

APPLICATION REPORT

XRF 169

IDENTIFICATION OF STEEL GRADES OF CUTTING CHIPS USING ZSX Primus

INTRODUCTION

In recent years, in the field of sample preparation for the X-ray fluorescence analysis, demands have been increasing for methods to analyze minute samples and very small quantities of samples. Although conventional instruments have been unable to analyze those samples, they are gradually widening their application ranges thanks to heightened sensitivity and developments in new analysis methods.

Using the ZSX Primus, which can make a qualitative analysis of a specified measurement position in an image of a sample surface that is photographed with a CCD camera, we succeeded in presuming the steel grade of an original material from results of qualitative and semiquantitative analyses of only one cutting chip. Details are described below:

1. SAMPLE PREPARATION AND INSTRUMENT

1.1 INSTRUMENT

Rigaku Industrial Corp. X-ray fluorescence spectrometer ZSX Primus

X-Ray Tube	4kW vertical Rh target type
Sample Observation Mechanism	CCD camera
Method for Specifying Measurement Position	Sample stage
Analysis Area	0.5mm dia.

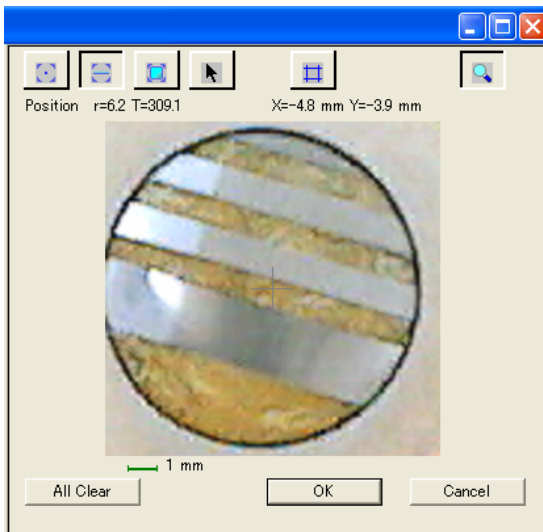
1.2 SAMPLE PREPARATION

A sample was put on an adhesive sheet and pressed before a measurement.

1.3 MEASURING CONDITIONS

Element	F-Mg	Al, Si	P, S	Cl	K, Ca	Ti-U
X-Ray tube	End window Rh tube					
kV-mA	30-100	30-100	30-100	30-100	40-75	50-60
Analysis Diameter	0.5mm					
Slit	S4	S4	S4	S2	S4	S2
Analyzing Crystal	RX25	PET	Ge	Ge	LiF200	LiF200
Detector	F-PC	F-PC	F-PC	F-PC	F-PC	SC
PHA	Differential					
X-Ray Path	Vacuum					

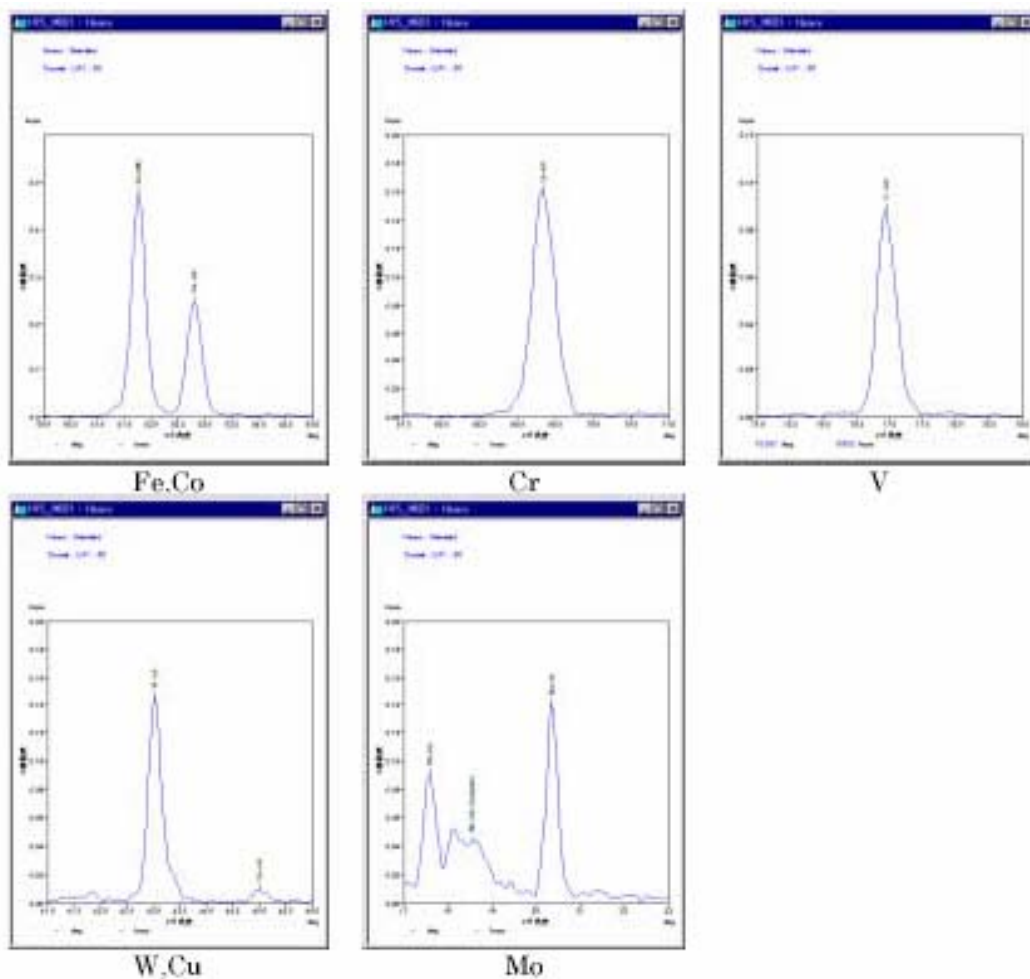
2. MEASUREMENT RESULT



Since the sample was a cutting chip, it was deformed and had the shape of a cone. After putting the sample on the adhesive sheet and pressing it, it was set in a holder for mapping.

The figure on the left shows the sample surface photographed with the CCD camera. A qualitative analysis was made for an analysis area of 0.5mm dia.

As the result of the qualitative analysis, Fe, Cr, V, W, Mo, etc. were detected. As shown below, satisfactory charts were obtained with good angular resolution, which is characteristic of a wavelength dispersion spectrometer, and low scattered ray (background) intensities.



The table below shows the result of a semiquantitative FP calculation for detected components using the sensitivity library stored in the instrument. From Metals Data Book of the Japanese Standards Association, we confirmed that the cutting chip was a kind of tool steel.

Table 1 SQX Analysis Result and Material Standard (mass%)

	SQX	SKH10 (Example)
Mn	0.33	<0.40
Cr	4.5	3.80-4.50
Mo	0.69	-
V	4.1	4.20-5.20
W	11	11.50-13.50
Co	5.0	4.20-5.20
Cu	0.20	-
Fe	74	Residue

3. CONCLUSION

We found that we can obtain satisfactory data even from a very small quantity of sample like that described above if we select an appropriate sample preparation procedure and a suitable analysis method. As for chip samples, we found that the steel grade can be identified successfully even for one chip if we take care about the information depth of X-rays and the sample is thicker than the effective depth. Since in the X-ray fluorescence analysis a sample after a measurement can be recovered and therefore we can assess quantified values even for a very small quantity of sample, new applications can be expected from now on.

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