NON-DESTRUCTIVE IMAGING OF MARTIAN METEORITE CHASSIGNY AND QUANTIFICATION OF PLATINUM GROUP METALS FROM ARCHEAN SPHERULE LAYERS IN THE BARBERTON GREENSTONE BELT, SOUTH AFRICA USING LOW VOLTAGE FEG SEM/EDS

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Introduction: Technological developments in energy-dispersive X-ray spectrometers (EDS), research concerning atomic data and modern data processing invite for challenging analytical approaches. We explain how an annular silicon drift detector (SDD) resolves textures and compositions below micrometre-scale using low accelerating voltages (≤8 kV). A novel methodology: non-destructive, non-invasive X-ray analysis using ultra-low beam current (~10 pA) under high vacuum will be demonstrated using a sample of the martian meteorite *Chassigny* (NHM sample, BM.1985,M173). We further highlight the possibilities and limitations of quantititative analysis of low to intermediate X-ray lines with a spatial resolution of ~60 nm. This will be demonstrated on polished samples of extraterrestrial platinum group metal (PGM) phases hosted by Ni-Cr spinels of the Barberton Greenstone Belt (BGB) [1].

Methods: A chip sample of *Chassigny* was analysed by hyperspectral imaging at 48 nm pixel size, 8 kV and 11 pA. Analysis made use of an annular BRUKER SDD [2] (inserted between the pole piece and sample) fitted to an FEI Quanta 650 FEG SEM. 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.

Quantification of the transition elements is challenging due to significant peak overlaps in the low to intermediate energy range. For the lowest energy range (<1 kV), the absorption edges within the bremsstrahlung background, the energy dependence of the efficiency and the uncertainties of absorption effects result to higher errors [3]. A systematic study on 8 PGM-rich phases 400 - 1200 nm in size from the CT3 drill core has been carried out [4]. Hyperspectal databases were acquired at 6 kV and 77 pA. Quantification was performed with pure element reference samples (L- and M-line families of Ru, Rh, Pd, Re, Os, Ir, Pt: ~2-3 keV; Ni-L, Fe Cu-L: <1 keV).

Results: *Calcite in Chassigny*: Elemental mapping of larger crystals which are associated with shocked silicates revealed thin coatings (Fig. 1A). SE imaging (Fig. 1B) shows a crystallisation sequence and pore space which may indicate shock induced thermal processes. *PGM-rich phases at BGB:* Quantification of maps with 4x4 binning of ~5 nm pixels (Fig. 1C) and area spectra show the presence of Ir, Pt, Ru, Rh, Os, Ni. Chemical zonation patterns will help to better understand formation conditions (exsolution from spinel, precipitation from melt, condensation from the impact vapour plume) [4]. EDS deconvolution algorithms allow the quantification of pure PGM phases with totals close to 100 mass%. Contribution of elements with X-ray lines <1 keV (e.g. Ni-L) leads to higher totals up to 105 mass% due to the inaccuracy of bremsstrahlung background removal and uncertainties of absorption effects.

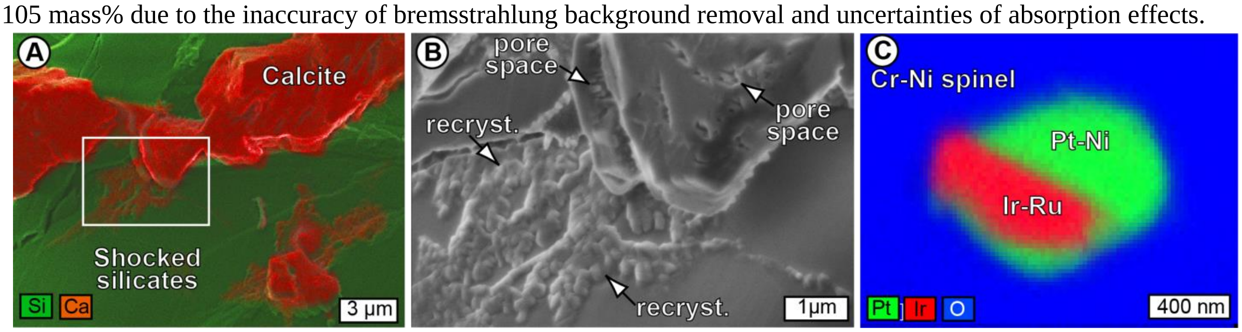

Fig. 1 (A,B) *Calcite in Chassigny*. EDS map (A) overlain with SE micrograph shows thin coating (8 kV, 11 pA, ~4.5 kcps, 48 nm pixels, 17.5 h). SE micrograph (B) of the rectangle shown in (A) reveals pore space in large calcite grain and fine-grained recrystallised domains. (C) *PGM-rich phases at BGB:* Net intensity map reveals zonation with a core of Ir-Ru with minor Rh-Ni and a rim of Pt-Ni rim with minor Ir-Rh (6 kV, 77 pA, ~17 kcps, 87 min).

Conclusions: FEG SEM/EDS at low accelerating voltages and low beam current with an annular SDD provides high spatial resolution and high detection sensitivity without the necessity of applying a conductive coating or working in low 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 will stimulate new approaches for planetary sciences and allows to preselect samples for further TEM investigations.

References: [1] Mohr-Westheide T. et al. (2015) *Geology* 43: 299-302. [2] Terborg R. et al. (2017) *Microsc. Today* 25: 30–35. [3] Pinard P. T. et al. (2015) *Microscop. Microanal.* 21 (Suppl 3). [4] Mohr-Westheide T. et al. (abstract #6127, submitted) *80th Annual Meeting of The Meteoritical Society, Santa Fe, USA, July 2017.*