
EMISSION SPECTROSCOPY IN THE STEEL INDUSTRY

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Large scale production of high grade electric furnace steels demands rapid, accurate analytical control. Without exaggeration it might be said that the analytical laboratory controls the steel industry—it is impossible to make good steel without close chemical control.

To produce a heat of alloy steel, the furnace is charged with steel scrap or pig iron. After the melting period, when the molten bath has been thoroughly mixed, a sample is cast and sent to the chemical laboratory. A complete analysis is made for elements which appear in the specification of the steel on order. This analysis is reported to the furnace operator, where it is used as a basis for calculating the ferro-alloys additions which adjust the alloy contents to the desired specification. The ferro-alloys additions are allowed to melt and diffuse uniformly throughout the bath after which another sample is sent to the laboratory. If the analysis of this test indicates that the various elements are present in the proper proportions, the heat is tapped and cast into ingots.

The importance of speed, by which the chemical elements are analyzed and reported, is obvious since the furnace operators cannot proceed with the processing until the exact analysis of the bath is known. The molten steel is maintained under a fused blanket of slag, which may be oxidizing or reducing in nature, and may

vary the percentage of certain elements. Therefore, to be of value, the chemical test must be rapidly analyzed, so that the analysis represents the conditions in the bath before any changes can take place.

Until comparatively recently this chemical control had been accomplished by wet chemical methods. While the accuracy of wet analysis is not questioned, the speed is usually much too slow for present-day production. Ordinary elements such as Mn, Cr, Ni, Mo, Cu and Va may be analyzed within a half hour. Other elements like Si, W, Al, Zr, Ti, B and Co may require as long as 8 to 12 hours to determine the amount present. Often a series of heats is scheduled in which the analysis of the first heat is used as a basis for producing the other heats, hence a prolonged analysis delays production until it can be finished. In the usual steel mill control laboratory one analysis is required, for each element, so that time delays may be held to a minimum. It is evident that quite a staff of chemists is necessary if very complex analysis is required. A laboratory of this type required much valuable floor space with quite a sum spent for chemicals, glassware and apparatus.

Therefore, the need for a more rapid chemical report made the emission-type spectrograph a boon to laboratories. While the spectrograph had been used for quite some time in other fields, the first instrument to be used successfully for ferrous melting control was installed in a foundry in 1935. Since that date many have appeared in the control laboratories of hot-metal industries.

A Baird Spectrograph was installed in the Timken Laboratory in 1943 for the analysis of tests from the melt shop. This instrument is equipped with a three meter focal length Wood's grating, ruled 15,000 lines per inch, set in an Eagle mounting. Samples for spectrographic analysis are cast in the form of rods, $\frac{7}{32}$ inches in diameter, by pouring the molten metal into a mold or sucking it up in a glass tube. The cast pins are sent to the laboratory by a pneumatic tube system, where they are carefully ground to a point of 140° included angle and placed in the electrode holders.

Power from a high voltage arc or spark source is applied and spectral lines from the emitted light are photographed on Eastman spectrum analysis No. 1 plates. The plate is then developed, washed, dried and cooled to room temperature in a dark room. The density of the line for the element required is read on a densitometer and calculated to percent, after which it is reported by telautograph to the furnace operator.

The headaches caused by interfering elements in wet chemistry are eliminated. Spectrographic analysis avoids the necessity of waiting several hours for certain elements, as when wet chemical methods are employed. It is reported that a large steel producer increased tonnage approximately 30 percent after a spectrograph was installed, since heats could be diverted to other specifications prior to tapping, when detrimental elements were present.

With the spectrograph there is a substantial saving in time over the wet chemical methods for special elements such as aluminum, but these elements are not usually requested for control analysis. Control elements are normally Mn, Cr, Ni, Mo, V and Cu. Depending on the number of elements requested, a report of spectrographic analysis can be completed in from 10 to 25 minutes, which time is not too much better than a well organized wet chemical laboratory. Approximately the same number of employees are necessary, if delays are to be held to a minimum.

The last word in rapid control analysis was realized with the development of the direct-reading spectrometer. In this instrument, the photographic stage has been entirely replaced by sensitive electron multiplier tubes, which pick up the emitted light at the various wavelengths and report it as percentage on calibrated clock dials within 35 seconds after a sample has been placed in the electrode holders.

The use of photo tubes eliminates all of the possible errors associated with plates or film, dark room developing and processing, densitometer readings and calculating boards.

A Baird Associates-Dow Direct-Reading Spectrometer was installed in the Timken control laboratory in May 1947 and was the first instrument of this type to be used in the steel industry. A simultaneous analysis of alloys is made; therefore, a total of eight chemical elements can be reported to the melt shop approximately six minutes after a sample has been delivered to the laboratory.

For over three years the direct reader has been used to furnish analyses of all types of steel, except the stainless grades, for elements in the following ranges:

Element	Calibration Range	Element	Calibration Range
Manganese	0.05-1.60	Copper	0.02 -1.20
Silicon	0.01-1.00	Vanadium	0.01 -0.30
Chrome	0.03-7.00	Tungsten	0.05 -5.40
Nickel	0.02-5.00	Aluminum	0.002-1.60
Molybdenum	0.01-1.00	Tin	0.007-0.10

The direct reader is operated 24 hours a day, seven days a week, with one operator on each eight-hour shift, instead of three, as with the spectrograph. The operator can very easily take care of the necessary elements, on all tests, from seven electric and three openhearth furnaces. Operators were selected from the chemical laboratory force, with the thought in mind that, since the number of chemists would be reduced when the spectrometer was installed, it would be essential that these operators could return to the chemical laboratory and carry on control analysis in case of a major spectrometer breakdown. To date this has not been necessary.

The spectrometer is located in an air-conditioned room, which is maintained at a temperature of 75° F and 45 percent relative humidity. It is felt that air conditioning is quite essential to good operation; that is, the atmosphere surrounding the analytical gap is held uniform, but more important, the electronic circuits are not subjected to absorption of moisture due to varying atmospheric conditions.

The instrument is well insulated against shock, the customary vibration found in steel and rolling mills causing no interference. The exit slits, which pass the emitted light from the grating to the photomultiplier tubes, are of the fixed type. In over three years they have never been changed or adjusted, due to varying mechanical or physical changes in the spectrometer. In a recent check, it was found that the light from the spectral lines was still passing through the exact center of each exit slit, which is thought to be quite remarkable, since the light is reflected approximately 24 ft. and the slightest movement will cause the light to miss these narrow slits which are from 50 to 200 microns in width.

Sixty-five electronic tubes are used in the various circuits in the spectrometer, and to date there has never been a shutdown caused by a burned-out tube. During vacation period shutdowns, the tubes are checked and weak tubes are replaced, even though they cause no trouble in operation. Many of the original tubes are still in use, which indicates well-designed circuits.

The location of trouble has been simplified since a channel has been designed for each element and the circuits which control that channel are built into a drawer, which is easily replaced with a spare drawer in case of trouble. The troublesome drawer is then repaired by the operator and held as a spare for future use. Tube fatigue is practically unknown, as shown by experiments in which from 20 to 40 exposures are made in rapid succession with no abnormal variations.

In order to check the effect of a shutdown, the spectrometer was standardized before it was turned off for a three-week vacation period. At the conclusion of this time the power was turned on and the electronic circuits allowed to warm up for 20 minutes. The same standard pins were then sparked and the analyses for all elements were in excellent agreement with the results obtained before the shutdown.

In a recent survey of a two-year period, it was found that results from the

spectrometer were used to control the melting of 14,184 heats of steel. This constituted a total of 951,207 tons, on which 75,470 tests were submitted, making a grand total of 1,021,250 determinations, reported by the aid of the direct-reader.

The real value of savings, possible through rapid control analysis, is apparent since melt shop time can be calculated at approximately one dollar per minute. It is obvious that any gain in analytical time will effect quite a saving, especially when considered on an annual basis. Costly erosion of ceramic furnace linings may be reduced, for with rapid analysis the bath no longer has to be held at approximately 3000° F. while waiting for preliminary analysis before adjusting the alloy content.

It is felt that in the near future this type of instrument will be used quite extensively. With the improvement in accuracy and the shorter period required to report analysis, it is felt that the direct-reading spectrometer is, by far, the greatest contribution that the control laboratory has ever made towards effecting a savings in the melting department. This is especially true today with the emphasis being placed on steel production due to the present emergency.
