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

Report Type:

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	Bowling Green, OH 43402		
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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: 10-01-02 to 12-31-02

Date of Report: 1-28-03;

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

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 7), it was stated that the challenges confronting the attainment of 72 hours of reasonably continuous operation was the sticking problem.

The work performed during the past quarter to solve the sticking problem focused on the following approaches:

- Approach A Development of a Start-up Practice to Prevent Early Sticking Conditions;
- Approach B Work Related to the Prevention of Sticking in the Reactor

 Through Selection of Materials and Incorporating Structural

 Changes;
- Approach C Improvement to Lances and Lance Practice to Overcome Sticking in the Reactor;
- Approach D Poking at Discharge End as an Interim Answer to Relieve

 Excessive Pushing Pressures to Overcome Sticking;
- Approach E Work Towards Prevention of Sticking in the Drums.

Other work performed in addition to addressing the sticking problem included the following:

- Item 1 Process Integration with Homogenizer
- Item 2 Analyses of Various Steels Produced From Process
- Item 3 Conceptual Illustration of Pioneer Plant and Scale Model of Same (Unfinished)
- Item 4 Coordination of Activities with U.S. Steel

Approach A - Development of a Start-Up Practice to Prevent Early Sticking Conditions

In starting the operation from cold, there is a tendency to freeze material that had been reduced and which was transformed to the eutectic phase. Such freezing of the material takes the form of a crust or "skull" that adheres to the refractory at the discharge end of the reactor. The hotter the discharge end the less the tendency to adhere.

An initial approach for cold start-up was the use of an oxy-acetylene torch inserted through a sight port to provide ignition, followed by oxygen injection. This approach made possible high temperature localized heating but was not conducive to the rapid propagation of heat to the cold refractory lining.

A second approach was to utilize a start-up burner of large capacity fired from the bottom into the discharge end of the reactor (photograph #1), without operating the pushing mechanism until a temperature of 2000°F is attained at the discharge end of the reactor. This approach was adopted; it gave the process a means for quick warm-up prior to beginning the pushing of material into the reactor in order to minimize premature freezing.

Approach B - Work Related to the Prevention of Sticking in the Reactor Through Selection of Materials and Incorporating Structural Changes

The sticking of material to the walls of the reactor is caused by the silica in the ore and the silica in the coal ash which is acidic in nature; it reacts with the basic properties of the refractory lining. The lining is basic to withstand the high temperature needed for the reactions to take place. Initially, the build-up begins at the walls (photograph #2) and proceeds to an advanced stage (photograph #3) that causes the shut down of operations by inability to push ore and coal into the reactor.

Since prevention of sticking is the ultimate goal, a graphitic refractory cement (C-34) made by UCAR (successor of Union Carbide) was recommended by U.S. Steel Research (photograph #4); this material was intended to be used as a thick paint to give the refractory walls non-wetting properties, the refractory walls being made of X-9 plaster which is good for 3000°F and made by Vesuvius. This C-34 cement paint unfortunately did not withstand the oxidizing effect from the oxygen injected by the lance.

It was then decided to use the C-34 not as a paint but as an integral component of the X-9 refractory by mixing 75% of X-9 with 25% of C-34 (termed option #1) to form a composite (photograph #5) and initially tried as a local coupon enveloping a thermocouple (photograph #6). The results were encouraging by virtue of the smooth surface resulting after 10 hours of continuous operation when compared to the surface of an X-9 coupon (photograph #7). Based on these results Calderon proceeded to reconstruct the discharge end of the reactor with this composite refractory with a ratio 66% (X-9) + 34% (C-34) (photograph #8) in order to prevent or mitigate "sticking". A twenty-four hour test followed;

unfortunately, the composite material failed by virtue that the carbon in the composite was consumed by the oxygen and the properties of the residual X-9 deteriorated.

Since extensive discussions had taken place for several years of using Silicon Carbide (SiC) which was Nitride bonded for application in the commercial reactor for making coke and for the pilot reactor for producing iron units, it was decided as Option #2 to embed some coupons of SiC in the X-9 refractory of the elbow of the reactor and subject them to operating conditions. A burner head of Nitride bonded SiC which had been obtained from Vesuvius was smashed with a sledge hammer and some shattered pieces were embedded in the refractory as planned. The reactor was operated for 24 hours. Upon extraction of the coupons, severe deterioration had taken place (photograph #9), by virtue of exposure to oxygen. It is evident that it would have been a major set back both economically and technically to this project had Calderon constructed a SiC reactor at the pilot scale originally contemplated in the program.

At this juncture it was decided to forgo any refractory material that has carbon as a constituent, and a consultation took place between Calderon and Inland Fire Brick Co. of Cleveland, the refractory company that installed the original lining in the reactor. This company recommended a material with non-wetting properties which could be suitable for this application. The material chosen was PA748, a product made by Norton Refractories, to be applied as a heavy paint over the X-9 of Vesuvius. This was done and the unit placed into operation. It was found that the material would stand the temperature but would tend to flake away from the wall of the reactor.

As Option #3, it was decided to mix 50% of X-9 and 50% of PA748 (photograph#10) to form a composite refractory material and reline the elbow of the reactor, while at the same

time make structural changes by increasing the taper of the elbow and also by increasing the slope at the discharge end of the elbow in order to facilitate the drop-off of reduced material at a temperature ranging from 2400° F to 2500° F. A device simulating a set of calipers was made to aid in establishing uniformity to the compounded taper and slope (photograph #11).

This Option #3 has been tested during several runs, the results are inconclusive by virtue that this composite working lining tends to leave the backup lining which abutts the steel shell of the elbow. The crusts (build-up) seems to have decreased but not eliminated.

The work regarding the selection of the right refractory for this application is proceeding with due diligence supported by actual tests. Towards the end of the quarter, representatives of both Harbison Walker and St. Gobain (successor of Carborundum) visited the Calderon facility and have given recommendations of refractory materials to be tried. Harbison Walker recommended a product called "Ruby" which is made from 40% $Cr_2O_3 + 60\%$ Al_2O_3 and St. Gobain recommended a product called "Coranit" made from 70% $Al_2O_3 + 30\%$ SiO_2 which is nitride bonded. The Ruby is currently in use in the Delta Section of electric arc furnaces in many melting shops and the Coranit is used in large blast furnaces in Europe to form a cup at the bottom of a blast furnace, areas known for intensive thermal attack. Samples of these materials are scheduled to be received and tested shortly.

Approach C - Improvement to Lances and Lance Practice to Overcome Sticking in the Reactor

In addition to the extensive work taking place with respect to finding the suitable refractories, work was also done in the area of the oxygen injection lances. This work encompassed the use of copper for the entire lance versus stainless steel with a copper

tip (photograph #12). Also lances of various lengths to maximize penetration into the charge for the efficient generation from the coal the following:

- Thermal energy to effect the reactions;
- Reducing gases; and
- Carbon

Because of using longer lances for deep penetration into the reactor the runway of the Lance Penetration System (LPS) had to be extended (photograph #13). Nozzles of various configurations (photograph #14) were made and tried in order to direct the oxygen jet away from the refractory walls in an attempt to prevent the excessive heating of the refractory and thus minimizing its softening; such softening adds to the sticking phenomena.

It has been found that by observation of a pressure gauge installed in the hydraulic line which feeds oil into the pushing cylinders of the raw material charging ram, it can be predicted when sticking begins to occur. Normally it is preferred to operate at a pushing of 250 psi. When the pressure increases to around 500 psi it is an indication that sticking is taking place (photograph #15). Often the pressure drops down on its own indicating that the ram force succeeded in dislodging the stuck material. At other times the pressure continues to climb without stopping until there is a forced shut down; this condition results in a poor product and an aborted run.

Approach D - Poking at Discharge End as an Interim Answer to Relieve Excessive

Pushing Pressures to Overcome Sticking:

To overcome aborted runs, poking ports where installed in the elbow which makes possible the removal of pipe caps and the introduction of a poker through the elbow in order to reach the discharge end of the reactor to dislodge the stuck material (photograph #16) and

thus maintain continuity to the operation; however, when poking takes place the steady state condition is disturbed and sub-standard material is generated. It is for this reason that it is imperative to prevent sticking and all effort is concentrated in achieving this. With persistence, the chances are that the problem of sticking will be solved. Calderon's team, U.S. Steel's Research and the qualified vendors are all committed to this task.

Approach E - Work Towards Prevention of Sticking in the Collection Drums

As has been reported in previous quarterly reports, sticking of the material to the refractory lining also occurs in the collection drums. In an attempt to solve this problem as option #1, sheet metal liners were introduced to line the inside of the drums to separate the refractory of the drum from the hot material falling into it (photograph #17). Because of the intense heat at the discharge end of the elbow of the reactor, the liners are destroyed as also shown by photograph #17. This arrangement was replaced by option #2 by providing obtuse tapers to the walls of the drums during the installation of the refractory of the drums (photograph #18). Such design has met with partial success, as the material still sticks to the refractory but not as severely. It is evident that once a suitable non-wetting lining is found for the reactor, such material will also serve to line the drums.

Other work performed during the quarter, in addition to the work done to address the sticking problem, is discussed as follows.

<u>Item 1 - Process Integration with Homogenizer</u>

This work revolved around the melting of the material produced to develop an acceptable practice with respect to:

- Suitability of induction melting of the material produced taking into consideration slag attack of furnace walls by the silica contained in the gangue of the ore and in the ash of the coal; and
- Capability of making steels and irons of various carbon content from the material produced.

With respect to using induction melting as a tool it has been found that the reduced material from the reactor being magnetic gets attracted to the wall of the hearth of the furnace by virtue of the coil surrounding the hearth. This phenomena tends to prevent the material from penetrating the slag to be homogenized in the bath of the metal beneath the slag. A graphitic dunker was made in order to force the material into the slag and bath (photograph #19). Discussions took place with Ajax, the maker of the furnace, and it was concluded that a bottom stirring plug in combination with a furnace extension may solve the problem, the stirring plug with argon injection to cause agitation, and the extension to keep the material charged at such a distance from the furnace coil as to minimize the magnetic effect from the coil. Modifications were made to the furnace and both stirring plug and furnace extension were installed (photographs #20 and #21).

The homogenizer was operated using a molten heel practice wherein ingots of metal previously cast would be melted first (photograph #22) and then the reduced material added to finish the heat. The ratio that was charged included 130 lbs of heel and 90 lbs of material which was slowly added to the molten heel while the metal was stirred by means of nitrogen being injected into the plug. Because of the availability of nitrogen in the laboratory it was decided to use it instead of argon which in commercial application argon would be the choice as the stirring gas. During this work it was discovered that the bath depth in relation to the

thickness of the slag is excessive causing the material charged to not penetrate through the slag readily (photograph #23), forcing the use of a bar to dislodge the semi-molten material from the wall and into the bath (photograph #24). To prevent attack of the lining at the slag line, CaC₂ was gradually added with the material in order to neutralize the acidity of the slag in order to prevent attack of the lining.

From the work done on the homogenizer, it is concluded that a furnace with a large shallow bath to maintain the thickness of the slag to a minimum is the correct approach wherein argon stirring of the molten bath and a foamy slag are incorporated. In other words, instead of using a tubler-shaped furnace a shallow bowl-shaped furnace should be used. Steps have been taken to look for a small used arc furnace to replace the induction furnace. In the meantime, work will continue with the existing furnace.

<u>Item 2 - Analyses of Various Steels Produced from Process</u>

Despite the problems experienced by the sticking of material in the reactor and those of the furnace just described, iron and steel of various commercial grades were made directly from ore concentrate and coal by the Calderon process. Samples were sent to Bowser Morner, a testing laboratory, to test samples taken from the various heats made. It is very encouraging to find that the process is capable of making the following:

- cast iron of 3.51% carbon (test #05);
- low carbon iron of 2.62% carbon (test #014-2);
- high carbon steel of 0.94% carbon (test #10F);
- medium carbon steel of 0.55% carbon (test #12F); and
- low carbon steel of 0.06% carbon (test #12H).

Test results from Bowser Morner are attached.

It appears that the process is capable of making all grades of irons and steels including the ultra low carbon steel (interslilial free steel). There is no denial as evidenced by the attached results that the metal has a very high sulfur content which must be addressed to make the process viable.

<u>Item 3 - Conceptual Illustration of Pioneer Plant and Scale Model of Same</u>

As illustrated in the layout of a commercial plant as conceived by Calderon which is marked Item 3, a desulfurization station is included. It is planned to tap the steel from the furnace/homogenizer into a ladle as a rimming steel. The initial step after the tap is to vacuum degas it in order to decarburize the heat; the second step is to desulfurize it by injection of a desulfurizer and to decant the slag; the third step is to alloy the heat and adjust the temperature before sending the ladle to the caster. This is the contemplated practice for most steels. Construction of a model of this Pioneer Plant at a scale of 3/16" to the foot has begun (photograph-Item 3(a)).

<u>Item 4 - Coordination of Activities with U.S. Steel</u>

On November 11, 2002, a group of four U.S. Steel representatives led by Dr. Patula, Director of U.S. Steel Research in Monroeville, visited the Calderon facility in Bowling Green, Ohio to:

- Observe an on-going test;
- Discuss the 72-hour test; and
- Discuss the game plan for the advancement of the Calderon technology;

The on-going test went well and Calderon was complimented for the progress achieved. The problem of the material sticking to the discharging end of the reactor had been

known to U.S. Steel as communicated to U.S. Steel verbally from time to time and as reported to NETL in the quarterly reports, copies of which being regularly sent to U.S. Steel.

Calderon presented to U.S. Steel a Work Plan (Item 4(a)), towards the performance of the 72-hour test. U.S. Steel indicated that the work plan was reasonable and that it would be issuing a Test Plan for the 72-hour test as a recommendation. On November 27, 2002 Calderon received the Test Plan which is attached (Item 4(b)). Calderon is in general agreement with the Test Plan recommended by U.S. Steel.

Calderon presented its ideas regarding the commercial application of its technology using the layout (Item 3) as a guide. The issue of desulfurization was broached and the manner of separating the -1/4" fines from the +1/4" material. All parties are aware that the sticking problem of material at the exit of the reactor must be solved as a priority, and U.S. Steel has assured continued assistance. A meeting has been arranged for the 6th of February in Bowling Green, at which time Mr. Ainsworth, U.S. Steel's refractory expert, will attend to review the work done as well as the input from the refractory vendors, and give his recommendation.

Conclusion

The work in the last quarter was very intensive and it shall continue to be so to find the

answer to prevent sticking. All effort will be concentrated to solve the "sticking" problem in

order to proceed with the 72 hour test. Work will also be directed towards desulfurization in

the ladle after killing the heat in order to establish an acceptable practice for managing the

high sulfur content in the metal, an inherent factor when using coal.

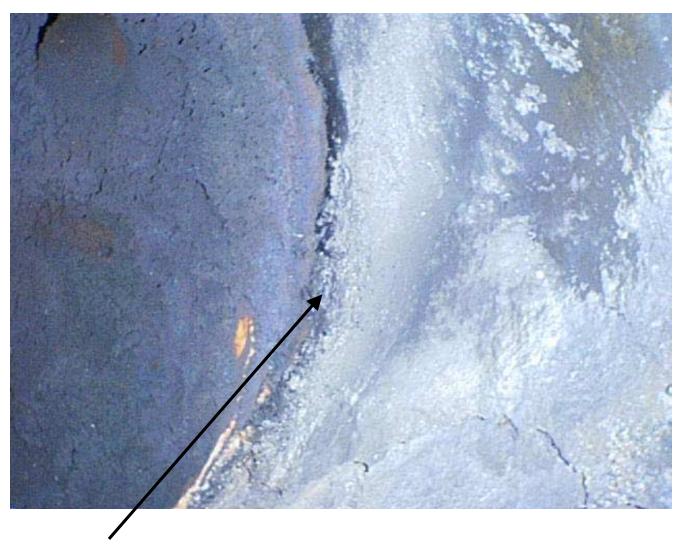
Submitted by:

Albert Calderon

Project Director



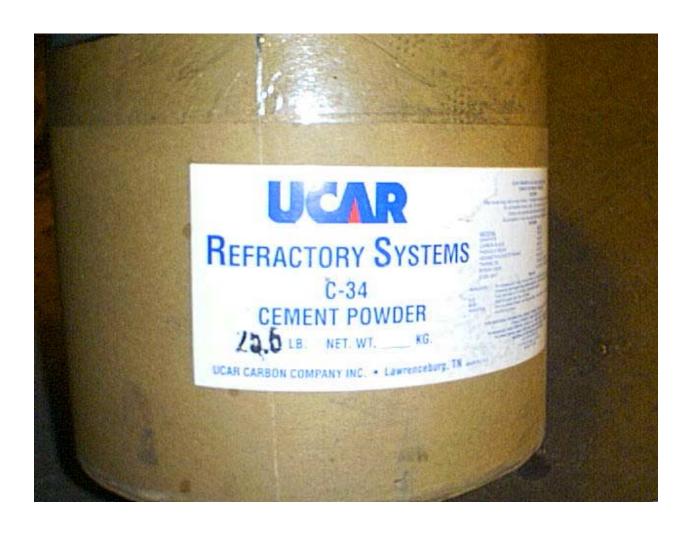
1. Start-up Burner.



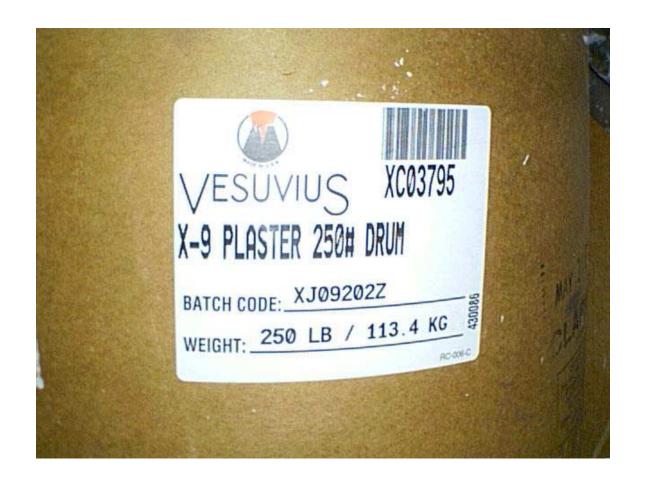
2. Initial build-up at discharge end of Elbow increasing pushing pressure.

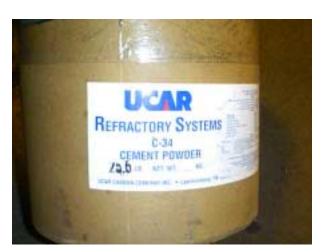


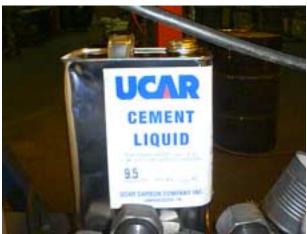
3. Advanced build-up at discharge end of Elbow sufficient enough to cause shut-down by inability to push.



4. U.S.S. Research Recommendation to use as Parting Material.

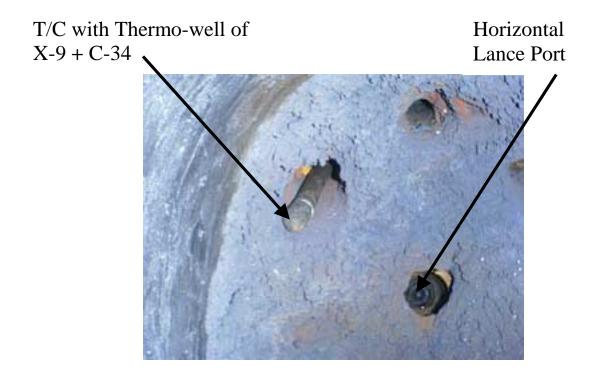




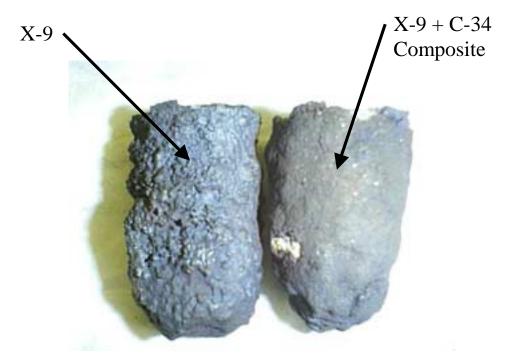


5. Option #1: Trial with composite refractory material.

(75% X-9) + (25% C-34)



6. Close-up of Elbow interior



7. Close-up of both Thermo-wells after use.





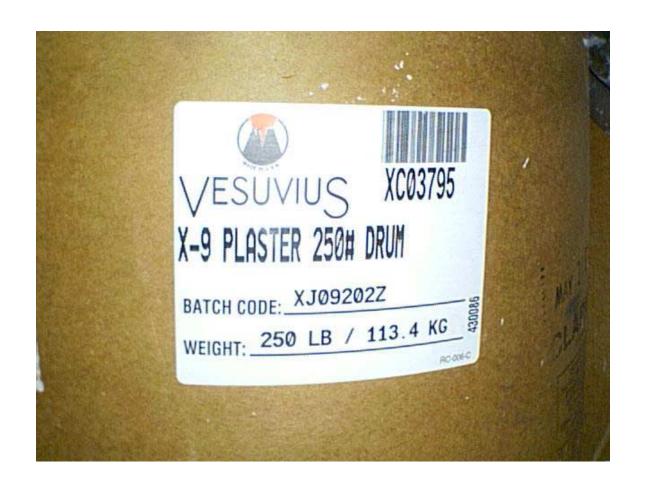
8. Lining of Elbow with a composite of 66% X-9 + 34% C-34 to prevent or mitigate sticking.







9. Option #2: Silicon Carbide Nitride Bonded

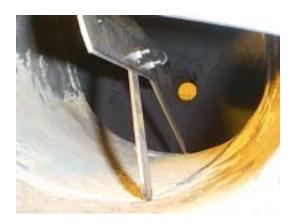






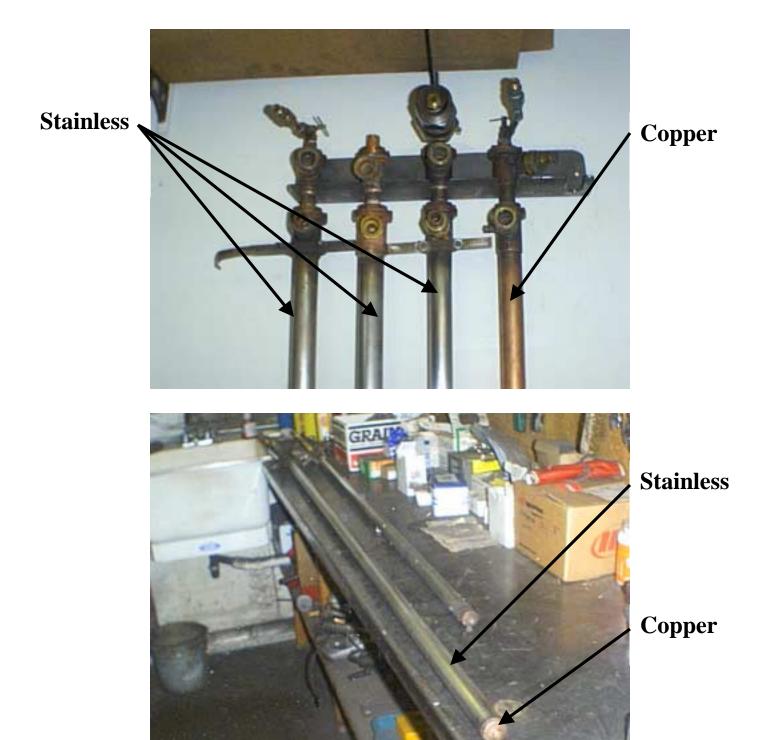
10. Option #3: Composite refractory material. (50% X-9) + (50% PA748)







11. Structural changes to mitigate sticking.

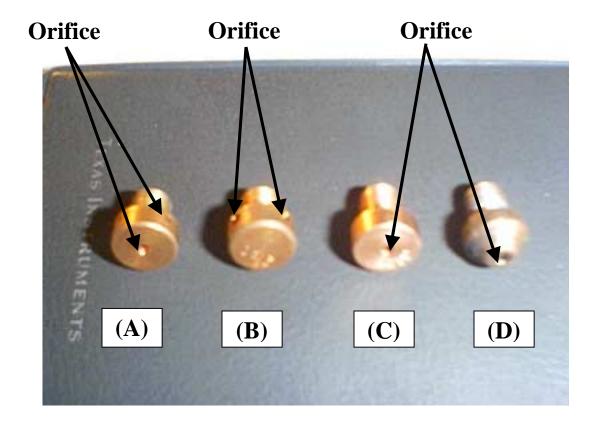


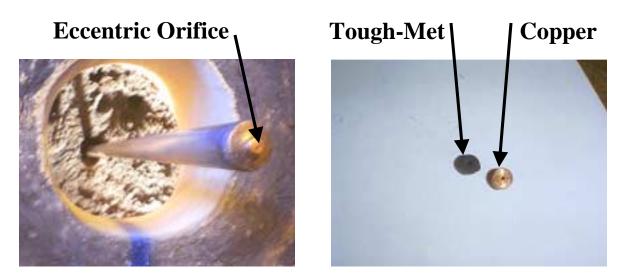
12. Materials of construction of lances and lengths





13. Modification to the Lance Penetration System (LPS).





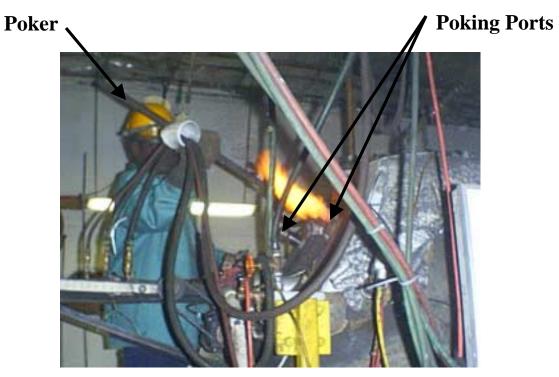
14. Lance nozzles configuration and material of construction.





15. Pressure alarm leading to sticking.





16. Interim step for continuity of operation.



17. Prevention of sticking in the drums: **Option #1**



18. Prevention of sticking in the drums: Option #2





19. Material floating and graphite dunker



20. Perforated plug for insection of gas to cause stirring



21. Furnace extension with plug stirring.



22. Previously cast ingots being melted to form the molten heel.



23. Material floating at the slag line.



24. Forcing material below slag line for reactivity.



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LABORATORY REPORT

TO: ALBERT CALDERON

CALDERON ENERGY COMPANY

500 LEHMAN AVE.

BOWLING GREEN, OH 43402

Report Date: 01/22/03 Job Number: 10010193

Group No. : 41410 Sample No. : 300432

Auth/P.O.# : PDU/DRI-1449 B

Sample Identification: 05 Date Received: 01/16/03 Date Sampled:

Analysi	s Description		Result	Units		Method	i
Phosphorus	CAST TRO	0. 0. 0.	024 409	% % %	BMI BMI BMI BMI BMI	34-065 34-060 34-060 34-065 34-060	
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Submitted by,

Thomas W. K Thomas M. Ryan, Senior Chemist Analytical Services Division



Item 2 continued

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LABORATORY REPORT

TO: ALBERT CALDERON

CALDERON ENERGY COMPANY

500 LEHMAN AVE.

BOWLING GREEN, OH 43402

Report Date: 01/22/03 Job Number : 10010193 Group No. : 41410 Sample No. : 300431

Auth/P.O.# : PDU/DRI-1449 B

Sample Identification: 014-2

Date Sampled:

Date Received: 01/16/03

Analysis Description		Result	Units		Method	
	CARBON IRON -	2.62 <0.01 0.039 0.363 0.02	% % % %	BMI BMI BMI BMI	34-065 34-065 34-065 34-065	
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		•				
						1.5.45 - 46.55

Submitted by,

Thomas M. Ryan, Senior Chemist Analytical Services Division



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Units

LABORATORY REPORT

Result

TO: ALBERT CALDERON

CALDERON ENERGY COMPANY

500 LEHMAN AVE.

BOWLING GREEN, OH 43402

Analysis Description

Report Date: 01/22/03

Job Number : 10010193

Group No. : 41410 Sample No. : 300434

Auth/P.O.# : PDU/DRI-1449 B

Method

Sample Identification: 10F

Date Sampled:

Date Received: 01/16/03

	•				
Manganese Phosphorus	HIGH CARBON STEEL -	<0.01 0.009	96 96 96 96	BMI BMI BMI BMI BMI	34-065 34-060 34-065 34-065 34-060
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Submitted by,

Thomas M. Ryan, Senior Chemist Analytical Services Division



Item 2 continued

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LABORATORY REPORT

TO: ALBERT CALDERON

CALDERON ENERGY COMPANY

500 LEHMAN AVE.

BOWLING GREEN, OH 43402

Report Date: 01/22/03 Job Number: 10010193 Group No.: 41410

Sample No. : 300436

Auth/P.O.# : PDU/DRI-1449 B

Sample Identification: 12F

Date Sampled:

Date Received: 01/16/03

Analysis Description	Result	Units		Method		
Carbon - MED. CARBON STEEL -	0.55	%		BMI	34-065	
Manganese	<0.01	%		BMI	34-060	
Phosphorus	0.057	%		BMI	34-060	
Sulfur	0.534	% %		BMI	34-065	
Silicon	0.02	*		BMI	34-060	

Submitted by,

Thomas M. Ryan, Senior Chemist Analytical Services Division



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LABORATORY REPORT

TO: ALBERT CALDERON

CALDERON ENERGY COMPANY

500 LEHMAN AVE.

BOWLING GREEN, OH 43402

Report Date: 01/22/03

Job Number : 10010193

Group No. : 41410 Sample No. : 300435

Auth/P.O.# : PDU/DRI-1449 B

Sample Identification: 12H

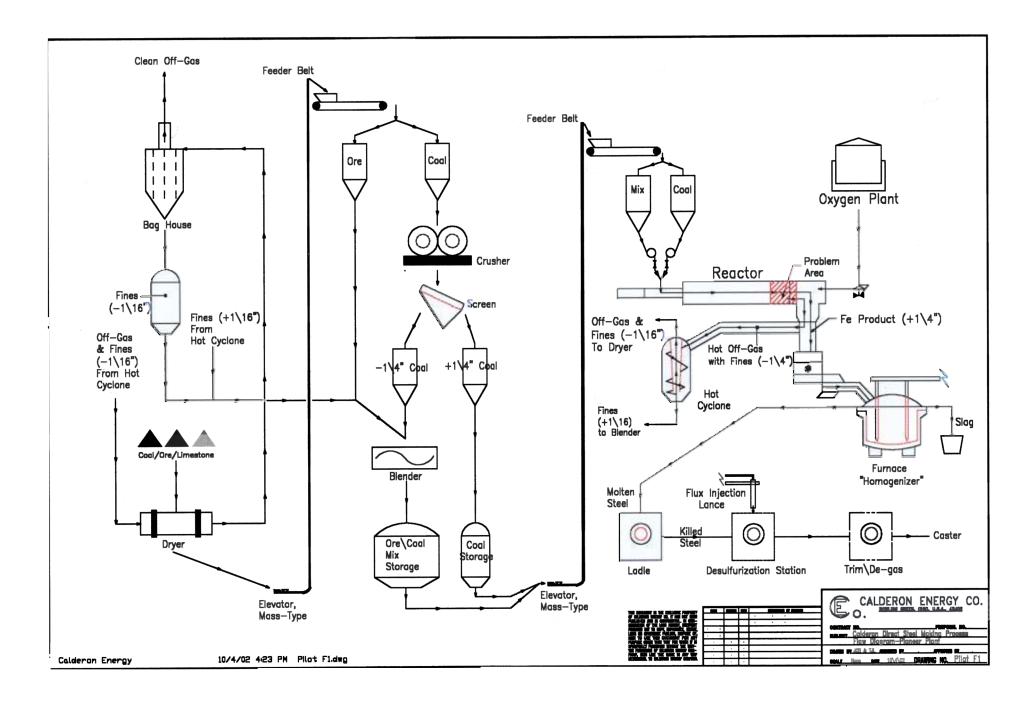
Date Sampled: Date Received: 01/16/03

Analysis Description		Result	Units		Method		
Carbon - Lo Manganese Phosphorus Sulfur Silicon	W CARBON STEEL -	0.06 <0.01 0.049 0.588 <0.01	8 8 8 8	BMI BMI BMI BMI	34-065 34-060 34-060 34-065 34-060		
		*					
4							

Submitted by,

Thomas M. Ryan, Senior Chemist Analytical Services Division

Thomas M. Ryan





Item 3(a). Model of pioneer plant at a scale of 3/16" to the foot (unfinished).

AS REPORTED - **STICKING** OF THE MATERIAL TO THE LINING OF THE REACTOR IS THE PROBLEM THAT HAS PREVENTED THE PERFORMANCE OF THE 72 HOUR TEST

WORK PLAN TOWARDS THE PERFORMANCE OF THE 72 HOUR TEST

- 1. SOLVE THE **STICKING** PROBLEM
- HAVE A DRY RUN OF 36 HRS (50% OF 72 HR TEST) AT REASONABLY CONTINUOUS STEADY STATE WITH U.S.S's PRESENCE – (4 TO 5 DRUMS)
- 3. SHIP THE DRUMS TO U.S.S. FOR EVALUATION
- 4. DEPENDING ON EVALUATION:
 - IF POSITIVE SET DETAILS OF 72 HR TEST AND PROCEED
 WITH U.S.S. PRESENCE
 - IF NEGATIVE -
 - (A) CORRECT DISCREPANCY WITH ADDITIONAL TESTING; AND
 - (B) HAVE ANOTHER DRY RUN OF 36 HRS AT STEADY STATE SHIP DRUMS TO U.S.S. FOR EVALUATION
- 5. IF POSITIVE SET DETAILS OF 72 HOUR TEST AND PROCEED WITH U.S.S. PRESENCE

Item 4(b)

CALDERON TEST PLAN

Calderon Energy is planning to continuously operate the bench scale unit for extended periods of time, 36 to 72 hours, with the objective of producing material that is, on average, 80 percent metallized. To facilitate the test analysis, the following recommendations are offered.

Recording of Test Data

All data pertaining to the test should be recorded. This should include all feed weights, number of pushes per hour, and operating variables, such as, temperatures, oxygen gas flow, periodic gas analysis, and operating delays, etc. In addition, all abnormal conditions are to be noted.

Mass Balance

The purpose of analyzing the mass balance is to account for all the iron fed to the process. If all the iron can not be accounted for (within reason), the balance is incomplete, and the tests results will be unreliable. All scales used to measure the feed material and product should be calibrated and checked for accuracy on a regular basis.

To establish accurate feed composition, a 100 gram grab sample of the feed (blended ore and coal fines) and coal should be collected every hour. These samples will be composited and analyzed for iron and other major constituents.

Product Analysis for Metallization

All of the product should be weighed and analyzed to complete the mass balance. Initial weights of each drum and refractory should be recorded. If U.S. Steel is going to analyze the product, then all the drums of product from a test should be shipped to the Tech Center. Even the material remaining in the reactor after a test should be placed in a drum so that all the material can be accounted for. Similarly, any solid material collected from the off gas stream should be shipped will the drums of product.

The contents of each drum will be weighed and analyzed to determine the amount of iron and degree of metallization within each drum. In the past, the product stuck to the refractory and they were separated by breaking the materials. Comparing the refractory weights before and after can help determine if all the refractory was removed from the product.

The product from each drum will be screened to 0.25 inch. The oversized material will be crushed and screened again. The crushing and screening will be repeated until the oversized material can not be crushed. Both the oversized and undersized material will be weighed. All minus 0.25 inch material will be riffled into four samples of about 200 grams each. At least two samples will be submitted to the UEC Chemistry Lab for analysis. It will be assumed that the material greater than 0.25 inch will have a typical hot metal chemistry.

Item 4(b) continued

Based on the analysis and the product weight of each drum, the total iron and metallic iron weights will be determined. Summation of all product drum weights will provide the total iron and metallic iron weights for the test. This will be compared to total amount of iron fed to calculate the iron balance. The percent metallization will be calculated for the product that was generated during steady state conditions, dividing the metallic iron content by the total iron content.