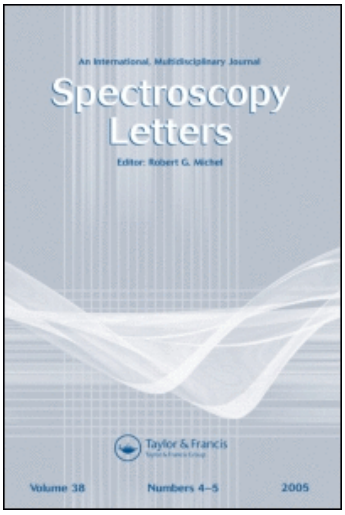

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UTILIZATIONS OF ARGON PLASMA JET AND AEROSOL DESOLVATION SYSTEM FOR EMISSION SPECTROSCOPY

KEY WORDS: emission spectroscopy, argon plasma jet, ultrasonic nebulizer, aerosol desolvation

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INTRODUCTION

During the past decade, various types of DC arc plasma jet, as a new light source, have been developed for emission spectroscopy. The investigated light source has a horizontal graphite cathode and an axial graphite anode. In nitrogen plasma jet, this source is characterized by unstable plasma flame and intense CN band emission caused by the formation of cyanogen flame. In this work, argon was used as a plasma gas for the improvement of stability on a flame and the suppression of interference by CN band emission. Further, desolvation system combined with ultrasonic nebulizer was investigated.

EXPERIMENTAL

A cross-section schematic diagram of the DC arc plasma jet source (Nakano Electronics Co.) is given in Fig. 1. Operating currents at 180 volts were 10 A for nitrogen gas and 40 A for argon gas, respectively. Sample aerosol is generated with a pneumatic or an ultrasonic nebulizer. In the pneumatic nebulization, a gas flow of 4.0 l/min (1.5 kg/cm^2) is used to carry

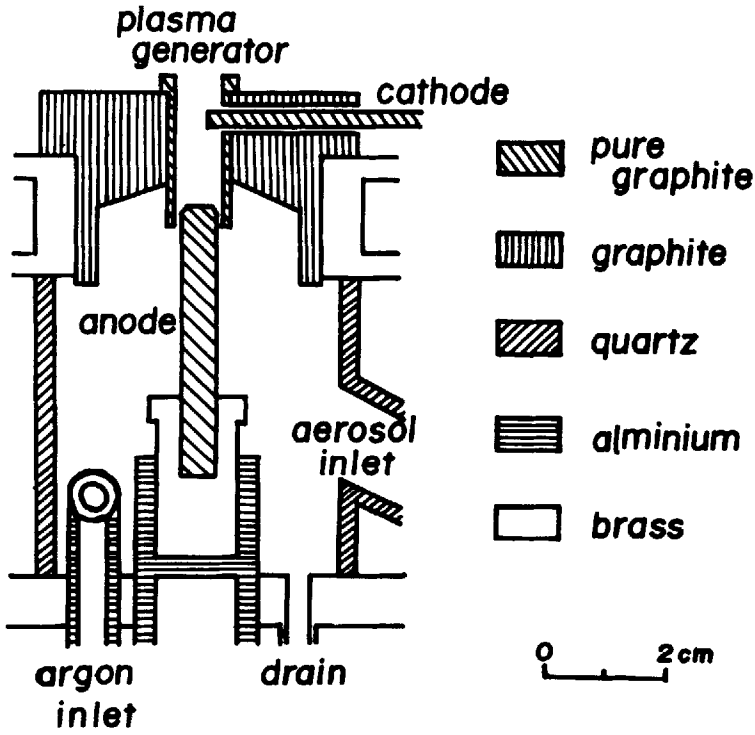

FIG. 1

Cross-section schematic diagram of the source

the sample aerosol to the plasma. Aspirating rate was about 1.5 ml/min. The ultrasonic nebulizer (1.25 MHz, 24 W) was combined with the aerosol desolvation system (tubes were made of quartz), as shown in Fig. 2. A sample solution of 5 ml was placed in the cell, with the bottom made of polyethylene film(0.03 mm thickness). The temperature in the center of the heated chamber was maintained at 140 ± 4 °C. It was made use of an auxiliary plasma gas that argon was flowed at 2.0 l/min.

The spectrum was photographed in a Eastman Kodak SA-1 plate by JEOL JSG-125B asymmetric Czerny-Turner mounting (1.25 m) spectrograph with plane-grating. The range of measurement on the spectra is from 250 to 390 nm and 10 μm slit was used.

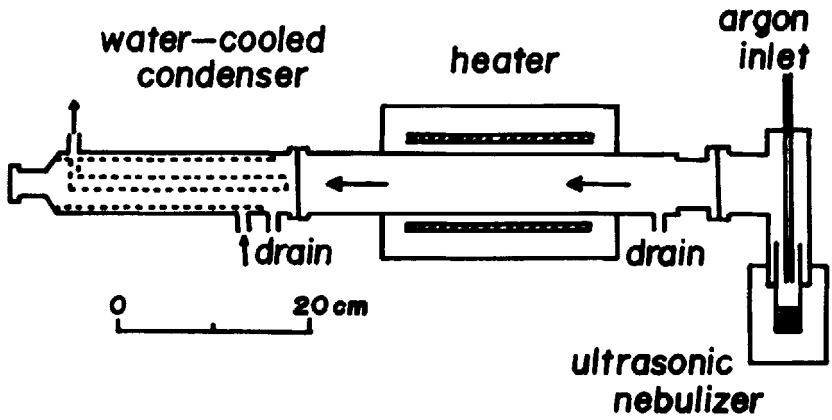

FIG. 2

Aerosol desolvation system

Standard solutions were prepared from the nitrates of metals and standardized by chelatometric titration. Sample solutions were diluted with 0.1 N nitric acid to the required concentration.

RESULTS AND DISCUSSION

In this apparatus, Ishida[1-3] and Nakajima[4] used nitrogen as a plasma gas. Its advantages are the enhancement of spectral line intensities and easy ignition with low operating current. However, background intensity observed was strong owing to the formation of cyanogen. In additional, it caused poor stability in the arcing that cathode spot extremely danced on the tip of the cathode. Graphite electrodes were consumed during the arcing. The position of horizontal cathode tip had influence upon the intensity to be obtained. On the improvement of the disadvantages in utilization of plasma jet, it was attempted to drive the horizontal cathode to the center of the plasma generator at the rate of about 0.5 mm/min. The anode was also consumed at the rate of about 0.3 mm/min, however, it had less effects on the intensity. Even if, the cathode tip position corrected, flame stability was not improved. Further, background intensity was little suppressed as shown in Fig. 3(A), for a case of iron

spectrum by way of example, recorded on the position of 8 mm height from the top of the plasma generator.

Then, a spectrum in argon flow was observed on the position of 4 mm height from the top of the generator. Fig. 3(B) shows this result. As is evident from Fig. 3(B), the background intensity decreases. Argon plasma gave poor sensitivity of metals in comparison with nitrogen. The argon plasma was formed easily at more than 20 A of operating current. Moreover,

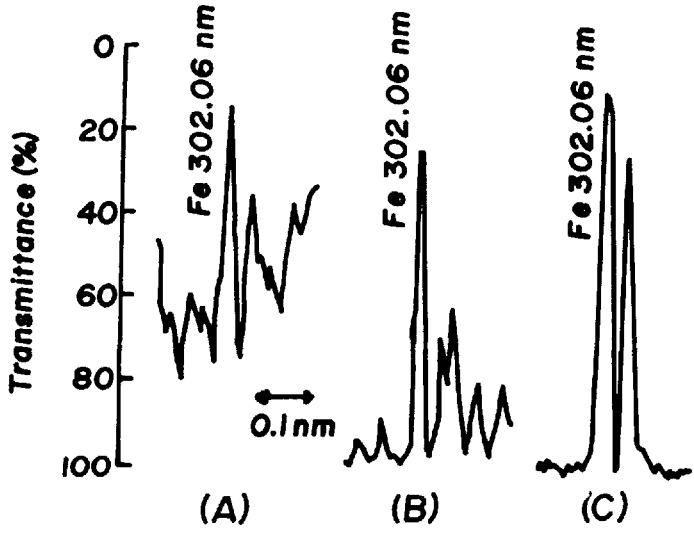

FIG. 3

Spectra of 10 ppm Fe solution

(A) nitrogen plasma, (B) argon plasma,
(C) argon plasma with desolvation system
(30 sec exposure)

cathode spot dancing and electrode consumption were less than in nitrogen plasma jet. In this case, proper mobility of cathode driving was 0.3 mm/min, and the rate of anode consumption was 0.2 mm/min.

With pneumatic nebulizer, the amount of a sample introduced to the flame was not only small portion (ca 8 %) of aspirated solution, but also OH band emission was strongly observed. However, the ultrasonic nebulization with desolvation system gave an increase of the spectral line intensity attended with the suppression of background, as shown in Fig. 3(C). For example, in case of ultrasonic nebulizing, the line intensity of iron was increased with time as shown in Fig. 4. This tendency was almost the same to other metals. It seemed that the less amount of sample solution in the cell, the more nebulized. Behaviors of intensity variation were similar at flow rates up to 6 ml/min, as is clear from Fig. 4. Cathode consumption was negligible against the intensity in practical.

Detection levels of chromium, manganese, iron, nickel, copper, and lead are tabulated in Table 1. Operating conditions of this experiment were as follows: using the ultrasonic nebulizer and the flow rate of argon gas 8 l/min. The cathode was not drived and exposure time was 60 sec after 2 minutes from the start of the nebulization.

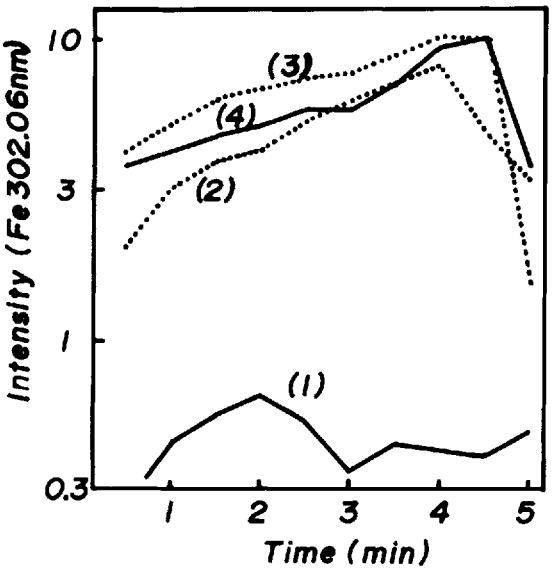

FIG. 4

Effects of argon flow rate on the emission intensity

argon flow rate: (1) 4 l/min, (2) 6 l/min,
(3) 8 l/min, (4) 10 l/min
(30 sec exposure, Fe 10 ppm solution)

TABLE 1

Detection Level of Various Elements

Element	Fe	Mn	Cu	Cr	Ni	Pb
Analytical line (nm)	302.1	279.4	327.4	302.2	300.2	283.3
0.03 ppm						
0.05 ppm			o			
0.1 ppm	o	o	o			o
0.3 ppm	o	o	o	o	o	o
0.5 ppm	o	o	o	o	o	o

(60 sec exposure)

The relative standard deviation on the intensity measured was 6.9 % in case of 0.5 ppm level of iron in the sample solution.

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