SIMS
Application Note 276

SIMS

Secondary Ion Mass Spectrometry

Glass Coatings

Summary

Coated glass plays a major energy conservation role in modern buildings; because of this the glass coating industry has grown enormously in recent years. Typical low emissivity glass comprises a thin silver layer sandwiched between other metals and dielectrics. The extreme sensitivity of SIMS means that it can play a crucial role in determining the composition and failure mechanisms of this material.

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Introduction

The requirement for energy efficient buildings has led to the development of glass coatings capable of reflecting heat (infrared radiation) whilst passing visible light with little degradation of color or perceived clarity. In addition, the coatings provide an opportunity to add color to the exterior of a glass clad building, resulting in exciting architectural possibilities.

The active part of most low emissivity (low-e) glass is a thin layer of sputter deposited metallic silver, typically only 10 nm thick. This is protected by other metallic and dielectric layers, giving a layer stack of around 100nm thick. Non-uniformities in thickness or composition lead to obvious optical variation and degrade performance.

In-situ failure of the layers can be extremely expensive, especially if a large building has to be reglazed, or many production units are affected. Failure may be caused by inappropriate use of chemicals, incorrect handling, or a manufacturing defect. Secondary ion mass spectrometry (SIMS) provides a rapid and cost effective analysis tool either for production control or failure analysis.

SIMS is able to evaluate layer composition and detect contaminants such as chlorine and sulfur that can directly attack and darken the silver layer.

SIMS

Secondary Ion Mass Spectrometry uses a focused, monoenergetic, chemically pure ion beam of typically 1-10 keV to sputter erode the surface under analysis. Ionized secondary particles are then analysed and detected in the mass spectrometer. At very low ion beam currents analysis is confined to the top few monolayers – excellent for detection of surface contamination. As the ion beam dose is increased and sputtering becomes more aggressive, subsequently deeper layers are exposed and concentration as function of depth can be determined.

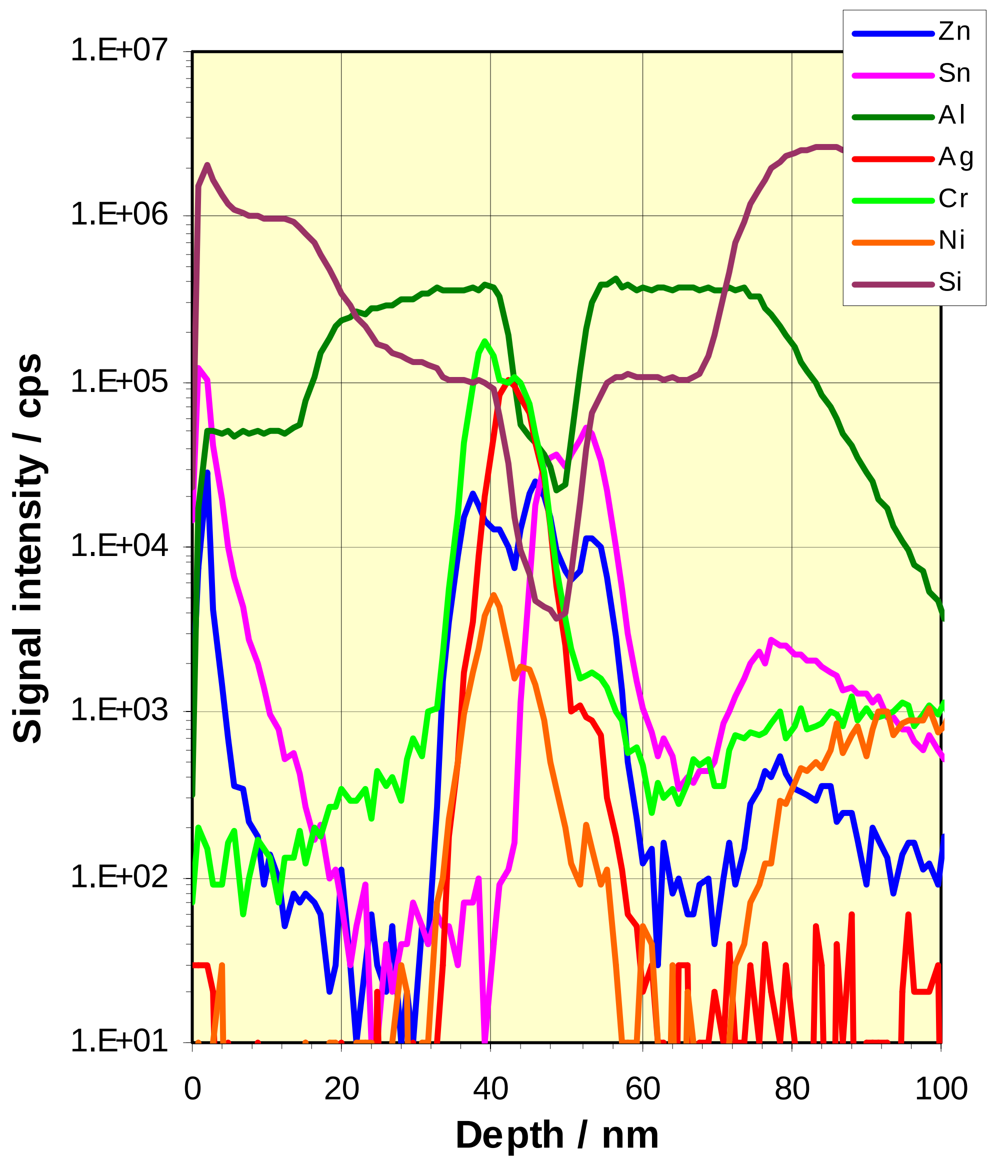
Generally SIMS depth profiles are presented with logarithmic concentration scales owing to the extreme dynamic range that can be achieved, ranging from ppb to bulk in the same analysis.

A flood of low energy electrons is used during analysis of insulating samples, such as glass, to prevent the buildup of surface charge.

The analysis presented here was made using the Hiden SIMS workstation, a complete and highly flexible quadrupole SIMS instrument equipped with the IG20 gas ion gun and MAXIM SIMS analyzer. The component parts are also available separately enabling high performance SIMS to be configured on a customers existing instrument (such as XPS or Auger), or optimizing the analysis for a particular part of the process flow.

Depth Profile Analysis

The SIMS depth profile shown below was collected using 5keV Ar ions focused to an 80µm spot and rastered over an area of 400 x 550µm. Positive secondary ions were collected and a 500eV electron flood was employed to prevent surface charging.

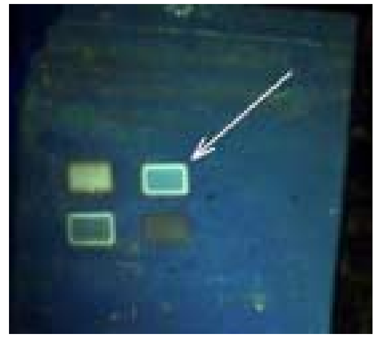


SIMS depth profile of a low-e glass sample

The SIMS depth profile agrees well with the design specification shown below,

Beginning at the exposed surface, the first layer is extremely thin and is partly consumed by the pre-equilibrium region at the start of the analysis. However, zinc and tin signals are clearly present at the very surface. There is a high silicon signal (rising to a level almost of that in the glass substrate) suggesting that a thin SiO2 layer may exist in the vicinity of the ZnSnOx.

The silicon nitride layer is characterised by a uniform concentration of silicon, however, this layer also contains aluminium, estimated to be ~7% (atomic).



Optical image of sample during analysis as seen by instrument camera system

Beneath the SiN layer lies a similar thickness of AlN. Interestingly, throughout this layer the Cr signal is rising, albeit from three orders of magnitude below the eventual peak. SIMS is perfectly suited to the investigation of this type of low concentration feature and for the analysis presented here it was necessary to significantly reduce the sensitivity to ensure that the peak of the Cr signal did not saturate the detector.

The region below the AlN contains the thin silver layer and its associated thin protective barrier layers containing Zn, Al, O Ni and Cr. The design thickness of the NiCrOx layer is only 1 nm and there has been some mixing of this into the silver layer during analysis.

Immediately below the silver, the thin Zn and ZnSnO layers are visible, before the final AlN layer and the glass substrate.

In conclusion, the *Hiden SIMS Workstation* is easily able to perform sensitive depth profile analysis on glass coatings, revealing layers of only a few nanometers thick and simultaneously observing low concentrations.