

Glass Analysis

Glass is a crucial element in numerous applications. We have unmatched expertise in glass analysis, research, and investigation.

Today there is more glass in use than ever before. Glass is a critical design component encompassing environmental protection, appearance, safety, light transmission, and thermal management. The increasing importance and sophistication of glass means that more advanced analyses are needed to address manufacturing issues. EAG has been analyzing glasses for over 20 years including verification tests, research and development support and failure analysis investigations. From composition, to surface modification, to contamination, to delamination and corrosion of coatings, EAG has unmatched expertise.

Glass Composition

Glass development and glass product verification requires the measurement of composition with high accuracy.

Major elements

Wavelength Dispersive X-ray Fluorescence (WD-XRF) can measure major and minor element glass composition.

WD-XRF features:

- Be-U elemental coverage
- Compositions accurate to within 5-10% for major elements
- Non-destructive analysis
- Standard method within the glass industry

WD-XRF: Selected Compounds

SRM 93a borosilicate glass (wt%)		
	Measured	Actual
B2O3	12.00	12.56
Na2O	4.17	3.98
MgO	0.005	0.005
AI2O3	2.35	2.28
SiO2	81.3	80.8
CI	0.078	0.060
K2O	0.016	0.014
CaO	0.016	0.01
TiO2	0.016	0.014
ZrO2	0.040	0.042

Trace elements

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) measures low concentrations and trace level concentrations with high accuracy. ICP-MS features:

- Almost full periodic table elemental coverage
- Trace level detection limits down to the ppm range
- Trace level components accurate to within 20%

Inclusions and Small Area

Laser Ablation ICP-MS (LA-ICP-MS) measures areas as small as 4 μ m. Sampling depth 1 μ m. LA-ICP-MS features:

- · Identify inclusions
- · Identify glass type
- · Analyze thin films on glass

ICP-MS: Selected Elements

NIST 611 Trace Elements in Glass (ppm wt)				
	Measured	Actual		
В	465	485		
Na	10.2 wt%	10.2 wt%		
Mg	483	465		
Al	1.05 wt%	1.04 wt%		
К	452	486		
Fe	474	457		
Со	413	405		
Мо	359	377		
Eu	477	461		
Та	391	377		

LA-ICP-MS: Selected Elements

NIST 611 Trace Elements in Glass (ppm wt)			
	Measured	Actual	
В	474	485	
Na	9.9 wt%	10.2 wt%	
Mg	570	465	
Al	1.10 wt%	1.04 wt%	
К	483	486	
Fe	456	457	
Со	407	405	
Мо	409	377	
Eu	471	461	
Та	440	377	





Surface Contamination

Surface particle and residue contamination can affect appearance, coating adhesion and film integrity.

Wash and rinse: Thin film trace level contamination

Adhesion, reliability and appearance of coatings can be affected by surface residue. The efficacy of the cleaning process should be well understood and identification of any residues should be investigated.

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) has very high sensitivity and a very shallow sampling depth making it ideal for detecting organic and inorganic surface residues.

Residues can have a detrimental effect on coating adhesion, appearance, yields and performance.

TOF-SIMS survery analysis shows:

- · Identity of residues
- Difference between 'good' and 'bad'
- · ppm detection limits

Particles and surface defects

Surface particles, defects, discolorations can affect appearance. Pinholes may affect corrosion of silver layers.

Auger Electron Spectroscopy (Auger) has very shallow sampling depth and can also examine sub-micron features. Auger can examine small particles, stained areas and corrosion sites.

Auger analysis can determine:

- · Particle composition
- Discoloration composition
- Defect locations and residues
- · Corrosion site products

TOF-SIMS Survey



Auger Survey







Secondary Electron image: appearance of defects



Glass surface modification

Exposure of glass surfaces to various treatments can result in a change in surface composition and chemistry. The penetration depth of these changes and the composition can be measured by Secondary Ion Mass Spectrometry (SIMS).

De-alkalization by hydration

Hydration is the process where hydronium ions diffuse into a glass surface and exchange with alkali and other metals. Hydronium concentration and ingress depth can be measured using SIMS. The depletion of alkali concentration at the surface and the depth of depletion can also be measured.

SIMS Profile



Ion exchange

Potassium can be substituted for sodium in the glass matrix using an ion exchange process.

The resulting glass is much stronger than before. SIMS concentration profiles of Na and K reveals how far into the material the exchange process has occurred.

SIMS Profile of Mobile Elements



SIMS Profile of a Newly Cleaned Surface



Minimal Artifacts

Alkali metals are also called 'mobile ions' because they are easily moved by electric fields such as those caused by the SIMS primary ion charged particle beam. With the correct analysis conditions, we can minimize migration caused by SIMS. Here we show a glass cleaved surface profile demonstrating SIMS has caused almost no profile distortions on this fresh fracture surface.

Coating Identification: Layer Structure and Composition

Investigation of the layer structure, identity and composition of an unknown coating system can be done in several ways.

Composition

An unknown glass coating structure was profiled by X-ray Photoelectron Spectroscopy (XPS). All layustifers were identified and the composition of each layer was determined. Compositions are accurate. Layer thicknesses are approximate.

Structure

An unknown glass coating structure was cross-sectioned and then analyzed by Scanning Transmission Electron Microscopy - Energy Dispersive X-ray Spectroscopy (STEM-EDS). We can observe the layer structure, thickness and uniformity, and all layers were identified. Layer thickness and uniformity is accurate. Composition is approximate.

Trace Level and Interdiffusion

An unknown glass coating structure was depth profiled by SIMS. We can observe the major element profiles along with the trace element profiles including Na diffusion from the substrate glass. Composition can be accurately determined with standards. Trace element concentrations are accurate. Layer thicknesses are approximate, in the absense of correlation to other measurements.



STEM-EDS Cross Section



—	Silicon
—	Titanium
—	Copper
	Silver
—	Tin
—	Nickel
	Zinc
	Chromium



Tempered glass analysis

Annealing and tempering of glass will change the glass and coating microstructure and cause interdiffusion between layers. SIMS depth profiling shows diffusion of major and minor elements between layers. TEM shows changes in microstructure.

Before Temper 100 1E+22 0 0 Ν Zn 1F+21 Approx. O, N, Al, Cr, Ag (atom%) Zn 10 (ato 1E+20 Mg S 1E+19 Na, 1E+18 4 1E+17 120 0.1 20 40 60 80 100 Ó Depth (nm)



Interfacial ayer





SIMS shows major diffusion of Na after tempering, from the glass substrate through all of the coating layers and to the surface. Si and N have diffused from the SiAIN layers into the adjoining layers.

TEM shows no change in coating layer thicknesses after tempering. However the interfaces between layers have become more defined and one interfacial layer has disappeared.

TCO analysis

Many glass coating systems include a TCO (transparent conductive oxide) layer to make electrical contact with the layer structure. For solar applications, TCO layers need to be optically transparent with a bandgap that minimizes light trapping yet still has high electrical conductivity.





Grain Orientation

TCO conductivity can be affected by dopants, contaminants, grain size, and grain orientation. GIXRD (Glancing Incidence X-Ray Diffraction) compares 2 TCOs: the red curve is from a low conductivity film while the black is from a higher conductivity film. The textures are quite different.

Thickness/ density/ roughness

TCO thickness, roughness and density can be measured quantitatively without the need for standards using XRR (X-Ray Reflectivity). Critical angle reveals density, the slope after the critical angle reveals roughness, and interference fringes reveals thickness. Comparison of two coatings shows similar thickness and density, but different surface and interface roughness.

Composition

Dopants can increase the electrical conductivity of TCOs but dopants can also reduce optical transmission. SIMS depth profiling reveals dopant concentration. The Zn:O ratio was calibrated by RBS (Rutherford Backscattering Spectrometry).

Glass Analysis

ParticlesDefects

• Composition



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- Layer Structure
- Contamination

- Coating Identification
- Glass Surface Modification