

LIGHTWEIGHT ALUMINA REFRACTORY AGGREGATE
Final Report

By
T. G. Swansiger
A. Pearson

July 16, 1996

Work Performed Under Contract No. DE-FC07-89ID12903

For
Idaho Operations Office
Idaho Falls, Idaho

By
Aluminum Company of America
Alcoa Center, PA

DISCLAIMER

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

This report has been reproduced directly from the best available copy.

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831; prices available from (423) 576-8401.

Available to the public from the U.S. Department of Commerce, Technology Administration, National Technical Information Services, Springfield, VA 22161, (703) 487-4650.

DISCLAIMER

**Portions of this document may be illegible
in electronic image products. Images are
produced from the best available original
document.**

**LIGHTWEIGHT ALUMINA REFRACTORY AGGREGATE
FINAL REPORT**

By

T. G. Swansiger and A. Pearson

Report No. 06-96-14

1996 July 16

Work Performed Under Contract No. DE-FC07-89ID12903

**Prepared for
Idaho Operations Office**

**Prepared by
Aluminum Company of America
Alcoa Technical Center
100 Technical Drive
Alcoa Center, PA 15069-0001**

1996 July 16
Report No. 06-96-14

Lightweight Alumina Refractory Aggregate

Final Report

Contract No. DE-FC07-89ID12903

Prepared By:
T. G. Swansiger (Technical Specialist)
A. Pearson (Technical Consultant)

ALUMINUM COMPANY OF AMERICA
ALCOA TECHNICAL CENTER
ALCOA CENTER, PA 15069

Prepared For:
Idaho Operations Office
Idaho Falls, Idaho

Sponsored by The Office of the
Assistant Secretary for Energy
Efficiency and Renewable Energy

Office of Industrial Technologies
Washington, DC

ABSTRACT

An Alcoa/DOE contract was formulated, based on a proposal submitted by Alcoa in response to DOE Solicitation for Federal Assistance for Refractory Materials Research and Development. This application was for cost-sharing research for development of new refractory materials or improvements to existing refractory materials, with end-use applications directed toward those refractories used by the aluminum, glass, cement, and iron and steel industries. The specific objective of this contract was to develop a lightweight, high alumina refractory aggregate for use in a variety of high performance insulating (low thermal conductivity) refractory applications.

A new, high alumina, lightweight aggregate process was developed through bench and pilot-scale experimentation, involving extrusion of a blend of calcined and activated alumina powders and organic extrusion aids and binders. The new aggregate with a bulk density approaching 2.5 g/cc exhibited reduced thermal conductivity and adequate fired strength, compared to dense tabular aggregate. Refractory manufacturers were moderately enthusiastic over the preliminary characterization results, especially with respect to reduced thermal conductivity and much greater aggregate strength, compared to existing lightweight aggregates.

Alcoa prepared an economic analysis for the production of lightweight aggregate using previous research results and estimates of production costs, based on a retrofit of this process into existing Alcoa production facilities for dense aggregate. Based on favorable technical and economic results and industrial interest, continuation of the contract to full-scale demonstration (final phase) was warranted if the market survey indicated the need for 20,000 mt/yr plant capacity.

Although the new lightweight aggregate process showed technical and economic promise, the formal market survey was the overriding factor dictating whether to continue with this development into the full-scale demonstration. As the final phase of the contract continued, Alcoa became aware of a new, competing lightweight aggregate material, developed and produced by another company. This competing aggregate had a significantly more favorable cost base than the Alcoa/DOE material, due to cheap raw materials and relatively few processing steps (thermal treatment of aluminum dross residue). In late 1995, Alcoa became a distributor of this new aggregate, named Plasmal.TM Alcoa estimated that 75% or more of the market originally envisioned for the experimental Alcoa/DOE aggregate would be taken by the less expensive PlasmalTM aggregate. Consequently, the remaining market was judged to be insufficient to justify continuation of the Alcoa/DOE contract to the actual full-scale demonstration of the process. This information was discussed with the DOE in 1996 March and it was mutually agreed to terminate this contract in an orderly manner without the full-scale demonstration.

SUMMARY

This contract was based on a proposal submitted by Alcoa in response to DOE Solicitation for Federal Assistance for Refractory Materials Research and Development. This proposal was for cost-sharing research for development of new refractory materials or improvements to existing refractory materials, with end-use applications directed toward those refractories used by the aluminum, glass, cement, and iron and steel industries. The specific objective of this contract was to develop a lightweight, high alumina refractory aggregate for use in a variety of high performance insulating (low thermal conductivity) refractory applications.

Following a comprehensive literature review to assess current state-of-the-art, various forming approaches and ceramic formulations were evaluated in 3 phases of development work characterized by bench-scale, pilot-scale, and full-scale demonstration of the process.

From bench-scale experiments (Phase I), it was determined that a sintered alumina aggregate with a bulk density below 2.5 g/l should be the project goal. Both paste and pelletized formulations were developed and characterized. Preliminary economic analyses for promising approaches were performed. At the conclusion of this phase, it was judged that extrusion was the preferred forming method for the formulation that consisted of a blend of calcined and activated aluminas and organic extrusion aids and binders. It was judged that the technical aspects of the process, preliminary process economics, and the potential market were all favorable enough to justify continuation into the pilot-scale development (Phase II).

In Phase II of the contract, kilogram quantities of materials were produced at the pilot-scale for further testing and performance evaluations that more closely simulated actual application conditions. For the first time, thermal characterization of the new aggregate in a brick or castable end product was performed. As anticipated, thermal conductivity of the new aggregate resulted in significantly improving the insulating properties of the brick or castable. Refractory manufacturers were moderately enthusiastic over the preliminary characterization results, especially with respect to reduced thermal conductivity and much greater aggregate strength, compared to existing lightweight aggregates. Alcoa prepared an economic analysis for the production of lightweight aggregate using previous research results and estimates of production costs, based on a retrofit of this process into existing Alcoa production facilities for dense aggregate. Based on these technical and economic results and industrial interest, continuation of the contract to full-scale demonstration (Phase III) was warranted.

Prior to embarking on the full-scale demonstration, resolution of three remaining technical issues was required. These issues were a) fired density slightly above target, b) poor intermediate strength after binder burnout but before sintering, and c) resistance to penetration by molten steel slag. The first two of these technical issues were addressed and solved by modification of the ceramic formulation. A preliminary indication of the resistance to molten steel slag was assessed in a cup test. Compared to the dense aggregate

control sample, the resistance to penetration exhibited by the lightweight aggregate was slightly less. Process economics were again revisited based on two production levels, up-to-date raw material costs, and the assumption of a retrofit into the Arkansas production facility. For a 10,000 mt/yr production level, a lightweight aggregate selling price of 35% more than T-64 on a volume basis would be required for a favorable return on the capital investment. This 35% value exceeded the contract target of 25%. For a 20,000 mt/yr production level, a lightweight aggregate selling price of 15% more than T-64 on a volume basis would be required for a favorable return on the capital investment.

Although the new lightweight aggregate process showed technical and economic promise, the formal market survey was the overriding factor determining whether to continue with this development into the full-scale demonstration. As the Phase III work continued, Alcoa became aware of a new lightweight aggregate material, developed and produced by another company. This new material exhibited physical properties similar to the Alcoa/DOE experimental aggregate, both being improvements over currently available aggregates. However, this competing aggregate had a favorable cost base, compared to the Alcoa/DOE material, due to cheap raw materials and relatively few processing steps (thermal treatment of aluminum dross residue). In late 1995, Alcoa became a distributor of this new aggregate, named Plasmal.TM In light of this new development, Alcoa estimated that 75% or more of the market originally envisioned for the experimental Alcoa/DOE aggregate would be taken by the less expensive PlasmalTM aggregate. Consequently, the remaining market was judged to be inadequate to justify continuation of the Alcoa/DOE contract to the actual full-scale demonstration of the process. This information was discussed with the DOE in 1996 March and it was mutually agreed to terminate this contract without the full-scale demonstration.

TABLE OF CONTENTS

ABSTRACT	i
SUMMARY.....	ii
TABLE OF CONTENTS.....	iv
OBJECTIVE.....	1
CONTRACT OVERVIEW	2
CONTRACT PHASES	2
Phase I - Literature Review and Bench-scale Experiments	2
Phase II - Pilot-scale Experiments	4
Market Survey for Lightweight Aggregate	5
Phase III - Full-scale Process Demonstration.....	5
Technical Issues Remaining to be Solved	5
Process Economics Feasibility Study.....	6
Full-scale Demonstration Plans	6
Revised Market Survey for Lightweight Aggregate and Project Termination	7
CONCLUSIONS.....	7
REFERENCES	8

OBJECTIVE

The specific objective of this contract between Alcoa and the Department of Energy was to develop a lightweight, high alumina refractory aggregate for use in a variety of high performance insulating (low thermal conductivity) refractory applications. The following specifications were targeted:

- Compared to Alcoa TabularTM alumina, achieve a 30% reduction in particle density from approximately 3.6 to 2.5 g/cc.
- Compared to existing high alumina aggregate (TabularTM/Fused), retain ~50% of the crushing strength.
- Compared to Alcoa TabularTM alumina, develop a lightweight aggregate product having a selling price not to exceed 25% more than the present selling price of TabularTM, on a product volume basis.

Secondary objectives also to be met, some of which impact the primary objectives, included developing an aggregate that: (a) was 99+ % alumina, (b) possessed thermal stability similar to TabularTM, and (c) was process compatible with existing tabular production facilities.

Specific goals for this contract dealt with bench-scale and pilot-scale work to demonstrate further the feasibility of producing lightweight aggregates. The term 'feasibility' included both technical and economic aspects of the process. Prior to embarking on the full-scale demonstration wherein 190.5 mt (420,000) lbs of aggregate would be prepared, the following three goals needed to be attainable:

1. A process demonstrated at a pilot scale and defined in sufficient detail to allow capital and operating cost projections for a full-scale process.
2. Convincing evidence that this type of aggregate would find a significant market (say >10,000 mt/yr) in insulating applications at the projected selling price to yield an adequate return on investment.
3. A belief by Alcoa that the desired material could be manufactured with the required quality and for a cost that would justify new capital expenditure.

CONTRACT OVERVIEW

This contract was based on a proposal submitted by Alcoa in response to DOE Solicitation for Federal Assistance Application No. DE-PS07-88ID12782 for Refractory Materials Research and Development. This application was for cost-sharing research for the development of new refractory materials or improvements to existing refractory materials, with end-use applications directed toward those refractories used by the aluminum, glass, cement, and iron and steel industries. These new or improved refractory materials should have the potential to lead to a reduction of energy consumption in one or more of the above end-use industries of 0.01 quadrillion BTU's annually. If successful, this energy saving would enhance the competitive position of the domestic end-use industries.

CONTRACT PHASES

The contract was broken down into three phases: a) bench-scale development, b) pilot-scale development, and c) full-scale demonstration. It was planned that the work would be performed in the sequence indicated. However, there was iteration between bench and pilot scale experiments as approaches were explored and new information developed. The project was managed by the Chemical Systems Division, but other Alcoa Technical Center (ATC) Divisions were accessible for expertise in ceramics, polymers, and surface chemistry. In addition, support groups such as the Information Department and Analytical Chemistry were utilized. The contract also involved working with the Product Development Group of the Industrial Chemicals' Business Unit located at Alcoa's Arkansas Operations that had the ultimate responsibility for translating any process developed to commercial production. Two refractory manufacturers were also involved in material preparation and evaluation.

Phase I - Literature Review and Bench-scale Experiments

Period: October 20, 1989 to May 31, 1991

Scope: The objective of this contract phase was to identify preferred approaches to the development of lightweight refractory aggregates. Included were a literature search, a review of prior related research, identification and evaluation of new approaches, performance of bench-scale experiments, selection of preferred approaches, and the decision to proceed or not to pilot-scale development of the process.

This contract specified that a technology or literature review be conducted as a prelude to experimental work. In addition, the contract specified that the technology review be summarized in a written report as part of the Phase I task list. Alcoa chose to separate the "Summary of Technology Report" from the report of experimental results conducted in Phase I of the contract.

This phase was the initial information gathering and screening stage of the project. The existing literature was studied and summarized to identify approaches to manufacturing low density alumina aggregate. In addition, Alcoa experts in related areas, including alumina, sintering, polymer science, and emulsion chemistry were consulted, as needed. Also, personnel of the Alcoa Arkansas Operations Technology Group were consulted for their experience as well as guidance as to the compatibility of potential approaches with existing operations.

An Alcoa Progress Report was issued representing a compilation of pertinent information resulting from a literature survey dealing with alumina refractory aggregate.¹ The literature survey included: a) a computerized search of Alcoa written reports and letters, b) a manual search of Alcoa Ceramic Division files, primarily to recover documents before 1960, and c) a computerized search of published literature and patents in several data bases, including World Aluminum Abstracts (1968-April 1990 update tape), Chemical Abstracts (1967-April 1990 update tape), Ceramic Abstracts (1967-April 1990 update tape), and PROMT (Prefabricated Overview of Markets and Technology (1972-December 1989). The references were catalogued using the computer spreadsheet package EXCEL™. Information tabulated included author(s), title, source, report/journal type, report/journal date, volume, pages, relevance designation, file number, and a document reference number.

With this background, it was decided that a sintered alumina aggregate with bulk density below 2.5 g/cc should be the project goal. New approaches to produce such a material on the bench-scale were postulated and performed for preliminary verification of processing approaches. Materials were purchased or synthesized as necessary, and test specimens were formed into green shapes and sintered. Bulk density, shrinkage, crushing strength, and microstructure were characterized as the screening parameters. The green forming method depended on the specific approach being investigated and included dry pressing, slurry casting, extrusion, and ball forming. Both paste and pelletized formulations were developed on the bench-scale to suit commercial mixing equipment available from toll processors and to minimize capital expenditure for the commercial process. Preliminary economic analysis for promising approaches was generated in this step, with the involvement of the Arkansas Technology Group. At the conclusion of this phase, it was judged that the approach decided upon (extrusion and sintering) was promising enough and potential markets large enough to justify continuation into Phase II. Details of the Phase I program and results were reported in an Alcoa Progress Report.²

Phase II - Pilot-scale Experiments

Period: September 01, 1991 to September 30, 1994

Scope: The objective of this contract phase was to determine the feasibility of producing lightweight aggregates and refractories at pilot-scale. Tasks included production of kilogram quantities of lightweight refractory materials, determination of new refractory formulation performance, and determination of feasibility to proceed or not to full-scale demonstration of the process.

In this phase of the experimental plan, kilogram quantities of materials were produced at the pilot-scale for further testing and performance evaluations that more closely simulated actual application conditions. In this phase, there was increased involvement of the Arkansas Technology Group as well as input and testing by two refractory manufacturers. Because of the variety of application and end-use conditions, it was viewed as essential that the expertise and facilities of refractory producers be utilized in the technical evaluation of candidate materials. Processes that still looked encouraging during this phase underwent a more critical economic analysis.

Alcoa prepared and executed an experimental plan for pilot-scale production of lightweight aggregate in sufficient quantities for preliminary evaluation by refractory manufacturers. Prior to the production, testing, and characterization experimental work, Alcoa obtained the input of refractory manufacturers with regard to the quantities of aggregate needed for fabricating specimens and the tests to be performed on these specimens.

Using pilot-scale processing equipment, Alcoa prepared new lightweight aggregates using the extrusion/sintering approach developed in Phase I of this project. Production of these new lightweight aggregates was performed under simulated full-scale, time-temperature production conditions at bench-scale and pilot-scale, utilizing ~1100 kg of aluminous materials. The lightweight aggregates were characterized for density and strength to ensure suitability for use in refractory formulations.

Refractory manufacturers prepared several refractory formulations (bricks and castables) from several formulations of the new lightweight aggregate. Refractory specimens were characterized against standard materials for physical and thermal properties including density and thermal conductivity. As anticipated, thermal conductivity of the new formulations had significantly improved insulating properties, reflecting the lower aggregate densities. Refractory manufacturers were moderately enthusiastic over the preliminary characterization results, especially with respect to reduced thermal conductivity and much greater aggregate strength, compared to existing lightweight aggregates.

Alcoa prepared an economic analysis (for internal Alcoa use only) for the production of lightweight aggregate using previous research results and estimates of production costs, based on a retrofit of this process into existing Alcoa production facilities for dense aggregate. Industrial interest in pursuing the best alternatives identified were solicited

from the refractory manufacturers who characterized the refractory specimens. These data were analyzed to determine if the technical, economic, and industrial interest factors supported effort toward full-scale evaluation.

Reporting of the acquired data included an Alcoa Progress Report on the pilot-scale effort.³ Based on these technical and economic results, continuation of the contract to the full-scale demonstration was warranted. This report included a recommendation to proceed into Phase III, pending the outcome of the market survey.

Market Survey for Lightweight Aggregate

After the Phase II report was submitted, Alcoa performed an internally funded study to determine if sufficient markets for the target lightweight aggregate were available to justify continuation of the development effort. Although specifics of this study are proprietary, Alcoa concluded that the North American market for this type of product was about 5,000 to 8,000 mt/yr, with application primarily in the chemical and ceramic industries. If the material could be used in direct contact with molten steel (and slag), the total North American market would increase to an estimated 10,000 to 18,000 mt/yr. Based on this assessment, it was decided to continue into Phase III of the development project.

Phase III - Full-scale Process Demonstration

Period: January, 1995 to March, 1996 (Contract terminated before completion)

Scope: The objective of this contract phase was to evaluate the feasibility of producing lightweight aggregates and refractories at full-scale. Included were the selection and evaluation of full-scale aggregate and refractory production approaches, provision of refractories for evaluation by end-users, performance of in-plant trials, evaluation of end-use performance, and assessment of refractory manufacturer and end-user interest.

Technical Issues Remaining to be Solved

Prior to embarking on the full-scale demonstration, resolution of three remaining technical issues was required.

- Fired density (slightly higher than target)
- Improve the intermediate strength (too weak after binder burnout, before sintering)
- Molten steel slag penetration resistance (unknown)

Additional pilot-scale experimental work in Task 1 of Phase III was aimed at: a) modifying the formulation to reduce the fired density to attain the 2.5 g/cc maximum target, and b) improving the intermediate strength of the lightweight aggregate. It was recognized during the Phase II work that the aggregate shapes were very weak in the intermediate temperature range of heating (after binder burn-out but before sintering). This problem needed to be solved to allow successful processing in commercial sintering equipment. To assess the slag penetration resistance, a sample of LWA from Phase II

work was incorporated into a separately funded Alcoa study of dense and lightweight aggregates in brick upon exposure to molten steel slag. The above technical issues were addressed and solved to the extent that they were not considered impediments to continuing with the contract. Details of the Phase III/Task 1 program and results were documented in an Alcoa Progress Report.⁴

Process Economics Feasibility Study

At this milestone point in the process development (1996 February), the economics were revisited for the modified composition that continued to utilize Eirich mixers to prepare moist granules for extrusion. The process was subjected to an economic feasibility study for production levels of 10,000 and 20,000 mt/year. This study used the most up-to-date raw material costs and the assumption of a retrofit into the Arkansas plant production facility. For these assumptions: a) a 10,000 mt/yr production level, b) realistic process recycle and losses, and c) a \$0.66/lb selling price (35% more than T-64 selling price on a volume basis), the economics would be favorable (~18% R.O.I.C.). For these assumptions: a) a 20,000 mt/yr production level, b) realistic process recycle and losses, and c) a \$0.60/lb selling price (15% more than T-64 on a volume basis), the economics would be favorable (~17% R.O.I.C.). The economy of scale was evident for the two projections of a 10,000 or 20,000 mt/yr production level. However, for the 10,000 mt/yr production level, the required LWA selling price exceeded the maximum level of 25% set forth in the project objectives. Details of the economic feasibility study were reported in Reference 4, Table 7.

Full-scale Demonstration Plans

The quantity of lightweight refractory aggregate to be produced as proof of full-scale process feasibility was determined by one key factor: a full-scale sintering campaign at Arkansas Operations. The estimate planned for processing 190.5 mt (420,000 lb) of aluminous raw materials, resulting in 108.9 mt (240,000 lb) of fired refractory shapes meeting the target specifications. After crushing to the desired aggregate sizes, it was assumed that a minimum of 54.4 mt (120,000 lb) would be available for fabrication into end-use products by two refractory manufacturers with each refractory manufacturer (R-M) to receive 27.2 mt (60,000 lb) of aggregate sized to their specification. Each R-M would prepare at least two insulating products for testing by two or more end-users. Phase III would involve toll processing outside of Alcoa for the raw material mixing, extrusion, and drying. Alcoa's Arkansas plant would fire, crush, and size the refractory.

Prior to the primary production run, pre-qualification of the toll processor using 7.26 mt (16,000 lb) of aluminous raw materials would be performed to assure satisfactory performance of equipment for the mixing, extrusion, and drying steps. As indicated in the following section, the project was terminated just prior to detailed logistical planning to prepare for execution of the full-scale demonstration.

Revised Market Survey for Lightweight Aggregate and Project Termination

As stated in prior reports, the formal market survey would be the overriding factor determining whether to continue with this development into the full-scale demonstration. As the work in Phase III of this project was underway, Alcoa became aware of a new lightweight aggregate material produced by thermal treatment of aluminum dross residue. This new material, developed and produced by another company, had physical properties somewhat similar to the Alcoa/DOE experimental aggregate. As was the case with the Alcoa/DOE aggregate under development, this dross-based aggregate exhibited significant performance enhancements over commercially available lightweight aggregates. Additionally, this competing aggregate had a much more favorable cost base, compared to the Alcoa/DOE LWA material, due to cheap raw materials and relatively few processing steps. In late 1995, Alcoa became a distributor of this new aggregate, named Plasmal.TM

In light of this new development, Alcoa estimated that 75% or more of the market originally envisioned for the experimental aggregate will be taken by the less expensive PlasmalTM aggregate. Consequently, the remaining market was judged to be not large enough to justify continuation of the Alcoa/DOE project. This information was discussed with DOE (1996 March) and it was mutually agreed to terminate this contract.

CONCLUSIONS

1. A new process for a high alumina, lightweight aggregate (2.5 g/cc bulk density) was developed on the bench and pilot-scale. This extrusion process utilized calcined and activated aluminas, an organic extrusion aid, and an organic binder in the refractory composition.
2. Technical problems dealing with attainment of the target fired density and poor intermediate strength after binder burnout but before sintering were addressed and solved.
3. Product characterization of this new aggregate revealed adequate fired strength and a significant reduction in thermal conductivity of bricks or castables that was a function of the reduced bulk density, compared to dense, tabular aggregate.
4. Process economics, dependent on the size of the retrofit plant and the assumed selling price of the new aggregate, were favorable for a 20,000 mt/yr production level and a selling price 15% more than T-64 on a volume basis.

5. The market for the new lightweight aggregate appeared to be favorable and would have justified full-scale demonstration of the process. However, a lower cost, competing material (PlasmaTM) became available and was estimated to fill 75% or more of the market originally envisioned for the high performance, Alcoa/DOE aggregate.

REFERENCES

1. Swansiger, T. G., "Lightweight Alumina Refractory Aggregate Phase I/Task 1 Summary of Literature Review," Alcoa Progress Report No. 6-90-40, DOE Contract No. FC07-89ID12903, December 1990.
2. Swansiger, T. G., "Lightweight Alumina Refractory Aggregate Phase I/Task 5 - Report on Experimental Results," Alcoa Progress Report No. 06-91-17, DOE Contract No. FC07-89ID12903, August 1991.
3. Swansiger, T. G. and Pearson, A., "Lightweight Alumina Refractory Aggregate Phase II-Pilot-scale Development," DOE Energy Conservation - Topical Report, DOE Contract No. FC07-89ID12903, November 1994.
4. Swansiger, T. G. and Pearson, A., "Lightweight Alumina Refractory Aggregate Phase III/Task 1-Resolution of Technical Issues," Alcoa Progress Report No. 6-96-11, DOE Contract No. FC07-89ID12903, July 16, 1996.