MS39-01 **The interplay between x-ray powder and electron diffraction: the case of experimental petrology.** Mauro Gemmi[a], [a]*Center for Nanotechnology Innovation@NEST, Istituto Italiano di Tecnologia, Pisa, Italy.*
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Multiphase powder samples, containing unknown crystalline phases, require a multi-technique approach to be investigated. Powder x-ray diffraction gives a global information on the sample but, because of the peak overlapping, is unable to identify all the phases, if some of them are unknown. Electron diffraction is complementary to x-ray, each powder grain can be investigated as a single crystal.

Experimental petrology is a perfect field to exploit the combination of these two techniques. The output of a high pressure – high temperature experiment, which simulates conditions of the earth interior, is a capsule of less than $1mm^3$ containing micrometric grains of several phases.

We will show here a combined investigation of electron and x-ray diffraction of samples having different composition in the MgO-Al_2O_3-SiO_2-H_2O system, at P,T condition around 5GPa and 700 °C, a model system for a subducted slab. After a preliminary screening of the sample with the electron microprobe to identify the composition of the larger crystal grains, x-ray laboratory data have been used to check if new unknown phases are present. Those samples showing an x-ray patterns with peaks that could not be indexed have been investigated with the TEM. Three new unknown phases have been identified. The big step forward made by electron diffraction as a structure solution technique due to precession electron diffraction [1] and automated electron diffraction tomography (ADT) [2] allows to use TEM as a single crystal diffractometer with a high chance of success in the structure solution. Two out of three of the identified phases have been solved with ADT. The first phase having ideal composition of $Mg_2Al(OH)_2AlSi_2O_6$ is a new type of hydrous pyroxenes (HAPY) containing water as OH group. Since it has been discovered at 5.4 GPa and 720°C, it can promote the H_2O transport beyond the chlorite breakdown [3]. A second experiment done with a bulk composition tuned on the chemical composition given by the structure solution gave a sample with HAPY as the main phase. A synchrotron radiation diffraction experiment on this sample allows the refinement of the structure. A second phase of unknown chemical composition has been solved with ADT. It is a monoclinic phase made up of a stacking of octahedral layer of brucite type with thetrahedral layers of isolated SiO_4 tetrahedra, a topology completely unknown among minerals. The third identified phase could not be solved, since it is disordered. However, the 3D reconstruction of its reciprocal space with ADT, indicates that this phases is formed by a disordered stacking of octahedral brucite layers and tetrahedral layers.

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MS39-02 **Electron microscopy as seen through the eyes of a powder diffractionist.** Lynne B. McCusker, Christian Baerlocher, *Laboratory of Crystallography, ETH Zurich, 8093 Zurich, Switzerland,*
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Electron microscopy has always been viewed by powder diffractionists as a valuable complementary technique that can be used in difficult situations. For example, a selected area electron diffraction (SAED) pattern can be very useful in resolving unit cell and space group ambiguities, which are common in powder diffraction. Scanning electron microscopy (SEM) images can be used to check for the presence of impurities, which can confuse the interpretation of a powder diffraction pattern, or to see the crystallite morphology, when a preferred orientation of the crystallites in the powder sample is suspected. If it is possible to record a high resolution transmission electron microscopy (HRTEM) image of the material, additional information can be gleaned (e.g. the presence and nature of stacking faults or point defects, which are difficult to characterize with powder diffraction data, or the channel system of a microporous material, which can be useful in the elucidation of the structure from powder diffraction data). It was not until 2006, however, that powder diffractionists recognized that electron microscopy data could also be used actively in the structure determination process [1]. An HRTEM image is a projection of the potential of the structure, and a Fourier transform of this image yields not only the amplitudes, but also the phases of the structure factors of the reflections contributing to that projection. Although a potential map is different from an electron density map, the two are closely related and the phases obtained from the HRTEM image are good estimates of the corresponding phases for the X-ray diffraction case. In other words, the HRTEM image can be used to obtain some starting phase information for structure determination from powder diffraction data. In the solution of three complex zeolite structures, the use of such phases in conjunction with high-resolution powder diffraction data was crucial to the structure determination [1-3]. Of course, it is not always possible to record a good HRTEM image, especially if the sample is beam sensitive. To circumvent this problem, the much simpler precession electron diffraction (PED) technique can be applied. The intensities measured using a PED attachment, while not completely kinematic, suffer much less from multiple diffraction that do those measured in a normal SAED experiment. As a result, reflections that are weak in a PED pattern can also be expected to be weak in the X-ray powder pattern and this information can be used to improve the estimation of the relative intensities of overlapping reflections. Furthermore, the intensities in the PED data have proven to be sufficiently reliable that it is possible to apply the charge-flipping algorithm [4] to estimate phases for the reflections in the projection, and these can be used in the same way as those obtained from an HRTEM image [5]. The fact that less-than-ideal PED intensities could be used for phase retrieval led to the idea of trying to do the same thing with intensities derived from a powder diffraction pattern. Indeed, it proved to be possible to obtain reliable phases from low-resolution, 2-dimensional subsets of the full 3-dimensional data [6]. This approach was applied successfully to the zeolite SSZ-82, which could not be solved directly from the powder diffraction data [7]. The complementarity of X-ray powder diffraction and electron microscopy data is a treasure that will continue to be mined by structural crystallographers.