Production of Aluminum-Silicon Alloy and Ferrosilicon and Commercial Purity Aluminum by the Direct Reduction Process

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Work Performed Under
Contract EC-77-C-01-5089

Third Interim Technical Report, Phase B
for the Period 1979 March 01 - 1979 May 31

1979 June
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PRODUCTION OF ALUMINUM-SILICON ALLOY AND FERROSILICON
AND COMMERCIAL PURITY ALUMINUM BY THE
DIRECT REDUCTION PROCESS

THIRD INTERIM TECHNICAL REPORT, PHASE "B"
FOR THE PERIOD 1979 MARCH 01 - 1979 MAY 31

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JUNE 1979

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ALCOA LABORATORIES
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FORWARD

This is the third interim technical report, Phase "B", submitted in accordance with the requirements of Contract No. EC-77-C-01-5089, a three-year cost-sharing agreement between the Department of Energy and Alcoa. The report describes work performed in the third quarter of the second year of the program.
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ABSTRACT

Phase "B" of a three year cost-sharing contract between the Department of Energy and Alcoa was started on 1978-09-01. Phase "B" is 16 months long and will include four quarterly reports plus an annual report. At the end of the ninth month the program for Phase "B" is estimated as 41.7% complete with 56.6% of the funding expended.

The effect of volatile sweep rate on bridge formation and product yield was determined in the bench reactor. Higher volatile rates resulted in decreased yields and wider zones of condensation in the shaft. Attempts to charge ores and reduction carbon separately produced very low yields of metallic alloy. Remelt tests of reactor alloy products required temperatures of 1900°C for samples high in Ti. Low Ti-containing alloys melted at 1300°C. Operation of the O₂ injected, side entry bench reactor was erratic and inconsistent due to formation of chemical burden bridging. Ferrosilicon alloy products containing 2 to 14% Al were made in VSR-1 runs with heat of reaction partially supplied by O₂ combustion of metallurgical coke. Tendency for burden bridging was reduced by slipping the furnace and by rodding with a level probe. Alloy product accumulated at the O₂ inlet area due to high heat losses. Cold modeling studies demonstrated that mechanical burden bridging was caused by plugging of the voids with fines generated in the raceway. Proper sizing of coke particles minimized fines formation. Rotating augers were also tested in the cold model as well as at operating conditions in the crucible reactor. The auger successfully kept chemical bridges from forming in these preliminary tests. A char injection system was designed for the VSR unit. Compressive strength and abrasion resistance were measured on calcined briquettes and extrusions containing ores and coke. Briquettes were much stronger; abrasion for both agglomerates was comparable.

Fractional crystallization and remelt experiments were conducted in the bench units to determine the effect of Ti level on the melt properties and the effects of high Fe and high Si on crystallizer products. Higher Si and/or Fe content reduced the recovery of suitable alloy product. The multipurpose high temperature induction furnace was refractory lined, sintered and started up. Fabrication of the holding ladle shell and accessory equipment was completed. Detailed design of several pilot crystallizer components was completed and part of the crystallizer construction materials was received.

Tests were continued to determine the effects of system operating variables on purifier efficiency and productivity. Detailed design of components and procurement of construction materials for the pilot unit were continued.
Primary activities in the fourth three-month period will include: design of a bench reactor for continuous operation with reflux to improve the simulation of the pilot unit; determination of effects of non-stoichiometric carbon on reactor yield; operation of VSR-3 with an auger and supplying all process heat electrically to demonstrate steady state, continuous reduction of high and low Fe burdens; evaluation of ball forming agglomerating techniques; fabrication of pilot crystallizer components and completion of detailed design drawings; lining of the holding ladle and lid; continuation of detailed design and materials procurement for the pilot commercial grade Al purifier, as well as initiation of construction of the pilot unit.
DISCUSSION

The primary objective of the three year program is to demonstrate technical feasibility of a pilot sized direct reduction process for producing aluminum and aluminum-silicon alloy. The process includes three major tasks, reduction to produce impure alloy, alloy purification and purification to commercial grade aluminum. Goals for the second phase are to build and operate a vertical shaft pilot reactor, to modify the reactor design for optimum performance, to complete detailed design, construction and installation of the purification pilot units, and to start up the purification units.

In the third period of Phase "B" Carnegie-Mellon University completed assembly of a high temperature, graphite resistance furnace as well as the control equipment for the furnace operation. Furnace shakedown was initiated. Professor G.K. Sigworth continued to work on thermochemical modeling involving vapor loss reactions and heat flow in the carbothermic reactor. Professor Julian Szekely consulted for 2 days on Alcoa's kinetics modeling program. Koppers Company prepared a complete engineering design for a system to inject char into the Alcoa pilot reactor. Specifications and equipment prices were supplied. Koppers also arranged for shipment of COED char from the Bureau of Mines at Bruceton, PA to Alcoa.

The status of the Direct Reduction project was reviewed for DOE in three separate sessions during the third period.

Subcontract authorization was given by DOE for Koppers Company, Professor Szekely and Carnegie-Mellon University for Phase "B" activities. DOE approval was requested for two additional consultants, Professor T.A. Engh and Professor K. Motzfeldt.

A modification to the contract was implemented in which DOE delegated DCASMA, Pittsburgh as the contract administration office.

Alcoa submitted information to the Office of Inspector General regarding capitalized equipment and government property.

Progress for the three main tasks is reported by sub-task as identified in the modified project outlines submitted on 1978 August 16. It is estimated that for Phase "B" the reduction task is 44% complete, the alloy purification task 47% complete, and the purification to commercial grade aluminum task is 25% complete at the end of the third period.
PROGRESS

A. REDUCTION - PHASE "B"

Task No. 1: Calculate Heat and Mass Balance

The new computer code for calculation of heat and mass balances was completed and has been used in test programs involving silica and carbon.

Task completion is 60%.

Task No. 2: Continue Reaction Kinetic Studies

A series of runs were made to evaluate the effects of volatile flow rate on product yield and solid burden bridging. At low volatile rates the location of bridge formation was a function of temperature gradient rather than flow rate, and was concentrated in a shorter height. As volatile rate increased the condensation of volatiles was spread over greater heights. Product yield as metal collected at the hearth decreased as volatile rates increased.

Task completion is 60%.

Task No. 3: Continue Reaction Mechanism Studies

Several runs were made to determine the effect of carbon concentration and relative location to ores in the burden formulation. Excess carbon resulted in formation of metal carbides. Separation of carbon from ores (i.e. poor mixing) resulted in very low product yields.

Remelt tests of metal-slag mixtures indicated that high concentrations of Ti and/or carbides produced high melting points for the metal portions and made separation of metal from slag difficult.

Task completion is 60%.

Task No. 4: Supply Burden Materials

Several batches of both extrusions and briquettes were prepared for the experimental reactors using different formulations of ores and carbon.

Task completion is 60%.
Task No. 5: Evaluate Burden Preparation Methods

Compressive strength and abrasion resistance were measured on calcined briquettes and extrusions prepared for reactor studies. Briquettes were significantly stronger than extrusions whereas abrasion resistance was similar for both agglomerates.

Task completion is 30%.

Task No. 6: Evaluate Burden Materials

Materials tested included ore extrudates without coke. The extrusions maintained high strength up to 1600°C but did shrink noticeably.

Task completion is 40%.

Task No. 7: Develop Analyses and Properties of Critical Compounds

Some preliminary work was started to evaluate X-ray fluorescence as a bulk analysis method. Comparisons will be made with samples analyzed by microprobe and chemical techniques.

Task completion is 10%.

Task No. 8: Complete Construction of Pilot Reactor, VSR-1

Task completion is 100%.

Task No. 9: Effects of VSR Operating Parameters

Four continuous runs were made using O₂ combustion. The first run was on metallurgical coke. Most of the coke ash was entrained in the exit gases rather than form an oxide slag. The raceway pattern determined by burn-off of graphite pegs in the grate was larger than desired. The second and third runs included ore-coke mixtures. Alloy product was formed in both runs but failed to flow through the grate due to high heat losses in the tuyere-grate region. Investigation of the alloy samples indicated primarily FeSi with 3-4% Ti and C and 2-14% Al. The low Al was due to insufficient temperature and/or residence time. In the fourth run the induction coil was repositioned to provide additional heat below the tuyeres. Heat supply problems still existed, however. During most of the run bed movement was achieved by slipping the burden, i.e. turning off the O₂ flow for short periods. Autopsy of the charge verified that bridging was occurring at several locations up the shaft. Cold modeling studies were continued and demonstrated that mechanical burden bridging was caused by plugging of the bed with
fines generated in the raceway. Proper sizing of coke particles minimizes the formation of fines. The model also showed that raceway size was a direct function of tuyere velocity only at low flow rates. As velocity increased, the coke fines controlled raceway size by restricting further growth.

Task completion is 30%.

Task No. 10: Effects of Operating Parameters in Bench Reactors

A series of runs were made in the oxygen injected bench scale reactor with a side entry tuyere. Various burden formulations and several different carbon types were tested. Operations were hampered by formation of burden bridging in almost every run. An attempt was made to eliminate bridging by increasing the high temperature zone to spread out volatile condensation over more of the bed volume. The run resulted in moving the bridged area higher up the shaft, but condensation still occurred over a short vertical distance and a bridge formed. Larger ore and coke particle sizes also failed to eliminate chemical bridging. Particle size-O₂ flow rate combinations that produced burden movement in the VSR reactor did not prove successful in the bench reactor, probably due to the difference in reactor top temperature.

Task completion is 50%.

Task No. 11: Evaluate Materials of Construction

During operation of VSR-1 the quartz shell developed a number of cracks. Attempts to repair the cracks with high temperature epoxy resulted in severe spalling of the quartz and necessitated trials with possible substitute materials in the interim between receipt of a new quartz unit. Transite pipe was tried but failed in the drying out stage. Fireclay pipe was tested next, but it also failed during the first heat-up. Computer calculations to determine the temperature and heat losses through transite and fireclay verified that transite could not be used and fireclay was limited for the current sized reactor due to a deficiency in insulation.

Task completion is 30%.

Task No. 12: Evaluate Alternative Heating Processes

A solids feeding system and burner for combusting O₂ and powdered char was designed for pilot reactor VSR-1. A paper
study was made to evaluate the effects of preheated and unheated char as a replacement for metallurgical coke charged to the reactor for heating purposes. Replacing coke with char results in an increase in carbon consumption and sweep gas generated in the reactor. The ability to preheat the char and $O_2$ reduce these disadvantages.

Task completion is 30%.

Task No. 13: Modify VSR Design

In order to improve the temperature profile of VSR-1 one of the induction coils was located below the $O_2$ tuyeres. This reactor configuration was denoted as VSR-2. To assist in burden movement a rotating auger was designed and tested in both a cold model and at temperature in the crucible reactor. The addition of the auger to VSR-2 was acknowledged as VSR-3. In this quarter a more efficient dust collection system was designed to process reactor off gases.

Task completion is 50%.

Task No. 14: VSR Design Modification Construction

A new quartz shell was installed to facilitate the VSR-2 modification of tuyere-coil location. The auger system including drive motor, shaft and auger was installed in VSR-3. Construction of the new dust collector was started. A microcomputer control was developed for the solids charging system.

Task completion is 35%.

Task No. 15: Develop Ore Beneficiation Methods

This task was not started yet.

Task completion is 0%.

Task No. 16, Supportive Phase Identification and Task No. 17, Supportive Analytical continued during this quarter.

Task completion is 55%.
B. ALLOY PURIFICATION - PHASE "B"

Task No. 1: Support Pilot Plant Operations

An insulating material for the pilot alloy purification unit was tested in a bench scale furnace at operating conditions. In particular, effects of compression on material integrity and life were determined.

Task completion is 50%.

Task No. 2: Determine Cooling Effects on Morphology

No progress to report.

Task completion is 0%.

Task No. 3: Establish Effect of Impurities

Nine runs were made investigating the effects of various concentrations of Ti, Fe and Si in the charge alloy. In the quaternary system Al-Si-Fe-Ti, the range of Ti contents studied resulted in about 35% product recovery. High Fe in the Al-Si-Fe system concentrates as an intermetallic formed at specific temperature ranges. Combinations of high Si and high Fe reduced alloy recovery below 35%.

Task completion is 50%.

Task No. 4: Fabricate and Install Multipurpose Furnace

The pilot melting furnace was lined.

Task completion is 100%.

Task No. 5: Install Furnace Instruments and Controls

Task completion is 100%.

Task No. 6: Fabricate, Install Crystallization Vessel and Heating System

The induction coil was received. Detailed design of the crystallizer assembly was completed. Refractory lining and insulation materials plus some structural steel members were received.

Task completion is 45%.
Task No. 7: Fabricate and Install System

Design drawings were completed and an order placed for fabrication.

Task completion is 25%.

Task No. 8: Fabricate and Install Cooling System

Design drawings were completed.

Task completion is 25%.

Task No. 9: Fabricate and Install Product Removal System

Fabrication of the holding ladle shell, lid, trunion and combustion system platform were completed. Refractory lining materials were received.

Task completion is 50%.

Task No. 10: Install Crystallizer Instruments, Controls

No progress to report.

Task completion is 0%.

Task No. 11: Start and Shake Down Multipurpose Furnace

The induction power supply and furnace were tested. Sintering of the furnace lining was completed.

Task completion is 75%.

Task No. 12: Start and Shake Down Crystallization System

No progress to report.

Task completion is 0%.

Task No. 13: Supportive Analysis

Chemical analyses were provided for starting alloys, product drains and remelt cuts from bench scale runs.

Task completion is 60%.
Task No. 14: Supportive Mechanical Engineering

Engineering drawings of crystallizer details were continued.

Task completion is 95%.

Task No. 15: Supportive Phase Analysis

Phase analyses were obtained for several samples from the bench scale runs using scanning electron microscopy and electron microprobe. Volume fractions of phases present were also completed.

Task completion is 55%.

Task No. 16: Supportive Facilities Engineering

All subcontractor work on site preparation was completed in this quarter.

Task completion is 100%.
C. PURIFICATION TO COMMERCIAL GRADE ALUMINUM - PHASE "B"

Task No. 1: Support Pilot Plant Work

Tests were continued to determine the effects of operating variables on unit operation. Five runs were made to define the optimum unit configuration for highest efficiency and productivity at the lowest energy requirement.

Task completion is 60%.

Task No. 2: Continue Evaluation of Types

Attempts to operate the large bench scale unit for long run times failed again due to a leak-out of molten material through the container, causing failure of the electric furnace. Repair of the unit was initiated.

Task completion is 40%.

Task No. 3: Fabricate, Install Unit

Detailed design work and procurement for the pilot unit was continued. Several critical materials were received and firm delivery dates were established for other long lead items.

Task completion is 20%.

Task No. 4: Fabricate, Install Residue Removal System

Several alternative protective coatings were considered for the residue removal container.

Task completion is 15%.

Task No. 5: Fabricate, Install Product Removal System

No progress to report.

Task completion is 10%.

Task No. 6: Fabricate, Install Alloy Charging System

The pilot crystallizer holding ladle will be used to charge alloy during the start-up of the aluminum purifier. Design work has not started yet on a smaller capacity molten metal transfer system.

Task completion is 15%. 
Task No. 7: Install Instruments and Controls

No progress to report.

Task completion is 10%.

Task No. 8: Fabricate, Install Handling System

Design of the system was continued.

Task completion is 10%.

Task No. 9: Start and Shake Down System

No progress to report.

Task completion is 5%.

Tasks No. 10 and 11: Supportive Analytical and Supportive Phase Identification are 55% complete.
PHASE "B" FOURTH PERIOD PROGRAM

Technical

Reduction: The reaction kinetics modeling program will be applied to Si production as a test of the computer program. Fundamental studies will be continued on phase equilibria of Al-Si-O-C and Al-Si-C. The effects of smaller particle size and non-stoichiometric C on bench reactor operation and yield will be determined. Various burden support materials will be tested. The effect of C on viscosity and melting temperature of alloy product will be measured using synthetic alloys. A continuous operation, refluxing bench reactor will be designed. The pilot reactor (VSR-3) will be operated with a rotating auger and using all electrical heat to demonstrate steady state, continuous operation for both high and low Fe burdens. Detailed material balances will be made. An optical microscope technique will be developed to assist phase identification analyses of reactor products. Burden preparation by ball forming techniques will be evaluated.

Alloy Purification: Tests will be continued on high Fe containing alloys in the bench crystallizer. Reactor product samples will be remelted to correlate melting point with alloy concentration. Component parts of the pilot crystallizer will be fabricated. Combustion systems will be designed and installed. The holding ladle construction including a gas-fired lid will be completed. All design detail drawings will be completed.

Purification to Commercial Grade Aluminum: The bench scale units will be operated to continue evaluating effects of operating parameters as well as several methods for heating the pilot unit. Detailed design of the pilot system and procurement of materials will be continued. Construction of the pilot unit will be initiated.

COST SUMMARY

Expenditures for the third three months of Phase "B" totaled $423,886. Distribution was $228,424 for reduction, $159,354 for alloy purification, and $36,108 for purification to commercial grade aluminum. Total cumulative spending through the first 21 months of the contract was $2,212,653. Actual spending for Phase "B" is compared to estimated spending in Figure 1.
ASSIGNED PERSONNEL

The actual man-hours expended by engineers and technicians for the third three months of Phase "B" are shown in Table 1 and compared to estimated man-hours. Through the first nine months cumulative engineer man-hours were 7.0% above estimate. Cumulative technician man-hours were 2.8% above estimate. Total cumulative man-hours were 4.4% above estimate.

TASK/MILESTONE SCHEDULE

Attachment 2 shows the task-time relationship for the 3 major tasks from initiation of the current contract in DOE fiscal year 1977, 4th quarter, through completion of the current contract in DOE fiscal year 1981, first quarter. Tasks for proposed extended phases through demonstration of the process are also indicated. Completed milestones are noted by filled in circles.
### TABLE 1

**Summary of Man-Hours Expended**

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**Phase A**

- FY 77: Design pilot
- FY 78: Start-up
- FY 79: Pilot run
- FY 80: Pilot run
- FY 81: Pilot run
- FY 82: Pilot run
- FY 83: Pilot run
- FY 84: Pilot run
- FY 85: Pilot run
- FY 86: Pilot run
- FY 87: Pilot run
- FY 88: Pilot run
- FY 89: Pilot run
- FY 90: Pilot run

**Phase B**

- FY 77: Detailed design
- FY 78: Start-up
- FY 79: Pilot run
- FY 80: Pilot run
- FY 81: Pilot run
- FY 82: Pilot run
- FY 83: Pilot run
- FY 84: Pilot run
- FY 85: Pilot run
- FY 86: Pilot run
- FY 87: Pilot run
- FY 88: Pilot run
- FY 89: Pilot run
- FY 90: Pilot run

**Phase C**

- FY 77: Complete demo.
- FY 78: Plant design
- FY 79: Start-up
- FY 80: Plant design
- FY 81: Start-up
- FY 82: Plant design
- FY 83: Start-up
- FY 84: Plant design
- FY 85: Start-up
- FY 86: Plant design
- FY 87: Start-up
- FY 88: Plant design
- FY 89: Start-up
- FY 90: Plant design

*Completed as follows:*