CERAMIC MEMBRANE ENABLING TECHNOLOGY FOR IMPROVED IGCC EFFICIENCY

QUARTERLY TECHNICAL PROGRESS REPORT

For Reporting Period starting October 1, 2001 and ending December 31, 2001

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ABSTRACT:

This quarterly technical progress report will summarize work accomplished for Phase 1 Program during the quarter October to December 2001. In task 1 optimization of the substrate material has yielded substantial improvements to membrane life. In task 2, composite development has enabled 50% of the target flux under Type 1B process conditions. In task 3, manufacturing development has demonstrated that 36" long tubes can be produced. The work in task 4 has demonstrated that composite OTM elements can produce oxygen at greater than 95% purity for more than 500 hours of the target flux. In task 5 construction of the multi-tube OTM reactor is completed and initial startup testing was carried out.

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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 tubes can be fabricated using methods that can become economically viable. Material and composite development are required to produce OTM tubes that are capable of a commercial flux and that have sufficient mechanical robustness for commercial life. The target flux will be demonstrated on 6" tubes of a material that can be used for pilot plant demonstration.

In the first quarter of the third year of the program, work has focussed on improving the mechanical properties of OTM substrate materials, fabricating 36" long composite tubes for the pilot reactor, demonstrating stable operation of PSO1d OTM membranes and initializing the O1 reactor for operation. The major accomplishments this quarter were

- **500 hour life was demonstrated on a PSO1d OTM tube with an oxygen flux of 80% of the commercial target at greater than the purity target under high pressure operation.**
- 36 " long composite PSO1d tubes were fabricated
- O1 pilot reactor operation was started

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 properties of the substrate material have shown that substantial increase in the mechanical integrity of the element is achievable. This development indicates that commercial life can be obtained.

Work has continued on subtle doping of PSO1d to increase its oxygen transport properties. Initial tests on dense discs indicate that flux improvements greater than 20% are possible.

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 to be obtained. This technology has now been applied to larger tubes.

C.3. Manufacturing Development Results and Discussion

Improvements to the manufacturing process were developed that have enabled fabrication of 36" long composite elements of PSO1d. These elements will be used in the O1 pilot reactor.

C.4. Process Development Results and Discussion

A composite tube has produced oxygen under conditions similar to IGCC operation with a **flux greater than 75% of the commercial target and purity greater than 95% for more than 500 hours**.

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

Construction of the OTM pilot plant has been completed. Initial startup procedures have been carried out. Testing of PSO1d tubes will begin next quarter.

D. Conclusion

Progress has been made in all tasks toward achieving the DOE-IGCC program objectives. In task 1, improvements to the substrate material indicate that commercial life can be achieved. In task 2, composite elements of larger size have been produced to the same high quality as smaller tubes that obtained the 2001 oxygen flux target. In task 3 improvements to the processing of OTM elements has enabled fabrication of 36" long composite PSO1d elements. In task 4, a composite tube has produced oxygen under conditions similar to IGCC operation with a **flux greater than 75% of the commercial target and purity greater than 95% for more than 500 hours**. In task 5 construction of the O-1 reactor was completed and initial startup procedures were carried out.

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

Prasad, R., Chen, J., van Hassel, B., Sirman, J., White, J., "Advances in Oxygen Transport Membrane Technology for Integrated Oxygen Production in IGCC", copyright 2001, presented at the 18th Pittsburgh Coal Conference, December 2001.