CERAMIC MEMBRANE ENABLING TECHNOLOGY FOR IMPROVED IGCC EFFICIENCY

QUARTERLY TECHNICAL PROGRESS REPORT

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Praxair Program Manager: **Ravi Prasad**
DOE Program Manager: **Jenny Tennant**

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Submitted by:

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

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ABSTRACT:

This quarterly technical progress report will summarize work accomplished for Phase 1 Program during the quarter July to September 2002. In task 1 characterization of PSO1x has shown no decrease in strength at operating temperature. In task 2, composite development has demonstrated the ability to fabricate membranes of the new material PSO1x. In task 3, increased length elements have been fabricated. The work in task 4 testing of PSO1x has demonstrated oxygen purity of greater than 95% after more than 500 hours of testing. In task 5 the multi-element OTM reactor has been operated and produced oxygen at greater than target purity and flux.

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A. Executive Summary

The objectives of the third year of the program are to operate a laboratory scale pilot reactor that can produce 200-300 CFH oxygen. Manufacturing technology will be developed to demonstrate that commercial size elements can be fabricated using methods that can become economically viable. Material and composite development are required to produce OTM elements that are capable of a commercial flux and that have sufficient mechanical robustness for commercial life. The target flux will be demonstrated on composite elements of a material that can be used for pilot plant demonstration.

In the fourth quarter of the third year of the program, work has focussed on characterizing the new OTM, PSO1x, and operating the O1 reactor. The major accomplishments this quarter were

- **High temperature strength of PSO1x is as high as the room temperature strength**

- **The O1 reactor was operated at target flux and target purity using shorter composite tubes.**

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 longer single element tester has been designed and constructed.

C. Results and Discussion

C.1. OTM Materials Development Results and Discussion

Characterization of PSO1x continued. The material has high temperature strength similar to the room temperature strength. Samples exposed to conditions similar to expected operation for 50 hours also showed no decrease in strength.

C.2. Composite OTM Development Results and Discussion

High quality composite elements of PSO1d have been routinely prepared using a variety of processing methods. These composite elements are gas tight and have enabled the 2001 target oxygen flux, life and thermal and pressure cycling to be obtained. These elements have been used successfully in O-1 to yield target flux and purity.

C.3. Manufacturing Development Results and Discussion

Improvements to the manufacturing process have been used to fabricate 20" long composite elements of PSO1d, and shorter elements of PSO1x.

C.4. Process Development Results and Discussion

A composite element of PSO1x has produced oxygen under conditions similar to IGCC operation with target purity for greater than 500 hours.

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

The O-1 reactor has been operated using high flux, composite PSO1d elements. The reactor is being operated at 900^{o}C with a pressure differential of 275 psi across the membrane. The target flux per tube was exceeded and >95% oxygen purity were obtained and the reactor continues to operate.

D. Conclusion

Progress has been made in all tasks toward achieving the DOE-IGCC program objectives. In task 1, improvements to the membrane material show the strength of PSO1x is as high at high temperature as at room temperature. In task 2, composite elements of PSO1d and PSO1x can be routinely prepared. In task 3, longer composite PSO1d OTM elements and short PSO1x composite elements can be fabricated using the technology developed in task 2. In task 4, a composite element of PSO1x has produced oxygen at purity greater than 95% after 500 hours. In task 5, oxygen was produced in the multi-tube O-1 reactor at greater than target purity and target flux per tube.

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.

F. List of Publications

[1] "Advances in OTM Technology for IGCC", Ravi Prasad, Jack Chen, Bart van Hassel, John Sirman, James White, Prasad Apte, Tim Aaron, Eric Shreiber, Presented at the 19th Annual International Pittsburgh Coal Conference, Pittsburgh, 9/26/02.