The Nuclear Microprobe: A Tool for Light Element Profiling

P. BERGER, P. TROCELLIER, D. BOUTARD, H. KHODJA and J-P. GALLIEN

Laboratoire Pierre SUE (CEA/CNRS)
CEA/Saclay F-91191 Gif s/ Yvette FRANCE

Understanding the properties of solid-state electrochemical devices requires spatial determination of elemental compositions. The chemical potential gradients and differential diffusion rates induce spatial compositional gradients in different scales ranging from a few nanometers to micrometers. As the elemental transport deals mainly with light elements, appropriate analytical techniques are scarce. In this presentation different analytical techniques will be compared and challenges will be presented.

The nuclear microprobe uses the interactions of a focused ion beam of MeV light ions (\(^1\)H, \(^2\)H, \(^3\)He, \(^4\)He, ...) with the target to determine local elemental concentrations at the \(\mu\)m scale. The major analytical techniques related to nuclear microprobe will be reviewed. These techniques usually are based on the spectrometry of the X and gamma-rays and the particles that are scattered or produced by nuclear reactions.

The particle induced X-ray emission (PIXE) technique is suitable for the trace elements with \(Z \geq 11\). In addition, the elastic scattering and associated nuclear reactions are employed for different surface characterization. The Rutherford BackScattering (RBS technique) is pertinent for medium and high \(Z\) elements determination (sensitivity proportional to the \(Z^2\)). In some cases, an enhancement of the cross-sections of diffusion by more than an order of magnitude allows however the use of elastic scattering for the light elements (Particle Enhanced Scattering, PES). The depth resolution capability and compositional changes will be discussed in relation to other techniques. The utilization of energy resonances (sharp increase in cross-sections) will be presented to analyze materials at a well-defined depth.

The nuclear reaction analysis technique (NRA) is also suitable for light elements, especially from Li to F with isotopic selectivity. It has depth profiling capabilities, but usually with a lower depth resolution than RBS. For the very light elements, such as hydrogen or deuterium, the elastic recoil detection analysis technique (ERDA) is preferred to the NRA for its better sensitivity. Its depth resolution capability is essential to evidence surface hydrogen enrichment or to measure shallow depth hydrogen composition without interference from possible surface hydrolysis.

Fig. 1. PES and NRA spectrum induced by 3100 keV protons on Li-Ca intercalated graphite.

These techniques, RBS, PES and NRA, are often performed simultaneously. Figure 1 shows an example of detection of both PES backscattered protons and NRA alpha particles, induced by a proton beam on a lithium-calcium intercalation graphite compound.

As elastic diffusion and nuclear reactions deal with nuclear properties, the measured signals are independent of the chemical bonding. A good accuracy of the determined compositions may then be freely obtained without reference samples or with the help of standards whose composition is not close to the phases to be determined. The compositions are derived from the raw spectra with the use of appropriate simulation programs.