Chapter 52. Nuclear Microprobe Using Elastic Recoil Detection (Erd) For Hydrogen Profiling in High Temperature Protonic Conductors

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Published Online: 26 MAR 2008
DOI: 10.1002/9780470291184.ch52

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How to Cite

doi: 10.1002/9780470291184.ch52

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Summary

The interaction between hydrogen and various high temperature protonic conductors (HTPC) has not been clearly understood due to poor densification and unreacted secondary phases. The melt-processing technique is used in producing fully dense simple SrCe$_{0.9}$Y$_{0.1}$O$_{3-\delta}$ and complex Sr$_3$Ca$_{1+x}$Nb$_{2+x}$O$_{9-\delta}$ perovskites that can not be achieved by solid-state sintering. The possibilities of ion beam analysis have been investigated to quantify hydrogen distribution in HTPC perovskites subjected to water heat treatment. Nuclear microprobe technique is based on the interactions of a focused ion beam of MeV light ions ($^1$H, $^2$H, $^3$He, $^4$He,..) with the sample to be analyzed to determine local elemental concentrations at the $\mu$m$^3$ scale. The elastic recoil detection analysis technique (ERDA) has been carried out using $^4$He$^+$ microbeams and detecting the resulting recoil protons. Mappings of longitudinal sections of water treated SrCeO$_3$ and Sr(Ca$_{1/3}$Nb$_{2/3}$)O$_3$ perovskites have been achieved. The water treatment strongly alters the surface of simple SrCe$_{0.9}$Y$_{0.1}$O$_{3-\delta}$ perovskite. From Rutherford Back Scattering measurements (RBS), both Ce depletion and surface re-deposition is evidenced. The ERDA investigations on water treated
Sr$_3$Ca$_{1+x}$Nb$_{2+x}$O$_{9.5}$ perovskite did not exhibit any spatial difference for the hydrogen incorporation from the surface to the centre. The amount of hydrogen incorporation for Sr$_3$Ca$_{1+x}$Nb$_{2+x}$O$_{9.5}$ was low and required further development of two less conventional techniques, ERDA in forward geometry and forward elastic diffusion $^1$H(p, p)$^1$H with coincidence detection.