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The Herakles 3D X-ray Scanner: Concept, Characterization and Application Complementary to Synchrotron Radiation Based Experiments.

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At Ghent University, the Herakles 3D X-ray scanner was developed, creating a unique, state-of-the-art scientific instrument which includes three X-ray based 3D methodologies in a single setup.[1] The scanner incorporates µCT, confocal XRF and XRF-CT analysis. Morphological information is retrieved using the µCT end-station of the instrument with spatial resolutions down to 700 nm, while the two XRF techniques provide the 3D elemental distribution. The confocal XRF setup has a micro-voxel of 30 x 30 x 10 µm³ (xyz), while the XRF-CT has a spatial resolution of 20 µm. Both XRF end-stations have trace-element sensitivity. The techniques are physically integrated using high-precision air-bearing motors, while dedicated control software is developed for the integrated experiments. A typical experiment on the instrument starts at the µCT end-station, yielding a detailed image of the interior of the scanned object. This information is then used to select the desired regions to perform 3D XRF analysis.

For many applications, high spatial resolution (i.e. nano-scale) chemical information is needed. The current state-of-the-art in laboratory based XRF imaging instruments cannot achieve scans with this kind of resolution. However, synchrotron radiation (SR) based experiments at high-end setups such as beamline ID16 of the European Synchrotron Radiation Facility (ESRF, Grenoble, France)[2,3] offer the possibility to use a diverse set of X-ray based analysis techniques with sub-micron spatial resolution (100 nm or smaller). A drawback of SR based experiments is the typically restricted experiment time which is available at these facilities. High-end SR based and laboratory based setups are working at very different levels of spatial resolution, hence they can be highly complementary. Employing this observation, a procedure was developed using the Herakles 3D X-ray scanner to optimize and complement the output of SR experiments by preliminary characterization of the samples, providing a detailed roadmap for the beamtime and aiding in region of interest (ROI) selection and in the meantime yielding valuable bulk information, which would be unfeasible to extract at the SR facility. Clearly, this type of experimental protocol could be extrapolated to other laboratory setups. In this paper we present a case study on microkrystite spherules to demonstrate the impact of combining laboratory and synchrotron experiments on the output of a beamtime.

Abnormally high concentrations of siderophile Ir and other platinum group elements (PGEs, i.e. Re, Os, Ir, Ru, Pt, Rh, Pd) are found in the Cretaceous-Paleogene boundary clay layer at Gubbio (Italy) and Caravaca (Spain).[4,5] These findings were confirmed at various locations worldwide. These geochemical anomalies could stem from the ejection of large volumes of projectile material upon the impact of a large asteroid with Earth's crust.[6] A considerable part of the clay layer consists of so-called microkrystites which could potentially host a fraction of this extraterrestrial PGE component.[5] Microkrystites are small glassy spherules (> 1 mm) stemming from solidified droplets from melt or condensed from vapor. Microkrystites are part of the distal impact ejecta found well away from the crater of large impacts (> 10 times the crater diameter). The current composition of these spherules is mainly defined by replacement products. The

remnant textural characteristics and the alteration products are indicative of the spherules' original compositions. Until today the main composing phases of the microkrystites, consisting of replaced material, received little attention.

The aim during the beamtime at ESRF ID16B was to better understand the distribution of PGEs in a set of Cretaceous-Paleogene boundary microkrystite spherules. A large set of spherules was available for investigation. Due to the restricted measurement time (4 days of beamtime), it was impossible to analyze all of these particles. In a first step to select an optimized data set, maximizing the amount of valuable information present in the chosen samples, a set of 14 microkrystite samples were mounted on a polymer tip for ease of handling during the experiments. Damaged samples were excluded based on optical microscopy. During a next step, all of these mounted particles were intensively scanned using the laboratory setup, using both the µCT end-station and conventional XRF scans at the Herakles 3D X-ray scanner. µCT scans were performed using the W tube at 70 kV and 3 W with 2 µm voxel size (1001 projections of 500 ms), while the XRF scans were performed at the confocal XRF end station, with the Mo tube at 40 kV and 0.6 mA (20 µm step size, 5 s live time).These measurements took quite some time (around 8 h each), however, on this lab instrument the total scanning time was not a constraint. The retrieved data sets were then analyzed carefully, focusing on interesting internal features visualized with the µCT and the elemental signature represented by the sum spectrum of the XRF scans. Using this information, a priority list could be made for the beamtime, making sure the most interesting samples would be scanned first, maximizing the amount of information gathered during the experiment. Furthermore, the data obtained through these preliminary scans could be used to interpret the beamtime data later on. This is demonstrated by the data shown in Figure 1. In the lab, an important fraction of Ni was detected, while the µCT showed some small bright spots dispersed throughout the sample. Ni could be indicative of Ni-rich spinels which were one of the phases of interest in this research. During the ID16B experiments, small, Ni-rich inclusions could be detected, with sizes in the same order of magnitude as the inclusions found via lab-µCT, confirming their identification as Ni-spinels. Furthermore, due to the small beamsize (50 nm) at ID16B compared to the size of the spherules (around 400 µm) the lab-XRF yielded indispensable bulk info, which was unfeasible to obtain during the SR experiment.

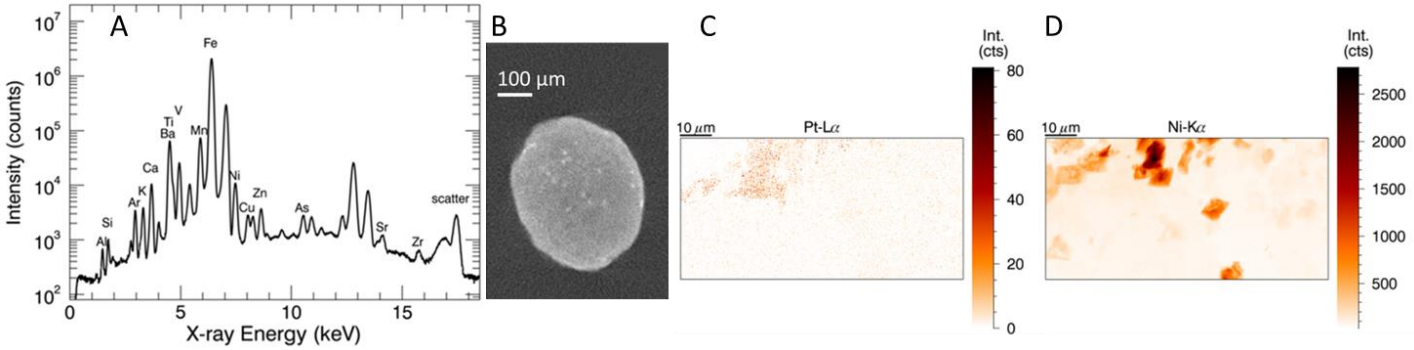

Figure 1 Analysis of microkrystite spherule at Herakles and ESRF ID16B. (A) sum spectrum of lab-XRF scan (40 kV, 0.6 mA, 5 s LT/point) (B) virtual slice of lab-µCT scan (70 kV, 3W, 1001 proj., 0.5 s/proj.) (C + D) Pt and Ni XRF maps at ID16B (17.5 keV, 200 nm steps, 0.1 s dwell time)

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