Some Recent Trends on Iron and Steel Research and Control Methods

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Blast furnace practice

(*a*) *Investigations on raw-materials and sintering characteristics*: Although in recent years a considerable amount of work has been done towards beneficiation and treatment of blast furnace raw materials leading to increased production in a given furnace, there is considerable scope for further investigation in this field with reference to indigenous materials and particularly sintering of ore blended with other materials. Scientific definition and standardisation of so-called "Sintering characteristics", "Reducibility of Sinter", need further studies and the effects of different constituents and sizes on the rate of sinter production and allied activities may very well lead to fruitful results. Some of the recent trends in this connection are:

(*i*) quick analysis of constituents in the sintered material by X-ray fluoroxopy—particularly the FeO, Fe_2O_3 and Fe_3O_4 contents by diffraction before and after a given treatment.

(*ii*) effects of injection of oxygen on the one hand and reducing gases on the other during sintering. It has been found for example that injection of O_2 instead of raising the maximum temperature Tmax and narrowing the zone of combustion, lowers the Tmax value and widens the zone of combustion. This is because the surface activity of coke increases with oxygen pressure and hence the ignition temperature is lowered with the result that combustion starts before the charge is preheated to a higher temperature. This results in a widening of the combustion zone and lowering of Tmax.

(*iii*) investigations on "Reducibility of Sinter" either by varying the CO concentration at definite time intervals and measuring the amount of oxygen removal or preferably by the SCICE-method—developed recently by the British Iron and Steel Research Association.

(*iv*) blending of CaO in sinter is helpful in reducing the thermal requirements and hence coke consumption and therefore helpful for reducing S in pig iron.

(*b*) *Investigations on distribution of raw materials and velocity of upward gases in blast furnace*: Information regarding the distribution of raw materials on the blast furnace top, the velocity and distribution of upward gases, the nature of descent of stock and other allied information are often helpful for suggesting corrective measures leading to decreased frequency of trouble and therefore increased production. The following simple methods are sometimes adopted for this purpose:

(*i*) A rod conveniently calibrated to read directly in inches or cm is pushed in a known distance through a tube in the brickwork. A weight is then allowed to drop at these known distances and the corresponding heights are measured at different intervals of time. From these data the contour of the charge level may easily be plotted or directly shown.

(*ii*) the nature of the stock level may be obtained by a radioactive source R emitting γ-ray and a detector element D. A series of such R-D sets or a rotating R-D pair gives valuable infomation regarding the nature of the descent or otherwise of the stock. Hanging or slipping may be early detected by this method.

(*iii*) velocity of upward gases and their distribution may be measured by injecting radioactive gas samples at the tuyeres for a given time interval $\triangle t$ and noting the activities of different gas samples collected at known intervals of time (say every second) in a sample collector vessel. Plotting the activities of the various samples as function of time indicates the average time taken by the upward gases. Maldistribution of gas velocities leading to "channeling" and other troubles may be located and corrective measures adopted.

(*c*) *Erosion and accidental damages to shaft lining in the blast furnace*: These are sometimes detected by placing radioactive samples like radio-cobalt at different places in the brickwork and recording periodically the activities of these samples. Precautions, however, are necessary to make allowances in the recording for the diffusion of the radio-cobalt within the brickwork. Diffusion may be minimised by sealing the radioactive samples in suitable capsules.

(*d*) *Increased production of pig iron with control of sulphur and silicon*: It is well known that use of increasing amounts of suitably prepared sinter in the burden and pressurised top lead to considerable

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increase in the production of pig iron.

The advantages of low Si and low S iron for steel-making purposes are quite well known. Indian raw materials (high Al_2O_3 ore, limestone with lower available CaO, high ash coke, etc.) do not lend much flexibility in this connection. Nevertheless from the results of investigations carried out in large pilot plants and/or blast furnaces in other countries, it appears that there are definite possibilities of improving the quality and quantity of Indian pig iron by some of the following methods:

(*i*) gradual introduction of "all-sinter" burden,

(*ii*) use of low ash coal—better washing or blending,

(*iii*) suitable investigations on the treatment of high alumina ore leading to a low alumina-silica ratio,

(*iv*) investigations on the blending of increasing amounts of lime or calcined dolomite in the sinter,

(*v*) gradual enrichment of the blast,

(*vi*) introduction of small amounts of lime powder through the blast,

(*vii*) use of higher blast temperature,

(*viii*) standardisation of tuyere metal specification and use of special metal tip tuyeres to minimise frequency of leaky tuyeres,

(*ix*) conditioning of the blast at a constant but low humidity level. Though injection of steam often results in smoother working and greater efficiency of reduction, it definitely increases the thermal requirements in the hearth, and therefore demands either a lighter burden or a higher blast temperature. Under Indian conditions, the moisture content in the blast may preferably be kept constant at that low level which on the one hand yields some of the advantages of steam injection and yet helps not to increase unnecessarily the coke consumption. Investigations on this optimum moisture content of the blast under Indian conditions may yield encouraging results.

In short, under Indian conditions gradual introduction of all factors and environments that help to cut down the thermal requirements of the furnace would help to increase the production at a given coke rate. The dangers of thermal deficiency in the hearth are quite well known. The Blast Furnace Manager, naturally, therefore is indifferent and even suspicious of cutting down coke in the normal burden. Nevertheless, a well planned and systematic investigation and careful introduction of some of the new features—one or a complementary pair at a time, and in small magnitudes to start with—may definitely be helpful and lead to increased production.

Steel-making practice

(*a*) *Increased steel production*: Fundamentally speaking, the rate of production of steel depends essentially on the following two factors:

(*i*) increased rate of heat transfer to the bath, and

(*ii*) increased rate of oxygen input into the bath.

From both these points of view, the open hearth process is highly inefficient, although nearly 90% of the steel production today is done by this process. Those who are familiar with slag-metal kinetic problems, therefore, always support the recent trend of making steel by one or other of the pneumatic processes—(originally discovered nearly a hundred years ago by Sir Henry Bessemer), e.g., the converter process with or without oxygen enrichment and with or without lime injection—the well known L-D., the O.L.P. and similar processes.

The heat transfer from the gas to the slag phase and thence to the metal phase and the oxygen diffusion from the slag phase to the metal phase obey more or less similar quantitative relations, namely, the quantity gradient vector is proportional to the potential gradient vector where the activity gradient for the oxygen transfer or the thermal potential gradient for the heat transfer are the driving forces. This hypothesis is amply corroborated by many of the recent data collected on slag-metal equilibria, the heat transfer and oxygen transfer problems. Unless aided by the vigorous lime boil or other stirring effects, the heat transfer in open hearth is essentially non-convective. Similarly, the diffusion coefficient of oxygen being low the slag-metal oxygen transfer to the bath is also low. The variations in the tap to tap time from as low as 6 hrs. to as high as 14 hrs., apparently under similar condition, are explainable on the basis of the different rates of heat transfer and oxygen transfer which may be very slow, yet very fast. To be able to understand some of the anomalies in basic open hearth practices and to be able to correlate some of the data on a more scientific and rational basis, investigations have been directed more intensively on slag-metal control methods and collection of some of the fundamental properties of slag and metal.

(*b*) *Investigations on slag and metal control methods*: The importance of slag and metal control in steel-making practices cannot be overestimated. The effects of activity coefficients and temperature on Slag Thermodynamics, the effects of viscosity, temperature and composition on Slag-Kinetics with particular reference to S and P removal are well known. But inspite of the work by Chipman, Richardson and others, reliable data for many of the activity coefficients of elements in slag and metal and fundamental properties of slag and metal are still lacking.

While the Pancake and/or the fluidimeter methods of slag control are practised in some plants, the tendency in recent years has been, as it should be, towards a more rational and scientific approach on the following lines:

(*i*) Careful sampling methods both for slag and metal—particularly for samples used for the estimation of gas contents.

(*ii*) Quick analyses of elements and constituents of slag and metal by the Quantometer and/or X-ray Fluoroscopy.

(*iii*) Measurement of absolute viscosity of slag by measuring the torque on a Mo or Pt rod or bulb at different temperatures. It may be possible to get a correlation between the conventional fluidimeter data and the absolute data for routine plant control.

(*iv*) Quick measurement of liquidus points of slag either by the micropyrometer or the Thermocouple Tip method. These data are helpful to know the rates of change of viscosity or fluidity as function of temperature on superheating or cooling above or below the liquidus. Nucleation and growth of crystal in a slag mass as function of the rate of change of viscosity offer another fruitful field of fundamental and applied research.

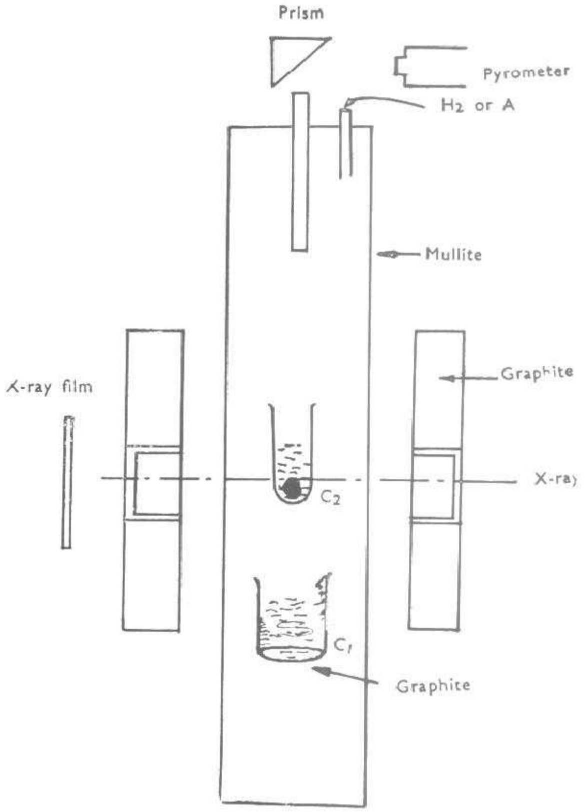
(*v*) Determination of activity coefficients of elements under a given set of conditions.

(*vi*) Determination of heat capacitance and conductivity of slags. These data are helpful for a scientific and quantitative approach to many slag-metal problems.

(*vii*) Determination of surface tension or rather the interfacial tension of liquid iron or steel by the liquid drop method. Although the detailed mechanism of transport of S″ and P″ ions through the slag-metal interface is not well known, there is no doubt that such transport processes are affected by diffusion and interfacial properties. For gas-metal interfacial tension measurement, the metal drop rests on an alumina disc and rod. For the measurement of interfacial tension between metal and slag, the slag is fused in a Pt or Mo crucible C, and the metal in an Al_2O_3 crucible C2. When both are molten crucible C2, with the side holes, is immersed into C1. After the slag flows into C2, it is raised up in position so that the slag-metal interface is in line with the X-ray beam and plate. After developing the X-ray image is magnified and a simple measurement of the dimensions of the drop gives the value of the constant B and the radius of curvature R and hence the surface tension r can be computed from the following relation :

$$Y=\frac{gdR^2}{B}$$ where d=density of metal, g=acceleration due to gravity.

For a given slag and metal and starting at some sulphur distribution ratio at equilibrium one may follow the changes in the shape of the liquid drop and hence its interfacial tension with progressive desulphurisation of the metal at a given temperature. It



Line diagram of apparatus for the determination of interfacial tension between metal and slag.

has been found for example that sulphur and oxygen are the two most surface active elements. It has also been found that sulphur in steel for a given slag environment decreases the interfacial tension of steel at a greater rate than oxygen.

(*c*) *Investigation on refractories* : It is not often realised how much the rate of iron and steel production depends on the proper quality of the refractory materials used in high temperature furnaces and regenerators. In basic open hearth, for example, the thermal potential of the flame is limited by the maximum temperature of the silica roof. This explains the modern tendency for the use of all basic open hearth furnaces. Similarly, the gas or air pre-heat temperatures are limited by the regenerator refractories. To be able to understand more quantitatively the surface and volume properties of refractories with regard to steel plant practices, the following few investigations are worth mentioning :

(*i*) Quick method of analysis of the composition of refractories by X-ray Fluoroscopy.

(*ii*) Studies on different phases in refractory materials at high temperatures by high temperature X-ray diffraction camera.

(*iii*) Precision thermal conductivity tests up to 1,400°C and standardisation of the results remembering that conductivity is not a linear function of temperature.

(*iv*) Standardisation of abrasive test for refractories particularly at high temperatures.

(*v*) Studies on the behaviour of thermal dialatation characteristics on heating or cooling at a given rate in a furnace with optical arrangements.

(*vi*) Sonic method of testing modulus of elasticity and spalling tendency.

Rolling mill practice

(*a*) *Continuous casting of steel*: Though originally conceived by Sir Henry Bessemer successful continuous casting of steel is of comparatively recent origin. The advantages of continuous castings are well known. For this country the most important advantage is that by eliminating the very costly blooming mill and its accessories, the ingot casting bays, the soaking pits, etc., continuous casting methods offer the possibility of obtaining higher yields and better quality with much lower capital cost and foreign exchange. In the future extension programme of the Indian steel mills, continuous casting should receive serious attention, particularly because several industrial plants are already working satisfactorily in Europe and the U.S.A. The few problems that still create trouble will very likely be solved within the next two or three years when our Third Five Year Plan and the expansion of steel programmes are expected to come into operation.

(*b*) *Automation-control and testing*: Apart from the trend towards more and more automation in the mill area, more and more of X-ray and γ-ray techniques are being used for fast and efficient routine control and testing of gauges and defects. Small amounts of radio P and S are added before teeming or casting and the rolled material is then subjected to auto-radiography for segregation. Radio Co source is used to detect more quickly and correctly piping defects and pipy portions of blooms or billets are quickly and correctly sheared off by the γ-ray detector mechanism.

Analytical methods

As a tool of fast quantitative analysis, the spectrograph has been and is still being used almost universally in steel plant central laboratories. For plate variable characteristics, want of sufficient speed and other inherent difficulties, the spectrograph could not be used in the steel plant stage laboratories. In recent years with the aid of photomultiplier tube, Geiger counters, etc. analytical speeds and reproducibility factors have been enormously improved and instruments under the name of Quantometer, Quantovacs Grating Polychrometer etc. are increasingly coming into use in steel plant stage laboratories. For the analysis of all the common elements in steel one needs a vacuum instrument particularly for C, S, and P. Recently however attempts have been made to analyse some of these elements without the vacuum instrument. For example the C++ Sparkline 2296A have been succesfully used for samples from 0·02 to 0·3% C. As this happens to be rather weak in intensity special photometer tubes have to be used. Moreover as the second ionisation potential of C is comparatively very high, one has to use special excitation conditions (22,000 volts) with a very small spark gap (not exceeding 1 mm). Similarly, the 2149 P line in the second order has been utilised for the analysis of P without the vacuum instrument. It appears that some very useful exploratory work could be done in this field to extend the range of analysis of C and P without the vacuum instrument.

Various other physical methods are being increasingly used these days for rapid chemical as well as structural analysis. The polarograph, the X-ray Fluoroscope and similar other devices are well known. But most of these methods yield only integrated or average result of the sample under consideration. Determination of the variation of composition within a small zone of a sample, fast analysis of microconstituents or phases of information regarding the composition at or near grain-boundaries or interphases or segregates have so far been very difficult to determine with any degree of precision. The electron microprobe analyser offers a very quick and efficient tool for high precision of the composition and structure of metals and alloys. The basic principles are extremely simple. A finely focussed beam of electrons called electroprobe about one micron in diameter is projected on a very small volume of the sample which emits white as well as characteristics X-radiation. The characteristics radiations are analysed with curve crystal Geiger counter vacuum spectograph. An optical microscope with a reflecting objective helps to observe the surface of the sample in the region to be analysed. The instrument consists of four main parts:

(*i*) The electronprobe generator and electron optic system

(*ii*) The Optical Microscope

(*iii*) The sample holder and shift mechanisms

(*iv*) The X-ray Spectrograph

The accelerating potential (10 to 35 kV) is taken on the minimum side of the excitation potential, to keep the volume of the analysed zone a minimum.

A sample holder carries the sample under investigation and a reference sample carrier containing 40 or more samples of pure elements which serve as standards for quantitative analysis. Any one of these pure elements can be quickly substituted under the probe in place of the sample. The ratio of the intensity I of the element x in the sample to the ratio I_x of the metal gives directly a quantitative estimation of the element in question in the region of the probe. The comparison of these ratios for the same wave length makes the method absolute and this is a very great advantage. Because preparation of a series of standard alloys which will be homogeneous on the small scale are completely avoided. Correction for self-absorption can be easily made from graphs or equations and the precision is about 1%. The only disadvantage is that it is difficult to extend the limit of analysis beyond atomic No. 12 (Mg). In ferrous samples therefore the analysis of such element as C, N, O, etc. are excluded, Nevertheless, the instrument has well demonstrated its versatile uses for studies of segregation, diffusion, identification and analysis of inclusions, carbides and other phases and finally the microanalysis of minerals. The work so far carried out at IRSID, France shows that it is possible to carry out local analysis of non-conductors, such as ores, as well as metals for alloys of one of more phases and further to measure all the variations of compositions within one phase such as a segregate.

The Microanalyser has also been used to find the composition of non-metallic inclusion, of sulphates, carbides and oxides formed by internal or surface oxidation. By analysing the surrounding matrix the distribution coefficient of various elements between precipitates and matrix is automatically obtained. If the precipitates have been obtained by prolonged annealing at a fixed temperature it is possible to establish even equilibrium diagram. Moreover, due to the high speed of analysis this instrument can produce a volume of work in a matter of hours which a group of experienced chemists might take weeks or even months to do.

DISCUSSIONS

Mr. B. L. Sen, N.M.L.: I want to seek some clarifications from Dr. Chatterjee regarding oxygen enrichment during sintering. I had the opportunity of working with the field trial team of the British Iron and Steel Research Association, Iron Making Division and had the opportunity of observing the experiments described in the paper. These were actually the observations of preliminary trials there, and it was subsequently seen that these curves obtained with oxygen enrichment did not truly represent the reaction zone as is normally available in sintering processes but likely to vary greatly according to the depth of bed and grain size of the raw materials. The effect of oxygen enrichment and the final curve obtained can be drawn, and explained only after taking into account the variables, namely, depth of sinter bed, grain sizes of the raw material permeability, etc. I would appreciate Dr. Chatterjee's further comments on this point.

Regarding the radio-active tracer element, advantages of which have been theoretically established, it was found that its use involved excessive man power, besides a number of practical difficulties. Except the well known Pulse method I am not aware of any further development in this direction to ease out the complication of handling the equipment and in obtaining any conclusive data, but I feel that industrial organisations are not very serious about it mainly due to the considerable labour, precision work and cost of the experimental equipment involved in it.

Dr. G. P. Chatterjee (Author): Referring to the question of oxygen enrichment in the blast furnace, I wanted to emphasise the fact that in India we should explore the advantages due to oxygen enrichment alone without having recourse to humidification of the blast. In other countries, there is a tendency to humidify the blast along with oxygen enrichment, to control the temperature and also to get the benefit of H_2 reduction. In this country however it is not necessary for this simultaneous humidification to take effect along with oxygen enrichment. If oxygen enrichment is found to create difficulties such as high silica iron, leaky tuyeres, etc. then certainly steps other than humidification should be taken. Humidification under Indian conditions should be done with great caution as it takes away a large share of heat by the endothermic reaction. It is necessary that we explore all means of cutting down the thermal requirements of the furnace and thereby cut down the coke consumption and also help better sulphur and silica control. I agree that the question is still open and even though some of the advantages of humidification are apparently too obvious, we shall have to investigate and establish the optimum levels of oxygen enrichment and humidification with regard to our local conditions.

With regard to Mr. Sen's question on oxygen enrichment during sintering, it may be stated that I have not discussed the depth of bed of sinter and grain size of fuel. The plot I have represented is the result of some of the most recent observations not only by Dr. Voice but also by others with regard to oxygen enrichment. Injection of oxygen instead of raising the maximum temperature of the bed (Tmax) and narrowing the zone of combustion, lowers Tmax and widens the zone. This is because the surface activity of coke increases with oxygen pressure and hence the ignition temperature is lowered with the result that combustion starts before the charge is pre-heated to a higher temperature. This leads to a widening of the combustion zone and a lowering of Tmax.

Referring to the point raised by Mr. Sen on the radio-active tracer element, I may indicate that it is not proper to say that the advantages of the radio-active technique have been only theoretically established. It is also not right to state that radio-active methods of analysis need more man power. Compared to a routine chemical laboratory and other conventional methods of analysis, the personnel requirements for physical methods of analysis (including radio-active method) is comparatively much less. The radio-active technique is, no doubt, a comparatively new one, but potentially, it is a very powerful technique to get valuable information of operating data from almost inaccessible regions of furnaces or equipment and as such, it is being used in many modern iron and steel plants as well as in other industries. I have therefore attempted to stress that this country should form a strong nucleus for radio-active methods of analysis including tracer technique.