Report Title:

PHASE II CALDERON PROCESS TO PRODUCE
DIRECT REDUCED IRON
RESEARCH AND DEVELOPMENT PROJECT

Report Type:

QUARTERLY

Reporting Period Start Date: 01/01/2002 End Date: 03/31/2002

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Report Issue Date: 04/25/2002

DOE Award No.: DE- FC22 -95PC92638

Submitting

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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

Reporting Period: 1-01-02 to 3-31-02

Date of Report: 4-25-02;

Phase II Award Date: 6-23-00; Anticipated Completion Date: 12-03-03

Total Project: \$ 25,814,000.00 Budget II Total DOE Share This Action: \$6,457,000.00

Contracting Officer's Representative (COR): Carl Maronde;

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.

U.S. Steel and Calderon teamed up to jointly work together to demonstrate that the Calderon technology can produce iron units from iron concentrate (ore) and coal quite competitively by eliminating pelletizing, sintering, coking and blast furnace operation. Such demonstration would also prove that, potentially, a significant reduction in pollution would occur including the minimization of greenhouse gases and specifically CO₂ by virtue of Calderon's technology being efficient and closed.

Accomplishments and Discussion

During the past quarter additional progress was made in the following areas:

- Laboratory work done to evaluate aspects of the Calderon process performed at U.S. Steel's Research and Technology Center;
- Integration of reduction with melting;
- Preparations for the extended continuous test of 72 hours at reasonably steady state for an overall mass balance performance; and
- Initial Trial Test.

Laboratory Testing of the Calderon Ironmaking Process at U.S. Steel's Tech Center

To gain a better understanding of the process, the Tech Center performed a number of tests in both the Raw Materials and Refractory laboratories. The purpose of the tests was to simulate the proposed process conditions and obtain qualitative heat and mass transfer information. All the tests were performed in a mixture of 75% Mintac (Minnesota taconite) concentrate and 25% Maple Creek coal.

The tests results indicated that the process will require a large amount of high temperature radiation heat transfer to be viable. The Tech Center did not take into account the energy contributed by the fuel in the core of the charge. The convective and conductive heat transfer are expected to have minor influence on the process.

Such test results suggest that an operation wherein a combination of energy input at the core and at the exit of the reactor (elbow) would yield the most efficient reduction. In commercial application the reactor should be of sufficient length to maximize residence time within the reactor via the coal (fuel) in the core and of a rather large diameter in order to maximize the amount of material exposed to the high temperature radiation at the exit of the reactor. The bench scale has been provided with provisions for supplying energy at the core as well as at the exit of the reactor (page 7) and has been operated in this mode.

Integration of Reduction with Melting

It has been observed that the iron/carbon material emerging out of the reactor is not uniform. Based on this fact, consideration has been given that the material discharge from the reactor needs to be homogenized and purified by a melting step wherein the iron/steel can be separated from its impurities through the formation of a slag which is lighter than the iron/steel to make possible its removal. Therefore the integration of reduction and

homogenization of the product has become a necessary task in order to produce an iron or a steel of homogeneous quality.

A 200 lbs/hr induction furnace was selected for integration to the bench scale reactor with the goal of furnishing adequate capacity once the output/hr of the bench scale is optimized. An order for the furnace was placed with Ajax Magnethermic, a company with extensive expertise in induction heating and melting. The controls cabinet and the cooling system for the furnace coil did not have to be purchased as they were retrieved from the Alliance, Ohio gasification project of Calderon. This equipment was moved to Bowling Green and will be assembled when the furnace is delivered in May.

Additional experience has been accumulated towards integration by converting iron/carbon product (feedstock) made in the reactor to something useful such as medallions that are cast from a feedstock that was exclusively made directly from concentrate and coal based exclusively. Arrangements were made with Cast Masters (a foundry in Bowling Green) to use one of its induction furnaces of approximately the same capacity as that ordered from Ajax. Attached are photographs which pictorially illustrate the steps taken to make the medallions.

The feedstock was delivered in cardboard boxes to the foundry (page 8). During the melting and working of the heat (page 9, top left) the reaction was over-active. In order to subside the reaction, carbon was added during the working of the heat (top right). The slagging was performed manually by removal of the slag with a steel bar (middle left). While the working and slagging of the heat was taking place, a ladle was preheated (bottom left) to be in condition to receive the heat with minimum heat loss at the completion of working the heat (middle right).

When the heat was being tapped into the hot ladle (page 10, top right) the investment molds which had been preheated for several hours in a heating furnace (top left) were removed from the heating furnace and placed on a stand. Once the furnace tap was completed, the heat in the hot ladle was moved to the investment molds and the molten metal cast into the center riser (tree) of the molds (middle left) allowing the metal to flow into the twelve cavities of the tree with each cavity being used to form one medallion. After completion of the cast the molds were left to cool (middle right). Upon cooling the investment envelope was broken (bottom left), and the sand was separated from the metallic tree by means of a vibrating shake-out. Each medallion was then sawed off from the center riser and surface ground, yielding a finished medallion (page 11). The metal was analyzed and the analysis is shown on page 12. It was found that the carbon content of the metal can be varied by carbon addition, an important advantage demonstrating that steels or irons of various carbon content can be made by the Calderon process for different applications. It is to be noted that the analysis shows a carbon average of 2.134% C. This carbon increase was accidentally caused when carbon was added to degass the heat (page 9, top right).

Preparations for the 72 Hour Steady State Test

In preparation for such test certain changes had to be made to some equipment and relining of same. The 3-foot original reactor was relined with a larger taper and its inlet was made of stainless steel to avoid hang-ups (page 13, top left). The elbow was also relined in order to have its inlet match the outlet of the reactor (top right). A transition spool was made with provisions for a rope seal to interconnect the elbow with the collection drums. Two pallet lifters with base plates were provided in such a way as to have each collection drum secured to the lifter. The transition and collection drums were also lined (bottom left and right). Also

provisions were made to the drums for nitrogen or argon injection while cooling the product. The new charger delivered at the end of the previous quarter, was installed, piped and wired; it was put into operation and debugged.

Testing Procedure

It was agreed by U.S. Steel and Calderon that at least one short test will be performed to demonstrate the product collection procedure and provide an analysis of the material before the extended test. If the analysis did not meet 80% average metallization, additional tests would be run to reach the 80% goal of metallization. This was accomplished. The collection drum was positioned under the transition spool and raised by the pallet lifter so that the top rim of the drum presses against the seal imbedded in the spool (page 14, top left). After ignition, the pusher (top right) was sequenced to receive the feed of ore mix with coal and charged into the reactor at a slow speed controlled by hydraulic valves and timer (bottom left) in order to expose the ore and coal face to radiant heat at the discharge end of the reactor, at such a rate as to establish a pre-set residence time. The material was discharged into the insulated drum sporadically because of inherent conditions within the reactor. The exhaust gases exited from the process through a port provided in the spool (see red-hot exhaust, right bottom).

Initially, the material produced during the pre-steady period was collected in the bottom of the drum. Once a period of 3 hours elapsed the charging of raw materials and the oxygen injection was interrupted. the drum was dropped from under the spool and pulled away from the spool. A piece of ceramic blanket (parting medium) was immediately dropped on the hot material in the drum and the drum was moved back to engage the spool and be in a condition to receive the product at steady state as the charging and oxygen injection was resumed.

The steady state lasted for 5.5 hours at which time, the unit was shut down and the material was cooled with nitrogen; it was shipped the next day to U.S. Steel for analysis. The results will be forthcoming in three to four weeks from U.S. Steel.

Conclusion

The work performed to-date is encouraging by virtue that product was produced with the lowest cost raw material (ore concentrate) , and the energy source being exclusively coal. The product was melted and cast. The equipment has been debugged and preparations are taking place towards the integration of the process to produce directly molten iron and/or molten steel. Also it is planned to conclude the 72 hours test at reasonably continuous steady state during next quarter.

Submitted by:

Albert Calderon
Project Director

Calderon Reactor that directly converted iron ore concentrate and coal with oxygen into iron-steel/carbon product

**The iron-steel/carbon product
from the reactor
as delivered in boxes to the foundry**

When working the heat in the induction furnace the heat was over-active.

Carbon was added to heat to de-oxidize it.

Slagging the heat by removing the slag with a bar.

The heat prior to tapping

The ladle to receive the heat was preheated during the working of the heat.

Melting & Working the Heat

The investment molds in the form of "trees" containing twelve cavities (per "tree") were preheated overnight.

Tapping the heat into the preheated ladle

Casting from the ladle into the preheated "trees".

Casting completed.

Partial break-up of investment mold during cooling of the cast.

Tapping and Casting the Heat into Medallions

**Medallion cast on March 26, 2002 from
iron directly made from U.S.S. ore concentrate
and coal via Calderon's unique Direct
Iron/Steel Making Process
(Analysis of metal on page 12)**

REFRACTORY & INSULATION WORK

**Reactor Outlet
accentuating taper**

**Elbow lining &
hole for lance penetration**

**Transition spool showing
refractory lining & seal**

**Pallet lifter showing
drums with lining**

MECHANICAL & HYDRAULIC WORK

**Collection drum on pallet
lifter sealed to
transition spool.**

View of new charger.

**Controls for hydraulic
system of pusher.**

Red hot exhaust.