CERAMIC MEMBRANE ENABLING TECHNOLOGY FOR IMPROVED IGCC EFFICIENCY

QUARTERLY TECHNICAL PROGRESS REPORT

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ABSTRACT:

This quarterly technical progress report will summarize work accomplished for Phase 1 Program during the quarter April to June 2002. In task 1 improvements to the membrane material have shown increased flux, stability and strength. In task 2, composite development has demonstrated the ability to cycle membranes. In task 3, scale-up issues associated with manufacturing large elements have been identified and are being addressed. The work in task 4 has demonstrated that composite OTM elements can produce oxygen at greater than 95% purity after 10 thermal and pressure cycles. In task 5 the multi-tube OTM reactor has been operated and produced oxygen.

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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 6" elements of a material that can be used for pilot plant demonstration.

In the third quarter of the third year of the program, work has focussed on improving the properties of the OTM material, demonstrating cyclability of PSO1d composite elements under simulated Type 1A IGCC conditions, and operating the O1 reactor. The major accomplishments this quarter were

- **A new material, PSO1x has been demonstrated to have superior flux, mechanical strength and stability than PSO1d.**
- **10 complete cycles were achieved using a composite PSO1d element between room temperature with no pressure differential to 275psig at 900^{o}C. The oxygen purity was > 95% after every cycle.**
- **Three tests were completed in the O1 pilot reactor using two dense PSO1d elements. Oxygen production was recorded during each test**.

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 PSO1d to increase its oxygen transport properties continued. The next composition, labeled PSO1x, shows significant improvement to oxygen flux under a variety of processing conditions. PSO1x also shows substantial (>40%) improvement to the mechanical strength and is more stable in conditions that are present under certain IGCC process cycles.

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. This technology has now been applied to larger elements.

C.3. Manufacturing Development Results and Discussion

Improvements to the manufacturing process have been used to fabricate large composite elements of PSO1d. Issues associated with scale-up are being addressed.

C.4. Process Development Results and Discussion

A composite element has produced oxygen under conditions similar to IGCC operation with **flux greater than 75% of the commercial target and purity greater than 95% O_2 after 10 thermal and pressure cycles.**

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

Three tests have been completed in the O-1 reactor using PSO1d elements. In each test oxygen has been produced. This is the first multi-element OTM reactor to produce oxygen.

D. Conclusion

Progress has been made in all tasks toward achieving the DOE-IGCC program objectives. In task 1, improvements to the membrane material indicate that oxygen flux, stability and strength can be further increased. In task 2, composite elements of capable of producing 10 thermal cycles have been prepared. In task 3, 36" long composite PSO1d OTM elements can be fabricated. In task 4, a composite element has produced oxygen under conditions similar to IGCC operation with a **flux greater than 75% of the commercial target and purity greater than 95% after 10 thermal cycles**. In task 5 oxygen was produced in the multi-element O-1 reactor during three separate tests.

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.