Analytical Science　　Prof.**Kazuaki Wagatsuma** (2000.6~)

【Staff Members】

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【Research Activities】

We have been studying the development of new instrumental methods in quantitative elemental analysis.

1. Rapid analytical techniques for industrial use.

Glow discharge optical emission spectrometry (GD-OES) is the most promising technique for the direct determination of solid samples, enabling rapid response of analytical values for process and quality control. We have reported on several novel techniques on the glow discharge emission source in order to improve the detection sensitivity and the analytical precision in GD-OES. A radio-frequency glow discharge plasma source that we have newly developed could excite analyte atoms to give 10-20 times larger emission intensities compared to those by the conventional glow discharge plasma sources. With the bias-current-conduced glow discharge source to be employed in GD-OES, trace elements at a few ppm level could be directly determined. Furthermore, we have developed a voltage modulation technique for controlling the glow discharge plasma, resulting in smaller variations in the emission intensities. This could contribute to the improvement in the analytical precision in GD-OES. These researches were performed in cooperation with Rigaku Industrial Corp. and Kawasaki Steel Corp., and a research group in The Institute for Steel and Iron of Japan. The grants from The Kawatetsu 21st. Foundation and The Institute for Steel and Iron of Japan were provided. Other analytical techniques for rapid quantification have been proposed by use of laser ablation and/or laser-induced plasmas. For this purpose, we have developed two laser excitation sources in atomic emission spectrometry: one is a laser-induced plasma source under reduced argon pressures and the other is a laser-ablation source associated with a helium glow discharge plasma. The former could be employed for on-line analysis of practical materials such as scrapped metals, while the latter could be employed for the fast and precise determination of minor elements in various materials. The emission signals from the laser-induced plasma source are easily detected because the background intensity is very small; it is therefore suitable for the excitation source for on-line analysis. The optimum operation conditions were determined so that the signal-to-background ratio of the emission signals could be maximized. The laser-ablation source with a high-frequency Nd:YAG laser can be employed for the sample introduction to the helium glow discharge plasma, which works as a reliable excitation source because the glow discharge plasma as well as the laser sampling are very stable. Regarding this research, we got the grants from Ministry of the Environment and The Steel Industry Foundation for the Advancement of Environment Protection Technology (SEPT).

2. Quantitative analysis of trace amounts of impurity elements

We have studied the determination of trace elements in high-purity materials yielding important information which contributes to the progress of recent material science. Several techniques for sample pre-treatment in graphite furnace atomic absorption spectrometry (GF-AAS) and inductively-coupled plasma mass spectrometry (ICP-MS) have been reported: co-precipitation with palladium in GF-AAS and with manganese dioxide in ICP-MS. Furthermore, a new pre-treatment technique was developed to reduce the contamination from surrounding gases in the determination of carbon and sulfur by combustion IR-absorption analysis. We have been taking part in cooperative analysis for certified standard materials in The Japan Iron and Steel Federation.

3. Research on >seeds= analytical techniques

We have studied a unique application of the glow discharge plasma as a fast electron source as well as an X-ray source, which is expected to be compact sources in X-ray or electron spectrometry. Further, we have reported on noble techniques for surface analysis: glancing-takeoff angle X-ray fluorescence spectrometry and glancing-exit electron-probe micro analysis (EPMA). New analytical applications in a microwave-induced plasma (MIP) -optical emission spectrometry with an Okamoto-cavity have been investigated. Aqueous samples can be easily introduced into this plasma, because the plasma is sustained at high microwave power. We found that this MIP using nitrogen-oxygen mixed gas could be employed for the direct analysis of organic solvents.

4. Fundamental study in analytical spectroscopy

Mechanisms on the electron emission such as Auger electron and X-ray photoelectron in electron spectroscopy have been studied for obtaining the detailed information from their complicated spectra as well as for performing the quantitative estimation. We have been investigating the excitation mechanisms on atomic emissions excited from various plasmas: ICP, MIP, glow discharge, spark discharge, and laser-induced plasmas, in order to find the criteria for the optimum operation conditions in each spectrometric analysis as well as to compile the database of spectral lines

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2. K. Wagatsuma and H. Matsuta: "Control of bias d.c. current in radiofrequency-powered glow discharge source and its emission characteristics",Spectrochim. Acta, 54B (1999) 527-535.

3. K. Tsuji, K. Wagatsuma, R. Nullens, and R.E. Van Grieken: "Grazing exit electron probe microanalysis for surface and particle analysis", Anal. Chem., 71 (1999) 2497-2501.

4. H. Matsuta and K. Wagatsuma: "Emission characteristics of a low-pressure laser-induced plasma: Selective excitation of ionic emission lines of copper", Appl. Spectrosc., 56 (2002) 1165-1169.

5. K. Wagatsuma, Y. Danzaki, and T. Nakahara: "Comparative study on excitation mechanism of chromium emission lines in argon radio-frequency inductively-coupled plasma, nitrogen microwave induced plasma, and argon or nitrogen glow discharge plasmas", Spectrosc. Lett., 36 (2003) 99-115.

【Plan】

1. Analytical Applications

(1) We will prepare an analytical instrument equipped with the improved glow discharge excitation source as a glow-discharge optical emission spectrometer, so that it can be tested in actual and routine analysis for the process control in cooperation with steel-making companies.

(2) In laser-induced plasma spectrometry, the plasma chambers will be prepared to be applied for the rapid analysis of various types of materials.

(3) For ultra trace analysis, we will proceed to study better pre-concentration and separation techniques without any contamination in inductively-coupled optical emission/mass spectrometry

2. New analytical methods

(1) We will develop a combined emission source of a He glow discharge plasma with laser ablation, and make the prototype apparatus to investigate the emission characteristics of the plasma. Also a dual-cathode hollow cathode emission source will be developed. It is expected that these advanced excitation sources can be employed for controlling the excitation process and the sampling process individually, yielding better analytical performance in glow discharge optical emission spectrometry.

(2) We will proceed to investigate the emission characteristics of the microwave plasma with the Okamoto-cavity, especially in the case where various gases including mixed gases are used as the plasma gas.

(3) By employing an image spectrometer equipped with a CCD detector, a new detection method in atomic emission spectrometry will be investigated. This technique can be applied to the observation on the spatial distribution of a spark discharge plasma or three-dimensional analysis of emissions from a glow discharge plasma.

3. Fundamental Studies

(1) We will further measure the spectrum of various compounds in X-ray photoelectron spectroscopy, in order to understand the process of the electron ejection in more detail.

(2) In atomic emission spectroscopy, the database of spectral lines, which are emitted from various plasma sources, will be further complied. Based upon these data, the excitation mechanisms continue to be investigated.