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

Fourth Interim Technical Report, Phase B, June 1—August 31, 1979

By
M. J. Bruno

September 1979

Work Performed Under Contract No. EC-77-C-01-5089

Aluminum Company of America
Alcoa Laboratories
Alcoa Center, Pennsylvania

U. S. DEPARTMENT OF ENERGY

Division of Industrial Energy Conservation

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FOURTH INTERIM TECHNICAL REPORT, PHASE "B"
FOR THE PERIOD 1979 JUNE 01 - 1979 AUGUST 31

MARSHALL J. BRUNO

SEPTEMBER 1979

ALUMINUM COMPANY OF AMERICA
ALCOA LABORATORIES
ALCOA CENTER, PA 15069

PREPARED FOR THE
DEPARTMENT OF ENERGY
OFFICE OF THE ASSISTANT SECRETARY FOR
CONSERVATION AND SOLAR APPLICATIONS
DIVISION OF INDUSTRIAL ENERGY CONSERVATION
UNDER CONTRACT EC-77-C-01-5089

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This is the fourth 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 fourth quarter of the second year of the program.

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FIGURE

1	PHASE "B" COST CHART

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 twelfth month the program for Phase "B" is estimated as 59.2% complete with 80.9% of the funding expended.

A series of calculated energy balances were made on alternate thermal processes for Al and Al-Si production. The computer modeling program was checked for validity by modeling low shaft Si production and comparing to known commercial conditions. Burden materials ground to -65 mesh prior to agglomeration compared favorably to -200 mesh grinds. Ball forming agglomeration was developed for bauxite-clay-coke mixes. The balls produced had higher strength and abrasion resistance than extrusions made from the same formulations. It was determined that Neutron Activation Analysis for Al, Si, Fe and oxygen combined with analysis of Ti by atomic absorption and carbon by combustion provided the best analytical procedure for reactor metal samples. A series of induction heated runs were made in VSR-3: continuous, steady-state operation produced significant quantities of coalesced metal alloy; individual metal yields for Al, Si, Fe and Ti ranged from 64 to 93%. The mechanical auger was successfully operated to minimize burden bridging during the induction heated runs. Revisions were made to the VSR burden support grate-liner assembly, the tuyere to shell seals and the auger design, improving reliability of reactor operation. Extensive phase analyses were completed on VSR-3 metal product samples by optical microscope techniques, resulting in identification of phases and understanding of relationships between phases formed.

Bench scale crystallizer tests were made on high Fe alloys. Special remelting procedures did not succeed; however, interrupted crystallization showed promise. Component parts of the pilot crystallizer were fabricated and all lining materials were received. The alloy product holding ladle was lined. All detail drawings for the pilot unit and accessories were completed. Data was developed on six elements/compounds containing Al, Si, Fe and Ti to facilitate quantitative phase analysis by optical microscopy.

Bench scale tests were made on the Al purifier to evaluate a special startup heating technique for the pilot unit. The effects of metal skim on purifier operation were determined. Construction of the pilot unit shell was started. Detailed design was continued. Heat transfer and stress calculations were completed for the pilot system.

Primary activities in the last period of Phase "B" will include: startup and continuous operation of the refluxing bench reactor to study burden formulations, $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios and various coke types; establishment of controlling parameters for optimum pellet formation on a bench scale pelletizer, followed by production and evaluation of pellets in large quantities on a pilot sized pelletizer; completion of the induction heated VSR runs on low Fe_2O_3 burdens and various concentrations of reduction coke; operation of VSR-3 using the oxygen-coke combustion heat source to attain steady-state, continuous operation; completing construction and installation of the pilot crystallizer; completing detailed design and most of the construction of the pilot Al purifier; operation of the bench purifier to determine the effect of metal head on unit performance.

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 fourth period of Phase "B" Carnegie-Mellon University completed shakedown of their high temperature furnace. Professor Julian Szekely consulted 5 days on kinetics modeling and process energy balances. Professor K. Motzfeldt initiated experiments to determine the phase diagram of the Al-Si-C system, including the solubility of carbon in liquid metal alloy. Professor T.A. Engh consulted for 2.5 months on the carbothermic reduction task including the following: reduction of coke in the alloy production stage; utilization of electric heat in the high temperature zones; mathematical modeling of the reaction system.

The Department of Energy approved subcontracts for Professors Engh and Motzfeldt and retained Professor J.F. Elliott as a technical monitor of the Direct Reduction contract.

The status of the project was reviewed with the DOE project manager twice during this period.

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 66% complete, the alloy purification task 59% complete, and the purification to commercial grade aluminum task is 39% complete at the end of the fourth period.

PROGRESS

A. REDUCTION - PHASE "B"

Task No. 1: Calculate Heat and Mass Balance

Coding was initiated on a scaled-down version of the kinetic program and will be used to calculate equilibrium heat and mass balances. A series of energy balances on alternative thermal processes for production of Al and Al-Si were completed.

Task completion is 75%.

Task No. 2: Continue Reaction Kinetic Studies

The computer kinetic modeling program was revised to expand the range of rate equations it can use and to consider external heating and/or cooling. A model was completed for silicon production in a low shaft furnace as a means of checking the mechanics of the modeling program.

Task completion is 75%.

Task No. 3: Continue Reaction Mechanism Studies

A series of runs were made in the bench reactor to compare several candidate materials as a "deadman" for the shaft reactor. Variables of bulk density, surface area, geometric shape and surface tension (between molten alloy and the material) were evaluated.

The effects of burden particle size, agglomerate shape and carbon stoichiometry on bench reactor performance were determined in CO sweep type runs. Burden materials ground to -65 mesh prior to agglomeration reacted similarly to agglomerates made from -200 mesh blends. Agglomerate shapes including extrusions, balls and briquettes produced similar product yields and burden movement during reaction. Results for substoichiometric carbon runs were not conclusive.

Further remelt tests of VSR alloy products were made during this period. Results similar to previous tests were obtained. Alloy collected in the VSR product crucible melted in the range of 1200°-1300°C. This alloy contained 3 to 5% Ti and was the only sample with high Ti levels to remelt at a reasonably low temperature, indicating the importance of other interactions on melting characteristics such as Fe or C, or rate of cooling. Alloy recovered from the same VSR

run external to and below the product crucible had a much higher melting range, 1300-2200°C, and analyzed as high in Fe, C and Ti.

Task completion is 80%.

Task No. 4: Supply Burden Materials

Burden materials were prepared for both the bench and pilot reactors, including extrusions and balls. About 700 lb of calcined metallurgical coke was crushed and screened to be used as heating coke in VSR tests.

Task completion is 75%.

Task No. 5: Evaluate Burden Preparation Methods

Ball forming agglomeration was developed for bauxite-clay-coke blends using an Eirich mixer. Calcining tests on these balls indicated that fast heat-up to end temperature produced agglomerates with higher compressive strengths and abrasion resistance than balls calcined by slow heat-up.

Task completion is 70%.

Task No. 6: Evaluate Burden Materials

Briquettes were made from two blends and compared. Burden containing bauxite-clay-coke-hematite were stronger and more abrasion resistant than briquettes of alumina-quartz-coal-hematite. Balls and extrusions were prepared from a 10% Fe_2O_3 formulation. The balls were superior in strength properties. Abrasion resistance was measured in a modified tumbler test. A series of burdens made from particles ground to -200 mesh--including extrusions, briquettes and balls--were tested in the bench reactor. All three agglomerates retained their strength after exposure to reactor environment.

Task completion is 80%.

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

Alternate methods were investigated to more rapidly analyze product samples from VSR runs. The best procedure appears to be analysis of Al, Si, Fe and oxygen by Neutron Activation Analysis plus total carbon by combustion and Ti by Atomic Absorption. Quantometer dilution remelt and X-ray fluorescence analyses were not successful for VSR samples due to the high melting point and X-ray absorption properties of the alloy.

Task completion is 80%.

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

Task completion is 100%.

Task No. 9: Effects of VSR Operating Parameters

A series of runs were made in VSR-3 using 100% induction heat. The objective of the runs was to establish steady-state operation with continuous metal production. CO sweep was introduced in several runs. The burdens charged contained both high (~12%) and low (~2%) concentrations of Fe_2O_3 and less than stoichiometric concentrations of reducing carbon. Silica to alumina weight ratios ranged from 0.51 to 0.72.

This series of runs demonstrated the following: metal could be produced continuously at steady state; substoichiometric carbon affected metal yield and degree of unreacted burden; in carbon deficient charges, Al_2O_3 slag was formed; the alloy produced appeared to be fluid; the bulk of reaction took place adjacent to the reactor walls in an electrically heated (by induction) mode of operation.

Material balances were determined for Al, Si, Fe and Ti and metal yields were calculated. Metal balances ranged from 85 to 98%. Metal yields varied from 64 to 93%.

Performance of the mechanical auger was monitored during the runs. Start-up and shutdown procedures were established to avoid premature failure of the auger. The auger was successful in minimizing burden bridging for induction heated runs with no excess upward gas flow.

Task completion is 70%.

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

Several runs were made in a special bench reactor containing a series of pressure taps along the vertical distance to obtain a pressure profile during operation. The objective of this set-up was to monitor bridge formation with respect to run conditions of time and temperature. The data showed the expected trend of decreasing pressure toward the top of the reactor. In one run an indication was given that a bridge was forming but the results were not conclusive. It was decided that the pressure data did not provide a good indication of bridging in the bench reactor. In one run it was demonstrated that alloy was produced and separated from the burden in an environment containing both excess heating coke and oxygen.

Task completion is 75%.

Task No. 11: Evaluate Materials of Construction

No progress.

Task completion is 30%.

Task No. 12: Evaluate Alternative Heating Processes

No Progress.

Task completion is 30%.

Task No. 13: Modify VSR Design

A number of modifications were planned and implemented to improve operation of VSR-3. The first long term continuous run demonstrated the need for a leakproof grate-liner assembly since a significant amount of alloy product bypassed the crucible hearth and flowed around it via the seam between the grate and the liner. A one-piece assembly was designed to eliminate the seam.

Close fitting water jacketed assemblies were designed to improve sealing between the quartz shell and the tuyere and sight tube ports. The cooling water maintained the sealing properties of the silicone rubber material used to effect the seals.

The rotating mechanical auger installed in VSR-3 to aid burden movement went through several design changes to obtain improved strength and reduced tendency for transporting particles and mixing burden layers.

Task completion is 75%.

Task No. 14: VSR Design Modification Construction

The design modifications described in Task No. 13 were implemented. In addition, the reactor tube was replaced with a better grade of graphite, which enhanced the induction capabilities of the system. The induction controls were adjusted to attain more power, increasing the supplied kW to 72 at reaction temperature. The grate location in relation to the tuyere entries was changed several times to accommodate injection of CO for the sweep runs.

Task completion is 70%.

Task No. 15: Develop Ore Beneficiation Methods

No progress.

Task completion is 0%.

Task No. 16: Supportive Phase Identification

Alloy product samples from VSR-3 runs were examined by optical microscopy and electron microprobe to determine the phases present and their relationships. Major phases were identified as FeSi_2Al_4 , Al-Si and SiC. Minor phases included FeSiAl_5 and $\text{Ti}_2\text{Fe}_2\text{Si}_3\text{Al}_3$ (approximate).

Task completion is 75%.

Task No. 17: Supportive Analytical

A large number of samples were analyzed by various techniques during this period.

Task completion is 75%.

B. ALLOY PURIFICATION - PHASE "B"

Task No. 1: Support Pilot Plant Operations

No progress. Task completion is 50%.

Task No. 2: Determine Cooling Effects on Morphology

No progress. Task completion is 0%.

Task No. 3: Establish Effect of Impurities

Experiments were continued in the bench crystallizer on high Fe alloys. Remelt cuts from several previous tests were combined and recrystallized. The procedure did not favorably redistribute the concentration of Fe in the alloy drained. Another procedure for handling high Fe alloy was more positive. In this case, crystallization was interrupted at certain temperatures to avoid peritectic reactions. The Fe was concentrated in a high temperature cut.

Task completion is 75%.

Task No. 4: Fabricate and Install Multipurpose Furnace

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

Molds were built for casting the pilot crystallizer taphole and top plate. All refractory and lining materials have been received. The crystallizer lid with gas-fired heating was fabricated and lined.

Task completion is 60%.

Task No. 7: Fabricate and Install System

Fabrication of the system was completed except for one control unit.

Task completion is 90%.

Task No. 8: Fabricate and Install Cooling System

The cooling system fabrication was completed.

Task completion is 100%.

Task No. 9: Fabricate and Install Product Removal System

The holding ladle was lined with refractory. Combustion piping of the lid burners was started.

Task completion is 80%.

Task No. 10: Install Crystallizer Instruments, Controls

Installation of controls was started.

Task completion is 20%.

Task No. 11: Start and Shake Down Multipurpose Furnace

Task completion is 100%.

Task No. 12: Start and Shake Down Crystallization System

No progress. Task completion is 0%.

Task No. 13: Supportive Analysis

Chemical analyses were completed for bench scale runs during the quarter.

Task completion is 75%.

Task No. 14: Supportive Mechanical Engineering

All drawings were completed.

Task completion is 100%.

Task No. 15: Supportive Phase Analysis

Six samples were analyzed for phases using electron microprobe, followed by determination of volume percents by Modal

analysis. Data was developed on 6 elements/compounds containing Al, Si, Fe and Ti to permit routine identification by their optical properties.

Task completion is 75%.

Task No. 16: Supportive Facilities Engineering

Task completion is 100%.

C. PURIFICATION TO COMMERCIAL GRADE ALUMINUM - PHASE "B"

Task No. 1: Support Pilot Plant Work

A number of bench experiments were directed toward fundamental aspects relating to operation of the pilot unit. These tests included evaluation of the effect of commercial grade purity components on metal purity and demonstration of a special heating technique for start-up mode.

Task completion is 80%.

Task No. 2: Continue Evaluation of Types

Bench runs were made to determine the effect of skim on critical components of the unit. Although the unit efficiency was reduced slightly, design features of the pilot unit should handle any skim problems. The life of a critical material of construction was retested in an extended bench test lasting 22 days. Results verified previous evaluations.

Task completion is 80%.

Task No. 3: Fabricate, Install Unit

Construction was started on the unit steel shell. Additional refractory and insulation lining materials were received. Detailed design of unit components was continued. Unit heat transfer calculations were revised. Stress analysis calculations were made for critical materials.

Task completion is 35%.

Task No. 4: Fabricate, Install Residue Removal System

Techniques for sealing the residue removal chamber as well as critical dimensions of the chamber were determined.

Task completion is 25%.

Task No. 5: Fabricate, Install Product Removal System

Tapping tube materials, size and location were selected for the pilot unit.

Task completion is 15%.

Task No. 6: Fabricate, Install Alloy Charging System

Design was started on a small capacity molten metal transfer system.

Task completion is 20%.

Task No. 7: Install Instruments and Controls

Access ports were designed into the unit lid to provide capability for measurement of temperature, fluid level and fluid concentration.

Task completion is 15%.

Task No. 8: Fabricate, Install Handling System

Design of the system was continued. A test was initiated on a candidate construction material.

Task completion is 20%.

Task No. 9: Start and Shake Down System

No progress.

Task completion is 5%.

Tasks No. 10 and 11: Supportive Analytical and Supportive Phase Identification are 75% complete.

PHASE "B" FIFTH PERIOD PROGRAM

Administrative

The Baseline Cost Report for DOE fiscal year 1980 will be submitted. Approval to proceed with Phase "C" will be requested. The sub-tasks under the 3 major tasks will be revised. Increased expenditure limits for subcontractors will be requested to cover Phase "C" work. A list of current time and materials subcontracts will be submitted.

Technical

Reduction: The equilibrium heat and mass balance computer program will be revised to incorporate the latest data base. Development of the Al-O-Si-C kinetics model will be continued. The refluxing bench reactor will be started up and operated continuously to more closely simulate the pilot unit. Burden formulations, coke types and $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios will be studied. Burden materials will be prepared on a bench scale pelletizer to establish controlling parameters. Balls will then be produced on a pilot sized disc pelletizer and evaluated for compressive strength and abrasion resistance. The series of pilot VSR runs using all electric heat will be completed, testing low Fe_2O_3 burden formulations and various concentrations of coke. The VSR unit will then be operated using O_2 combustion of coke to supply part of the heat, the mechanical auger to assist burden movement, and a modified induction heating system to supply additional power for offsetting heat losses. The burden support grate position will be varied to obtain molten metal flow to the product crucible. Data from VSR oxygen runs will be used to design an alternate reactor. Process economic studies will be conducted, with emphasis on char generation costs.

Alloy Purification: All phases of design and construction will be continued for the pilot crystallizer. Bench scale units will continue to be operated to evaluate techniques for purifying alloys high in Fe and Ti impurities. Additional VSR alloy product remelts will be made to correlate melt properties with temperature.

Purification to Commercial Grade Aluminum: Design and construction of the pilot unit will be completed. Bench scale operations will be conducted to determine the effect of metal head on unit performance, and to test several materials of construction planned for use in the pilot unit.

COST SUMMARY

Expenditures for the fourth three months of Phase "B" totaled \$502,318. Distribution was \$259,220 for reduction, \$185,640 for alloy purification, and \$57,458 for purification to commercial grade aluminum. Total cumulative spending through the first 24 months of the contract was \$2,714,971. 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 fourth three months of Phase "B" are shown in Table 1 and compared to estimated man-hours. Through the first twelve months cumulative engineer man-hours were 5.0% above estimate. Cumulative technician man-hours were 15.8% above estimate. Total cumulative man-hours were 11.5% 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.

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FIGURE 1 - Phase "B" Cost Chart: Total Expenditures vs. Time

TABLE 1

Summary of Man-Hours Expended

	Months 1-3 Man-hr		Months 4-6 Man-hr		Months 7-9 Man-hr		Months 10-12 Man-hr		Months 13-16 Man-hr		Cumulative Man-hr		Cumulative Per Cent Deviation
	<u>Actual</u>	<u>Est.</u>	<u>Actual</u>	<u>Est.</u>	<u>Actual</u>	<u>Est.</u>	<u>Actual</u>	<u>Est.</u>	<u>Actual</u>	<u>Est.</u>	<u>Actual</u>	<u>Est.</u>	
Engineers	3190	2737	3355	3230	3556	3475	3659	3658			13760	13100	+5.0
Technicians	<u>4714</u>	<u>4046</u>	<u>5026</u>	<u>5287</u>	<u>5676</u>	<u>5664</u>	<u>7565</u>	<u>4853</u>			<u>22981</u>	<u>19850</u>	+15.8
TOTAL	7904	6783	8381	8517	9232	9139	11224	8511			36741	32950	+11.5

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Attachment 2

Direct Reduction Project

Task/Milestone Schedule

	FY 77				FY 78				FY 79				FY 80				FY 81				FY 82	FY 83	FY 84	FY 85	FY 86	FY 87	FY 88	FY 89	FY 90
	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4									
					PHASE A				PHASE B				PHASE C																
Contract Procurement				▲				▲									▲											▲	
				Contract Awarded				Rev. Work Statement Approval for B																					
Reduction				▲	●		●		●	●	●		○				○	▲			▲		▲	▲	▲			▲	
				Compt. Progn. Heat, Mass Bal.			Designed Pilot VSR-1		Installed Pilot VSR-1	VSR-1 Start-up	VSR-2 Start-up		VSR-3 Start-up				Scale-up Design			Complete Dem. Plant Design			Dem. Plant Installation, Start-up	Start comm. Plant Design	Start comm. Plant Install.		Complete comm. plant install. start-up		
Alloy Purification				▲	●	●	●	●		●	○	○					○	▲					▲	▲	▲			▲	
				Est. Yield, Purity in Existing Pilot		Det. Effects of Impurities	Designed High Temp. Furnace	Designed pilot Unit		Installed High Temp. Furnace	Install Pilot Unit	Pilot System Start-up					Scale-up Design												
Purification to Commercial Purity Al				▲	●				●		●		○	○			○	▲					▲	▲	▲			▲	
				Det Efficiency					Tested Critical Mat'l. of Constr.		Design Pilot Unit	Install Pilot Start up Pilot					Scale-up Design												

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