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Application News

Shimadzu Energy Dispersive X-Ray Fluorescence Spectrometer EDX-8100

Introduction of Quantitative Analysis of Aluminum Alloys and Matching Function

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User Benefits

- Enables analysis of Mg in aluminum alloys, which was difficult with conventional EDXRF.
- The series of aluminum alloys can be distinguished by using the matching function.
- Supports diverse size of samples, from small specimens to A4 size.

■ Introduction

In aluminum alloys, elements such as copper, manganese, and silicon, are added to aluminum to improve its machinability, abrasion resistance and corrosion resistance. Depending on the composition, aluminum alloys are classified as 1000 to 8000 series.

The energy dispersive X-ray fluorescence spectrometer (EDXRF) is widely used for quality control of aluminum alloys and acceptance inspections of recycled materials. However, analysis of light elements (particularly Mg) had been difficult until now due to the inadequate sensitivity and resolution. The Shimadzu EDX-8100 offers greatly enhanced performance in the light element region, enabling quantitative analysis of Mg at levels of 0.1 % or less.

This article introduces the following:

1. Quantitative analysis and accuracy evaluation by the calibration curve method (preparation of calibration curves using standard samples and repeatability testing)
2. FP (fundamental parameter) qualitative/quantitative analysis and matching function

1. Quantitative Analysis by Calibration Curve Method

■ Samples

- Aluminum alloy standard samples manufactured by Arconic Corporation
  ① MSS-1100, ② MSS-2024, ③ MSS-3004,
  ④ MSS-5182, ⑤ MSS-7075
- Pure aluminum sample (purity: 99.99 % or higher);
  Total 6 samples

Table 1 shows the standard values of the aluminum alloys.

Table 1 Standard Values of Aluminum Alloys

Unit [wt%]

	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti
①MSS -1100	0.18	0.50	0.15	0.040	0.030	<0.0050	0.080	0.020
②MSS -2024	0.20	0.35	4.6	0.65	1.6	0.060	0.10	0.030
③MSS -3004	0.18	0.50	0.15	1.2	1.1	<0.0050	0.050	0.020
④MSS -5182	0.15	0.20	0.05	0.35	4.6	0.030	0.050	0.030
⑤MSS -7075	0.16	0.15	1.6	0.080	2.6	0.20	5.8	0.040

■ Elements

Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti

■ Calibration Curves

Fig. 1 shows the calibration curves. Considering the influence of absorption excitation between elements, matrix element correction by the dj method was applied for Mg, and correction for overlap with Mn and Cr by the lj method was applied for Fe and Mn, respectively.

■ Qualitative Profile Around Mg Peak

Fig. 2 shows the profile around the Mg peak. Although detection had been difficult until now, the peak for MgKα can be clearly confirmed.

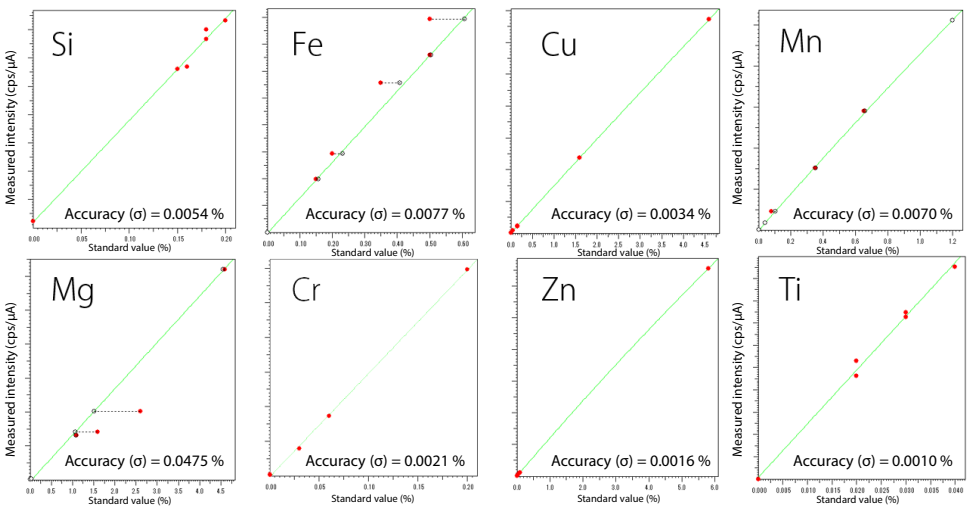

Fig. 1 Calibration Curves and Degree of Accuracy (σ)

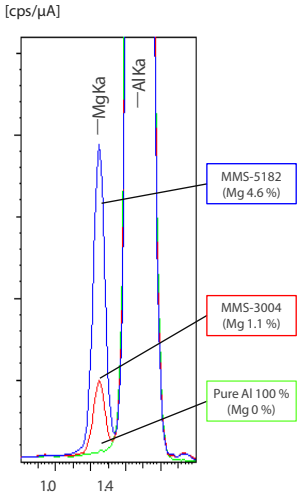

Fig. 2 Profile Around Peak of Mg

■ Repeatability

Table 2 shows the results of a simple repeatability test (n = 10) of standard sample ② MSS-2024.

Table 2 Repeatability of MSS-2024

Unit [wt%]

	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti
Standard value	0.20	0.35	4.6	0.65	1.6	0.060	0.10	0.030
Average value	0.196	0.364	4.598	0.658	1.596	0.0588	0.103	0.0298
Standard deviation	0.0050	0.0015	0.0034	0.0016	0.0082	0.0007	0.0008	0.0013
Repeatability [%]	2.5	0.4	0.07	0.2	0.5	1.3	0.8	4.4

2. FP Qualitative/Quantitative Analysis and Matching Function

■ Outline

The matching analysis function compares the analysis results with preregistered library data and searches for substances which are close to the sample.

Fig. 3 shows the matching results for standard sample ③ MSS-3004 using the FP qualitative/quantitative analysis results and "Al Alloy + Castings Library."

Because Al_3004 was the highest hit in the matching results, the sample was identified as aluminum alloy 3004 in the JIS standard.

■ Analysis Conditions ①: Calibration Curve Method

Table 3 Analysis Conditions (Calibration Curve Method)

Instrument	:	EDX-8100
Elements	:	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti
Analysis group	:	Calibration curve method
Detector	:	SDD
X-ray tube	:	Rh target
Tube voltage	:	15 [kV] (Si, Mg) 50 [kV] (Fe, Cu, Mn, Cr, Zn, Ti)
Tube current	:	Auto [μA]
Collimator	:	10 [mmφ]
Primary filter	:	None (Si, Mg), #2 (Ti), #3 (Fe, Mn, Cr), #4 (Cu, Zn)
Atmosphere	:	Vacuum
Integration time	:	300 [s] + 180 [s] × 3 (#2, #3, #4)
Dead time	:	Max. 30 [%]

■ Analysis Conditions ②: FP Method

Table 4 Analysis Conditions (FP Method)

Instrument	:	EDX-8100
Elements	:	C-U
Analysis group	:	Qualitative/quantitative analysis
Detector	:	SDD
X-ray tube	:	Rh target
Tube voltage	:	15 [kV] (C-Sc), 50 [kV] (Ti-U)
Tube current	:	Auto [μA]
Collimator	:	10 [mmφ]
Primary filter	:	None (C-Sc), (Ti-U), #3 (Cr-Fe), #4 (Cu-As, Pb)
Atmosphere	:	Vacuum
Integration time	:	300 [s] (C-Sc) 100 [s] × 3 (Ti-U), (Cr-Fe), (Cu-As, Pb)
Dead time	:	Max. 30 [%]

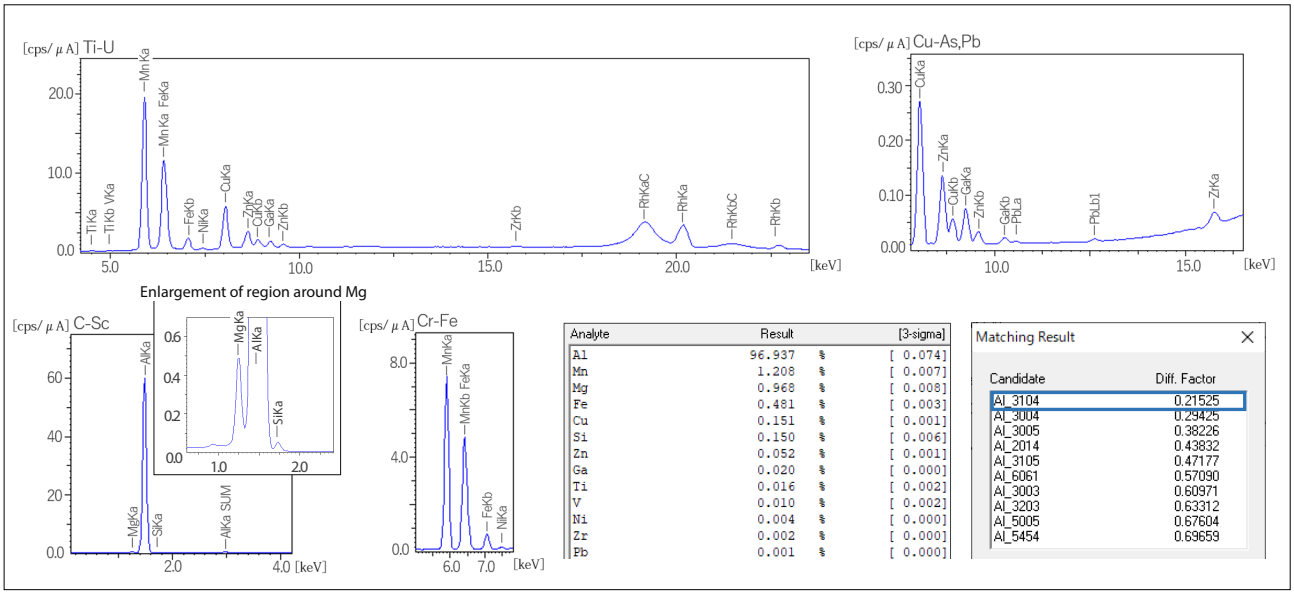

Fig. 3 Results of FP Qualitative/Quantitative Analysis and Results of Matching Search

■ Conclusion

Because the EDX-8100 has greatly improved sensitivity for light elements, highly accurate analysis of light elements is also possible. In particular, although analysis of Mg in aluminum alloys is difficult, excellent sensitivity and accuracy are possible even with contents of approximately 1 wt%.

In addition, it is also possible to distinguish series of aluminum alloys by using the matching search function, which is included in the analysis software as a standard feature. This function is a useful tool for process control, quality control, and acceptance inspections of recycled materials.

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