Application Note WDXRF1091

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XRF1091 - Beryllium analysis in beryllium copper alloy using ZSX Primus IV with RX85

Introduction

Beryllium copper alloy is almost as strong as steel, and is the strongest among copper alloys. In addition, it has non-magnetic and non-sparking characteristics, high electrical conductivity and ductility. Owing to these features, beryllium copper has many uses, such as: springs, electric connectors, tools in environments with explosive vapors and gases, and musical instrument components. Since the characteristics and uses of beryllium copper alloys depend on the beryllium concentration, it is important to analyze beryllium in beryllium copper.

Beryllium is the lightest element that can be analyzed by XRF spectrometry. Its element line, Be-Kα, has a very long wavelength, 11.4 nm (or very low energy, 0.109 keV) and very shallow critical depth. Therefore, X-ray intensities of Be-Kα are significantly affected by the surface condition of specimens. For beryllium analysis by XRF spectrometry, surface treatment is essential.

Owing to the long wavelength of Be-Kα, beryllium analysis requires high-power wavelength-dispersive X-ray fluorescence (WDXRF) spectrometers equipped with an analyzing crystal with high reflectivity for Be-Kα.

This application note demonstrates beryllium analysis in beryllium copper alloy.

Instrument and measurement

ZSX Primus IV and ZSX Primus IVi, a sequential WDXRF spectrometer, are equipped with a 4 kW X-ray tube with a Rh target featuring an ultra-thin beryllium window, which has a strong advantage for lighter element analysis, and four analyzing crystals covering $_{8}$O to $_{96}$Cm. In this analysis, a new synthetic multilayer analyzer RX85 (optional) was also mounted in the spectrometer.

For beryllium analysis, APC (Auto Pressure Control), a function that keeps the vacuum levels in the sample and optical chambers constant during measurement, is effective. Without APC, changes in the vacuum level in the X-ray path would cause the X-ray intensities of Be-Kα to fluctuate because of the characteristic line's long wavelength.

The system software is designed for ease of use in routine analyses. The Flowbar in quantitative analysis guides users in establishing calibration. The Sample ID Table and the Program Operation help operators carry out daily analysis.

Measurements were performed on the ZSX Primus IV with a 4 kW X-ray tube operating at 30 kV and 100 mA using the RX85 multilayer analyzer and the S8 high-sensitivity slit (optional). The counting time was 300 seconds only for peak.

Standard and sample preparation

Certified reference materials of beryllium copper alloy provided by CTIF (France) were used for calibration. Each sample was polished using a belt surfacer with a #240 alumina abrasive belt.

Analysis result

Figure 1 shows the calibration curve obtained with CTIF CRMs and a pure copper metal.

The accuracy of calibration is 0.12 mass% and the lower limit of detection (LLD) is 0.08 mass%.

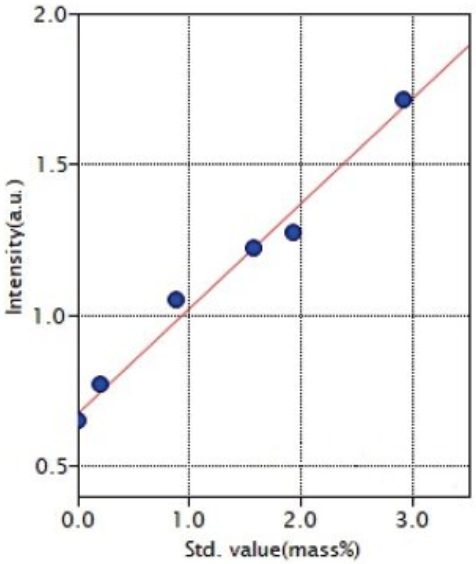

Figure 1: Beryllium calibration curve

The accuracy of calibration is calculated by the following formula:

$$Accuracy = \sqrt{\frac{\sum_i (C_i - \hat{C}_i)^2}{n-2}}$$

C_i: calculated value of standard sample
$\hat{C}_i$: reference value of standard sample
n : number of standard samples.

The LLD is calculated by the following formula:

$$LLD = 3 \cdot \frac{1}{m} \cdot \sigma_B = \frac{3}{m} \cdot \sqrt{\frac{I_B}{1000 \cdot t}}$$

$m{{ widgetHtmlContent }}nbsp; : sensitivity of calibration (kcps/mass%)
$\sigma_B{{ widgetHtmlContent }}nbsp; : standard deviation of blank intensity (kcps)
$I_B{{ widgetHtmlContent }}nbsp; : blank intensity (kcps)
$t{{ widgetHtmlContent }}nbsp; : counting time (s)

Repeatability tests were carried out using two of the CRMs used for the calibration. Each specimen was polished and then measured, quantified with the calibration. This process was repeated ten times. The test results are shown in Table 1.

Table 1: Repeatability test results. (mass%)

Cycle	CTIF 4873	CTIF 4766
n=1	0.23	1.57
2	0.27	1.55
3	0.24	1.63
4	0.25	1.60
5	0.22	1.58
6	0.27	1.62
7	0.22	1.57
8	0.22	1.58
9	0.16	1.64
10	0.23	1.59
Average	0.23	1.59
Standard deviation	0.033	0.029
RSD	14.3%	1.9%

Conclusion

Beryllium in beryllium copper alloy can be routinely analyzed with ZSX Primus IV equipped with a 4 kW X-ray tube with a Rh target featuring an ultra-thin beryllium window, and the multilayer analyzer RX85. It is necessary to carefully polish the surface of specimens prior to measurement. The measurement demonstrated in this application can also be performed with ZSX Primus IVi, a tube-below sequential wavelength dispersive X-ray fluorescence (WDXRF) spectrometer.

Related products

ZSX Primus IV

High power, tube above, sequential WDXRF spectrometer wi th new ZSX Guidance expert system software

ZSX Primus IV*i*

High-power, tube-below, sequential WDXRF spectrometer wi th new ZSX Guidance expert system software