CERAMIC MEMBRANE ENABLING TECHNOLOGY FOR IMPROVED IGCC EFFICIENCY

ANNUAL TECHNICAL PROGRESS REPORT

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ABSTRACT:

This yearly technical progress report will summarize work accomplished for Phase 1 Program during the program year 2000 / 2001. In task 1, the lead material composition was modified to enable superior fluxes and its mechanical properties improved. In task 2, composite OTM elements were fabricated that enable oxygen production at the commercial target purity and 75% of the target flux. In task 3, manufacturing development demonstrated the technology to fabricate an OTM tube of the size required for the multi-tube tester. The work in task 4 has enabled a preferred composite architecture and process conditions to be predicted. In task 5, the multi-tube reactor is designed and fabrication almost complete.

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A. Executive Summary

The objective of this program is to improve process economics and efficiency and to reduce waste for coal-based IGCC power generation by advancing the development and commercial deployment of ceramic membrane systems that produce oxygen at lower cost than conventional systems.

The work undertaken in phase 1 of this program has been divided into 7 development tasks. These development tasks are

1) Materials development
2) Composite development
3) Manufacturing development
4) Process development
5) Lab-scale reactor system
6) Preliminary business development
7) Program management and reporting

Task 1 involves the development of a materials system that has the desired oxygen transport properties combined with the required stability to meet commercial targets. Task 2 focuses on defining the desired composite and substrate architecture, producing an effective interface layer and developing suitable fabrication techniques for the manufacture of composite OTM elements necessary for tasks 4 and 5. The work in task 3 is to demonstrate large-scale production of OTM composite elements. The work in task 4 involves experimental testing of OTM elements in single element flux testers at ambient and high pressures, and the development of oxygen transport models for single element composite OTM and multi-element OTM systems, and a reliability model. The objectives of task 5 are to design, construct and operate a multi-tube laboratory tester.

The objectives of the second year of the program are to:

- optimize a material composition that has high performance as a composite membrane for IGCC applications,
- demonstrate oxygen transport through composite OTM elements at 75% of the commercial target under simulated IGCC conditions
- demonstrate oxygen purity > 95% from composite OTM
- demonstrate technology to fabricate a tube of the size required for the laboratory reactor
- design and construct a multi-tube laboratory reactor

The work to achieve these objectives has led to several major accomplishments, summarized below.

- The lead material, PSO1d, was modified to improve the surface exchange coefficient enabling the flux target to be obtained.
- 75% of the commercial oxygen flux target has been exceeded under simulated IGCC conditions using composite tubes of modified PSO1d.

- The program oxygen purity target was exceeded under simulated IGCC conditions using composite tubes of modified PSO1d.
- A composite tube of PSO1d of the required size was manufactured.

B. Experimental Methods

B.1. OTM Materials Development Experimental Methods

Characterization of OTM and substrate materials has been undertaken using many different experimental procedures. These include permeation, crystallographic, thermomechanical, thermochemical and electrochemical measurements. Standard equipment such as XRD, SEM, dilatometry and TGA/DSC were used. In addition oxygen permeation testers were used to measure the oxygen flux of OTM discs. The permeation test facility was described in the DOE IGCC first annual report [1].

B.2. Composite OTM Development Experimental Methods

Various fabrication routes have been developed to prepare composite OTM samples. Small samples are first prepared and the fabrication routes that are most promising are further refined to enable larger OTM elements to be prepared. The fabrication routes used are proprietary information and included in the Appendix.

B.3. Manufacturing Development Experimental Methods

Fabrication routes developed in task 2 have been used for the manufacture of OTM elements for testing in the high-pressure permeation testers used in task 4.

B.4. Process Development Experimental Methods

Composite OTM elements of the required geometry prepared using methods developed in prior work have been tested for high temperature permeation utilizing the high-pressure test facility and method previously described in the DOE IGCC first annual report [1]. A method of increasing the driving force for oxygen transport has been added to the flux tester.

C. Results and Discussion

C.1. OTM Materials Development Results and Discussion

Modification of the OTM material has shown that significant improvement can be made to the oxygen flux without compromise to the mechanical integrity of the element. Figure 1 shows the flux enhancements possible with this modification.

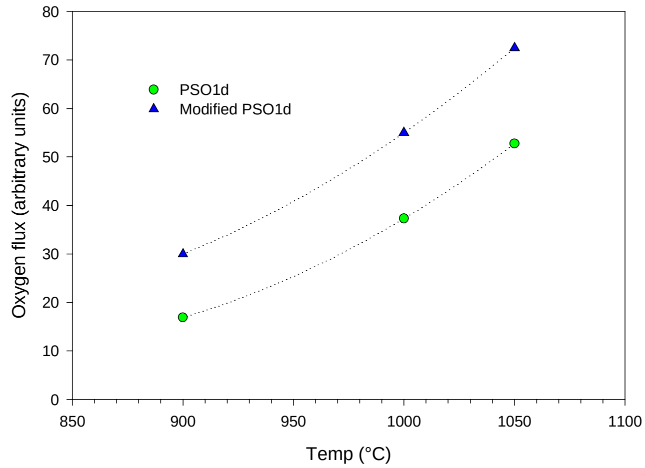

Figure 1. Oxygen flux of unmodified and modified PSO1d using a helium purge.

C.2. Composite OTM Development Results and Discussion

Composite OTM tubes have demonstrated that oxygen can be produced at the commercial purity target. They have also demonstrated oxygen production at 70% of the commercial flux target under simulated IGCC conditions. This is an extremely encouraging result and demonstrates that OTM technology is viable for commercial operation.

C.3. Manufacturing Development Results and Discussion

Improvements to the fabrication process were developed that have significantly improved the yield of OTM elements. Further modification to the process is continuing to ensure this increased yield applies to larger OTM elements. Fabrication technology has been demonstrated that enables the production of pre-commercial length tubes.

C.4. Process Development Results and Discussion

Composite tubes have routinely produced oxygen under conditions similar to IGCC operation with a **flux greater than 75% of the commercial target and purity greater than 95%**. Figure 2 illustrates the oxygen flux obtained under simulated IGCC conditions. Under conditions with an increased driving force the commercial target flux has been obtained. Stable operation has been demonstrated at greater than 250 psi.

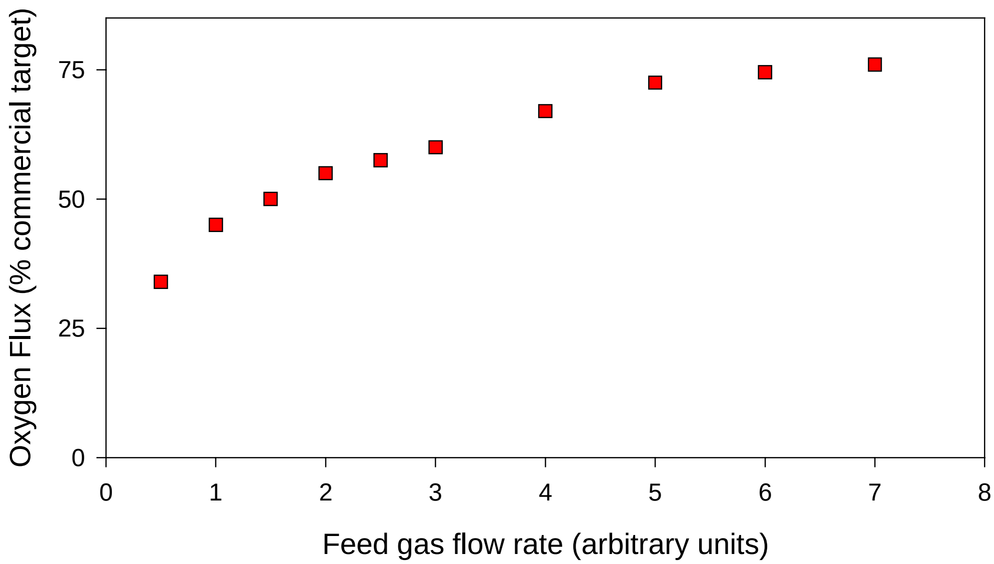

Figure 2. Oxygen flux of composite OTM tube at 900°C as
a function of feed flow rate at a pressure differential of 275 psi.

A high temperature seal has been developed that allows oxygen to be produced at the commercial target purity.

C.5. O-1 Pilot Reactor Development Results and Discussion

Design of the OTM multi tube pilot plant has been completed. Construction is underway and is expected to be completed in October.

D. Conclusion

Good progress has been made in all tasks toward achieving the DOE-IGCC program objectives. In task 1, the lead material, PSO1d, has been modified to improve the surface exchange coefficient. This has enabled significant improvement to the oxygen flux. In task 2, fabrication processes have been developed, from which tubes have been produced that allow oxygen production at the commercial purity target and 75% of the commercial flux target. In task 3, fabrication technology has been demonstrated that enables the production of pre-commercial length tubes. In task 4, process conditions have been determined that allow oxygen production at 75% of the commercial flux target. Stable operation at a pressure differential of 250 psi was demonstrated. In task 5, a laboratory scale, multi-tube tester has been designed and construction almost completed.

E. References

1. **Prasad, Ravi, "Ceramic Membrane Enabling Technology for Improved IGCC Efficiency" 1st Annual Technical Progress Report for US DOE Award No. DE-FC26-99FT40437, October 2000.**