Combination of X-ray Absorption with Raman Spectroscopies : at the macro- and micrometer scale

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The use of complementary techniques in Materials Science is a well-established prerequisite for accessing to a deeper structural description of a given material. This approach which offers a simultaneous access to different kinds of information for the same material has great advantages with respect to separate experiments to rid oneself of errors due to differences in sample environment, thermal history, ageing, temperature, sample preparation. Even more important is the possibility to resolve ambiguities in the understanding of processing by allowing accurate determination of the order of occurrence of the chemical and physical events with a high time framing rate. Additionally, the results from one technique can be used as external constraints on the analysis of the other data.

In the past years, we have successfully developed a new experimental approach for studying materials processing based on the simultaneous combination of X-ray Absorption Spectroscopy (XAS) with techniques routinely applied in materials science, such UV-Vis or Raman spectroscopies [1]. Dealing with the combination with Raman spectroscopy, the powerful of such approach has been first probed at the macro-scale with experiments at LURE. Indeed, the flexibility for the focal length of the Raman optics connected by optical fibers to the Raman spectrometer (here the RXN1 model from Kaiser Optical Systems, Inc) makes appreciably easy the design of sample environment to study systems under constraints. Therefore, a lot of systems were investigated by combined XAS and Raman spectroscopies. Among them we can mention, the study of ionic conduction into $K(CF_3SO_3)$-doped polyelectrolytes at the potassium K-edge (3.6 keV), of the oxydation of ethanol by Ce(IV) at the Ce L_3 edge (5.7 keV), of the thermally induced spin transition in the $Fe(NCS)_2(o\text{-}phen)_2$ complex at the iron K-edge (7.1 keV), of Cisplatin based drug release at the Pt L_3 edge (11.5 keV) of the sol-gel transition of zirconia based systems (18 keV), of the rehydration process of Mo-based heterogeneous catalysts (20 keV) and the study of hydrolysis-condensation of $SnCl_4.5H_2O$ in ethanolic medium at the tin K-edge (29.2 keV). Some examples of studies belonging to this list will be presented [1-2] with the main purpose to emphasize how complementary are the information obtained by both techniques. It is noteworthy that such combination is today routinely proposed on the SAMBA beamline at SOLEIL.

Recently, we have succesfully undertaken to combine micro X-ray absorption (µ-XAS) and micro-Raman (µ-Raman) on the LUCIA beamline at SLS. Both techniques allow to give a detailed insight of spatially ill-structured materials at the micrometer scale. The original set-up developed on LUCIA will be presented, and its applications for the study of minerals [3] and of the alteration of samples belonging to cultural heritage will be discussed.

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