

**Development of ITM Oxygen Technology for Integration in IGCC & Other  
Advanced Power Generation Systems  
(ITM Oxygen)**

**Technical Progress Report**

for the period

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### **Task 1.1: Optimize ITM Oxygen Materials**

Air Products and Ceramatec, Inc. continued to measure physical and chemical property data for the ITM Oxygen material AP2K. The Pennsylvania State University (Penn State) performed quantitative tensile creep rate measurements for the ITM Oxygen material AP2K, using samples fabricated by Ceramatec.

Penn State has completed several experiments to measure the high-temperature behavior of AP98 and AP2K samples under oxygen permeation conditions using the specialized apparatus commissioned during the prior quarter: this test provides physical property data and is also a candidate test to validate McDermott Technology, Inc.'s thermal and mechanical modeling of ITM Oxygen structures. Penn State, Air Products and McDermott Technology (see Task 2.3) collaborated for the analysis and interpretation of the experimental data, and further definition of experimental conditions.

Eltron Research, Inc. has determined the sintering conditions appropriate for the preparation of membrane samples for all eight material compositions being investigated under a designed experiment study for ITM Oxygen. The goal is to identify an optimized membrane composition and a preferred catalyst that enhances the rate of oxygen exchange between the gas phase and the membrane.

### **Task 1.2: Lifetime Testing of Optimized ITM Oxygen Materials**

Air Products performed multiple additional oxygen flux tests with subscale membranes and laboratory-scale, supported, thin-film membranes. Ceramatec prepared all of the membrane samples under Task 3.2. A subscale membrane exceeded the Phase I flux target, achieving 125% of the target oxygen flux at a feed-air pressure of 200 psig in a Vacuum Permeate-Equipped Short Loop Test Rig unit. This is a record performance for an ITM Oxygen supported, thin-film membrane at a feed-air pressure and conditions representative of a commercial process.

During January-March, Air Products continued several long-term oxygen flux tests. Two tests with subscale membranes, initiated in December 2000, were operated in excess of 1950 hrs and 2350 hrs in the Vacuum Permeate-Equipped Short Loop Test Rig units at a feed-air pressure ratio of 14 and the full operating temperature targeted for ITM Oxygen. In addition, two laboratory-scale membranes surpassed 2700 hrs on-stream at atmospheric pressure feed-air and a vacuum-permeate, completing long-term testing at a driving force and operating temperature considered typical for commercial operation. Both laboratory-scale membrane samples produced steady oxygen fluxes with time.

Air Products completed additional performance tests of ceramic-to-metal seal assemblies for the ITM Oxygen module. A full-size ceramic-to-metal seal between a metal header and a ceramic, full-size oxygen-product pipe performed successfully during a 400 hr test at 200 psig in a Short Loop Test Rig unit. The full-size ceramic-to-metal seal is required for testing membrane modules in the TDU. Ceramatec prepared the full-size oxygen-product pipe under Task 3.2.

Oxygen flux tests are progressing well at Eltron Research, with data obtained for all eight material compositions being investigated under a designed experiment study for ITM Oxygen. Membrane testing is continuing to complete the full set of experimental test conditions for each composition, and to extend the performance database with repeat samples. Eltron Research and Air Products will perform a statistical analysis of the performance database to develop a property/composition model. The goal is to identify an optimized membrane composition and catalyst.

### **Task 2.1: Gas Turbine Integration**

Concepts NREC, Inc. has completed its initial review of the design and performance of commercially available engines against the requirements for integration in ITM Oxygen oxygen/power co-production applications. Concepts NREC identified several engines including separate compressor and expander components for further analysis during Phase I, and a performance model has been calibrated for each candidate engine. Parametric calculations have been completed on a number of these candidate engine models to evaluate options for system integration in ITM Oxygen oxygen/power co-production applications. Initial contacts were made with the candidate engine vendors to solicit their input for a more detailed analysis during Phase I.

### **Task 2.3: Thermal and Mechanical Analysis of Membranes and Seals**

McDermott Technology continued its development of thermal and mechanical models to analyze Penn State's test calibration procedure and measurement of the high-temperature behavior of ITM Oxygen materials under oxygen permeation conditions (see Task 1.2 above). McDermott Technology proposed several modifications for the test procedure.

McDermott Technology also completed an initial analysis of the full-size oxygen-product pipe, to estimate and define reasonable tolerances for fabrication.

### **Task 2.4: Conceptual Process Vessel Design Engineering**

McDermott Technology completed a series of cost sensitivity studies for conceptual, commercial-size, ITM Oxygen process vessel designs. Cost estimates for process vessels to house full-size membrane modules (see Task 3.1) were consistent with the economic target for ITM Oxygen. From several design variables identified at a November 2000 review meeting between Air Products, Ceramatec, and McDermott Technology, McDermott's sensitivity study has identified the major independent design variable that significantly impacts vessel cost. McDermott Technology also completed preliminary computational fluid dynamic (CFD) analyses to examine airflow distribution within an ITM Oxygen conceptual vessel design.

### **Task 2.6: Process Engineering and Economic Evaluation**

Under Task 2.6.1, Texaco, Inc. and Air Products constructed the third case for process engineering and economic evaluation of ITM Oxygen-integrated IGCC power plants employing Texaco's gasification technology. Several candidate ideas were considered, including alternative and/or further heat integration, all based around the same state-of-the-art turbine engine used for the ITM Oxygen-integrated base case and the cryogenic ASU-integrated base case. Texaco and Air Products will follow the scope and work plan used successfully for the base cases to complete case #3.

### **Task 3.1: Membrane Design and Engineering**

McDermott Technology completed the "pressure-collapse-test" shakedown trials of strain-gauged ITM Oxygen membranes, at ambient temperature using a specialized apparatus. The "pressure-collapse-test" is a candidate for validating McDermott Technology's approach and property assumptions used for mechanical modeling of membrane structures. Membranes were successfully tested up to a pressure ratio of approximately 100 the operating pressure limit of the test system with strain data collected from multiple elements on the membrane surface. A pressure ratio of 100 is approximately 5-times the pressure ratio for representative F/G-class turbines and typical feed-air pressure ratios envisaged for the ITM Oxygen process. McDermott Technology has completed its analysis of the initial "pressure-collapse-test" using three-dimensional, finite element analysis (FEA) models. The strain behavior predicted is in general agreement with the measured values from the strain-gauged membrane, providing preliminary validation of the modeling. Further pressure-collapse-testing is planned at typical feed-air pressure ratios envisaged for the ITM Oxygen process, to permit additional comparison to the analytical predictions and validation of the modeling approach.

Air Products extended further the module size optimization analysis based on economic and engineering models by including membrane module fabrication yields and process vessel cost. Several design sensitivities were considered under Milestone 3.1.3 Complete the Design and Engineering of a Large-Module of Full-Size Membranes. As a result, a specification was recommended for an optimum (i.e., full-size) ITM Oxygen module.

### **Task 3.2: Membrane Fabrication**

Ceramatec continued its baseline ceramic processing development for the ITM Oxygen full-size membrane. Scaleup has been substantial to date as a result of ceramic processing tools installed for the Stage I and Stage II ceramic processing development facility (PDF). When expressed as the number of full-size membranes processed per month, Ceramatec has achieved a seven-fold increase in throughput over a nine-month period: Ceramatec transitioned to full-size membranes in July 2000. Membrane throughput during January-March was more than a three-fold increase versus the prior quarter. Ceramatec also realized a three-fold improvement in the per-quarter yield of testable, full-size

membranes due to a planned process change implemented midway through this quarter, although these membranes did not meet the most stringent leak-rate criterion. Ceramatec is continuing to develop control charts to assess its baseline ceramic processing and to identify the process changes required to prepare, in high yields, membranes that meet the most stringent criterion for testing in the TDU under Task 4.3.

### **Task 3.3: Membrane Fabrication Pilot Line**

The contractor has completed construction of Ceramatec's Stage II ceramic processing development facility (PDF). The Stage II structure received a certificate of compliance, and a Process Hazards Analysis is underway for the facility. Under Milestone 3.3.2 Install Membrane Fabrication Pilot Line, Ceramatec initiated the installation of processing tools and equipment into the Stage II PDF. The major ceramic processing tool outstanding for Stage II is scheduled for vendor buyoff in April, and delivery and installation in May. The Stage II facility will be used to further the baseline ceramic processing development for the ITM Oxygen full-size membrane.

### **Task 3.4: Subscale Module Fabrication**

Ceramatec continued to develop processing for the assembly of membrane modules from full-size components, and inventories of all ceramic components have been established. Initial module assembly trials have been unsuccessful. A collaborative experimental plan was instituted and is underway at Ceramatec and Air Products to isolate and eliminate the root cause factors. Ceramatec will install additional equipment resources in the following quarter to facilitate module fabrication development. Membrane modules will be tested in the TDU under Task 4.3.

### **Task 3.5: Advanced Module Development**

Ceramatec recommended a preferred system, processing conditions and procedure for initial membrane module assembly trials of its alternative bonding method. The preferred system has successfully produced hermetic test assemblies of the ITM Oxygen material AP2K. Ceramatec will prepare "advanced" subscale membrane modules for testing at Air Products in the Vacuum Permeate-Equipped Short Loop Test Rig under Task 1.2. Ceramatec also initiated development of the plan for examining alternate or "advanced" ceramic fabrication approaches for ITM structures.

### **Task 4.2: Design and Construction of the TDU**

In January, Air Products completed equipment installation, field wiring and the Operational Readiness Inspection for the Technology Development Unit (TDU). The TDU extends the analytical capabilities of the Prototype Development Unit (PDU) by incorporating instrumentation to measure the combustion products in the feed and non-permeate streams. The TDU was also upgraded to a PLC-based control system and a retractable igniter for the combustor. Equipment shakedown continued throughout the quarter, including the PLC system, air compressor, heaters and the combustor igniter: the unit will be

commissioned under Milestone 4.2.2. The TDU is designed to demonstrate commercially relevant oxygen fluxes and recoveries with full-size ITM Oxygen membrane components, in a vitiated-air stream directly heated by a pre-combustor to simulate closely the commercial process conditions. The oxygen vacuum-permeate system is sized for a maximum production of 0.1 TPD.