Application Note

XRF 1084

Mapping and Small Spot Analysis with a General-Purpose XRF Spectrometer

Application

geology,
rock
mineralogy

Instrument

Wavelength dispersive X-ray fluorescence spectrometer
ZSX Primus IV, ZSX Primus

ZSX Primus IV

Keywords

MicroMapping
small spot analysis
mapping analysis
distribution
digital image

Introduction

One of the features of X-ray fluorescence (XRF) spectrometry is that a large area, such as 30 mm in diameter, can be measured on the surface of a specimen, which appropriately represents an analysis sample.
On the other hand, there has been a demand to measure a small spot on the surface on a specimen (point analysis) or to obtain information of elemental distribution on the surface of a specimen (mapping analysis). Although there are XRF spectrometers dedicated for point or mapping analysis available in the market, it is not possible to perform large area analysis with these models.
Rigaku ZSX Primus IV and ZSX Primus, general-purpose wavelength-dispersive XRF (WD-XRF) spectrometers, enable point and mapping analysis.
This note demonstrates point and mapping analysis function of ZSX Primus IV/Primus by analyzing a granite rock chip.

Instrument

ZSX Primus IV/Primus have a six-position diaphragm changer with the analysis areas 0.5, 1, 10, 20, 30 and 35 mm in diameter, and are capable of both small-spot and large-area analyses.
ZSX Primus IV/Primus also have a sample stage, which can move a specimen to a specified position.

In sequential WD-XRF spectrometer, the X-ray tube is placed diagonally to the sample, as close to the sample as possible, in order to optimize the X-ray intensities of both the primary X-ray beam and fluorescent X-rays generated in the sample. Therefore, the distribution of the primary X-rays on the sample is not concentric. As shown in Figure 1, the center of the sample is not the point with the highest sensitivity but that point is shifted to the side for the X-ray tube.
In conventional XRF spectrometers, for small-spot analysis, the measurement position is the center of the sample. Therefore, as shown in the right side of Figure 1, the X-ray intensity of the primary X-ray beam at the measurement position is almost half of the highest intensity.
On ZSX Primus IV/Primus, for small-spot analysis, the measurement position is set at the spot with the highest X-ray intensity. Since ZSX Primus IV/Primus have the sample stage with r-θ driving, any position on the sample can be measured with the same sensitivity by automatically moving the sample accordingly. Even spots near the rim of the measurement area have the same sensitivity.
ZSX Primus IV/Primus have MicroMapping function, where a digital image of the sample surface is captured and it is possible to specify an analysis area, a line or spots on the captured image.

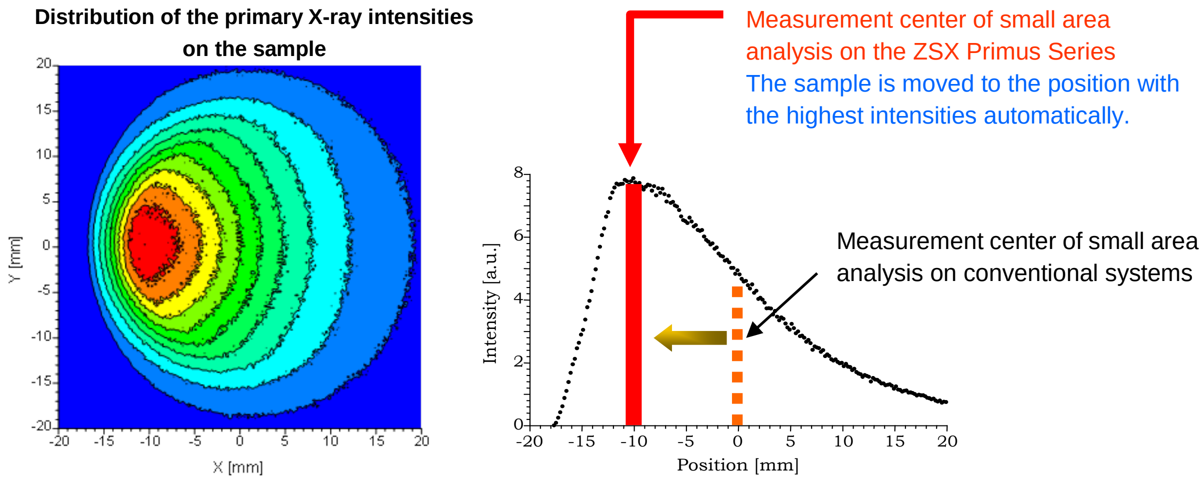

Figure 1 Distribution of the primary X-ray beam.

In MicroMapping of ZSX Primus IV/Primus, the sample is moved by the sample stage, but the optics does not move; therefore, it is possible to perform qualitative, quantitative or semi-quantitative analysis with the same sensitivity for each spot in MicroMapping.

Measurement

A rock chip of granite was measured by MicroMapping with ZSX Primus IV. The rock chip is a remaining part of a piece of granite for making a thin section.
The measurement spot size was 0.5 mm in diameter.

Analysis Results

Figure 2 shows a captured digital image of the granite sample.
First, SQX analysis was carried out for four spots on the granite samples. Figure 3 shows the measured spots.

Table 1 SQX result of the spots measured by MicroMapping.

Component	Measured points			(in mass%)
	A	B	C	D
Na_2O	-	-	3.4	-
MgO	7.2	4.9	-	-
Al_2O_3	12.1	7.9	19.4	0.46
SiO_2	41.1	41.6	64.7	99.0
P_2O_5	-	2.7	-	-
K_2O	2.6	1.9	10.8	0.10
CaO	0.92	6.7	1.2	0.05
TiO_2	1.6	4.6	-	-
MnO	0.58	0.37	-	-
Fe_2O_3	33.2	28.3	0.41	0.35
Rb_2O	0.04	-	0.03	-
SrO	-	-	0.03	-
ZrO_2	-	0.85	0.03	0.02

- : “not detected”

Table 1 shows SQX analysis results, which shows the results match the minerals at the measured spots.

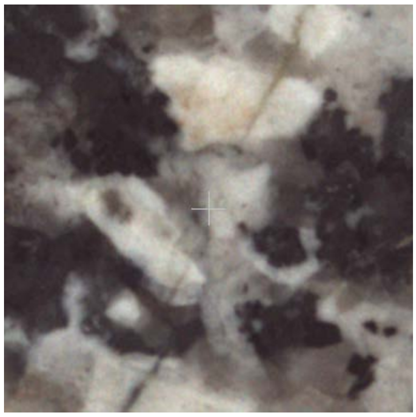

Figure 2 Captured digital image of the granite sample.

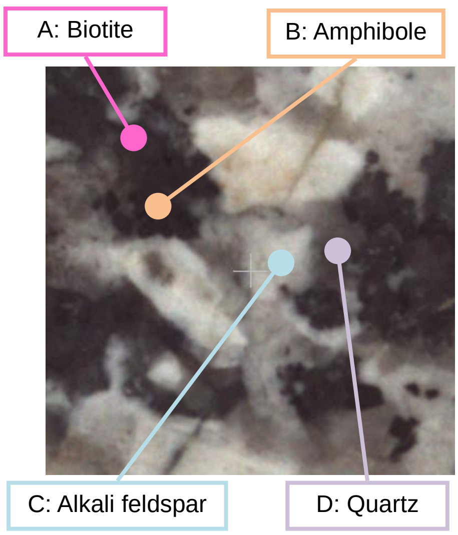

Figure 3 Position of the measured spots.

Thus, the SQX results of the small spot analysis can help identify minerals on the area of the captured image.

Figure 4 shows qualitative charts for representative element lines. It is possible to visually compare X-ray intensity of each spot.

Next, mapping analysis was carried out.
The area indicated by a red rectangle in Figure 5 shows the measured area of the mapping analysis.

Figure 6 shows mapping analysis results by 2D viewer for X-ray intensities of Mg-Kα, Si-Kα, K-Kα and Ca-Kα lines.
The analysis results show that the X-ray intensity distribution of each element matches minerals of the captured image.

Figure 7 shows Mg-Kα intensities by 3D Bird Viewer. Mapping analysis results can be shown by 2D or 3D viewer.

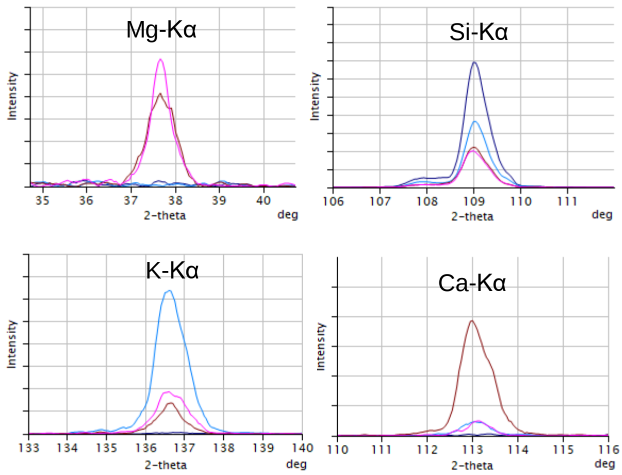

Figure 4 Qualitative chart of Mg-Kα, Si-Kα, K-Kα and Ca-Kα for the measured spots.
A: biotite
B: amphibole
C: alkali feldspar
D: quartz

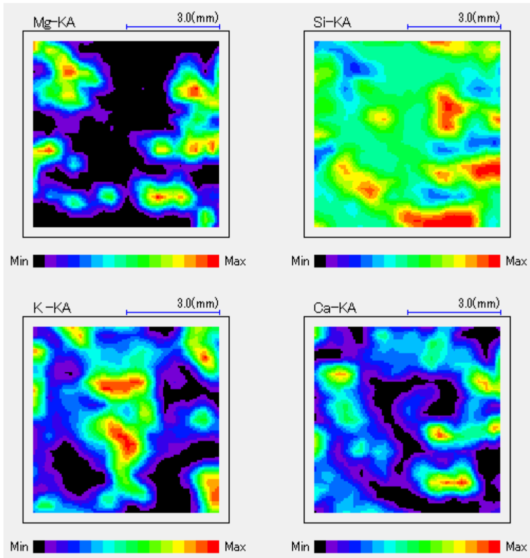

Figure 6 Mapping analysis results by 2D Viewer; X-ray intensities of Na, Mg, K and Fe Kα lines.

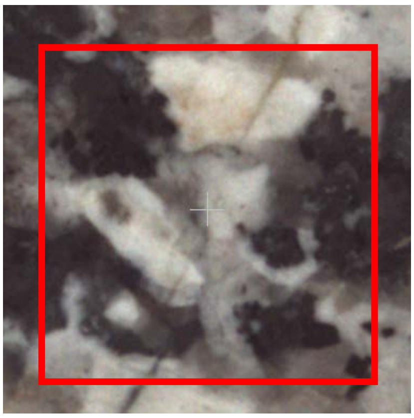

Figure 5 Analysis area of the mapping analysis.

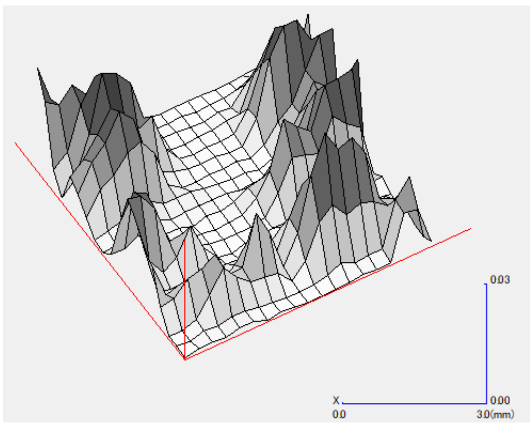

Figure 7 Mapping analysis results by 3D Bird Viewer; X-ray intensities of K Kα line.

Conclusions

Rigaku ZSX Primus IV (tube-above) and ZSX Primus (tube-below), which are general-purpose sequential wavelength-dispersive XRF spectrometers, are capable of point and mapping analysis with small-spot measurement, down to 0.5 mm in diameter.

ZSX Primus IV/Primus have a sample stage, which moves a specimen at a specified position; therefore, it is possible to measure any point on the surface of the specimen at the same optical condition, which enables accurate quantitative, semi-quantitative (standardless) and qualitative analysis for a small spot.

ZSX Primus IV/Primus can be equipped with a digital camera. On a digital image by the camera, point(s), a line or an area (mapping) for small-spot measurement on a digital image of a specimen can be specified.

With ZSX Primus IV and ZSX Primus, it is possible to quickly check inclusions/defects or inhomogeneity on samples without any complex sample preparation, such as EPMA method, as well as conduct daily routine analysis with large-measurement area (e.g. 30 mm in diameter).

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