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DIRECT REDUCED IRON
RESEARCH AND DEVELOPMENT PROJECT

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Principal Author(s): ALBERT CALDERON

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Submitting
Organization(s)

CALDERON ENERGY COMPANY

500 Lehman Avenue

Name & Address

P.O. Box 126

Bowling Green, OH 43402

(1)

(2)

(3)

(4)

(5)

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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: 7-01-02 to 9-30-02

Date of Report: 10-29-02;

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Total Project: \$ 14,732,316.00 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.

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.

Accomplishments and Discussion

In the “Conclusion” section of the last quarterly report (page 8), it was stated that the challenges confronting the attainment of 72 hours of reasonably continuous operation were:

- Thermocouple life in a hostile environment; and
- Sticking of the material produced in the reactor.

Also, reference was made to a plan 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 of the reactor with the homogenizer.

The work performed during the past quarter for the advancement of the Calderon technology for the making of iron units by making use of iron ore concentrate and coal, for which this report is presented covered the following areas:-

- Item A - Development and Application of a Thermocouple Device to Withstand the Hostile Environment in the Elbow (Discharge End of Reactor).
- Item B - Development Work to Eliminate the Sticking of Material at the Discharge End of the Reactor and Collection Drums.
- Item C - Work Relating to the Prevention of Refractory Wear in the Reactor.
- Item D - Completion of Construction and Integration Work Pertaining to the Reactor and Homogenizer.

Item A - Development and Application of Special Thermocouple Device

The Calderon process like any high temperature process requires the accurate recording of temperatures in order to control the process. High temperature readings in excess of 2200°F demand the mounting of thermocouple wires within a ceramic sheath which is quite fragile. The tip of such a sheath is shown in photograph 1 which was taken after the cool-down of the reactor discharge, the thermocouple being a “B” type, and having been newly installed with its sheath prior to the run. Photograph 2 shows the build-up at the tip of the same sheath after three runs. Such build-up gives erroneous readings causing process control problems and the making of inconsistent product.

For this reason, it was decided to develop a water-cooled thermocouple device with a disposable tip that can be inserted through a port of the reactor which can at least last several hours of operation (the aim of 4 plus hours) and its tip being changed during the run within a few minutes by removing the device from the reactor and replacing it without interruption to the operation. Such device was constructed as shown in photograph 3; it was put into use. A port in the elbow of the reactor was provided for mounting the device; photograph 4 shows the port and the device in the process of being inserted through the port. Photograph 5 shows the tip of the thermocouple with the build-up after several hours of use, and photograph 6 shows the thermocouple with a new tip prior to insertion into the port. This device has worked rather well, as long as its fragile quartz tube does not penetrate deeply into the elbow or touch the material being discharged from the elbow. U.S. Steel’s Research suggested the use of an optical pyrometer for high temperature readings. Arrangements were made for the acquisition of such pyrometer, and delivery will take place soon.

Item B - Development work to Eliminate Sticking

The most vexing challenge in the project to-date has been the sticking of the metallized material at the discharge end of the reactor and to the walls of the collection drums. This sticking problem at the reactor disrupts steady state operation and often causes the shut down of operations by the inability to push the charge through the reactor. The material freezes and the pusher ram becomes immobile even though the hydraulic pressure peaks to its maximum (1500 psi). When there are no stickers the operating pushing pressure hovers between 150 and 300 psi. Some of the sticking is mild as shown in photograph 7, and some is very severe as shown in photograph 8. The metal “sticker” removed from the discharge end of the reactor after cool-down of a severe “sticker” is shown in photograph 9; such sticker may weigh as much as 50 pounds.

The sticking of the material to the collection drums lining shown in photograph 10, makes it difficult to remove the material from the drums, often necessitating the destruction of the drum, a costly issue.

To prevent sticking at the discharge end of the reactor two approaches have been used. The first approach is the provision of a drum preheat practice of at least three hours prior to placing the collection drum under the reactor using the burner shown in photographs 11 and 12. The second approach is the trying of various settings for a lance practice using the Lance Penetration System (LPS) shown in photograph 13. These settings include:-

- The depth of penetration of the lance into the tunnel formed by the partial combustion of the coal core, shown in photograph 14, in order to reach the coal core which is the source of the thermal energy, the carbon, and the

reducing gases; the encapsulation of the coal core within the annulus of the ore-and-coal mix is shown by photographs 15 and 16;

- The feed and dwell of the LPS, in order to prevent the localized over-heating of the material; and
- The rate of oxygen feed by the lance in order to prevent over-oxidation of the material.

The sticking problem at the discharge end of the reactor has not been eliminated at this writing but has been mitigated, shown in photograph 17. Additional work is under way to solve this problem.

With respect to the sticking problem in the collection drums, an approach consisting of using a fiber liner surrounded by a ceramic blanket shown in photograph 18 and 19 has been tried. The liner disintegrated after a single use. A refractory coat called "X-9" was brushed on the fiber lining, photograph 20, to give a hot parting medium between the material and the liner as shown in photograph 21. The destruction of the fiber lining after a single use persisted. Work is to continue in order to obtain multi-use of drum linings.

Item C - Work Relating to the Prevention of Refractory Wear in the Reactor

Since oxygen is used to combust part of the coal in the core, it is important to combust the coal sub-stoichiometrically and to keep such combustion within the core because of the high prevailing temperatures. Excessive refractory wear was noticed at the top of the entry of the elbow which connects to the end of the reactor, photograph 22. Even though such wear is repairable, photograph 23, effort was expended to find out the reason for such wear. It was discovered that the deposit build-up at the tip of the lance nozzle, photographs 24 and 25, redirects the oxygen injection pattern thus impinging on the refractory.

To avoid such redirection of the oxygen from the lance, a scraper which peels off the build-up on the lance tip was developed, photograph 26, which has been quite successful in keeping the lance tip free of build-up. It is fortunate that the build-up chills on the water cooled lance and is easily scraped. The scraper is fixed while the lance moves to effect the scraping. Photographs 27, 28 and 29 show various positions of the lance in relation to the scraper.

Item D - Completion of Construction and Integration Work Pertaining to the Reactor and Homogenizer

Construction work was completed at the larger site "the New Laboratory" and all services were brought to the homogenizer to make it operative (photograph 30). Also, the reactor (photograph 31) was moved from the machine shop which occupies an area of 1,500 square feet to the New Laboratory which has an area of 10,000 square feet; services were brought to the reactor at its new location to make it operative.

The integration of the reactor with the homogenizer has been withheld until the problem of sticking at the discharge end of the reactor is overcome. Iron/steel product made in the reactor and fed into drums continued and some of this product (photograph 32) has been melted and cast into pigs (photographs 33 through 39).

Conclusion

As of this writing the longest run with acceptable product as tested by grinding for rough metallization evaluation has been 33 hours of continuous operation. The work during the coming quarter is to concentrate on eliminating the sticking problem and extending the operation to 72 hours. Additional work will also encompass integrating the reactor to the homogenizer in an attempt to eliminate the sticking problem by virtue that homogenizer (furnace) possesses a source of heat; whereas, the collection drum which is a "heat sink" because it does not have a source of heat, may be causing the discharge of the reactor to cool thereby freezing the material at the discharge end.

Submitted by:

Albert Calderon
Project Director

1: Build-up at the discharge end of the Reactor.
(Sheath of Thermocouple after a single run.)

2: Build-up at the discharge end of the Reactor.
(Sheath of Thermocouple after 3rd run.)

3: Water-Cooled disposable tip Thermocouple.

4: Water-Cooled disposable tip Thermocouple being inserted into port at discharge end of the Reactor.

5: Thermocouple disposable tip covered by deposit after several hours of continuous monitoring.

6: Thermocouple with new disposable tip.

8: Build-up (severe) at entrance of elbow.

9: Metallic “sticker” removed from entrance of elbow.

Collection
Drum Rim

Refractory
Lining
Build-Up

Ceramic
Blanket
Insulation.

10:

Metallized Iron
Product

Refractory Lining

11: Burner applied to preheat collection drum before being placed under the Reactor.

12: Drum Preheat. (Close-up)

13: Lance penetration system. (LPS)

14:

Tunnel formed by combusting coal core with lance, yielding thermal energy and reducing gasses.

Refractory insulation

Coal core

15:

Annulus of mix made up of
ore concentrate and coal.

Reactor Flange

16: Reactor as viewed when separated from the elbow.

17: Improved lance practice, mitigating build-up.

18: Fiber lining trials for collecting drum.

19: Drum lined with a fiber liner.

20: Drum lined with fiber and painted with parting material.

21: Collecting drum full of material immediately after extraction from under reactor.

22: Refractory wear at elbow.

23: Refractory repair at elbow.

24: Cause of redirection of oxygen injection.

25: Lance tip build-up.

26: Scraper to peel off lance build-up.

27: Lance & scraper with lance in extended position.

28: Lance & scraper with lance in retracted position before being scraped.

29: Lance & scraper with lance in retracted position after being scraped.

30: Induction melter. “Homogenizer”

31: Calderon Reactor that directly converts iron ore concentrate and coal with oxygen into iron/steel product.

32: Iron\steel product (feedstock) for homogenizer. (Melter)

33: Iron\steel product being homogenized before being killed.

34: Molten metal killed with carbon.

35: Molten Metal being slagged.

36: Heat of molten metal being tapped.

37: Molten metal being cast into pigs.

38: Pigs being air cooled.

39: Pig exclusively made from Iron Ore fines and Coal via the Calderon Process.