rocklets and regolith, as well as enabling its sampling. Some instruments are also vital for contamination knowledge. In addition, we are aware that over the next few years will see many new developments that will be of advantage to the curation facility. Some of these we cannot discuss in our report for reasons of industrial confidentiality.

Notably, there is considerable expertise within Europe for the minimally destructive physical and chemical characterization of geological samples that is required. A range of companies, from small local enterprises to large global multinational companies, can have a role to play.