Non Destructive Analyses of (Extra-) Terrestrial Materials by Combining Digital Optical Microscopy with LIBS (Element Analyses) and Micro Raman Spectroscopy – A New Approach

V.H. Hoffmann^{1,2}, M. Junge^{1,2}, F.Hentschel², W.W. Schmahl^{1,2}, M. Kaliwoda^{1,2}

¹Department of Earth- and Environmental Sciences, University of Munich; ²Mineralogical State Collection Munich (MSM-SNSB), Munich, Germany.

In this contribution we will present a new approach for the full analyses of highly sensitive or rare, valuable (extra-) terrestrial materials. The method is nearly non-destructive and will significantly reduce the material loss or unwanted influence to the samples / particles under investigation. The approach was specifically developed for returned samples for example from asteroids (Hayabusa 1,2; Osiris Rex, and others) [1,3,4]. It allows to obtain high resolution (in x/y/z) and sensitivity mineralogical data with minimum sample modifications. All investigations can be preformed in atmospheric conditions without any preparation, so directly on the samples as obtained from the various sources.

Due to the significant brecciation and very fine grained matrix / phases, experiments on primitive carbonaceous chondrites are quite complex. Many phases in these primitive space materials are extremely sensitive against (even minor, or local) heating effects, and therefore any kind of preparation (cutting/grinding etc.), specifically in terrestrial atmospheric conditions should be minimized. In order to avoid any such effects we prefer to investigate - whenever possible - naturally broken unprepared sample materials. The representativity of the data obtained on the often small amounts of available sample material was also topic of our studies: large sets of high resolution mappings in 2D/3D can help to overcome the problem of tiny samples / fragments. Our experiences from the earlier investigations on Hayabusa 1 materials (asteroid Itokawa) were highly profitable in this context [2].

(1) Digital Optical Microscopy

Up to now, the surface morphology and mineralogy of the samples is/was pre-investigated routinely by SEM whereby in most cases carbon (or other) coatings are basic requirement. Raman experiments on such samples were then impossible because coatings on rough/raw samples cannot be removed and even more serious, investigating carbon phases was also blocked. High resolution digital microscopy (Keyence VHX950F system) can completely overcome all these severe disadvantages. The technique provides full control of sample materials by pre-selection of particles/areas in 2D/3D for the Raman experiments planed exactly on the same samples as a follow-up step (3). The capabilities of our approach will be demonstrated on a selected set of meteorite samples and terrestrial equivalents. The methodology is very well suited for a fast characterization and classification of tiny samples / fragile materials such as carbonaceous chondrites or returned samples [1-5].

(2) Element Analyses by LIBS

As a next step element analyses can directly be performed with the same instrument on the pre-selected spots or particles by LIBS – LASER Induced Breakdown Spectroscopy. No vacuum, coating or pre-preparation are required which for example means no limitations to sample sizes or shapes. The short-time LASER – material interaction causes only very minor material consumption (LASER spot diameter about 10 microns). Within few seconds the element spectra can be obtained for a large number of elements.

(3) Micro Raman Spectroscopy

Finally the samples or particles under investigation can be transferred to our Micro Raman Spectrometer. As a last step of our approach, a large number of Raman spectra can be obtained by high resolution mapping of the pre-selected spots or particles. We use a Horiba Jobin Yvon Micro Raman Spectrometer (XploRa One) at the Mineralogical State Collection Munich (MSM) for our experiments which is best suited for investigating non-prepared surfaces - to avoid any influence on preparation-sensitive extraterrestrial phases or tiny rare materials. Raman spectroscopy is a mostly non-destructive technique for systematic phase analyses specifically on very small, < 50-100 μ m sized particles or even subsurface inclusions. Performing successful Micro Raman experiments on highly fragile space materials such as carbonaceous chondrites, requires the design of a highly sophisticated experimental setup to avoid or at least minimize alteration effects already during the measurements on the one hand and to guarantee a reasonable signal/noise relation on the other.

Further details and a set of examples will be provided by our iposter contribution.

Acknowledgements

We highly appreciate the support of Th. Koch from Keyence Germany GmbH, specifically concerning the application and detailed test of LIBS on a set of our samples.

References

[2] Mikouchi T. and Hayabusa Consortium, 2014. Earth, Planets and Space, 66/82, 9pp

[3] https://mars.nasa.gov/mars2020/

- $[4] \ https://www.esa.int/Science_Exploration/Human_and_Robotic_Exploration/Exploration/ExoMars$
- [5] Meteoritical Bulletin Database, 09/2023.

^[1] https://www.isas.jaxa.jp/en/missions/spacecraft/past/ hayabusa.html