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XRF Analysis of Impurity Metal Elements in Iron Base Alloy

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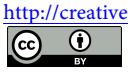

Abstract

The content of Si, Mn, V, Cu and other impurity elements in tool steel samples was analyzed by wavelength dispersive X-ray fluorescence spectrometry (XRF). Using Ag target X-ray tube and the SDD detector (FWHM < 135 eV@5.9 keV). The best working conditions of the instrument are tested before measurement, including tube pressure, flow, filter used and measuring time. The repeatability of the instrument is verified to meet the standard. Finally, the detection limit of the instrument is tested. In the analysis and comparison, linear regression method is used to eliminate the matrix effect among the elements in the sample, and it is found that the multiple linear regression method has a good correction effect on the matrix effect. The results show that the average relative errors of Si, Mn, V and Cu are 3.24%, 3.05%, 0.29% and 0.59% respectively by using the optimal linear regression analysis. The method improves the control requirements of impurity elements in iron base alloy.

Subject Areas

Nuclear Technology and Nuclear Instruments

Keywords

Iron Base Alloy, XRF, Matrix Effect, Linear Regression

1. Introduction

With the rapid development of China's iron and steel industry and national economy, the production capacity of the iron and steel industry is saturated, and the demand for high-performance steel is increasing due to its wide range of uses [1]. Adding specific elements to iron-based alloys will make them have certain characteristics, such as adding CR to iron-based alloys to increase their cor-

rosion resistance; the addition of Mn increases its firmness and strength. Due to the continuous innovation of iron making technology, the requirements for the proportion of elements in iron-based alloys are more strict [2] [3]. It is necessary to study and control the proportion of trace elements in iron-based alloys to improve the properties of iron-based alloys [4].

The analysis of iron-based alloys is mainly to determine the contents of various major and trace elements in various iron-based alloys, so as to control the proportion of elements in iron-based alloys. The contents of Impurity Elements Si, Mn, V and Cu in iron-based alloys were analyzed by X-ray fluorescence analysis.

2. Basic Principle

According to Moseley's law, the X-ray excites the target element in the sample to be tested to produce the characteristic X-ray of the target element, and its intensity is directly proportional to the content (W_i) of the target element in the sample to be tested. The calculation formula is as follows:

$$W_i = A + B \times I_i \tag{1}$$

where, I_i is the characteristic X-ray intensity of the target element in the aluminum alloy; A and B are the undetermined coefficient.

3. Selection of Experimental Instruments and Optimum Working Conditions

3.1. Preparation of Experimental Instruments

The energy dispersive X-ray fluorescence analyzer has high detection efficiency, compact spectrometer, convenient installation and use process, and is suitable for on-site or on-line analysis [5]. Malvern Panalytical is the manufacturer of energy dispersive X-ray fluorescence analyzer selected in this paper, and the instrument model is Epsilon 1. The working voltage range of the instrument is 10 kV - 50 kV and the working current range is 1 μA - 500 μA. It can be measured with or without Cu-50, Cu-300, Al-50, Al-200, Ag and Ti filters.

3.2. Repeatability Inspection of Experimental Instruments

Set the instrument parameters with voltage of 15 kV and current of 15 μA. The same stainless steel sample BX1 was measured 10 times without filter, and the repeatability of the instrument was measured and calculated. According to JJF1047-2011 calibration specification for energy dispersive X-ray fluorescence analyzer, the repeatability of the instrument shall be less than or equal to 3%, that is, the relative standard deviation (RSD) shall be less than or equal to 3%.

When measuring element Si, RSD is 2.364%; when measuring element V, RSD is 0.065%; when measuring element Mn, RSD is 0.00005%; when measuring element Cu, RSD is 0.002%. The RSD calculated in this paper meets the standard, and the repeatability of the instrument is good.

3.3. X-Light Tube Best Working Parameter Selection

The spectrum used to excite the sample is a continuous spectrum in the X-ray primary spectrum, while the working voltage of the X-ray tube and the change in operating current will affect the strength of the continuous spectrum [6]. Therefore, it is necessary to determine the optimal operating voltage and current before starting the sample. The instrument used herein is divided into four channels for measurement, namely Na-Si, K-V, Cr-Co, Ni-Mo, and four measurement channels can employ different operating conditions, and the four elements Si, V, Mn, and Cu analyzed herein are also among these four measurement channels.

The working voltages of the four measuring channels Na-Si, K-V, Cr-Co and Ni-Mo are 25 kV, 38 kV, 32 kV and 22 kV respectively, and the working currents are respectively 15 μA, 14 μA, 45 μA and 145 μA respectively. The filters are Ti, Al-50, Al-200 and Al-200 respectively, and the measurement time of each channel is 600 s.

4. Results and Analysis

4.1. Analysis of Impurity Elements

Select tool steel GSB03-1366 for measurement. The data analysis results of Elements Si, V, Mn and Cu are shown in the table below:

Table 1 shows that binary linear regression also has a good effect on the analysis of Si element in tool steel, in which the maximum absolute error is −0.022%, the average absolute error is 0.008%, the maximum relative error is −7.94%, and the average relative error is 3.24%.

Table 2 shows that the effect of ternary linear regression analysis on the analysis of V content in tool steel is the best, in which the maximum absolute error is −0.011%, the average absolute error is 0.005%, the maximum relative error is −4.66%, and the average relative error is 3.05%.

Table 3 shows that when considering Mn and Cu elements, the influence of other elements can be better eliminated by using quaternary linear regression analysis, with minimal error, and has a good effect in the analysis of tool steel.

Table 1. Analysis results of Si in tool steel samples.

	Si			
Sample no	Recommended value (%)	Binary regression analysis value (%)	Absolute error (%)	Relative error (%)
GF1	0.184	0.197	0.013	7.07
GF2	0.371	0.372	0.001	0.27
GF5	0.700	0.703	0.003	0.43
GF7	0.188	0.195	0.007	3.72
GF9	0.518	0.518	0.000	0.00
GF12	0.277	0.255	−0.022	−7.94
Average value	0.361	0.373	0.008	3.24

Table 2. Analysis results of V in tool steel samples.

	V			
Sample no	Recommended value (%)	Ternary regression analysis value (%)	Absolute error (%)	Relative error (%)
GF2	0.040	0.041	0.001	2.50
GF5	0.204	0.198	−0.006	−2.94
GF6	0.773	0.777	0.004	0.52
GF9	0.236	0.225	−0.011	−4.66
GF12	0.043	0.045	0.002	4.65
Average value	0.259	0.257	0.005	3.05

Table 3. Analysis results of Mn and Cu in tool steel samples.

	Mn				Cu			
Sample no	Recommended value (%)	Quaternion regression analysis value (%)	Absolute error (%)	Relative error (%)	Recommended value (%)	Quaternion regression analysis value (%)	Absolute error (%)	Relative error (%)
GF1	0.518	0.520	0.002	0.39	0.055	0.054	−0.001	−1.82
GF2	0.424	0.422	−0.002	−0.47	0.078	0.078	0.000	0.00
GF5	0.398	0.396	−0.002	−0.50	0.105	0.105	0.000	0.00
GF7	1.312	1.311	−0.001	−0.08	0.835	0.838	0.003	0.36
GF9	0.642	0.644	0.002	0.31	0.076	0.077	0.001	1.32
GF12	0.556	0.556	0.000	0.00	2.883	2.882	−0.001	−0.03
Average value	0.642	0.642	0.002	0.29	0.672	0.672	0.001	0.59

The maximum absolute errors of Mn and Cu quaternion linear regression were −0.002% and 0.003%, the average absolute errors were 0.002% and 0.001%, the maximum relative errors were −0.50% and −1.82%, and the average relative errors were 0.29% and 0.59% respectively.

From the above analysis of Tables 1-3, it can be seen that multiple linear regression analysis has a good effect in analyzing samples with many kinds of elements, and can well eliminate the influence of other elements. Especially for iron-based alloys, the various elements contained in them are basically adjacent in the periodic table. When analyzing the content of one element, the matrix effect caused by multiple elements should be eliminated.

4.2. Calculation of Detection Limit

The detection limit is the minimum value that a certain element in the sample can be accurately detected. The detection limit includes method detection limit and instrument detection limit. The detection limit of the method reflects the sensitivity of the analytical method. The content corresponding to the standard deviation of 3 times the blank value is taken as the minimum detection limit.

Table 4. Experimental results of detection limits of each element.

Element name	Sensitivity factor	Background count	Detection limit (%)
Si	64.020	8292	0.0071
V	1750.854	66000	0.0005
Mn	2578.715	150120	0.0008
Cu	2920.467	454696	0.0011

Select the lowest group of elements in all samples to calculate the detection limit, and the results are shown in Table 4.

Table 4 shows that the detection limit of the instrument meets the analysis needs of Impurity Elements Si, V, Mn and Cu.

4.3. Conclusion

In this paper, after the best working parameters of the instrument are determined through experiments, the tool steel samples are measured. When processing the data, the best multiple linear regression analysis is used to deal with the matrix effect of each element. The measurement results meet the expectations, and it is concluded that the multiple linear regression method has a good correction effect on the matrix effect.

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Conflicts of Interest

The authors declare no conflicts of interest.

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