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PHASE II CALDERON PROCESS TO PRODUCE
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

Reporting Period: 1-1-05 to 3-31-05

Date of Report: 4-26-05;

Phase II Award Date: 6-23-00;   Anticipated Completion Date: 12-03-05

Total Project: \$ 14,732,316.00   Total DOE Share This Action: \$6,457,000.00

Contracting Officer's Representative (COR): John Stipanovich;

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. In 2000, U.S. Steel teamed up with Calderon for a joint effort which will last 42 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 by using iron ore concentrate and coal as raw materials, both materials being appreciably lower in cost than using iron pellets and coke.

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Executive Summary

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 will produce in a closed system iron units from iron concentrate (ore) and coal competitively by eliminating pelletizing, sintering, coking and blast furnace operation. If such process steps could be eliminated, a huge reduction in polluting emissions and greenhouse gases (including CO₂) relating to steelmaking would ensue. Such reduction will restructure the steel industry away from the very energy-intensive steelmaking steps currently practiced and drastically reduce costs of making steel.

The development of a technology to lower U.S. steelmaking costs and become globally competitive is a priority of major importance. Therefore, the development work which Calderon is conducting presently under this Agreement with the U.S. Department of Energy becomes more crucial than ever.

Consistent with the objectives set in the Conclusion of the previous report twelve test runs were conducted with a duration of 588 hours using 16,973 lbs. of ore concentrate and 12,124 lbs. of steam coal. Two runs were 84 hours each, the longest ever attained; they were ended on schedule.

Experimental

During the 1st Quarter of 2005 which this report covers, twelve experiments were conducted; they numbered from Test Runs #167 through #178 inclusive. Table 1 shows the Test Run numbers, the dates at which the tests were started, the duration of each test, and the number of pushing charges for each test. The total number of hours tested were 588 hours: 16 minutes and the total number of pushes were 6466. It is to be noted that each of the twelve Test Runs was pre-planned depending upon a definitive objective in order to add to the knowledge for the ultimate purpose of making hot metal while at the same satisfying U.S. Steel regarding the target of conducting three 72 hour tests at reasonably steady state with 80% (or better) metallization.

Results and Discussion:

During the Quarter the following results were obtained: Test Run #169 lasted 84 hrs: 15 minutes and Test Run #170 lasted 84 hrs: 00 of which 12 hours were allotted for start-up and 72 hours for steady state operation. No pushing problems were experienced during these two long tests, the longest we have achieved to date. This is an important milestone because it indicates that 72 hours of operation at reasonably steady state as set by U.S. Steel, is attainable.

As to the 80% metallization Calderon took the approach that the entire iron/carbon material called "intermediate" will be melted (homogenized), slagged-off and poured into pigs (see photograph #1). The pigs upon solidification were stacked on a pallet (see photograph #2) with the intention of sending them to U.S. Steel together with the collected dust, scrapings, and the data sheets of the entire Run including the melting logs, for an in depth analysis.

TABLE 1

Objective	Test Run #	Date	Duration	Number of Pushes
Run 84 hrs	167	Jan 9th	56 hrs: 45 mins.	629
Run 84 hrs	168	Jan 12th	40 hrs: 30 mins.	436
Run 84 hrs	169	Jan 17th	84 hrs: 15 mins.	932
Run 84 hrs	170	Feb 14th	84 hrs: 00 mins.	939
Run Test to Homogenize	171	Feb 21st	20 hrs: 42 mins.	222
Run Test to Homogenize	172	Feb 24th	20 hrs: 30 mins.	217
Run for 80% Metallization	173	Mar 1st	59 hrs: 28 mins.	663
Run for 80% Metallization	174	Mar 8th	59 hrs: 00 mins.	668
Run Test to Homogenize	175	Mar 14th	21 hrs: 00 mins.	231
Run Test to Homogenize	176	Mar 17th	21 hrs: 40 mins.	218
Run for 80% Metallization	177	Mar 22nd	59 hrs: 26 mins.	653
Run for 80% Metallization	178	Mar 29th	61 hrs: 00 mins.	658

Total number of hours - 588 hrs: 16 mins.

Total number of pushes - 6466

Total weight of ore concentrate used - 16,973 lbs.

Total weight of steam coal used - 12,124 lbs.

Contact was made with U.S. Steel with respect to Calderon's preparedness to ship the above. U.S. Steel responded with a letter (see Exhibit 1) stating (in substance) that Calderon should focus on making DRI of 80% metallization as agreed in 2002, and was amenable to help Calderon achieve that target.

The iron/carbon material that Calderon is capable of producing (see photograph 3) is not a DRI as is known in the industry, but a new, non-uniform, chunky, crude material with properties approaching metallic steel/iron containing gangue and carbon, which when ground sparks like steel (see photograph #4). In Calderon's opinion the logical approach is to recover the metallic iron from this crude material which we are now calling "intermediate", by melting it in order to homogenize it, thus removing the gangue and excess carbon as a slag.

U.S. Steel has good reason to be concerned about directly reduced iron (DRI) that is not at least 80% metallized by virtue of problems experienced by others with unreduced pellets or briquettes which caused the following problems when attempting to melt them:

- a) Inefficiency of heat transfer of the unreduced part of the pellet or briquette;
- b) Difficulty in melting by virtue of the pellet or briquette tending to float on the slag and molten bath;
- c) Damage to furnace refractory by virtue of chemical attack; and
- d) Kinetics of slag-metal interface reactions.

In as much as Calderon is eager to satisfy U.S. Steel, Calderon replied to U.S. Steel that it would persist in its testing program (see Exhibit 2) to produce an intermediate which will average at least 80% metallization and ship it as is (similar to that shown in photograph #5) to U.S. Steel for an in depth analysis including the performance of a mass balance. Conjunctively, Calderon informed U.S. Steel that it intends to continue to do developmental

work towards producing hot metal by homogenizing the intermediate and overcome the concerns listed above. Calderon believes these concerns can be dealt with by:

a) Having sufficient carbon integrated with the intermediate which is inherent in Calderon's process, to insure that sufficient reductant is present in the homogenizer to reduce unreduced ore;

b) Stirring the bath inductively and via bottom stir plugs to enhance the formation of uniform hot metal;

c) Neutralizing the acidity of the slag floating on the molten bath with additions;

d) Providing a dunking action to force the hot intermediate fed into the homogenizer by forcing it through the slag and submerging it into the molten bath.

In the last Quarterly Report reference was made that Calderon was constructing a "dunker" to submerge the intermediate into the molten bath; the dunker was completed, mounted to the side of the homogenizer and placed into operation. This dunker which is adapted to impart a repetitive up-and-down motion in order to submerge the intermediate into the molten bath through the slag comprises a vertical slide mounted on a frame which is equipped with an inverted "L" shaped arm to which a graphitic head is fastened; see photographs #6 and #7. In operation, the graphitic head is partly immersed in the molten bath and the graphitic head is made to rotate while it is raised and lowered into the bath, thus the entire area that makes up the top of the homogenizer is covered by the sweep of the graphitic head; see photograph #8 which shows the graphitic head partially immersed in the bath of molten metal. The dunker has worked better than expected in its capability to submerge the intermediate into the bath. Surprisingly, it was noticed that the rate at which the carbon in the intermediate goes into solution is markedly increased, and the metal becomes rapidly

quiescent quite rapidly by virtue of the accelerated de-oxidation by means of the excess carbon in the intermediate.

Since the homogenizer (coreless induction furnace) being used has no top, covering it with a blanket kept the conditions within the homogenizer reducing and an off-gas in the form of CO was formed with its combustion taking place as it was exhausted through the pouring spout of the homogenizer; see photograph #9. Within the furnace the temperature did rise dramatically to such an extent that a hot spot through a thick insulating ceramic blanket developed; see photograph #10. In these times when the cost of energy is so high and natural gas is no exception, the formation of a combustible gas as a by product source of thermal energy would be most welcomed by the steel industry.

Referring to Table 1, Test Runs #167 and #168 were scheduled to run 84 hrs each. Neither reached that goal. Test Run #167 was aborted after 56 hrs: 45 minutes, the cause being the pusher ram stalled. After cool down, the unit was disassembled and found material stuck at the discharge. Upon examination it was discovered that during start-up the oxygen input from the lances was higher than the three previous tests; this was done in an attempt to shorten start-up time; melting of the material could have taken place in the early part of this test. It was decided that from then on, the start-up would be slow to avoid pushing problems by preventing the melting of material at the discharge.

Test Run #168 was also aborted after 40 hrs: 30 minutes, the cause being a stuck sliding gate. Efforts were expended to get it un-stuck, but the transition above the gate and below the elbow was too hot for the men to tackle. After cool down, frozen material build-up between the gate and the transition had to be removed. It was decided to make a change to the refractory configuration at the gate proper to overcome this problem.

It is to be noted that during all the test runs subsequent to Test Runs #167 and #168, a period of 491 hours of operation in which 5,401 pushes occurred, the pusher never stalled. During Test Run I-177, which was started on March 22nd, air was introduced together with the oxygen in the horizontal lance in order to moderate the temperature in the core to prevent melting within the core of the charge. The introduction of the air took place at push 508 of the Run at which time the pushing pressure was recorded to be 289 psi. At push 562 the pushing pressure increased to 695 psi; such pressure had not been seen since the stalling of Test Run #167, a period of 81 days earlier during which time 5,088 pushes had occurred. It was decided to shut the air off and go back to the previous setting for oxygen without air.

With respect to the sliding gate sticking, the gate stuck at push 541 of Test Run #174; after trying to move the actuator back and forth for about 5 minutes, the gate became unstuck. During test Run #178 at push 595 the slide gate stuck; after several tries to get it unstuck, it would not budge. It was decided to drop the transition from the elbow, move the gate housing out and get it unstuck while hot, a hot, arduous job, but the men succeeded in putting the gate back into operation with a penalty of one-half hour loss to the operation and a loss of temperature from 2372°F to 2181°F at the discharge of the elbow. The sticking occurred @ 10:45 A.M. and it took close to 2-1/2 hours to recover to the temperature range at which steady state is maintained.

The duration of the runs were set with the following objectives:-The objective of Test Runs #167, #168, #169, and #170 was to attain 84 hrs of operation of which 12 hours would be the start-up from cold and 72 hours of reasonably steady state. As mentioned above, Test Runs #167 and #168 were aborted whereas Test Runs #169 and #170 did attain the 84 hrs

duration. The objective of Test Runs #171 and #172 were scheduled as very short runs to improve operation at homogenizing hot intermediate.

After receiving notice of U.S. Steel's position that DRI and not hot metal be made of 80% metallization, we decided to focus on U.S. Steel's position by running two consecutive 60-hour tests and select one drum of intermediate that we thought would be suitable to send to U.S. Steel for analysis, as a guide; such drum was delivered to U.S. Steel. Interspersed between the tests for U.S. Steel, we conducted short tests of about 20 hrs each; twelve hours for start-up and eight hours to homogenize the intermediate while it was produced at steady state in order to interest a third party in our process to make hot metal from ore concentrate and steam coal which the industry badly needs, and as outlined in our letter to U.S. Steel Exhibit 2. Test Runs #173 and #174, and test Runs #177 and #178 were relatively of long duration to concentrate on quality in order to attain 80% metallization. Test Runs #175 and #176 were relatively of short duration to improve on the practice to make hot metal.

Conclusion

Substantial progress was made during the Quarter covered by this report. Developmental work will continue during the next Quarter following the above procedure to satisfy both U.S. Steel's goal of making satisfactory DRI and Calderon's objective of producing hot metal from the intermediate. Also effort will be expended to overcome the problem of the sliding gate sticking. Even though such occurrence was the exception when compared to the large tonnage of intermediate handled by the gate. Interruption to the steady state operation which causes deterioration of the material within the reactor as well as heat loss which takes time to recover, must be prevented from happening.

Since Calderon is convinced that its process can be a contribution to the steel industry of major beneficial consequences, and the remaining funds at the Department of Energy are quite low with U.S. Steel's contribution having been expended, Calderon is increasing its contribution to 90% temporarily until additional funds are made available for this most important technological development.

Submitted by:

Albert Calderon
Project Director

References - Not Applicable

The work performed in this quarter which the report covers, was original work. No reference material was relied upon for the work.

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Joseph D. Defilippi
Division Manager – Product Technology and
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March 7, 2005

Mr. Albert Calderon
President
Calderon Energy Company
P.O. Box 126
Bowling Green, Ohio 43402

Dear Albert:

Thank you for your letter dated February 16, updating your progress on the Direct Ironmaking project. It is indeed encouraging that operating periods in excess of 72 hours can be achieved without the major difficulties experienced in the past. We know that long term continuous operation has been a major obstacle and we congratulate you on your success.

Given the non-uniformity of the product, we agree that it would be desirable to have a process that could accept a widely variant feed material. However, fundamentally, processes typically operate most efficiently and economically when feed material quality is held within a very tight range. This concept has been demonstrated repeatedly throughout the development of the steel industry and has been recently demonstrated by a few domestic steel companies that unsuccessfully attempted to make steel from an inconsistent DRI product.

Thus, we believe the project emphasis should be on producing a high quality, consistent, DRI material. If such a material can be produced economically, it is likely that it will find a place in the industry as a steel-making feed stock.

U.S. Steel still insists that the results of the project are judged against the mutually agreed upon objectives, that is, to produce DRI of at least 80 percent metallization, from a steady-state, continuous, 72-hour trial, and that the process repeatability be demonstrated by performing three separate tests that duplicate its success. To date, the project has failed to meet these objectives.

Although U.S. Steel has provided all of the in-kind services committed to in the Calderon/U.S. Steel Project Teaming Agreement dated December 28, 2000, we are still interested in this project. Please let us know how we can assist Calderon Energy

Mr. Albert Calderon

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in meeting the objective. We will consider all requests that help the production of a high quality DRI product, but please realize that requests for assistance will be judged on their merits against the current workload allocation and current priorities.

Sincerely,

J. D. DeFilippi

VIA OVERNIGHT COURIER

March 14, 2005

Dr. Joseph D. Defilippi
Division Manager -- Product Technology
and Acting General Manager -- Research
U.S. Steel Corporation
Research and Technology Center
4000 Tech Center Drive
Monroeville, PA 15146

Dear Dr. Defilippi:

I thank you for your constructive letter of March 7, and especially for U.S.Steel's position regarding the offer to assist Calderon in meeting the objective of "producing DRI at least 80% metallization from a steady state, continuous, 72 hour trial and that the process repeatability be demonstrated by performing three separate tests that duplicate its success."

We have a problem relating to avalanches that take place during the discharging of hot material from the end of the reactor which tend to drag partially un-reacted material from the reactor. This condition must be solved or mitigated in order to achieve the percentage of metallization agreed upon.

We would like to apprise U.S.Steel that Calderon has made two decisions:

(1) First, we will continue testing with emphasis on solving or mitigating the above-mentioned problem to enable us to meet the metallization objective of at least 80%, during the three separate tests mentioned above.

(2) Second, as an independent set of tests, we will continue making reduced material and converting it to hot metal. We need to pursue this technical direction, apart from the DRI tests, because we believe that the cost savings and merit of our process as applied to making hot metal directly are compelling. We intend to seek a third party who would be willing to work with us to make hot metal despite the non-uniformity of the reduced material produced.

If we succeed in the DRI metallization objective of at least 80% as agreed, well and good. If we fail to meet the 80% metallization objective, but we continue proving that hot metal can be made directly despite the non-uniformity of the material, we will invite U.S.Steel to join us. Our agreement provides that we have the right to pursue the

CALDERON ENERGY CO.
BOWLING GREEN, OHIO 43402

Mr. Joseph Defilippi
U.S.Steel Corporation
March 14, 2005
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hot metal application independently of U.S.Steel, but that U.S.Steel has the election to participate in a hot metal development phase we pursue with any third party. As we consolidate interest in our hot metal process with a third party, we will be providing U.S.Steel with a formal written inquiry, as required by the Agreement, asking whether U.S.Steel elects to participate in such hot metal development phase.

We would like U.S.Steel to be aware that the remaining funds from DOE are around \$200,000. We have decided to greatly increase Calderon's cost sharing contribution in order to advance our technology, whether we make DRI or hot metal. This will extend the life of the Cooperative Agreement with U.S.DOE, as well as the project.

Therefore, in the next few days I shall be calling Dave Rohaus for a meeting to take place in Bowling Green or Pittsburgh, but preferably in Bowling Green to show him the progress we have made since his last visit, and to initiate a meaningful work plan towards the conducting of the three 72-hour Test Runs.

I still look forward to meeting you personally.

Sincerely,

Albert Calderon
President

cc: Dave Rohaus

Photograph 1

Photograph 2

Photograph 3

Photograph 4

Photograph 5

Dunker

**Graphitic
Head**

Photograph 6

**Graphitic Head
Holder**

Vertical Slide

Motorized Drive

Height Adjuster

**Photograph 7
Dunker in Homogenizer**

**Bottom Part of
Holder**

Molten Metal

**Graphitic
Head**

**Photograph 8
Dunker in Action**

Photograph 9

Photograph 10