Application Brief

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Latest Technology : The Bench Top X-ray Fluorescence Analyzer

1. Overview

Fluorescent X-ray analysis makes possible non-contact, non-destructive analysis and is widely used because of high repeatability in quantitative results. The predominant method is wavelength dispersion fluorescent X-rays from samples with spectral crystals. Over 10 years of technological progress, in particular, improvements in high-resolution detectors and increased speed of personal computers has broadened the use of bench-top energy dispersion analyzers, which have superior operation capabilities.

This application brief introduces the new technology of bench top X-ray fluorescence analyzers.

2. Analysis Conditions

The energy dispersion X-ray fluorescence analyzer is capable of multi-element, simultaneous measurement and simple measurement pre-treatment. However, it displays low sensitivity in measuring light elements. Here we test the improvements from improved sensitivity of light element measurements. Below is a summary of improvements.

- Carbon detection with HTW detector.
- Improved light element excitation with low volt excitation and a new type of X-ray tube.
- Improved P/B ratio by use of primary filter.

3. Configuration

A photograph of the system is shown Figure 1. Figure 2 displays a block diagram of the system.

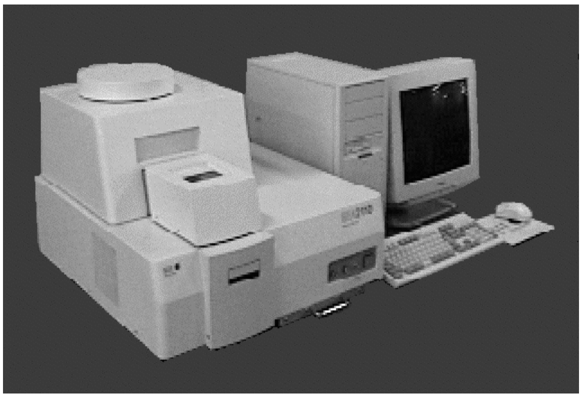

Figure 1 SEA2100 Bench Top Analyzer

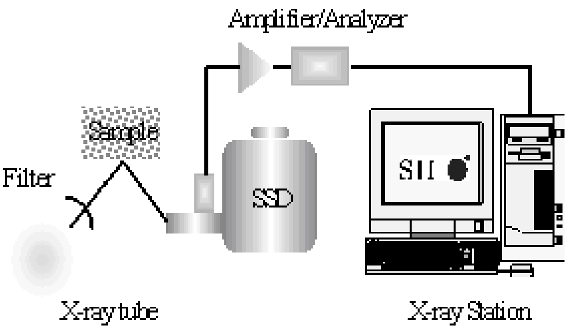

Figure 2 Block Diagram of the System

The measurement head is the primary X-ray instrument and has underside irradiation. The detector is a Si(Li) semi-conductor detector. The data processor is an IBM-PC compatible personal computer that features Windows OS. The X-ray Station software is common among the X-ray product group at Hitachi High-Tech Science Corporation.

4. HTW Detector

The High Transmission Window (HTW) enables a higher transmission of low energy X-rays because it is thinner than conventional Be windows. Figure 3 shows a comparison with the transmission of conventional detectors.

The solid line is of the HTW. The broken line is of conventional detectors. Transmission for the carbon K line shows excellent characteristics at a percentage of several 10s. By using HTW the P/B ratio can be improved for light elements such as Na, Mg, Al, and Si.

Figure 4 shows a spectrum a measurement of a NaCl thin film standard sample (50.1 μg/cm^2 on mylar film). Na intensity with the HTW is twice that of prior intensities.

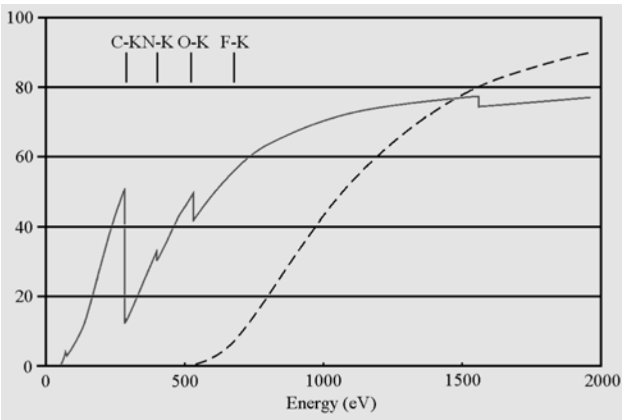

Figure 3 Transmission Comparison

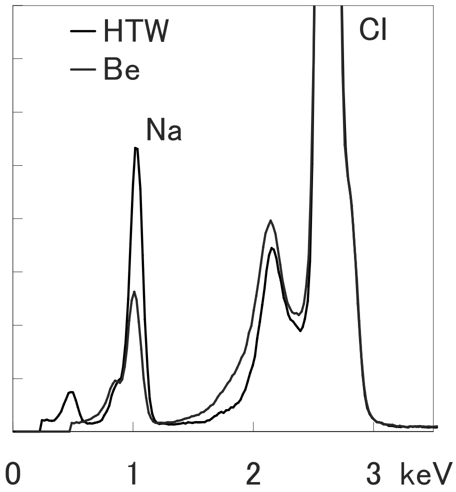

Figure 4 NaCl Thin Film Standard

5. Effectiveness of new X-ray tube

Modifications were made aimed at improving sensitivity of light elements in X-ray generation as well as detection.

Inside the X-ray tube, thermions discharged from the filament speed up and generate X-rays by colliding with the metal target.

Figure 5 shows conditions near the metal target.

Loss of low energy fluorescent X-rays can be controlled and the distribution of X-rays shifted to lower energies by increasing the output angle of the target and making the material of the X-ray tube window thinner.

Figure 6 shows an example of the effect of light element analysis using the new X-ray tube. The sample contains Al, Ti, Fe, and Zn. Measurement conditions include an acceleration voltage of 15 kV and vacuum atmosphere.

What is clear from this spectrum is that the Al intensity improved but the intensities of Ti, Fe, and Zn did not change much. In other words, the excitation rate of light elements improved selectively.

The effect of the new X-ray tube is that the P/B ratio of the Al peak improved 15%.

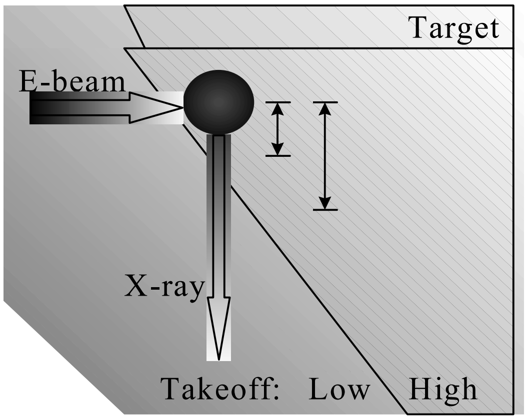

Figure 5 Improved X-ray Tube

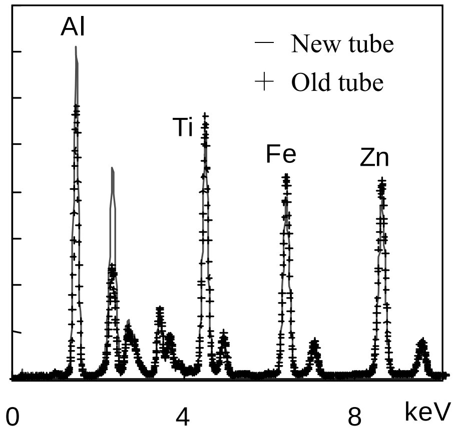

Figure 6 Effect of New X-ray Tube

6. Effectiveness of Low Voltage Excitation

This section shows the effect of low voltage excitation. In the case of energy dispersion fluorescent X-ray analysis, when analyzing very light elements within a heavy element matrix, the detector is saturated by fluorescent X-rays from the excitation effect of the very heavy elements. Intensities of targeted light elements cannot be sufficiently detected. Thus, 5 kV low voltage excitation was added to the 50 kV and 15 kV acceleration voltages already in use.

The expectation using low voltage excitation is that the fluorescent X-rays of heavy elements that make up the main matrix will not be excited but that there will be an increase effect of low energy fluorescent X-rays.

Figure 7 shows the measurement of a sample of soda lime glass. Measurements were performed under conditions of 15 kV and 5 kV. The atmosphere in both measurements was a vacuum.

Using low voltage excitation, the excitation effect of Ca, of which the main matrix consists, decreases. Relatively speaking, the excitation effect of light elements improves and the P/B ratio of light elements Na, Mg, and Al improve markedly. The P/B ratio of oxygen also improved.

In this way, improvement of the intensity of Na within the glass doubled by using the low voltage method.

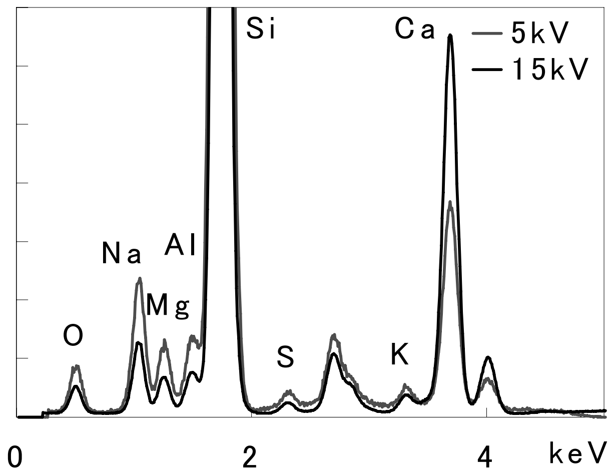

Figure 7 Low Voltage Excitation Effect

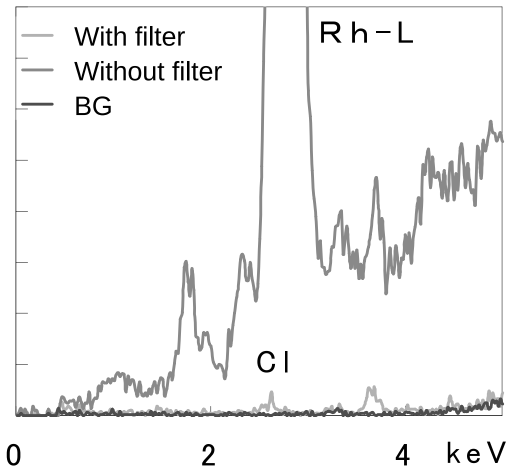

Figure 8 Effect of Primary Filter

7. Effectiveness of a Primary Filter

The primary filter is placed between the X-ray tube and sample. There are many varieties depending on the use.

Shown here is one typical use, which is the effect of removing scattered X-rays and improving the P/B ratio.

An instrument that uses Rh as the target material in the X-ray tube has difficulty analyzing trace amounts of Chlorine due to overlap with Rh scattered X-rays.

By using a primary filter and removing characteristic X-rays of the target material, we are able to lower the background effect and improve the P/B ratio.

Figure 8 shows the effect of the primary filter.

The sample is a resin that included trace amounts of chlorine. The trace Cl peak is completely hidden in the scattered X-rays of the target material when the filter is not used, but can be analyzed when the filter is used.

This is an example of selectively controlling the primary X-ray quality of radiation.

8. Summary

With the development of this instrument, the generating system and detection system, which are basic parts of energy dispersion fluorescent X-ray analyzers, were changed with an aim to improve sensitivity especially in light element analysis.

This is the start of measuring ultra-light elements (C to F) and the realization of improved overall sensitivity.