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APPLICATION NOTE

Alloy Characterization using Wavelength Dispersive X-Ray Fluorescence Spectroscopy (WDXRF)

INTRODUCTION

Alloy composition is of critical importance for product design, failure analysis, and quality control. For example, choosing the correct alloy for the environmental and physical stresses to which a device or part will be subjected is paramount to avoid early failures from corrosion, fracturing, or other mechanisms. When failures do happen a comprehensive root cause analysis needs to involve verification that the correct alloy was actually used.

Another common example of an alloy-related failure is when small metal flakes or shavings are found in products and it is critical to determine their source. Identifying the alloy can very often pinpoint the source of the particles by matching the alloy of the flakes to that of known components in the system used to fabricate the contaminated material.

Finally, quality control when manufacturing alloys for precise applications is critical to minimize failure mechanisms by making sure that the compositions of the materials used meet exacting specifications.

Wavelength Dispersive X-Ray Fluorescence Spectroscopy (WDXRF) is an ideal analytical tool for determining or verifying alloy compositions. As a full survey technique with strong standardless quantification capabilities, simple alloy compositions can typically be determined with little or no need for preliminary information about the material. In more demanding applications, WDXRF can be used with calibration standards to provide the utmost accuracy to verify that compositions meet even the most stringent requirements.

DISCUSSION

PART 1: SIMPLE ALLOY DETERMINATION

A simple determination of the alloy class can usually be accomplished using WDXRF without the need for matched standards by simply using Fundamental Parameters (FP), a "standardless" method relying on instrument sensitivity factors derived from pure elements. Table 1 shows results from three stainless steel alloys analyzed by this method. The measured compositions can be subsequently compared to known alloy specifications to confirm the particular alloy identity. Three very similar 300 series stainless steels (301, 303, and 304) were clearly distinguished and identified from the combination of their Ni and Cr concentrations. Trace elements with concentration specifications, such as sulfur and phosphorus, can similarly be used to identify a particular alloy. Additionally, the results from two aluminum alloys, 5052 and 6061 show accurate quantification to enable the alloy identification (Table 2). XRF has the ability to measure matrix compositions as well as trace elements to provide a nearly full survey; however, the C, N, and O concentrations are best measured by IGA, which complements the XRF results. The accuracy of the FP algorithm is enhanced with WDXRF measurements due to the very high energy resolution and low backgrounds associated with the technique.

PART 2: HIGH ACCURACY ALLOY CHARACTERIZATION

In cases when it is necessary to know an alloy composition with very high accuracy, either as a buyer or supplier, WDXRF has unrivaled accuracy and precision when the appropriate calibration standards are available. An example of this is the iron-nickel controlled expansion alloy system, which is used in applications where a specific thermal expansion is required over a given temperature range. These alloys are used as glass sealing alloys where a hermetic seal is required between glass and metal and are also used when a near zero coefficient of expansion is necessary (e.g. some electronic devices; precision laser equipment; and lead frames in integrated circuit packaging). The amount of nickel in the alloy determines the thermal properties and may range from approximately 20 to 54% Ni by weight, depending on the application. WDXRF is an ideal technique to provide very accurate determination of the Ni content in these alloys, with the use of certified standards for calibration. In the example shown (Table 3), the nickel content was measured with a relative accuracy of 0.11%. Additionally, trace metals in the alloy were measured with relative accuracies ranging from <1 to ~9%. The standard deviation from repeat measurements are also provided to demonstrate the precision.

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CONCLUSION

Many thousands of ferrous and non-ferrous alloys exist to fill niches for very specific applications, and new alloys are being created all the time to address high-end manufacturing and technology needs. WDXRF is an analytical tool with an inherent precision and accuracy such that it is an invaluable tool for alloy characterizations. First level survey inspections are easily accomplished on any material using Fundamental Parameters, whereas availability of standards for calibration provides the means for QC analysis when the highest degree of accuracy is required.

Table 1. Standardless Quantification of Stainless Steel Alloys (in Wt%)[a]

	SAE 301		SAE 303		SAE 304	
	Measured	Actual	Measured	Actual	Measured	Actual
Si	0.64[b]	0.53	0.449	0.415	0.53	0.56
P	0.007	0.006	0.031	0.028	0.0398	0.0402
S	0.0089	0.0019	0.465	0.326	0.0277	0.0188
Cr	17.0	17.0	17.21	17.23	18.21	18.17
Mn	1.73	1.67	1.87	1.80	1.762	1.706
Ni	7.15	7.12	8.22	8.17	8.37	8.20

[a]Partial element list. Iron (Fe) is the principal balance element.

[b]Measured concentrations are listed to the same decimal place as the certified value

Table 2. Standardless Quantification of Aluminum Alloys (in Wt%)[a]

	5052 Al		6061 A1	
	Measured	Actual	Measured	Actual
Mg	1.00[b]	0.995	2.66	2.58
Si	0.639	0.638	0.154	0.149
Ti	0.0435	0.0418	0.0102	0.0103
V	0.0119	0.0111	0.0110	0.0107
Cr	0.255	0.231	0.265	0.244
Mn	0.0557	0.0509	0.0562	0.0527
Fe	0.376	0.357	0.216	0.200
Ni	0.573	0.0506	0.055	0.048
Cu	0.324	0.297	0.07	0.06
Zn	0.08	0.08	0.085	0.083
Ga	0.022	0.0201	0.0217	0.0202

[a]Partial element list. Aluminum (Al) is the principal balance element.

[b]Measured concentrations are listed to the same decimal place as the certified value

Table 3. High Accuracy Quantification of NiFe Alloy (in Wt%)[a]

	Measured	Actual	Relative Accuracy (%)	Standard Deviation[c]
Ni	25.07[b]	25.10	0.11	0.014
Al	0.107	0.103	3.6	0.0004
S	0.58	0.58	0.05	0.001
Cr	0.0351	0.0334	5.1	0.0005
Mn	0.0110	0.0121	9.1	0.0006
Co	0.752	0.746	0.80	0.001

[a]Partial element list. Iron (Fe) is the principal balance element.

[b]Measured concentrations are listed to the same decimal place as the certified value

[c]Standard deviation from four repeat measurements