X-ray Fluorescence microscopy and micro-spectroscopy: Multidisciplinary tools

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The X-ray microanalysis techniques follow today the evident trend in the development of nano-technologies by pushing further spatial resolution. Hence, considering the concomitant developments of laboratory instruments and dedicated synchrotron beam lines worldwide, a very competitive context can be anticipated for the coming years. Towards this perspective, synchrotron based analytical techniques (diffraction, imaging and spectro-microscopies) will play an important role by offering unique capabilities in the study of complex systems. Ultimately, this complexity can be envisioned in three dimensions: compositional, temporal and spatial. Typical experiments can be broadly divided into two categories. On one hand, morphological studies, which require high spatial resolution and are, therefore, well adapted to 2D or 3D full-field imaging microscopy. On the other hand, studies dealing with co-localization and/or speciation of trace elements in heterogeneous systems. Scanning X-ray microprobes using various detection modes – transmission and fluorescence - are better suited for the latter cases, which often require both low detection limits and spectroscopic analysis capabilities for chemical composition and chemical state, respectively.

Compared to other techniques, Synchrotron X-Ray Fluorescence (SXRF) microprobes display a unique combination of features. Today, SXRF microprobes using undulator sources provide micron spatial resolution and sub-ppm detection limits for Z>20. When associated with a high collection detection system, the radiation damage is minimal and accurate quantification is possible. Furthermore, the possibility of in-situ experiments remains a unique attribute of synchrotron based analytical methods. Physical penetration of hard X-rays enables specific sample environments to be developed to study realistic systems in their near-native environment rather than model systems. Ability to analyze in-situ in environmental chambers such as high or low temperature, high pressure, or wet cells explains the increasing interest from communities such as Planetary and Earth, environmental science and microbiology. More recently, fluorescence tomography has been developed, where 2-D slices are obtained through a 3-D object without physical sectioning.

Among the 40 beamlines in operation at the European Synchrotron Radiation Facility (Grenoble, France), three beamlines are fully dedicated to X-ray microscopy and micro-spectroscopy techniques in the multi-keV energy range. The main fields of applications are driven by the unique attributes of X-ray microscopy in this spectral range: i) access to K-absorption edges and fluorescence emission lines of medium-light elements and L,M - edges of heavy materials for micro-spectroscopy, chemical or trace element mapping; ii) higher penetration depths compared to soft X-rays allowing imaging of thicker samples; iii) favorable wavelengths for diffraction studies and iv) generally large focal lengths and depths of focus which are advantageous for the use of specific sample environments (in-situ, high pressure, controlled temperatures….).

This presentation will be biased towards sub-micron microscopy developed on the X-ray microscopy beamlines at the European Synchrotron Radiation Facility (Grenoble). Following a brief account on the characteristics of these instruments, strengths and weaknesses of X-ray microscopy and spectro-microscopy techniques in the 1-20keV range will be discussed and illustrated by examples of applications.