Investigations of several variables in the Dwight-Lloyd McWane ironmaking process

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INVESTIGATIONS OF SEVERAL VARIABLES IN
THE DWIGHT-LLOYD McWANE IRONMAKING
PROCESS

BY
JAMES ORLAN HOOD, JR.
1945 -

A
THESIS
submitted to the faculty of
UNIVERSITY OF MISSOURI - ROLLA
in partial fulfillment of the requirements for the
Degree of
MASTER OF SCIENCE IN METALLURGICAL ENGINEERING
Rolla, Missouri
1970

Approved by
(advisor)
ABSTRACT

The Dwight-Lloyd McWane Ironmaking Process (D-LM) is the first commercial process of its particular type and one of the few commercial processes involving prereduction of iron ore in existence today. This presentation reviews some promising ironmaking processes which involve iron ore prereduction, with special attention being given the D-LM process. Although the D-LM process is continuous from iron ore to high quality molten cast iron, the research and discussion herein is limited to principal variables involved in the production of high quality carbonized pellets in the prereduction phase of the operation. The variables of process temperature and pellet carbon content are studied and related to the chemical property of prereduction of oxides, the physical property of pellet crush strength, and the electrical property of pellet resistance. Through the use of a bench-scale test devised herein, it is found that maximum prereduction of iron oxides is obtained at approximately 20 percent pellet carbon and a carbonizing temperature of 2000°F., carbonized pellet crush strength increases drastically as pellet carbon increases above 25 percent and reaches a maximum at 1800°F. carbonizing temperature, and electrical resistance of pellets decreases rapidly as both pellet carbon and carbonizing temperature increase. With the use of these relationships it is proposed
that the bench-scale prereduction apparatus utilized in re­search be used as a quality control device in the prereduction step of the D-LM ironmaking process.
ACKNOWLEDGEMENT

This presentation was made possible by the assistance and guidance of Mr. Warren C. Jeffery, General Manager of Product Development, McWane Cast Iron Pipe Company, Birmingham, Alabama. My sincere thanks to Mr. Jeffery and also to Mr. Glenn E. Waldrop, McWane Iron Company, Theodore, Alabama for his assistance in chemical analyses and laboratory technique.
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I. INTRODUCTION

The Dwight-Lloyd McWane Process (D-LM) for the making of pig iron is one of many iron-making processes which has certain problems arising out of its inherent characteristics. These problems concern mainly raw materials quality and quantity, and temperature and atmosphere control in the high-temperature zone utilized in prereducing the oxides of iron in the form of pellets, i.e., carbonizing.

The basic variables and properties responsible for these problems are related in this presentation in an effort to design a bench-scale test that would adequately characterize the raw material, filter cake, to permit prediction of the properties of the product after carbonizing in the D-LM prereduction circuit.

Low values of oxygen removal during prereduction increases the work load for the electric smelting furnace. Low pellet crush strengths present problems in electric smelting of the self-fluxing, self-reducing pellets due to fines created in handling and charging. Fines, in turn, produce a highly electrical resistant load detrimental to efficient electrical smelting.
II. REVIEW OF LITERATURE

A. THE FLUIDIZED BED PROCESSES

1. ESSO RESEARCH-LITTLE DIRECT IRON PROCESS\textsuperscript{1}

The process (FIGURE 1) was developed by Arthur D. Little, Inc., Esso Research and Engineering Company, The Texas Company, and The M. A. Hanna Company.

This process is a continuous one operating at substantially atmospheric pressure. It is based on the partial combustion of natural gas with air to essentially carbon monoxide and hydrogen. With the air preheated to 1500-1600°F. and the natural gas preheated to 600-700°F., the partial combustion reaction provides sufficient heat for the reduction reactions, and produces a reducing gas that is about 20.5\% carbon monoxide, 41\% hydrogen, and 38.5\% nitrogen.

Ore is first fed to a fluidized bed preheater where a portion of the exit gas from the reducing reactor is burned completely to preheat the ore to 1500-1600°F. The preheated ore is then passed by gravity to the reducing reactor where it proceeds downward through two or three reducing fluidized bed stages, contacting high temperature reducing gas. The exit gas has a calorific value of 120 B.T.U. per cubic foot and is not recycled. Instead, it passes through a dust separator and is then used to preheat the ore, air, and natural gas. It also provides fuel for a power plant, similar to the
FIGURE 1.--THE ESSO RESEARCH-LITTLE PROCESS$^{2}$
practice with a blast furnace.

The reduced ore passes to an ore cooler where it contacts cool exit gas, and is then agglomerated before melting by briquetting or by squeeze rolls.

There is no specific data available concerning quality control of product, definite type of ore best suited, and fuel requirements. The main operating difficulty mentioned was sticking of the ore in the fluid-bed gas reactor.

2. H-IRON PROCESS

The H-Iron Process (FIGURE 2) was developed by Hydrocarbon Research, Inc. and Bethlehem Steel Corporation. The nature of the process concerns the production of pure hydrogen, fluidized reductions of the ore in several stages, and briquetting of the product.

Iron ore fines, 20 to 325 mesh, are first fed to a rotary drier or shaft furnace which removes moisture and preheats the ore to the reduction temperature of 900°F. The preheated ore is then transferred pneumatically to a vertical charging cylinder and then batch charged under hydrogen pressure to the reduction vessel. Dry hydrogen preheated to 100°F. and under a pressure of 400 p.s.i. is passed slowly through the charge to accomplish the reduction. A modified fluid bed with calming devices is used.

The exit gas from the reducing vessel, still maintained under pressure, is cooled to about 100°F. to remove water vapor, and some gas is vented, as needed, to remove other impurities. Make-up hydrogen is added at this point and the
FIGURE 2.—THE H-IRON PROCESS
hydrogen is recompressed, reheated, and returned to the reducing vessel.

The reduction step is a batch operation with a single ore charge being reduced to iron in about eight hours. It is then transferred by dense-phase hydrogen transport to a dump hopper. The resulting reduced iron powder is pyrophoric (ignites spontaneously) and must be agglomerated by hot compression under reducing conditions through squeeze rolls to powder metal plates which can be broken to platelets or chips by breaker rolls. These are then treated with oil to prevent reoxidation during storage.

No data is available as to the hydrogen content of the H-Iron or its possible effect on steel quality. At the low reduction temperature used, hydrogen does not remove much sulphur during the actual reduction operation.

The flow of solids through the H-Iron reducer is shown by FIGURE 3, while the flow of hydrogen through the same system is shown by FIGURE 4.

A question of utilization of H-Iron chips in electric furnaces was encountered where chips made up a large percentage of the charge. Electric furnace heats were made with about 63% of the charge consisting of H-Iron chips, 90% reduced, and made from Cerro Bolivar ore. These trials indicated some deficiencies in the shape of the compacted H-Iron when used in high percentages and not thoroughly mixed with the scrap. The ability to control carbon at melt temperatures and the metallic yields were consistent with
FIGURE 3.—FLOW OF SOLIDS THROUGH H-IRON REDUCER

FIGURE 4.—FLOW OF HYDROGEN THROUGH H-IRON REDUCER
expectations, but heat times were 55% and power consumption was 30% above normal. Research is now being conducted to effect a new shape of H-Iron compact to reduce excessive heat times and power consumption.

3. NU-IRON PROCESS

The Nu-Iron Process (FIGURE 5) was developed by United States Steel Corporation based on recent patents to E. R. Shipley, assigned to United States Steel Corporation. Continuous gaseous reduction of iron ore in a fluidized bed system constitutes the nature of the process.

High-grade ore fines are fed continuously to a preheater where the temperature is raised to 700°F. by sensible heat from the exit gas produced by the subsequent reducing reactors. This preheated ore then passes to an ore heater where the ore is heated to about 1700°F. by combustion of natural gas or other suitable fuel. It is not known whether the ore preheater and ore heater are fluid bed reactors, but they could be, as fluidized solid/gas contact would be the most practical method of carrying out the preheating step. The ore at 1700°F. then passes to a primary reactor where the temperature is maintained at about 1300°F. and the ore is reduced essentially to FeO. The sensible heat in the ore supplies a part of the heat needed for the endothermic reducing reactions of this primary reactor. The partially reduced ore then passes to a second fluid bed reactor, maintained also at 1300°F., where the FeO is reduced to metallic iron. No information has been published as to quality control of the
FIGURE 5.—THE NU-IRON PROCESS
product or percentage recovery of iron from the ore.

The reduction step is a continuous operation whereby ore from the heater is passed countercurrent to reducing gas in two fluid bed stages. The exit gas, after passing through the ore preheater, consists of carbon monoxide, carbon dioxide, hydrogen, water vapor, and nitrogen, and passes to a cyclone, waste heat boiler, and electrostatic precipitator to eliminate dust and recover water vapor. Make-up reducing gas from the reforming unit is added at this point and the gas compressed to 60 p.s.i. The carbon dioxide is removed in a monoethanolamine scrubbing tower. The resulting gas, consisting essentially of carbon monoxide and hydrogen, is then reheated to 1600°F. in a gas-fired heater, fired with either natural gas or spent gas bled from the system.

This process is in general similar to the other fluidized bed processes, such as H-Iron, except that it is continuous and operates at a lower pressure and a higher temperature than H-Iron. True fluidization of solids is attained in the reducing reactors and probably also in the ore preheater and heater. Moderate pressures of 60 p.s.i. are used to increase utilization of the reducing gas per pass and probably to minimize the carry-over of dust from the fluidized beds. The unused gas is recycled so that essentially all the hydrogen is eventually used as a reducing agent, except for that which is bled from the system to maintain nitrogen content at 10 percent or less.
4. CRITIQUE OF FLUIDIZED BED PROCESSES

The three preceding processes, Esso Research-Little Direct Iron Process, H-Iron Process, and Nu-Iron Process, involve a fluidized bed reaction whereby finely divided particles of iron ore are reduced by gaseous reductants consisting mostly of hydrogen and carbon monoxide. The fluidized bed is a relatively new concept in reducing iron ores and is still in its developmental stages. Problems have been encountered concerning pressurized feeding of the reducing gases to properly suspend the "fluid bed" of iron ore particles to allow maximum exposure of each particle to reducing gases passing through the bed. The heated particles have a tendency to agglomerate, thus causing the suspended bed of fine iron ore particles to collapse.

The reduction of iron ores in the fluidized bed processes occurs at a very rapid rate, but the price of a very complex system must be paid to achieve this rapid reduction. The product obtained via these type processes is reduced iron of about 90% purity in the form of platelets, briquettes, or powder, and is difficult to melt efficiently in an electric furnace operation. It can be, and usually is, used as a "synthetic scrap" and included in the charging mix in blast furnaces, electric furnaces, or cupolas.

This type process and product is unsuited for the McWane organization since a molten product of casting quality is desired.
B. THE ROTARY KILN PROCESSES

1. KRUPP-RENN DIRECT IRON PROCESS

The Krupp-Renn Process (FIGURE 6) was proposed by Johannsen and developed by Krupp in Germany prior to World War II.

Crushed ore, limestone or dolomite, and coke breeze, bituminous coal fines or other low-grade solid fuels are charged to a long rotary kiln similar to a cement kiln which is usually fired with pulverized coal. The charge materials move counter-current to the products of combustion. Conditions are controlled so that reduction of the iron is virtually complete about two-thirds of the way through the kiln, and so that in the final section (nodulizing zone) a temperature of 2200-2400°F. is reached. A relatively small heat flame is used to control the temperature in the nodulizing zone within narrow limits as most of the fuel is charged with the ore. Air is blown under pressure to the nodulizing zone to give rapid combustion of the carbon monoxide generated in the reducing zone. The temperature in the nodulizing zone is such that the slag just becomes viscous and the reduced iron particles ball up into nodules in the slag. The semi-solid mass discharged from the kiln is sprayed with water, crushed and ground, and the nodules recovered by screening and magnetic concentration. The nodules vary in size from two inches down and average about 1/4 inch in diameter. The recovered grains smaller than 1/16 inch are recharged to the kiln and eventually recovered as larger nodules.

Silica and titanium are almost completely eliminated in
1 - IRON ORE CRUSHERS  
2 - FUEL  
3 - IRON ORE  
4 - MAGNETIC MIDDINGS  
5 - MIXER - FEEDER  
6 - ROTARY KILN  
7 - PREHEAT ZONE  
8 - REDUCTION ZONE  
9 - BALLING ZONE  
10 - PULVERIZED COAL BURNER  
11 - CONVEYOR  
12 - GRINDING MILL  
13 - MAGNETIC SEPARATOR  
14 - IRON NODULES  
15 - SLAG

FIGURE 6.--THE KRUPP-RENN PROCESS
the Krupp-Renn Process. Most of the nickel, about 30% of the manganese, and very little chromium are recovered in the nodules, which are high in sulphur unless low sulphur fuel is used. The nodules usually analyze 94% iron plus nickel, 0.9% carbon, and about 3% adhering slag. With low-sulphur anthracite fines used as a reductant, sulphur is about 0.1%, while with coke breeze, sulphur is from 0.6 to 0.8%. The metallic iron represents about 99% of the total iron content. Recovery of iron from the ore is quite high, 92 to 93%.

Some difficulties are experienced in the process with ring formations in the kiln which considerably increases refractory costs. The very big disadvantage of the process, however, is the large sulphur pick-up (at least 30% of that in the fuel) and only nodules made from low sulphur fuel have been found suitable as "synthetic scrap" for steel-making furnaces. The large sulphur pick-up results from the highly acid slag which is necessary for proper slag viscosity required for nodulizing.

2. R-N DIRECT IRON PROCESS

The R-N Direct Iron Process (FIGURE 7) was developed by Republic Steel Corporation and National Lead Company.

The process involves the direct reduction of iron ore carried out in a rotary kiln in a controlled atmosphere and a controlled temperature range which is below the melting point of iron and of practically all the constituents of the charge. With U. S. Southern Clinton ores this range is approximately 1800-2000°F; other ores may require a different range of
FIGURE 7.—THE R-N DIRECT IRON PROCESS"
temperatures. Low grade solid fuels, such as coke breeze, bituminous or lignite char, and anthracite fines, can be used as the reducing agent. A large excess of carbon is used, but most of this is recycled. The kiln is heated with gas or oil and a system of air pipes spaced along the length of the kiln controls the temperature and insures effective utilization of the carbon monoxide given off during the reduction. The ore is fed in the form of screened material up to one inch top size or as pelletized fines, along with limestone and dolomitic limestone, depending upon requirements and the solid reductant. The product from the kiln is cooled either slowly or rapidly as requirements dictate which maintains the iron in a passive condition to corrosion and prevents reoxidation and loss of metal. The kiln discharge materials are separated by screening and by gravity and magnetic means. The excess solid carbon fuel is cleaned, dewatered, and returned to the kiln. The magnetic portion containing the reduced or metallic iron is freed of gangue by stage-grinding and magnetic and gravity separation procedures. The high-grade product is pressed without binder into cylindrical briquettes weighing 5-25 pounds. The second grade product is made into smaller briquettes suitable for blast furnace feed.

Sulphur, phosphorous, silica, and titanium in the process materials are eliminated to a high degree and carbon in the product is less than 1%.

Depending on the quality of the ore, concentrates in the range of 95% total iron (90% or better metallic iron) and
less than 2-3% silica can be produced with overall iron recovery of 85-90%.

With some low-grade ores, the process gives two products. A high-grade product of 60-80% of the overall products contains 90-95% total iron (about 90% metallic iron) and less than 3% silica. The remaining 20-40% of the overall product may contain 80-90% total iron (60-80% metallic iron) and up to 8% silica. The richer grade of product is suitable feed for electric or open-hearth furnaces. The second grade of product is particularly good for the blast furnace.

3. ORCARB PROCESS FOR DIRECT IRON ORE REDUCTION

The Orcarb Process (FIGURE 8) was developed by Carl E. Lesher, an independent consulting engineer, and the Swindell-Dressler Corporation.

It is not a sponge iron process nor is it designed to make synthetic scrap. The process replaces sintering or pelletizing, coke ovens, and the blast furnace. It substitutes electric power for coke in the final melting and smelting steps and thus requires but half the normal amount of coking coal. The two steps, reduction of oxides and smelting to metal, are accomplished at their respective temperature levels and are separately controlled.

Fine-sized ore is preheated to 1000°F. or more in a direct-fired rotary kiln and delivered continuously at a regulated rate to a mixing conveyor where it meets and is thoroughly mixed with a regulated amount of fine coal—minus four mesh—and delivered continuously to the Orcarb retort.
FIGURE 8.—THE ORCARB PROCESS
The Orcarb retort is a horizontal rotating cylinder, externally heated and enclosed in an insulated, stationary, outer cover. The charge enters at one end and the products, solids, and gases, leave at the other end. Tar vapor is condensed and the vapor used for process heat. The process is said to be simple to operate when properly instrumented. The bed material in the retort is maintained at 900-950°F. It is reported that there has been no formation of rings or crusts on the interior wall of the retort.

The Orcarb retort discharge plus the required flux is delivered continuously to the prereduction kiln, and heated to 1900-2000°F. It then proceeds continuously to the electric furnace, entering through the furnace roof directly onto the molten bath where it is rapidly smelted; the feed seldom accumulates to a depth of more than a few inches. Gases evolved in the electric furnace--largely carbon monoxide--are conducted to the discharge end of the prereduction kiln where air for combustion is admitted. The waste gases are vented through a dust collector to atmosphere. No auxiliary fuel has been required to maintain the 1900-2000°F. in the kiln. The absence of clinker accretion in the kiln, even at high temperatures, is a decided advantage and is ascribed to the carbon coating on the ore particles which prevents fusing.

Process electric furnace smelting affords close metallurgical control of the product, but no details are available as to control of various impurities, other than silicon and carbon control which is normally afforded by the electric
furnace pig-iron process. Also, there is no information available on the percentage recovery of iron from the ore.

This process in general is somewhat similar to the Dwight-Lloyd-McWane and Strategic-Udy processes. Its unique feature is the low temperature carbonization, which bonds or covers the ore particles with low temperature coke that is much more reactive and effective than other forms of high temperature carbon. It is claimed that this low temperature coke makes feasible 70 to 80% reduction of the iron oxides in the prereduction kiln, which should materially reduce the electric furnace power consumption enough to facilitate an economically sound process. However, the use of three units before the electric furnace—ore heating kiln, Orcarb kiln, or retort, and the prereduction kiln—make it a more complicated process than either the Dwight-Lloyd-McWane or the Strategic-Udy process, each of which has only one main process unit preceding the electric furnace.

4. STRATEGIC-UDY DIRECT IRON PROCESS

The Strategic-Udy Process (FIGURE 9) was developed by Strategic Materials Corporation, Buffalo, New York, and Montreal, Canada.

In the process, a mixture of iron ore, flux, and carbonaceous reductant is continuously fed to a rotary kiln where the iron ore is heated and partially reduced while the flux (limestone or dolomite) is calcined and combined with the ore. The maximum temperature of the free-flowing discharge from the kiln is controlled at some point between 1800 and 2300°F.,
FIGURE 9.--THE STRATEGIC-UDY DIRECT IRON PROCESS

[Diagram showing a flowchart of the Strategic-UDY Direct Iron Process, including components such as TRACK HOPPER, VIB. FEEDER, VIB. SCREEN, CRUSHER, DUST COLLECTOR, FAN, ORE LIMESTONE COAL STORAGE BINS, FLO-CONTROL FEEDERS, COLLECTING CONV., KILN, AUXILIARY FUEL + AIR, WEIGH HOPPERS, FEED HOPPER, ELECTRIC FURNACE, PIG MOLD, and SLAG LADLE CAP.]

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varying for different ore. Beyond this temperature, the materials start to cake and adhere to the kiln walls forming a ring. Sufficient coal is added to the kiln to accomplish the two-step reduction that is required. The coal which is not utilized in prereduction or burned in the kiln is coked in the kiln and passed with the prereduced material for use as a preheated reductant in the electric furnace.

To save thermal and electrical energy, the prereduced ore from the kiln is charged directly to the smelting zone of the electric furnace through insulated feed control bins and controlled feeders. An open bath technique is employed with the electrodes from one-half inch above the molten slag to three inches submerged in the slag, depending on the needs of the particular operation. The charge material enters the periphery of the furnace and forms its own angle of repose. Thus, the space above the electrodes is open and free flow of carbon monoxide gas produced by the reduction reactions is possible. The molten bath furnishes a constant electrical resistance, thus permitting high and uniform power inputs. This charging technique permits use of fines without interference to operation. The reaction between the coked coal and the iron oxides is extremely rapid, resulting in fast liberation of carbon monoxide, which is collected and burned in the kiln to supply a major portion of the heat required there.

The balance of the operation is similar to conventional smelting, with the iron and slag being tapped intermittently.
Silica and titanium are removed to a high degree and it is claimed that selective removal of manganese, chromium, cobalt, nickel, phosphorous, and sulphur are possible. Recovery of iron up to 95% is claimed, although recovery may be decreased by possible increases in slag volume.

Like the Dwight-Lloyd-McWane Process, the Strategic-Udy uses a combination of metallurgical equipment, each of which is established and well-proven. It is of particular interest for the production of foundry iron and semi-steel. The process can make low-carbon hot metal available, thus shortening the time of making a heat of steel in the open hearth or electric finishing furnace. Similarly, hot metal of controlled carbon content can be supplied for bessemer or basic oxygen steel making.

5. CRITIQUE OF ROTARY KILN PROCESSES

In the four preceding processes, Krupp-Renn Direct Iron Process, R-N Direct Iron Process, Orcarb Process, Strategic-Udy Process, one or more rotary kilns is used in the pre-reduction step in each process. Although rotary kilns are more established in the direct iron ore reduction field than are fluidized bed vessels, there are still some problems yet unsolved though some sources of information state that rotary kilns exhibit no problems on pilot plant scale.

The most prominent deficiency in rotary kilns is the formation of rings or crusts of the hot prereduction mix on the refractory-lined walls of the kiln which necessitates a process shutdown for relining of the walls or repair of the
refractory. Since the charge in the kiln is made up of separate entities of ore, coal, and flux, there is an inherent characteristic of possible segregation of the charge upon introduction into the smelting furnace.

In the D-LM Process, the coal, iron ore, and flux are finely divided and intimately mixed in the form of pellets and after prereduction, each individual pellet is self-fluxing and self-reducing which eliminates the possibility of segregation upon charging the prereduced material into the electric smelting furnace.

Other than the unique feature of intimately mixed pellets, the D-LM Process utilizes equipment which has been tried and proven in the Metallurgical and Mineral Dressing fields. The basic equipment involved is ball mills for grinding ore, coal, and flux, vacuum disc filters which extract water from the slurry in the grinding circuit and form a filter cake, concave disc pelletizers which form the filter cake into spherical pellets, the travelling grate for drying and prereducing the pellets, and finally the submerged arc electric furnace for smelting.

C. DWIGHT-LLOYD McWANE PROCESS

The Dwight-Lloyd McWane Process (FIGURES 10 and 11) was developed for McWane Cast Iron Pipe Company by the Dwight-Lloyd Research Laboratories in Cleveland, Ohio.

The initial steps of the process are proportioning and blending of iron ore, coal, and flux. Proportioning is continuously achieved by weighing feeders which stratify the
Iron Ore Coal Flux

Proportioning

Grinding

Filtering

Balling

Carbonizing

General Requirements

(Per ton of metal)

- Iron Ore: 1.4-1.9 tons
- Coal: 0.6-0.9 tons
- Flux: 0.2-0.6 tons
- Electrodes: 4-10 lbs
- Electricity: 750-1000 Kwh

FIGURE 10.—THE D-LM PROCESS
FIGURE 11.--DRAFT FLOW DIAGRAM OF TRAVELING GRATE SYSTEM
ingredients on a common conveyor and primary blending is accomplished by the transfer of the materials. Proportions are predesigned from analytical determinations and metallurgical calculations for making target grades of slag and metal. Feed-back data from carbonizing and smelting operations provide continuous correction of proportions. Rough proportions of raw materials are 60% ore, 30% coal, and 10% flux.

Wet grinding and filtration are performed by ball milling and vacuum filtration operations. These operations provide a satisfactory mesh of grind and residual moisture content for green ball formation, and a uniform high specific surface of the intermixed minerals for the solid-solid reactions of carbonizing. Size analyses of some of the different blends which have been favorably balled and carbonized are shown in TABLE 1.

Balling and carbonizing are carried out by disc pelletizing followed by Dwight-Lloyd travelling grate processing for drying and high-temperature firing of the green balls. Relatively large-sized green pellets on the order of one inch in diameter are favored for deep bed carbonizing operations. Flat disc slopes at high rotational speeds and carefully adjusted feed locations are required for production of the large-sized green pellets by an open circuit balling arrangement. The large green pellets have relatively low density when contrasted to normal iron pellets and can be transported and handled without unusual difficulty. The green pellets are formed with about 13% moisture and do not require addition of
TABLE 1
SCREEN ANALYSES OF VARIOUS BURDEN BLENDS TREATED BY BALLING AND CARBONIZING IN PILOT PLANT CIRCUIT

<table>
<thead>
<tr>
<th>MESH</th>
<th>BLEND A</th>
<th>BLEND B</th>
<th>BLEND C</th>
</tr>
</thead>
<tbody>
<tr>
<td>+14M</td>
<td>0.0</td>
<td>0.3</td>
<td>0.0</td>
</tr>
<tr>
<td>20</td>
<td>1.4</td>
<td>0.8</td>
<td>0.0</td>
</tr>
<tr>
<td>28</td>
<td>2.4</td>
<td>1.1</td>
<td>1.2</td>
</tr>
<tr>
<td>35</td>
<td>4.7</td>
<td>3.0</td>
<td>1.6</td>
</tr>
<tr>
<td>48</td>
<td>8.1</td>
<td>5.7</td>
<td>2.4</td>
</tr>
<tr>
<td>65</td>
<td>16.1</td>
<td>10.7</td>
<td>3.8</td>
</tr>
<tr>
<td>100</td>
<td>33.3</td>
<td>21.6</td>
<td>9.0</td>
</tr>
<tr>
<td>150</td>
<td>47.1</td>
<td>31.1</td>
<td>25.8</td>
</tr>
<tr>
<td>200</td>
<td>59.1</td>
<td>42.5</td>
<td>36.1</td>
</tr>
<tr>
<td>325</td>
<td>65.3</td>
<td>58.3</td>
<td>52.1</td>
</tr>
<tr>
<td>-325M</td>
<td>34.7</td>
<td>41.7</td>
<td>47.9</td>
</tr>
</tbody>
</table>
a separate binding agent. Usually the clay constituents of coal provide sufficient binding properties for the green pellets as can be noted from the green pellet properties shown in TABLE 2.

High temperature conversion of green pellets to hot carbonized pellets is performed by a travelling grate processing system. Warm recycle gases dry the green pellets initially in a drying zone and a high temperature induced draft fires the pellets in a carbonizing zone.

The primary objective of effective carbonization is obtained by heating the bed in stages to a temperature above 2000°F. with a draft of controlled composition. The complete carbonizing system requires close integration of the generated and used process gases throughout the length of the carbonizing machine. Control of the bed zone temperatures and oxidation levels has a strong influence on the chemical and physical qualities of carbonized pellets.

Coal of the pellet matrix initiates primary reactions of carbonizing during the heating operation. Cracking of coal provides many reactants including carbon, hydrogen, hydrocarbons, amines, water, and oxides of carbon. These co-react with oxygen, iron ore, and limestone at elevated temperature to cause the following major reactions coincidental with pyrolysis of coal and combustion of hydrocarbons.
TABLE 2
GREEN PELLET PROPERTIES

<table>
<thead>
<tr>
<th></th>
<th>CARBONIZED PELLET PROCESS</th>
<th>OXIDIZED PELLET PROCESS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size</td>
<td>-1&quot; + 3/4&quot;</td>
<td>64-82%</td>
</tr>
<tr>
<td></td>
<td>-1/2&quot; + 3/8&quot;</td>
<td>0-1%</td>
</tr>
<tr>
<td>Moisture</td>
<td>% H$_2$O</td>
<td>12.3-14.7%</td>
</tr>
<tr>
<td>Crush load</td>
<td>Lbs.--wet</td>
<td>7.0-9.0 lbs.</td>
</tr>
<tr>
<td></td>
<td>Lbs.--dry</td>
<td>6.0-12.0 lbs.</td>
</tr>
<tr>
<td>Impact</td>
<td>No.--12&quot;--wet</td>
<td>4-5</td>
</tr>
</tbody>
</table>
\[
\begin{align*}
C+O_2 &= CO_2 \\
2C+O_2 &= 2CO \\
C+CO_2 &= 2CO \\
CaCO_3 &= CO_2 + CaO \\
2Fe_2O_3 + C &= CO_2 + 4FeO \\
Fe_2O_3 + 3C + CO &= 3CO + 2Fe + CO_2 + FeO \\
2Fe_2O_3 + H_2 + CO &= H_2O + 2FeO + CO_2 + Fe \\
Fe_2O_3 + H_2 &= H_2O + Fe
\end{align*}
\]

The principal chemical beneficitation of the pellets is brought about by devolatilizing coal and limestone and deoxidizing ore. Analyses of chemical and materials balance show significant elimination of sulphur and phosphorous probably due to localized pyrolysis and hydrogenation of sulphur as well as reduction and volatilization of phosphates.

Carbonized pellets are self-fluxing and self-reducing in composition. Basic and acid constituents comprise a basicity of one, and the carbon to oxygen ratio is sufficient for complete reaction to the target grade of metal. The composition of carbonized pellets varies widely with the grades of raw materials used for production. Ranges of various constituents in some of the different carbonized pellets treated in the D-LM process are shown in TABLE 3. Continuous samples are obtained during charging of the furnace storage bin to provide periodic determinations of the physical and chemical properties of pellets as quality control of the intermediate
TABLE 3
RANGE OF CONSTITUENTS AND RESULTS FROM VARIOUS QUALITIES OF CARBONIZED PELLETS TREATED IN D-LM PROCESS

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Lower Range Data</th>
<th>Upper Range Data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe total</td>
<td>49.8%</td>
<td>61.0%</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>1.8</td>
<td>3.0</td>
</tr>
<tr>
<td>FeO</td>
<td>39.1</td>
<td>44.7</td>
</tr>
<tr>
<td>Fe$_{metallic}$</td>
<td>13.8</td>
<td>26.9</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>7.8</td>
<td>10.7</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>1.9</td>
<td>4.2</td>
</tr>
<tr>
<td>CaO</td>
<td>7.3</td>
<td>9.7</td>
</tr>
<tr>
<td>MgO</td>
<td>0.2</td>
<td>2.9</td>
</tr>
<tr>
<td>S</td>
<td>0.1</td>
<td>0.3</td>
</tr>
<tr>
<td>V.M.</td>
<td>0.3</td>
<td>1.5</td>
</tr>
<tr>
<td>F.C.</td>
<td>7.9</td>
<td>11.0</td>
</tr>
<tr>
<td>C/O--Ratio</td>
<td>0.8</td>
<td>0.9</td>
</tr>
<tr>
<td>O$_2$ Removal--%</td>
<td>41</td>
<td>62</td>
</tr>
<tr>
<td>Slag volume--lbs.</td>
<td>450</td>
<td>1300</td>
</tr>
<tr>
<td>Basicity ratio</td>
<td>0.8</td>
<td>1.2</td>
</tr>
</tbody>
</table>
product.

Continuous smelting in a submerged arc electric furnace is performed by allowing the "self-smelting" carbonized pellets to descend into the very high temperature zone of the furnace near the slag-burden interface. Here the hearth and liquid-state reactions take place while sustained by electro-thermal energy.

Carbonized pellets withstanding crushing loads of several hundred pounds can be made with selected raw materials and favorable conditions of processing. The general strength qualities have been determined to be satisfactory for the smelting requirements of the low shaft electric furnace. Nevertheless, all practical material handling features are designed to minimize loads and impacts upon the carbonized pellets.

A diagram illustrating a concept of the smelting furnace conditions is given in FIGURE 12. It can be noted that pre-heated pellets of approximately 1800°F. descend into the furnace and contact hotter generated gases ascending from the reaction zone of the hearth. These gases are composed largely of CO which performs indirect reduction as shaft reactions.

After heat transfer and shaft reactions, the pellets enter the very high temperature zone of the crucible which liquefies them into droplets while completing the smelting reactions to form slag and high carbon molten iron. The principal endothermic hearth reactions are the following:
FIGURE 12.—SECTION AND THERMAL GRADIENT FOR SUBMERGED ARC SMELTING OF D-LM PELLETS
FeO + C = CO + Fe \hspace{1cm} \Delta H = 67,151 \text{ cal.}
SiO_2 + 2C = 2CO + Si \hspace{1cm} \Delta H = 274,102 \text{ cal.}
3Fe + C = Fe_3C \hspace{1cm} \Delta H = 10,764 \text{ cal.}
FeS + CaO + C = CO + CaS + Fe \hspace{1cm} \Delta H = 59,251 \text{ cal.}

Liquid iron and slag at a temperature of about 2600°F. are accumulated at the furnace hearth where they are periodically tapped while the furnace is maintained under full load. Slag is skimmed from the flowing metal in a runner while the iron is directed to a holding-shaping arc furnace.

A holding and shaping operation is used for adjusting the composition of hot metal to a specific custom-made grade. Though reasonable uniform composition of metal can be maintained from the smelting furnace, a very narrow limit of a specified grade requires final adjustment. The arc furnace for holding and shaping is used for making these adjustments between tapping periods. These are made by temperature control of the holding-shaping furnace and metallurgical additions of ferroalloys, cold metal, and oxidants as well as changes of slags.

Due to the high purity of the metal produced by the D-LM process, it is especially suited for the casting of ductile iron. In the production of ductile iron, the presence of minute amounts of minor elements have major effects on the graphite and matrix structures. By minimizing the presence of trace elements in D-LM metal, the difficulty of controlling ductile iron castings is reduced drastically. TABLE 4 compares the trace element content of D-LM metal with other
### Table 4

**Trace Element Content of Typical Pig Irons and Direct Reduction Metals Used for Ductile Iron Production**

<table>
<thead>
<tr>
<th>Type Metal</th>
<th>D-LM Metal</th>
<th>Blast Furnace Pig Iron &quot;C&quot;*</th>
<th>Blast Furnace Pig Iron &quot;D&quot;*</th>
<th>Direct Reduction Metal &quot;S&quot;*</th>
<th>Blast Furnace Pig Iron &quot;T&quot;*</th>
<th>Blast Furnace Pig Iron &quot;U&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum, %</td>
<td>&lt;0.001</td>
<td>0.010</td>
<td>0.010</td>
<td>0.015</td>
<td>0.010</td>
<td>0.009</td>
</tr>
<tr>
<td>Antimony, %</td>
<td>N.D.</td>
<td>0.001</td>
<td>0.001</td>
<td>&lt;0.001</td>
<td>0.001</td>
<td>N.D.</td>
</tr>
<tr>
<td>Arsenic, %</td>
<td>N.D.</td>
<td>0.010</td>
<td>0.022</td>
<td>0.001</td>
<td>0.010</td>
<td>0.004</td>
</tr>
<tr>
<td>Boron, %</td>
<td>&lt;0.0005</td>
<td>&lt;0.0005</td>
<td>0.0005</td>
<td>&lt;0.0005</td>
<td>&lt;0.0005</td>
<td>&lt;0.0005</td>
</tr>
<tr>
<td>Chromium, %</td>
<td>0.011</td>
<td>0.020</td>
<td>0.020</td>
<td>0.018</td>
<td>0.025</td>
<td>0.050</td>
</tr>
<tr>
<td>Cobalt, %</td>
<td>N.D.</td>
<td>0.015</td>
<td>0.060</td>
<td>0.060</td>
<td>0.015</td>
<td>N.D.</td>
</tr>
<tr>
<td>Copper, %</td>
<td>N.D.</td>
<td>0.030</td>
<td>0.150</td>
<td>0.040</td>
<td>0.040</td>
<td>0.081</td>
</tr>
<tr>
<td>Lead, %</td>
<td>N.D.</td>
<td>0.001</td>
<td>0.001</td>
<td>0.001</td>
<td>0.001</td>
<td>N.D.</td>
</tr>
<tr>
<td>Molybdenum, %</td>
<td>0.003</td>
<td>0.005</td>
<td>0.005</td>
<td>0.001</td>
<td>0.005</td>
<td>0.004</td>
</tr>
<tr>
<td>Nickel, %</td>
<td>N.D.</td>
<td>0.003</td>
<td>0.005</td>
<td>0.085</td>
<td>0.005</td>
<td>0.019</td>
</tr>
<tr>
<td>Tin, %</td>
<td>0.005</td>
<td>0.003</td>
<td>0.003</td>
<td>0.001</td>
<td>0.005</td>
<td>0.006</td>
</tr>
<tr>
<td>Titanium, %</td>
<td>0.015</td>
<td>0.108</td>
<td>0.050</td>
<td>0.018</td>
<td>0.030</td>
<td>0.185</td>
</tr>
<tr>
<td>Vanadium, %</td>
<td>&lt;0.001</td>
<td>0.047</td>
<td>0.010</td>
<td>0.020</td>
<td>0.040</td>
<td>0.049</td>
</tr>
<tr>
<td>Total trace elements, %</td>
<td>0.0365</td>
<td>0.2535</td>
<td>0.3375</td>
<td>0.2615</td>
<td>0.1875</td>
<td>0.4075</td>
</tr>
</tbody>
</table>

N.D. = Not detected spectrographically.

direct reduction metals and blast furnace pig irons. The total trace element content of D-LM metal is shown to be only a fraction of that contained in other metals currently used for ductile iron castings.

Ductile iron castings produced from pilot plant D-LM metal were found to have a desirable graphite and matrix structure when treated with magnesium ferrosilicon. FIGURE 13 shows the typical as-cast ductile iron microstructure of such a casting.

Because of its flexibility and versatility and due to the rapidity of the metallurgical reactions, the D-LM process has tremendous potential for the production of gray and ductile iron castings, and hot metal for steel-making and ferro-alloy production.

Medium to large foundries such as automotive and pipe foundries can use a D-LM unit as the continuous source of molten metal for producing such castings. This makes possible the shipment of finished products directly from iron ore or coal mining areas.
FIGURE 13.--DUCTILE IRON MICROSTRUCTURE
III. DEVELOPMENT OF A BENCH-SCALE TEST

A. NEED FOR A BENCH-SCALE TEST

Since the commercialization in May, 1969, of the D-LM Process by McWane Iron Company in Theodore, Alabama, certain problems concerning the quality of carbonized pellets to be used as charge material for a submerged arc smelting furnace have been encountered. These problems concern mainly raw materials quality and quantity, and temperature and atmosphere control in the high-temperature zone utilized in prereducing the oxides of iron, i.e., carbonizing.

Low values of oxygen removal during prereduction increases the work load for the electric smelting furnace. Low pellet crush strengths present problems in electric smelting of the self-fluxing, self-reducing pellets due to fines created in handling and charging. Fines, in turn, produce a highly electrical resistant load detrimental to effective electrical smelting.

When the above mentioned problems are detected, especially low pellet crush strengths, the carbonized pellets entering the electric smelting furnace must be diverted since proper smelting is impossible. Usually raw material corrections have to be made which necessitates changing the filter cake composition in the initial phase of the process and allowing sufficient time for the change to be reflected in
the carbonized pellets (sometimes as long as two to three hours operating time). This last (non-productive) operating time can be eliminated by the use of a bench-scale test which can predict the properties of a particular filter cake mix before balling and carbonizing take place.

B. PARAMETERS THAT NEED TO BE MONITORED

In developing a bench-scale test to predict the properties of filter cake, certain parameters must be monitored closely in order to isolate variables related to carbonizing temperature and filter cake composition.

To relate such properties as percent prereduction of carbonized pellets (oxygen removal), carbonized pellet crush strength, and carbonized pellet electrical resistance, to carbonizing temperature as a variable, such parameters as exposure time in the carbonizing zone, reaction atmosphere composition, initial compaction of the green pellet, green pellet size and shape, and filter cake composition must be monitored closely and held as near constant values as possible.

Likewise, to relate percent prereduction, crush strength, and electrical resistance to filter cake composition as a variable (i.e., % C in the filter cake), the parameters of exposure time in the carbonizing zone, reaction atmosphere composition, initial compaction of the green pellet, green pellet size and shape, and the temperature in the carbonizing zone must be closely monitored and held as near constant values as possible.
C. EXPERIMENTAL APPARATUS AND PROCEDURE

1. BENCH-SCALE CARBONIZING APPARATUS

FIGURE 14 depicts the apparatus utilized as a carbonizing system during research in an effort to obtain a parallel for the commercial carbonizing system. It consists mainly of a Lindberg tube furnace to provide the required temperature for reaction, a Leeds and Northrup potentiometer to provide accurate temperature control of the tube furnace, and a commercially pure nitrogen source to provide an inert atmosphere for the pellet during processing.

A needle valve and a gas flow meter serve to maintain the nitrogen flow at three cubic feet per hour. The composition of the thermocouple in the carbonizing zone is platinum-platinum + 10% rhodium. The tube furnace temperature was set by adjusting a rheostat built into the furnace unit.

The fact that commercially pure nitrogen was used as a pellet atmosphere in the bench-scale operation is the outstanding difference between the bench-scale and commercial operations. This was done to eliminate a number of variables inherent in the commercial process, in which air and combustion gases from the pellets make up the atmosphere in the carbonizing zone. Via the nitrogen atmosphere, it is possible to isolate and study variables related to temperature and composition.

2. PELLET PREPARATION APPARATUS

FIGURE 15 shows the apparatus which converts filter cake, from the actual plant grinding circuit, to green
FIGURE 14.--BENCH-SCALE CARBONIZING APPARATUS
FIGURE 15.--PELLET PREPARATION APPARATUS
pellets. About 10 grams of filter cake (13% moisture) is introduced into the brass tube. Then the brass rod is inserted and the filter cake is compacted with five drops of the one pound hammer. The brass tube is then rapped lightly a few times to free the newly formed green pellet from the sides of the tube, and with the brass rod the pellet is pushed from the tube. The more compact end of the pellet is nearer the brass rod, so the pellet is sized to a length of 1¼ inches by trimming away the less compact end with a sharp knife.

The pellet is then placed on a ceramic support and placed in an oven in an air atmosphere at approximately 180°F. until dry (about 30 minutes).

3. PROCEDURE IN STUDYING THE TEMPERATURE VARIABLE

In studying the temperature variable, green pellet composition was constant (TABLE 5), exposure time to carbonizing temperatures was constant, and the reaction atmosphere was made uniformly inert in all pellet tests by using commercially pure nitrogen.

Preliminary tests were performed at constant temperature and composition to determine the exposure time to carbonizing temperatures at which maximum prereduction was reached. Results of these tests are shown in APPENDICES 1 and 2, and the nominal time chosen for all pellet tests was twenty minutes. This twenty minutes at carbonizing temperatures also approximates the actual time that pellets are exposed to high temperatures in the carbonizing zone in the commercial plant.
TABLE 5
GREEN PELLET ANALYSIS

*% $C_t = 21.75$
% $CO_2 = 5.20$
% $Fe_t = 39.51$

*Note: $C_t$ includes the carbon present in $CO_2$. 
Prepared and dried pellets were placed in the preheat zone of the tube at approximately 450°F. for five minutes. Here the pellets were preheated as they are in the actual drying hood in the plant. Then the pellets were moved into the carbonizing zone at various temperatures for twenty minutes. After carbonizing, the pellets were moved into the cooling zone of the tube and cooled until hand warm. This reduction in temperature in the inert atmosphere prevents oxidation of the pellets when stored in air. The pellets were contained in a desiccator until prepared for chemical or physical analysis.

4. PROCEDURE IN STUDYING THE CARBON VARIABLE.

To study the effects of varying amounts of carbon in the green pellets, the constant temperature of 2000°F. was chosen for carbonizing. This choice was governed by the study of the temperature variable and deemed the most suitable since a very desirable combination of three important properties was obtained at this temperature.

Time in the carbonizing zone was twenty minutes and the flow of nitrogen through the tube furnace was three cubic feet per hour.

Other processing details pertinent to the bench-scale carbonizing operation were exactly the same in the carbon study as they were in the study of the temperature variable.

Percent carbon in the filter cake was varied by adding raw materials, excluding coal, to filter cake from the plant.
(about 20% carbon) to lower the carbon percent, and by adding coal to raise the carbon percent. The additive raw material mix used to lower carbon was prepared by drying and grinding ore, oyster shells, and sand individually and then mixing them by weight in the following ratio: 62 units of ore, 10 units of oyster shell (CaCO$_3$) and 2 units of sand. Then by alternate screening and grinding the proper mesh size distribution was obtained (APPENDIX 4).

5. PROCEDURE IN DETERMINING PERCENT PREREDUCTION

APPENDIX 3 shows the method used to obtain the percent prereduction of carbonized pellets.

In preparation for the computation of percent prereduction, the carbonized pellets were crushed and ground to minus 200 mesh (Tyler Screen Series) using an agate mortar and pestle. It is necessary that the sample be ground very fine to insure complete solution during chemical analysis.

The pellets were then analyzed chemically for ferrous iron (Fe$^{++}$$_{\text{anal.}}$), metallic iron (Fe$^0$), and total iron (Fe$_t$). Since the chemical analysis used for determining ferrous iron (Fe$^{++}$$_{\text{anal.}}$) includes metallic iron (Fe$^0$), it is necessary to subtract the metallic iron result from the ferrous iron result to obtain the actual percentage ferrous iron (Fe$^{++}$$_{\text{actual}}$) present in the sample.

6. PROCEDURE IN DETERMINING PELLET CRUSH STRENGTH

The pellet crush strengths were measured using a soil-test stability testing machine, which utilizes a standardized
proving ring and a dial indicator to measure deflection of the proving ring. Deflections were converted to pounds of total force required to crush the pellets loaded in compression in accordance with the conversion table supplied with the standardized proving ring. The cylindrical pellets were not subjected to an axial load, but to a load that was perpendicular to the central axis of the cylinder. Axial loading was not utilized due to an inability to obtain a proper contact between the small end surfaces of the cylindrical pellets and the loading faces of the testing equipment.

7. **PROCEDURE IN DETERMINING THE ELECTRICAL RESISTANCE OF PELLETS**

Resistance measurements of carbonized pellets were attempted using copper caps as contacts on the ends of the cylindrical pellets. Measurements taken thusly were very erratic due to inadequate contact between the copper caps and the coarse-grained surface of the pellets.

To obtain a smoother, more adequate contact surface, the pellets were crushed and ground in an agate mortar and pestle to minus 200 mesh (Tyler Screen Series). With the aid of 5 percent by weight boric acid powder as a binding agent, the crushed and ground samples were pressed under 77,000 p.s.i. into discs measuring one inch in diameter by one-eighth inch thick. Four resistance measurements were taken on the circular surface of each disc across the diameter of an inscribed concentric circle one-half inch in diameter at 45 degree intervals. These measurements provided consistent results
for each sample, and the four measurements were averaged to obtain a resultant resistance measurement.

D. RESULTS AND DISCUSSION

1. THE EFFECT OF TEMPERATURE ON PERCENT PREREDUCTION

Percent prereduction was calculated for samples of constant composition carbonized at various temperatures; the results are displayed graphically in FIGURE 16 and shown in tabular form in TABLE 6.

From the graph it can be noted from the slope of the curve that above 2000°F., increases in percent prereduction, with unit increases in temperature, are not as great as those corresponding increases below 2000°F. This tends to indicate that the maximum efficient operating temperature is approximately 2000°F. which correlates with the established operating temperature in the plant carbonizing zone.

2. THE EFFECT OF TEMPERATURE ON PELLET CRUSH STRENGTH

Results of pellet crush strength tests performed on samples carbonized at various temperatures are depicted in FIGURE 17. The graph shows that maximum crush strength is obtained at approximately 1800°F. and that this maximum strength is maintained well above the operating temperature of 2000°F. Therefore, if pellets are carbonized at the plant's operating temperature, it is assured that all pellets have obtained maximum crush strength.
FIGURE 16.--THE EFFECT OF TEMPERATURE ON PERCENT PREREDUCTION
**TABLE 6**

ANALYSES OF CARBONIZED PELLETS USED IN TESTING

THE EFFECT OF TEMPERATURE ON PER CENT PREREDUCTION

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Carb. Zone Temp.</th>
<th>%Fe$^{++}$&lt;sub&gt;anal&lt;/sub&gt;</th>
<th>%Fe$^{O}$</th>
<th>%Fe$^{++}$&lt;sub&gt;actual&lt;/sub&gt;</th>
<th>%Fet</th>
<th>%IO$_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1625°F</td>
<td>45.34</td>
<td>2.59</td>
<td>42.75</td>
<td>45.50</td>
<td>36.99</td>
</tr>
<tr>
<td>2</td>
<td>1785°F</td>
<td>50.04</td>
<td>17.81</td>
<td>32.23</td>
<td>50.36</td>
<td>56.67</td>
</tr>
<tr>
<td>3</td>
<td>1930°F</td>
<td>53.92</td>
<td>35.62</td>
<td>18.30</td>
<td>53.92</td>
<td>77.35</td>
</tr>
<tr>
<td>4</td>
<td>2000°F</td>
<td>58.30</td>
<td>45.98</td>
<td>12.32</td>
<td>59.59</td>
<td>83.97</td>
</tr>
<tr>
<td>5</td>
<td>2108°F</td>
<td>58.62</td>
<td>53.44</td>
<td>5.18</td>
<td>59.75</td>
<td>92.30</td>
</tr>
<tr>
<td>6</td>
<td>2175°F</td>
<td>60.40</td>
<td>55.71</td>
<td>4.69</td>
<td>60.89</td>
<td>94.05</td>
</tr>
<tr>
<td>7</td>
<td>2217°F</td>
<td>60.24</td>
<td>55.06</td>
<td>5.18</td>
<td>60.24</td>
<td>94.26</td>
</tr>
<tr>
<td>8</td>
<td>2322°F</td>
<td>60.73</td>
<td>58.29</td>
<td>2.44</td>
<td>61.37</td>
<td>96.30</td>
</tr>
<tr>
<td>9</td>
<td>2395°F</td>
<td>60.72</td>
<td>59.27</td>
<td>1.45</td>
<td>61.05</td>
<td>97.87</td>
</tr>
</tbody>
</table>
FIGURE 17. -- THE EFFECT OF TEMPERATURE ON CRUSH STRENGTH
3. THE EFFECT OF TEMPERATURE ON THE ELECTRICAL RESISTANCE OF PELLETS

Results of resistance measurements taken on samples carbonized at various temperatures are shown in FIGURE 18. The resistance decreases as temperature increases due to the increase in reduction of iron oxides to metallic iron. A minimum resistance is approached in the temperature range of 1950-2000°F.; beyond this range, decreases in resistance with unit increases in temperature are very small.

4. THE EFFECT OF CARBON ON PERCENT PREREDUCTION

Percent prereduction was calculated for samples of varying percentages of carbon which were carbonized at 2000°F. The results are depicted graphically in FIGURE 19 and shown in tabular form in TABLE 7. TABLE 8 shows the filter cake analysis for each green pellet sample.

It can be seen from FIGURE 19 that maximum prereduction is obtained when 20 percent carbon is present in the green pellet. Less carbon is inadequate to remove the required oxygen from the iron oxide for maximum prereduction to occur. Percent prereduction remains constant after 20 percent carbon due to the constant temperature of 2000°F. At higher temperatures the prereduction would approach 100 percent.

5. THE EFFECT OF CARBON ON THE CRUSH STRENGTH OF PELLETS

Results of crush strength tests are illustrated in FIGURE 20. TABLE 9 lists the filter cake analysis for each green pellet tested.
FIGURE 18.--THE EFFECT OF TEMPERATURE ON RESISTANCE
FIGURE 19.—THE EFFECT OF CARBON ON PERCENT PREREDUCTION
TABLE 7
ANALYSES OF CARBONIZED PELLETS USED IN TESTING
THE EFFECT OF CARBON ON PER CENT PREREDUCTION

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>$%\text{Fe}^{++}\text{anal}$</th>
<th>$%\text{Fe}^0$</th>
<th>$%\text{Fe}^{++}\text{actual}$</th>
<th>$%\text{Fe}_t$</th>
<th>$%\text{IO}_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>62.51</td>
<td>10.36</td>
<td>52.15</td>
<td>62.67</td>
<td>44.25</td>
</tr>
<tr>
<td>2</td>
<td>64.61</td>
<td>43.07</td>
<td>21.54</td>
<td>64.94</td>
<td>77.36</td>
</tr>
<tr>
<td>3</td>
<td>59.75</td>
<td>47.29</td>
<td>12.46</td>
<td>59.75</td>
<td>86.09</td>
</tr>
<tr>
<td>4</td>
<td>54.57</td>
<td>44.69</td>
<td>9.88</td>
<td>53.38</td>
<td>86.64</td>
</tr>
<tr>
<td>5</td>
<td>48.90</td>
<td>39.51</td>
<td>9.39</td>
<td>49.88</td>
<td>85.49</td>
</tr>
</tbody>
</table>
TABLE 8
ANALYSES OF FILTER CAKE USED IN TESTING THE EFFECT OF CARBON ON PER CENT PREREDUCTION

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>$%C_t$</th>
<th>$%CO_2$</th>
<th>$%Fe_t$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.00</td>
<td>5.87</td>
<td>51.48</td>
</tr>
<tr>
<td>2</td>
<td>14.13</td>
<td>5.50</td>
<td>46.44</td>
</tr>
<tr>
<td>3</td>
<td>21.75</td>
<td>5.20</td>
<td>39.51</td>
</tr>
<tr>
<td>4</td>
<td>25.26</td>
<td>4.80</td>
<td>35.42</td>
</tr>
<tr>
<td>5</td>
<td>30.33</td>
<td>4.29</td>
<td>33.22</td>
</tr>
</tbody>
</table>
FIGURE 20.--THE EFFECT OF CARBON ON CRUSH STRENGTH
### TABLE 9

**ANALYSES OF FILTER CAKE USED IN TESTING THE EFFECT OF CARBON ON PELLET CRUSH STRENGTH**

<table>
<thead>
<tr>
<th>Sample No.</th>
<th><em>%C_t</em></th>
<th>%CO₂</th>
<th>%Fe_t</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>14.37</td>
<td>5.20</td>
<td>45.18</td>
</tr>
<tr>
<td>2</td>
<td>21.44</td>
<td>4.67</td>
<td>41.72</td>
</tr>
<tr>
<td>3</td>
<td>26.51</td>
<td>4.23</td>
<td>36.52</td>
</tr>
<tr>
<td>4</td>
<td>34.00</td>
<td>3.60</td>
<td>31.80</td>
</tr>
</tbody>
</table>

*Note: C_t includes the carbon present in CO₂.*
The graph in FIGURE 20 shows that very high crush strengths occur at higher percentages of carbon. It can be inferred that the coal in the green pellet is responsible for the crush strength since the carbon is provided by coal additions.

The particular coal used in these tests initially exhibits its characteristic plasticity, or fluidity, at 745°F\textsuperscript{16}. At 810°F\textsuperscript{16}, maximum fluidity is achieved and the fluid coal enters, by capillary action, the void spaces among the constituent grains of raw materials within the pellet. The end of coal fluidity and the beginning of pellet bonding occurs at 860°F\textsuperscript{16}, after volatile matter is driven from the coal and coking, or charring, of the coal takes place. Thus the coke bond, or "char bond,"\textsuperscript{14} is responsible for the crush strengths achieved in pellets and increasing percentages of coal (carbon) provide corresponding increases in pellet crush strengths.

6. THE EFFECT OF CARBON ON THE ELECTRICAL RESISTANCE OF PELLETS

Results of the resistance measurements are represented in FIGURE 21. Filter cake analyses of the green pellets tested are shown in TABLE 10.

The graph in FIGURE 21 indicates very high resistance and drastic increases in resistance as the carbon decreases below 15 percent. Decreases in resistance are caused simultaneously by two mechanisms: (1) increasing percentages of conductive metallic iron as a result of increasing
FIGURE 21.—THE EFFECT OF CARBON ON RESISTANCE
TABLE 10
ANALYSES OF FILTER CAKE USED IN TESTING THE EFFECT OF CARBON ON ELECTRICAL RESISTANCE OF PELLETS

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>*%C_t</th>
<th>%CO_2</th>
<th>%Fe_t</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15.93</td>
<td>5.60</td>
<td>45.44</td>
</tr>
<tr>
<td>2</td>
<td>21.18</td>
<td>5.22</td>
<td>40.51</td>
</tr>
<tr>
<td>3</td>
<td>32.19</td>
<td>4.10</td>
<td>32.22</td>
</tr>
</tbody>
</table>

*Note: C_\text{t} includes the carbon present in CO_2.*
prereduction, (2) increasing amounts of conductive carbon present in the pellet. Although these two mechanisms simultaneously reduce resistance, it is thought that the resistance decrease below 20 percent carbon is mostly due to increasing amounts of conductive carbon present in the pellet. This is in agreement with the results depicted in FIGURE 19 where the maximum percent prereduction is reached at approximately 20 percent carbon.
IV. CONCLUSION

It is concluded from the foregoing research and discussion that physical, chemical, and electrical properties of carbonized pellets are definitely predictable using a bench-scale carbonizing apparatus. It should be noted here, however, that only maximum values and general trends in the research performed were compared to values in the actual plant, and it is, indeed, only these maximums and trends that actually conform. This is thought to be true because of the difference in atmospheric conditions in the reaction zone—the inert nitrogen atmosphere in the bench-scale carbonizing, and the air and combustion gases in the plant carbonizing. But the mere fact that maximum values can be assumed correct and that the general trends do exist, provides basis for a "tailor-made" bench-scale test that also agrees with plant results numerically. Even more support for agreement between bench-scale and plant can be derived from the fact that even though the plant reaction atmosphere differs in composition from the bench-scale, it is virtually constant in composition.

For example, in bench tests relating carbonizing zone temperature and prereduction of pellets, maximum efficient prereduction was obtained at approximately 2000°F. The percent prereduction at this temperature is 83.97 which is very much higher than the average plant prereduction range of
52-57 percent. By either increasing pellet size or decreasing exposure time in the carbonizing zone, results obtained on a bench-scale could be made to correspond more closely with plant operations. It is believed that the entire curve in FIGURE 16 could be lowered on the prereduction scale to indicate a maximum prereduction of 52-57 percent at 2000°F. APPENDIX 1 tends to indicate that increasing pellet size rather than decreasing exposure time would be the better choice because exposure time in the range of 52-57 percent prereduction is very critical.

In studying the temperature effects on the crush strength of pellets, FIGURE 17 shows maximum crush strengths of 10 pounds at about 1800°F. Actual crush strengths for D-LM pellets are in the range of 80-100 pounds which is 8 to 10 times the maximum crush strengths obtained with bench-scale tests. With modifications similar to those concerning prereduction, crush strengths obtained on a bench-scale can approach the actual crush strengths of D-LM pellets. The most probable method to obtain this correlation is to increase the degree of compaction of the green pellet before carbonizing, thus producing more intimate contact among the particles of raw materials and increasing the coke bond or "char bond" formed by the fluidized and devolatilized coal. Also, increasing pellet size, or even a combination of increasing pellet size and compaction, would tend to increase crush strengths to provide the desired correlation.

It is evident, then, from the foregoing conclusions and
proposals that bench-scale apparatus cannot be utilized to establish norms and standards for the production of D-LM pellets. But bench testing can be made to conform to established values and relationships and can be an invaluable aid in their preservation when used as a quality control device in the prereduction of D-LM pellets.
APPENDIX 1.--THE EFFECT OF TIME ON PERCENT PREREDECTION
APPENDIX 2

ANALYSES OF CARBONIZED PELLETS USED IN TESTING THE EFFECT OF TIME ON PER CENT PREREDUCTION

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Time in Carb Zone</th>
<th>( %\text{Fe}^{++} \text{ anal} )</th>
<th>( %\text{Fe}^{0} )</th>
<th>( %\text{Fe}^{++} \text{ actual} )</th>
<th>( %\text{Fe}_t )</th>
<th>( %\text{IO}_2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5 min</td>
<td>50.68</td>
<td>24.29</td>
<td>26.39</td>
<td>51.50</td>
<td>64.23</td>
</tr>
<tr>
<td>2</td>
<td>10 min</td>
<td>54.90</td>
<td>43.72</td>
<td>11.18</td>
<td>55.38</td>
<td>85.61</td>
</tr>
<tr>
<td>3</td>
<td>15 min</td>
<td>56.52</td>
<td>48.58</td>
<td>7.94</td>
<td>56.84</td>
<td>90.11</td>
</tr>
<tr>
<td>4</td>
<td>20 min</td>
<td>53.92</td>
<td>48.58</td>
<td>5.34</td>
<td>53.92</td>
<td>93.40</td>
</tr>
<tr>
<td>5</td>
<td>25 min</td>
<td>55.54</td>
<td>47.93</td>
<td>7.61</td>
<td>55.54</td>
<td>90.85</td>
</tr>
<tr>
<td>6</td>
<td>30 min</td>
<td>56.52</td>
<td>50.52</td>
<td>6.00</td>
<td>57.81</td>
<td>90.85</td>
</tr>
</tbody>
</table>

FILTER CAKE ANALYSIS FOR ABOVE SAMPLES

\[ \%\text{C} = 21.75 \]

\[ \%\text{CO}_2 = 5.20 \]

\[ \%\text{Fe}_t = 39.51 \]
APPENDIX 3

PROCEDURE FOR DETERMINING PERCENT IRON REDUCTION
IN CARBONIZED PELLETS

The percent reduction is calculated on the basis of percent oxygen removed from the iron ore. Metallic iron represents total oxygen removal according to the reaction

\[ \text{Fe}_2\text{O}_3 + 3\text{C} = 3\text{CO} + 2\text{Fe}, \]

whereas ferrous iron (i.e., FeO) represents only 1/3 oxygen removal according to the reaction

\[ 2\text{Fe}_2\text{O}_3 + \text{C} = \text{CO}_2 + 4\text{FeO}. \]

Therefore, percent reduction of ore, or the index of oxygen \((\text{IO}_2)\), is given by

\[
\text{% Reduction} = \left(\frac{\text{AMOUNT OF IRON REDUCED} \times 100}{\text{TOTAL AMOUNT OF IRON}}\right)
\]

or,

\[
\text{% Reduction} = \frac{\text{Fe}^0 + \frac{1}{3}\text{Fe}^{++} \times 100}{\text{Fe}_t}
\]

where:

- \(\text{Fe}^0\) = Metallic Iron
- \(\text{Fe}^{++}\) = Ferrous Iron
- \(\text{Fe}_t\) = Total Iron

Sample calculations

#1  \(\text{Fe}^0 = 10\%\)
\(\text{Fe}^{++} = 38\%\)
\(\text{Fe}_t = 53\%\)

\[
\text{% Reduction} = \frac{10 + \frac{1}{3}(38) \times 100}{53} = 42.8
\]
#2

Fe₀ = 25.20%
Fe⁺⁺ = 34.09%
Feₜ = 60.74%

\[
\text{% Reduction} = \frac{25.20 + \frac{1}{3}(34.09) \times 100}{60.74} = 60.2
\]
## APPENDIX 4

### TYPICAL DRY FILTER CAKE SCREEN ANALYSIS

*(TYLER SCREEN SERIES)*

<table>
<thead>
<tr>
<th>Mesh</th>
<th>Weight Retained</th>
<th>Individual % Retained</th>
<th>Cumulative % Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>+ 35</td>
<td>5</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>+ 48</td>
<td>37</td>
<td>7.4</td>
<td>8.4</td>
</tr>
<tr>
<td>+ 65</td>
<td>52</td>
<td>10.4</td>
<td>18.8</td>
</tr>
<tr>
<td>+100</td>
<td>126</td>
<td>25.2</td>
<td>44.0</td>
</tr>
<tr>
<td>+150</td>
<td>130</td>
<td>26.4</td>
<td>70.4</td>
</tr>
<tr>
<td>+200</td>
<td>89</td>
<td>17.8</td>
<td>88.2</td>
</tr>
<tr>
<td>+270</td>
<td>37</td>
<td>7.4</td>
<td>95.6</td>
</tr>
<tr>
<td>+325</td>
<td>6</td>
<td>1.2</td>
<td>96.8</td>
</tr>
<tr>
<td>-325</td>
<td>18</td>
<td>3.2</td>
<td>100.4</td>
</tr>
<tr>
<td>Totals</td>
<td>500</td>
<td>100.0</td>
<td></td>
</tr>
</tbody>
</table>
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VIII. VITA

James Orlan Hood, Jr. was born on August 15, 1945, in Amory, Mississippi. He received his primary and secondary education in Birmingham, Alabama. He has received his college education from the University of Missouri-Rolla, in Rolla, Missouri. He received a Bachelor of Science Degree in Metallurgical Engineering from the University of Missouri-Rolla, in Rolla, Missouri, in August, 1968.

He has been enrolled in the Graduate School of the University of Missouri-Rolla since September, 1968. During the period June, 1969 to January, 1970 he worked at McWane Iron Company in Theodore, Alabama in conjunction with the University of Missouri-Rolla as an "Industrial Intern" to research and write his thesis for his Master of Science Degree in Metallurgical Engineering.