

THE SPECTRO-CHEMICAL ANALYSIS OF THE BEARING ALLOYS

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ABSTRACT.—The technique of spectro-chemical analysis of the bearing alloys with copper base has been developed by means of Hilger medium quartz spectrograph. The ranges of the different elements determined are:

Sn—6.50 to 7.90%, Pb—3.0 to 3.65%, Ni—0.70 to 1.0%

The determinations agree well with those found chemically.

INTRODUCTION

The technique of spectro-chemical analysis of the copper base alloys used in the sheet mill as bearing material at Tata has been developed. Its composition is:

Sn—6.50 to 7.9%, Pb—3.0 to 3.50%, Ni—0.70 to 1.0%.

the balance being copper and the trace elements. In the industrial laboratories of different countries the compositions of various copper base alloys are being spectro-chemically determined; but the estimation of such high per cents of both Sn and Pb in an alloy has been rare. From the Bell Telephone Laboratories Jaycox (1945) has published a very comprehensive paper on the "The spectro-chemical analysis of copper base alloys." The percentages of Al, Pb and Sn in the alloys vary from 0.30 to 15.0; for some elements it was as high as 40.0. As it is not feasible to determine spectro-chemically such high per cents by arcing and sparking the solid samples directly, the samples have been taken into solutions diluting the latter with copper nitrate. The ranges of different alloying elements have been thereby reduced to 0.01—2.0 % with respect to copper. The spectra have been then obtained by drying the solutions on graphite electrodes and by exciting in the D.C. arc. Besides this additional chemical work there is difficulty in having the same amounts of different solutions dried in the same way on the electrodes. Moreover, the inaccuracy—say in 2.0 % determination will be increased twenty times if the actual percentage is 40.0. The method adopted in the present paper is quite different from the above in as much as the solid samples themselves have been used for sparking even up to 7.8%.

A few papers from the Russian sources have recently been published on the spectro-chemical analysis of the copper base alloys:

- (I) Spectro-chemical analysis of Pb in brasses, (1945)
- (II) Spectro-chemical analysis of tin bronzes, (1945)
- (III) Spectro-chemical analysis of bronzes, (1945)

As only the abstracts of these papers have been published in English or American journals, the methods adopted in or other details of these works are not known to the present authors.

In the Bragg Laboratory, U. K., a large number of gun metal samples were spectro-chemically analysed. Zinc, lead and nickel were determined by the spark method and the trace elements by the D. C. arc technique. They, however, found it more convenient to determine tin *chemically*. The percentage ranges of the elements estimated by the spark method were :

Zn—0.70 to 6.0, Pb—0.10 to 4.0, Ni—0.05 to 0.4, Fe—0.05 to 0.4.

Zinc was used partly to replace tin as a war-measure. In the investigation to be reported the percentage of tin determined is about 7.8%. It can be spectro-chemically determined from the same spark spectra used for the estimation of Ni and Pb. There is thus a considerable saving of time if Sn is determined spectro-chemically instead of chemically. Some Fe-lines are also found on the plates which are strong enough to permit the photometric measurements of their blackenings. The percentage of Fe, however, has not been determined for want of suitable standards.

EXPERIMENTAL

Hilger medium quartz spectrograph has been used for photographing the spectra. The following are the conditions for excitation :

Voltage	...	10,000
Capacity005 μ F
Inductance	...	0.06 or 0.13 mh
Spark gap	...	2.0 mm.
Upper electrodes	...	Pointed carbon rods
Lower electrodes	...	Conical sample with flat top about 2 mm in diameter.

Five standard samples of variable compositions have kindly been prepared by the Tata Metallurgical Department in the small high frequency furnace of the Research Laboratory for this investigation. It became necessary on account of the fact that the variation in the compositions of the alloy made at Tata is not sufficiently large to enable one to draw standard curves properly. The spark spectra of at least four of these standards have been photographed on each plate. With the help of the curves drawn from the spectra of these standards the compositions of the unknown samples have been determined. It is expected that these standards are more segregation-free than the alloys made in the works. They give almost always smooth curves whereas there have been observed occasional variations in the readings obtained for the same unknown samples from different plates which cannot be attributed to experimental error. However, the results obtained for the unknown samples agree very favourably with those estimated chemically. It would have been ideal to re-melt the unknown samples and make fresh rods from them under the same laboratory conditions used for making the standards.

An attempt has been made to improve the accuracy of the results by the emulsion calibration of the photographic plates. The method adopted for this purpose was the one developed by Churchill (1944). This is known as "Two

lines" method and consists of giving a series of exposures on the photographic plate of the spark spectrum of one representative sample used in the investigation; every thing else remains the same excepting the illumination which is varied. In the present investigation the variation has been effected by gradually changing the time of exposures. Two lines with appropriate blackening containing the range of blackenings to be met within the course of the investigation are selected from these spectra. For comparing the intensities of the spectral lines used for quantitative analysis a non-recording microphotometer has been used. The light from a steady source, a lamp running at a constant voltage, passing through a system of lens and prism, falls on the photographic plate containing the spectra of the various samples. After its passage through the selected spectral lines another system of lenses and a mirror reflect the patch of light on the slit of a photo-cell. The current generated in the photo-cell is measured by a galvanometer. The photographic plate which is placed on a horizontal platform can be given two motions at right angles to each other in the horizontal plane and a rotation about a vertical axis; with these each and every line belonging to the different spectra photographed on the plate can be placed in the path of the light and their intensity, rather blackening, measured. The photometric readings of these lines for the whole series of spectra are plotted one against the other. The final emulsion curve is prepared from these preliminary curve with the help of an approximate ratio of the intensities of the two lines, if the actual ratio is not known. The intensity ratio of the spectral lines used for percentage determinations is then obtained by referring their photometric readings to the emulsion curve. The correction thus obtained has not been applied to all the plates taken during the investigation but where applied a decided improvement has been noticed. A few results which are very bad, probably due to segregation or other defects have not improved at all, on the contrary some of them have even become worse. The introduction of an auxiliary spark gap in series with the analytical gap, which makes the discharge more violent, has not been found to increase the accuracy of the result whether corrected or not.

RESULTS

Table I contains the results obtained for a few samples of the bearing metals. The samples have been used more than once, the results are concordant and agree well with the chemical determinations* The few results which differ considerably from those obtained chemically cannot be ascribed to the experimental errors; they are perhaps due to segregation or other defects inherent in the samples. It should be remembered that in the chemical analysis, one gets a kind of average effect but in the spectro-chemical analysis, specially when the method of excitation of the

* Chemical determinations have been made by Mr. R. C. Dutt, the Chemist of the Research Laboratory.

spectrum by spark is employed only a very small area on the surface of the rod samples is made to emit light. If it happens that a segregate or an inhomogeneity is present on the surface the nature of the light emitted, and consequently the analysis obtained from it, will not be a representative one.

The techniques are still being improved and it is hoped that not only the higher percent of the above elements but also the trace elements present in this kind of alloys will be correctly evaluated from the same spectra.

TABLE I

Sn		Pb		Ni	
Chem	Sp Chem	Chem	Sp Chem	Chem.	Sp. Chem.
7.84	7.78	3.63	3.65	0.91	0.93
7.00	7.05	3.08	3.00	0.70	0.70
—	—	3.08	3.00	0.70	0.72
7.00	7.00	3.08	3.05	0.70	0.70
6.93	6.80	3.36	3.36	1.00	0.98
7.09	—	—	—	—	—
6.54	6.58	3.25	3.16	0.88	0.92
6.80	6.70	—	—	1.06	1.05
6.54	6.60	3.25	3.20	0.88	0.92
6.35	6.35	3.15	3.23	0.91	1.00
6.35	6.32	3.15	3.32	0.91	0.98
6.30	6.30	3.46	3.40	0.94	0.92
7.84	7.85	—	—	—	—
7.00	7.00	—	—	—	—
7.00	7.00	—	—	—	—
7.00	7.00	—	—	—	—
6.54	6.54	—	—	—	—
6.54	6.40	—	—	—	—
6.30	6.35	3.46	3.38	0.94	0.92
6.80	6.84	3.22	3.15	1.06	1.10
6.35	6.45	3.15	3.20	0.94	0.94
7.25	7.25	3.15	3.35	1.00	0.97
6.30	6.30	3.46	3.50	0.94	0.95
6.54	6.55	3.25	3.33	0.88	0.87
7.00	7.00	2.98	2.85	0.70	0.70
6.30	6.30	3.45	3.50	0.94	0.92
6.54	6.60	3.25	3.33	0.88	1.02
7.00	6.60	2.98	2.93	0.70	0.73
6.80	6.90	3.22	3.20	1.06	1.10
6.35	6.20	3.15	3.20	0.94	0.95
7.25	7.25	3.15	3.36	1.00	1.02

The bold figures are some of the values which have been corrected by the emulsion calibration curve.

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