CERAMIC MEMBRANE ENABLING TECHNOLOGY FOR IMPROVED IGCC EFFICIENCY

QUARTERLY TECHNICAL PROGRESS REPORT

For Reporting Period Starting April 1, 2001 and Ending June 30, 2001

Principal Investigator: Ravi Prasad
DOE Program Manager: Ted McMahon

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Submitted by:

Praxair, Inc.
175 East Park Drive
Tonawanda, NY 14150

ABSTRACT:

This quarterly technical progress report will summarize work accomplished for Phase 1 Program during the quarter April to June 2001. In task 1 optimization of the membrane architecture has showed the potential for significant improvements to membrane performance. In task 2, **improved composite elements have been prepared that have demonstrated 75% of the commercial target flux**. In task 3, control of fabrication steps has resulted in a significant increase in yield of OTN elements. The work in task 4 has demonstrated that composite OTM elements can produce oxygen at greater than 95% purity and 75% of the target flux. In task 5 work the design of a multi-tube OTM reactor is completed and construction will begin next quarter.

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A. Executive Summary

The objectives of the second year of the program are to define a material composition and composite architecture that enable the oxygen flux and stability targets to be obtained in high-pressure flux tests. Composite technology will be developed to enable the production of high-quality, defect free membranes of a thickness that allows the oxygen flux target to be obtained. The fabrication technology will be scaled up to produce three feet composite tubes with the desired leak rate. A laboratory scale, multi-tube pilot reactor will be designed and constructed to produce oxygen.

In the third quarter of the second year of the program, work has focussed on materials optimization, composite and manufacturing development and oxygen flux testing at high pressures. This work has led to several major achievements, summarized below.

- **Oxygen has been produced under conditions similar to IGCC operation using composite OTM elements at a flux greater than the 2001 target.** Under conditions with a greater driving force the commercial target flux has been met.
- Methods to significantly increase the oxygen flux without compromise to its mechanical integrity have been identified.
- Composite OTM elements have demonstrated stable operation at $\Delta P > 250$ psi
- Design of the pilot plant is complete and construction will begin next quarter.

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

Improvements to the surface properties of the OTM material have shown that significant improvement can be made to the oxygen flux without compromise to the mechanical integrity of the element.

Work has continued on new compositions that have significant benefits in certain IGCC process conditions. Larger batches of powder have been ordered to enable composite development of these materials.

C.2. Composite OTM Development Results and Discussion

High quality composite elements of PSO1d can be routinely prepared using a variety of processing methods. These composite elements are gas tight and have enabled the 2001 target oxygen flux to be obtained. Composite elements comprising improved compositions and structures have also been manufactured.

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.

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%**. Under conditions with an increased driving force the commercial target flux has been obtained.

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

Design of the OTM pilot plant has been completed. Parts have been ordered and contracts awarded. Construction will begin next quarter.

D. Conclusion

Progress has been made in all tasks toward achieving the DOE-IGCC program objectives. In task 1, a method of increasing the oxygen flux without compromise to the mechanical integrity was identified. In task 2, composite elements have been produced that meet the 2001 oxygen flux target. In task 3 improvements to the processing of OTM elements has led to an increase in the yield of OTM elements. In task 4, **an oxygen flux greater than 75% of the commercial flux target was met under conditions similar to the IGCC process**. The commercial target flux was obtained under conditions with an increased driving force. In task 5 the design of the O-1 reactor was completed. Construction will begin next quarter.

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.