

## Emission Spectrographic Analysis of Aluminum Base Materials by DC-ARC Excitation

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**Summary:** An emission spectrographic method employing dc-arc excitation has been described for the determination of Bi, Cr, Cu, Fe, Mg, Mn, Ni, Pb, Si and Zn in aluminum base materials. The samples were dissolved in nitric acid, dried and converted to oxide form by heating in a tube furnace at 900°C under oxygen atmosphere before analysis. Treated samples were mixed with graphite in 2:1 ratio and excited in 10 Amp dc-arc for 30 seconds. The lowest detection limits for different elements lie between 10 and 40 ppm.

### Introduction

Aluminum base alloys find their wide spread use in general purpose as well as specific applications where high strength to weight ratio, ductility and corrosion resistance are the properties dictating their use [1]. Both the alloying elements and the impurities present induce specific changes in physical and mechanical properties. The alloying elements affect fluidity, hardness, corrosion resistance and machinability of the aluminum alloys [2]. Therefore the exact knowledge of the composition of the alloys alongwith the picture of impurities would be necessary to delineate the application of these alloys for specific purposes.

Emission spectrography has been employed in the past for the analysis of aluminum alloys [3-11]. Different methods employing point to plane technique [6-8] for the analyses of such alloys have been adopted by ASTM. Neil [9] and Jaycox [10] have used dc-arc while Olenovich *et al.* [11] have utilized ac-arc as excitation sources. In the present paper, an emission spectrographic method employing dc-arc as the excitation source has been described for the determination of impurities and alloying elements in aluminum base materials.

### Results and Discussion

Before conducting the actual analyses various exposure parameters were studied. Graphite was used as a buffer because it resulted in smooth exposure and a stable arc. The volatilization behaviour of elements under study was mapped by utilizing "Moving Plate Technique". The results for some of the elements

are presented in Figure 1. It is obvious from Figure 1 that maximum volatilization of alloying impurities in aluminum oxide occurs in 30 seconds. Thus, an exposure time of 30 seconds was selected.

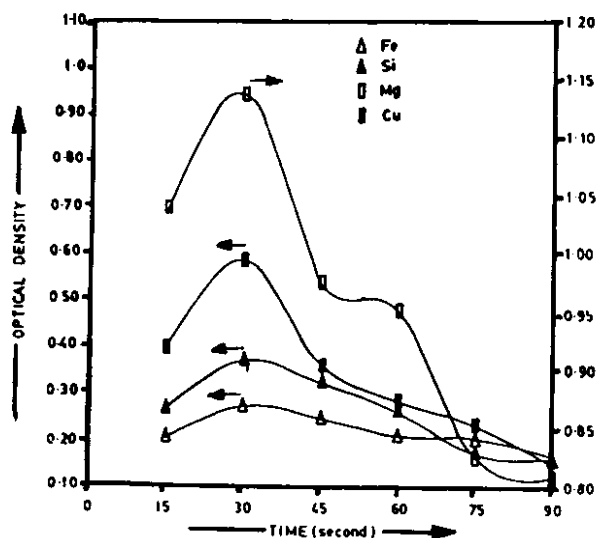


Fig:1 Volatilization behaviour of some alloying elements.

Effect of arc power on the detection of various elements was studied by varying the current level in the range of 5-15 Amp. The results depicted in Figure 2 show that 10 Amp excitation current was the most suitable as at higher current values significant background was observed which resulted in a decrease in the signal to background ratio. Similarly, the amount of charge and type of sample electrodes were selected keeping in view the concentration range of various elements under

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Table-1: Details of equipment and instrumental conditions

a) Equipment	
Jarrell Ash 3.4 meter Ebert Spectrograph with a plane grating (591 grooves/mm) having a dispersion of 2.5°/mm in second order along with a standard varisource.	
b) Excitation and Photographic Processing	
Spectral region	2200-3400 Å
Arc current (dc)	10 amp
Slit width	10 micron
Analytical gap	3.0 mm
Exposure time	30 sec
Electrodes	High purity graphite Ringsdorf, Germany
i) Counter	RW0063
ii) Sample	RW0006
Photographic emulsion	Kodak spectrum analysis No. 1
Plate processing	3 min at 18°C each in Kodak D-19 developer and Kodak rapid fixer

Table-2: Detection limits of selected analytical lines

Element	Wavelength (Å)	Range of estimation (%)
Bi	3067.72	0.002 to 1.0
Cr	2835.63	0.002 to 0.5
Cu	3247.54	0.004 to 2.0
Fe	2488.14	0.004 to 2.0
Pb	2833.07	0.007 to 1.0
Mg	2779.83	0.001 to 0.5
Mn	2794.82	0.002 to 1.0
Ni	3002.49	0.004 to 0.5
Si	2514.32	0.0325 to 5.0
Zn	3345.02	0.031 to 0.5

investigation. The aim of the present work was to produce best suited exposure conditions for the elements commonly associated with aluminum base materials rather than optimizing the exposure conditions for minimum detection limits. The detection limits for different elements are in the range of 10 to 40 ppm. The selected analytical lines and the range of detection are given in Table 2. The working curves for these elements plotted after emulsion calibration (using 2-step filter method) are reproduced in Figure 3 and 4.

The analysis data on three mixtures along with the known values are presented in Table-3. Moreover these mixture were also analyzed using atomic absorption spectrophotometry (AAS), the results of which are depicted in Table-3. Silicon was however estimated by spectrophotometry for comparison purposes. It is evident from the results in Table-3 that emission spectrographic data compares well with that of AAS thus suggesting that this method can be used for analysis of aluminum base materials. This method is being employed in our laboratory for last many years.

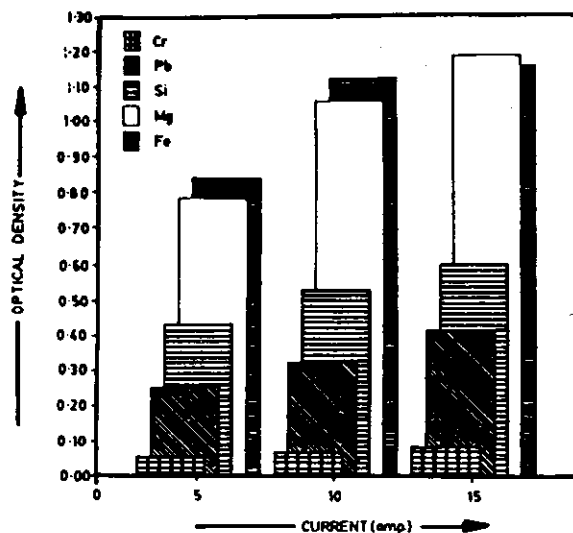


Fig. 2: Effect of arc power on spectral intensity.

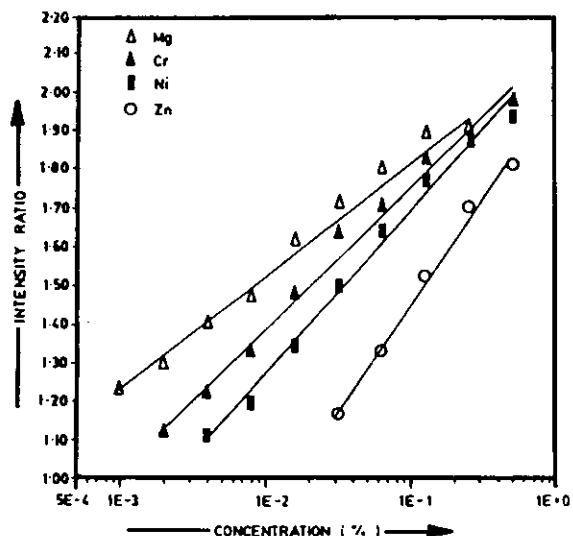


Fig. 3: Working curves of Mg, Cr, Ni and Zn in Al - Matrix.

## Experimental

### Chemicals

All chemicals used for the preparation of standard/standard mixtures were of spec pure grade supplied by Johnson and Matthey Company, UK. The acids were of Analar grade. Recommended Kodak chemicals were used for photographic processing [12].

### Equipment

Details of the equipment and instrumental conditions are enlised in Table-1.

Table-3: A comparison of analyses by Emission Spectrography and Atomic Absorption Spectrophotometry

Element	Mixture No. 1			Mixture No. 2			Mixture No. 3		
	Concentration (%)			Concentration (%)			Concentration (%)		
	Known value	Analyzed by		Known value	Analyzed by		Known value	Analyzed by	
	ES	ASS		ES	AAS		ES	AAS	
Bi	1.0	0.99	1.08	0.030	0.028	0.032	0.004	0.0045	0.0043
Cr	0.5	0.51	0.52	0.015	0.014	0.016	0.005	0.0045	0.0049
Cu	2.0	2.01	1.97	0.060	0.059	0.057	0.008	0.0087	0.0079
Fe	2.0	2.01	1.98	0.100	0.103	0.099	0.040	0.0391	0.0413
Pb	1.0	1.04	1.04	0.030	0.029	0.030	0.010	0.0098	0.0102
Mg	0.5	0.48	0.52	0.010	0.009	0.010	0.002	0.0018	0.0021
Mn	1.0	0.99	0.97	0.020	0.020	0.019	0.004	0.0041	0.0038
Ni	0.5	0.49	0.50	0.010	0.009	0.011	0.007	0.0068	0.0071
Si	5.0	5.08	5.07*	0.100	0.102	0.108*	0.040	0.0390	0.0421*
Zn	0.5	0.51	0.52	0.110	0.099	0.104	0.042	0.0392	0.0417

\*Analyzed by spectrophotometry

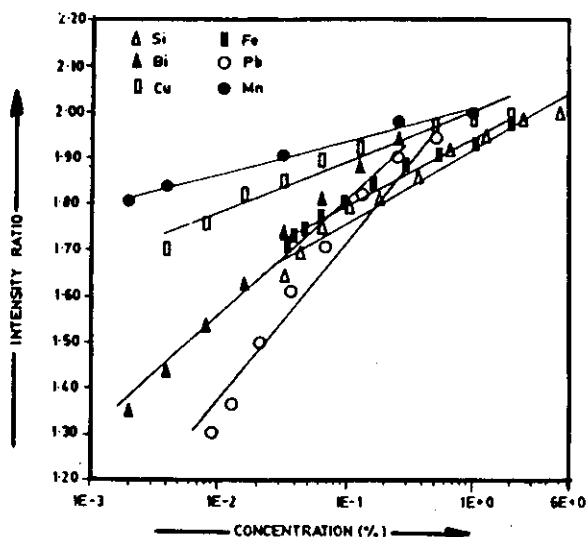


Fig. 4: Working curves of Si, Bi, Cu, Fe, Pb and Mn in Al matrix.

#### Preparation of standards

Oxides of Bi, Cr, Cu, Fe, Mg, Mn, Ni, Pb, Si and Zn in appropriate quantities were added to aluminum oxide, so that the concentration of these impurities was 1, 0.5, 2.0, 2.0, 0.5, 1.0, 0.5, 1.0, 5.0 and 0.5 percent respectively. Mixing was carried out in plastic wares. A series of standards were prepared by diluting the above standard with aluminum oxide powder.

#### Pre-treatment of samples for analysis

The samples to be analyzed were dissolved in dilute nitric acid and evaporated to dryness at low temperature. The residue was converted to oxide form by heating at 900°C in a tube furnace over a constant supply of oxygen gas. The oxide

thus formed was ground to fine powder in a mortar and mixed with graphite in 1:2 ratio. Plastic bottles and mixing tools were employed where appropriate. For excitation, a 20mg charge was placed in a high purity graphite electrode.

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