

Pelletizing

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INTRODUCTION

DURING the last 15 or 20 years we have witnessed an extraordinary increase in the quantity of artificial agglomerates used in blast-furnace burdens. This expansion has occurred in two fields, the first being sintering, a technique applied particularly to the agglomeration of low grade 'earthy' ores and to screenings from high quality lump.

If ores are to be sintered successfully it is essential that a bed of sinter mix be prepared through which air can be drawn at an adequate rate without a prohibitive pressure drop. It follows that research workers, designers, and machine operators are continuously occupied with schemes for increasing bed permeability and maintaining it constant at a suitable level; the problem is clearly most acute with very fine ores. Examples of difficult ores are Norwegian magnetite concentrates from Sydvaranger, as used at the Workington (UK) sinter plant of The United Steel Companies, or the naturally fine specular hematites from Yampi Sound (Western Australia) used by the Broken Hill Proprietary Company at its sinter plants on the east coast of Australia.

The sintering problems which such materials present are by no means insuperable but the attendant difficulties, e.g. high fuel consumption and low specific outputs, have given added impetus to the development of another technique which forms the main subject of this paper: pelletizing.

HISTORICAL DEVELOPMENT OF PELLETIZING

The principal nursery of this technique and the source from which the flow of successful modern development has sprung is the Mines Experiment Station of the University of Minnesota, USA.

During the 1940s research workers at this station under the direction of Dr Davis examined the problems of utilizing the low-grade iron ores of Minnesota. Particular attention was given to the ferruginous rocks adjacent to the main ore bodies of the Mesabi range.¹ These low-grade ores (25–30% Fe) are quite exceptionally hard and abrasive and are known locally as 'taconites'; they are the original material from which high grade Mesabi hematites have been evolved by natural leaching and oxidation. The recoverable iron mineral is finely disseminated magnetite and the ore must be ground to about 80% —325 mesh for liberation, the concentrate containing about 65% iron and 8% silica.

By 1945 research and development at the Station had made considerable progress; not only had a promising concentration technique been evolved but novel ways had been investigated of using the very fine concentrate produced. The wet concentrate was 'balled' in a rotating drum² and then hardened by suitable heat treatment in a shaft kiln. The hard pellets (about $\frac{1}{2}$ –1 in dia.) were thought to be a suitable blast-furnace feed material and subsequent tests in a small experimental blast-furnace were encouraging. These results attracted the attention of iron makers all over the world and inspired some particularly energetic and successful work in Sweden.^{3,4,37}

In the USA the steel and ore companies saw in this

SYNOPSIS

An attempt is made to present a logical account of iron-ore pelletizing operations (balling and hardening) with particular reference to laboratory techniques. The factors leading to the growth of the industry are dealt with historically, concluding with an examination of pellets as a blast-furnace feed. A section on the physics of balling includes an account of laboratory techniques for the production and testing of green balls. The firing of green balls is dealt with both theoretically (in terms of heat transfer with chemical reaction in a packed bed) and experimentally, followed by a discussion of test requirements and methods. The paper concludes with a short discussion of the utility of test-work in relation to practical plant design and a brief survey of the relative position of sintering and pelletizing in the world today.

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technique a means of prolonging the rich but dwindling ore resources of Minnesota and Michigan by making available hitherto unusable material. New companies were formed to explore these possibilities, and research was intensified. By 1949 it was generally agreed in the USA (and Sweden) that the best way to prepare balls from a concentrate was in a rotating drum, but opinion was divided on the heat treatment method.

It was essential that the apparatus used, whatever its form, should:

- (i) give close temperature control
- (ii) require the minimum amount of fuel, i.e. should recuperate sensible heat
- (iii) be reasonably trouble-free and reliable in operation
- (iv) have an adequate unit output.

At first vertical shaft kilns were used exclusively in pilot-plant research, moist balls of concentrate being fed into the top and moving downwards against an ascending flow of hot gases which first dried them, and then elevated their temperature to the hardening region. Hardened pellets were withdrawn from the bottom of the kiln. Early work on shaft kilns has been discussed by Haley and others.^{5,6,3}

Although the process is intrinsically simple, and therefore attractive, it proved in practice to have some awkward features. Amongst the difficulties which were most acute in the early 1950s may be mentioned:

- (i) the difficulty of securing even gas distribution
- (ii) the difficulty of securing even stock descent, uniform pellet treatment and trouble free discharge of product
- (iii) the difficulty of securing a high output from one unit
- (vi) uncertainty about 'scaling-up' and the most appropriate kiln shape.

These problems caused the Reserve Mining Company in the USA to seek another way forward.⁵ The new line of advance had its genesis in the 'Lepol kiln process', used in the cement industry. This process, which was developed in Europe, consists of a balling unit feeding a moving grate on which the balls are dried and partly hardened. The grate discharges into a rotating kiln where the burning process is finished; the hot gases from the kiln are ducted back to the grate where they perform the drying and hardening functions just mentioned.

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TABLE I Pelletizing production data (approximately correct to mid 1962)

Company	Location	Process	Capacity, long tons/a
Reserve Mining	Minnesota	Grate	9 000 000
Columbia Geneva	Wyoming	Grate	1 500 000
M. A. Hanna	Canada	Grate	5 000 000
"	Michigan	Grate	1 250 000
Cleveland Cliffs	Michigan	Grate	660 000
Int Nickel	Canada	Grate	300 000
Cleveland Cliffs	Michigan	Grate-kiln	1 800 000
Erie Mining	Minnesota	Vertical Shaft	1 500 000
Bethlehem Steel	Canada and USA	Vertical Shaft	3 330 000
			30 340 000

Mitchell⁷ has reported that Dr Leliep of the Allis-Chalmers Company suggested that the balling drum and grate might well be used for producing burned pellets from fine magnetite concentrates. If such an arrangement could be designed to dry and fire the pellets and to recuperate the sensible heat of the product, it might well solve the problems of fine magnetite agglomeration relatively cheaply.

Research was continued in the Allis-Chalmers laboratories with encouraging results, and in 1954 the Reserve Mining Company commissioned a 1000 tons/d experimental machine based on these principles, but designed mechanically on sinter machine lines by the Arthur G. McKee Company. The pellets were dried by blowing hot air upwards through the bed, and then hardened by drawing hot gases downwards from special furnaces. Arrangements were made to cool these pellets on the strand and the hot air recovered was used to dry the wet balls at the feed end, thus reducing fuel consumption. Useful operating experience was obtained and after a few months trial the Reserve management placed orders (April 1954) for the design of six large machines to be installed at Silver Bay, on the western shores of Lake Superior. Haley³⁶ has described these early developments.

Other companies, however, persisted with the development of the vertical kiln process, again for magnetite concentrates, and their progress also justified the erection of some large installations. The Erie Mining Company plant at Hoyt Lakes is the largest of its kind; it was commissioned in 1957 and includes 24 vertical shaft furnaces.⁸

In the 1950s, therefore, both vertical shaft kilns and moving grate machines were being developed and applied to the pelletizing of fine magnetite concentrates. Magnetite concentrates are relatively easy to pelletize: the particles are granular (rather than plate-like) in shape, with a high surface area, and with a surface uncontaminated by flotation agents; if properly treated they will oxidize during firing, giving a useful heat release. Oxidation is also associated with grain growth and recrystallization which contribute to the development of the requisite final strength. However, not all ferrous concentrates are magnetites; thus in the state of Michigan there are large deposits of jaspilite in which the iron mineral is a finely divided specular hematite. These ores have been called the Michigan counterpart of the Minnesota taconites, but the iron mineral is recoverable by flotation and gravity methods, rather than by magnetic concentration.

Such hematite concentrates present special problems and their exploitation has been a major concern of the Cleveland Cliffs Iron Company. In 1956 they began operation at Eagle Mills (Michigan) with a grate machine,⁸ but since then two very successful larger plants have

TABLE II Effect of burden and wind rate blast on Armco 28 ft furnace, Middletown, USA

Period: August 1954	June 1961	Notes
Burden lb/nthm		
Ore	2 594	
Pellets (Reserve)	649	3 080
Stone and/etc.	676	440
Wind, ft ³ /min	83 000	123 000
Hot metal make, tons/d	1 462	2 942
Coke rate, lb/nthm	1 623	1 304

nthm = net ton hot metal

been built using the *grate-kiln* system, this being, in fact, the Lepol process as a whole applied directly to the iron ore problem.⁹ The system has been developed by Allis-Chalmers and the plants themselves have been engineered and constructed by the McKee Company. In general it may be said that the Lepol process (drying and preheating on a grate and hardening in a kiln) has proved very successful with hematite concentrates (which do not have the advantage of an exothermic heat of oxidation) whilst for magnetites the 'straight grate' or vertical shafts are the usual tools.

PRESENT SITUATION

The efforts made during the last decade have been successful, and a major new industry has been established and is still rapidly expanding. An appreciation of the magnitude of this industry in the USA and Canada will be obtained from the production data given in Table I

EFFECTS OF PELLETS ON BLAST-FURNACE OPERATION

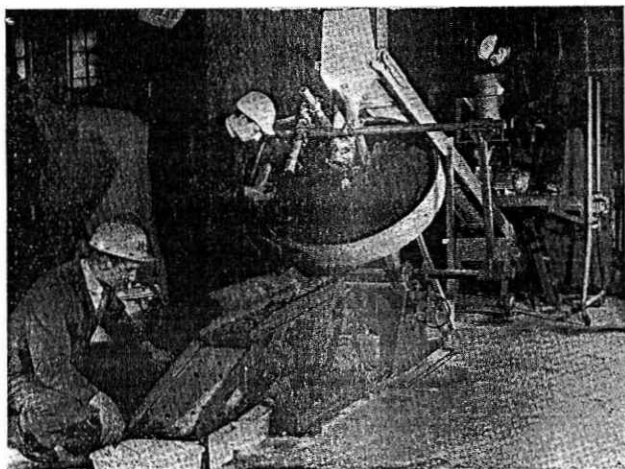
It should be noted that the expansion of pelletizing capacity has been accelerated by the rapidly accumulating evidence that pellets are an excellent furnace feed. Olt¹⁰ has pointed out that pellet properties are almost ideal, since they are:

- (i) uniform in composition and size
- (ii) highly oxidized
- (iii) high in bulk density
- (iv) capable of being evenly distributed across a blast-furnace stack, thus reducing channelling.

The effect of using pellets, relative to prepared lump ore burden, can be quite dramatic, as instanced in the now classical data¹⁰ given in Table II.

It will be seen from Table II that the use of pellets enabled the Middletown furnace to be blown at a much higher rate, and yet with much reduced coke rates.

This furnace still holds the US record for sustained production; by comparison the best results with self-fluxing sinter are 2211 nthm/d, with a coke rate of 1200 lb/nthm (Gary no. 7, 28 ft hearth). Lander and Banks¹¹ have compared Middletown and Gary practice by using Flint's coefficients, and infer that the lower coke rate (with Gary sinter) can be explained in terms of coke chemistry, blast temperature and moisture, slag volume, and hot metal silicon content. They conclude that pellets are still the best available burden, and that the development of self-fluxing pellets would further improve the situation. On the other hand the production of a stabilized self-fluxing sinter with a controlled particle size distribution and structure is still worth attempting. They go on to make a different comparison: between 100% pellets (Erie taconite concentrates) and the normal Indiana Harbor burden of 50% ore and 50% Erie pellets. The 100% pellet burden enabled the furnace to be blown at higher rates (production increased from 2311 to 2777 nthm/day) with an increased coke rate (from 1160 to 1221 lb/nthm, which they do not believe to be significant as the increase was



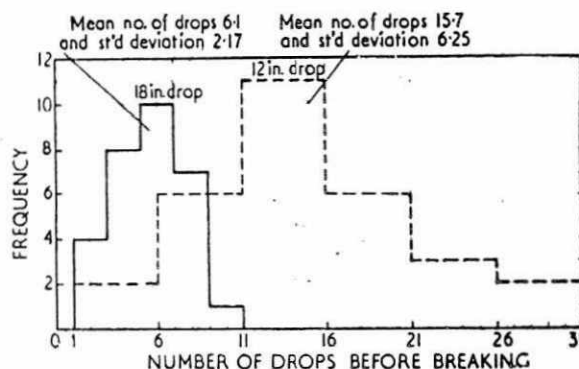
1 Head Wrightson laboratory balling disk

maintained after the trial period). The highest daily production rate, with 100% pellets was 3095 nthm. Reference should also be made to the work of Liggett and Killian,^{12,13} particularly for data on furnaces using moderate proportions of pellets.

A less satisfactory comparison has been made by Heynert *et al.*,¹⁴ on the Phoenix-Rheinrohr no. 7 furnace with 50% ore plus 50% pellets as against 50% ore plus 50% sinter. They found no difference in blast-furnace productivity, and a slightly higher coke rate with pellets. However, the pellets used were not ideal: Malmberget shaft-furnace pellets (about 20mm dia.) intended for use in Bessemer and LD converters. What is more, the burden used during the pellet trials contained about 21% of highly irreducible Wabana ore, compared with less than 6% during the normal period. Linder has calculated that this factor alone is sufficient to explain the relatively poor results with pellets. At the same time a 50% ore/50% pellet charge is almost certainly not ideal from the point of view of gas flow.

A comprehensive series of trials comparing various pellets and sinters has been run on the US Bureau of Mines Experimental Blast Furnace (Mahan *et al.*¹⁵). With so small a furnace it is difficult to know what faith to put in the results; however, in other trials, comparisons with large furnaces have been encouraging. The results may be summarized as follows: 100% pellets are superior to 100% fluxed sinter, both with respect to driving rate and coke rate; 100% grate-kiln pellets appear to be slightly superior to 100% updraught grate (experimental) pellets. It should be noted that the latter comparison between pellets is a little doubtful in that the slag volume was distinctly lower with the grate-kiln product (517 lb/ton compared with 605 lb/ton for the updraught grate product). It may be noted also that the grate-kiln pellets (89.8%Fe₂O₃, 1.0%FeO) used were rather more oxidized than the straight-grate product (80.9%Fe₂O₃, 9.6%FeO), and much more so than the fluxed sinter (60.8%Fe₂O₃, 23.9%FeO). Mahan concludes that pellets perform significantly better than comparable sinters.

Poos and Linder¹⁶ have recently considered the general problem of reducibility of burden components in relation to furnace performance. Their data indicate generally high reducibilities for such pellets as were tested (although the reducibility of sinters seems to increase more rapidly at small particle sizes (~1cm)). It is, however, interesting to note that, where reducibility could be compared with actual furnace operation, the highest percentage of indirect reduction (r_{co} %) observed was obtained with 100% pellet burdens.



2 Green ball drop test, ground natural specular hematite

Klemantaski and Wild¹⁷ have examined the behaviour of 100% pellet burdens by the SCICE technique, and have found good general agreement with operating results from blast-furnaces, and conclude that segregation and burden breakdown (which do not occur with SCICE) 'cannot exert a major influence on coke rate in furnace operation under these conditions'.

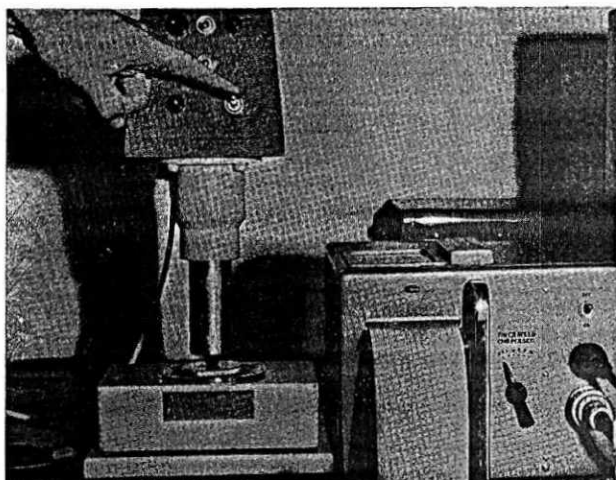
We may, then, conclude that pellets provide a singularly good blast-furnace burden, particularly when used 100%. They provide the obvious method of agglomeration of very fine concentrates or very fine naturally-occurring materials (e.g. possibly 'Indian blue dust'). On the other hand the days of sinter are by no means numbered. In the UK¹⁸ the production of closely-sized sinter by re-sintering sinter fines has given large increases in output and has been proved economic (at least in a particular case). In the USSR furnaces are driven at exceedingly high rates with 100% sinter burdens of poor quality (and coke of excellent quality) by using high top pressures, and by continual adjustment of the charging sequence as indicated by gas analyses and temperature measurement. In Japan exceedingly low coke rates result from high iron burdens and very careful size preparation of both the ore and sinter charged. There is clearly no universal road to successful burden preparation or blast-furnace operation.

However, it is not surprising that blast-furnace operators in Europe have imported experimental shiploads of pellets and that a worldwide interest has developed. The authors' company is actively involved in these matters, and we have extended our ore preparation laboratory appropriately, to enable pelletizing studies to be made of ferruginous materials (and indeed of all other minerals to which the technique is applicable). These laboratory studies complement the hard-won process and engineering 'know-how' of our US associates (McKee).

From this sketch of the origin and development of pelletizing processes we now consider fundamentals and laboratory techniques. We shall use the terminology of Goldstick,¹⁹ in which 'pelletizing' is taken to mean the entire process of making a hard pellet by first forming a 'ball' and then firing it; 'balling' is the first step in this process, the formation of an unfired spherical agglomerate, in this case a 'green pellet' or 'ball', by rolling moist powder in a drum or on a disk, or similar device.

PRINCIPLES OF BALLING

The technique of balling a fine iron ore by moistening and rolling is not new. The experiments on the production of heat-hardened pellets by Andersson² in Sweden in the early years of the century, foreshadowed much of the later work. However, as stated above, most modern work in the iron ore field stems from the Mines Experimental Station in Minnesota.



3 Head Wrightson ball compression strength tester

The balling stage is of crucial importance to the success of the pelletizing operation as a whole because the firing equipment, whatever its nature, requires a steady supply, at rates of 50–100 tons/h or more, of green pellets with closely controlled diameters and properties. Valuable discussions on the technology of balling equipment have been given, e.g. by Haley and Apulji³⁶; Haley⁵; and Greaves and English.²⁰ Newitt and Conway-Jones²¹ and Bhrany *et al.*²² have studied the kinetics of balling in drums and on disks.

Balls (i.e. green pellets) must be strong enough to withstand the mechanical shocks of transport and loading into the firing apparatus without fracture or excessive deformation. In addition they must be able to survive the stresses imposed by the subsequent heat treatment (i.e. drying and the internal structural changes brought about by firing). Experience shows that the properties of balls are largely governed by:

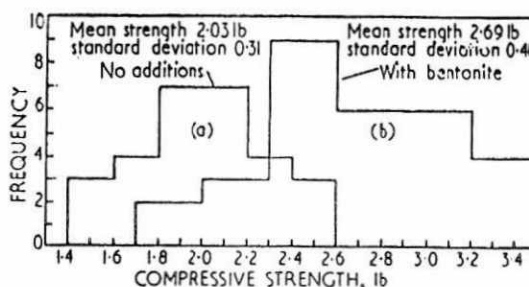
- (i) 'finesness' of the ore
- (ii) moisture content of the balls
- (iii) additives used (if any)
- (iv) shape of the particles
- (v) surface properties of the particles, e.g. the presence or absence of hydrophobic films.

All these factors must be reviewed in the course of laboratory investigation.

Particle size and surface area

In the literature there has been rather undue emphasis on the relationship between *mesh size* and ease of balling. Thus Davis and Wade have stated that a typical magnetic taconite ore must be ground 100% 65 mesh and 60% –325 mesh (Tyler) for successful balling. However, in practice the concentration requirements for this ore demand that 80% of the concentrate should pass 325 mesh; this last figure is widely and erroneously quoted as being the degree of fineness necessary for balling *any* iron ore.

A much more useful figure to quote for such a typical magnetic taconite concentrate would be the surface area (about 1 700 cm²/g) as measured by an air permeability method (e.g. the Blaine apparatus²³). We have found this a useful criterion and a natural specular hematite and a natural limonite will both pelletize readily when ground to this degree. It will be shown later that a surface area term enters naturally and directly into theoretical equations for ball strength. Size analyses are also made on these materials and for determination in the sub-sieve range (–325 mesh to 80 mesh) we have found the Bahco centrifugal classifier most useful (at least as reliable



4 Green ball compression test (Swedish natural magnetite)

as, and quicker than, the Haultain Infrasiser). Magnetite particles may need demagnetizing to prevent magnetic flocculation during sizing operations. The sub-sieve range of sizing is the most important, as these fractions make a major contribution to the total surface area. 'Slime-free' but otherwise substantially fine materials may not ball at all; hence the grinding operations must be chosen with regard both to release analysis and pelletizing requirements.

Shape and size analysis obviously affect the closeness and effectiveness of packing in a purely mechanical sense; the difficulties with the platelike particles of specular hematite are well known.

Moisture content of the balls

The role of water in balling is discussed in detail below, but it may be noted here that precise regulation of water content is required, the optimum level being determined experimentally. Typical examples are as follows:

- (i) natural American magnetite ~9½% (with bentonite)
- (ii) natural Swedish magnetite ~9% (without bentonite)
- (iii) highly porous artificial magnetite ~14½%
- (iv) less porous artificial magnetite ~9½%
- (v) ground natural specular hematite ~8%
- (vi) ground limestone ~13%

Practical difficulties can arise in control when concentrates are dewatered on filters; it may be difficult to achieve an adequately low moisture level.

Additives

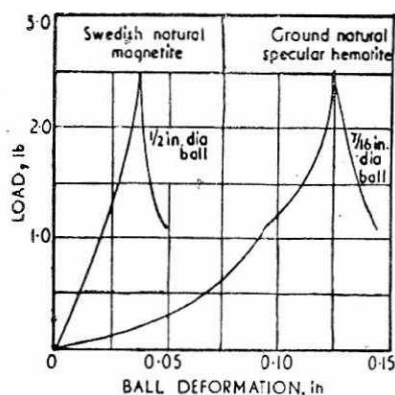
These are used to:

- (i) increase the green strength and/or
- (ii) to increase the dry strength and improve drying behaviour, and possibly the fired strength
- (iii) to produce a self-fluxing pellet.

Many benders have been tried but bentonite is the one most used in the USA. It gives a stronger wet and dry ball in the green state and may also give an increase in fired strength. The optimum addition appears to be around 0.5% and this is usually the current practice when using the natural bentonites from Wyoming or South Dakota. In some cases artificial bentonites may be economically used. In Sweden the use of ferrous sulphate has been successfully developed in relation to shaft kiln operations.

Dolomite or limestone is sometimes used in addition to bentonite^{24, 25} and this work has been carried to its logical conclusion at the Republic plant (grate-kiln) of the Cleveland Cliffs Company where self-fluxing pellets have been produced. Many other additives have been tried experimentally, and for certain applications Ca(OH)₂ is particularly promising.

The use of carbon should be noted. In some instances it may prove useful to supplement the heat supplied in the firing process by the heat of oxidation of carbon (the



5 Two typical green ball compression test traces

carbon being either 'rolled' on to the surface of the ball, or dispersed through the ball, as found most suitable).

Surface properties

It is to be expected, and experience confirms, that balling behaviour is modified by the presence of contaminants on particle surfaces. For example, hematite flotation concentrates which are both slime free and coated with hydrophobic reagents must be reground before balling.

Balling test procedure

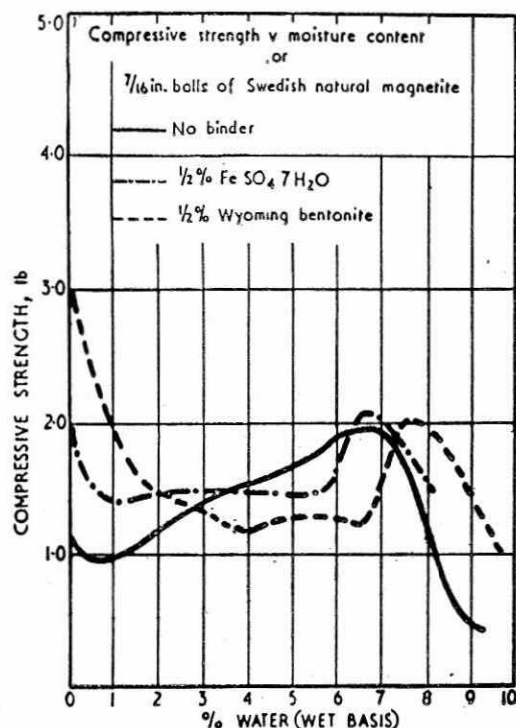
Whilst we believe that balling drums are the best industrial devices we find it more convenient to use a balling disk in the laboratory; these disks may be operated at various speeds and slopes (Fig. 1). Preliminary tests to assess grind, additives, approximate water contents, etc. are, however, made in a very small batch drum.

For the main test the feed material is thoroughly mixed in a paddle mixer, where the moisture level is raised almost to the required level and any additives thoroughly dispersed. This mix is then transferred to a feed bin, and discharged from a rotating feed table via a 'fluffer' to the disk. The speed and slope of this disk are adjusted and the rest of the moisture added by suitably directed spray.

Balling is continuous, the product is continuously screened and the undersize recycled. To produce balls with properties akin to those which will be produced on an industrial scale this operation must be carried out in a standard way by skilled and experienced laboratory staff. In general we find it convenient to work with an initial charge of 250–280 lb of damp feed, which produces sufficient $\frac{1}{2}$ – $\frac{3}{4}$ in green balls for green ball tests and one firing experiment. We would stress that the firing experiments are carried out immediately; owing to ageing effects green balls should not be stored.

Cohesion of green balls and testing procedures

In 1944 Firth¹ observed that balls produced by rolling in a drum developed densities that could only be obtained in powder compacts by the application of very high pressures, e.g. of the order of (at least) 10 000 lb/in². He suggested that similar pressures were generated at the ball surface where very small elements, even a few grains, would carry its entire weight. In 1950 Tigerschiöld and Ilmoni³ rejected this explanation and argued that the 'compression' of the ball was caused by internal contractile forces generated by surface tension and capillarity: they said, 'Balling is caused by the fortunate co-operation between the capillary forces and the vibration massage achieved by rolling.' They presented their views in a quantitative way and drew on related researches in the field of soil mechanics.



6 Changes occurring in ball strength during drying

They pointed out that the height to which a liquid will rise in a capillary is given by:

$$Z = \frac{2\alpha \cos i}{r\rho g}$$

where Z = capillary rise, cm; α = the surface tension, dyn/cm; i = the angle of meniscus at the wall; r = the average pore radius, cm; g = 981 cm/s²; ρ = density of liquid.

If $i=0$, and water is used at room temperature, then $h=0.15$ cm wg.

Tigerschiöld and Ilmoni then calculated the average value of r as a function of the specific area S of the concentrate, the true density of the concentrate ρ_s and the porosity ϵ of the ball (these being measurable quantities) and proceeded to estimate the capillary rise Z , which they equated with the suction, or negative pressure.

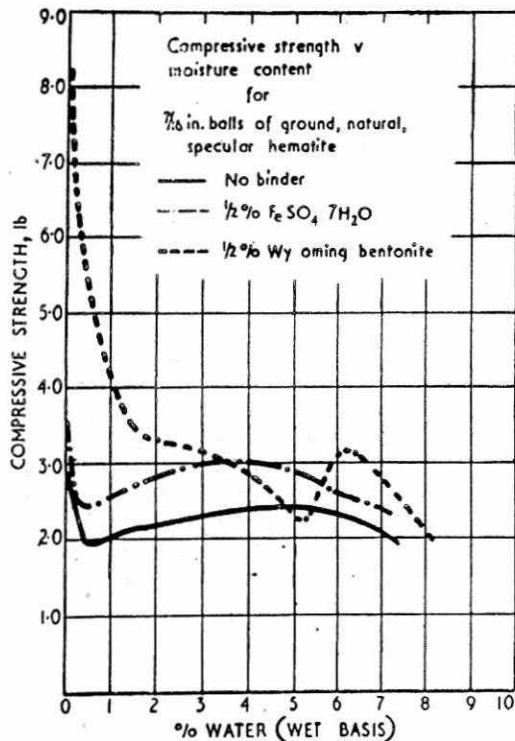
Thus $Z = \{(0.075 \times S \times \rho_s (1 - \epsilon)) / \epsilon\}$ cm where Z the suction in cm wg is equivalent to $Z \times 10^{-3}$ kg/cm² negative pressure.

Values of Z estimated for different pellets were found to be linearly related to the measured crushing strength. Philippoff²⁰ criticized these estimates of the contractile force. He argued that the very high suctions calculated by Tigerschiöld could not be developed in pellets because their limited dimensions never allow Z to exceed a few centimetres. Tigerschiöld and Ilmoni, however, adhered to their view, and there is now general agreement on the major role of 'surface tension forces' in the coherence of wet balls, if not on the quantitative theory.

In 1958 Newitt and Conway-Jones²¹ made an important contribution on balling, with the legitimate simplification (for the purpose of identifying governing factors) of working with commercially uninteresting materials (washed and graded sands and silts). Like Tigerschiöld and Ilmoni they drew on kindred work in the field of soil mechanics and in particular on the work of Fisher²⁷ and Haines.²⁸

Newitt and Conway-Jones, following Haines, identify three states in water-particle systems:

- (i) the 'pendular', when water is present at points of grain-contact only



7 Changes occurring in ball strength during drying

- (ii) the 'funicular', when some pores are fully occupied by water
- (iii) the 'capillary', when all pores are filled, but the surface of the system is not covered by a film.

Considering the capillary state, they observed that any removal of water, by evaporation or otherwise, from a granule containing moisture in the capillary state will produce a curvature of the water surface in the interstices of the surface layer of particles, thus setting up a 'suction-potential' in the ball. The magnitude of this quantity will increase rapidly as moisture is progressively removed until a value known as the 'entry suction' is reached, when air begins to enter the surface pores. The 'suction-potential' within the pellet then increases very slowly during the funicular state which follows.

The entry suction (or suction-potential, usually expressed in cm wg) gives a measure of the resultant forces tending to hold the particles together. For a saturated bed of randomly packed particles of irregular shape, but roughly uniform size, the entry-suction P_e is derived as follows:

$$P_e = x' \left(\frac{1-\epsilon}{\epsilon} \right) \frac{T}{r} \quad \dots (1)$$

where x' is a constant (~ 4); ϵ is the porosity; T is the surface tension, and r is the mean particle radius.

As the saturation s decreases, and the funicular state is entered, the suction potential will increase slowly (e.g. for a medium sand with $P_e = 45$ cm wg, P might increase to 60 cm at 20% saturation).

The cohesive stress f is approximately given by:

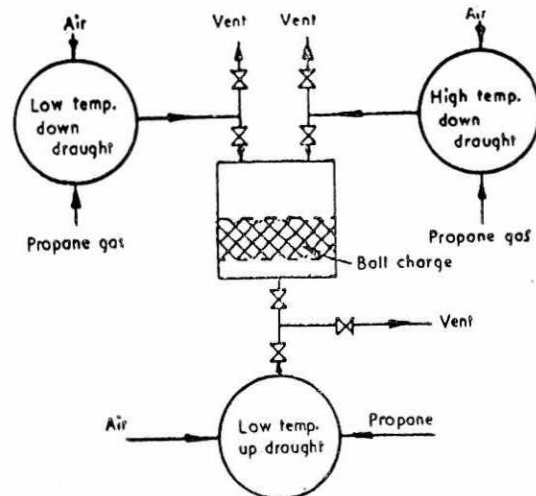
$$f = sP \quad \dots (2)$$

this being the stress which is exerted per unit area of a plane in the granules.

Newitt and Jones, Greaves and English³⁸ and other workers have observed that the size and 'strength' of balls are related by an equation of the form:

$$L = kD^2$$

where L = breaking load; D = ball dia., k = a characteristic constant (ball strength factor).



8 Line diagram of pellet hardening apparatus

If the strength of the ball is proportional to the theoretical cohesive forces then:

$$k = Kf = KsP \quad \dots (3)$$

where K is defined as the 'ball cohesion factor'.

Now K , s , and P may be measured and Newitt and Jones report that several sets of data were correlated closely by giving K the value 0.7. As the theory of plastic deformation confirms that a value of 0.7 is of the right magnitude the explanation of cohesion which they advance appears to be valid.

The maximum strengths of granules formed from uniformly sized sand may be estimated in a similar way. Since a granule at maximum strength is almost completely saturated:

$$sP = P_e$$

$$\text{whence } k = KsP = 4.0 K \left(\frac{1-\epsilon}{\epsilon} \right) \frac{T}{r} \text{ g/cm}^2 \quad \dots (4)$$

and expressing r in terms of particle surface area, density and shape we obtain:

$$k = KC \left(\frac{1-\epsilon}{\epsilon} \right) T \rho_s S^3 \quad \dots (5)$$

where C = constant; ρ_s = particle density; S = specific surface area.

This is an interesting relation which makes explicit the dependence of K on porosity, surface tension, and specific surface. Newitt and Jones and other workers confirm that strength varies directly with surface tension. Calculated values of k based on this equation are not very accurate when mixed particle sizes are used, but the form of the dependence is as predicted.

It will be noted that equation (5) indicates that strength will increase as the porosity decreases; the rolling action of the drum contributes to this by reducing the internal pore space.

In 1961 Rumpf²⁹ presented a very full discussion of the bonding mechanisms in pelletizing. Taking account of surface tension effects he made estimates of the tensile strengths of agglomerates, and developed a technique for measuring these experimentally. He also takes account of van der Waals forces (although, in practical situations, capillary forces predominate). In discussion Goldstick¹⁹ pointed out that in a comprehensive treatment magnetic forces (where relevant) can also be taken into account.

Green ball testing

For purposes of plant design green balls are subjected to two practical mechanical tests: drop tests and compression tests. At the same time moisture content is

determined by drying two or more adequate samples of the green pellets at 100°C.

Drop test

This test is designed to assess the ability of the balls to withstand handling (transport to, and dropping on to the moving grate). The test is essentially a manual one: balls are dropped from 12in and 18in and the average number of drops which they can withstand is noted. We normally standardize on 30 $\frac{7}{16}$ in balls, although for crucial cases a more comprehensive and statistically controlled test may be necessary. Drop numbers of about 6 and 15 are satisfactory values for 18in and 12in drops respectively. Too low a value indicates brittle balls and too high a value would give poor bed permeability. Figure 2 shows a typical histogram obtained for such a test.

Compression tests

Tigerschiöld and Ilmoni³ have described a method of loading green pellets by means of an air plunger device; the air pressure could be built up very rapidly until the pellet failed. We have not found this system to give results of sufficient reproducibility. We have therefore built a device (Fig. 3) employing a mechanically driven plunger which moves downwards at a constant rate of 0.45in/min. The fixed platen is in fact a load cell coupled to a high speed recorder which gives a strength/time trace on a strip chart. It should be noted that whilst the load at fracture is an important property the plasticity of the pellet is also relevant. Thus a very brittle pellet may shatter so that the resultant fine particles both lower the permeability of the bed and also cause dust problems. On the other hand excessively plastic pellets may squash in the bed and this will again hamper bed permeability. Hence it is useful to build displacement transducers into the test device so that a true load/deformation curve may be plotted.

In general, wet compression strengths should lie between a minimum of about 2 lb and a maximum of about 3 lb. The same apparatus may also be used for dry (but not fired) compressive strengths: a good dry strength is needed for that critical phase during drying where the whole weight of the bed is supported by the 'just dry' balls lying on the updraught part of the grate.

It should be pointed out that human error creeps into both drop and compressive strengths in the selection of balls to be tested. Care must be taken to size all the balls to be tested as closely as possible (using gauges) and not to change the properties of the balls by handling. This question of handling is particularly important for balls made from hematites or clayey ores: even gentle pressure between finger and thumb will alter the shape and cause a characteristic loss of sheen on the ball's surface (i.e. water is being withdrawn into the capillaries). There is always a great temptation to choose the best looking balls (i.e. those which are most spherical and smooth), whereas in fact what is required is a truly representative sample.

Figure 4 shows a histogram which indicates the kind of spread which may be expected in compression tests of this kind, and Figs. 5-7 illustrate some of the data obtained with the apparatus just described. Figure 5 demonstrates that specular hematite pellets are more plastic than a natural magnetite, though the crushing loads for the case presented are similar.

Figures 6 and 7 illustrate the changes which occur in ball strength on drying. Two materials were used: a natural magnetite and a ground specular hematite, and

three cases were studied: (a) no binder, (b) $\frac{1}{2}\%$ $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, and (c) $\frac{1}{2}\%$ Wyoming bentonite. The test batches were prepared in a small drum and all had identical rolling times, although these were not necessarily ideal for both materials.

The following inferences may be drawn:

- (i) the use of bentonite increases the water requirement
- (ii) the maximum wet strengths are developed when a binder is used
- (iii) ball strength increases as drying begins
- (iv) binders enhance the dry strength; the effect was most marked in the case of bentonite with specular hematite.

Firing of green balls

Firing experiments are run in the laboratory to simulate industrial cycles already in use and to develop new ones. A flow sheet of the main test rig is shown in Fig. 8.

The grate (bearing the green ball charge) has a hearth area of 1 ft². Three propane-fired combustion chambers (working at different temperature levels) have been found necessary to give a thorough simulation of all phases of complex updraught/downdraught pellet hardening cycles; they are connected to the top and bottom of the bed through a series of high temperature valves. The mass-flow, temperature, and oxygen content of gases entering the bed are all capable of precise regulation, and bed temperatures, emergent gas temperature and pressure drop are continuously recorded.

In a 'straight grate' process the usual bed depth is 12in although this can be varied to suit individual requirements. In general we have found it useful to carry out preliminary shallow-bed studies (2-6in). A special rig is available for this preliminary work and it is possible from the results to decide upon the degree of regrind, the binders and fluxes to be used, gas flow rates, and to get a general indication of the required drying temperatures (the drying stages being very critical). Some 10-20 deep bed tests are usually necessary to optimize a cycle when a new material is being tested.

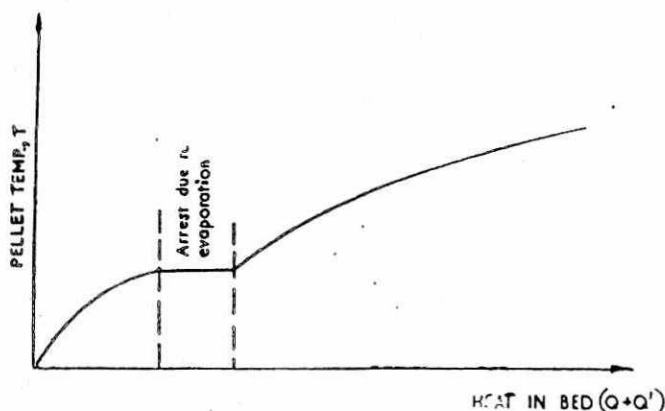
Whilst it may be found that, with the straight grate machine, a 'Reserve' type of cycle works very well with most natural magnetites, quite different cycles may be necessary for other materials. Even so it is useful in many cases to start off with a Reserve type cycle in mind: (a) updraught drying, (b) downdraught drying, (c) downdraught preheat, (d) downdraught ignition, (e) downdraught recuperation, (f) updraught cooling.

The development of an experimental sequence is essentially empirical, involving a good deal of practical experience, and reference should here be made to the excellent papers of Phelps and Anthes³⁰ and of Burge and Wakeman.²⁴ Nonetheless progress has been made in our laboratory by a purely theoretical study of thermal changes in the pellet bed. A brief treatment of these fundamental principles will form a useful prelude to a description of laboratory techniques.

The purpose of the pellet hardening operation is, first, to dry the green balls, and, second, to bring about recrystallization and bonding between the component particles. All this must be done without disruption of the pellets (by the evolution of steam or other gases, or by stresses resulting from thermal shock or phase changes) and at maximum thermal efficiency.

Heat transfer during pellet hardening operations

The problem of heat transfer in packed beds with heat sources and sinks has not been given much attention



9 Temperature/enthalpy diagram

apart from the paper by Elliott and Humbert.³¹ Our solution (due to Mr P. Butterfield) has been to analyse the separate parts of the process, to express the mechanism mathematically, and then to synthesize the elements of the process using a digital computer. In this way a working mathematical model has been obtained which can be checked by comparison with experimental results.

In general it may be said that the temperature profiles in the bed may be reasonably explained in terms of heat transfer with heat sinks (latent heat of evaporation) and heat sources (heat of oxidation).

Evaporation of water

It is assumed that no water evaporates at a particular point of the bed until the temperature reaches an 'evaporating' temperature, and that the temperature does not rise again until all the water at that point has been given off. The wet bulb temperature and not the boiling point has been used for this 'evaporating' temperature since it is an evaporating rather than boiling action. The wet bulb temperature depends upon the gas condition, but a temperature of 60°C is sufficiently accurate for most purposes.

It is also assumed that the steam passes out of the bed without further phase change and without taking any further part in heat transfer; a more complex model can be formulated to take account of continuous condensation ahead of the heat transfer front, with subsequent re-evaporation.

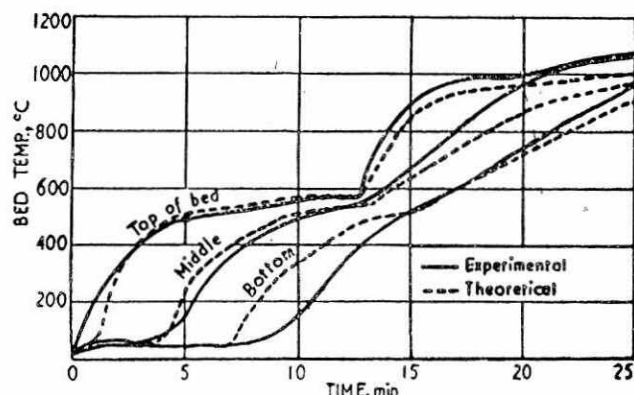
Oxidation

The most difficult part of the process is to analyse the oxidation of the ore (where this is relevant, e.g. magnetites), and a much simplified model has been taken. We have assumed that oxidation takes place only above some known temperature, T_o , and that above this temperature the reaction proceeds at a known constant rate, independent of temperature, liberating heat at R Btu/h ft³. Unfortunately, neither T_o nor R are known with any accuracy, and it is necessary, as they depend upon the reactivity of the concentrate under study, to rely on experience built up by observation of experimental work.

Heat balance

If Q Btu/ft³ is the heat transferred to the bed from the gas and Q' Btu/ft³ is the heat liberated by oxidation then it is possible to relate T , the bed temperature, to $(Q+Q')$ as in Fig. 9 using a heat balance.

This relation between T and $(Q+Q')$ must take account of the varying specific heat of the bed. Note that an arrest can be expected in the temperature enthalpy dia-



10 Comparison between experimental and theoretical temperatures

gram, on account of the latent heat of evaporation.

Rate of heat transfer

The rate of heat transfer to the solid is given by:

$$\frac{\partial Q}{\partial \theta} = h(t - T) \quad \dots \quad (6)$$

where θ is time, h is the volumetric heat transfer coefficient, and t is the gas temperature.

The correlation for the heat transfer coefficient, h , has been taken from Lof and Hawley³³ using a temperature correction from Furnas³⁴:

$$h = 0.79(W/d)^{0.7} \left\{ \frac{(T+t)/2 + 460}{660} \right\}^{0.3} \quad \dots \quad (7)$$

where W lb/h ft² is the flow rate, and d ft is the pellet diameter.

Temperature gradient

Consider a slice of the bed of cross-sectional area A ft² and thickness Δx . At a given time let the gas temperature entering this slice be t , and leaving be t' . Then the rate at which heat is being transferred to the slice is $AW C_p (t - t')$ Btu/h. Dividing this quantity by $A \Delta x$, the volume of the slice, we find that heat is being transferred to the bed at a rate of $WC_p (t - t')/\Delta x$ Btu/h ft³. Now as Δx tends to zero, $(t - t')/\Delta x$ tends to $-\partial t/\partial x$, where x is distance in the direction of gas flow. Hence:

$$\frac{\partial Q}{\partial \theta} = -WC_p \frac{\partial t}{\partial x}$$

which, using equation (6), can be written,

$$WC_p \frac{\partial t}{\partial x} = -h(t - T) \quad \dots \quad (8)$$

Synthesis using a computer

Equations (6), (7) and (8) describe the process subject to our assumptions, and these equations have been solved on a digital computer by the following procedure:

- (i) the temperature is known initially at all parts of the bed, and so equation (8) may then be solved to give the respective gas temperatures
- (ii) with t and T known, we find $\partial Q/\partial \theta$ from equation (6) and, by adding the quantity $(\partial Q/\partial \theta)\Delta \theta$ to the original value of Q , we find the new value of Q after a small period of time $\Delta \theta$
- (iii) provided $T \geq T_o$ and oxidation has not been completed, $R\Delta \theta$ is added to Q'
- (iv) the new values of T are found using a graph such as Fig. 9
- (v) with the new values of T the whole procedure is repeated as many times as necessary.

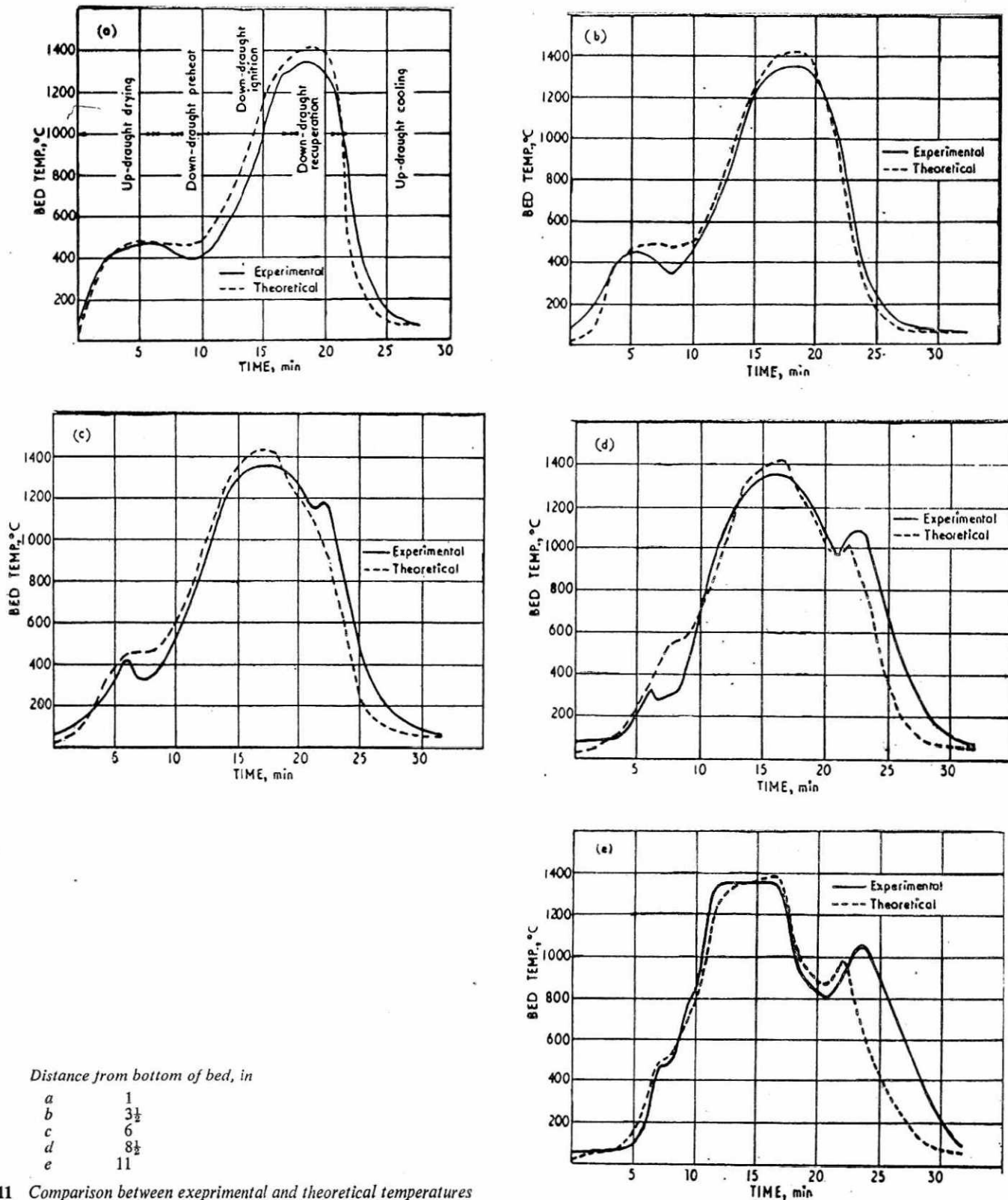
The whole process is carried out at high speed on a digital computer and temperatures are punched out at regular intervals.

Results

It is interesting to compare theory and practice for two series of trials.

The first comparison is shown in Fig. 10 (for a down-draught drying and firing cycle) and it will be seen that

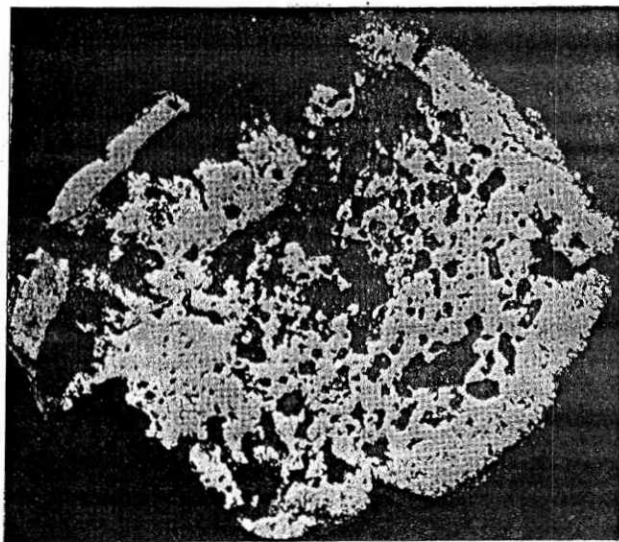
there are discrepancies. In the first part of the test, the theoretical temperature rises earlier than the experimental, and in the second part of the test the theoretical temperatures are generally too low. It is known that in this (early) experimental test large heat losses occurred from the rig and this could delay the arrival of the 'heat front' to the bottom of the bed. The problem of oxidation is also a difficult one and may not have been adequately included in the model. Nonetheless the general agreement between the curves is encouraging.



Distance from bottom of bed, in

a	1
b	3½
c	6
d	8½
e	11

11 Comparison between experimental and theoretical temperatures



12 Spongy particle of artificial magnetite

×2.4

The second comparison is shown in Figs. 11a-e (for a Scandinavian magnetite). Here agreement is much better, there being no serious differences until the cooling section where the theoretical temperature drops too quickly. It is likely that some oxidation was in practice delayed into the updraught cooling period, thus slowing the rate of cooling of the bed.

The approach is clearly encouraging, and the method is even now used to supplement experimental work. Thus the effect of altering any of the many parameters can often be fairly accurately assessed by this method, so that it is possible to optimize the process before doing the final experimental runs.

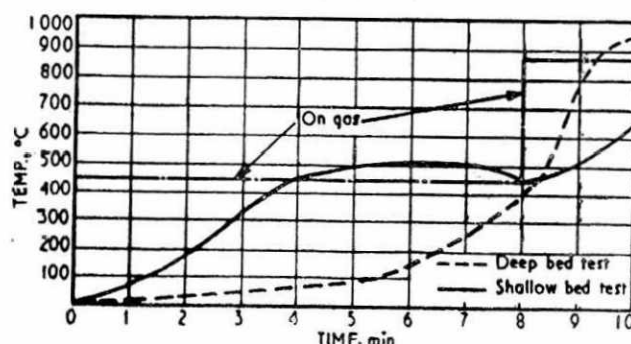
The theory obviously tells us nothing of the recrystallization processes, or the final pellet strength, but it is possible, by experience, to link the various temperature gradients with the actual behaviour of pellets on a grate.

Laboratory tests

It is probably best to discuss the experimental development of a cycle in terms of an actual example, in this case an artificial magnetite which presented many difficulties. The magnetite particles were spongy rather than solid particles (Fig. 12) giving a very high surface area. In consequence the magnetite was extremely reactive and readily oxidized, and in addition the moisture content of green balls was exceptionally high.

Shallow bed tests were performed to determine the amount of regrind necessary on part of the concentrates. In other words it was not possible to infer the amount of re-grind required merely from the properties of the green pellets. At first 0.5% Wyoming bentonite was used as a binder but in later tests an 'artificial bentonite' was substituted and the effects of an addition of recycled ground pellets were tried. It might be added that the material balled very easily and gave satisfactory green ball properties in practically all cases. In due course these shallow bed tests yielded very satisfactory pellets.

The first deep bed tests proper were carried out using 'Reserve' cycle times and temperatures, the normal drying temperature of 450°C for both up- and down-draught phases having been proved satisfactory in the shallow bed test. However, the resultant pellets were badly cracked and a large proportion of them had broken down completely into chippings. This major difference of



13 Comparison between deep and shallow bed tests, showing effect of temperature gradients

behaviour between shallow and deep bed tests is unusual. It was also noted that shrinkage of the bed was severe (15–20%).

It seemed possible that the cracking could arise either from reoxidation beginning at unusually low temperatures (this artificial magnetite being extremely reactive) or from sulphur evolution (the sulphur content was 1%). Attempts were made to diminish the rate of sulphur emission during the high temperature preheat phase (by using different degrees, and/or several steps of preheat). This treatment did little or nothing to prevent cracking. Further tests were then carried out, and by holding the process at various stages in the cycle it was established that breakdown occurred early in the cycle. Successively lower preheat temperatures were tried and the product pellets improved progressively as the preheat temperature was lowered. However, even at the lowest preheat temperatures (approaching the drying temperatures) the final product was still not acceptable.

The whole problem was then reassessed in terms of the following possible causes of cracking:

- (i) sulphur emission
- (ii) rapid reoxidation at a high or intermediate temperature
- (iii) rapid re-oxidation at a low temperature
- (iv) inadequate drying causing spalling when preheating
- (v) rapid shrinkage of the pellets
- (vi) thermal shock due to too rapid temperature changes during any part of the cycle.

The first approach (sulphur emission) had not proved fruitful. In general, disruption of the pellets was shown to occur at low temperatures, ruling out the second possibility. Pressure drop readings showed that shrinkage occurred largely during the ignition phase (1315°C). In general temperature traces indicated that drying was adequate (and indeed the drying phase had been extended to quite exceptional lengths).

It was then noticed that the rate of temperature rise was much more severe during high and intermediate temperature phases of the operation in the shallow bed tests than in the deep bed tests, whilst the reverse was true in the lower temperature phases (see Fig. 13). Thus the centre of the shallow beds rose at the rate of 150 deg C/min during up- and down-draught drying at 454°C, and during the preheat at 871°C, whereas the centre of the deep beds had temperature gradients ranging from 250 deg C/min and 800 deg C/min during the same periods. The lowest gradients occurred with the lowest preheat temperatures. At the same time careful examination of the temperature recorder charts showed the very striking effects of reoxidation. At each thermocouple level the heat of oxidation drove the bed temperature to above that of the current on-gas temperature.

TABLE III Details of laboratory cycle

Phase	Time, min	Flow ft ³ /min STP	Temp., °C
Updraught drying	7.5	360	385
Downdraught drying	9.0	320	330
Preheat	2.0	280	870
Ignition	10.0	240	1315
Recuperation	2.5	240	760

The heat released was passed successively downwards causing a leap-frogging effect in the temperature traces. The net result was that by the time the centre of the bed was reached the rate of temperature rise was excessive. The centre of the deep beds always got the worst treatment due to the selection of an up- and downdraught type of cycle. However, with an all downdraught cycle the bottom of the bed was subjected to an even worse treatment.

It therefore became clear that oxidation was well advanced even during the drying phases and that these must therefore be extended to allow oxidation to go to completion at these temperatures and gas flows. It thus became possible to achieve a cycle with a low-temperature updraught drying phase and an even lower temperature downdraught drying phase such that oxidation was essentially completed within these two periods; pellet strength was excellent and breakdown (concentrated at the bed centre) was within reasonable limits. Table III illustrates the laboratory cycle chosen, and from this, by experience, a satisfactory industrial cycle can be formulated.

TESTING OF FIRED PELLETS

Much useful information can be obtained by unloading the pot grate very carefully and noting various points of interest.

Strength tests

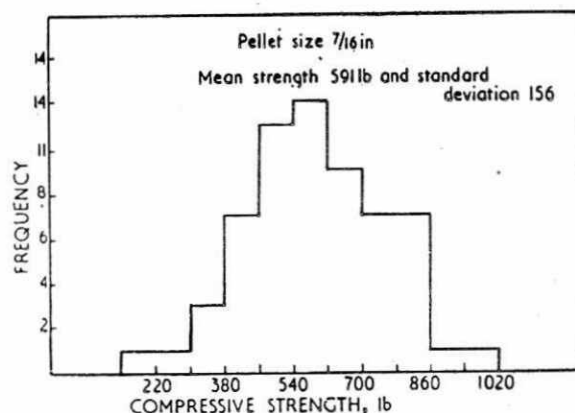
Two tests have been adopted in the USA for assessing 'strength' and we use the same procedures in our own laboratory. Carefully sized pellets are taken from the top, middle, and bottom of the bed; we normally test a total of 60, though the number may vary with the experimental scatter experienced.

(a) Compressive strengths

The pellets are crushed in a standard compression testing machine accurate to 1 lb. A typical histogram for a series of tests is given in Fig. 14. Commercially satisfactory pellets usually have compressive strengths of 300 lb or more.

(b) Tumble test

The presence of excessive fines can spoil a high pellet burden, so a test has been adopted in the USA to assess abrasion resistance. A standard ASTM coke abrasion drum is used (36 in dia. × 18 in, 25 rev/min). Two 2 in high lifters are fitted inside the drum. A 50 lb charge of ($-\frac{1}{2} + \frac{1}{4}$ in) pellets is loaded into the drum and tumbled for 8 min. The discharged products are sized and the cumulative percentages passing $\frac{1}{4}$ in and 28 mesh determined. There is general agreement that 6–8%–28 mesh is satisfactory. Inspection of the $-\frac{1}{4}$ in + 28 mesh fraction is useful because it can indicate what (if any) breakdown by 'chipping' has occurred, as distinct from abrasion. The product of a test run is completely screened to assess breakdown and shrinkage occurring during firing; artificial magnetites and natural limonites have been observed to shrink considerably.



14 Fired pellet compression test (ground natural specular hematite)

Macro- and chemical examination of fired pellets

The oxidation of Fe_3O_4 to Fe_2O_3 is an important feature of magnetite pelletizing operations, and requires careful control of the oxygen potential of the firing gases in experimental work if results comparable with practice are to be achieved.

The practical significance of oxidation is threefold. First, the exothermic heat of oxidation (about 300 000 Btu/ton of pellets) is a significant factor in the heat balance, affecting both plant design and conversion costs. Secondly, oxidation is an important factor in the bonding mechanism of pellets. Thirdly, blast-furnace operators prefer a well oxidized pellet.

The progress of oxidation (beginning at the surface and proceeding inwards) can be usefully studied by carefully sectioning and polishing. Figure 15 shows cored pellets; the boundaries between the outer Fe_2O_3 and the cores are clearly seen. Shrinkage cracks are also present at the interface.

Oxidation should also be assessed (in the overall sense) by analysis for Fe^{++} ; great care must be taken with sampling and with analytical methods if satisfactory results are to be achieved.

The mechanism of bonding in magnetite pellets has been particularly well studied by Cooke and Ban^{34,35} and a useful review is given by Tigerschiöld.⁴ An increase in strength is found even at relatively low temperature ($\sim 300^\circ\text{C}$) by surface oxidation of the magnetite to hematite, binding the grains at points of contact. In the presence of sufficient oxygen hematite will form progressively, recrystallizing at 1100–1200°C. If insufficient air is available (or in the presence of carbon) retained magnetite will recrystallize at about 900°C. In the 1000–1200°C range some slag formation can occur (at these temperatures between SiO_2 and FeO , but not with Fe_2O_3), resulting in a weak bond, and also (by coating the grain) hindering further oxidation of Fe_3O_4 . It may also be noted that Fe_2O_3 will revert to Fe_3O_4 at temperatures in the region of 1300°C if the partial pressure of oxygen in the environment becomes too low.

Utility of test work

Test work of the kind described is an essential preliminary to the preparation of pelletizing schemes. Some materials (e.g. natural magnetite concentrates) are so well understood, by virtue of accumulated experience, that little more than confirmatory tests are usually required.

Other materials, such as artificial magnetites and hematites, and fine natural hematites (blue dust, concentrates, etc.), are less well known, and careful studies are essential. In all cases both experimental skill and engineering



15 Pellets from laboratory tests exhibiting Fe_2O_3 rims and recrystallized cores of original magnetite

×2.4

know-how are necessary if laboratory results are to be successfully transformed into practical cycles and machine design.

CONCLUSIONS

The object of this paper has been to survey the pelletizing of iron ore, to present as logical a background to both balling and firing operations as seems possible at the moment, and to indicate the nature of the laboratory work upon which practical plant designs are based. No attempt has been made to describe actual operating plants, but much information will be found in the literature: for horizontal grate machines attention may be drawn to the paper by Haley and Apuli.³⁶

In general it has been seen that the laboratory work necessary for pelletizing operations is much more complex than is required for comparable studies in sintering. The equipment required is considerable, and so is the skill required in experiment and interpretation if meaningful results are to be obtained.

Some attempt has been made to indicate the merits of pellets as a blast-furnace burden. The situation here is changing rapidly and there is no simple answer to the pellet versus sinter controversy. However, the following remarks seem to be relevant:

1. 100% pellets provide about the best blast-furnace burden available. The use of fluxed pellets, together with sized coke (or even coke pellets) seems to be the logical conclusion to the search for a 'chemically engineered' burden.
2. Pellets can be made economically from fine concentrates, or from naturally occurring fine materials, after suitable regrinding if this should be necessary. Hence pelletizing, (in association with concentration processes where necessary) can make possible the opening-up of hitherto unusable iron ore deposits.
3. Existing ore-preparation plant/sinter plant/blast-furnace plant combinations can yield excellent results if sufficient care is taken in screening into the blast-furnace skip. Hence, where there is a large existing capital outlay in the form of sinter plants, and where ores suitable for sintering are in good supply, blast-furnace performance is perhaps best uprated by the installation of much more comprehensive burden screening plant. This obviously increases the circulating load to the sinter plant, but in some cases this will not be disadvantageous (the proportion of return fines in circulation on existing plants is rarely optimized).
4. Pellets can be transported with relatively little degradation. Hence the possibility opens up of installing pelletizing plants at the mine, and of transporting the pellets by rail or ship to distant users. The world market for such a product is large, and the outlook for such enterprise is good, parti-

cularly since pellets should sell at a premium relative either to concentrates or direct shipping ore.

5. Pellets also form an excellent burden for direct reduction processes, and are therefore of interest wherever a small-scale iron industry is envisaged. A combined grate, reducing-kiln operation is particularly attractive, the green balls being dried and partially hardened on the grate, and then not merely hardened, but reduced in the kiln. The process can be preceded or followed by appropriate mineral dressing operations, and can provide a feed for cupola and/or electric furnace melting and refining, either in an integrated sense or elsewhere.

Finally, it is to be hoped that the information presented will enable prospective users of pelletizing plant the better to assess their own needs, and the sometimes conflicting claims of designers and manufacturers.

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