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DIRECT REDUCED IRON
RESEARCH AND DEVELOPMENT PROJECT

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QUARTERLY TECHNICAL PROGRESS REPORT
PHASE II CALDERON PROCESS TO PRODUCE DIRECT REDUCED IRON
RESEARCH AND DEVELOPMENT PROJECT

CALDERON ENERGY COMPANY
COOPERATIVE AGREEMENT NO. DE-FC22-95PC92638

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Project Director: Albert Calderon

Assistant Project Director: Reina Calderon

Abstract

This project was initially targeted to the making of coke for blast furnaces by using proprietary technology of Calderon in a phased approach, and Phase I was successfully completed. The project was then re-directed to the making of iron units. U.S. Steel teamed up with Calderon for a joint effort which will last 30 months to produce directly reduced iron with the potential of converting it into molten iron or steel consistent with the Roadmap recommendations of 1998 prepared by the Steel Industry in cooperation with the Department of Energy.

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Introduction

The commercialization path of the Calderon technology for making a feedstock for steelmaking with assistance from DOE initially focused on making coke and work was done which proved that the Calderon technology is capable of making good coke for hard driving blast furnaces. U.S. Steel which participated in such demonstration felt that the Calderon technology would be more meaningful in lowering the costs of making steel by adapting it to the making of iron - thus obviating the need for coke.

On page 6 of the July 2002 publication of "Iron & Steelmaker (I & SM)", which covered the International Report on steel for Europe, it was reported that the chairman of the Iron and Steel Institute and president of the German Steel Federation, Mr. Dieter Ameling made reference to the necessity of reducing CO₂. He said the following: "... that in some areas, the burden imposed by politicians has reached the breaking point. During the past year, the discussion about the reduction of CO₂ emissions has intensified. Some policies currently brought forward would deeply affect the steel industry since they would narrowly limit production and market potential to the EAF path. Eventually, oxygen steel production would have to be abandoned completely in Germany, with job losses as a consequence. Some political forces even claim it is about time to initiate a restructuring of the German industry "away from energy-intensive old economy industry sectors"."

The fact that U.S. Steel and Calderon teamed up to jointly work together to demonstrate that the Calderon technology will produce in a closed system iron units from iron concentrate (ore) and coal competitively by eliminating pelletizing, sintering, coking, blast furnace operation and possibly doing away with the BOF and the EAF by making steel directly, a huge reduction in CO₂ generation relating to steelmaking would ensue. Such

reduction will restructure the steel industry away from the very energy-intensive steelmaking steps currently practiced and which are highlighted in the attached flowline diagram.

Accomplishments and Discussion

The work performed during the past quarter for the advancement of the Calderon technology for the making of iron units for which this report is presented covered the following areas:

Item A - Improvement to Material Drying

Item B - Experimentation with Disposable Thermocouples

Item C - Oxygen Lance Practice

Item D - Improvements to Lance Construction

Item E - Collection Drum Insulation

Item F - Collection Drum Operation

Item G - Product Analysis

Item H - Entrained Dust in Off-Gas

Item I - Preparation for Integration of Process

Item A - Improvement to Material Drying

Since the ore concentrate and in some cases the coal, that is received from U.S. Steel has moisture, drying of the ore and coal mix has been a standard practice. As previously reported a drier was built which consisted of placing the mix in the a drum and rotating it over a gas fired burner (photo #1 left) until the mix is elevated to a temperature of 220°F. This approach worked but the time to dry the mix was slow in relation to the charging sequence of the mix into the reactor. It was found that the heat loss through the steel shell of the drum

was excessive thus rendering the drying operation quite slow. An insulated dog house was designed and constructed (photo #1 right and photo #2). This dog house was light enough to enable two people to handle it off and on the drier. This arrangement has worked well and the drying operation was greatly accelerated which resulted in coordinating the drying of the mix with the charging of the mix.

Item B - Experimentation with Disposable Thermocouples.

The reactor and the discharge elbow are equipped with five thermocouples (TC) which consist of three “K” , one “S” and one “B”. The “K” type which are good for continuous operation up to 2200° are used during heat up of the reactor and elbow and are withdrawn once they reach a temperature of 2150°F. The “S” and the “B” which are capable to operate up to 3000°F are used for steady state operation; these TC’s are encased in ceramic shields which cannot take mechanical abuse. They also operate erratically when deposits of metal or carbon build-up occurs on their tips. Since the accurate reading of temperature is essential at steady state at a range of 2300° to 2600° (and above in case of excursions), TC problems have been rampant including their frequent replacement.

To overcome this problem, trials were conducted with disposable TC’s similar to those used in dipping the TC into molten metal with a wand (photos #3 and #4). Unfortunately the wand and its protective insulation sleeve were overheated and destroyed during the trials. Work has begun to construct a replaceable tip TC that has a water cooled wand for high temperature continuous duty wherein the tip could be replaced in a few minutes time when failure occurs or replaced routinely prior to failure. The cost of such tip is minimal (\$1.50/tip).

Item C - Oxygen Lance Practice

The oxygen which is delivered in banks of 12 bottles per pallet (photo #5), services the horizontal lance, the vertical and the elbow ports by means of separate regulators. The vertical lance and the elbow ports have been trouble free. The horizontal lance which feeds oxygen into the core of the reactor in order to convert the coal to thermal energy, to produce reducing gases and carbon at a controlled rate was provided with a specially designed motorized lance insertion device (LID) that is adapted to travel on a track and programmed to travel with respect to depth of insertion and dwell time (photos #6, #7, and #8). A lance practice was initiated in order to enable the process to predictably produce a consistent acceptable product suitable for homogenization in a melter.

Item D - Improvement to Lance Construction

In the operation of the water cooled oxygen lances, the practice has evolved into having the vertical lance in a fixed position with its tip inserted two inches into the elbow. With respect to the horizontal lance which moves in and out of the core, the original construction consisted of the entire lance being made of copper tubing. With use it was discovered that the copper would not take the abuse within the tunnel created by the combustion of the coal core. A change was made to replace the copper tubing with stainless steel, leaving the tip of copper unchanged. It was found that cleaning the stainless steel lance of deposits was much easier but a problem developed at the joint of the copper tip and the stainless tubing (photo #9) . A change was made by increasing the length of the copper tip (photo #10) which has worked well. The build-up of deposits at the tip has been solved by increasing the injection pressure of the oxygen to maintain the discharge nozzle of the lance open (photo #11).

Item E - Collection Drum Insulation

It was agreed by U.S. Steel and Calderon that a 72 hour test of reasonably continuous operation take place. It was proposed that the material be collected in insulated drums, cooled and shipped to U.S. Steel for analysis and percent metallization, and also that a mass balance would be conducted. Three drums were insulated similar to the one shown in photograph #12. In operation, the material produced stuck to the walls of the drum (photo #13) and the way to remove it was to torch the drum (photos #14 and #15).

In an attempt to solve this problem three additional drums were lined with a composite lining. One such drum with the nitrogen injection pipe at the bottom for cooling the product, is shown in photo #16.

Item F - Collection Drum Operations

In operation, the drum was positioned under the elbow by means of a pallet lifter and raised against the discharge of the elbow to seal it. After collecting the product of the run the drum while hot was dropped and pulled out from under the hot elbow (photos #17, #18 and #19). The hot drum (photo #20) was then sealed with a cover (photo #21) and purged with nitrogen to cool it (photo #22); additional cooling was provided by pouring water on top of the cover (photo #23). To check whether the drum was under positive pressure while cooling a rubber glove was installed (photo #24) at the discharge of the drum cover and upon its inflation positive pressure within the drum was verified. Once the drum was cold, the material was dumped on a cement driveway for inspection (photo #25).

Item G - Product Analysis

In order to more fully understand the process and develop a practice wherein acceptable product would be consistently produced, a procedure for analysis was developed wherein the drum contents were discharged as a long mound (photo #26) with temperatures written in chalk at equally spaced points along the length of the mound, the temperatures being taken from the data sheets which were kept during the run itself.

Upon inspection samples were taken from the mound and sanded on a belt to indicate by means of sparking whether the material is a steel, an iron, or overoxidized or un-reduced material. In conducting runs knowledge has been accumulated regarding the material produced. Photograph #27 is a good sample. It indicates the various colors representing different temperatures at which the material was subjected to and the effects of oxygen while the material is formed. If the material is to be graded from 1 to 5 in the absence of wet chemistry, with 1 being the most sought for quality and 5 the least desired, it is believed that the silvery colored would be graded 1, the golden 2, the purple 3, the blue 4, and the gray 5. It has been discovered that the golden, the purple and the blue material when wire-brushed the silvery shine appears indicating that the hues are superficial. Since there is yet more work to be done to overcome operational problems , the goal as of this writing is to consistently produce material similar to that shown in photo #28.

To date two drums of material have been sent to U.S. Steel for analysis for percent of metallization. The results obtained regarding the first drum have shown a metallization of 64%; regarding the second drum the metallization was slightly under 80%. The goal is to obtain a consistent metallization of 80% on an average. With respect to the second drum, the data sheets for that particular run were included. Based on such data U.S. Steel did

inquire as to the discrepancy in weight of materials charged into the reactor when compared to the weight of the material in the drum. Prior to shipment the material was dumped on the concrete driveway for inspection and determination as to the suitability of the material for shipment. Once the decision was made to ship, this material was scooped from the concrete and put into a non-lined drum. It can be said that there were some losses of material during the transfer from lined drum to ground and from ground to unlined drum, but such losses were not accounted for. It is believed that the bulk of the differences in weight of material charged and material recovered consist in losses traceable to various factors including the accuracy of the scales, but those that are outstanding include the fines entrained in the off-gas which will be discussed in Item H that follows, and material that fuses to the refractory wall of the elbow and the refractory wall of the drum. Once consistency of product is obtained, optimization of the process will ensue which will include the tracking of losses of material including dust. Since the ore is quite fine (80% minus 200 mesh) it is quite easy for it to be entrained in the off gas.

Item H - Entrained Dust in Off-Gas

The tests to-date have shown that depending upon the operation, substantial amounts of dust are entrained in the off-gas. A trap has been provided downstream of the exit port of the off-gas to attempt to collect the fines; photo #29 shows the fines discharge and the pile collected. Further, still finer particles escape through the stack shown in photo #30. It is to be noted that when the rain cap of the stack is brought down for analysis (photos #31 and #32), there is evidence that dust cleanup of the off-gas in a baghouse would have to be an essential part of a commercial operation once the off-gas is cooled. Also the introduction of

the recovered dust from the baghouse into the charging system would also be imperative in order to avoid land filling and loss of iron and carbon units.

Item I - Preparation for Integration of the Process

As mentioned in the previous report that an induction furnace had been ordered from Ajax Magnethermic for homogenizing the product made in the reactor. During the period for which this report is concerned, the furnace was delivered (photo #33), and about 75% of the work relating to utilities to service the furnace and reactor has been accomplished. Miscellaneous piping and electrical conduits (photo #34), a cooling tower (photo #35) and transformers (photo #36) have been installed. A control cabinet operating at a frequency of 3000 was also put in place (photos #37 and #38).

Conclusion

As of this writing the longest run has been 18.5 hours of continuous operation. This must extend to 72 hours producing on the average a product of 80% metallization. The challenges confronting the attainment of 72 hours of reasonably continuous operation are:

- Thermocouple life in a hostile environment; and
- Sticking of material at the discharge end of the reactor during extended operation.

Once these challenges are met it is projected that consistency of product will be obtained. An all out effort to reach consistency is in place.

In addition to the operation of the reactor feeding material into the drums, the plan is to begin operating the homogenizer with cold material produced from the reactor to learn the operation of melting and slagging in preparation for the integration step which would follow, taking into consideration that the material would be hot when discharged from the reactor into

the homogenizer. At such time the problems encountered with the collection drums would be immaterial since feeding into drums is only a temporary matter prompted by the 72 hour test.

Submitted by:

Albert Calderon
Project Director

The raw materials of steelmaking must be brought together, often from hundreds of miles away, and smelted in a blast furnace to produce most of the iron that goes into steelmaking furnaces. Air and oxygen are among the most important raw materials in iron and steelmaking.

Molten steel must solidify before it can be made into finished products by the industry's rolling mills and forging presses. The metal is usually formed first at high temperature, after which it may be cold-formed into additional products.

A FLOWLINE ON STEELMAKING

This is a simplified road map through the complex world of steelmaking. Each stop along the routes from raw materials to mill products contained in this chart can itself be charted. From this overall view, one major point emerges: Many operations—involving much equipment and large numbers of men—are required to produce civilization's principal and least expensive metal.

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