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EBSD: a powerful technique to obtain crystal and structural data of sub-micrometric mineral samples

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The number of new mineral species yearly submitted for approval to the IMA-CNMCN (Commission on New Minerals, Nomenclature and Classification) in the recent years is stabilized around 80-100 proposals.
Even if crystal data acquisition and structure solution are still widely based on X-ray diffraction (XRD), the number of proposals where electron diffraction techniques are used is increasing. The main reasons of a such relatively recent approach is that the new mineral species often correspond to exsolutions, inclusions or just intergrowths whose dimensions are well below the limit suitable for single crystal XRD, i.e. the dimensions of available single crystals are smaller than 10 microns. However, often new minerals species having dimensions down to few hundred nano-meters are still visible in routine SEM analyses. At this scale handling potential new species to obtain the necessary crystallographic information is still possible using FIB (Focused Ion Beam) to prepare samples for TEM (Transmission Electron Microscopy) and/or EBSD (Electron Backscattered Diffraction) observations.
Both techniques represent the latest technological approaches to sub-micron down to nano-phase characterization. The main differences between FIB-TEM and EBSD techniques rely on instrumentation costs - millions towards hundred of thousand euros, respectively - less sample induced electron damaging for FIB-TEM and time consuming experiments. If in the FIB-TEM case at least two separate instruments are needed, for an EBSD detector mounted on the same SEM-EDS-WDS used for routine analyses one single session theoretically would be sufficient.
On the other hand, as the possibility of exploiting EBSD to obtain crystal and structural data for a mineral sample relies on the availability of a perfectly polished mineral surface and, even more, on a model structure – often represented by a synthetic inorganic compound - for the calculation of simulated patterns, the limitations in this specific field are evident.
An important advantage of EBSD on FIB-TEM is that the analysis of the pattern can be automatically done by "ad hoc" software and, even people "ignorant" of crystallography, can benefit of the results. Instead, with FIB-TEM method the interpretation of the diffracted pattern still largely depends on the crystallographic capability of the investigator. Practically the pattern of Kikuchi lines collected by an EBSD detector is analyzed and indexed by the "ad hoc" software via comparison with oriented patterns calculated from known structures that are contained in a database of some 150,000 crystalline compounds.
In order to show how EBSD is a powerful and sometimes the only available technique to investigate possible new mineral species, the method will be illustrated by several examples among which one sub-micrometric intergrown phase related to NaAlSiO4 and its hydrate equivalent both recently submitted to IMA-CNMCN.