

**Decision Point 3
of
Statement of Project Objectives (SOPO)**

TOPICAL REPORT

1 February 2012

Tasks 28, 29, 30

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**“Recovery Act: Development of ITM Oxygen Technology for Integration
with Advanced Industrial Systems”**

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ABSTRACT

Air Products is carrying out a scope of work under Phase 5 of the ITM Oxygen Cooperative Agreement to design, build, and operate a ceramic membrane fabrication facility (the “CerFab”) to enable production of membrane modules to supply a conceptual 2000 ton per day (TPD) ITM Oxygen facility (the “ITM Oxygen Development Facility”), and to perform supporting development tasks in materials development and engineering development toward industrial, carbon capture and sequestration applications. Air Products is executing this project under the American Recovery and Reinvestment Act (ARRA) with the objective to accelerate the adoption of ITM Oxygen technology to help meet the country’s goals for deploying clean power plants. The objective of this Topical Report is to address the requirements of Decision Point 3 (DP3), which pertains to the status of all Tasks within Phase 5 and most notably the project status of the CerFab (Task 30) prior to authorization of funds for equipment purchase and construction of the facility. The intent of the DP3 is to provide the opportunity for DOE-NETL to review the status of these tasks and to make recommendations on forward project direction, including a recommendation to pass into Budget Period 8. In the area of Materials Development, Air Products has specified a high pressure dilatometer system which will enable measurements of material expansion of ITM ceramic compounds at very high oxygen partial pressures consistent with CCS applications. Under Task 28.2, subcontractor Ceramatec has made significant progress since DP2 in materials selection and process development and improvement for advanced architecture module fabrication. Ceramatec has determined a materials specification, and has selected a process for making the material. Ceramatec has further developed and selected the process for applying the membrane to unsintered advanced architecture wafers with a Two Step process. Ceramatec has built submodules meeting leak rate specifications and intact 1-TPD modules compromised of advanced architecture components. Equipment and processes required for implementation of the advanced architecture design in the CerFab have been identified and are included in the Task 30 scope of equipment supply.

Under Task 29.0, Air Products has developed conceptual ideas for implementation of ITM Oxygen in a 2000 TPD test unit. These concepts are proposed for scale up of the ITM technology to large scale with reduced commercial risk.

Under Task 30, Air Products and Ceramatec have collaborated to re-design the CerFab process with the advanced architecture wafer production process as primary, and laid out the new process in the Tooele building; the required

process equipment have been specified. The project team updated the project cost and schedule estimates, including all environmental permit schedules. The team also re-assessed project risk and costs for the commissioning and operating phases of the program. Newly projected costs for the CerFab, for which details are provided here as attachments, indicate a savings of approximately \$100,000 relative to the original estimate at the time of Phase 5 contract definitization. However, an assessment of risks associated with the equipment, process, and operations of the CerFab indicate that approximately \$2.9 million of costs should be expected to address various issues in the project. In addition, \$1.6 million of additional spending is expected relative to the original estimate to operate the facility during Task 30.2. Air Products proposes a plan to re-direct funds from Tasks 28, 29, and 31 to Task 30 as necessary to allow the CerFab to meet the Phase 5 program objectives.

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Executive Summary

Air Products is executing this Recovery Act project with the objective to accelerate the adoption of ITM Oxygen technology to help meet the country's goals for deploying clean power plants. As part of this objective, Air Products is designing, building, and operating a ceramic membrane fabrication facility (the "CerFab") to enable production of membrane modules to supply a conceptual 2000 ton per day (TPD) ITM Oxygen facility (the "ITM Oxygen Development Facility").

The objective of this application is to address the requirements of Decision Point 3 (DP3) of the above-referenced SOPO which pertains to the status of all Tasks within Phase 5 and most notably the project status of the CerFab (Task 30) prior to authorization of funds for equipment purchase and construction of the facility. The intent of the DP3 is to provide the opportunity for DOE-NETL to review the status of these tasks and to make recommendations on forward project direction, including a recommendation to pass into Budget Period 8. DP3 includes description in the following areas:

- 1.0 Summary of Status of Task 28.0 "Ceramic Materials and Manufacturing Development for the Industrial and CCS Applications"
- 2.0 Task 28.2 "Advanced Module Development"
- 3.0 Summary of Status of Task 29.0 "Engineering Development for Industrial and CCS Applications"
- 4.0 Summary of Status of Task 30.0 "Ceramic Membrane Module Fabrication Facility"
- 5.0 Discussion of overall project management
- 6.0 Project Status

Under Task 28.0, Air Products has placed an order for a required analytical device which will extend the range of critical measurements of ITM ceramic material properties to the full range of operating conditions anticipated in industrial and CCS applications. A revised project schedule indicates the system will be commissioned by Q4FY12. Under Task 28.2, subcontractor Ceramatec has made significant progress since DP2 in materials selection and process development and improvement for advanced architecture module fabrication. Ceramatec has determined material stream characteristics for producing improved advanced architecture membrane wafers, and has selected a process for making the material. A process involving applying membranes to advanced architecture wafers has been developed and selected for use in the CerFab. Submodules meeting leak rate specification have been made using advanced architecture wafers derived from the selected material stream. Equipment and processes required for implementation of the advanced architecture design in the CerFab have been identified and are included in the Task 30 scope of equipment supply. Plans for additional process development focused on yield enhancement have been made.

Under Task 29.0, Air Products has developed conceptual ideas for implementation of ITM Oxygen in conjunction with cryogenic oxygen production facilities as a basis for a 2000 TPD test unit. These hybrid concepts are proposed for scale up of the ITM technology to large scale with reduced commercial risk.

Under Task 30, Air Products and Ceramatec have collaborated to re-design the CerFab process with the advanced architecture wafer production process as the

primary process, and laid out the new process in the Tooele building; the required process equipment has been specified. The project team obtained updated quotations and made updated project cost and schedule estimates, including all environmental permit schedules. The team also re-assessed project risk and costs for the commissioning and operating phases of the program. Newly projected costs for the CerFab indicate a savings of approximately \$100,000 relative to the original estimate at the time of Phase 5 contract definitization. An assessment of risks associated with the equipment, process, and operations of the CerFab indicate that approximately \$2.9 million of costs should be expected to address various risk issues in the project. In addition, \$1.6 million of additional spending is expected relative to the original estimate to operate the facility during Task 30.2. Air Products proposes a plan to re-direct funds from Tasks 28, 29, and 31 to Task 30 as necessary to allow the CerFab to meet the Phase 5 program objectives.

Report Details

A. Recovery Act Objectives

The objective of this project is to accelerate the adoption of ITM Oxygen technology by developing and constructing systems and infrastructure that will enable manufacturing of ITM membrane modules and the integration of the ITM technology toward deployment at industrial-energy plant scales. This objective includes the optimization of the materials processing technology and refinement of the module fabrication techniques to supply a conceptual 2000 ton per day (TPD) ITM oxygen facility (the “ITM Oxygen Development Facility”). The objective further includes the operation of the fabrication facility that demonstrates the capability of producing components and devices for separating oxygen from air and oxygen-containing streams. The key metrics for this effort include:

- Development of materials processing techniques and capacity to supply ceramic modules that meet specifications (each module capable of producing 1 ton per day of 99% purity oxygen) in support of the oxygen production goals for the ITM Oxygen Development Facility.
- Development of module designs and manufacturing techniques to enable 99% reliability of constructed and individual modules that are capable of supplying the ITM Oxygen Development Facility achieving 50-85% oxygen recovery.
- Development of integrated infrastructure, facilities, and quality control to enable constructed modules to be supplied to the ITM Oxygen Development Facility which is based on a process that achieves lower cost oxygen than is supplied by comparable commercial cryogenic air separation units.
- Development of a conceptual and detailed process design and appropriate budgetary cost models for the ITM Oxygen Development Facility that meets scale-up and operational needs for a pre-commercial unit that supports the goals of the DOE NETL Gasification Program and Industrial Carbon Capture and Storage (ICCS) Program.

This development and construction effort will occur concurrently with the Phase III effort to construct and demonstrate a 100 TPD Intermediate Scale Test Unit (ISTU) of an Oxygen separation plant and pursuant to the objectives and provisions of the American Recovery and Reinvestment Act (the “ARRA”).

The objective of this application is to address the requirements of Decision Point 3 (DP3) of the above referenced SOPO. The requirements of DP3 are described within Task 30.1.3.4 and prior to initiating Task 30.1.4 (“Equipment procurement and installation”) of the SOPO. For ease of reference, the entire text of Task the Decision Point 3 is referenced below.

Excerpted from SOPO:

DECISION POINT 3 - Review Readiness for Procurement

There shall be Decision Point at the completion of task 30.1.3.4. The Recipient shall submit a “Decision Point Application” directly to the NETL FPM and the DOE Contract Specialist no later than 30 days prior to the completing task 30.1.3.4 including 1) prior to procurement of equipment, materials and supplies, 2) prior to major modifications of the ceramic membrane module manufacturing facility and 3) upon

completion and approval of any permitting and environmental related determinations as specified by the DOE Contract Officer at Decision Point 2.

The Decision Point Application shall include the following information:

- 1) *A report on the Recipient's progress towards meeting the objectives of the project, including any significant findings, conclusions, or developments;*
- 2) *A detailed budget and supporting justification for the upcoming Subphase if additional funds are requested, a reduction of funds is anticipated, or a budget for the upcoming phase was not approved at the time of award; and*
- 3) *A description of the Recipient's plans for the conduct of the project during the upcoming Subphase.*

The decision to initiate task 30.1.4 will be based on the DOE's review of the technical accomplishments of the preceding work, programmatic changes and/or the availability of funding. If, at the time of the Decision Point, it is advantageous to the Government to continue the project, as determined by the sole discretion of the DOE, the DOE Contracting Officer shall decide whether or not to continue the Phase V project and shall notify the Recipient in writing in a timely notification. Work on subsequent Tasks and Subtasks shall not begin in the absence of written approval by the Contracting Officer.

In the event the DOE does not grant a favorable determination, the Contracting Officer shall notify the Recipient in writing of such decision and performance under Phase V of this Cooperative Agreement shall be considered complete at the Decision Point. The DOE reserves the right to de-obligate any remaining funds obligated to Phase V of the Cooperative Agreement.

Accordingly, Air Products, working with its subcontractor, Ceramatec, has assembled this Application Package which addresses progress made under Phase 5 to-date. As described in the statement of work, Task 28.0 involves development of advanced ceramic component architectures and fabrication methods. To-date in the project, these activities have been focused on components that feature an advanced architectural structure, rather than the standard structure developed under Phases 1-2. Exploratory work under Phase 3 of the program has shown that the advanced components have superior reliability to the standard components that were developed and that have been produced in large numbers under Phase 3. Air Products and Ceramatec have put special emphasis on developing the advanced architectures and production methods with the aim that such processes can be introduced to the CerFab with minimal disruption to the overall project and will enhance the overall production process while preserving its flexibility to produce either advanced or standard parts. Further development of the advanced architecture technology has led to its adoption as the primary module architecture around which development activities and CerFab process design have been based during Phase 5.

In addition, Air Products intends that this document will provide supporting information to allow the DOE to understand better the CerFab project scope, budget and schedule, technical and project risks in the CerFab project, and Air Products' recommendations toward forward program emphasis among the various tasks.

In particular, this package includes the following sections:

- 1.0 Summary of Status of Task 28.0 “Ceramic Materials and Manufacturing Development for the Industrial and CCS Applications”
 - 1.1 Objectives of Task 28
 - 1.2 Work under SubTask 28.1.3.1 High temperature materials properties
- 2.0 Task 28.2 “Advanced Module Development”
 - 2.1 Background
 - 2.2 Results
 - 2.3 Expected Future Work
 - 2.4 Specific Equipment and Processes Required for Introduction of Advanced Architecture Module Production in the CerFab
- 3.0 Summary of Status of Task 29.0 “Engineering Development for Industrial and CCS Applications”
- 4.0 Summary of Status of Task 30.0 “Ceramic Membrane Module Fabrication Facility”
 - 4.1. Project Scope
 - 4.2. Project Schedule
 - 4.3. Summary and Status of the environmental approvals required
 - 4.4. Commissioning plan leading up to DP4
 - 4.5. Project Budget
- 5.0 Discussion of overall project management
 - 5.1 Budget Trade-offs within the Phase 5 project
- 6.0 Project Status

B. Supporting Information

1. Summary of Status of Task 28.0 “Ceramic Materials and Manufacturing Development for the Industrial and CCS Applications”

1.1 Objectives

This section addresses progress under Task 28.0. Air Products and Ceramatec have been progressing under Tasks 28.1, 28.2, and 28.3. One of the main objectives of Task 28.1 is the establishment of a high temperature/high pressure dilatometer instrument as described in DP2. Work toward that objective is described below. In addition, under Task 28.2, Ceramatec is working to define processing steps that will enable advanced architecture wafer production at the CerFab. Work under Task 28.3 is also progressing, which supports all production steps at increased scale.

1.2. Work under Subtask 28.1.3.1. High temperature materials properties

1.2.1. Background

Air Products' ion transport membrane (ITM) material, like most materials, expands when heated. Unlike most materials, however, at a constant temperature, the ITM material contracts with increasing oxygen partial pressure and expands with decreasing oxygen partial pressure. In the presence of an oxygen partial pressure gradient, a differential expansion can be produced that results in a mechanical stress. This is analogous to the stress produced by a temperature gradient in conventional materials. To perform mechanical analysis of membrane modules and to predict stresses in advanced membranes during operation, the expansion behavior of the ITM membrane material is needed as a function of temperature and oxygen partial pressure. Since typical ITM Oxygen process conditions include operation with high pressure air, knowledge of the expansion behavior of the ITM membrane material at oxygen partial pressures in excess of one atmosphere is needed. Under Phase 3, dilatometry measurements have been extrapolated to high pressures to predict stress at ITM Oxygen operating conditions. To reduce technical risk in future development activities, particularly those involving a wider range of operating conditions as contemplated under Phase 5, mechanical modeling and stress predictions at operating conditions should be done based on data obtained at the actual temperature and oxygen partial pressure conditions and not rely on extrapolations.

A dilatometer is an analytic device to accurately measure length changes of a sample in a controlled atmosphere as a function of temperature. Commercially available dilatometers are limited to operation at relatively low pressures, typically operating with a maximum pressure of one atm. To measure expansion at higher oxygen partial pressures, a dilatometer capable of operating in high pressure air is needed. A dilatometer vendor has proposed a design for a high pressure dilatometer to allow operation at the required conditions. The data from this unit will allow calculation of the stresses of the ITM membrane at actual process conditions anticipated in Industrial and CCS applications.

1.2.2. Dilatometer Project

Air Products developed the scope and refined the cost estimate for the dilatometer project; the results were presented in the Decision Point 2 application package. The project consists of procuring and installing the device as well as its associated high pressure gas handling system. An updated project schedule is presented in Table 1.2.1.

Table 1.2.1 Dilatometer Project Schedule

Sept 2011	Preliminary Hazard Review Completed
9/30/2011	Approval to proceed received from DOE
10/25/2011	PO placed with dilatometer supplier
Nov 2011	Preparation of laboratory space commenced
Dec 2011	Gas handling system components ordered
Jan 2012	Pressure vessel drawing approval by Air Products; Gas handling system construction complete
Feb 2012	Design Hazard Review complete
June 2012	Dilatometer delivered to Air Products
Q4FY12	Installation and commissioning completed; Operational Readiness Inspection completed;

1.2.3. Dilatometer System Design

Air Products placed a purchase order with the dilatometer supplier in Q1FY12 to deliver the high pressure instrumentation with high pressure handling capability. The device will meet all Air Products safety requirements for a high pressure system; the pressure vessel housing the electronics will be designed to the ASME pressure vessel code. A drawing of the pressure vessel is shown in Figure 1.2.1. The pressure vessel consists of an upper chamber housing the electronics and a lower chamber housing the sample and heating means.

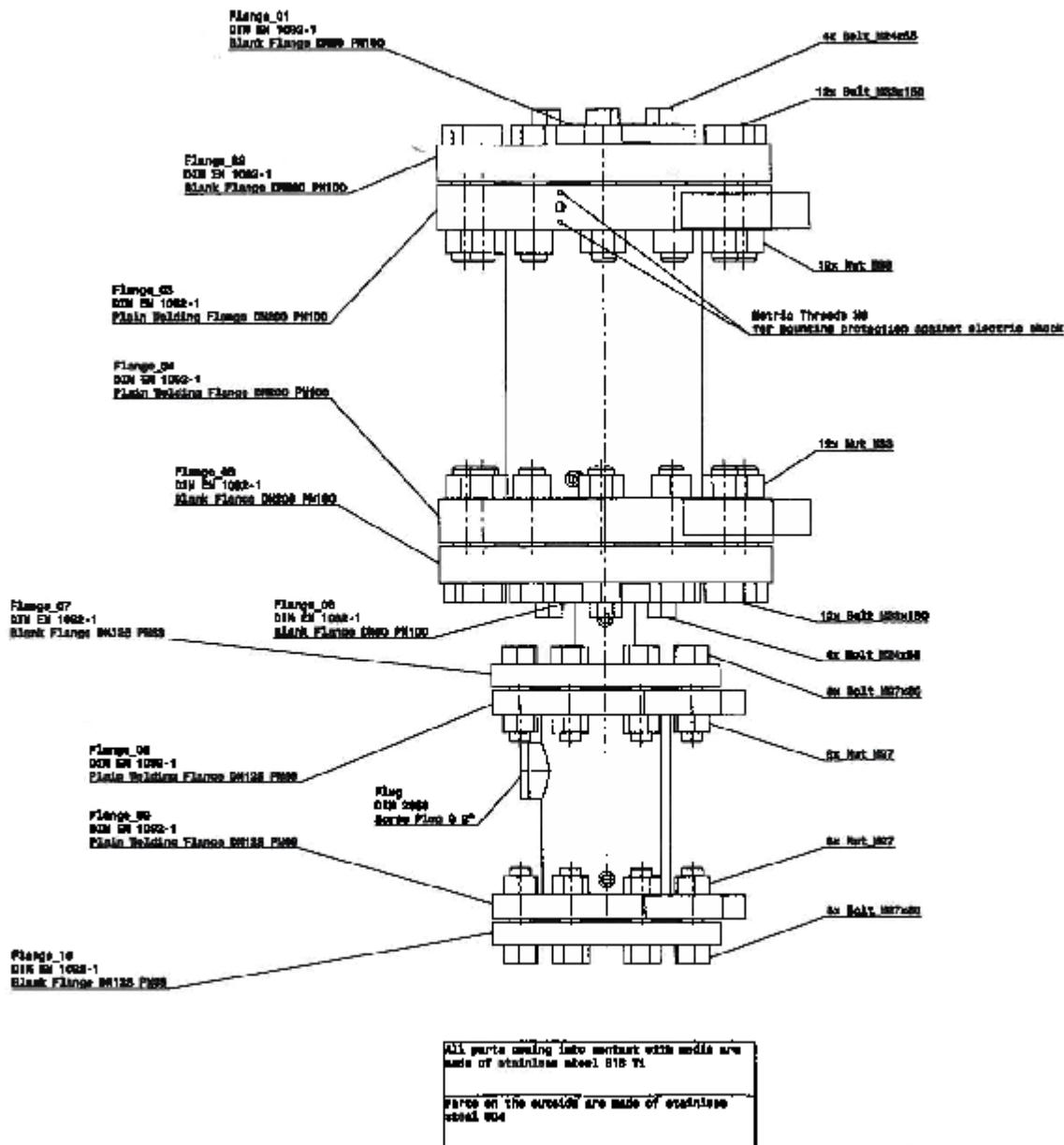


Figure 1.2.1. High Pressure Dilatometer Pressure Vessel

In addition, Air Products developed a gas handling system to enable controlled delivery of high pressure process gases to the device. A schematic of the gas handling system is shown in Figure 1.2.2.

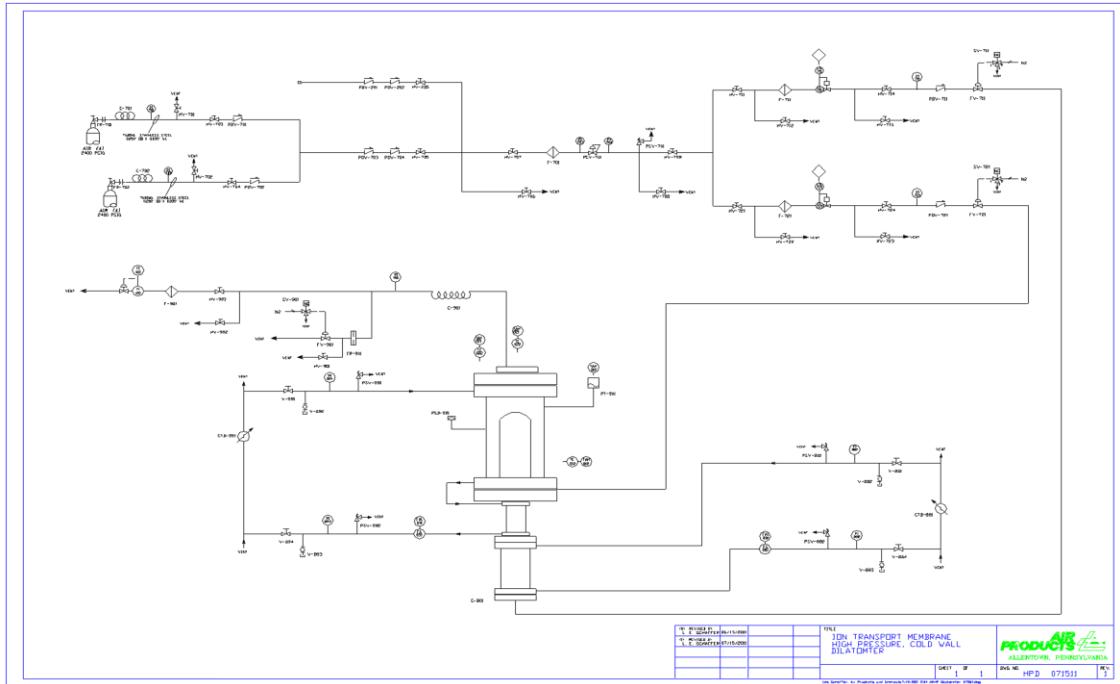


Figure 1.2.2. Gas Handling System schematic for the High Pressure Dilatometer System.

2.0 Work Performed under Task 28.2 Advanced Module Development

2.1 Background

As described in previous reports, finite-element-analysis (FEA) -driven work performed by Ceramatec indicated that structure architectures could be further improved resulting in lower stresses during unplanned cooldown scenarios. This result has been demonstrated experimentally both at Ceramatec and Air Products by several independent tests on material specimens, wafers, and test components. Consequently, Ceramatec began the development of "advanced architecture" module components. Similar to the standard module structure, all of the material in the advanced structure is the oxygen-conducting perovskite material used for the ITM. During Phase 3, methods were developed to make advanced module endcaps that met the requirements of sufficiently low leak rates to be used in modules. Similar methods have been employed in Phase 5 to make advanced architecture wafers.

Two parallel approaches for preparing advanced architecture wafers were investigated. The two approaches differ mainly in how the membrane is applied to the wafer. One method is a

“One Step” method, and involves sintering a portion of the wafer before addition of the membrane layer; this approach was based on the process that was developed in Phase III to make module end caps. The second approach, also initiated in Phase III, was based on the conventional process for making standard wafers. In this approach uses a “Two Step” method to apply the membrane to the wafer. After wafer fabrication, the methods of building submodules and modules from the advanced architecture wafers are the same as has been practiced for standard components.

Although wafers meeting leak-rate specifications were produced prior to Decision Point 2 by the One Step method, improvement of the yield of wafers meeting the leak specification was quite slow. In addition, the average leak rate for wafers obtained using the Two Step method were lower than that of those prepared with the One Step method. Since development of the Two Step process was initiated significantly later than the One Step process, relatively few wafers made with the Two Step process could be made prior to Decision Point 2. Nevertheless, it was shown that both types of wafers could cycle rapidly and that they could also be joined into submodules, although sufficient parts with low leak rates, were not available to make components with low enough leak rates to generate high purity oxygen. Three main areas requiring further development were identified: materials selection, process improvement, and assembly methods.

2.2. Results

2.2.1 Materials Selection

Initial investigation of advanced architecture wafers indicated that the material microstructure had to be engineered to meet requirements of product purity, chemical and thermal equilibration, and manufacturing simultaneously. The desired microstructure must be strong enough to handle the load required during assembly processes, yet must achieve the benefits of significantly reduced chemical expansion-induced residual stress, relative to standard structures. In addition, there is a concern that the materials may creep excessively during joining and possibly over the desired operational lifetime. To address these issues, studies were undertaken to develop processes capable of adjusting the microstructure of the material and its corresponding properties. An investigation of the effect of processing on microstructure and the resulting strength, creep behavior, shrinkage rates, elastic properties, thermal properties, and chemical expansion behavior was conducted. The starting powder properties and the effect of sintering temperature were characterized to allow engineering of the appropriate wafer powder stream and processing conditions.

Another constraint on advanced architecture materials is that they must be compatible with achieving an intact outer membrane. As described earlier, this can be accomplished either by fabricating a support structure that is amenable to application of a membrane that can subsequently be densified onto the support or by fabricating a structure that undergoes transformations along with the outer layer. Ceramatec investigated both approaches for wafer fabrication.

Support structures were selected for One Step route based on results of previous research in Phase III. The supports were made from powder that was processed to create a desired powder morphology. The powder morphology and process was chosen because it had been used to make advanced structures for module endcaps (domes) and basepeices earlier in Phase III and had the mechanical and chemical properties appeared to be adequate for those applications.

Selecting the powder morphology and process for making material for the Two Step method required additional experimental testing. The effect of starting powder, tape processing conditions, and sintering temperature on the material properties of standard control samples were measured. The following is a summary of the test parameters:

Starting powder: several different powders were examined and compared to the powder source used to produce internal support layers in standard wafers, since they are known to be compatible with the membrane layer. This powder is produced by milling calcined powder so that it passes through mesh screen, followed by additional particle size reduction and mixing. Some powders were mixed with higher surface area powder to study the effects of powder mixtures in controlling material properties.

Tape Processing: three basic processes were compared – the on-line production process for making tapes, small scale batching and casting using a labscale tape caster, and the blending of powders and organic materials using commercial dispersing equipment. All labscale casts were batched using the same formulations on the on-line production process. The formulation used with the disperser was made with reduced binder content which generally adjusts tape properties relative to those achieved with simple mixing.

Sintering Temperature: Control samples were sintered over a range of temperatures. Samples were held at temperature several hours to achieve near-final density.

The study indicated that a substantial amount of control over material layer properties could be exerted by selection of the correct particle morphology and sintering temperature. The sintering temperature was subsequently fixed at a temperature known to provide densification of the membrane layer and to be desirable from the perspective of operating and maintenance costs of kilns.

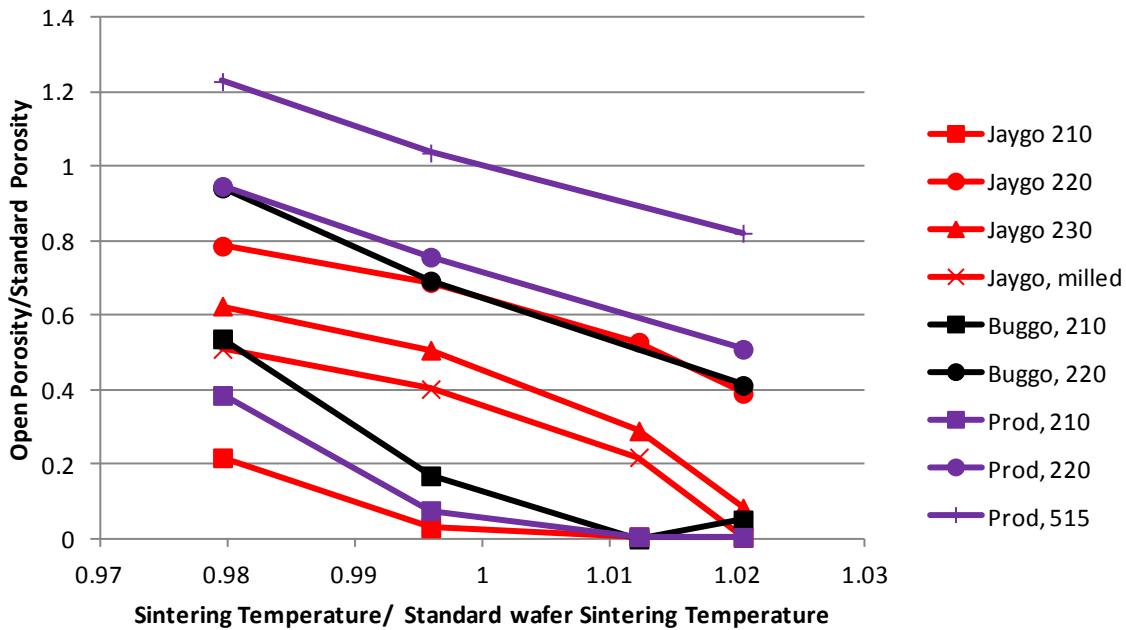


Figure 2.2.1 shows the effects of different starting powders on open porosity as a function of sintering temperature for a variety of different powder morphologies. As can be seen, the choice of starting powder can control part porosity, ranging from the porosity of standard wafers to no measurable open porosity. The data show tapes from different processing routes using the same powders.

As mentioned earlier, the shrinkage of the tapes for the inner layers must be compatible with the membrane tape shrinkage.

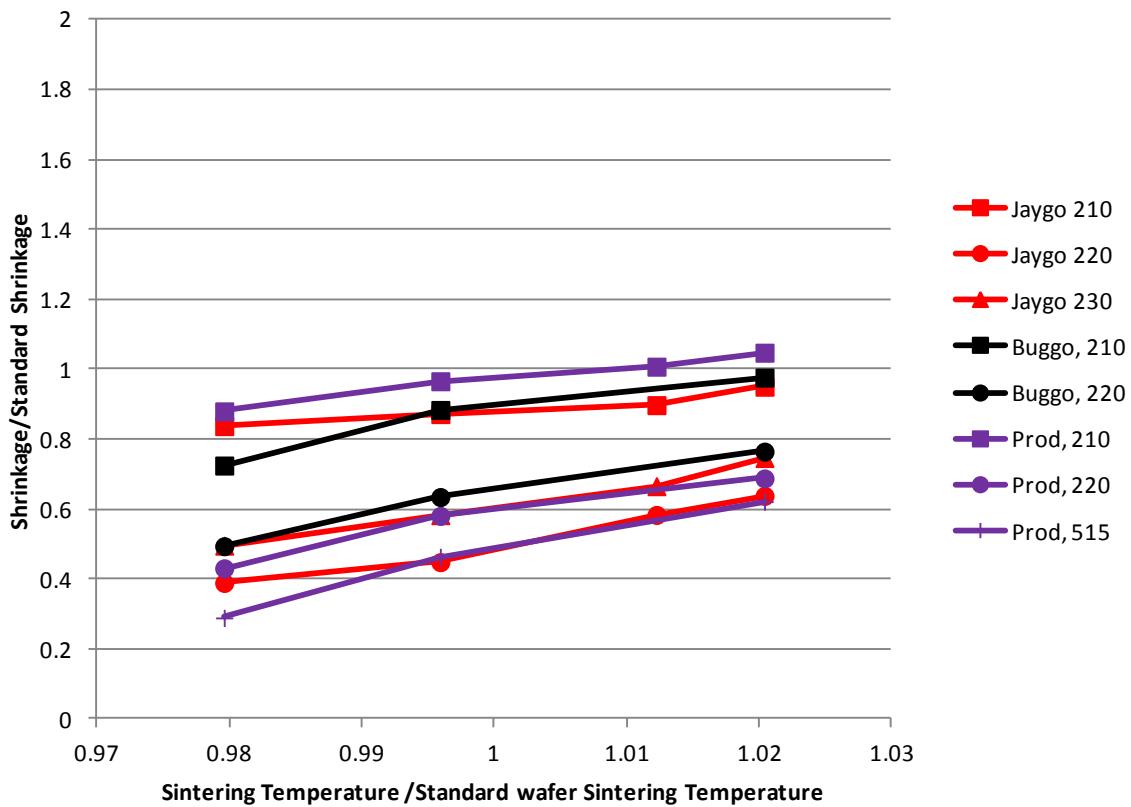


Figure 2.2.2 shows the shrinkage for the tapes shown in

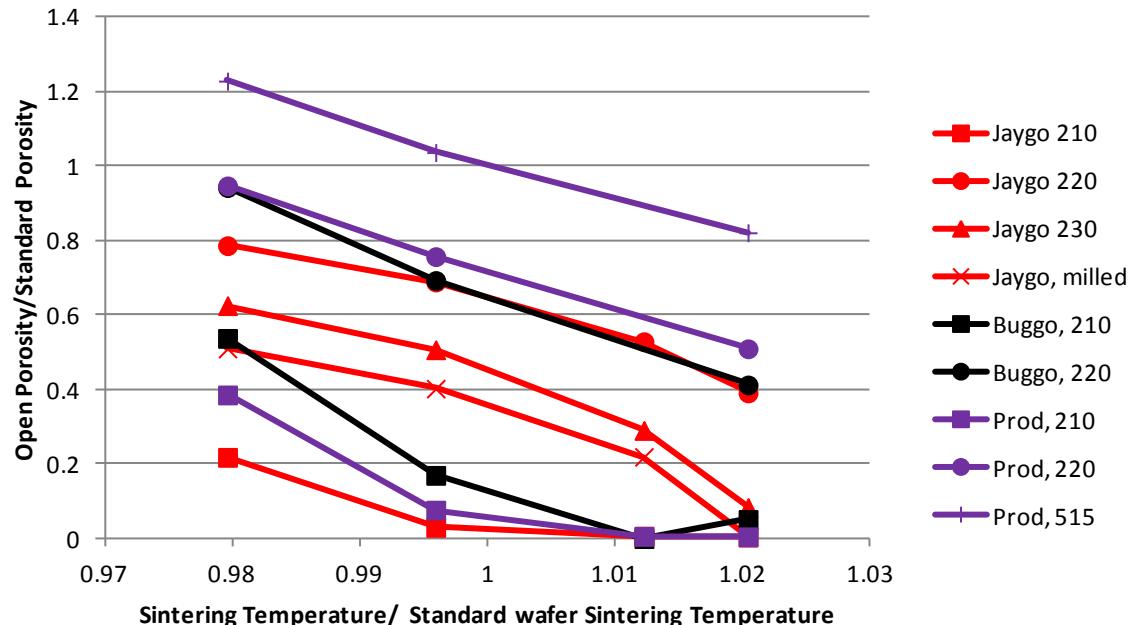


Figure 2.2.1, and shows that all of the tapes with higher porosities have much lower shrinkages. For comparison, the porosity values of a series of tapes made for a support layer

in standard wafers are shown in

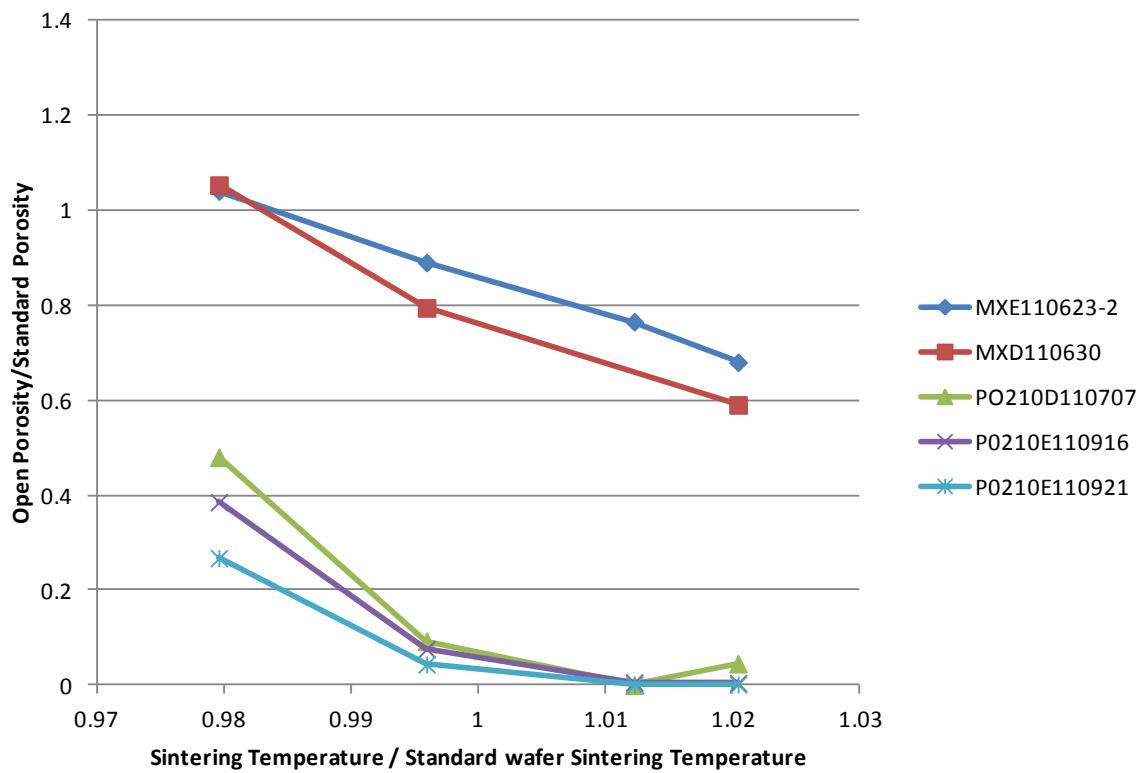


Figure 2.2.3. This figure shows typical variation from lot to lot. Also shown are the results for three tapes made with similar starting powders. The shrinkage of the tapes is shown in

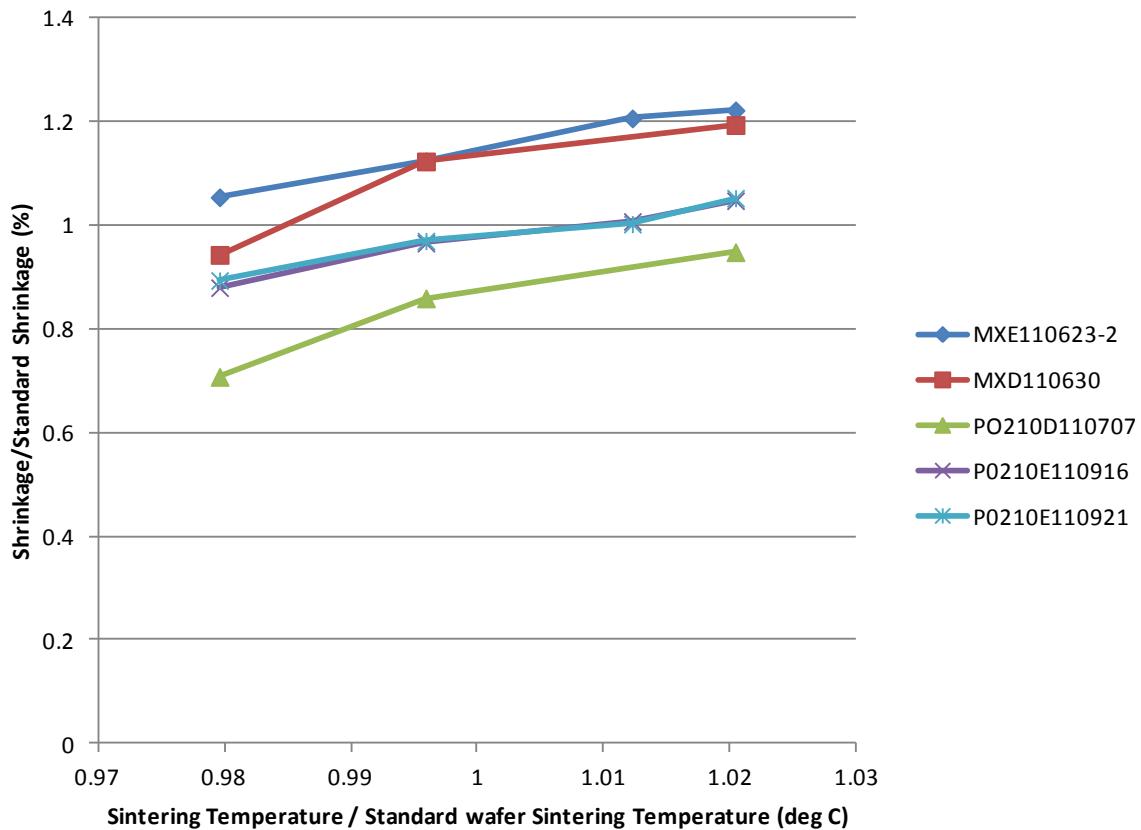


Figure 2.2.4. Based on these results, it is fairly clear that the “210” series material is the best fit to co-sinter with membrane tapes.

It should be noted that the 210-series powder has similar particle size distribution to the screened powder produced for standard wafers. The particle size distributions of several typical lots of powder from the screened powder source after being milled as per procedure and cast into tape are compared with those for several 210-series powder lots in

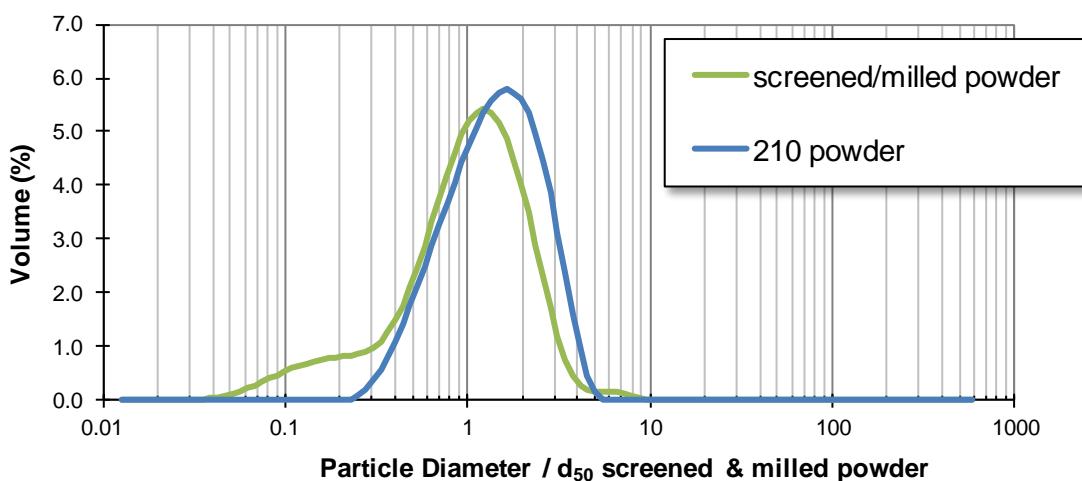


Figure 2.2.5. The only real difference is a distribution of finer powder, which has only a minor effect on tape sintering properties.

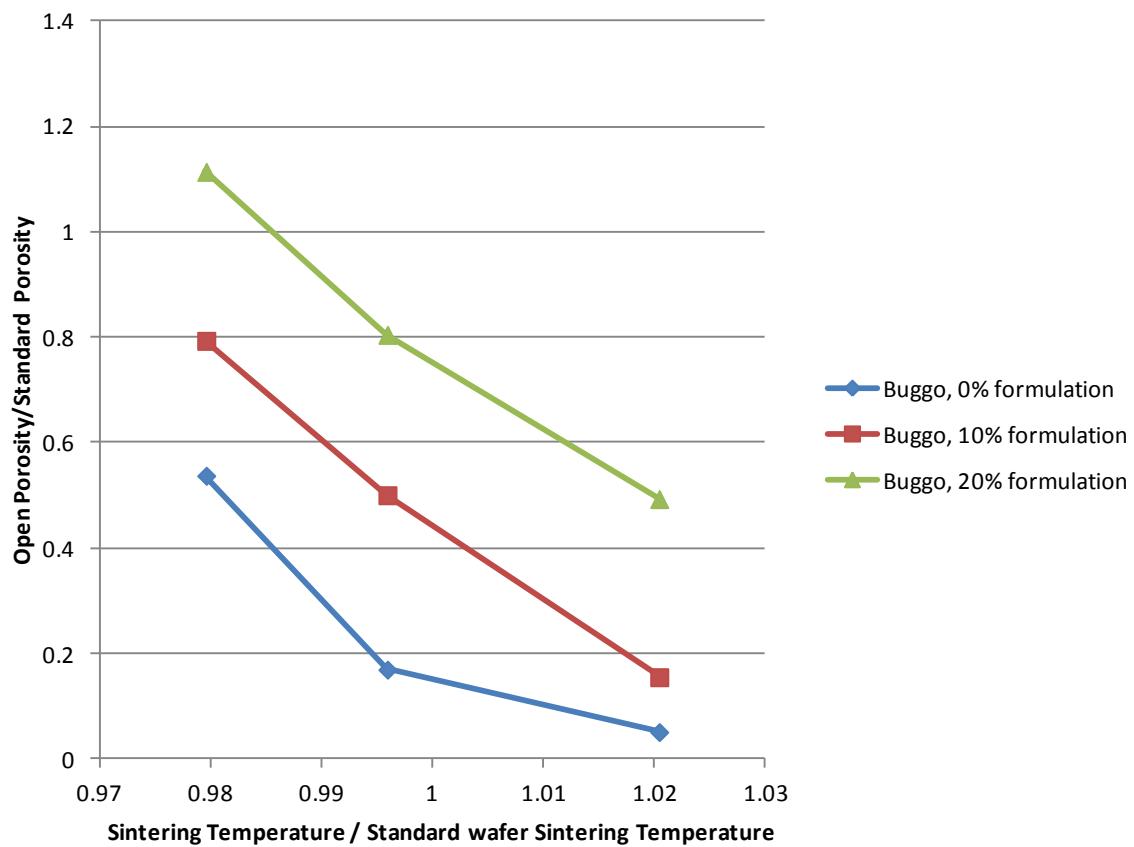


Figure 2.2.6 shows the effects of different slip formulations on the various powders . Here the data show that adjusting the slip formulation is effective in adjusting the porosity.

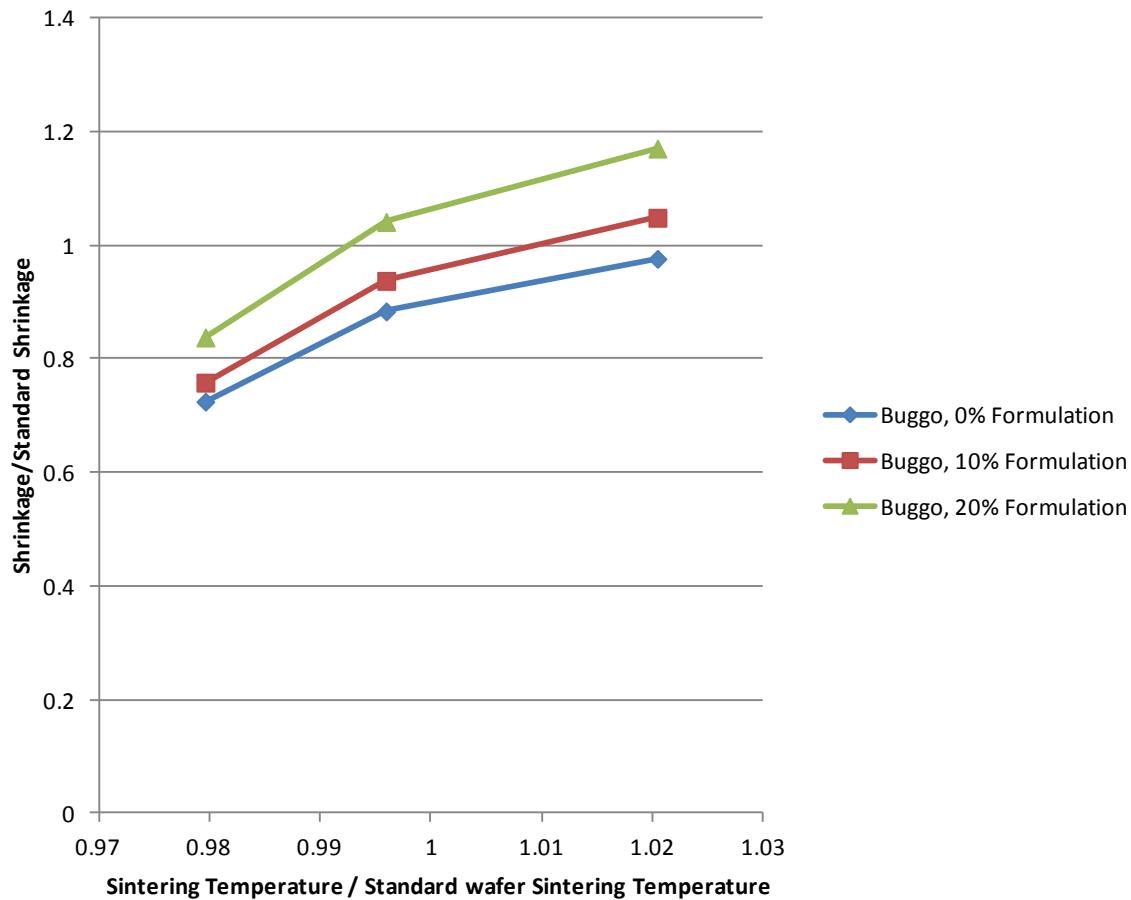


Figure 2.2.7 shows the shrinkage as a function of formulation.

Finally, one option available to control layer properties is the alteration of particle morphology. To this end, a baseline of the 210- and 220-series powders was selected and additions of 0, 10 and 20% fine powders were made. The results are shown in

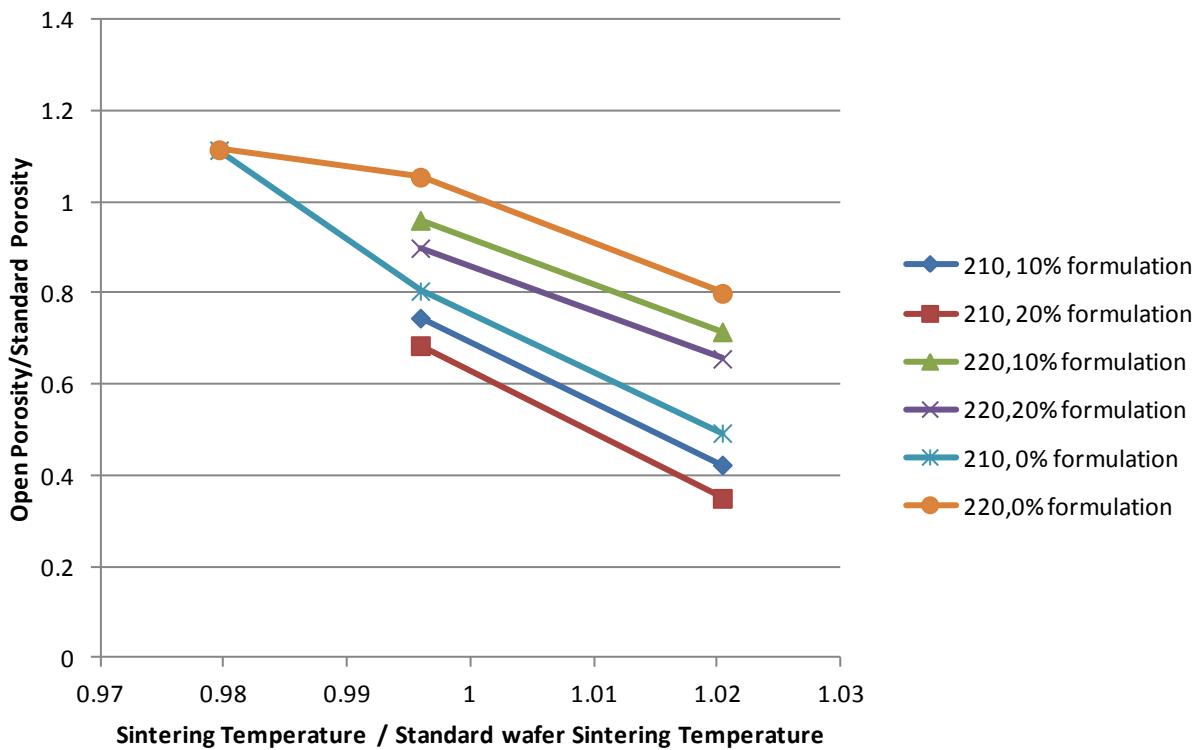


Figure 2.2.8 and

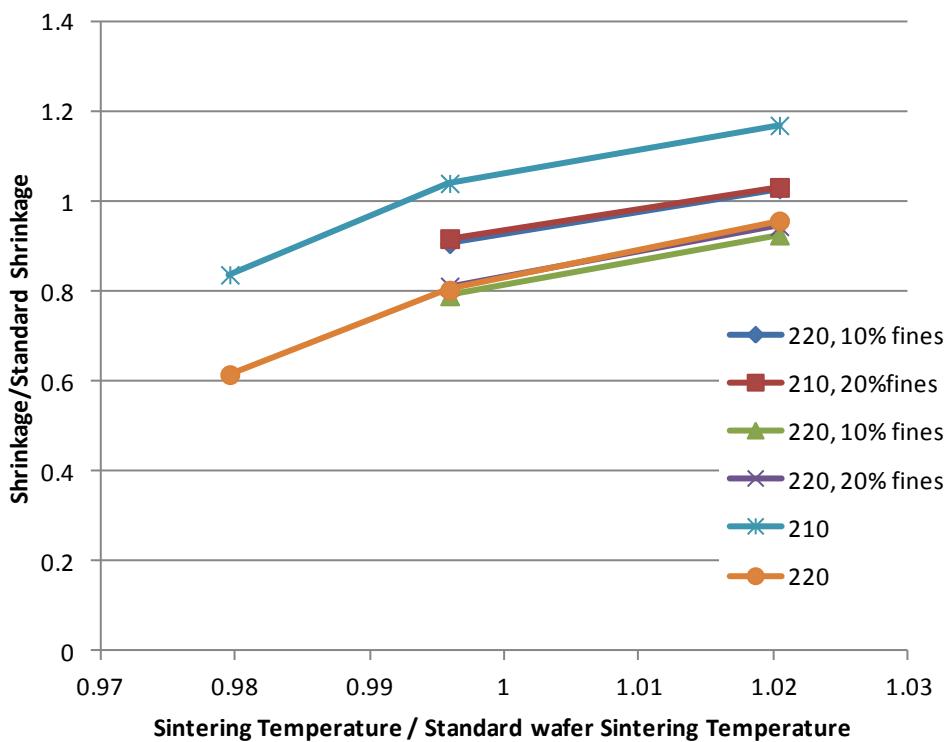


Figure 2.2.9 below, and show that the addition of finer powder has a lesser effect on

porosity than anticipated. In particular, the effects on shrinkage were minimal, likely due to the improved packing of a bi-modal distribution of powders.

From these data, the 210 series powder was selected as the best powder for production of wafers, although the results indicate that some slip re-formulation may be optimal.

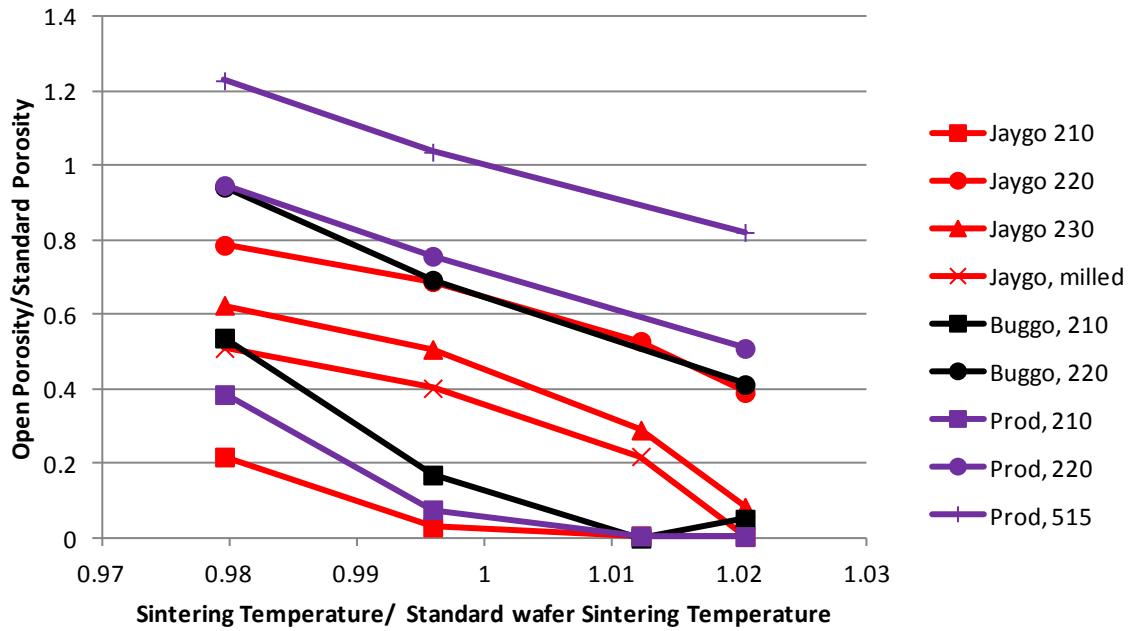


Figure 2.2.1 – Open porosity vs. sintering temperature for various tapes made with different powders.

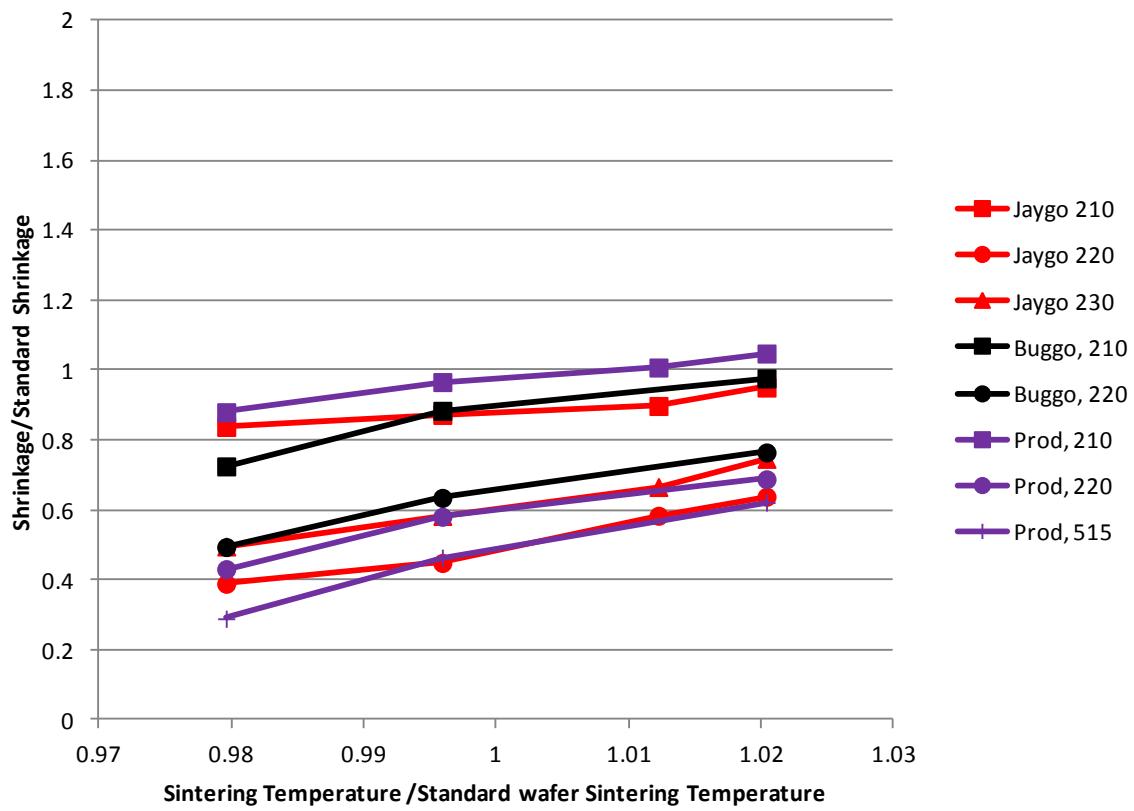


Figure 2.2.2 – Shrinkage vs. sintering temperature for various tapes made with different powders.

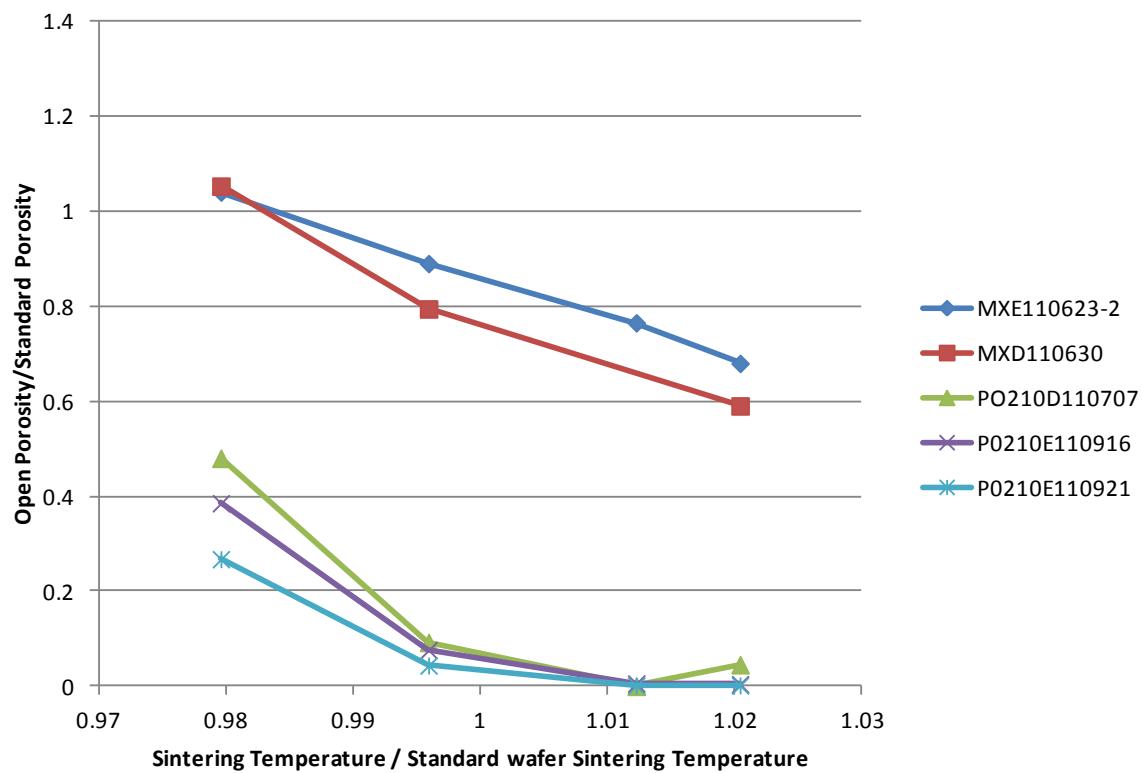


Figure 2.2.3 – Open porosity as a function of sintering temperature for tape lots made with sieved powder (MX), and inner layer tapes (PO) made with sieved powder.

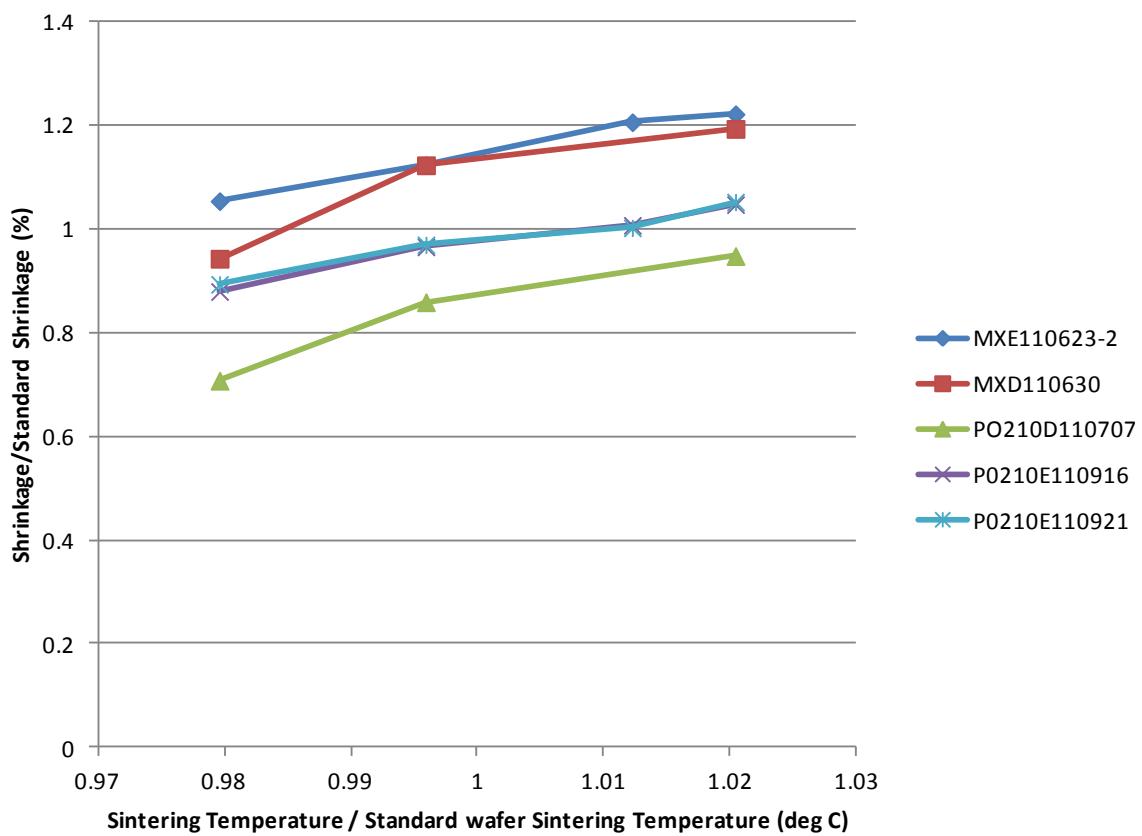


Figure 2.2.4 – Shrinkage as a function of sintering temperature of cast tapes.

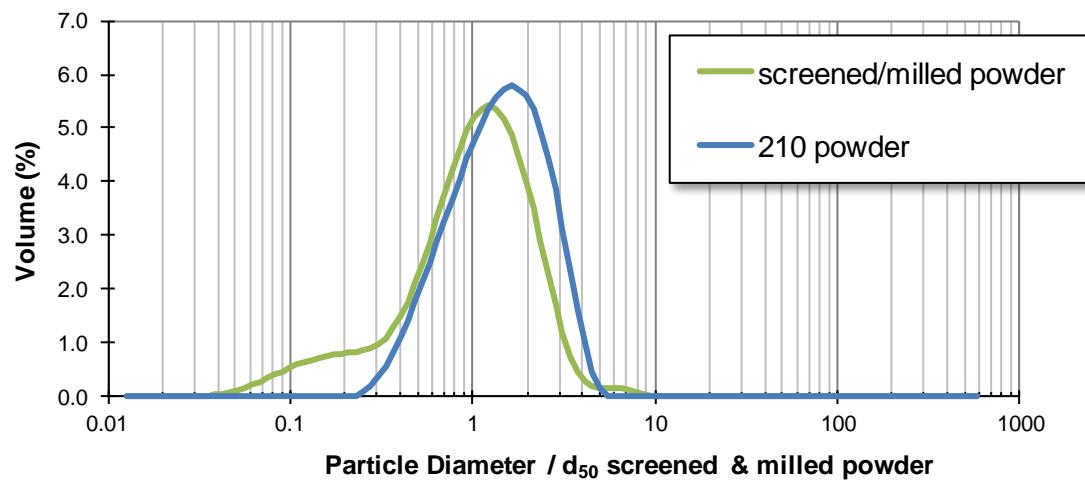


Figure 2.2.5 – Overlay of the PSDs of milled screened powder and "210" series powder, which shows similar particle sizes except for some fine particle distribution.

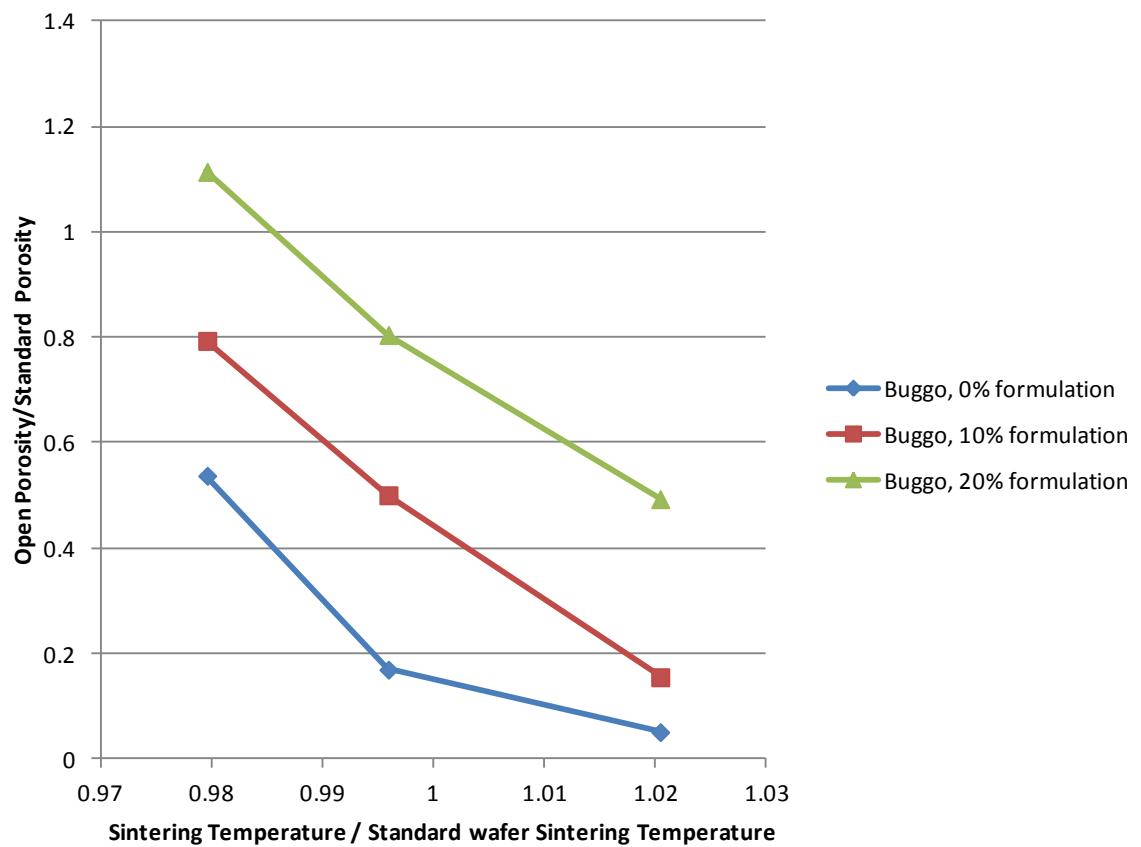


Figure 2.2.6 – Open porosity vs. sintering temperature for labscale cast tapes made from 210-series powder for different slip formulations .

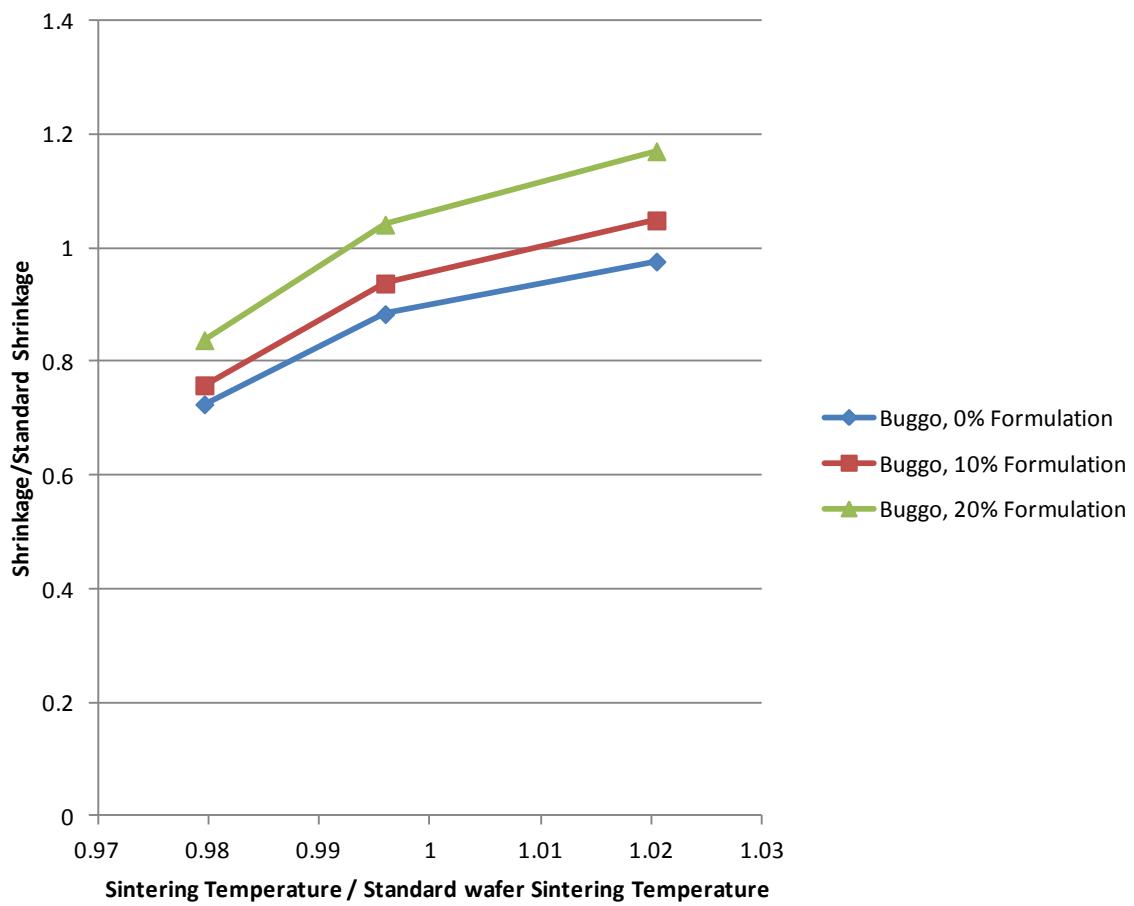


Figure 2.2.7 – Shrinkage vs. sintering temperature for labscale cast tapes made from 210-series powder for different slip formulations.

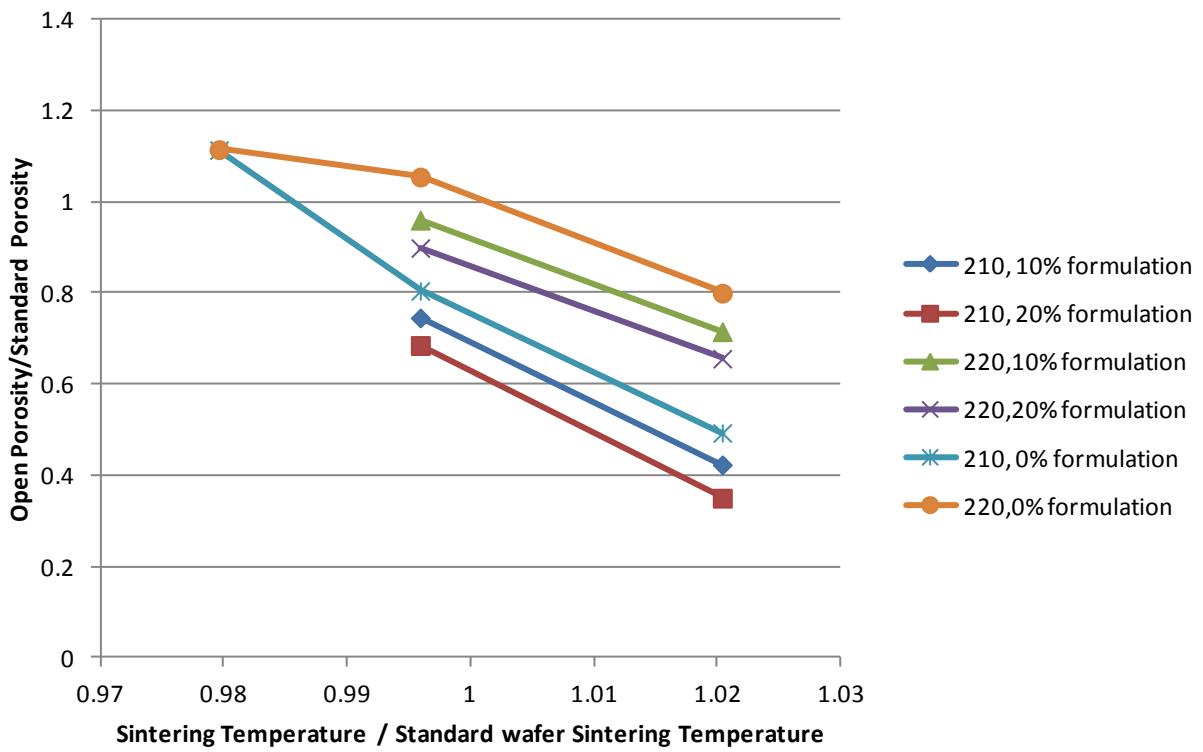


Figure 2.2.8 – Open porosity vs. sintering temperature for 210- and 220-series powders cast with various amounts of fine powder additions.

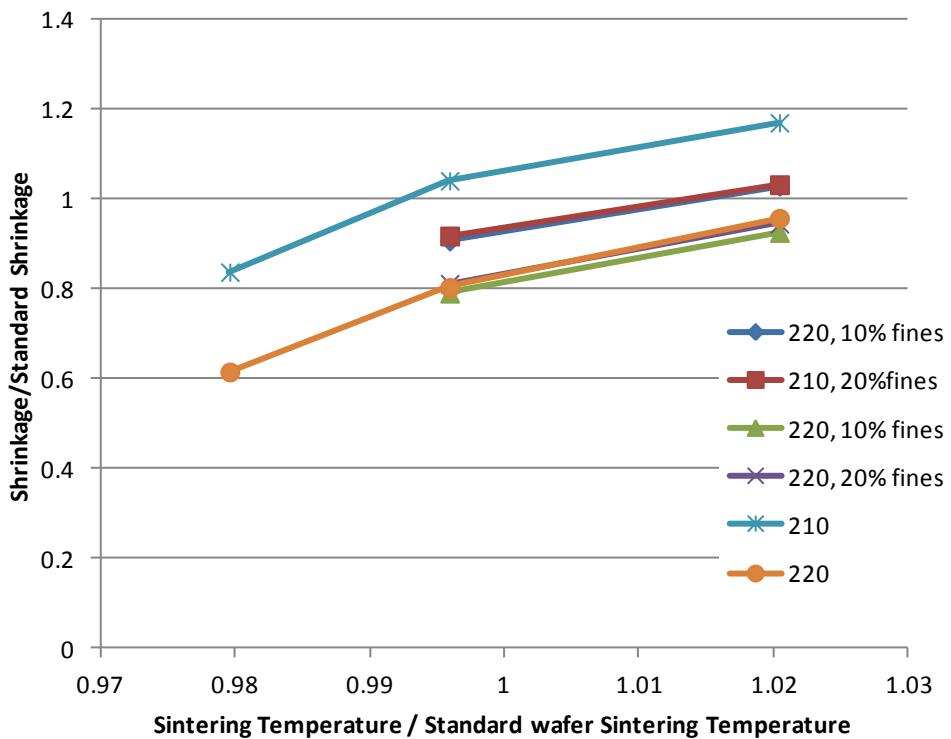


Figure 2.2.9 Shrinkage vs. sintering temperature for tapes with 210- and 220-series powder cast with varying amounts of fine powder additions.

2.2.2. Process Improvement

Subsequent to selection of the materials for advanced architecture wafers, viable processes for producing these wafers had to be developed. A manual system for executing the One Step process was designed and used by operators to produce approximately 2,000 wafers. At the same time, lab scale work was directed at investigations of the Two Step process using similar methodology. Multiple thermal cycles were used to achieve a leak tight component. The lab scale work produced the best results for wafers.

As efforts continued, the results indicated that the Two Step approach was more successful than the One Step approach. This is not surprising because, there is a higher probability for defects to occur during the One Step process. Typical leak rate distributions for wafers made by the One Step process are shown in Figure 2.2.10. Therefore, in Q4FY11, a decision was made to focus efforts only on wafers made with the Two Step process.

As mentioned earlier, initial wafers were made using a screened powder and processes for making the standard wafer internals because they were known to have good compatibility with the tape membrane. However, the morphology of the screened powder is not ideal for advanced architecture wafers because it leads to low strength materials.

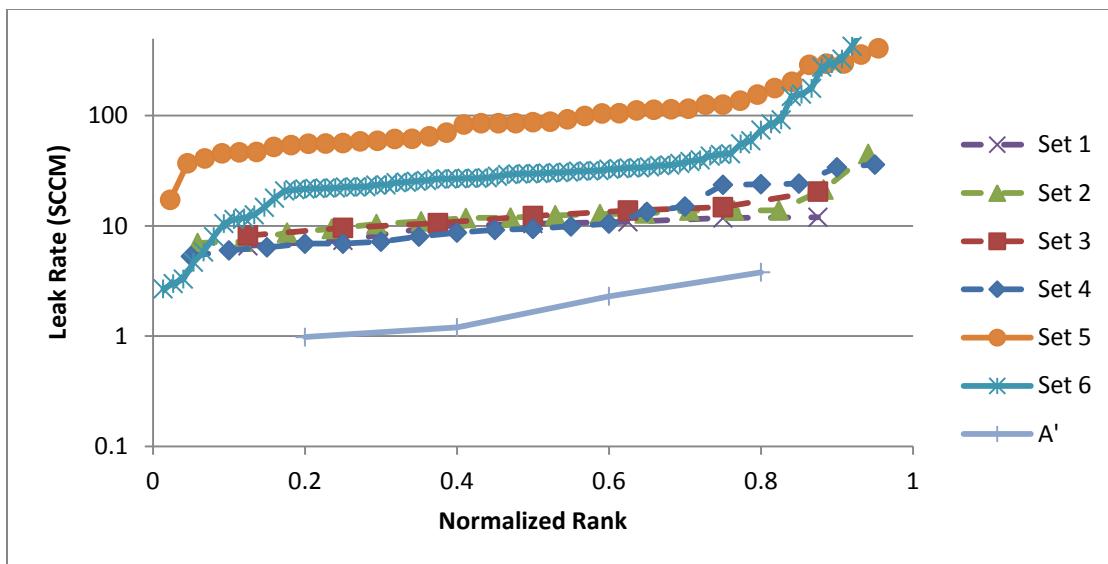


Figure 2.2.10 - Leak rate distributions for wafers made by the One Step process.

Measurements were made of both the strength values and compressive creep rate values of wafers made using screened powder process and although wafers equilibrated rapidly during thermal cycles and could be joined successfully, several wafers cracked during joining. In addition, the process for making powder and slip for these wafers using the screened powder is labor intensive and inefficient.

While efforts on material selection were concluding, wafers using the screened powder process were fabricated since wafers meeting leak rate specifications could be made in reasonable quantities by production operators. At the same time, work conducted in lab scale experiments to improve the yield of wafers demonstrated that equivalent or better yields of “in-spec.” wafers could be obtained by the Two Step method with a relatively low number of thermal cycles. After the desired 210-series powder stream was identified, wafers were made with the improved Two Step method. The combination of the selection of the 210 powder and the appropriate thermal cycles proved successful as equivalent or higher yields of in-spec. wafers were obtained than those for wafers made from screened powder. Pending joining results, the powder stream and processes for making these wafers will be adopted as the base-line method for making 1 TPD modules for performance testing.

2.2.3 Assembly Methods

Testing and development of submodule and module assembly methods also was conducted concurrently with work on materials selection and component development. This approach provided additional information relevant to component development as well as accelerated the implementation of selected materials and components. Initially during component development, wafers were made without spacers to save resources but allow for development of processing techniques. In joining experiments, spacers made from the same powder stream as the wafers were used in conjunction with these wafers. In addition, joining experiments were initiated prior to production of significant amounts of in-spec. wafers. In these experiments the success of the joining cycle could only be determined by the presence or lack of cracks, resulting flatness and alignment of components, and a limited amount of cross-sectional microscopy.

Joining results for wafers made with the One Step process from the 515-series powder, indicated that they were not strong enough to survive joining. Out of approximately 18 submodules made with wafers and separate spacers, only one survived without visible signs of cracking. Wafers made from an alternative 210-series powder were selected for wafer production before One Step wafers could be produced and joined.

Results of joining tests using wafers made with the Two Step method and using screened powder were much more encouraging. Again, wafers without spacers were used for initial testing. Only one of three stacks of three wafers survived the first attempt at joining without visible signs of cracking. Two types of spacers were processed with this type of wafer: (1) improved, standard spacers, referred to as the “OD2 spacer”; and (2) unsealed advanced architecture spacers. Using the OD2 spacer six out of six stacks of three wafers survived joining without visible signs of cracking. Using the unsealed advanced architecture spacer, thirty out of thirty-three submodules survived joining without visible signs of cracking. Obviously, the presence of a spacer improves the ability of wafers to survive the joining process. The thirty submodules were used to build two, 1-TPD modules and one, 0.5-TPD module.

Parameters for joining the 0.5 TPD module were determined during the joining process. This module came out with the top sub-module cracked down the middle. At that point, joining parameters for the two 1-TPD modules were altered *in situ*. Both of the 1-TPD modules were joined successfully without any signs of cracking or wafer failure. An image of one of the modules in the assembly furnace is shown in Figure 2.2.11. Since the wafer and spacers used for these experiments were not capable of producing high purity oxygen, leak rates for these modules could not be measured.

Initial attempts to join wafers made from the 210 series powder stream were also unsuccessful. It was found that the multiple thermal cycles used to obtain leak tight wafers diminished the ability of the modules to undergo subsequent thermal cycles. These experiments, therefore, discovered a limitation of the wafer processing method that was not apparent from leak rate measurements. The introduction of the Two Step method for

preparing wafers followed by limited thermal processing allowed for leak tight wafers to be made with sufficient



Figure 2.2.11 – One of the first 1-TPD modules made entirely from Advanced Architecture wafers in the assembly furnace.

ability to survive thermal cycles required for joining. A set of three sub-modules using these wafers were joined. The wafers were joined using standard procedures and materials and reduced thermal cycling. All three sub-modules came out intact, with leak rates lower than the sum of the starting wafers. A summary of leak rates of the wafers before and after sub-module joining are shown in Table 2.2.1. The decreasing leak rates may be due to the apparatus making the measurements, but the final leak rates have been confirmed. The results show that the initial joining trials for these new wafers were successful, with no major issues with either wafer integrity or leaks in the ceramic joints. Experiments on joining wafers made from the 210 series powder with slip modifications will be conducted in December.

Table 2.2.1 Leak rates of wafers and resulting sub-modules built from wafer made with 210 series powder.

Sub-Module ID	Leak Rates (sccm)	
	Sum of Wafers	Joined Sub-Module
TMLB3111-1	52.5	39.6
TMLB3111-2	85.0	80.1
TMLB3111-3	192.8	200.6

2.2.4. Summary

A powder stream, the “210” series, and a process for making it have been selected. A Two Step process for producing advanced architecture wafers has been developed and selected. In order for the selected powder to be compatible with the tape membrane, it was found that modifications to the powder are desirable. Currently, yields of wafers meeting leak-rate specification using the modified 210 powder have been encouraging. Submodules meeting leak rate specification have been made using wafers derived from various unmodified powders. Submodules made from wafers using modified powders will be joined in December.

2.3 Expected Future Work

Significant progress on the Advanced Module Development task has been made since DP2. Additional efforts on material characterization and selection are required to ensure that the component manufacturing yield and subsequent performance are acceptable. Further process definition and improvement are required also. The reliability of advanced modules and components needs to be assessed to ensure long-term success.

Efforts to date have identified a design window for advanced architecture wafers that can equilibrate during thermal cycles and that are compatible with the ITM. Future work must focus on confirming that the selected materials and processes produce components that also meet the requirements for joining, creep lifetime, shipping and handling, etc. Since the current yield of wafers capable of meeting leak rate specification needs improvement, additional process engineering is required, particularly in the areas of tape casting and thermal processing.

Although high volume manufacturing has always been considered during development of the methods used to fabricate advanced architecture components, this must be

demonstrated on a scale relevant to large-scale production. Although most process steps performed during fabrication of advanced architecture wafers utilize the same or similar equipment to that used for standard wafers, powder preparation equipment and membrane application equipment for large-scale manufacturing need to be specified and qualified. Although the primary path advanced architecture modules offer for reducing manufacturing cost is by increasing yield, due to the ability of such wafers to survive faster thermal cycles than standard ones, appropriate thermal profiles need to be developed and qualified. Furthermore, production of advanced architecture modules may lead to a different materials balance in the process flow with regard to reclaiming and utilizing scrap material. In summary, process conditions for advanced wafer, submodule, and module production need to be further refined.

In addition to developing advanced architecture components that are more durable with respect to required assembly conditions, additional design and analysis will also be performed to enhance the performance of modules with advanced components. These engineering efforts will focus on joint design, joint materials, and joint fabrication methods. The joint must meet several requirements simultaneously: compliance with wafer surface features during assembly, the ability to form a leak tight bond under conditions that do not negatively impact other components of the module, and sufficient mechanical durability for forces imposed during handling and operation. In addition to modifying the joint, the region of the wafer and the spacer around the joining area can be modified to improve the behavior of joints. Alternative methods of joining, or thermal cycles, may also be required for assembly of modules with advanced components or to improve their yield and performance.

Since mechanical failure of ceramic components is a potential reliability limiting mechanism for ITM systems, efforts will be made to characterize the reliability of modules with advanced components. The same approach as has been used to improve reliability of standard modules will be used: characterization of material strength distributions, numerical stress analysis, and experimental validation of reliability. The effects of process parameters on the material reliability on strength will be examined thoroughly. The objective of these efforts will be to identify materials, architectures, and operating conditions that obtain the most benefit from the ability of advanced architectures to reduce stresses in components without sacrificing reliability in other ways.

2.4 Specific Equipment and Processes Required for Introduction of Advanced Architecture Module Production in the CerFab

2.4.1. Equipment Requirements

The efforts on materials selection, process improvement, and assembly methods have confirmed that advanced architecture wafers, made using specific powder streams, can be made with conventional manufacturing techniques that can be sintered with modest thermal process requirements. Although additional details regarding the materials and processes remain to be determined, the basic equipment and process requirements for production of advanced architecture modules in the CerFab have been identified. Since multi-scale efforts to improve advanced component yield and performance continue, some equipment or processes to be used at the CerFab also require development.

One of the principal pieces of equipment required for introduction of advanced architecture module processing in the CerFab is powder processing system for producing the desired powder stream. Ceramatec has identified several companies that offer equipment capable of producing the desired powder stream; equipment specifications have been written by the CerFab engineering team as part of Task 30.

Currently, the advanced architecture wafers are processed by hand at Ceramatec using tooling and fixtures to obtain consistent results. A difference in the yield of in-spec. wafers has been observed for wafers processed with different tool sets. Therefore, additional work is required to determine the tool set, and corresponding procedure, to obtain a high yield, manufacturing friendly process. Subsequently, this process must be scaled for CerFab production, which is envisioned to include the use of automated wafer processing equipment.

In addition to equipment for producing powder for advanced architecture modules, equipment and processes are required for preparing slip (the mixture of organics and powder used for tape casting) and for casting modified slip at high production rates. Equipment for slip preparation has been specified, based on scaling the size of equipment used during Phase III at Ceramatec, and a new tape caster, similar to that specified for the CerFab, is expected to arrive at Ceramatec in Q2FY12. These tools will be used to develop the processes that will be practiced at the CerFab.

Another tool required for high volume production at the CerFab is a featuring machine for featuring the internal layers of the wafers. The tool required for the CerFab will use a more concentrated featuring pattern than has been used in typical applications. To address the customization of the featuring tool, an automated tool was specified and tested during Phase III and is expected to arrive at Ceramatec in Q2FY12. This tool will be used to develop and verify the processes that will be practiced in the CerFab.

Finally, to control capital equipment costs for the CerFab, the maximum length of the thermal cycles for wafer sintering, submodule joining, and module joining have been

identified. Although experimental cycles of almost the required length have been tested on a small scale, appropriate cycles that ensure high yields with larger equipment must be developed for the CerFab. These cycles will be developed using the furnaces available at Ceramatec.

2.4.2. Summary

Significant progress has been made since DP2 in materials selection and process development and improvement for advanced architecture module fabrication. Although assembly methods have been demonstrated for several versions of advanced architecture designs, these methods must be validated with the material and processes selected. Equipment and processes required for implementation of the advanced architecture design in the CerFab have been identified and are included in the Task 30 scope of equipment supply. Plans for additional process development focused on yield enhancement have been made.

3. Summary of Status of Task 29.0 “Engineering Development for Industrial and CCS Applications”

Task 29.0 is focused on engineering development of specific implementations of ITM Oxygen technology for oxygen/power production, and has two components: work on processes that feature or achieve low carbon dioxide emissions, and work on a 2000 TPD test unit design. Work up to Decision Point 2 was mainly focused on the former task, specifically Task 29.1.1.1 (“Process cycle and process economics development”) and identifying applications that require carbon reduction; work since Decision Point 2 has been primarily focused on the 2000 TPD test unit design, Task 29.1.2.1 (“Process cycle development”).

Air Products embarked on an effort to explore synergies of an ITM unit coexisting with a cryo ASU unit – the Hybrid ASU concept. *Prima facie*, it may appear somewhat quixotic that a hot ITM unit coexist synergistically with a super-cold cryo unit. Nevertheless, the concept appears to have some advantages. It is well known that almost all ITM Oxygen units have a need to generate excess power through expansion of the pressurized, hot vitiate – this is an integral part of the value of the technology. It is also well known that cryogenic ASU’s typically require electric power to drive the separation and refrigeration. Air Products developed a conceptual facility that overall makes 2000 TPD of gaseous oxygen (GOX) as a product. About 36% of the GOX is furnished by the ITM unit. The high pressure ITM vitiate further generates a net surplus of power, which is captively used by the Cryo ASU to make the remaining 64% of the GOX product. The overall facility is *karmaneutral* – that is, there is no net import or export of electric power. This particular facility is also configured to export pressurized gaseous nitrogen (GAN), as well as Liquid Argon (LAR) – all sourced from the cryo ASU alone. However, the ASU can easily be configured to vent the nitrogen and argon as gases at near ambient conditions.

Having a facility that is *karmaneutral* offers an advantage in siting, because the combined system is not subject to the requirements of an external customer of the cogenerated power. Further, the cryogenic unit backs up the ITM unit should the ITM unit need to be shut down for routine maintenance or by an unplanned event. Cryogenic technology is mature and has on-stream factors greater than 95%. The ITM test unit is expected to have the operating profile of a new technology, namely, more frequent startups and shutdowns than the plant based on the incumbent cryogenic technology. A commercial customer facility for oxygen at this scale would need a reliable supply. Thus having a cryogenic ASU as backup provides an alternative to a pipeline. The cryogenic facility can also be configured to have a liquefier and provide liquid storage for a few days, which provides further backup. The multiple gaseous and liquid products from the cryo ASU may also have commercial value to offset, in part, the cost of the test unit.

Table 3.1 below offers a preliminary glance at the relative utility consumption of such a Hybrid system (supplied only with natural gas (NG) fuel), relative to a cryo-only oxygen plant of identical oxygen and nitrogen production (supplied primarily with electric power)

at typical projected power and natural gas prices. The cryo -only system requires only electricity. The Hybrid system requires only natural gas. The fuel-to-electricity conversion implicit here is 57% (LHV basis), which is a respectable number compared to NGCC plants.

Table 3.1. Product flow and power input for an example Hybrid Cryo-ITM system.

	CRYO - ONLY	CRYO-ITM HYBRID
GOX, Nm ³ /h	58,380	58,380
GAN, Nm ³ /h	29,220	29,220
LAR, Nm ³ /h	2,005	1,060
5 yr POWER COST	\$138 MM	- \$1 MM
5 yr FUEL COST	\$0.8 MM	\$71 MM
5 yr UTILITY COST	\$139 MM	\$70 MM

A key decision variable is the configuration of air compression. A conventional gas turbine compresses air adiabatically, fires it with fuel in an integrated combustor, and then expands the hot pressurized offgas in an integral expander mounted on the same shaft as the compressor. The heat of compression is preserved in the system at the cost of increased compression power requirement. In contrast, the main air compressor (MAC) of a cryogenic ASU typically attempts to simulate isothermal compression by employing several intercooled stages of compression. The goal here is to minimize compression power, at the cost of low-grade heat discarded into cooling media. One of the goals of an integrated cryo+ITM Hybrid facility is to explore the possibility of integrating air compression, benefiting from economy of scale and higher efficiencies commercially available for larger scale equipment. Thus it is useful to explore staged-intercooled (SI) compression for the ITM unit. Of course, such a scheme bifurcates the innards of a conventional Gas Turbine into its constituent parts. The adiabatic compression portion is not necessary any more. Only the pressurized combustor(s) and the turbine need be retained, in a so-called “Combustor-expander”.

Figure 3.1 depicts one possible simulation of an ITM O₂ plant. Ambient air is compressed to 30 bara in a SI compression train, as discussed above. It is then preheated indirectly by heat exchange and then routed to the ITM unit. The oxygen is collected at 3 psig. The ITM oxygen is cooled by heat exchange, and then compressed to the product pressure of 31 bara.

The vitiated air from the ITM unit is fired with a modest amount of fuel in a pressurized combustor to source the majority of high grade heat required to preheat the ITM feed air. It still retains about 10% O₂, which is both sufficient to ensure a stable flame, as well as provide the oxygen in stoichiometric excess to combust the preheated turbine fuel. The turbine exhaust heat is recovered by heat exchange against incoming air, before being vented through a stack.

Another key feature in this flowsheet is the heat exchange network, which involves heat exchange to high temperatures.

One of the streams to be cooled is oxygen. Oxygen aggressively attacks metallic materials. Future work will examine the trade-offs with oxygen cooling schemes versus the cooling scheme as proposed here. We also need to investigate materials design for these schemes.

The oxygen permeate from the ITM unit can be pulled off at a vacuum. Operating the oxygen circuit below ambient conditions causes large heat exchange surfaces. In the example flowsheet, these issues have been avoided by pulling the oxygen off at a modest pressure of 3 psig. To maintain a reasonable driving force, the feed pressure has also been boosted to 30 bar. The ITM module operation and design needs to accommodate these elevated feed pressures.

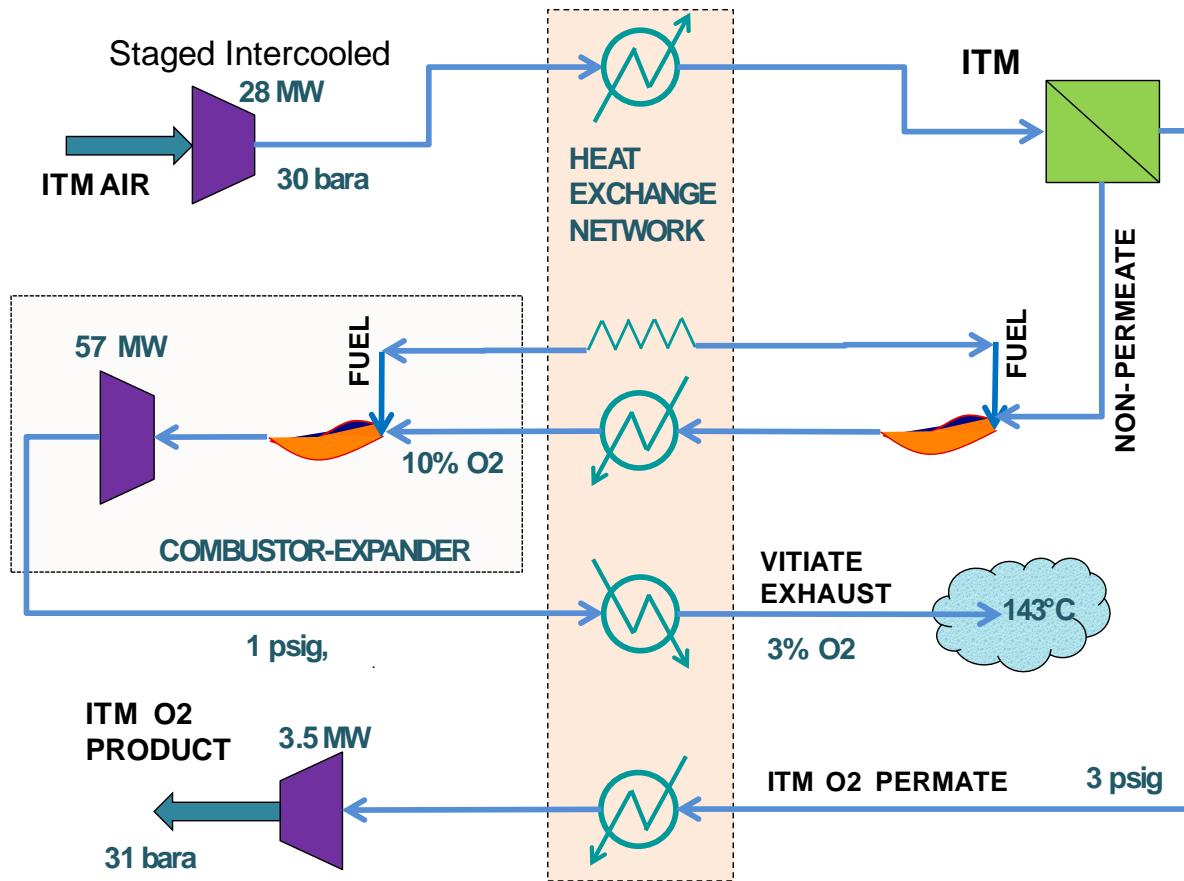


Figure 3.1. ITM Oxygen flow schematic with staged/intercooled compressor and separate combustor-expander.

A feature of the Hybrid concept studied here is that even though the overall facility makes 2000 TPD oxygen, the ITM plant itself generates only a portion of this – about 720 TPD. This is not an adequate scale-up from the 100 TPD ISTU of Phase III to be representative of large-scale oxygen production typical of low-carbon energy facilities. To scale the ITM facility to generate 2000 TPD on its own, then a *karmaneutral* Hybrid facility threefold as large is required. Current opportunities in North America for oxygen plants of such magnitude include gasification, carbon-to-chemicals and decarbonized power applications. Another alternative would be to have a Hybrid facility, but not insist it be *karmaneutral*. The bigger the ITM’s share of the oxygen generation, the more the power in excess of what can be captively used by the cryo unit. It is possible that this power will meet some or all the requirement of the oxygen-consuming facility as well.

The other feature that merits attention is the nitrogen coproduction requirement. IGCC requires compressed nitrogen co-product for the GT combustor to be used as a diluent — this controls the combustor temperature while providing motive fluid. There is a stringent specification on the

oxygen content of this nitrogen – typically <2%. The ITM can be used to process the vitiate stream to meet this spec by itself; however, blending the cryo-nitrogen with the vitiate opens up some interesting options. In this manner, it is quite possible to eliminate the combustor-expander machine entirely; and simply use the GT's own turbine to process all the ITM vitiate. Similar concepts might apply to decarbonized power facilities.

4. Summary of Status of Task 30.0 “Ceramic Membrane Module Fabrication Facility”

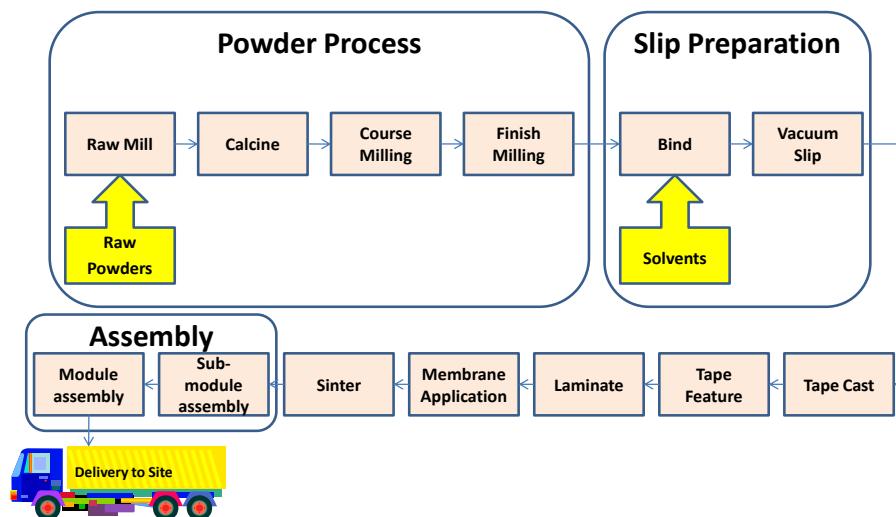
4.1. Project Scope

4.1.1 Preferred Process Scheme for the baseline Cerfab Production Process

This is an update of the process scheme outlined in Decision Point 2 Topical Report Appendix and includes expanded scope such that the Advanced Architecture design is the primary design basis for the facility. As was the case at Decision Point 2, the process is capable of both Standard and Advanced wafer architectures.

As shown in Figure 4.1, the proposed process consists of 13 sequential batch or semi-batch processing steps to transform the raw ITM powders into completed membrane modules ready for preparation for shipment. One additional step, Membrane Application, has been added for the processing of Advanced wafers .

CerFab Simplified Process Overview



1

Figure 4.1. Schematic of a ceramic-processing scheme proposed for the CerFab.

1. The first 4 steps are Powder Processing operations performed upon the raw ITM powder materials only, including the key Calcining step required to obtain the desired membrane powder structure. The process equipment used for these first 4 steps are identical for both Standard and Advanced Architecture wafers.
2. Steps 5-6 are Slip Preparation steps, during which the membrane powder is mixed with solvents, organic binders and plasticizers to form a pourable slip of the correct viscosity and composition.

3. Batches of slip are cast in step 7 to form continuous thin sheets of “green” tape which is formed by depositing slip on a plastic carrier film on a moving-belt tape casting machine, during which much of the solvents are extracted to leave a dried tape which can be handled in downstream processing steps.
4. Tape Featuring (Step 8) utilizes mechanical-press and laser-etching processes to form different layers of the ITM wafer structure, which are subsequently assembled in the following Lamination step (Step 9) into wafers.
5. Membrane Application (Step 10) is required to apply the ITM layer to the laminated wafer.
6. The final 3 steps of the process are the Thermal Processing steps carried out in high-temperature furnaces where the assembled wafers are first sintered to densify the ceramic materials, and subsequently assembled into sub-modules and then finally completed modules ready for packaging. All modules are leak-checked – a final step in this sub-process to determine quality.

4.1.2. Process Equipment Layout

Based upon the selected Tooele facility, Ceramatec have proposed a process equipment layout which is shown in Figure 4.2. This layout was based on updated quotations received for specific machinery selected for the proposed fabrication processes and now takes into account specific additional equipment needed for the production of Advanced Architecture wafers.

This proposed arrangement was arrived at by considering a number of sometimes conflicting criteria, namely:

- i. The arrangement needed to be efficient from the viewpoint of flow of materials throughout the process. All materials flow clockwise around the building from the Receiving/Material Storage area through to completely assembled modules ready for storage/preparation for shipping
- ii. The layout needs to meet all applicable safety requirements in terms of space allocation, segregation of hazardous materials, distance to escape routes etc.
- iii. The layout needs to minimize the refurbishment costs for the needs of Phase V. For example, furnaces are located closest to the incoming electrical power to minimize the expected high cost component of running electrical power to these high energy consumers.
- ii. The layout must recognize the restrictions of the existing building and the need to eliminate any demolition/re-construction of the main building structure (as a requirement for minimizing the potential of any environmental concerns).

Ceramatec received firm bids from Engineering-Procurement-Construction (EPC) contractors in which the proposed layout (Fig. 4.2) was refined.

Additionally Air Products intends to pursue option pricing to bring the kilns in closer to the other equipment in the building to improve efficiency of operations. This will make the electrical feeds longer and adjust duct work, so Air Products will analyze this potential change with the contractor to determine if moving the kilns is warranted based on overall cost.

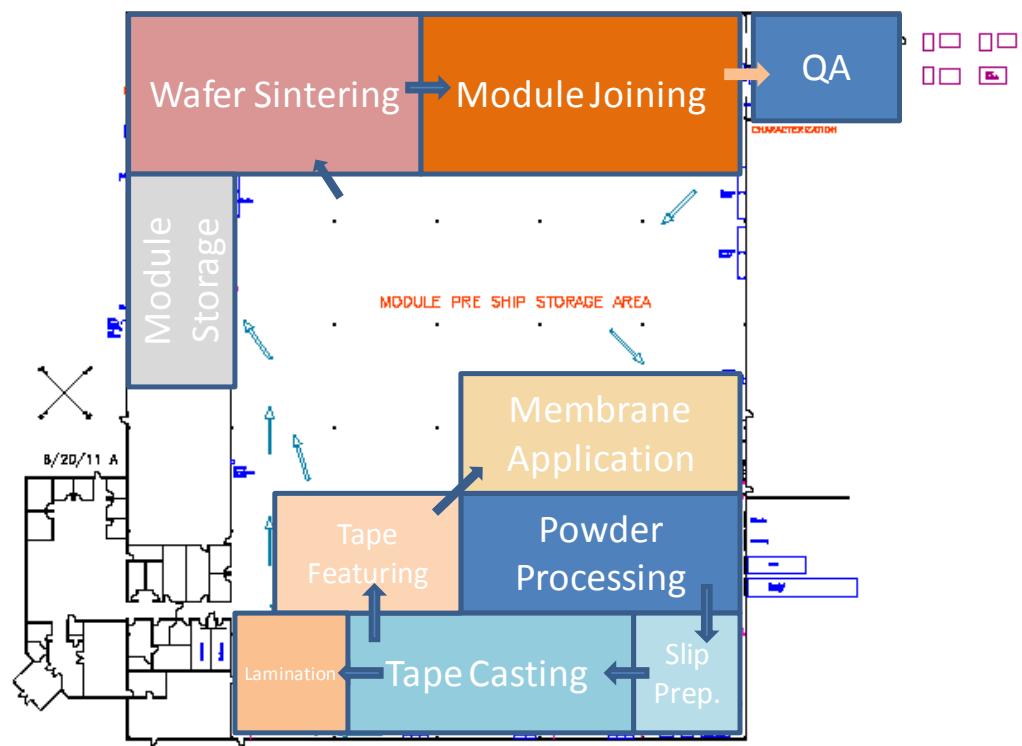


Figure 4.2. Process equipment layout for CerFab based upon the selected Tooele facility.

4.2 Project Schedule

4.2.1 Gantt chart

Since DP2, a more detailed schedule has been developed for Tasks 30.1-30.4 (see Figure 4.3 and Attachment 1). The project schedule is loaded with labor resources

Adjustments are made to the schedule based on period meetings between the Air Products and Ceramatec. A preliminary Commissioning schedule has been included; a refined schedule will be developed in the first quarter of 2012 to specify the staffing plan in more detail.

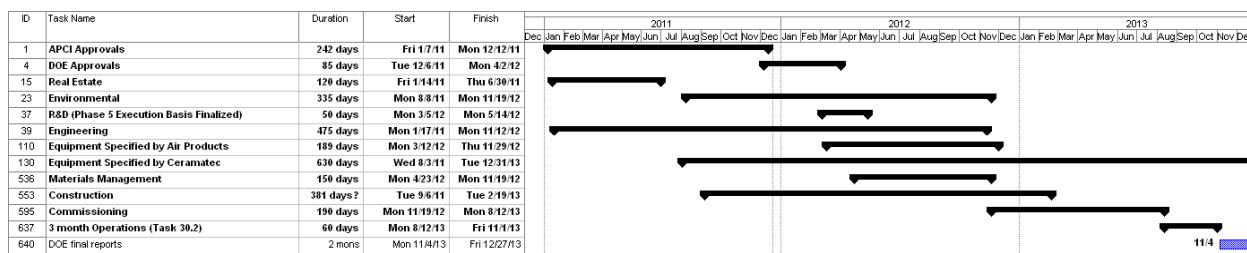


Figure 4.3. Simple Gantt chart for the CerFab.

4.2.2 Project Schedule Critical Path

Air Products has requested the DOE to authorize release of funding prior to Decision Point 3 for initial purchase order (PO) payments for the Tape Casting equipment and the Powder Processing equipment. The purchase orders for these items will be required in March 2012. The items are the critical path as they are part of the frontend processes that will be commissioned in the earlier phases of commissioning. Additionally, early release of funding has been requested to begin to fabricate the portions of the kiln furniture which are required as part of the Wafer Sintering process.

Following the long lead equipment purchases is the release of the construction contract so that the construction firm can order long lead items and complete the building re-fit prior to the equipment arriving on site later in 2012. The project schedule contains time at the back end to allow ample time for report writing and to close out the project with the DOE by the end of 2013.

4.3 Summary and Status of the environmental approvals required

4.3.1. Permitting Responsibility – Air Products will be responsible for obtaining all required environmental approvals, working cooperatively with the U.S. DOE and Ceramatec. The following approvals are either required or may be required:

- U.S. DOE National Environmental Policy Act (NEPA) review
- Air Emissions - Utah DEQ, Division of Air Pollution
- Wastewater - City of Tooele Uniform Zoning Code, Title 7, Chapters 5, 6, 7, and 8.
- Solid and Hazardous Waste – Utah DEQ, Division of Solid and Hazardous Waste
- Spill Prevention, Control, & Countermeasure Plan – Utah DEQ Division of Water Quality

4.3.2. Stakeholder Engagement – Air Products will pursue a comprehensive stakeholder engagement process to ensure concerns are heard and addressed through the approval, construction and operating phases of the project. Stakeholders include the U.S. DOE, Air Products, City of Tooele, State of Utah, owner of leased property (Conestoga).

4.3.3. General Environmental Control Strategies – Air Products will include in the process design those air and water emission controls necessary to comply with all regulatory limits and to be protective of the environment. Prior to submitting the air permit, Air Products completed a BACT analysis for emissions to determine if any additional controls were required. In addition, Air Products will implement various administrative controls (Spill Prevention, Training, Environmental Compliance Management System, Incident Investigation, etc.) to further reduce the likelihood of environmental impacts from the construction and operation of the CerFab facility.

4.3.4. Compliance Strategy – Air Products will engage the appropriate agencies and seek required permitting information by the end of Budget Period 7 so as to mitigate further impacts or requirements to the project. Air Products has submitted the air permit application to the Utah DAQ and maintains ongoing communications for status on the air permit. Air Products has obtained a Hazardous Waste ID number from the state of Utah and has received approval. Additionally Air Products has a national contract with a waste disposal service, who approved the profiles of both the flammable liquids and organic debris.

Table 4.3.1 Environmental Permits/Approvals (updated for DP3)

Permit/Approval [Agency]	Reason	Estimated Time to Obtain Permits & Status	When Required
National Environmental Policy Act, Finding of No Significant Impact, Categorical Exclusion B3.6	NEPA Section 102	Environmental Questionnaire completed. Categorical Exclusion approval timing based on air permit issuance. Submitted: May 2011 Re-submit after review: June 2011 Issue: Feb 2012	If CX is not given, we anticipate that an Environmental Assessment would take approximately 7 months.
Approval Order Utah Department of Environmental Quality, Division of Air Pollution	UDEQ Rule 307-101.	4-6 weeks to prepare the application and approximately 90 days for issuance. Submitted: 21 Oct 2011 Draft Air Permit release for public comment: mid. Jan 2012 Issue: late Feb 2012	Approval Order prerequisite for the CX/NEPA approval. Approval Order required prior to the installation of process equipment.
Wastewater Discharge Permit, City of Tooele	Uniform Zoning Code, Title 7, Chapters 5, 6, 7, & 8.	2 – 4 weeks to prepare and up to 60 days for approval Submit: Sep 2012 Issue: Nov 2012	Prior to process discharge.
Small Quantity Solid Waste Generator Registration UDEQ/USEPA	40 CFR Part 261	Registration submitted in November 2011 and Identification Number assigned. Issued: Nov 2011	Completed.
Spill Prevention, Control, and Countermeasure Plan - UDEQ	40 CFR 112	4-6 weeks Issue: Nov 2012	At start up

4.4 Commissioning plan leading up to DP4

Air Products will begin commissioning activity in February 2013. Equipment will be commissioned on an individual basis during Q2/Q3 FY13, culminating in process commissioning and Decision Point 4. Staffing will consist of technical and non-technical office personnel and shop personnel. Staffing will be added gradually as equipment is added and commissioning tasks proceed, culminating in complete staffing at commencement of Task 30.2.

A budget for both the commissioning period and operating period (Task 30.2) has been estimated according to this plan.

4.5 Project Budget

4.5.1. Equipment Cost Basis

Air Products completed what is termed a detailed cost estimate for the CerFab equipment and the overall project. The equipment contained in the Process Layout (see section 4.1.2) was costed based on equipment vendor quotations and/or engineering estimates. Costs and quotations were collected and tabulated.

Firm quotations were obtained for large and/or specialty equipment items (60% of the equipment total), while budgetary quotes were obtained for off-the-shelf items (32%). Firm quotes were obtained from three contractors to establish the cost of building modifications. Engineering costs were estimated by Air Products and Ceramatec. Approximately 8% of the equipment scope was estimated based on engineering judgment. The majority of these items fall into the category of tooling and are smaller value items.

Air Products also estimated a necessary contract engineering portion of the work to be performed by the local power company. The new equipment being added requires additional power feed. A preliminary review of the requirements has been conducted and, should any Right-of-Ways be required to bring in the additional power, the power company will conduct a paid engineering study which will take up to 90 days to complete at which time an estimate will be provided. Air Products based its estimate on the cost that the original building owner had paid in 2006 for similar work. This estimate plus five years escalation is what has been included in Air Products engineering cost estimate.

4.5.2. CerFab project costs

Overall project cost for the CerFab is estimated to be approximately \$115,000 lower than had been estimated at the time that the definitized contract was put in place. Significant reductions in cost were realized because of the selection of the Tooele building, which already had a substantial amount of installed infrastructure. Accordingly, building remodeling and facilities equipment requirements dropped. In addition, the emphasis in the facility design on advanced architecture component fabrication reduced the capacity or need for some process equipment.

Other areas increased in cost, most notably in the tape-casting, wafer sintering, and engineering, items. Tape-casting costs increased as the tape casting specification was further developed during 2011. Sintering costs increased as a result of the use of advanced architecture technology which requires more sophisticated and thorough thermal processing . Engineering costs increased substantially, mainly as a result of the emphasis on advanced architecture technology, which necessitated a re-work of the facility to enable full production with that ceramic architecture and the requirement to add additional specialty equipment items to the overall process as determined by the work under Task 28.2. Additional engineering staff was added relative to the original budget to manage the commissioning portion of the work.

4.5.3 Discussion of equipment that requires additional work for specification

As described in Section 2.5 above, some of the processes specific to advanced architecture wafer fabrication are still in development. Ceramatec has developed process concepts that Air Products has adopted as the design basis for the CerFab facility; additional work under Task 28 will be carried out during FY12 to enable a full equipment specification to be developed. At the time of this writing, one main piece of equipment requires detailed specification.

As part of Task 28, advanced architecture wafer fabrication is being carried out manually. When that work is complete, this process will be defined and implemented as an automated process under Task 30. Air Products and Ceramatec have built up the estimate for the required equipment using a quote for a similar type of equipment and estimating the remaining scope that would be required .

4.5.4 Commissioning and operating costs

Air Products has estimated the commissioning and operating costs based on the commissioning plan described in Section 4.4 above and the requirement for a one calendar-quarter year operating campaign under Task 30.2 to meet the Phase 5 objectives. Costs were developed based on the power and labor requirements of the process laid out above. In addition, Air Products has identified engineering activities associated with commissioning the custom equipment in CerFab that were not known when the Phase 5 contract was definitized. These costs are included in Table 4.5.1 in a simple breakdown of costs for commissioning and operation. These costs are evaluated against Phase 5 program spending in Sec. 5.1 below.

Table 4.5.1. Estimate of Commissioning and Operating Costs (without G&A)

Period	Cost, \$000's
Commissioning	\$ 3,915
Operation	\$ 2,517
Grand Total	\$6,432

4.5.5 Discussion of budget trade-offs within the CerFab project

Air Products and Ceramatec have collaborated to produce the process layout and project cost stack discussed above. The CerFab process has been optimized to limit overall costs while meeting the Phase 5 objectives and while balancing the technical risk in the project. The process has been re-optimized to emphasize advanced architecture wafer production, but retains its ability to produce standard architecture modules.

The capacity of the CerFab facility has been arrived at based on a normal material supply schedule for the air separation industry. Some variance in schedules exists under typical conditions. Thus, there is some ability to vary the final capacity of the CerFab facility while still achieving an objective of the Phase 5 program, which is to construct a facility capable of supplying membrane modules to a 2000 TPD ITM Oxygen plant. Therefore, should unforeseen events occur that require greater expenditure than is currently projected, the capacity of the CerFab could be reduced to maintain budget.

In addition, additional process equipment has been added to reduce risk in certain parts of the process. For example, the process has been designed with sufficient thermal processing equipment to address technical risk. One cost-saving step for the CerFab would be to accept the risk that the through-put target will not be made and eliminate the added thermal processing equipment.

5.0 Discussion of overall project management

5.1 Budget Trade-offs within the Phase 5 project

As part of good Project Management practice, Air Products is applying its standard Technical Risk Management Process (TRMP) to the CerFab project. The process is used to identify technical and project risks, to weigh their ramifications and probability of occurrence, and to assign each risk a monetary valuation which reflects the weighted expectation of additional expenditure associated with each risk. A risk assessment was carried out at the onset of the project, and has since been updated with the new information based on added emphasis on advanced architecture wafer technology and with feedback from equipment suppliers.

The Risk Assessment indicates that there are approximately \$2.9 million in estimated costs associated with equipment, process, and operations risk to Phase 5 objectives. The majority

of the risk is associated with the uncertainty in the advanced architecture equipment and operations, which is the subject of Task 28. Therefore, the risk can be assessed at various intervals based on the progress under Task 28. However, it seems prudent to plan for the possibility now that these expenditures would be necessary to complete the CerFab to meet the Phase 5 objectives. In that case, Air Products recommends that the Phase 5 program be conducted so as to make available to the CerFab project up to \$2.9 million in additional funds solely to address costs associated with the aforementioned risks.

In addition, as described in Section 4.5.5 above, \$6.4 million in operating costs are expected to be required to complete the commissioning and operating tasks, which exceeds the cost estimated for this activity at the time of contract definitization by \$1.6 million. This change is mainly attributed to under-estimation during definitization in the required expenditure during the operating portion of the work (Task 30.2). Therefore, Air Products further recommends that an additional \$1.6 million be spent under Task 30.2 for operating the CerFab to produce modules.

The necessary funds will be sourced from the other Phase 5 tasks. Air Products recommends that \$1.6 million for Task 30.2 be shifted from Task 29, “Engineering Development for Industrial and CCS Applications.” An appropriate reduction in level-of-effort to Task 29 and its sub-tasks will be made in consultation with the Federal Project Manager (FPM) while ensuring that Task 29 activities meet the Phase 5 program objectives. In addition, Air Products proposes that approximately \$1.9 million in Task 28 (Ceramic Materials and Manufacturing Development for the Industrial and CCS Applications”) spending be delayed until FY13 pending the outcome of on-going work under that Task to assess progress against the critical CerFab risks. Should specific issues identified as Phase 5 risks arise during commissioning/operations, these funds will be made available for addressing those risks. Finally, Air Products recommends that Air Products develop an alternative spending plan for Task 31 and be prepared to direct \$1 million of Task 31 funds to Task 30 to address Phase 5 risks as and if they arise during commissioning/operations of the CerFab. All of these decisions would be made in conjunction with the FPM prior to re-assigning program funds.

6.0 Project Status

Based on work completed through 30 Sep 2011, the Phase 5 task work is approximately 3% complete based on a cost-weighting of subtasks.

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ACRONYMS AND ABBREVIATIONS

APCI = Air Products and Chemicals, Inc.

ARRA = American Recovery and Reinvestment Act

CerFab = ceramic module fabrication facility

DEQ = Department of Environmental Quality

DP1= Decision Point 1

EPA=environmental protection agency

EPC= engineering/procurement/construction

GHR = gas heated reformer

HVAC = heating, ventilating and air conditioning

HAP = hazardous air pollutant

ICCS = industrial carbon capture and storage

ISTU = intermediate scale test unit

ITM = ion transport membrane

LAN=local area network

MeOH = methanol

NEPA = National Environmental Policy Act

NG = Natural Gas

PDF = process development facility

PA=public address or Pennsylvania

VOC = volatile organic compound

TPD = tons per day

SOPO = Statement of Project Objectives