T15 X-ray Photoemission Spectroscopy (XPS)- CEA

TEESMAT
Open Innovation Test Bed for Electrochemical Energy Storage Materials

How it works

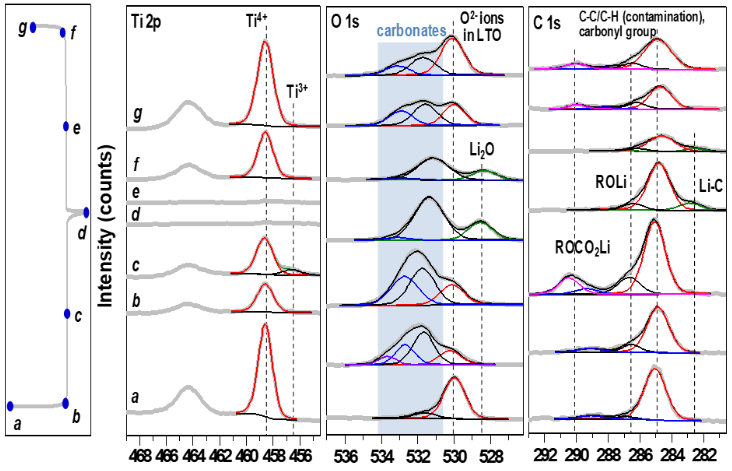
X-ray Photoelectron Spectroscopy or electron spectroscopy for surface analysis (XPS or ESCA), considered as surface sensitive technique (depth analyses is about 50 Å), is one of the most direct means of investigation for the chemical and electronic structure of materials. It consists of the analysis of the electrons emitted by a sample subjected to X-ray radiation. The concept of energy conservation law connect the kinetic energy E_{kin} of the ejected photoelectron k to vacuum with the binding energy E_b or ionization potential. The relation between the photon energy hν and the maximum kinetic energy of the emitted electrons, i.e. the fundamental photoelectric equation can be derived as follow:

$$E_{kin}=h\nu-E_b-q\varphi \ ,$$

where ϕ is a characteristic constant of the sample surface known as the work function.
The main use of XPS is to probe the core level which is a direct probe of the chemical environment of the atoms, hence its oxidation state. XPS is considered as a semi-quantitative technique as it can provide relative atomic concentrations for homogeneous and composite materials. Beside the core level analyses, XPS can provide additional information through the valence band, Auger transition, shake-up and shake-off analyses. In material science, the XPS is nowadays a well-established technique dedicates to study different materials such batteries, catalysis, semiconductors, etc. The main advantage of XPS techniques is the depth analyses probed by XPS allowing the access to the phenomena occurring at the first nanometer of the material surface. Combined to ion gun and gas cluster ion gun, the XPS can provide also chemical depth profiling across several hindered of nanometers.

What can be seen

XPS is now days one of direct and non-destructive tool to probe the redox process and the SEI (solid electrolyte interphase) growth in lithium ion battery. The XPS provide qualitative and quantitative chemical information (oxidation state, chemical bonds) of the elements present at the surface of the electrodes after the electrochemical operating. The figure illustrates the evolution of Ti 2p, C 1s and O 1s spectra recorded at the surface of Li4Ti5O12 electrode materials taken at different cot-off voltage of the first cycle Charge/discharge. We can clearly follow the evolution of lithium insertion and de-insertion through the redox process (Ti4+→ Ti3+) and the evolution of the SEI (formation and dissolution of alky-lithium carbonate). More details are reported in J. Mater. Chem. A, 2014, 2, 631-636.



J. Mater. Chem. A, 2014, 1–6

What kind of sample ?

Thin films, polymer, composite powder, low vapor liquid (ionic liquid)

Why is it useful ?

XPS can be used for sample qualification and control, atomic concentration control (detection limit of 0,1 at%), depth profiling, chemical structure, oxidation state

Investigation time-scale : hours to days depending to the case study

Maturity level : advanced

Grant Agreement
No 814106