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: 10-01-03 to 12-31-03

Date of Report: 1-27-04;

Phase II Award Date: 6-23-00;   Anticipated Completion Date: 12-03-04

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.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

TABLE OF CONTENTS

Executive Summary	Page 1
Experimental	Page 2
Results and Discussion	Page 3
Conclusion	Page 10
References	Page 10

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.

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 drastically reduce costs.

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. To demonstrate feasibility of the Calderon process U.S. Steel set a target for metallization of 80%. Calderon met 90% of the 80% target (72.3% metallization). Steps were taken during the past quarter to make changes to the equipment to overcome the impediment which prevent the attainment of 80% or better metallization of the iron ore concentrate. Work is proceeding to attain this objective.

Experimental

During the quarter for which period this report covers the following Runs were conducted using the newly installed vibrating conveyor:

Run #127 - October 28, 2003 -- Objective: 16 hour Run. Total number of hours from start-up: 5 hrs:16 mins. Number of pushes: 35. Material being processed reaching above 2400°F: None. Cause of shut down: Smoke leakage from burned holes through the sealing skirt

Run #128 - November 6, 2003 -- Objective: 16 hour Run. Total number of hours from start-up: 14 hrs:0 mins. Number of pushes: 100. Material being processed reaching above 2400°F: None. Cause of shut down: High hydraulic pushing pressure reaching limit – unable to push because of material hanging at reactor discharge.

Run #129 - November 11, 2003 -- Objective: 16 hour Run. Total number of hours from start-up: 15 hrs:8 mins. Number of pushes: 92. Material being processed reached above 2400°F in 11 hrs:0 mins. Cause of shut down: Product hanging on hot face at reactor discharge stalling horizontal lance.

Run #130 - November 17, 2003 -- Objective: 16 hour Run. Total number of hours from start-up: 15 hrs:55 mins. Number of pushes: 104. Material being processed reached above 2400°F in 6 hrs:55 mins. Cause of shut down: Scheduled shut down.

Run #131 - November 19, 2003 -- Objective: 24 hour Run. Total number of hours from start-up: 25 hrs:34 mins. Number of pushes: 216. Material being processed reached above 2400°F in 8 hrs:55 mins. Cause of shut down: Scheduled shut down.

Run #132 - November 25 & 26, 2003 -- Objective: 32 hour Run. Total number of hours from start-up: 31 hrs:10 mins. Number of pushes: 315. Material being processed reached above 2400°F in 6 hrs:10 mins. Cause of shut down: Scheduled shut down.

Run #133 - December 2, 3 & 4, 2003 -- Objective: 48 hour Run. Total number of hours from start-up: 47 hrs:50 mins. Number of pushes: 435. Material being processed reached above 2400°F in 6 hrs:5 mins. Cause of shut down: Scheduled shut down.

Run #134 - December 8, 9 & 10, 2003 -- Objective: 72 hour Run. Total number of hours from start-up: 62 hrs:10 mins. Number of pushes: 641. Material being processed reached above 2400°F in 7 hrs:10 mins. Cause of shut down: Unable to push because of material hanging at discharge of reactor.

Run #135 - December 29, 2003 -- Objective: 48 hour Run. Total number of hours from start-up: 12 hrs:45 mins. Number of pushes: 96. Material being processed reached above 2400°F in 11 hrs:55 mins. Cause of shut down: Refractory failure at transition.

Results and Discussion

Most of the month of October was devoted to integrating the vibrating conveyor to the reactor and its ancillaries such as the elbow, off-gas spool, transition, etc. A running track was also installed on the floor for the conveyor in order to accommodate the operating position and the retracted position, the retracted position being provided for maintenance access to the elbow, off-gas spool and transition. Photograph 1 shows the general arrangement in preparation of miscellaneous initial check-list tests prior to commencing the Runs. During the performance of the check list test it was discovered that the sealing of the cover to the conveyor proper was not adequate to contain emissions and an alternative had to be devised. As previously reported the plan was to have the cover fixed and the conveyor

vibrating with an adjustable seal in between; see photograph 15 of previous report. This feature did not work.

The steps taken to overcome this problem were to redo the section of the cover at the drop zone and fabricate a new transition; see photographs 2 and 4. Both the underneath side of this cover and the transition were insulated (see photographs 3 and 4) to withstand the high temperature prevailing in the drop zone; also, the cover and transition were equipped with mounting rings in order to fasten a sealing skirt made of fiber-glass cloth with the capability to collapse “accordion” like. The upper mounting ring was installed on the transition and the lower mounting ring on the conveyor cover; see photograph 5. A raising and lowering device attached to the off-gas exhaust spool (photograph 6) provided the capability to raise and lower the transition so as to interconnect the fixed exhaust spool to the cover which is an integral part of the vibrating conveyor. When needed the transition is dropped into the cover of the conveyor thus freeing the transition from the spool. The conveyor is then free to be rolled from under the exhaust spool (see photograph 7) and thus provide access to the exhaust spool, the elbow and the discharge of the reactor for maintaining them.

With respect to the sealing of the conveyor to the cover to prevent smoke and dust emissions, it was decided to use the ledge which circumscribes the entire conveyor (see photograph 8), as a surface to lay beads of a putty-like aluminized sealant (see photograph 9) which upon curing, a continuous leakproof seal is formed between the ledge and the cover as the bolts and nuts which fasten the cover to the conveyor; are tightened. Having made these changes during the first four weeks of October, it was decided to commence the test Runs. Initially, these tests were set to last two consecutive 8-hour shifts (16 hours in total),

and gradually ramp up to reach the 72 hour objective of reasonably continuous operation at steady state. A minimum temperature of 2400°F at the face of the material being processed just before discharging from the elbow, was set as the benchmark for the beginning of the steady state condition.

On October 28, Run #127 was initiated but was aborted after 5 hrs:16 mins. due to holes which developed in the sealing fiberglass skirt and smoke leakage became too excessive to continue the Run. The damage to the skirt is shown in photograph 11. The cause for this failure was attributed to heat. To overcome the problem it was decided to mount to the transition an additional protective skirt made of heavy, fire resistant, insulating ceramic cloth (see photograph 12) ahead of the fiberglass skirt in order to protect it.

On November 6, Run #128 was begun and lasted 14 hours, shy by 2 hours from the 16-hour objective. The cause for the shut-down was the inability to push attributed to frozen material at the discharge of the elbow. Material freezing at the discharge of the elbow is caused by excessive heat loss. Despite running for a period of 14 hrs., no steady state was attained. To overcome this problem, a curtain made of fire-resistant ceramic cloth was installed between the hot section and the cold section of the conveyor cover (see photograph 13) to maintain the drop zone hot by minimizing heat loss.

On November 11, Run #129 was conducted for 15 hrs:8mins. with steady state reaching after 11 hrs from start-up; still 52 minutes shy from the 16-hour objective. The cause for the shut-down was the inability of the horizontal lance to penetrate the coal core, which is attributed to material freezing at the discharge end of the elbow. Prior to shut-down the transition was dropped for inspection. Cool solidified material was spotted between the exhaust spool and the transition; see photograph 14. It was decided to drop the transition

and roll the conveyor out from under the exhaust spool in order to have access to scrape frozen material with a prying bar and dislodge the material stuck to the spool and elbow; see photographs 15 and 16. In addition to the problem of material sticking, smoke had been observed to leak again through the fiberglass sealing skirt while in operation and before the rolling of the conveyor from under the exhaust spool. Even though steady state was reached after 11 hrs from start-up, and scheduled shut down was only 52 minutes away, it was decided to shut down none-the-less.

Based on the data gathered during the first three Runs, it was evident that the sealing skirt at the transition needed to be improved to prevent failure of the fiberglass cloth to prevent emissions, and that the insulation of the hot end of the conveyor needed to be improved as the outer steel shell temperature at the drop point hovered around 470°F. It was decided to make the two following improvements before proceeding with additional Runs:- (i) the fiberglass cloth to be coated with the aluminized putty and (ii) redoing the lining of the conveyor at the drop point to reduce the shell temperature to minus 250°F.

In the improvement of the fiberglass skirt, a simple roller assembly was made where the skirt can be stretched to properly apply the aluminized sealant; see photographs 17 and 18. While aluminizing the skirt for the drop zone, the skirt used at the discharge of the conveyor which is not exposed to high temperature was also aluminized. After the application of the aluminized putty, the skirts were hung to dry overnight and were installed as shown in photographs 19 and 20.

With respect to relining the drop zone, it was decided to use a three part composite lining to insure a high degree of insulating property. The first part which was against the steel shell consisted of insulating boards; see photograph 21. Next, a form was made for the drop

zone to enable the pouring of an insulating castable; see photographs 22 and 23. Following the setting of the castable, a ruby refractory good for 3200°F and possessing abrasion resistant properties was rammed into place; see photograph 24. In addition the entire length of the conveyor beneath the cover beyond the drop zone was insulated with board and wool; see photograph 25.

Since the cloth curtain shown in photograph 13, was used for trial as a temporary measure, but proved that a temperature of 2400°F can be reached through its use, it was also decided to replace the cloth curtain with a more substantial structure which consisted of an insulated gate to minimize heat loss at the drop point. This gate possessed a steel backing-plate (see photograph 26) to which insulation was mounted to the side facing the heat, and a pivot was also provided for rotation; see photograph 27. Photographs 28, 29 and 30 show the gate in the closed, opened (ambushed), and intermediate positions respectively. This gate is operable from the outside of the drop zone by means of a handle; see photograph 31.

Having made the modifications as described, Run #130 occurred on November 17, with steady state being reached in only 6 hrs:55 mins. after start-up, and the unit was shut-down after 16 hrs (minus 5 mins) on schedule. For Run #131, it was decided to increase the Run to two 12 hour shifts; this Run took place on November 19, and lasted 25 hrs:34 mins. with steady state reaching almost 9 hours after start-up. Shut-down was a little after schedule in order to make up for the extra time taken to reach steady state.

The length of Run #132 was extended to 32 hours. It reached steady state in 6 hrs:10 mins. Total length of Run was 31 hrs:10 mins. It began on November 25th and ended on the 26th.

The length of Run #133 which took place on Dec 2nd, 3rd, and 4th, was extended to 48 hrs.; it operated for 47 hrs:50 mins. with steady state being reached 6 hrs:05 mins. after start-up. It was also shut down pretty much as scheduled.

With respect to Run #134, it was decided to operate for 72 hrs. It started on the 8th and ended on the 10th of December lasting 62 hrs:10 mins. with steady state reaching 7 hrs:10 mins. after start-up. The cause for the shut-down was the inability to push because of material hanging at discharge of reactor caused by melting in the core and collapse of the annulus.

Run #135 which was started on the 29th of December, was scheduled to operate 48 hours because of New Year's eve. It operated for only 12 hrs:45 mins. with steady state reaching 11 hrs:55 mins. after start-up. The cause for the shut down was failure of refractories at the transition. It is believed that the excessive time taken to reach steady state may have been caused by processed material slumping at the hot zone causing the gate not to fully close thus contributing to heat loss.

Another area where a small but significant improvement was made during the past quarter was the change in design of the oxygen injection nozzle for the horizontal lance, and also the material of construction of the lance itself. The horizontal lance plays a major role in converting the coal core to thermal energy to devolatilize the coal to release hot gases which include H_2 , CO, CH_4 , tars, light oils and ammonia, and some H_2S , O_2 , N_2 , and CO_2 . Of these H_2 and CO are the most important gases for the desired reducing reactions while the tars, light oils and ammonia upon cracking generate appreciable amounts of H_2 , the most reactive reducing agent. Heretofore, the nozzle of the horizontal lance protruded from the lance (see photograph 32) and its life span was good for about a Run of 24 hours provided

it did not encounter solid processed material as it penetrated the coal core. Since the coal temperature can easily reach 3000°F, the nozzle becomes soft despite its cooling and it takes little force to disfigure it thus skewing the orifice through which the oxygen yet passes. Such change of direction of the jet impinges upon the annulus of the mix and melting takes place resulting in cave-ins of the annulus; see photograph 33 which shows a hole in the annulus by virtue of a cave-in.

To address this problem, a nozzle which is flat (see photograph 34) and does not protrude (see photograph 35) was developed. This nozzle has two holes with solid bottoms in addition to the orifice for the oxygen jet, in order to engage a spanner wrench with the solid bottoms preventing water leakage. Such revised nozzle appears to concentrate the jet of oxygen into the coal core (see photograph 36) and renders it more constant to result in preventing cave-ins, in extending the life of the nozzle, and in providing a core hole which is concentric to the annulus of the mix (see photograph 37).

The second improvement which relates to the lance itself is that in the past not much attention was paid as to whether the body was made of stainless steel welded pipe or stainless steel seamless tubing. Recently, it has been discovered that welded pipe cracks at the seam; see photograph 32. A crack in the pipe reduces cooling of the nozzle through water loss; further, water leaks also cause upsets to the process. For these reasons, only stainless seamless tubing will be used from now on.

Conclusion

From the nine Runs that occurred during the past quarter, it has been learned that the maintenance of the drop zone at a temperature above 2400°F is imperative to prevent the material produced from freezing at the discharge of the elbow. It is also important to provide a non-protruding nozzle to the horizontal lance so that it remains cool and therefore strong and stable. Heat loss in general must be reduced to a minimum.

During the next quarter the work will continue to focus on attaining the 72 hour steady state test with such test being repeated two additional times as agreed with U.S. Steel. Integration with the melting furnace will follow once these tests are achieved.

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

1. General arrangement

2. Cover at the drop zone of conveyor

**Lower ring
for sealing
skirt**

3. Insulation underneath of cover at the drop (hot) zone of the conveyor

4. Transition between off-gas exhaust spool & conveyor

**Upper ring
for sealing
skirt**

5. Transition and conveyor positioned at drop zone

Upper sealing ring

Lower sealing ring

6. Device for raising and lowering transition for engagement and disengagement from off-gas exhaust spool and conveyor

Off-gas exhaust port

Sealing skirt when transition is in raised position

7. Conveyor moved away from off-gas exhaust spool

Spool

Transition

Collapsed sealing skirt

Conveyor

8. Conveyor without cover

Drop zone

**Sealing ledge to seal
cover to conveyor**

**Sealing skirt at
conveyor
discharge**

9. Sealing conveyor cover

Cover

Sealing ledge

**Aluminized
sealant gasket
between cover
and conveyor**

10. Cover bolted to conveyor

Cover

Bolts

Sealing ledge

Conveyor

11. Damaged sealing skirt

**Burned out
holes**

**Fiberglass
cloth**

12. Inner skirt of heavy, fire resistant ceramic cloth providing protection to sealing skirt at drop zone

**Upper sealing ring of
transition for mounting
fiberglass sealing skirt**

13. Fire resistant ceramic cloth curtain for separating drop zone from rest of conveyor for minimizing heat loss

14. Transition lowered from off-gas spool for inspection

15. Material stuck to spool and elbow being scraped

**Conveyor moved
away from
exhaust spool**

16. Removal of product stuck to exhaust spool and elbow

**Collapsed
sealing skirt**

17. Reinforcement of sealing skirt with aluminized sealant

Outer sealant coating

Inner sealant coating

18. Rollers specially made for spreading aluminized sealant on sealing skirt

19. Transition in place at drop zone with aluminized sealant skirt making sealed connection between fixed exhaust spool and vibrating conveyor

Exhaust spool

Transition

Skirt

Conveyor

20. Collection drum at discharge of conveyor with aluminized skirt connecting vibrating conveyor and fixed drum

21. Insulation board at the drop zone of conveyor

Insulation boards

22. Form for installation of insulating castable at drop zone of conveyor

Form

Reaction springs of conveyor

Vibrating conveyor

23. Insulating castable lining installed at the drop zone

Forms to hold castable in place

24. Ruby refractory laid on top of insulation at drop zone

25. Insulation along entire length of conveyor

Insulating wool

Insulating board

Sealing ledge

26. Metal plate backing for mounting insulation to form an insulated gate in order to maintain heat at the drop zone of conveyor

27. Insulating gate being mounted underneath cover of conveyor

Insulation board

Rotating shaft
used as pivot

28. Insulated gate in closed position

Gate
(closed)

Drop
zone

29. Insulated gate (unseen) in open position

Opening to allow product to leave drop zone when gate is opened

Drop Zone

30. Insulated gate in intermediate position

Gate

Drop Zone

31. External handle used to open and close gate

Conveyor

Handle

**32. Protruding lance nozzle
for oxygen injection**

Nozzle

Copper

Crack in stainless
welded pipe

**33. Irregular bore in core
caused by deflection of oxygen
jet from lance**

Cave-in hole
caused by melting

**34. Flat nozzles with holes for
spanner wrench**

Holes

Orifice for
oxygen jet

35. Flat lance nozzle for oxygen injection

**Flat
nozzle**

**Stainless
seamless tubing**

36. Coal core and annulus of mix as seen from charging end

**Coal
core**

**Annulus of mix made
up of ore concentrate
and coal**

37. Regular bore caused by proper consumption of coal core by oxygen jet