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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: 4-01-04 to 6-30-04

Date of Report: 7-28-04;

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.

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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.

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 CO<sub>2</sub> 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 2<sup>nd</sup> Quarter of 2004 which this report covers, thirteen experiments were conducted; they numbered from Test Run #139 through Test Run #151. The table that follows gives the dates, the length in hours, the number of pushes, the ore weight, the coal weight and the total weight of ore and coal charged of each test run.

| Test Run | Date From To | No. of Hours | No. of Pushes | Ore Wt. | Coal Wt. | Total Wt. Charged |
|----------|--------------|--------------|---------------|---------|----------|-------------------|
| #139     | 4-05 to 4-07 | 51:15        | 552           | 1,449   | 1,035    | 2,484             |
| #140     | 4-13 to 4-14 | 37:55        | 407           | 1,068   | 764      | 1,832             |
| #141     | 4-20 to 4-21 | 48:11        | 521           | 1,368   | 977      | 2,345             |
| #142     | 4-26 to 4-29 | 88:45        | 969           | 2,544   | 1,817    | 4,361             |
| #143     | 5-04 to 5-07 | 89:00        | 961           | 2,523   | 1,801    | 4,324             |
| #144     | 5-11 to 5-13 | 58:40        | 645           | 1,693   | 1,210    | 2,903             |
| #145     | 5-18 to 5-19 | 58:42        | 651           | 1,709   | 1,221    | 2,930             |
| #146     | 5-26 to 5-26 | 15:20        | 42            | 110     | 79       | 189               |
| #147     | 6-03 to 6-03 | 19:20        | 181           | 475     | 340      | 815               |
| #148     | 6-07 to 6-09 | 60:20        | 668           | 1,754   | 1,252    | 3,006             |
| #149     | 6-16 to 6-16 | 16:00        | 163           | 336     | 336      | 672               |
| #150     | 6-24 to 6-24 | 15:30        | 148           | 305     | 305      | 610               |
| #151     | 6-29 to 7-02 | 87:18        | 982           | 2,023   | 2,023    | 4,046             |
| TOTALS   |              | 646:16       | 6,890         | 17,357  | 13,160   | 30,517            |

These tests aggregated to 646 hours and 16 minutes of operation consisting of 6,890 pushes and consuming 17,357 lbs of ore and 13,160 lbs. of coal. The ratio per push of ore to coal in the mix in Test Run #139 through #148 inclusive consisted of 3 parts ore (42 oz. ore) and 1 part coal (14 oz. coal) in the annulus with one pound (16 oz) of coal in the core. There was

a change made at push 359 of Test Run #148 to the pattern of charging until the Test run was completed by charging the mix (still in 3 parts ore and 1 part coal) but as a slice of mix followed by a slice of coal obviating the annulus/core pattern; the slice of the mix was increased to 5½ lbs. per push and the slice of coal to 2.75 lbs. per push. Test Runs #149, #150 and #151 were conducted using the same pattern of charging employing the slice mode, and the proportions of ore and coal used in the latter part of Test Run #148.

### Results and Discussion

As stated in the Conclusion section of the previous Quarterly report, that since the metallized material has the tendency to increase its adhesive properties at the temperatures of metallization, effort would be expanded to develop a modified lance practice to minimize sticking and consequently reduce build-up. It was also stated that a monitoring procedure would be implemented that could give early notice that a build-up was occurring. Further it was mentioned that a simple device be used to dislodge the build-up from the discharge of the elbow and apron with minimum interruption to the operation.

During the span of time within the Quarter, five approaches were tested. The first was to change the cycle of the horizontal lance. The practice that had been used involved movement of the lance forward into the core a distance of 23 inches from the face of the material at the discharge end at a rate of 7/8 of an inch with a 30 seconds pause and retract at the same rate. By monitoring the temperature of the cooling water of the lance it was noticed that the water tended to boil when the lance was in the advanced forward position within the core by virtue of its long residence time, a sign which indicated that excessive heat was being removed from the material being reduced. To minimize heat loss from the material into the cooling lance water, the pauses were eliminated from the cycle so the lance traveled

uninterrupted from the home position to the end of the travel a distance of 30" and automatically reverse to home position.

Another change made to maintain heat at the end of the reactor, a ring was fabricated (see photograph #1) for sandwiching it between the reactor and the elbow. This ring was provided with circumferential ports (see photograph #2) for the injection of oxygen, air or air enriched with oxygen and lined with Ruby refractory; see photograph #3. The ring was then mounted to the elbow (see photograph #4) and the reactor was then bolted to the elbow with the ring in between; see photograph #5. A manifold equipped with valves was provided to control the flow of oxidant to the ten ports of the ring.

Another structural change made to the equipment to minimize sticking at the discharge end was to replace the gas exhaust spool which possessed tapered (converging) sides (see photograph #6) which interconnected the elbow (not shown in photograph #6) and the transition which is above the sliding gate shown in photograph #7. This replacement consisted of providing a new exhaust spool with a vertical wall at the discharge. Photographs #8, #9 and #10 show the progressive steps in the fabrication and the lining of the new spool. With the changes made a high thermal input via suppressed combustion of coal gases was obtained at the discharge end; see photographs #11, #12, and #13. Photograph #11 was taken while looking into the gas exhaust spool; photograph #12 was taken from beneath the sliding gate while the slide gate was in the process of opening; and photograph #13 was taken from beneath the sliding gate while the gate was completely opened showing the entire discharge all the way up to the ceiling of the elbow with the water cooled horizontal lance shown as a dark pipe. These changes made possible to confine high and adequate

temperature at the discharge end while still depending exclusively on thermal energy directly derived from the coal charged.

With respect to dislodging frozen material at the discharge end in order to prevent the interruption of operation, a platform above the elbow was constructed with a set of stairs leading to it; see photograph #14. An access port was provided in the roof of the elbow to enable the use of a jackhammer that can chisel the stuck material from above; see photograph #15. A special refractory cap was made (see photographs #16 and #17) to keep the port closed. When cleaning was necessary as detected by making use of a mirror (see photograph #18) positioned at floor level under the slide gate in the opened position, it would tell whether or not the discharge needs cleaning. If so, a worker climbed to the platform and extracted the refractory cap (see photograph #19) and by means of the jackhammer equipped with a chisel would dislodge the material stuck at the discharge end. The worker then would remove the jackhammer with red hot chisel extending from it (see photograph #21) and closed the port by placing the refractory cap back into the port.

During the early part of May Calderon received notice that Mr. Maddox, Acting Assistant Secretary of Energy in charge of fossil fuels, would be paying a visit to the Bowling Green facility on June 3<sup>rd</sup> to discuss Calderon's technology of coal gasification and observe the operation of its pilot unit including iron making. A dry run was set up for May 26<sup>th</sup> which included the making of metallized material from ore concentrate and coal, melting it, and casting the molten steel into molds. Once this would be done, the unit would be charged with high sulfur coal to demonstrate gas making in anticipation of Mr. Maddox's visit for the 3<sup>rd</sup> of June. Test Run #146 which represented the dry run was performed. Subsequent to this test and a few days prior to the 3<sup>rd</sup> of June, Calderon received notice that Mr. Maddox's visit would

be postponed to the 24<sup>th</sup> of June. Since all preparation had been made to run Test #147 for Mr. Maddox, it was conducted as if he were present. Test Run #148 which lasted 60 hrs: 20 mins. was started on June 7<sup>th</sup> and ended on June 9<sup>th</sup>. A second dry run Test Run #149 was conducted on June 16<sup>th</sup> in preparation of Mr. Maddox's visit and on the 24<sup>th</sup> of June, and Test Run #150 was conducted in Mr. Maddox's presence.

Test Run #150 was begun at midnight and when he arrived at 10:30 a.m. a short discussion took place which lasted about one hour. A crowd of about 20 people were present; Mr. Maddox representing DOE, representatives of the two Ohio Senators (DeWine and Voinovich), several representatives from the State of Ohio, the mayor and the Director of Utilities of the City of Bowling Green, the press and some of Calderon's employees. Mr. Maddox observed the unit in operation after an explanation of its components and the objective of making iron/steel from iron concentrate and coal. He saw reduced material being made and collected in a drum then while hot fed into an induction furnace; see photographs #22 and #23. The heat was first worked (see photograph #24) and then tapped at 2927°F into molds; see photographs #25, #26 and #27. A sample taken prior to the tap was cooled, ground to show that it was steel via a sparking test, and given to Mr. Maddox; he showed interest in taking it with him.

Following the iron making demonstration and while a working lunch was taking place, the workers at the unit purged it with high sulfur steam coal to demonstrate the gasification features of the Calderon technology. At 1:30 p.m. the meeting was adjourned. Before leaving he walked to the floor a second time and observed gas being made from coal (see photograph #28) while feeding crushed run-of-mine Ohio high sulfur coal as delivered

(unprepared 3" x 0"), into the reactor; no pulverization or slurry was used. In answering questions to the press, Mr. Maddox seemed to be impressed with Calderon's technology.

In summarizing the work done during the Quarter, the following can be stated:-

with respect to minimizing heat loss via the cooling of the horizontal lance, it is a step in the right direction as any loss of heat at the discharge causes build-up; more work will be done to optimize its operation. As to the introduction of oxidant between the reactor and elbow, there was evidence that additional heat was peripherally imparted to the material.

With respect to the change of the gas exhaust spool, there is evidence that the build-up is reduced by providing a straight vertical wall at the discharge instead of an inclined plane. As to the device for dislodging build-up from the top, it works when the material can be chiseled; however several disadvantages were discovered:-

(a) the operator must be very cautious as flames tend to exit the port (see photograph #20);

(b) the chisel at the end of the jackhammer because of the intense heat within the elbow, makes the chisel (see Photograph #21), blunt and therefore soft and useless in its capability to dislodge hot, stuck material;

(c) since all of the oxygen injected into the reactor and elbow is shut off as a matter of caution during the dislodgement of the stuck material, and it takes some interval of time to chisel out stubborn material, excessive heat loss from the unit is experienced; and

(d) poor visibility because of brightness prevents the operator to dislodge the stuck material efficiently. The dislodgement of stuck material was and continues to be used only as a last resort.

## Conclusion

From the thirteen tests conducted during last Quarter which comprised the consistent use of the slide gate, there is evidence that large amounts of thermal energy can be provided from the coal in Calderon's technology particularly with the new ratio of mix to coal used in Test Runs #149, #150, and #151 and the manner in which these materials were charged in successive slices; photographs #11, #12 and #13 show the brightness of color caused by the generous amount of thermal energy that can be released from the coal. In the experimental work performed the ratio of coal to ore was increased during the three above mentioned Test Runs. The purpose of this change was to shift the chemical equilibrium in favor of increased metallization due to higher concentrations of reducing gases. It has been determined that this change will not have a negative impact on the overall process economics of the process as the residual char is either recycled as a reductant or preferably gasified to produce useful, gaseous fuel which the steel plant can substitute for expensive natural gas which it buys

The plan is to continue testing in order to develop a practice wherein the sticking problem simply is prevented. It is becoming evident that such practice would have to be computer controlled. Heretofore, all adjustments have been made manually. In view of the process dealing with high temperatures with variables relating to swelling properties of materials charged, pushing pressures, water-cooled lances, oxygen injection through nozzles, foul conditions, adhesive properties of the metallized material, etc., it has been decided that steps will be taken to develop the hardware as well as the software needed to control the process by computer. Such development work will begin next quarter with the objective of putting such control means into use as soon as practical. For this reason an extension of the present contract will be requested soon at no additional cost to DOE or U.S. Steel. Calderon

is convinced that its process will deliver the results as agreed and it is accepting to advance the funds needed to bring the process to a successful conclusion.

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. Injection Ring for Oxidant**



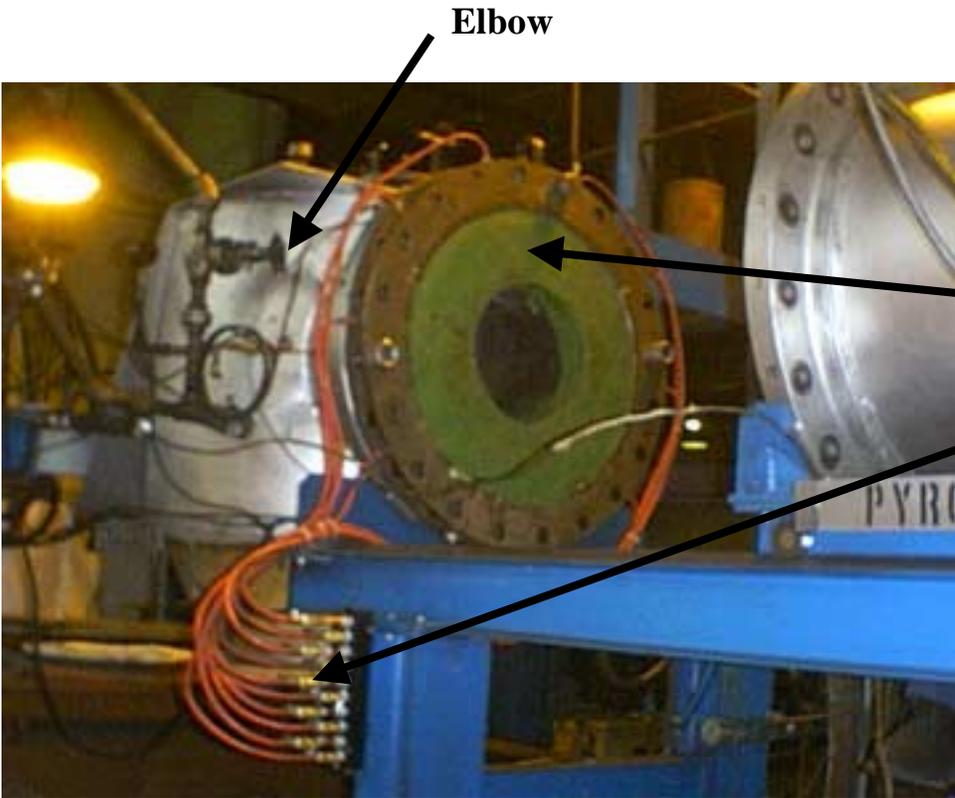
**2. Ports of Ring**



**3. Injection Ring Lined**

**Ports Through Lining**



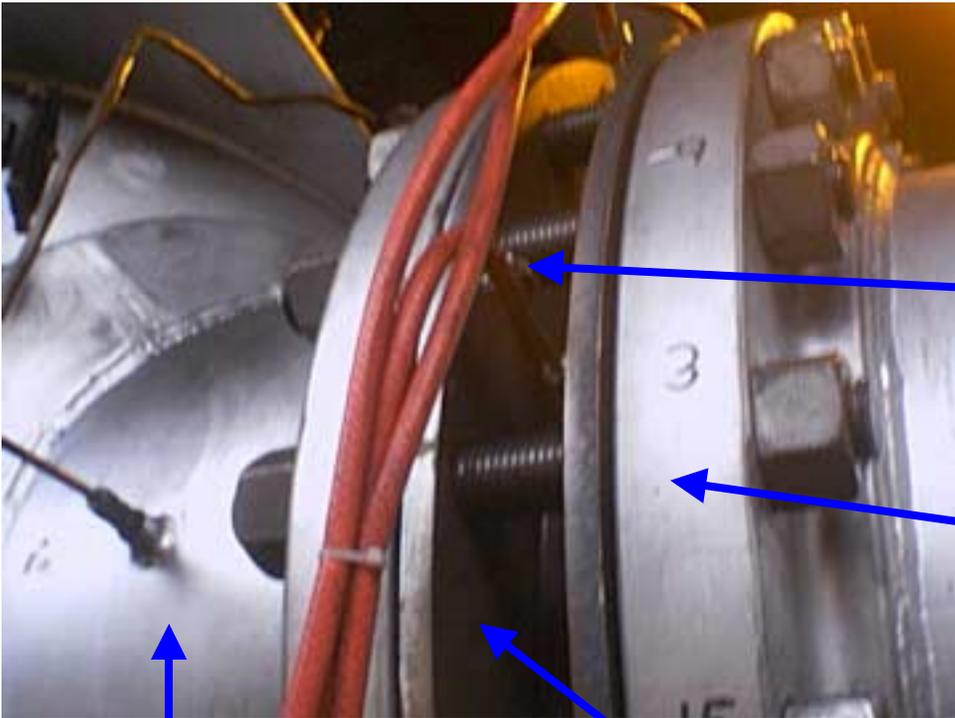


**Elbow**

**4. Injection Ring  
Positioned Against  
Elbow**

**Ring**

**Manifold with  
Individual Leads  
Equipped with  
Valves**



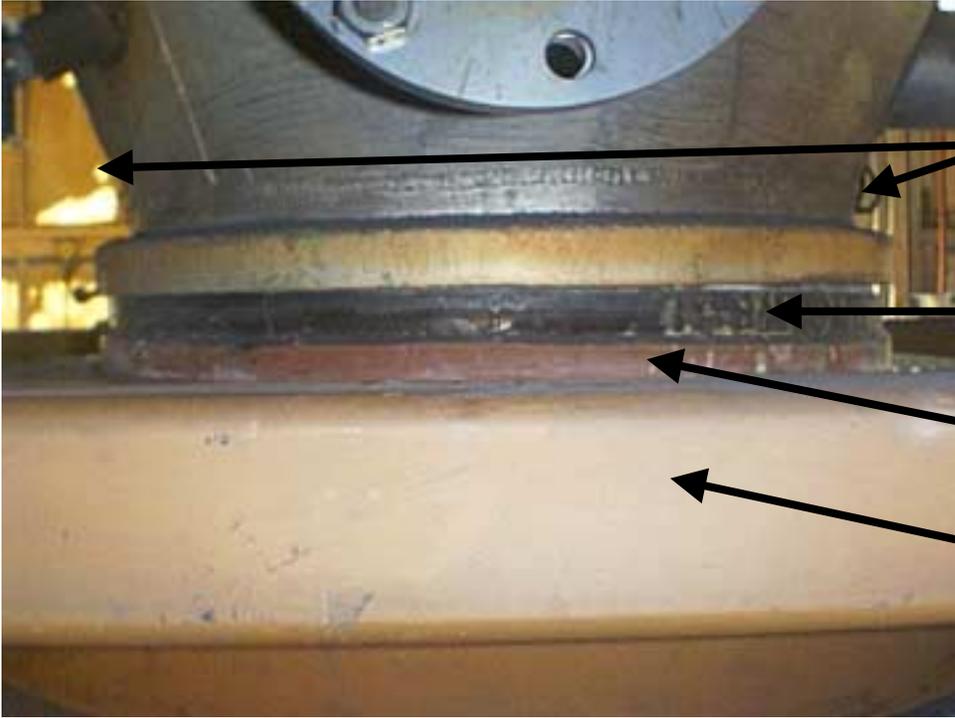
**5. Ring Sandwiched  
Between Reactor &  
Elbow**

**Port**

**Reactor**

**Elbow**

**Ring**



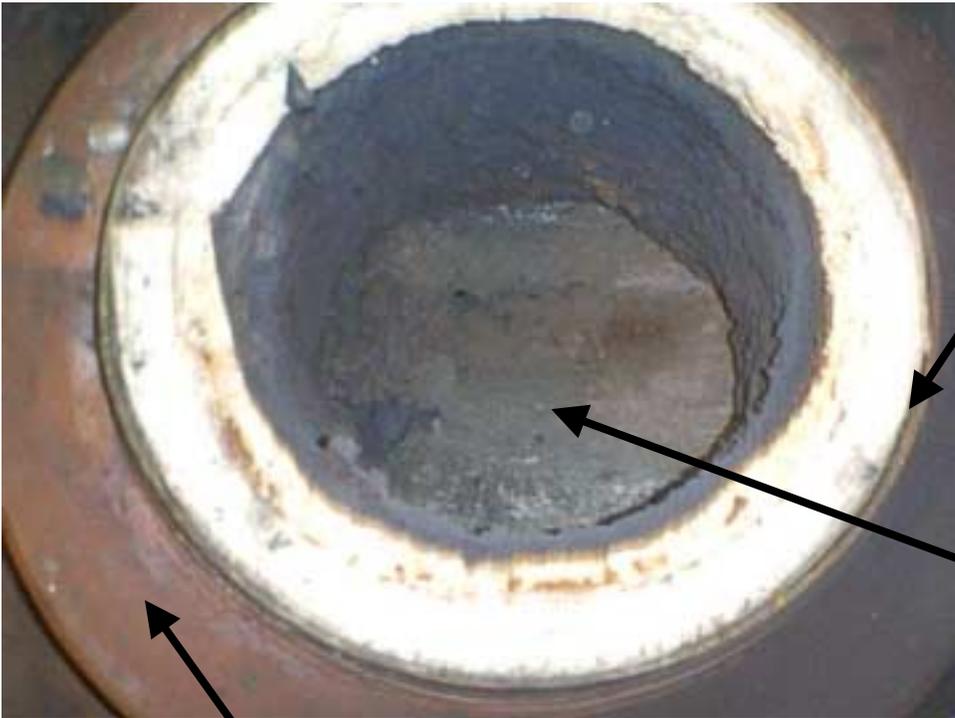
**6. Gas Exhaust Spool**

**Converging Sides**

**Sealing Ring**

**Transition**

**Sliding Gate Housing**



**7. Transition & Sliding Gate (seen from above)**

**Sealing Ring**

**Sliding Gate (closed position)**

**Transition**



**8. Gas Exhaust Spool with Vertical Walls**

Form for Installation of Insulation Lining

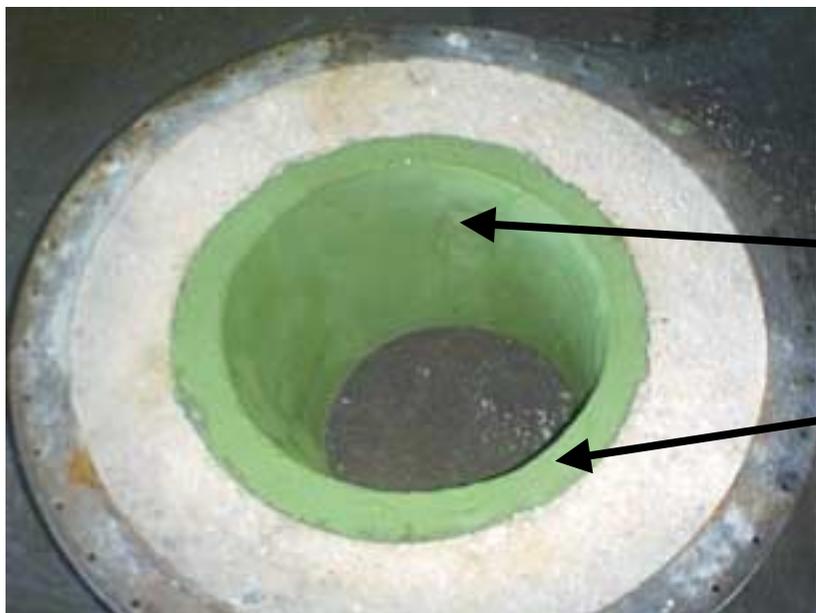
Shell



**9. Gas Exhaust Spool with Insulation Lining In Place**

Exhaust Port

Vertical Wall



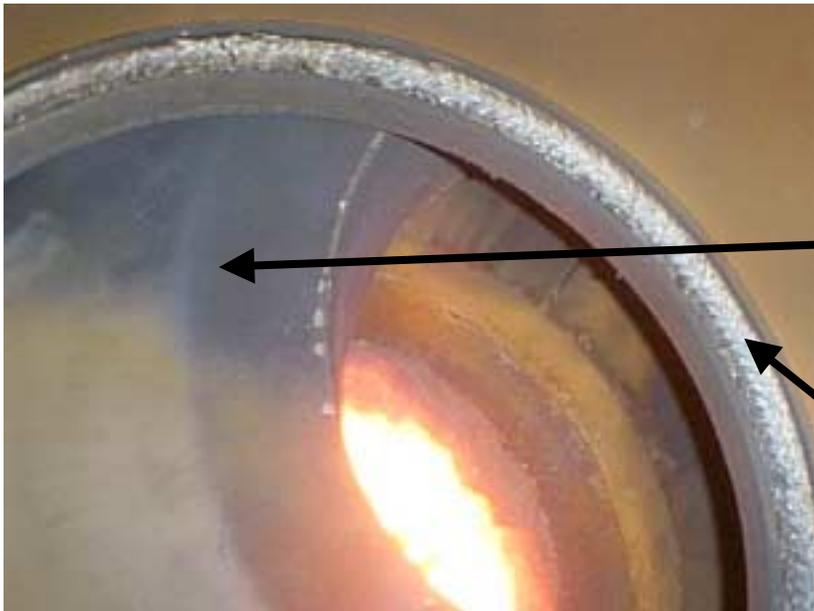
**10. Gas Exhaust Spool with Refractory In Place**

Exhaust Port

Ruby Refractory



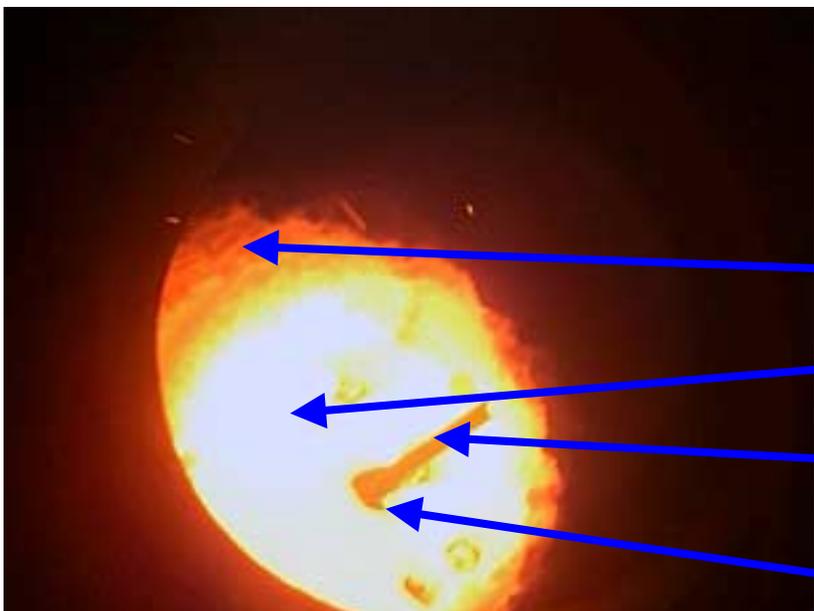
**11.** Internal view of the Gas Exhaust Spool as seen from clean-up port (port cover removed)



**12.** Partial internal view of discharge end as seen from beneath Slide Gate

Slide Gate (partially opened)

Circumferential Seal



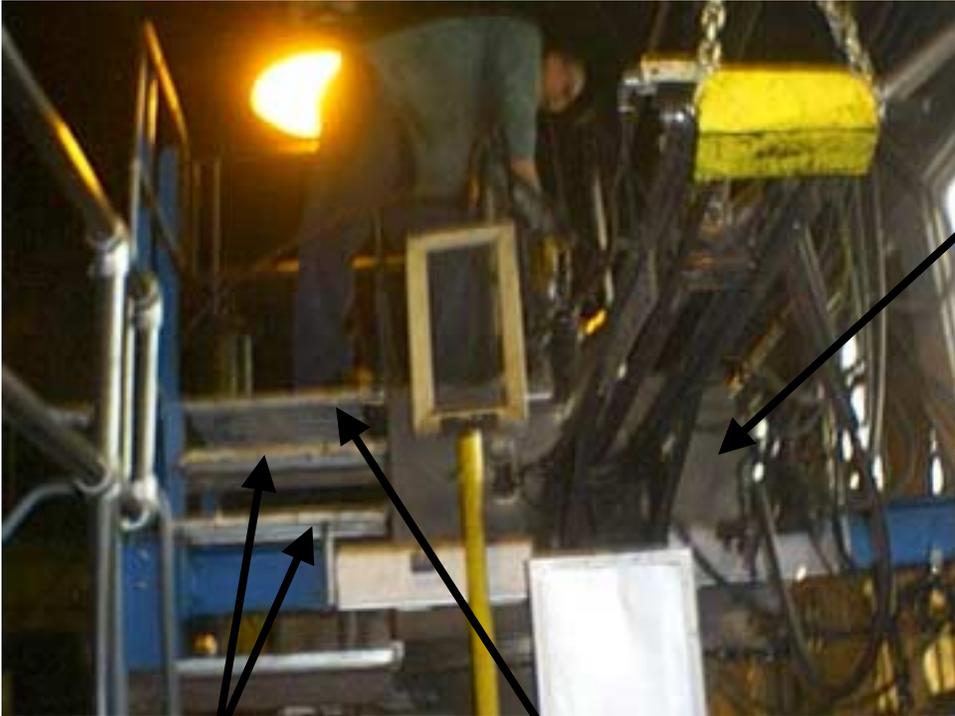
**13.** Internal view of discharge end as seen from beneath of Slide Gate (open condition)

Minor Build-Up

Ceiling of Elbow

Water-Cooled Horizontal Oxygen Lance

Entry Port for Lance

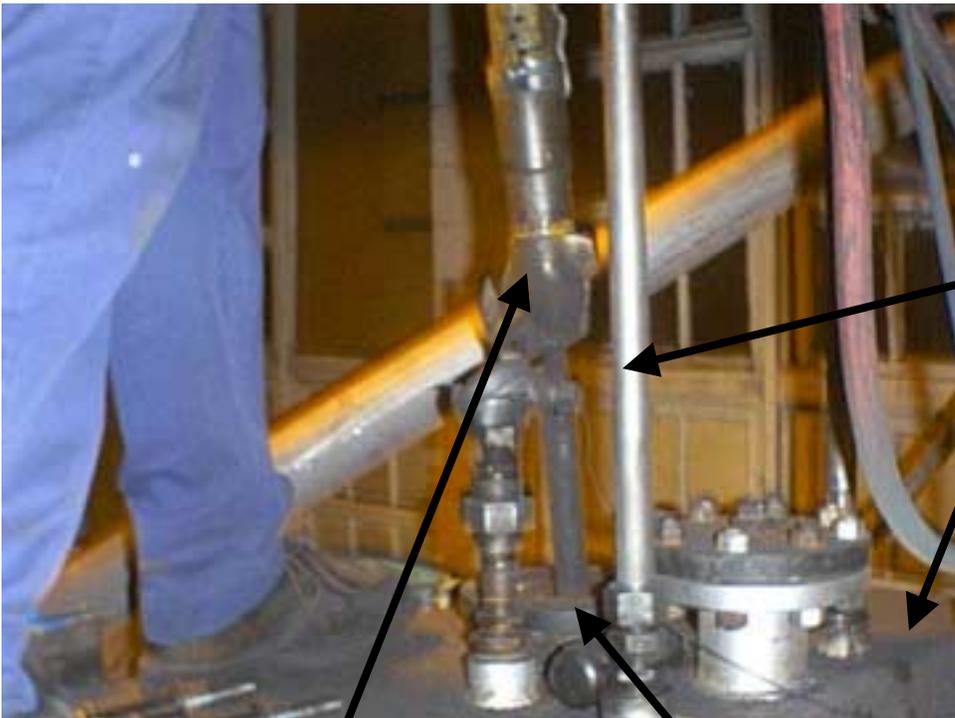


**14. Platform Erected above Elbow**

**Stairs**

**Platform**

**Elbow**



**15. Access Port for Dislodging Stuck Material**

**Pneumatic Jackhammer**

**Port**

**Vertical Lance**

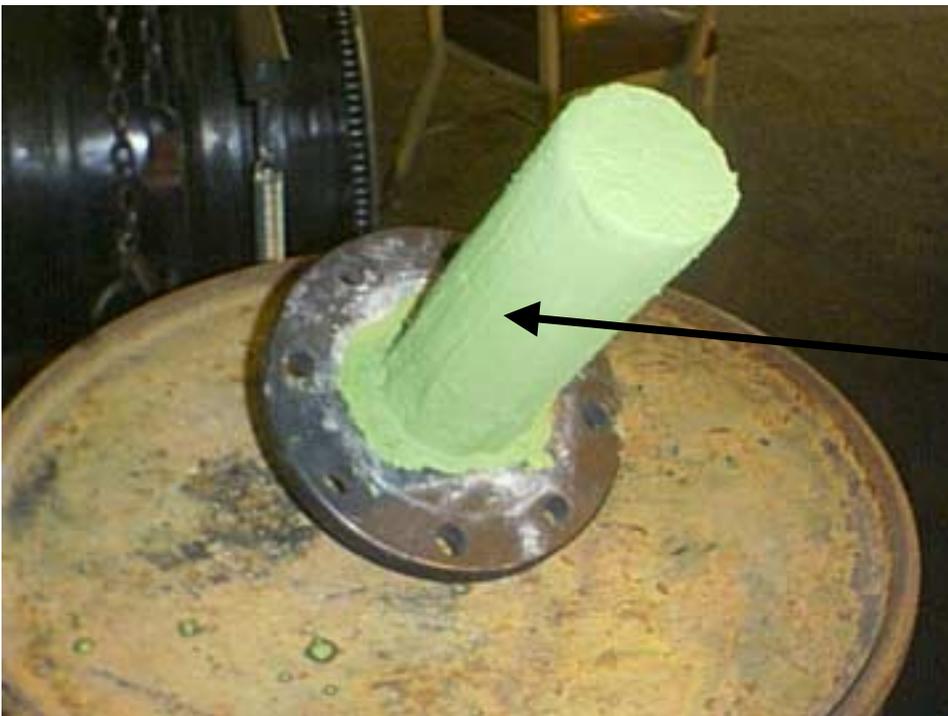
**Elbow Roof**



**16. Cap to Cover Access Port**

**Barbed Stem**

**Blind Flange**



**17. Cap to Cover Access Port with Refractory Lining**

**Refractory with Inbedded Barbed Stem**



**18. Stainless Detection Mirror**



**19. Worker Extracting Refractory Lined Cap**

**Reactor**

**Elbow**

**Jackhammer**



**20. Pneumatic Jackhammer in Action**

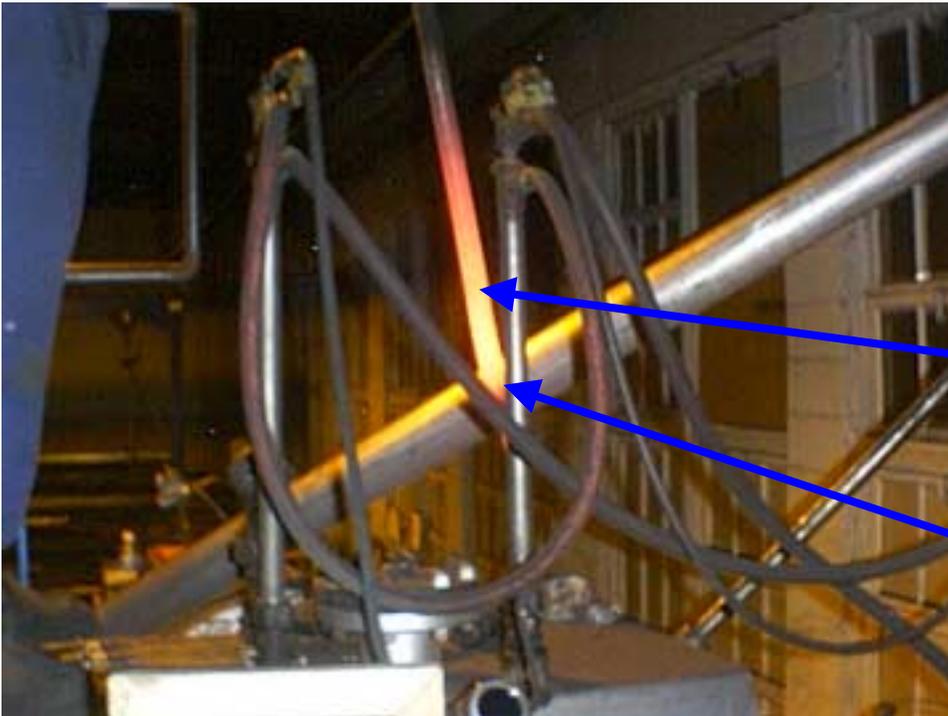
**Tongue of Flame Exiting Through Exhaust Port**

**Platform**

**21. Chisel of Jackhammer Being Withdrawn from Access Port**

**Red Hot Chisel**

**Blunt End**





**22.** Hot metallized material made from Ore Concentrate and Coal being removed from collection drum with tongs



**23.** Hot metallized material being transferred to melting furnace



**24.** Heat being worked in melting furnace



**25.** Indicator showing tapping temperature of 2927° F of steel heat



**26.** Tapping the heat made exclusively from Ore Concentrate reduced with coal

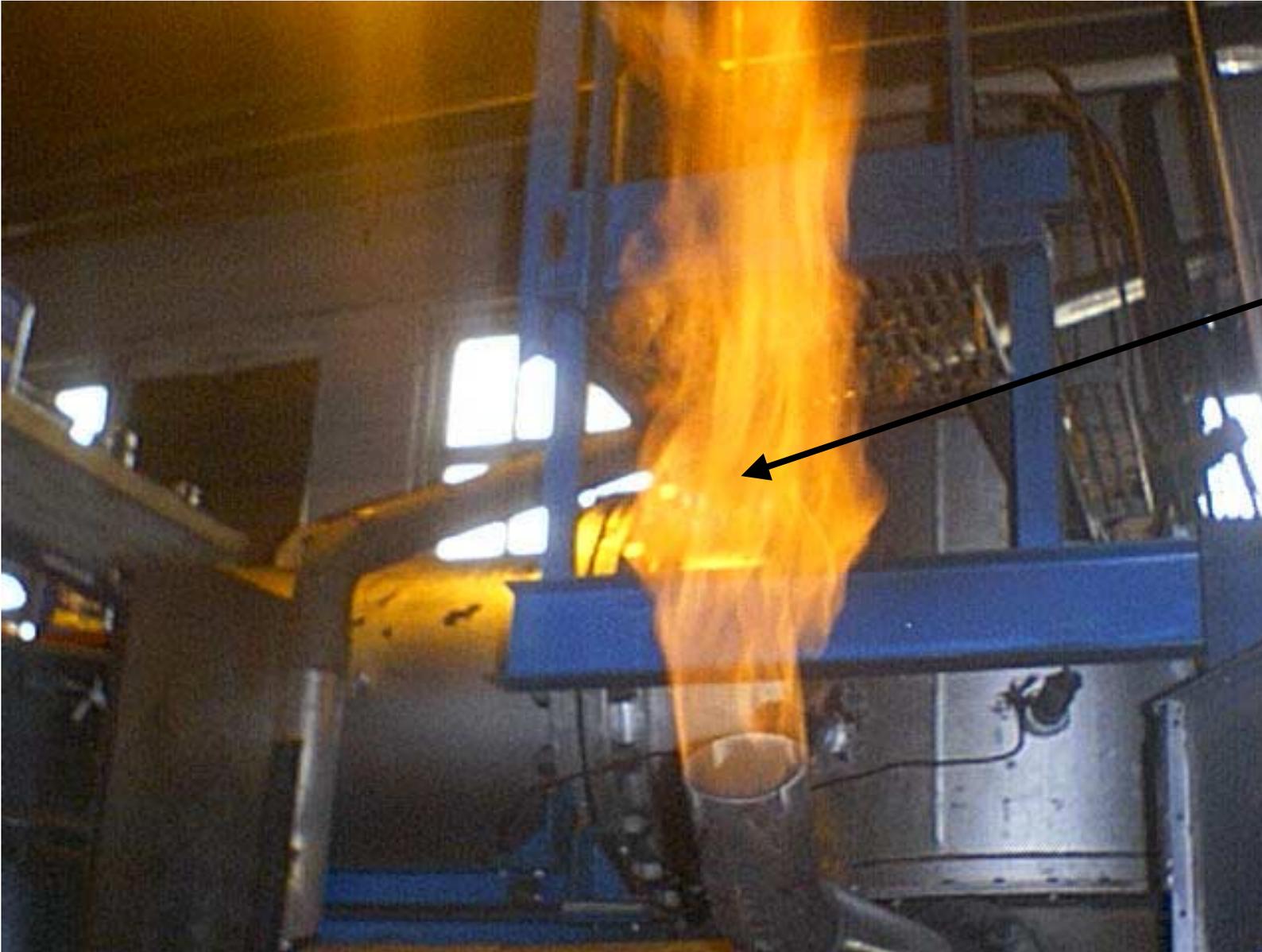
Molds on turntable used to accept the molten steel



**27.** Tap of heat completed

Melting furnace (tilted position)

28.



**Syngas  
Pyrolyzed from  
High Sulfur  
Coal Devoid of  
Hydrocarbons**