RECENT ADVANCES IN EDS AND EBSD TECHNOLOGY: REVOLUTIONIZING THE CHEMICAL ANALYSIS OF CHONDRITIC METEORITES AT THE MICRO- AND NANOMETER SCALE. J. Berlin, T. Salge, M. Falke, and D. Goran, Bruker Nano GmbH, Schwarzschildstrasse 12, 12489 Berlin, Germany (jana.berlin@bruker-nano.de).

Introduction: Electron microscopes equipped with energy-dispersive spectrometers (EDS) and/or electron backscatter diffraction (EBSD) detectors are widely used tools in meteorite research [e.g., 1-3]. Within the past decade, silicon drift detectors (SDD) – originally developed for space missions to avoid the use of liquid nitrogen for cooling, have become state-of-the-art in EDS, outperforming Si(Li) detectors in almost every aspect [4-6]. Here, latest advances in EDS and EBSD technology are discussed together with application examples of interest for meteoriticists.

Analysis of light elements: Ultrathin polymer windows on energy-dispersive spectrometers have made the detection of light elements, such as boron and even beryllium possible (Fig. 1a). In combination with the new SDD technology (improved energy resolution: FWHM of Mn-Kα ≤ 123 eV and C-Kα ≤ 46 eV even at 100,000 cps) and extended atomic databases (300 additional L, M and N lines in the low energy range of 0-6 kV [7]), the quantification of light elements has been significantly improved. Peak overlaps can be compensated by deconvolution, such as shown in Fig. 1b for N-Kα and Ti-L.

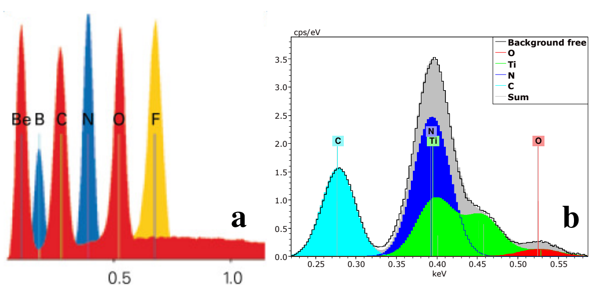

Fig. 1. a) 3 EDS spectra of compounds containing light elements. b) Deconvolution of Ti-L and N-Kα.

Multi detector systems: Because of the compact design and stability of SDDs, it is possible to use multiple EDS detectors on SEMs (Fig. 2a) and even TEMs. With each additional detector, the total solid angle and input count rate increase while the time needed for analysis decreases. This helps especially when samples are sensitive to the electron beam. Another advantage of multi detector systems is the reduction of shadows when dealing with samples that have a rough surface. To demonstrate this, Fig. 2b and 2c show EDS maps of the same area on a broken-off surface of the L5 chondrite Dhofar 446. The map in Fig. 2b was obtained with one detector within 1 hour, while the map in Fig. 2c was obtained with two detectors within 30 min. Analytical conditions were 20 kV accelerating voltage, 6 nA beam current and 100 kcps input count rate.

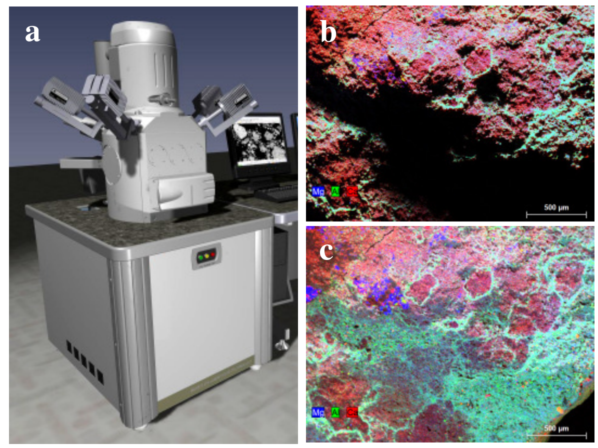

Fig. 2. a) SEM setup with four silicon drift EDS detectors. b+c) EDS maps (Mg=blue, Al=green, Ca=red) of the same area on a broken-off surface of L5 chondrite Dhofar 446 obtained with one XFlash®5010 detector within 1 hour in b) and with two XFlash® 5010 detectors within 30 min in c). A comparison of these 2 maps demonstrates the reduction of shadows when using more than 1 detector.

The analysis of rough surfaces with multiple EDS detectors opens up new possibilities for the analysis of meteorites. For example, potential meteorites can be examined without much time-consuming preparation. Studying the relationships between different terrestrial weathering products on broken-off surfaces, such as shown in Fig. 3, could also be more worthwhile than looking at them in thin section.

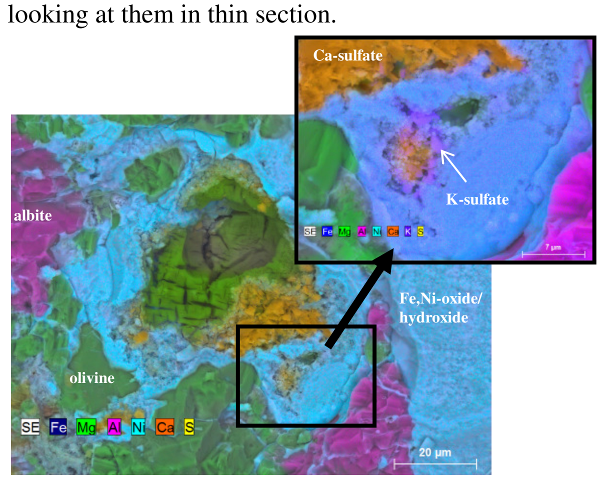

Fig. 3. EDS maps (mixed with SE image) of terrestrial weathering products on a broken-off surface of L5 chondrite Dhofar 446.

Multi-channel detectors: Fig. 4 shows a new type of EDS detector containing four sensors that are integrated on one chip, combined with a novel annular design. This detector is placed between the microscope pole piece and the sample (while the electron beam passes through the hole in the middle), providing optimum collection efficiancy at a maximum solid angle.

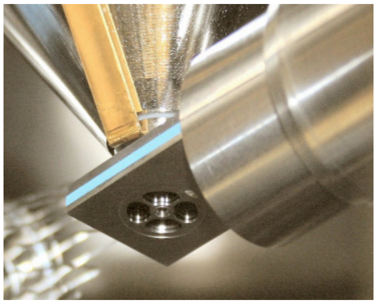

Fig. 4. SEM setup with an annular multi-channel SDD (XFlash® 5060F).

EBSD (and simultaneous EDS): EBSD detectors have become much faster (up to 800 patterns/s at 8x8 pixel binning) and easier to use (motorized insertion mechanism, signal and calibration assistants). A set of three forescattered (FSE) and two backscattered electron (BSE) detectors (Fig. 5a) mounted below and above the phosphor screen (which can easily be replaced), provide valuable additional information. Color coding and mixing of the individual detector signals produces powerful images revealing microstructural details invisible in the grayscale image (Fig. 5b+c).

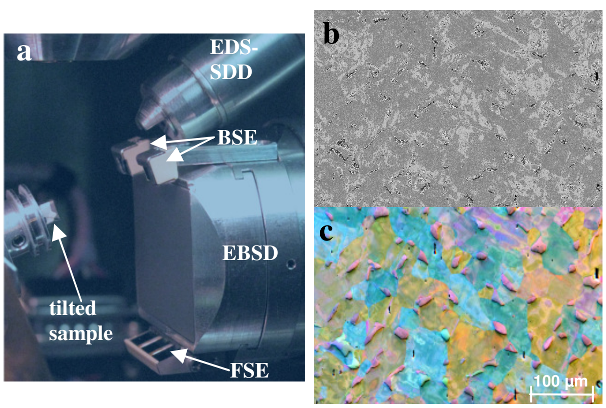

Fig. 5. a) SEM setup with EDS-SDD & EBSD detector (e^-Flash^{1000}). b) Grayscale FSE image of the Cape York iron meteorite. c) Color coded image produced by mixing the signals of the FSE detectors, showing microstructural details.

Furthermore, EBSD has been integrated into the EDS software, allowing the simultaneous acquisition of EBSD and EDS maps (see also setup in Fig. 5a), resulting in better discrimination of the phases if some of them happen to exhibit the same crystal structure.

Silicon drift EDS detectors in TEM: The easier cooling and much lighter weight compared to Si(Li) EDS detectors, makes SDDs highly suitable for TEM [6]. Successful atom column chemical mapping using SDD, Cs-correction and a relatively small beam current [8] proves that SDDs are predestined for atomic resolution instruments, which require high efficiency signal detection with low noise and no artifacts from interference.

Advances in the EDS software - hyperspectral imaging, quantitative maps and "Autophase": Nowadays, EDS intensity (or ROI = regions of interest) maps can be acquired and saved as hyperspectral databases (also called PTS: position-tagged spectroscopy) – a format that allows offline-evalution and quantification of regions of interest. Background-subtracted, quantified maps can be produced within minutes from such databases, also separating elements with overlapping peaks by deconvolution. An "Autophase" tool allows producing phase images (including modal analysis) either with automatic or user-guided phase detection algorithms. Further advances, such as job and stage control allow acquiring large scale element maps (e.g., tiling/stitching of a mosaic map that covers a whole thin section) and to run several different kind of jobs (analyses, linescans, maps) overnight.

Conclusion: Within the past decade, the silicon drift detector technology, advances in pulse processing, integration of EBSD into the EDS software as well as new software features have been revolutionizing the chemical analysis at micro- and nanometer scale.

References: [1] Keil K. et al. (2009) *Microsc. Microanal., 15,* 476-483. [2] Brearley A. (1993) *GCA, 57,* 1521-1550. [3] Watt L. et al. (2006) *MAPS, 41,* 989-1001. [4] Gauthier C. et al. (1994) *Nucl. Instrum. Meth. A, 349(1),* 258-262. [5] Çubukçu H. E. et al. (2008) *Micron, 39,* 88-94. [6] Allard L. F. and Rozeveld S. J. (2009) *Microsc. Microanal., 15 (Suppl 2),* 228-229. [7] Aßmann A. and Wendt M. (2003) *Spectrochim. Acta, 58,* 711-716. [8] Chu M.-W. et al. (2010) *Phys. Rev. Lett., 104,* 196101.