PILOT PLANTS FOR METALLURGICAL RESEARCH AT FRIED. KRUPP, ESSEN(*)

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The erection and operation of semi-commercial pilot plants for performing metallurgical tests and research has proved to be a valuable proposition in many industrial countries and will become more and more important in the future. By adopting this procedure, it will be possible for the reseearch engineer to obtain reliable information on metallurgical reactions and other phenomena with a relatively low expenditure of time and money. The results obtained in these pilot plants can be applied to large scale industrial plants, either direct by using the principle of similarity or indirect by interpreting the test results on the basis of empirical figures.

The following deals with examples of the pilot plants used by the Research Centre of FRIED, KRUPP, Essen, for experimental work on iron ore reduction outside the blast furnace and on oxygen steel-making using the LD- process.

The economic production of pig iron in the blast furnace requires the more important constituents of the burden, namely ore and coke, to have definite chemical and physical properties. For the iron-bearing materials such as ore, pellets or sinter, the critical factors are reducibility and resistance to softening under pressure. The fuel, which is coke in most cases, must possess a high resistance to abrasion in addition to good reactivity.

The basic conditions for successful blast furnace operation can no longer be fulfilled in many countries, so that a new approach must be made to the production of pig iron. In Germany, for instance, the necessity of using low-grade, high silica ores for the production of iron led to the introduction of the KRUPP- Renn process which was developed some 30 years ago. In this process, small-size ore is reduced in a rotary kiln fired with oil or pulverized coal to reach a temperature of 1250 to 1300°C in the final zone, the charge and heating gases passing through the kiln in opposite directions. In the final zone, the reduced iron particles weld together to form luppen which remain dispersed in the slow-flowing slag and are

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discharged with it. This process has proved successful in commercial application and has gained a prominent position among existing direct-reduction processes.

After World War II, there was a strong trend towards the development of new reduction processes, but in all of these processes the following requirements had to be taken into consideration:

1) Treatment of rich ore fines, ranging from grit to dust particle size.

2) Use of petroleum or natural gas or one of their by-products as reducing agent

3) Low first costs for large- scale industrial plants.

Messrs. FRIED. KRUPP of Essen resumed their research work on rotary kiln processes which had been interrupted during the war, and several years ago developed the KRUPP sponge iron process which, in contrast to the KRUPP- Renn process, is designed for the treatment of rich ore fines.

After the metallurgical research centre had been reconstructed, FRIED. KRUPP devoted their attention to obtaining closer information on the rotary kiln processes. Their object was, on the one hand, to widen the basic knowledge in the field of metallurgy and, on the other, to determine the potential applications of the rotary kiln processes. In addition, it was intended to examine the amenability of new, unknown ores to these processes by conducting suitable tests with small quantities of ore.

A start was made with the development of small-size rotary kilns for experimental work in the laboratory in which reduction tests of different kinds can be made under conditions similar to those existing in kilns of commercial size. The rotary ceramic tube, which is placed into an electrically heated furnace, can be charged with about 2000 or 3000 grams of ore plus the necessary amount of solid fuel. The operating conditions such as revolving speed, temperature, gas composition, flow rate of gas through the reduction zone, and the number of material samples can be varied within wide limits. The gases (CO and CO_2) evolved during reduction are continuously analysed. The tests are generally conducted in such a way that the feed in the tube is heated to the desired temperature, whereupon the tests proceed under isothermal conditions. The reactions taking place in the tube feed, especially the removal of oxygen from the ore to form metallic iron, are checked by making a chemical analysis of each of the samples that are continuously taken from the tube. Information is also

desirable on the changes of a more physical character the ore undergoes in the tube, and metallographic sections are used to demonstrate these changes.

The work carried out so far was directed not only to investigating the metallurgical reactions, but also to evaluating the metallurgical properties of ore and fuel. New testing methods had to be developed, since the methods of ore evaluation known so far were unsuitable for small-size and fine ores and, moreover, provided little or no information on the nature of the alternate reactions between ore, solid fuel, and kiln atmosphere.

To give an example of what information is obtainable from tests in a laboratory kiln, the reduction curves of various types of ores treated under the same conditions have been studied and it may be said that the graph clearly indicated the different characteristics of reducibility that could be determined by this testing method, information which is of practical value for starting tests in a semi-commercial pilot plant.

Since the laboratory kilns as described above lend themselves only to batch operation, it was found necessary to erect a pilot plant designed to furnish results comparable to those of a commercial plant. The charge is continuously fed into a 6.5 m long rotary kiln by means of a screw feeder, while the kiln product, after having been cooled to a large degree, is discharged into cars shielded by inert gas. For heating the kiln, a multi-fuel burner suitable for gas, pulverized coal and gas is used which permits the flow rate of fuel to be varied within wide limits to ensure good temperature control. The temperature within the free kiln space and inside the charge is continuously measured at 6 points by thermocouples and the measured values transmitted via electrical contact rings to the temperature recorders. Gas and charge samples are also continuously taken from inside the kiln without interrupting the process. Revolving speed, kiln inclination, temperature range, and kiln atmosphere can be varied to suit the desired conditions under which the process is desired to take place. The 6.5 m long rotary kiln is used for running smelting tests in cases where the amount of ore available for treatment is limited, and for studying basic metallurgical problems presented by the use of any type of ore or fuel.

The results obtained in this pilot plant are used as a basis for making further tests in a semi-commercial plant with the object of improving both the KRUPP- Renn process and the KRUPP sponge iron process. The semi-commercial plant comprises a 14 m long kiln with an inside shell diameter of 1.2 m which can be run at various speeds and different angles of inclination. The fuel burning equipment is so designed that pulverized coal, oil, coke-oven gas and blast furnace gas or any mixture of them can be used for firing the kiln, the various fuels being fed in measured quantities. In

addition, the kiln is provided with adjustable nozzles for injecting combustion air into the kiln space as well as with devices for taking gas and material samples. A large number of instruments is available for measuring temperature variations, and pressure or draught in the kiln as well as the composition of the waste gas are continuously checked. The discharge end of the kiln permits several methods to be employed for discharging the product from the kiln.

In addition to the uses described above, the rotary kiln is employed for conduction sintering and roasting tests and for studying details of volatilizing and clinkering.

The pilot plant which centers around the rotary kiln is complete with all necessary equipment for preparing the kiln feed (crushing, screening, coal pulverizing and blending, pelletizing) and also includes a variety of machines required for treating the kiln product by selective crushing, grinding, screening, sifting and magnetic separation.

The pilot plant is fitted out with the same equipment as commercial plant employing the various processes described above, only on a smaller scale.

The tests are carried are carried out in continuous operation and generally last 2 to 6 weeks. The results form the basis for the planning and construction of corresponding commercial plant. A comparison of the results obtained from a 14 m long kiln that has been operating for more than 5 years with those from full-scale plants has demonstrated that there is practically no difference in the quality of the kiln product.

In order to conduct metallurgical tests using oxygen steelmaking processes, particularly the LD-process, it was found necessary to establish a pilot plant. The purpose of this plant was not only to make fundamental investigations into the reactions taking place in the process, but also to furnish results which as far as possible could be applied to operations on a commercial scale. A converter of 2.5 to 3 tons capacity was considered adequate to meet these requirements. This converter size already ensures a favourable heat balance and also permits the shape of the converter to conform in most respects to that of those used in full scale production, consideration being given to the principles of similarity.

The complete pilot plant consists of a rotary melting furnace with oil-oxygen burners for providing the hot-metal to be blown, and the converter stand with the necessary auxiliary installations.

The advantage of a separate melting furnace for the hot-metal can be varied within wide limits. The drawback is the unavoidable waits between melts and the consequent loss of converter heat which must be made up by installing a device for keeping the converter hot. A portable gas burner has proved to answer the purpose and can also be used for preheating the converter after stoppages.

In the pilot converter, the ratio of bath depth to diameter has been taken 1:3 which is the customary ratio used for production converters. The distance of the nozzle to the surface of the bath can also be varied during the blow and can be adjusted to meet the desired test conditions. The head of the oxygen lance is provided with interchangeable nozzles which only take a few minutes to change. The converter is lined with a tar-dolomite ramming mixture which has proved satisfactory in intermittent operation with frequent complete cooling of the converter, and has a life equivalent to 60 - 80 heats.

Sampling can be done at any time without interrupting the blow. For this purpose an immersion mould welded to a rod, is lowered into the bath. The moulds are closed with wooden lids to prevent the ingress of slag. The samples are usually killed with aluminium previously placed in the mould. The slag adhering to the outer wall of the mould is taken for slag-testing. By this means, only the fluid, reactive slag is taken, a matter of great importance for metallurgical evaluation.

Special attention was devoted to measurement of temperature, it being highly desirable to find some means of measuring the temperature throughout the whole refining period of the heat and of recording it automatically. This problem was solved by incorporating thermocouples into the converter walls. The "Heratherm protective Tube", which is obtainable on the market, has been found to give a satisfactory service life. As long as the temperature does not arise beyond 1700°C they can remain in contact with hot metal or the steel bath for up to 50 minutes and are only slowly attacked even by slag containing fairly large amounts of FeO. The thermocouple protecting tubes are renewed after every heat.

Removing an old tube and replacing it with a new one by means of simple tool is a matter of few minutes only. The tube as it appears in the converter after a heat has been blown projects about 40 mm into the interior of the converter.

The temperature of the bath is registered continuously by a stripchart recorder at intervals of 1 second. The time lag in recording despite the relatively thickwalled tube, is so slight that even small fluctuations, such as take place when scrap or fluxes are added, can be picked up.

The operation of the 3 ton pilot converter and the possibility of reproducing the results were checked in a series of tests. Results obtained from three heats which were blown under the same working conditions, with only very slight differences in the charge and initial temperature have been studied. From this study it is apparent that the results are reproducible. In all three tests a hot metal of the usual composition with about 4% C, 1.7% Mn, 0.7% Si, 0.1% P, and 0.04% S was blown. The process corresponded to that employed commercially, the steel bath being cooled by additions of ore and crushed limestone.

The quantities given by analysis show very slight differences which hardly exceed the limit of error of chemical analyses. The differences in the final analysis are proportional to the differences in the composition of the hot metal.

The good reproducibility of the test results is by the study of temperature curves of the three heats which also clearly indicate the influence of the additions upon the temperature of the bath.

The refining and temperature curves confirm the suitability of the 3-ton pilot converter for accurate metallurgical investigations.

The object of the investigations carried out since the pilot plant came into operation was to study the reactions that take place in the processes employed in commercial plant and to develop processes permitting the use of pig iron charges of abnormal composition, as well as the use of KRUPP- Renn-luppen and products from other ore-reduction processes.

Reports on the progress of this research work are currently published in the periodical "Technische Mitteilungen KRUPP".

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