

Swarnakshi Halder

CA22M006

Course no- CA5030

Q. Application of xps on industrial technique.

Ans:

X-ray photoelectron Spectroscopy (XPS) is a widely-used technique for surface chemical analysis. In an industrial laboratory XPS can be used for a wide variety of applications ranging from identification of contamination on a surface to characterization of materials as process control, or as a method for characterizing new materials in a research environment.

X-ray Photoelectron Spectroscopy (XPS) is able to easily look at both conductive and non-conductive samples. XPS also has the advantage of being non-destructive, at least to a first approximation, and can quantify the detected elements and also their specific chemistry that is observed on the surface.

An industrial laboratory will see a wide variety of samples: conductors, insulators, polymers, thin films, and powders. Each sample might call for surface chemical analysis to, for example, determine the presence or degree of 'as received' contamination, or to determine if a surface treatment was effectively applied. Thin films might be examined as received or by sputter depth profiling to determine their composition and chemistry as a function of depth. In addition to inorganic thin film materials, XPS is commonly used to look at polymers.

Application of xps technique:

1.Li-ion batteries for mobile energy storage:

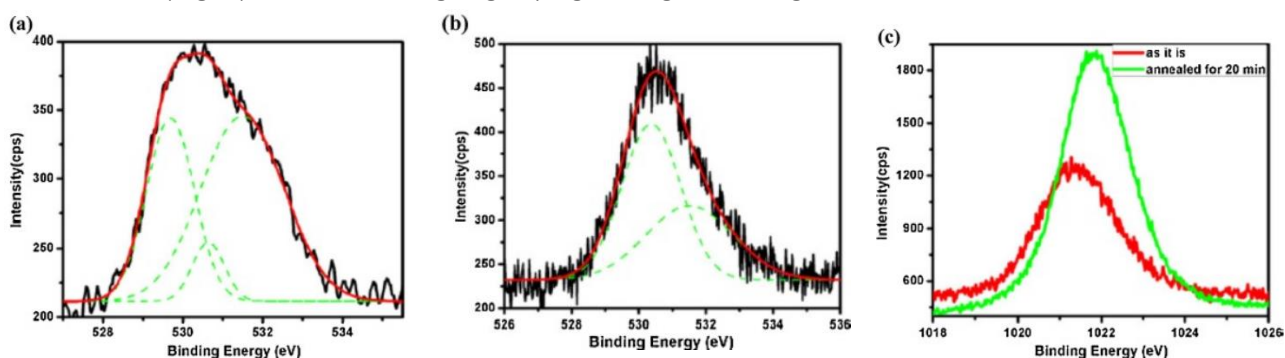
The lithium batteries were prepared with four different amounts (0, 0.05, 0.1 and 0.2 M) of LiPF₆ added dual-salt (LITFSI/ LiBOB) electrolytes with a Li anode and a LiNi_{0.4}Mn_{0.4}Co_{0.2}O₂ cathode. Li anodes obtained from these batteries (cycled several times) were used in XPS measurements. To avoid side reactions with ambient oxygen and moisture, Li metal samples were transported from the glovebox to the XPS chamber in a specially designed sealed container filled by Ar gas. Physical Electronics Quantera scanning X-ray microprobe was used to carry out the XPS analysis. All the excitations were done using monochromatic Al K α X-ray source. XPS measurements were performed at three different depths from the anode surface (0 nm, ~10 nm and ~25 nm) by slowly removing the anode materials by 2 keV Ar ion sputtering rastered over 3 mm x 3 mm area. During the sputtering, the chamber pressure was maintained at 2×10^9 Torr. Sputter rate was obtained by sputtering a 40 nm thick SiO₂ film on Si sample at the same experimental conditions.

The long-standing issue of lithium corrosion and dendrite growth on Li anodes was addressed and *ex-situ* XPS analysis played a crucial role in developing a modification strategy for the electrolyte through the understanding of the chemistry at the SEI layer.

2. Optoelectronic materials:

The effect of annealing ZnO thin films in an oxygen atmosphere was investigated using XPS. The ZnO films were deposited onto borosilicate glass substrates via pulsed DC magnetron sputtering. Annealing the film in oxygen led to an enhancement of electrical properties (conductivity, mobility, carrier concentration) in comparison to those measured for the as-deposited film. This enhancement in carrier transport was linked to reduced grain boundary potential scattering for charged carriers at grain boundaries due to incorporation of oxygen in oxygen-deficient regions.

The O $1s$ spectrum of the as-deposited film (Fig. a) was deconvoluted into three features: a peak at ~ 532 eV due to surface carbonyl and hydroxyl species, a peak at ~ 530.7 eV due to partially associated O^{2-} in oxygen deficient regions, and another peak at ~ 529.7 eV due to O^{2-} tetrahedrally bonded with Zn^{2+} corresponding to wurtzite ZnO. Annealing the ZnO thin film in an oxygen atmosphere for 20 min led to the disappearance of the spectral feature at ~ 530.7 eV (Fig. b). In addition, a shift of the Zn $2p_{3/2}$ peak position to higher binding energy was observed (Fig. c) after annealing, signifying stronger binding between Zn and O.



3. Biosensors:

XPS survey scan of the paper membrane dispersed with imidazole and SDS revealed the presence of photoelectron peaks corresponding to Na in addition to the C and O signals observed in the XPS analysis of the paper membrane. The Na $1s$, S $2p$ and C $1s$ spectral regions of both the blank paper and the imidazole and SDS-functionalized paper membrane point to substantial sample charging, due to which it becomes difficult to decipher if the functional groups are chemically bonded or physisorbed in the surface of the paper membrane. Since physisorption is typically through relatively weaker Van der Waals forces that are easily broken, the functional groups might desorb gradually over time, leading to decreased sensitivity of the POC biosensor.

Sathiyathan Felix et al. developed a non-enzymatic electrochemical sensor based on Cu_2O for glucose sensing. Since the electrocatalytic property of a sensor based on Cu materials strongly depends on size, distribution and shape, Cu_2O were synthesized in the form of

nanocubes using a one-pot wet chemical route with L-ascorbic acid as a reducing agent. XRD analysis of the nanocubes revealed the presence of phase pure Cu_2O . Based on the XPS analysis of the Cu $2p$ spectral region of the nanocubes, the presence of Cu^+ observed. However, the Cu $2p_{3/2}$ spectral feature appears to have a shoulder at higher binding energy, and a shake-up satellite peak that is typically associated with Cu^{2+} is also observed in the Cu $2p$ spectral region, suggesting the presence of some CuO , possibly in the surface of the nanocubes.