

Research Article

A Spectral Analysis of Tool Steel by a Low-voltage Spark Source and a Line Array Ultraviolet CCD Sensor

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Abstract

In this work, we propose a novel analysis lines and comparative lines of the six alloy elements of the tool steel (W, Cr, V, Si, Mn, Mo) within the bandwidth of 280nm~310nm wavelength for carrying out the simultaneous quantitative analysis for the six alloy elements of the tool steel. Steels have fundamentally different in their qualities depending on the types and amounts of elements added. In reality, special steels are produced for the several purposes. The correct and rapid analysis of the amounts of elements required and impurities involved for the steel produced is important for the quality of the product. The most frequently used additives in the metal industry are the six elements mentioned above. However, the method of simultaneous and rapid analysis of the above-mentioned elements is not completed. Here the key point is to choose an analysis line and a comparative line for each element used in the analysis. Especially, tool steel plays an important role in the machining industry. The ultraviolet sensors have been extensively used for the spectrum analysis in the steel industry in recent years. We propose a method that quickly analyses tool steel material by combining the ultraviolet sensor with a computer. We choose an analysis line and a comparative line for Si, Mn and Al, which are auxiliary impurities, and for W, Cr and V, which are main alloy components, and make a test curve. According to our recent work, this is the first paper that deals with the analysis of the tool steel by combination of ultraviolet sensor and computer.

Keywords

Tool Steel, Ultraviolet Array CCD Sensor, Spectrum Analysis

1. Introduction

Now spectroscopic analysis has found widely applications in various fields of science and technology and has been an important component indispensable to the production processes of metallurgical and mechanical plants.

The main advantages of the spectroscopic analysis are the fast analysis, the test of composition without any damage of the product, the guarantee of satisfactory accuracy and very high sensitivity, and the universality of the device, method and operations for analysis. Therefore, the spectroscopic

analysis method is economically superior to the chemical analysis one. Now, most of companies in the world have been evaluating quantitatively the chemical composition of the 70~90 percent of raw materials and finished products by the spectroscopic analysis [1-4, 15-20].

Optical emission spectrometry (OES) is a relatively old optical analytical method. In the previous spectroscopes, a human's eye, photosensitive materials or photoresistance, photoelectric cell and a photomultiplier using photoelectric

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effect had been made many uses of photosensors. Nowadays, the production of traditional film and color photographic sensitive materials had been stopped and then CCD (Charged Coupled Device) has been being employed as a main photo sensor. At the beginning, flame was used as a light source for OES. Later, arc/spark OES prevailed in the 1950s and the early 1960s due to its better excitation capability. Meanwhile, in the 1960s, inductively coupled plasma (ICP) was first adopted as the light source of OES by Greenfield [5] and Fassel [6] and until now OES has been dominated absolutely

by ICP-OES. In addition, direct current plasma (DCP); microwave plasma (MP), including microwave-induced plasma (MIP) and microwave plasma torch (MPT); glow discharge (GD), and laser-induced plasma (LIP) are playing an active role as the light source of OES.

Although laser-induced breakdown spectroscopy and others are widely used in the spectral analysis of steel [11-14] and high alloy steel [7-10], there are no papers analyzing tool steel using low voltage spark source.

Table 1 shows the general composition of some tool steels.

Table 1. General composition of some tool steels.

Sort of steel	C	Si	Mn	Cr	W	Mo	V
L18	0.7~0.8	0.2~1.2	<0.5	3.5~4.5	17.5~19	2.5~3.5	1.8~2.3
L9	0.7~0.9	0.2~1.2	<0.5	3.5~4.5	8.5~10	2.5~3.5	2~2.6
L12	0.8~0.9	0.2~1.2	<0.5	3.5~4.5	11~13	2.5~3.5	2.2~2.9

As shown in the table, the content of W, Cr and V in high speed steel is high in general. Therefore, it is very important to choose an appropriate analytical line and comparative line in view of the concentration of the mixture element to be analyzed and the sensitivity of sensor.

2. Experimental Setup

The optical system for the spectral analysis of tool steel is shown in Figure 1.

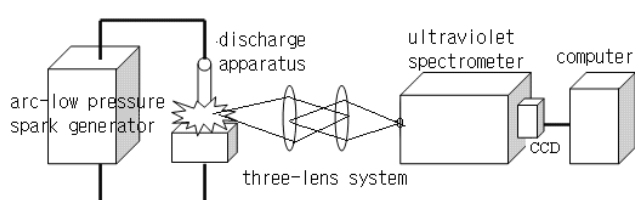


Figure 1. Optical system for tool steel analysis.

This device is a system consisting of a low-voltage spark generator, a discharger, tri-lens illuminating system, an ultraviolet spectroscopy and a computer.

The low low-voltage generator and discharger is intended for discharge of test metal piece.

The ray entering in the spectroscopy through the tri-lens illuminating system is classified by wavelength, and received by the ultraviolet CCD.

Then the information is sent to the computer and is analyzed and processed.

In ultraviolet band, all of element have spectrum and particularly, it is possible to perform spectral analysis for some elements only in ultraviolet band.

CCD sensor used is 14um and total number of pixels is 2048.

In spectral analysis, the accuracy of analysis depends on preparation of samples and discharge condition. It is important to prepare appropriate standard samples.

The standard test piece was chosen in which the concentration was deviated by 2~3 percent for the tool steel under consideration and other physical and chemical composition is almost similar. The standard sample and analytical sample has to be so processed by surface grinder as to have correctly parallel lower and upper face before analyzing work. In order to provide the stability of discharge, a sharp needle is placed by copper electrode.

Then needle should be set so that its cusp is 10~20mm apart from the copper electrode and 6~10mm towards the copper electrode from the center of discharger. On such a condition, the discharge is performed by low voltage arc. The most appropriate conditions for the analysis of tool steel are such that low voltage arc current is 3A, distance between the electrode and the test piece 0.7mm, gap of the main discharge 0.6mm, and 0.2mm.

Figure 2 shows the spectral line intensity distribution along the spectrum image and wavelength captured.

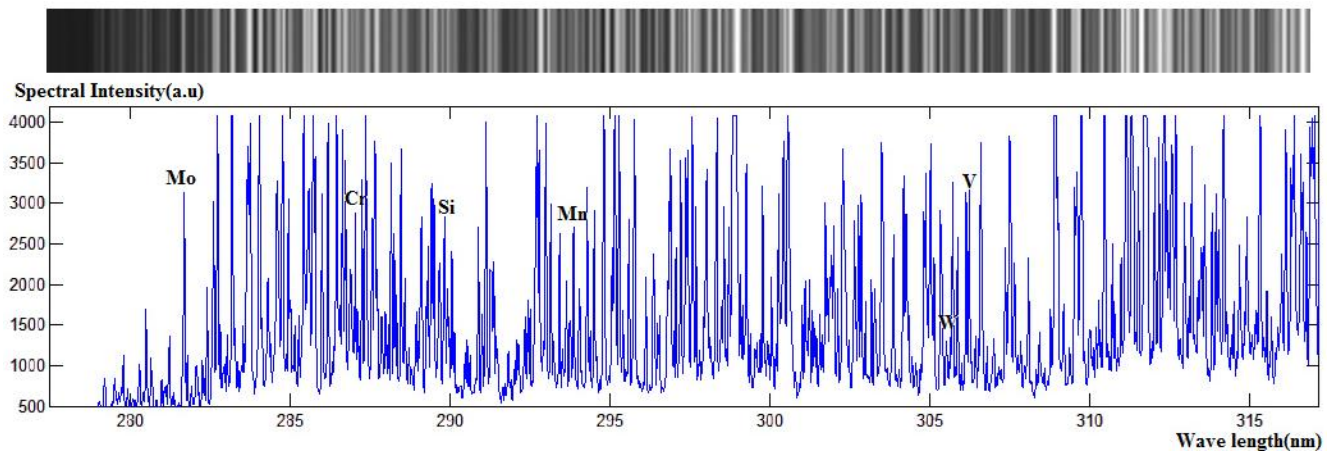


Figure 2. Photographed spectral image and Spectral line intensity distribution versus the wavelength.

3. Selection of Analysis Lines and Comparative Lines and Calibration Curve Fitting

As shown in the previous paragraph, the basic formula for quantitative spectral analysis is given by

$$\lg(I_a / I_c) = a + b \lg C \quad (1)$$

where C is the concentration of alloy element contained in tool steel, $R=I_a/I_c$ is the intensity ratio of the analytical line and the comparative one.

Given observation (I_a/I_c) for known different concentrations, a and b are coefficients determined by the least square method.

Letting $y=\lg R$ and $x=\lg C$, expression (2) is given by

$$y = a + bx \quad (2)$$

If we measure y_i logarithm of light intensity ratio, by taking test pieces with known concentration C_i ($i=1, 2, 3, \dots$), the least square method gives us a and b such that.

$$a = \frac{\sum_{i=1}^m y_i - b \sum_{i=1}^m x_i}{m}, b = \frac{m \sum_{i=1}^m x_i y_i - \sum_{i=1}^m x_i \sum_{i=1}^m y_i}{m \sum_{i=1}^m x_i^2 - \left(\sum_{i=1}^m x_i \right)^2} \quad (3)$$

The table 2 shows the wavelengths of the analytical line and the comparative one considered in our experiment.

Table 2. Wavelengths of the analytical line and the comparative one for analysis of tool steel.

Analytical line	Comparison line
W-305.13nm	Fe-295.39nm
Cr-284.98nm	Fe-284.56nm
Mo-281.62nm	Fe-282.86nm
Si-288.16nm	Fe-287.73nm
Mn-293.31nm	Fe-292.66nm
V-306.33nm	Fe-306.22nm

Based on the analytical line and the comparative line chosen in the above, a test curve is constructed we measured several standard samples with known concentrations six times and constructed the test curve according to equation (1) by the means of measured values. The figures below show the test curves of wolfram, Vanadium, Molybdenum, Chrome, Manganese and Silicon.

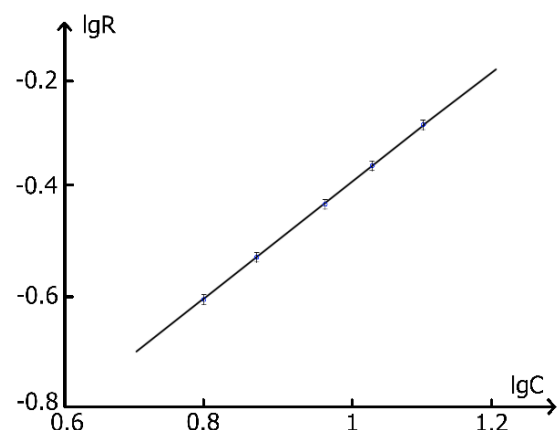


Figure 3. Test curve for Wolfram (W-294.70nm, Fe-295.39nm).

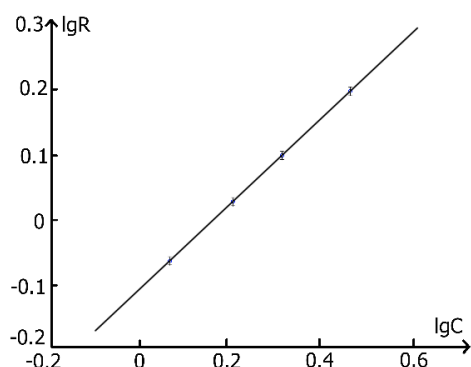


Figure 4. Test curve for Vanadium (V-306.33nm, Fe-306.22nm).

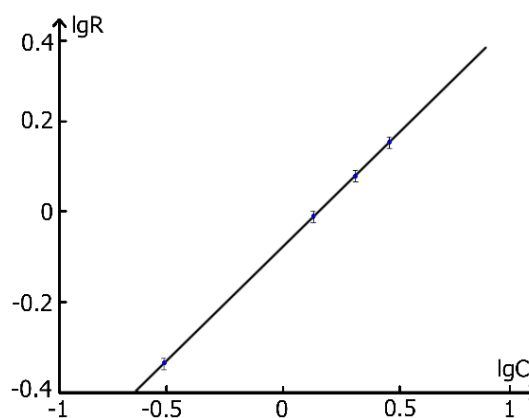


Figure 8. Test curve for Silicon (Si-288.16nm, Fe-287.73nm).

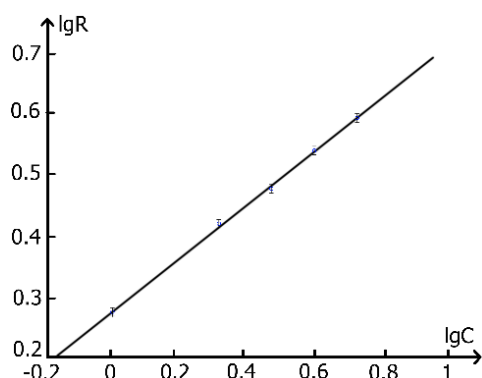


Figure 5. Test curve for Molybdenum (Mo-281.62nm, Fe-282.86nm).

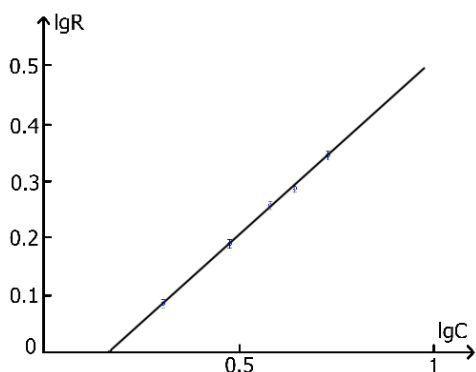


Figure 6. Test curve for Chrome (Cr-284.98nm, Fe-284.56nm).

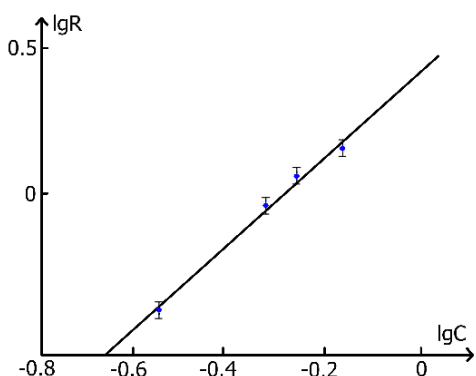


Figure 7. Test curve for Manganese (Mn-293.31nm, Fe-292.66nm).

We can see that linearity is well fitted in the log concentration-log intensity.

Ratio relation from these curves.

Table 3. Data of spectral and chemical analysis for tool steel L9.

Element	Chemical analysis (%)	Spectral analysis by CCD	
		Analytical concentration (%)	Relative error (%)
W	9.02	9.17	1.7
Cr	4.25	4.17	1.9
V	1.97	1.92	2.5
Mo	1.53	1.57	2.6
Si	1.22	1.26	3.3
Mn	0.27	0.26	3.7

We estimated concentrations of and manganese, and showed the values is compared with analysis data by photo in Figure 3.

The relative errors are 1.1~4 percent for the analysis elements.

On order to evaluate reproducibility, analysis of the same test piece was made ten times. Then relative standard deviation was 1~2.5 percent.

4. Conclusion

Taking into consideration of the choice principle of analytical and comparative line and in view of spectrum sensitivity of photo sensor, we have chosen the most appropriate analytical and comparative line within the bandwidth of 280nm~310nm wavelength and constructed test curves.

We have carried out the quantitative analysis for the six alloy elements of the tool steel P9 (W, Cr, V, Si, Mn, Mo). On

our study, the analysis time is under one minute, the relative error 1.1~4% and the relative standard deviation 1~2.5%.

Abbreviations

OES	Optical Emission Spectrometry
CCD	Charged Coupled Device
ICP	Inductively Coupled Plasma
DCP	Direct Current Plasma
MP	Microwave Plasma
MIP	Microwave-Induced Plasma
MPT	Microwave Plasma Torch
GD	Glow Discharge
LIP	Laser-Induced Plasma

Conflicts of Interest

The authors declare no conflicts of interest.

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