

SCOPE

The measurement of zirconium (Zr) conversion coating on aluminum is demonstrated.

BACKGROUND

Aluminum and steel are often coated with a protective conversion coating, also called passivate or passivation coating, to prevent oxidation and corrosion of the base metal. Conversion coatings include Cr, Ti, V, Mn, Ni, or Zr. A phosphate coating may be applied as well to minimize wear on cutting tools and stamping machines. Aluminum is often coated for use in aircraft parts, aluminum window frames and other similar industries where the aluminum is exposed to weathering. Steel for the automotive industry is typically first galvanized with a zinc coating before the conversion coating is applied. Protected steel is also used for outdoor sheds and other similar uses where steel is exposed to weathering. Conversion coating also help in the retention of paint for the final finished product.



INSTRUMENTATION

Model:	Rigaku NEX QC ⁺
X-ray tube:	50kV 4W Ag-anode
Detector:	High performance SDD
Film:	Mylar
Analysis Time:	300 sec
Atmosphere:	Air



SAMPLE PREPARATION

A test coupon is simply placed flat in the analysis chamber with the coated side facing down towards the X-ray beam. Test coupons must cover the 26mm diameter analysis aperture. Alternately, 32mm or 40mm circles can be cut for use with the autosampler trays or 32mm circles can be cut for use with the sample spinner. All samples must lie flat and cover the analysis aperture.

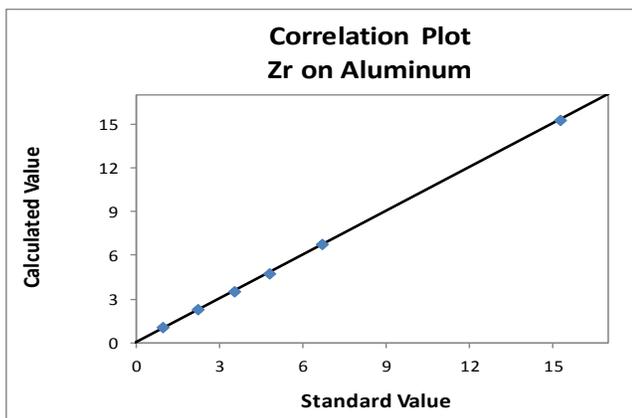
UNITS

1 mg/ft² = 10.8 mg/m²
1 mg/m² = 0.09 mg/ft²

CALIBRATION

An empirical calibration was built using a set of standards assayed by careful weigh-strip-weigh. The bare, uncoated aluminum sample was measured to generate special background correction that automatically compensates for the specific amount of background in each individual sample measured.

Element: Zr		RMS Dev: 0.060
Units: mg/ft ²		R ² Correlation: 0.99987
Sample I.D.	Standard Value	Calculated Value
STD 1	0.98	1.010
STD 2	2.21	2.267
STD 3	3.52	3.446
STD 4	4.81	4.738
STD 5	6.71	6.764
STD 6	15.26	15.265



RECOVERY & REPEATABILITY

To demonstrate repeatability (precision), three calibration standards were chosen. Each sample was measured in static position for ten repeat analyses using a total analysis time of 300 sec per measurement in air atmosphere, with typical results shown below.

Element: Zr		Units: mg/ft ²		
Sample ID	Standard Value	Average Value	Std Dev	% Relative
STD 1	0.98	1.082	0.029	3.0
STD 4	4.81	4.690	0.037	0.8
STD 6	15.26	15.217	0.024	0.2

DETECTION LIMITS

To determine the Lower Limit of Detection (LLD) ten repeat analyses of a bare, uncoated sample were measured using 300 sec measurement time per measurement and the standard deviation was calculated. The LLD is defined as three times the standard deviation. Detection limits depend on several factors, including measurement time,

Element	LLD in mg/ft ²	LLD in mg/m ²
Zr on Aluminum	0.08	0.82

SAMPLE SURFACE & POSITIONING

Aluminum may be surfaced with different patterns to give products various esthetic looks or physical properties. Such surfacing techniques may leave the aluminum smooth, may be brushed aluminum with a directional grain pattern, or may be a hatched or other pattern. The samples demonstrated here had a brushed directional grain pattern.

Various grain patterns may scatter X-rays in various ways and slightly degrade precision and detection limits. X-ray scatter effects can be minimized or eliminated by using longer measurement times, consistent positioning of pattern or by spinning the sample during analysis.

It should be noted that when samples have a grain pattern to them each sample should be positioned with the grain in the same, consistent position. In the case of brushed aluminum the grain should be positioned front-to-back from the operator rather than side-to-side. Alternately 32mm diameter flat circles can be stamped for use with the sample spinner.

CONCLUSION

The performance shown here demonstrates NEX QC⁺ provides excellent sensitivity and performance for the measurement of zirconium conversion coatings on aluminum.