Nuclear material investigations by advanced analytical techniques

C. Degueldre*, G. Kuri, A. Froideval, S. Cammelli, A. Orlov, J. Bertsch
LNM, NES, PSI Villigen, Switzerland. E-mail: claude.degueldre@psi.ch
* Corresponding author

Abstract - Advanced analytical techniques have been used at the Paul Scherrer Institute (PSI) to characterize nuclear materials during the last decade. The analyzed materials ranged from reactor pressure vessel steels, Zircaloy claddings to fuel samples. The processes studied included copper cluster build up, corrosion and behavior of PWR and BWR cladding materials as well as fuel defect development. The used techniques were muon spin resonance (muSR) spectroscopy for zirconium alloy defect characterization and hydrogen pickup while fuel element materials were analyzed by techniques derived from x-ray absorption spectroscopy (XAS).

Extensive investigations using advanced analytical techniques applied on nuclear materials have been carried out at the Laboratory for Nuclear Materials (LNM, former Laboratory for Materials Behaviour) over the last 10 years. The materials analyzed comprised reactor pressure vessel (RPV) steels [1], Zircaloys from claddings [2] as well as fuel samples [3]. The processes studied included copper cluster build up with the study of atomic clusters in neutron irradiated RPV surveillance samples by extended X-ray absorption fine structure spectroscopy, the corrosion of claddings by variable incidence angle – X-ray absorption spectroscopy (XAS) and the speciation of zirconium and niobium within the metal and oxide layer of a zirconium niobium cladding segment of a fuel rod corroded and irradiated in a pressurized water reactor (PWR) [4]. The behavior of PWR and BWR cladding material was also assessed by feeling defects in Zircaloy by extended X-ray absorption fine structure and muon spin relaxation (muSR) analyses using muons as probes, and, by muSR measurements on zirconia samples to quantify hydrogen pick up through the corrosion layer [5] using muon as an hydrogen (proton) isotope. In addition, Zircaloy-2 secondary phase precipitates (SPP) were analyzed by scanning transmission X-ray microscopy (STXM) [6] for characterizing Zircaloy SPP at the 20 nanometer level.

Combining the synchrotron irradiation based techniques, i.e. micro- x-ray fluorescence (XRF), micro- x-ray diffraction (XRD) and micro- x-ray absorption fine structure (XAFS) (see Fig. 1a,b,c) allows to reveal for example the local structure of main component atoms in a radioactive zinc ferrite deposit showing how versatile these techniques are for investigating the distribution of elemental, crystallographic and structural features for example in this case of corrosion product deposition on a BWR cladding. In the future, fuel defect analyses in their solid solutions are planed to be investigated for MOX samples. In these studies, XAS is used throughout in a 1D, 2D or even 3D mode. Such investigations are crucial to progress in the understanding of the processes occurring in the materials used in Gen II & III systems.

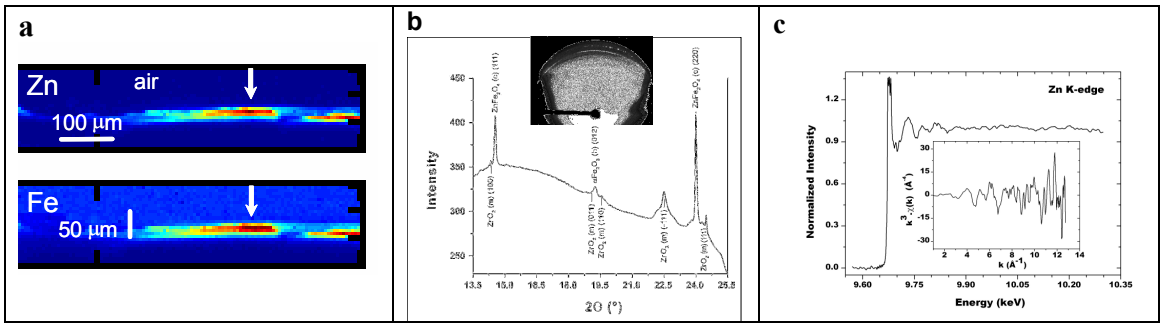

Fig.1: µXRF (**a**), µXRD (**b**) and µXAS (**c**) of Zn enriched spot in the corrosion deposit layer of a BWR cladding .

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