Application Note #2005

Analyzing Fluorine In-Situ During Semiconductor Manufacturing

Fluorine is the most reactive chemical element and the lightest of the halogen group. It is an essential raw material for semiconductor manufacturing. Fluorine compounds are used for a variety of applications including chemical vapor deposition, plasma etching, and cleaning.

A challenge in using fluorine compounds for applications like these is the ability to measure it during manufacture, especially as a residual. The semiconductor industry needed to find a solution. Handheld XRF was proposed for its portability, in-situ measurement capability, ability to provide fast results, and its nondestructive nature.

Most handheld XRF analyzers can measure elements as light as magnesium up to those as heavy as uranium. However, analysis of low atomic number elements like fluorine (#9) are challenging for most handheld XRFs.

Consequently, Bruker's advanced TRACER 5g handheld XRF was selected as the optimal solution for in-situ fluorine analysis. The TRACER 5g is specifically designed to dramatically improve sensitivity for light elements enabling measurements of sodium and fluorine, as well as for lower concentration measurements of magnesium and aluminum.

Non-Destructive Fluorine Analysis with TRACER 5g

Handheld X-ray fluorescence (XRF) analyzers are fast, multi-element analyzers which are straightforward to use and can be taken anywhere testing is needed, including the clean room. For light elements, such as fluorine, the simple procedure is to place the analyzer nose-down to the sample with the window removed, and to purge the nose with helium. The capability of the TRACER 5g handheld XRF to measure fluorine is quickly becoming critical for semiconductor manufacturing applications.

For instance, yttrium oxyfluoride (YOF), a plasma-resistant material, is replacing yttrium oxide (Y_2O_3) as the protective material in plasma process chambers for the manufacture of 3-D stacking circuits. It is critical to detect fluorine to differentiate which yttrium compound is being used. The TRACER 5g is capable of this fluorine measurement.

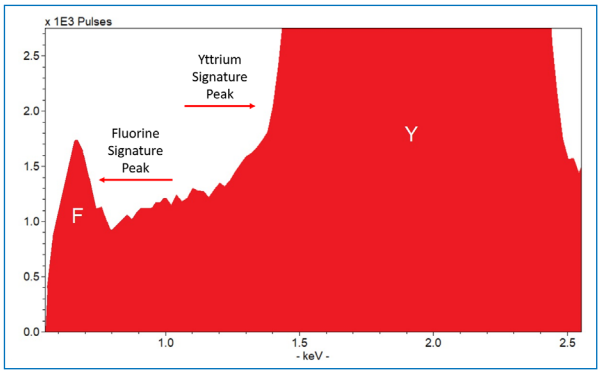
Fluorine Analysis with TRACER 5g Handheld XRF

Fluorine is a very low atomic number element, which means the XRF signal is very weak and absorbed by all materials including air. Typical XRF measurement depth of fluorine is only about 1µm, which means the measured surface needs to be clean and representative of the measured material. To get a strong enough XRF signal it is necessary to remove air and other materials absorbing the fluorine signal between sample and the detector:

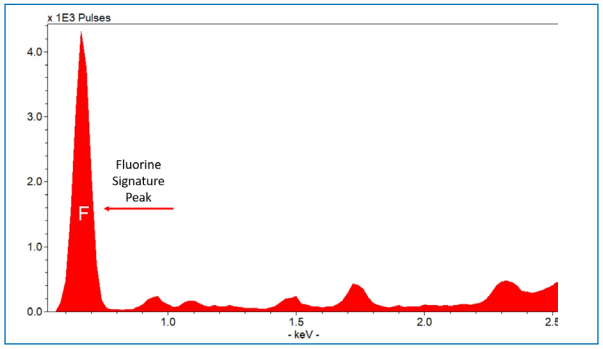
- The instrument protective window and any other film between the instrument detector and sample needs to be removed
- The instrument nose needs to be purged with helium, which absorbs the fluorine signal much less than air (N_2 , O_2 , Ar)
- In practice, this means an effective fluorine measurement requires a solid sample with a reasonably flat surface

Below are spectra of measurements of yttrium fluoride (YF3) coating used in the semiconductor industry and polytetrafluoroethylene (PTFE) tape with a TRACER 5g. The measurement conditions were as follows: 10kV / 100 µA; 30 sec measurement time; no filter; no protective window; industrial-grade He >99.9% purity.

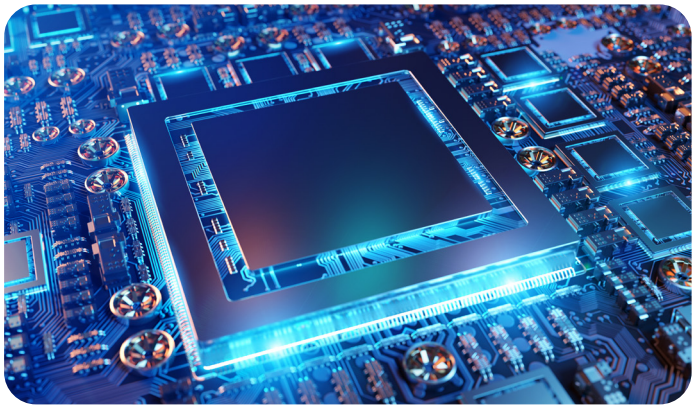
Fluorine limit of detection (LOD) is highly dependent on the application; it is typically between 1% to 10% depending on measured material and measurement conditions.



Yttrium fluoride (YF_3) coating measurement with TRACER 5g



Polytetrafluoroethylene (PTFE) tape measurement with TRACER 5g



TRACER 5g Handheld XRF for Fluorine Analysis

Complete User Control

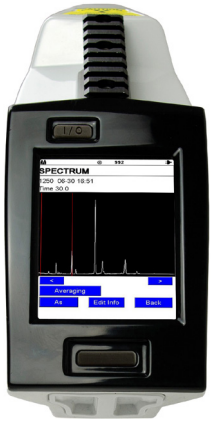
- Complete user control of excitation conditions - current, voltage, automated filter, manual filter, sample spot size, and atmosphere (vacuum, helium, or air)

Fast, Accurate Spot Positioning

- Internal camera for full view of sample spot with target area and reticle positioning for precision
- Remote view projection for challenging object positioning

Features

- Rh 50kV tube excitation source
- 1 µm Graphene window high resolution SDD detector
- Detector Shield™
- SharpBeam™ Geometry for minimized distance between sample and detctor
- 3 and 8 mm sample spot sizes
- Automated 4 filter wheel AND/ OR manual filter slot
- Integrated display embedded operating and analytical software
- Control, save, and send with USB, Wi-Fi, or Bluetooth
- Secure encrypted data storage
- Lightweight - only 1.9 kg / 4.1 lbs, including battery
- Non-destructive measurement



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