ASOMA® Application Note

AMETEK® PROCESS INSTRUMENTS

ASOMA® PHOENIX II

Determination of Conversion Coatings on Steel or Aluminum

Summary

All samples were analyzed using the ASOMA® PHOENIX II XRF Benchtop system. This report demonstrates the capability of the PHOENIX II to analyze conversion coatings on steel or aluminium.

Conversion coatings are used for barrier protection against oxidation and corrosion, as a primer for paint-ing, or as a primer for optimum tooling and cutting properties.

Coatings include Zn, Cr, Ti, V, Zr, P or Si compounds.

Proper monitoring of coat weight is important. Over coating may cause loss of quality and undue over ex-pense. Under coating causes loss of quality. ASOMA® coating measurements require little sample preparation. Simply cut a sample panel, place it in the sample chamber and analyze.

These benefits work together to maximize quality and reduce operational costs.

Introduction

The ASOMA® PHOENIX II benchtop XRF analyzer offers a fast, precise, simple and non-destructive analysis technique well suited for the determination of conversion coatings thickness (as given by coat weight) on steel or aluminium substrates. Galvanization makes use of zinc coatings. As hexavalent chromium is being phased out of use, coatings to replace chromate coatings include titanium, vanadium and zirconium. Coating used as both a paint primer and tooling preparation include phosphate compounds and silicon compounds (silane, silicone, etc.)

The PHOENIX II employs state-of-the-art optics. Polarization excitation offers unique benefits because it eliminates most of the back-ground scatter emerging from the X-ray tube before it arrives at the sample. This results in a dramatic improvement in peak-to-background signal, especially in highly scattering materials. This translates to vastly improved precision and lower detection limits than traditional direct excitation XRF systems can achieve.

The PHOENIX II uses an onboard PC computer with a simple touch screen interface. Thus, an external computer is not required. Data handling and results storage can be obtained on a thermal paper printout and are stored in the hard drive of the PHOENIX II. The data is readily transferred to a USB thumb-drive or a network Ethernet connection.

Calibrations are carried out using assayed standards. This ensures easy traceability of results for quality purposes. This initial calibration process is a “once only” procedure. Subsequently, the curve can be re-standardized, if required, by the touch of a button on the main analysis screen.

The PHOENIX II offers power, versatility and performance all in a small, compact, easy-to-use design.

Experimental Portion

Equipment

All measurements were conducted using a PHOENIX II XRF analyzer. Performance is shown for a measurement time of 100 seconds, except for Si on Cr coated Steel, which uses an analysis time of 200 seconds.

Sample Preparation

Simply cut an approximately 3 x 5 inch square or disk panel and place it coating side down over the aperture in the sample chamber.

Measurement Parameters

All measurement parameters are easily controlled through the touch screen on the display panel. Operators simply choose the correct method from the analysis screen (there may be more than one method stored, e.g. to deal with chromate or titanium) and then press the green ANALYZE button.

The results can be reported using a variety of different options: results are reported on the display screen; on a thermal paper printout; on an optional external printer; and in the database history within the analyzer.

Instrument Configuration

ASOMA® PHOENIX II

Excitation: 48 kV 50 W Air-cooled X-ray Tube
Detection: Gas-filled Proportional Counter
Analytes Optimization: X-ray voltage, current and X-ray filters
Atmosphere: Air
Options: HOPG crystal for polarized X-rays; Moveable secondary target; Detector filters; Polypropylene 4 µm film

Note: No consumable gases required.

Typical sample panel shown in analysis position

Handy Conversion Factors

1 mg/ft² = 10.8 mg/m²
1 mg/m² = 0.001 g/m²
t = (coat weight)/d
t = coating thickness
d = density of coating material

Note:

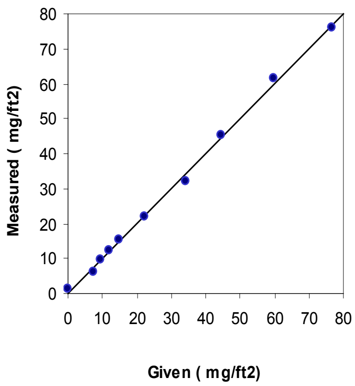
The following results sections show performance for a few typical coatings. The PHOENIX II is equipped to measure many others. Please contact AMETEK Process Instruments if you do not see your application listed here.

Results for Cr on Aluminum

Calibration for Cr on Aluminum

Element: Cr		
Units: mg/ft²	Std. Error of Estimate: 1.24	
Sample	Given	Measured
1	76.8	76.01
2	7.4	6.10
3	9.5	9.78
4	11.8	12.40
5	15.0	15.25
6	22.2	22.02
7	34.3	31.87
8	44.4	45.29
9	59.8	61.35
10	0.0	1.17

Correlation Plot
for Cr on Aluminum



Precision for Cr on Aluminum
10 repeat analyses at 100 seconds per measurement

Element: Cr				Units: mg/ft²
Sample	Given	Mean	Std. Dev.	%Rel.
1	76.8	78.66	0.43	0.6
6	22.2	22.26	0.19	0.9
5	15.0	15.61	0.15	1.0

Minimum Detection Limit (MDL)
Cr on Aluminum

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank uncoated sample. The following MDL was derived using this empirical method and applies to this matrix and coat weight range.

Element	Count Time	MDL
Cr	100 sec	0.7 mg/ft²

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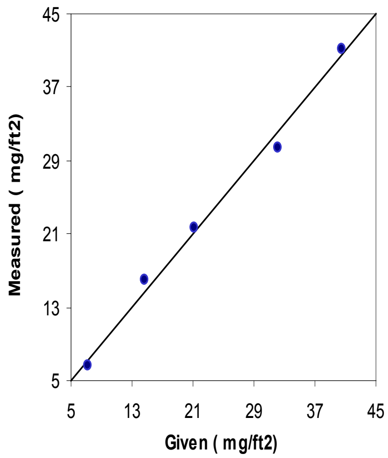
Results for Cr on Steel

Calibration for Cr on Steel

Element: Cr		
Units: mg/ft^2	Std. Error of Estimate: 1.45	
Sample	**Given**	**Measured**
20.a	40.5	41.2
15a	32.1	30.3
7a	21.2	21.6
4a	14.6	16.0
2a	7.2	6.6

Correlation Plot
for Cr on Steel



Precision for Cr on Steel
10 repeat analyses at 100 seconds per measurement

Element: Ca				Units: mg/ft^2
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
20a	40.5	39.4	1.0	2.6
7a	21.2	21.3	1.0	4.9
2a	7.2	7.00	1.3	18.0

Minimum Detection Limit (MDL)

Cr on Steel

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank uncoated sample. The following MDL was derived using this empirical method and applies to this matrix and coat weight range.

Element	Count Time	MDL
Cr	100 sec	3.1 mg/ft^2

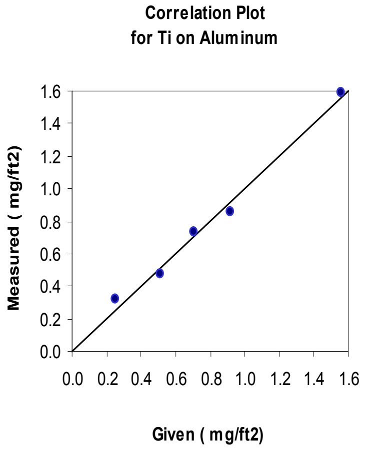
Results for Ti on Aluminum

Note:

Results for vanadium (V) coatings with be comparable to titanium (Ti) results.

Calibration for Ti on Aluminum

Element: Ti		
Units: mg/ft^2	Std. Error of Estimate: 0.053418	
Sample	**Given**	**Measured**
1	1.56	1.585
2	0.25	0.318
3	0.00	-0.008
4	0.51	0.474
5	0.71	0.729
6	0.92	0.853



Precision for Ti on Aluminum
10 repeat analyses at 100 seconds per measurement

Element: Ti			Units: mg/ft^2	
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
1	1.56	1.584	0.037	2.4
6	0.92	0.910	0.021	2.4
2	0.25	0.295	0.034	10.4

Minimum Detection Limit (MDL)

Ti on Aluminum

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank uncoated sample. The following MDL was derived using this empirical method and applies to this matrix and coat weight range.

Element	Count Time	MDL
Ti	100 sec	0.09 mg/ft2

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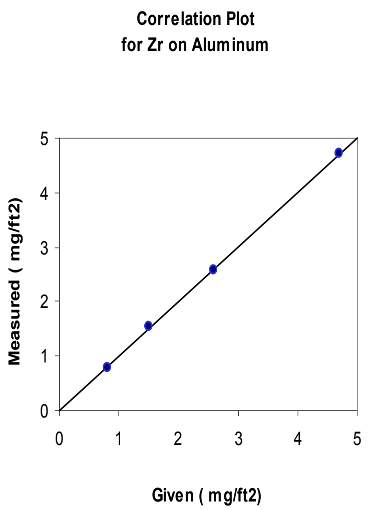
Results for Zr on Aluminum

Calibration for Zr on Aluminum

Element: Zr		Units: mg/ft²
RMS: 0.009	Std. Error of Estimate: 0.028487	
Sample	**Given**	**Measured**
1	4.7	4.70
2	0.8	0.79
3	1.5	1.53
10	2.6	2.58



Precision for Zr on Aluminum
10 repeat analyses at 100 seconds per measurement

Element: Zr				Units: mg/ft²
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
20a	40.5	39.4	1.0	2.6
7a	21.2	21.3	1.0	4.9
2a	7.2	7.00	1.3	18.0

Minimum Detection Limit (MDL)
Zr on Aluminum

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank uncoated sample. The following MDL was derived using this empirical method and applies to this matrix and coat weight range.

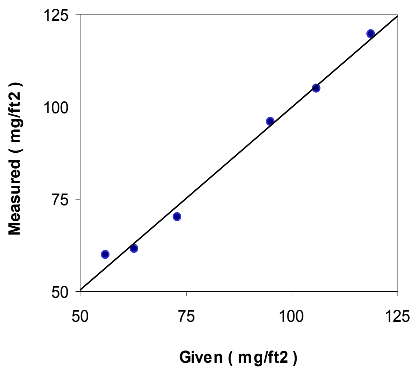
Element	Count Time	MDL
Zr	100 sec	0.025 mg/ft2

Results for P on Steel

Calibration for P on Steel

Element: P		Units: mg/ft^2
Std. Error of Estimate: 3.04		
Sample	**Given**	**Measured**
1	119	119.8
2	106	105.0
3	95	95.8
4	63	61.4
5	73	70.1
6	56	59.9

Correlation Plot for P on Steel



Precision for P on Steel

10 repeat analyses at 100 seconds per measurement

Element: P			Units: mg/ft^2	
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
1	119	119.9	0.53	0.44
6	56	57.6	0.18	0.31

Minimum Detection Limit (MDL)

P on Steel

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank uncoated sample. The following MDL was derived using this empirical method and applies to this matrix and coat weight range.

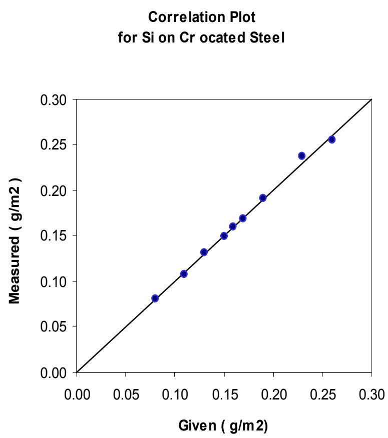
Element	Count Time	MDL
P	100 sec	1.2 mg/ft2

Results for Si on Cr Coated Steel

Calibration for Si on Cr Coated Steel

Element: Si		Units: mg/ft²
R.M.S.: 0.0010	Std. Error of Estimate: 0.0022716	
Sample	**Given**	**Measured**
1	0.26	0.255
2	0.19	0.191
3	0.16	0.160
4	0.15	0.149
5	0.13	0.131
6	0.11	0.108
7	0.08	0.081
8	0.17	0.169
9	0.23	0.237

Precision for Si on Cr Coated Steel



10 repeat analyses at 200 seconds per measurement

Element: Si			Units: g/m²	
Sample	**Given**	**Mean**	**Std. Dev.**	**% Rel.**
1	0.26	0.253	0.001	0.5
3	0.16	0.161	0.001	0.6
8	0.08	0.082	0.0005	0.6

Minimum Detection Limit (MDL)
Si on Cr Coated Steel

The Minimum Detection Limit (MDL) for an element is determined as three times the standard deviation of ten analyses of the blank uncoated sample. The following MDL was derived using this empirical method and applies to this matrix and coat weight range.

Element	Count Time	MDL
Si	200 sec	0.001 g/m²

Conclusion

As can be seen from the above data, the use of the PHOENIX II XRF system gives excellent performance when applied to the determination of conversion coatings on steel or aluminium. Results are rapid, precise and analysis is easily carried out, even by non-laboratory personnel. Because no consumable chemicals are used, the relative "cost of ownership" is much lower than other analytical techniques.

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