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Hybrid Vehicle Turbine Engine

HVTE-TS

Technology Support Project

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1995-1996 Annual Report

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FOREWORD

This report presents a summary of technical work accomplished on the Hybrid Vehicle Turbine Engine—Technology Support (HVTE-TS) Project during calendar years 1995 and 1996. Work was performed under an initial National Aeronautics and Space Administration (NASA) contract DEN3-336. As of September 1996 the contract administration was transferred to the U.S. Department of Energy (DoE) Chicago Operations Office, and renumbered as DE-AC02-96EE50453. **The report is arranged per the original work breakdown structure (WBS). Only WBS elements with activity in 1995-1996 are reported herein.**

This technology project is funded by the U.S. Department of Energy. Project management and technical direction in these reporting periods were provided by the NASA Lewis Research Center (LeRC).

The purpose of the HVTE-TS program is to develop gas turbine engine technology in support of DoE and automotive industry programs exploring the use of gas turbine generator sets in hybrid-electric automotive propulsion systems. The program focus is directed to the development of four key technologies to be applied to advanced turbogenerators for hybrid vehicles:

- Structural ceramic materials and processes
- Low emissions combustion systems
- Regenerators and seals systems
- Insulation systems and processes

The HVTE-TS program builds on the significant technology base established by the previous DoE/NASA Advanced Turbine Technology Applications Project (ATTAP).

Automotive gas turbine attractions include the following potential advantages:

- significantly increased fuel economy
- ability to meet federal emission standards with untreated exhaust
- ability to operate on a wide range of alternate fuels
- inherently smooth, low vibration operation

Allison Engine Company addressed the HVTE-TS Program with a team drawing upon:

- Allison's extensive ceramic design, analysis, and materials data base and expertise. Previous automotive turbine development background including:
 - substantial experience, design, and test capabilities
 - automotive gas turbine technology and hardware
 - test vehicle resources
- the infrastructure of expertise and resources in place in the American ceramics industry
- the working relationships between the ceramic industry and Allison
- the unique capabilities and resources at universities and national laboratories, such as:
 - High Temperature Materials Laboratory (HTML) at Oak Ridge National Laboratory (ORNL)
 - Argonne National Laboratory

In this arrangement, Allison serves as prime contractor. Major ceramics industry development subcontractors during 1995-1996 included Schuller International; Corning, Inc.; AlliedSignal Ceramic Components (ASCC); and Kyocera Industrial Ceramics Corporation. Norton Industrial Ceramics Corporation also provided ceramic component support with processing, fabrication, and characterization efforts leading to the delivery of silicon nitride rotors and scrolls to Allison.

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SUMMARY

HVTE-TS activities during 1995 and 1996 included: test-bed engine design and development; ceramic component design; materials and component characterization; ceramic component development and fabrication; ceramic component rig testing; and test-bed engine fabrication and testing.

In 1994, the original ATTAP technology focus was redirected and broadened to include key technologies needed for advanced gas turbine electric generator sets such as those under development in the Hybrid Vehicle Propulsion System Program (Hybrid). The application of the technology was redirected from a prime power role to a hybrid propulsion system role, where the turbine power plant drives a high speed alternator for electrical power generation.

Due to this redirection the program was renamed Hybrid Vehicle Turbine Engine—Technology Support (HVTE-TS). This new direction for HVTE-TS was maintained in 1995 and 1996.

Test-bed engine design and development continued with emphasis on the application and cyclic durability testing of ceramic hot flow path components.

Component design activities included a continuing effort to refine and improve ceramic regenerator design and analysis methods.

Allison materials characterization efforts have yielded additional information on:

- AlliedSignal Ceramic Components slip cast and gel cast AS800 Si_3N_4
- AC Cerama CSN101 isopressed Si_3N_4
- Kyocera Industrial Ceramics SN252 slip cast Si_3N_4
- Kyocera Industrial Ceramics SN253 slip cast Si_3N_4

Ceramic component process development and fabrication continued with several ceramic component subcontractors, including Schuller International, for insulation systems and processes; Corning, Incorporated, for extruded regenerator materials and processes; and several suppliers of structural ceramics components, such

as AlliedSignal Ceramic Components and Kyocera Industrial Ceramics.

Component rig activities were focused on completing the construction of test cell facilities, and initiating testing. Installation was completed for the hot gasifier rig, the regenerator disk performance rig, the regenerator transient durability rig, and the regenerator cold flow seal leakage rig.

Test-bed engine fabrication, testing, and development activities continued to verify improvements in ceramic component technology permitting the achievement of both program performance and durability goals.

Significant testing milestones were achieved in this time frame in two specific areas:

- Completion of a 300-hr cyclic durability test on an AGT-5 hot gasifier rig with a ceramic hot flow path, while operating up to 2500°F and 100% rated speed
- Completion of a 500-hr durability test of two one-piece extruded magnesium-aluminosilicate (MAS) regenerator disks operating at up to 100% rated flow and temperature

Engine and hot rig test hours were accumulated in 1995-1996 as summarized in Table I.

Table I.
Total test hours.

	Pre-1995	1995-96	Cumulative
Engine hours	2108	55	2163
Hot rig hours	2385	541	2926
Total hours	4493	596	5089

INTRODUCTION

This is the seventh of a series of annual reports documenting work performed on the ATTAP/HVTE-TS (Report number DoE 0336-7 was entitled "Ceramic Design Manual [WBS 6.4.5]"). This is a combined report to cover work performed in both 1995 and 1996. Work in 1995 and 1996 was conducted by a team directed by Allison Engine Company, with significant support from several domestic suppliers who are under development subcontracts. The U. S. Department of Energy (DoE) sponsored the work, which was initially managed and technically directed by NASA-Lewis Research Center under contract DEN3-336. The contract was modified in 1996 to implement management by the DoE Chicago Operations Office as new contract number DE-AC02-96EE50453. Technical direction was assumed by the Office of Transportation Technologies in Washington, D.C.

GOALS AND OBJECTIVES

Initially this effort was intended to advance the technological readiness of an automotive ceramic gas turbine engine. It aimed to develop and demonstrate structural ceramics having the potential for competitive automotive engine life cycle cost and for operating for 3500 hr (automotive engine life) in a turbine engine environment at temperatures up to 1371°C (2500°F). Project objectives were the following:

- Enhance the development of analytical tools for ceramic component design using the evolving ceramic properties data base

- Establish improved processes for fabricating advanced ceramic components
- Develop improved procedures for testing ceramic components
- Evaluate ceramic component reliability and durability in an engine environment

In the continuing program in 1995 and 1996, the objectives of the HVTE-TS program were the same as those of the original ATTAP, with additional focus as follows:

- Develop four key technologies to a level so a commercialization decision can be made on:
 - Structural ceramic materials and processes
 - Low emissions combustion systems
 - Regenerators and seal systems
 - Insulation systems and processes
- Make this technology applicable to the automotive gas turbine engines that form the basis of hybrid automotive propulsion systems consisting of combined batteries, electric drives, and on-board power generators

The relationship of the HVTE-TS project to the Hybrid Electric Vehicle (HEV) project is graphically depicted in Figure 1. Allison was a subcontractor to General Motors Corporation (GM) during the HEV project. Allison was contracted to design, develop, fabricate, and deliver gas turbine engine driven auxiliary power units (APUs)

Figure 1. HVTE-TS relationship to Hybrid turbine auxiliary power unit (APU) development.

which were utilized in GM's Hybrid Propulsion System Development Program (Hybrid). Both the HEV and HVTE-TS projects draw on the significant technology base established by ATTAP. As the key technologies are developed, the results of the technologies in materials, components, and manufacturing processes would be incorporated into the hybrid projects at various milestone stages of the program. Ultimately, the Hybrid program, with support from HVTE-TS, would lead to a solid basis for commercialization decision within a reasonable time frame.

continuous activities in ATTAP, and promising candidates were integrated into the program as merited. Similarly, those technologies and/or ceramic component suppliers that did not productively evolve to address program goals were deleted from the ATTAP effort. A similar approach was being continued in 1995-1996 with the HVTE-TS program, as shown in Figure 3.

This technology development effort continued as laid out in Figure 3, with milestones geared to those of the DoE's Hybrid Engine Program.

PROGRAM SCHEDULE AND CONTENT

Figure 2 shows the scheduled activities in the original ATTAP. Materials assessment occurred at the initiation of ATTAP and resulted in the targeting of ceramic component technology goals and the identification of materials, processes, and manufacturers to address those goals. The materials assessment was updated in Year 3 and again in Year 5, at which time the state of the art was reassessed for each component and required technology improvements were defined. The identification and evaluation of materials, processes, and manufacturers were ongoing,

Figure 2. ATTAP schedule.

Figure 3. HVTE-TS schedule (time phased to Hybrid program objectives).

A reference powertrain design (RPD) was established during the early phases of ATTAP and included preliminary design of a powertrain system to meet performance, cost, and reliability design goals. The RPD, as revised through the ATTAP, reflects current ceramic technology and goals, and is being carried through the HVTE-TS program. The initial Allison Hybrid engines were intended to operate at somewhat lower temperatures than the original RPD goals; however, for this technology program, it was decided to continue development with the original 1371°C (2500°F) turbine inlet temperature (TIT) goals. This will provide growth margins for future Hybrid configurations.

Test-bed engine development, shown as an intermittent activity during ATTAP, included efforts aimed at ensuring the availability and functionality of the AGT-5 gas turbine engine as the test-bed for the high temperature ceramic components. Engine development was not a primary focus of ATTAP, but those activities recognized the need to continue the evolution of the engine in order to handle additional power and thermal loads. The AGT-5 engine also served as a test-bed for design changes resulting from the integration of high temperature flow paths. The engine will continue to be used in this role in the HVTE-TS program.

Central to the logic of Allison's approach to ATTAP/HVTE-TS is the iterative component development cycle. The cycle, as illustrated in Figure 4, includes the design/fabrication/characterization/rig test/engine test sequence of activities. The development cycle reflects the anticipated improvements in ceramic materials

and associated component processing technologies and the incorporation of laboratory characterization data and rig/engine test results into succeeding designs. The initial design activity featured the then-current monolithic ceramic technology in the design of the gasifier turbine stage of the AGT-5 engine for 1371°C (2500°F) TIT plus other required hot flow-path pieces. The second design phase incorporated toughened monolithic materials, used in the same gasifier stage components. The third phase incorporated advanced (e.g., from ORNL's Ceramic Technology for Advanced Heat Engines [CTAHE] project) materials and processes as they became available. Succeeding design phases include other necessary ceramic components in the high-temperature test-bed engine, notably power turbine flow-path pieces. Additionally, as new material systems are developed at major ceramic suppliers, and manufacturing techniques and processes are developed, components from these programs will be incorporated into the design cycle as part of the HVTE-TS program, ultimately yielding component capability and process validation that can be transferred to application in hybrid engine components.

Component fabrication includes those process development activities by ceramic suppliers resulting in the fabrication of engine-usable components. Characterization involves the laboratory activities, at both Allison and outside suppliers, that measure and define the various properties and qualities of ceramic materials in either test bar form or in actual components. Examples are microstructural evaluation and measurements of density, strength, oxidation

Figure 4. Ceramic component development cycle.

resistance, toughness, etc. Included are the development and application of nondestructive evaluation (NDE) techniques.

Component rig activities include the development of rigs for component verification and testing (e.g., hot gasifier turbine rigs) as well as the actual testing activities. Test-bed engine testing includes the test activities associated with test-bed engine development plus the verification and development testing of the ceramic components. Each component development cycle (Figure 4) begins with design, followed by component fabrication, characterization, rig testing, and finally engine testing. The rigorous development process is iterative between the users and the ceramic supplier community, and ensures developing an understanding of the behavior of components in service and in continuous identification of areas for improvement.

TEST-BED ENGINE

Figure 5 shows the automotive gas turbine engine being used as the ceramic component development test-bed for ATTAP/HVTE-TS. This GM-developed engine, the AGT-5, is a two-shaft, regenerative configuration with axial-flow gasifier and power turbines. The engine produces approximately 110 horsepower (hp) at its original full-power TIT of 1038°C (1900°F).

The emissions and alternate fuels goals are considered achievable based on demonstrated GM experience. For example, the AGT 100 engine's (from the AGT project) combustion system has displayed laboratory steady-state emissions of oxides of nitrogen (NO_x), carbon monoxide (CO), and unburned hydrocarbons (UHC) well within the Tier 0 Federal Emissions Standards using diesel fuel, jet fuel, and methanol. Although such systems have demonstrated the potential for low

emission/alternate fuel gas turbine combustion, much work remains before achieving a fully functional system suitable for automotive applications. Such efforts were originally outside the scope of ATTAP, but the development of components, processes, and technologies in the HVTE-TS program will be incorporated into engines expected to be applied to the Hybrid program.

KEY TECHNOLOGIES

During ATTAP, emphasis was placed on ceramic component technology as development. The critical development components were selected because their functional success was critical to the viability of the ceramic automotive turbine engine, and each required further technological development to be reliable, durable, and cost effective in the automotive engine. The components included:

- Gasifier turbine rotor
- Gasifier turbine vanes
- Gasifier turbine scroll
- Regenerator disks
- Thermal insulation

With the new thrust of the HVTE-TS project to support of the Hybrid Propulsion System, the project is now structured to develop critical technologies to support a production feasibility demonstration in four years. The technology requirements of an advanced turbine driven on-board generator unit depend on the four key technologies as shown in Figure 6:

- Structural ceramic materials and processes
- Low emissions combustion systems
- Regenerators and seal systems
- Insulation systems and processes

Development of these four key technologies is based on the broad range of experience gained during the AGT project and ATTAP, and will be continued through the HVTE-TS program.

Activities in 1995 and 1996 were focused primarily on the durability demonstration of hot ceramic flow path components and extruded regenerator cores. At the conclusion of 1996 both of these goals were achieved with the successful completion of a 300-hr cyclic engine test at up to 2500°F peak operating temperatures and with the completion of a 500-hr durability test on a pair of extruded regenerator cores.

TE89-4564

Figure 5. ATTAP/HVTE-TS test-bed engine—
AGT/demonstration targets.

Figure 6. HVTE-TS Project key technologies.

VS94-2126
TE95-1646

**KEY TECHNOLOGIES
DEVELOPMENT:**

- STRUCTURAL CERAMICS
- LOW EMISSIONS COMBUSTION
- REGENERATOR SYSTEM
- INSULATION

1.0 ENGINE/POWERTRAIN DESIGN AND DEVELOPMENT, ANALYSIS, AND MATERIALS ASSESSMENT

1.4 TEST-BED ENGINE DESIGN AND DEVELOPMENT

1.4.2 Combustion Systems

The primary objective of this task was to meet California ultra-low emissions vehicle (ULEV) automotive emissions standards of 0.2 gm/mi NO_x, 1.7 gm/mi CO, and 0.04 gm/mi HC on diesel fuel without the benefit of exhaust gas after treatment. This was to be accomplished by design, fabrication, and test of a lean premixing/prevaporizing combustion system. However, due to funding limitations in the HVTE-TS program, the Low Emissions Combustion effort was terminated in early 1995.

In 1994, technical efforts focused on analyzing a lean premixing variable geometry poppet valve combustor design. Initial testing of this combustor (see 1993-1994 HVTE-TS Annual Report) indicated that ULEV emissions standards were achievable with this concept, but metal liner wall temperatures and ceramic valve seat temperatures were too high.

Prior to termination of effort in 1995, detail drawings for a poppet valve combustor redesign, which improved liner backside cooling, were completed. In addition, a four year combustion program was planned for calendar years 1995 through 1998. Summarized briefly, this plan contained the following elements:

Combustor Concepts for Rig and AGT-5 Engine Testing

- Poppet valve combustor
 - Variable geometry in premix zone only
 - Metal/ceramic components
 - Showerhead fuel injection
- Sliding pad combustor
 - Variable geometry in premix and dilution zones
 - Metal components
 - Showerhead fuel injection

- Indexing head combustor
 - Variable geometry in premix and dilution zones
 - Ceramic components
 - Single point fuel injection
- Catalytic combustor
 - Reduction of cold start hydrocarbon emissions
- Select best concept for further development

Premixing Zone Diagnostics

- Experimental subcontract to university
- Laser diagnostics to relate premix zone design parameters to mixing and evaporation

Fuel Nozzle Development

- Pure airblast
 - Uses combustion air to atomize fuel
 - Eliminates need for atomizing air pump
- Electrostatic
 - Transfers static charge to fuel to atomize
 - Eliminates need for atomizing air pump

Ceramic igniter

- Improved durability

2.0 CERAMIC COMPONENT DESIGN

2.1.4 Ceramic Rotary Regenerator System Design

The overall goal of the ceramic regenerator design was to analyze, design, and develop a rotary regenerator disk system for automotive applications.

2.1.4.1 Regenerator Performance

2.1.4.1.1 Objective/Approach

The objective of this task was to refine current analytical capabilities. The refinements included design studies to account for nonideal conditions encountered in the use of rotary regenerators. The approach was to build on the previous work and refine the performance analysis of the disk.

2.1.4.1.2 Accomplishments/Results

- Improved Methods for Predicting Regenerator Performance
- Accounted for Flow Distribution Results and Effects on Performance
- Accounted for Regenerator Seal Leakage Effects on Disk Performance
- Accounted for Through Wall Leakage of Disks on Disk Performance
- Explored the Thermal Capacity of a Disk

2.1.4.1.3 Discussion

Improved Regenerator Performance Predictions

Allison has developed computer codes for the analytical prediction of the thermal and pressure drop performance of a rotary regenerator. The rotary regenerator analysis requires significant iterations to achieve steady-state predictions due to the regenerator core temperatures varying with rotation. One of the codes is based on conventional heat exchanger theory. This program assumes uniform gas flow and temperature. It is used for all published performance predictions and parameter trade-off studies for engine cycle analyses.

The second program is a conduction/convection heat transfer model used to predict regenerator performance under nonuniform conditions (flow,

temperature, pressure, seal geometry, etc) and to predict regenerator core temperature.

The Allison computer codes were used extensively to support the regenerator system design and analysis effort for the hybrid electric vehicle program activities under the HVTE-TS project.

Regenerator Seal Leakage Effects on Disk Performance

Regenerator seal leakage is a penalty to engine cycle performance. High pressure air that leaks in and around the regenerator disk has two significant effects:

1. It changes the flow through both sides of the disk (increases the gas flow and decreases the airflow).
2. It injects additional air at different temperatures into other areas.

Figure 2.1.4-1 illustrates the various leakage paths around a disk. These leakage paths consist of leakage through the L seals, leakage between the primary seal faces and the disk, and leakage through the disk itself due to porosity. Figure 2.1.4-2 illustrates the reduction in effectiveness as a function of leakage. The predicted results agree with the observation that as regenerator leakage increases, the exhaust gas temperature increases. As gas effectiveness decreases with leakage, less heat is taken out of the exhaust and therefore the exhaust temperature increases. If the regenerator has a measurable leakage, the measured air and gas side effectiveness will be different. Even a

Figure 2.1.4-1. Various leakage paths in and around the disk and seals.

Figure 2.1.4-2. Leakage effects on effectiveness.

leakage at the maximum design goal of only 5% will cause an approximately 3% difference in effectiveness.

Through Wall Leakage of Disks

Regenerator seal leakage is the main source of leakage for the regenerator system, but the disk also contributes to the leakage. The ceramic materials used for regenerators are slightly porous and allow airflow through the walls when exposed to a differential pressure. The leakage through the walls of a disk are reported by Corning as part of the quality control testing on each disk. Corning measures the through wall leakage using a test fixture that exposes an area to a differential pressure of 20 psig. The leakage is through multiple walls for a distance set by the width of the test fixture seal. This test is illustrated in Figure 2.1.4-3. Corning measures the airflow (lb/sec) through the test area at several locations on a disk and then determines a mean leakage. This mean leakage is then divided by the leakage area (leakage perimeter $\times$ disk width) to arrive at a reported leakage per unit area (lb/sec/in.²).

Figure 2.1.4-4 illustrates the percent engine leakage over a range of disk leakages for a 10 kW and 40 kW Hybrid cycle point. The abscissa of these curves is the standard seal width (1.5 in.)

Figure 2.1.4-3. Test fixture to measure disk internal leakage.

leakage value so that a comparison can be made for current disk leakages and previously reported leakage values. The three highlighted disk leakage values are for extruded lithium-aluminosilicate (LAS), extruded magnesium-aluminosilicate (MAS), and a wrapped disk procurement specification limit. The extruded parts are well below the wrapped specification limit and may allow the limit to be reduced to approximately 3.0×10^{-5} lb/sec/in. If this limit can be maintained, the contribution to the total regenerator leakage from the disk would be less than 0.4%. With a goal of 3.5% leakage from the entire regenerator system, the disk would contribute approximately 11% of the total leakage.

Thermal Capacity of Disk Analysis

Optimization studies have been completed on the regenerator design to maximize performance. The study involved increasing the quantity of heat transferred by a disk to determine the maximum heat transfer capacity. To complete the study, two different disk sizes were analyzed: a 9.5-in. diameter disk and a 14.0-in. diameter disk. An engine cycle point originally designed for a 9.5-in. diameter disk was used for the analysis. To increase the amount of heat transfer relative to this design point, either the temperature difference or the flows had to be changed. The temperature difference could not be increased significantly because of material limitations so the air and gas flow were increased to increase the heat transfer. As the flows were increased to many times the design point flow, the heat transferred eventually peaked for each of the two disk sizes. Figure 2.1.4-5 illustrates both the heat transferred and the pressure drop incurred in each disk. The thermal capacity of the 9.5-in. diameter disk was 284%, which means that at the design point only 35% of the disk's thermal capacity was being used. In comparison, the 14-in. diameter disk has a 614%

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Figure 2.1.4-4. Estimated disk leakage at engine operating conditions.

capacity, which results in only 17% of its capacity being used at the design point. Figure 2.1.4-6 illustrates the same data but the percent thermal capacity used is plotted against the pressure drop incurred. This demonstrates that the curves for different regenerator sizes follow a very similar curve and that they all have a peak capacity when a pressure drop of approximately 34% is incurred. This curve demonstrates that increasing the pressure drop from 3.9%, DP/P for a 14-in. disk, to 7%, DP/P for a 9.5-in. disk, the thermal capacity doubles. With a doubled thermal capacity, the disk is approximately half the volume, which agrees with the volumes of the 9.5 and 14 in. diameter disks.

The drawback with achieving the maximum thermal capacity is that the pressure drop is near

34%, which is about 5 times greater than the design point pressure drop of 7%. Current engine cycle tailoring dictates that regenerator pressure drops remain near 5%, but these curves demonstrate that significantly smaller regenerators could be used if the pressure drop is allowed to increase near the 25 to 35% range. A constant pressure drop in this range is probably not acceptable, especially when an engine is attempting to produce maximum power. This pressure drop may be acceptable at less than peak power conditions when the engine would produce less power but operate more efficiently. This could be achieved through the use of a bypass valve to get maximum power when needed, but achieve maximum efficiency at part power with relatively small regenerator disks.

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Figure 2.1.4-5. Study of the maximum heat transfer available from a disk.

The results of this study are the following: (1) When a disk is sized for an engine cycle using current guidelines, the heat transferred cannot be increased significantly, even if the disk volume is increased significantly. The engine cycle conditions, temperatures, and airflows dictate the amount of maximum heat transfer. Disk sizing guidelines would size a disk to near 90% effectiveness, so little additional heat transfer can be achieved even if the effectiveness is increased to 92 or 95% with a bigger disk. (2) To minimize the size of a disk, the pressure drop should be as

high as possible. The slope of the curves in Figure 2.1.4-6 demonstrates that even small increases in pressure drop can increase the percent capacity, which means smaller disks. For example, for the engine cycle point used in this study, a disk would only have to be about 5.7 in. in diameter to exchange the same heat as the 9.5 or 14 in. diameter disks. The pressure drop for this 5.7-in. diameter disk would be approximately 34%, which is very high but may be tolerable at certain times as described previously.

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Figure 2.1.4-6. Percent of thermal capacity used versus pressure drop of disk.

2.1.4.2 Regenerator Mechanical Design

2.1.4.2.1 Objective/Approach

The objective of this task was to improve the stress analysis capability for the regenerative disk.

2.1.4.2.2 Accomplishments/Results

- Analyzed Material Properties Affecting Stress Analyses
- Described Useful Applications of Stress Analysis
- Successfully Simplified the Hub/Bearing Design
- Formulated a Thermal Stress Resistance Indicator for Material Screening

2.1.4.2.3 Discussion

Stress Analysis

An important design aspect of the regenerator disk is the stress induced due to mechanical and thermal loads. The current disk designs are under mechanical loads due to pressure differences and the drive torque, but the significant loads are thermal stresses. The temperature difference along the axis of the disk can be as high as 1400°F across a length of 2 or 3 in. These severe temperature gradients require the use of either low expansion materials to reduce the stresses or high strength materials to accommodate the stresses. The proper balance of material properties (expansion, strength, modulus, etc.) must be achieved. A range of materials have been considered, but the most successful have been the very low expansion ceramics. The three key parameters inducing the thermal stress are the thermal expansion, elastic modulus, and Poisson's ratio. The strength, or modulus of rupture (MOR), is the key parameter resisting the stresses. Using these properties, an indicator was formulated to roughly assess the stress/strength suitability of a material for a rotary disk regenerator. The equation for the indicator results from the basic stress analysis equations and is defined as:

$$I = \frac{MOR}{E \alpha} (1 - \nu)$$

This indicator is similar to the thermal shock parameter developed in other published reports. Table 2.1.4-I provides a brief list of materials that have been considered for regenerator disks and their indicator value. The table rates each of the materials against LAS because this is the primary material for automotive applications. LAS has a proven ability to resist thermal stresses. Aluminum silicate (AS) has a higher indicator than LAS and has also demonstrated very good thermal stress resistance, but is significantly more expensive than LAS. MAS is less expensive than LAS but has only one third the capability of LAS to resist thermal stresses. The two stainless steels are listed for reference but are not completely comparable because they fail in a ductile manner, which is different than the brittle failures of the ceramics. The conclusion from this phase of the stress study was that the low expansion materials definitely have the advantage in the property balance and are the preferred candidates.

Material property data were provided by Corning. MAS core material data are shown in Figures 2.1.4-7 and 2.1.4-8.

The variability of the material properties with temperature typically results in poor correlation of predicted results with test data.

Table 2.1.4-I. Comparison of regenerator materials and the susceptibility to thermal stress.

Material	Modulus of rupture, kpsi	E modulus, mpsi	Coeff of thermal expansion, in./in./°C	Poisson ratio	Indicator	% of LAS
LAS	8.0	10.0	40.0e-07	0.25	1500	100
AS	4.0	4.0	3.0e-07	0.12	2933	196
MAS	5.0	17.5	5.0e-07	0.22	446	30
Silicon nitride (2.7 g/cc)	38.0	23.2	29.0e-06	0.24	429	29
Silicon nitride (2.3 g/cc)	17.0	20.0	2.7e-06	0.24	239	16
Silicon carbide (SiC/SiC)	43.5	33.4	4.5e-06	0.19	235	16
430 stainless steel	85.0	28.5	1.2e-05	0.30	175	12
410 stainless steel	75.0	29.0	1.2e-05	0.30	152	10

Figure 2.1.4-7. Coefficient of thermal expansion for Corning MAS material.

Figure 2.1.4-8. Modulus of elasticity for Corning MAS material.

3.0 MATERIALS CHARACTERIZATION AND CERAMIC COMPONENT FABRICATION

This section describes the ongoing ceramic material and component fabrication, characterization, and development activities that are a key focus of the HVTE-TS program. The ceramic materials subsection documents the results of characterization and qualification of ceramic materials and components being developed for advanced gas turbine engine applications. This includes the characterization of material properties (microstructure, strength, and fracture toughness) on both test specimens and bars sectioned from components and the results of failure analyses of rig/engine tested components. The ceramic component fabrication subsection describes the ongoing ceramic component process development activities at the ceramic suppliers, including AlliedSignal Ceramic Components, Kyocera Industrial Ceramics, Norton Advanced Ceramics, and Schuller International.

Allison's approach to ceramic component technology development continues to be one of subcontracting process development to the domestic ceramic manufacturing community and working in an iterative development loop with those suppliers in areas of component design, fabrication, characterization, and rig/engine data feedback. While basic ceramic materials development is not part of this program, the program integrates material developments from DoE/ORNL programs, Defense Advanced Research Projects Agency (DARPA) material partnership programs, supplier in-house activities, and other sources as they become available for component fabrication efforts.

3.1 MATERIALS AND COMPONENT CHARACTERIZATION

3.1.1 Material Properties and Microstructure

3.1.1.1 Objective/Approach

The materials and component characterization efforts have focused on the testing and evaluation of candidate ceramic materials and components

being developed for use in advanced gas turbine engines. The primary objective of this task is to establish a data base of appropriate material characteristics to support the design, analysis, development, and testing of hot section ceramic components. A secondary objective is to evaluate new candidate ceramic materials and suppliers and to assess which, if any, should be used in subsequent component development efforts. The materials characterization activities have focused on microstructure, density, flexural strength, and fracture toughness evaluations of various ceramic materials. Fracture surface analysis is conducted to determine the nature and location of strength-controlling flaws. In addition, tensile strength and time dependent strength characteristics are evaluated for select materials.

3.1.1.2 Accomplishments/Results

The ceramic materials and components evaluated during this reporting period include:

- AlliedSignal Ceramic Components AS800 slip cast Si_3N_4
- AlliedSignal Ceramic Components AS800 gelcast Si_3N_4
- AC Cerama CSN101 isopressed Si_3N_4
- Kyocera Industrial Ceramics SN253 slip cast Si_3N_4
- Kyocera Industrial Ceramics SN281 slip cast Si_3N_4

3.1.1.3 Discussion

AlliedSignal Ceramic Components AS800 Silicon Nitride. Characterization of AlliedSignal Ceramic Components AS800 Si_3N_4 was conducted this reporting period. Material was evaluated from two distinct forming methods: slip casting and gelcasting. The slip cast material was fabricated by pressure slip casting using plaster molds, with the specimens sectioned from slip cast billets. The gelcast specimens were sectioned from gelcast billets formed utilizing hard tooling. The AS800 silicon nitride is an in-situ toughened material with a highly acicular microstructure, as shown in Figure 3.1.1-1. The interlocked acicular Si_3N_4 grains result in tortuous crack paths and the resulting crack deflection contributes to the

Figure 3.1.1-1. Microstructure of AlliedSignal Ceramic Components AS800 Si_3N_4 .

toughness of the AS800 material. The enhanced crack growth resistance of the in-situ toughened microstructure makes the material more damage tolerant, reducing the sensitivity to processing induced flaws such as machining damage, voids, and inclusions. This results in lower variation in the strength data (higher Weibull modulus) because the dominant fracture origins are Si_3N_4 grains and not processing flaws. The average density of the slip cast and sintered AS800 measured 3.325 g/cc (0.120 lb/in.³). The fracture toughness of the AS800 Si_3N_4 material was 8.4 MPa-m^{1/2} (7.6 ksi-in.^{1/2}) determined using the single edged notched beam technique.

The mechanical property results for the slip cast AS800 Si_3N_4 are summarized in Table 3.1.1-I. The typical fracture origins observed in the machined specimens at both room and elevated temperatures were surface flaws, as shown in Figure 3.1.1-2. A majority of these flaws were associated with large tabular $\beta\text{-Si}_3\text{N}_4$ grains on the specimen surface or near surface. The bars tested with an as-processed surface condition also had failures originating from surface flaws, frequently associated with shallow surface depressions (Figure 3.1.1-3).

Table 3.1.1-I.
Strength characteristics of AlliedSignal Ceramic Components AS800 silicon nitride material fabricated by pressure slip casting.

Temperature °C (°F)	MOR strength, MPa (ksi)	
	Machined surface	As-processed surface
25 (77)	728.1 (105.60) m=28.37	669.9 (97.16) m=12.84
1000 (1832)	646.2 (93.72)	621.9 (90.20)
1150 (2102)	625.6 (90.74)	581.4 (84.33)
1250 (2282)	613.5 (88.98)	564.4 (81.86)
1370 (2500)	577.7 (83.79)	480.9 (69.75)

Figure 3.1.1-2. Typical fracture origin (surface flaw) observed in slip cast ASCC AS800 Si_3N_4 specimens with a machined surface.

Figure 3.1.1-3. Typical fracture origin (surface flaw with shallow surface depression) observed in slip cast ASCC AS800 Si_3N_4 specimens with an as-processed surface.

Additional material strength characterization was conducted on AS800 Si_3N_4 material fabricated by gelcasting. The gelcasting process utilizes a small amount of organic monomer and crosslinker in the silicon nitride aqueous slurry. The monomer polymerizes upon heating and the resulting gel (which is $\approx 90\%$ water) rigidizes the silicon nitride slurry into the shape of the mold. The gelcasting process allows cycle times 50 to 80% shorter than slip casting, provides more uniform powder packing, and results in a much higher green strength. The average density of the gelcast and sintered AS800 measured 3.318 g/cc (0.120 lb/in.³).

The mechanical property results for the gelcast AS800 Si_3N_4 are summarized in Table 3.1.1-II. The typical fracture origins observed in the machined specimens at both room and elevated temperatures were surface flaws, as shown in Figure 3.1.1-4. A majority of these flaws were associated with large tabular $\beta\text{-Si}_3\text{N}_4$ grains on the specimen surface or near surface. Occasional fractures were observed to originate from relatively small (50-100 micron [0.002-0.004 in.]) internal and surface inclusions. The inclusion shown in Figure 3.1.1-5 consisted primarily of Fe, with minor constituents of Si and La. Gelcast AS800 test specimens with an as-processed surface condition were fabricated at AlliedSignal Ceramic Components (ASCC) for evaluation in 1997.

Table 3.1.1-II.

Strength characteristics of AlliedSignal Ceramic Components AS800 silicon nitride material fabricated by gelcasting.

Temperature, °C (°F)	MOR strength, MPa (ksi) machined surface
25 (77)	733.2 (106.34) m=16.11
1000 (1832)	683.8 (99.18)
1150 (2102)	586.0 (85.00)
1250 (2282)	545.0 (79.04)
1370 (2500)	520.0 (75.42)

Figure 3.1.1-4. Typical fracture origin (surface flaw with large $\beta\text{-Si}_3\text{N}_4$ grain) observed in gelcast ASCC AS800 silicon nitride.

Figure 3.1.1-5. Fracture origin (internal inclusion) observed in gelcast ASCC AS800 Si_3N_4 specimens. The inclusion consisted primarily of Fe, with minor amounts of Si and La.

AC Cerama CSN101 Silicon Nitride.

Characterization of AC Cerama CSN101 Si_3N_4 was conducted this reporting period. The CSN101 silicon nitride is a high temperature material developed for gas turbine engine components. The low level of sintering additive (2.5% yttria) results in excellent elevated temperature strength and creep resistance and good thermal shock resistance. The CSN101 test material was sectioned from cold isostatic pressed billets that were densified by glass encapsulated hot isostatic press (HIP) processing. While this material was

produced by cold isostatic pressing (CIPing), AC Cerama has a demonstrated capability for injection molding Si_3N_4 turbine components, including axial and radial turbine rotors, vanes, and duct structures. The CSN101 material had an average density of 3.218 g/cc (0.116 lb/in.³). The fracture toughness (K_{IC}) of this material was 5.6 MPa-m^{1/2} (5.1 ksi-in.^{1/2}) determined using the chevron notch method.

The strength characteristics of the AC Cerama CSN101 silicon nitride material are summarized in Table 3.1.1-III. The dominant fracture origins observed in the specimens were surface flaws, as shown in Figure 3.1.1-6. Several of the specimens had fractures initiating from small (10-20 micron [0.0004-0.0008 in.]) internal inclusions, shown in Figure 3.1.1-7. Energy dispersive X-ray spectroscopic analysis revealed these inclusions to consist primarily of iron silicide, with traces of yttrium present, presumably in the form of the sintering additive, yttrium oxide.

Table 3.1.1-III.

Strength characteristics of AC Cerama CSN101 silicon nitride.

Temperature, °C (°F)	MOR strength, MPa (ksi) machined surface
25 (77)	968.0 (140.40) m=15.02
1000 (1832)	742.1 (107.64)
1150 (2102)	690.8 (100.20)
1250 (2282)	721.8 (104.69)
1370 (2500)	588.8 (85.40)

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Figure 3.1.1-6. Typical fracture origin (surface flaw) observed in AC Cerama CSN101 Si_3N_4 .

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Figure 3.1.1-7. Fracture origin (internal inclusion of iron silicide) observed in AC Cerama CSN101 Si_3N_4 .

Kyocera SN253 Silicon Nitride. SN253 silicon nitride material from Kyocera Industrial Ceramics was evaluated this reporting period. The specimens with a machined surface were sectioned from the hub section of AGT-5 axial turbine rotors, while the specimens with an as-processed surface condition were obtained from slip cast billets. The microstructure of this material is similar to that of SN252 Si_3N_4 , with acicular beta- Si_3N_4 grains 10-50 microns (0.0004-0.002 in.) in length (shorter than those observed in the SN252 material) interspersed in a matrix of equiaxed Si_3N_4 grains. The average density of this material measured 3.455 g/cc (0.125 lb/in.³).

The mechanical property results are summarized in Table 3.1.1-IV. The typical fracture origins observed in the machined specimens at both room and elevated temperatures were surface flaws, as shown in Figure 3.1.1-8. The primary strength-controlling flaws observed in the specimens evaluated with an as-processed surface were also surface flaws, occasionally associated with shallow surface depressions, as shown in Figure 3.1.1-9.

Table 3.1.1-IV.
Strength characteristics of Kyocera SN253 silicon nitride material.

Temperature °C (°F)	MOR strength, MPa (ksi)	
	Machined surface	As-processed surface
25 (77)	780.3 (113.17) m=13.48	562.3 (81.55) m=18.48
1000 (1832)	776.4 (112.61)	546.3 (79.23)
1150 (2102)	689.8 (100.05)	567.0 (82.24)
1250 (2282)	685.3 (99.39)	576.5 (83.61)
1370 (2500)	573.1 (83.12)	457.5 (66.35)

Figure 3.1.1-8. Primary strength-controlling feature (surface flaw) observed in ground Kyocera SN253 Si_3N_4 .

Figure 3.1.1-9. Typical fracture origin (surface flaw associated with a shallow surface depression) observed in Kyocera SN253 Si_3N_4 evaluated with an as-processed surface condition.

Kyocera SN281 Silicon Nitride. The strength characteristics of Kyocera Industrial Ceramics SN281 silicon nitride were evaluated this reporting period. Two surface conditions (machined and as-processed) were evaluated. The flexural specimens with a machined surface condition were sectioned from the hub sections of hybrid molded rotors, while the as-processed specimens were obtained from hybrid molded billets. The microstructure of the material, shown in Figure 3.1.1-10, is similar to that of SN252 and SN253 Si_3N_4 , with long acicular grains 10-50 microns (0.0004-0.002 in.) in length dispersed in a matrix of relatively equiaxed silicon

Figure 3.1.1-10. Microstructure of Kyocera SN281 Si₃N₄.

nitride grains. The average density of this material measured 3.355 g/cc (0.121 lb/in.³). The fracture toughness of this material is similar to that of SN252 Si₃N₄, with a K_{IC} of 6.2 MPa-m^{1/2} (6.8 ksi-in.^{1/2}) measured using the chevron notch fracture toughness method.

The strength testing was conducted in accordance with ASTM-C1161 specifications. The mechanical property results are summarized in Table 3.1.1-V. The typical fracture origins observed in the machined specimens at both room and elevated temperatures were surface flaws, as shown in Figure 3.1.1-11. The machined specimens tested at ambient temperature had a lower strength and Weibull modulus than test data obtained from Kyocera. In addition, the room temperature strength was significantly lower than the 1000°C (1832°F) strength, indicating a potential surface flaw blunting/healing mechanism in the elevated temperature specimens. This indicates that the room temperature strength and Weibull modulus values could potentially be increased with a suitable heat treatment process. The fractured specimens were provided to Kyocera for investigation into this phenomenon. The primary

Table 3.1.1-V.
Strength characteristics of hybrid molded Kyocera SN281 silicon nitride material.

Temperature °C (°F)	MOR strength, MPa (ksi)	
	Machined surface	As-processed surface
25 (77)	965.8 (108.58) m=8.57	782.4 (87.96) m=12.93
1000 (1832)	1188.1 (133.58)	788.1 (88.60)
1150 (2102)	1045.4 (117.53)	792.6 (89.11)
1250 (2282)	813.4 (91.45)	684.4 (76.94)
1370 (2500)	777.9 (87.46)	617.4 (69.41)

Figure 3.1.1-11. Typical fracture origin (surface flaw) observed as the strength-controlling feature in ground Kyocera SN281 Si₃N₄.

strength-controlling features in the as-processed bars were surface flaws, as shown in Figure 3.1.1-12. One specimen had a fracture originating at a small inclusion located near the tensile surface. This inclusion, shown in Figure 3.1.1-13, measured approximately 50 microns (0.020 in.) in diameter. Energy dispersive spectroscopy revealed the inclusion to consist primarily of a rare earth oxide, presumably associated with the sintering additive. Kyocera claims a maximum use temperature for the SN281 Si₃N₄ material of 1500°C (2732°F). The furnace used at Allison for MOR testing is limited to approximately 1427°C (2600°F). Allison has shipped specimens to the ORNL HTML for flexural testing at 1500°C (2732°F).

Figure 3.1.1-12. Primary strength-controlling defect (surface flaw) observed in Kyocera SN281 Si_3N_4 tested with an as-processed surface.

Figure 3.1.1-13. Fracture origin (internal inclusion consisting of a rare earth oxide) observed in Kyocera SN281 Si_3N_4 .

3.1.3 Failure Analysis

3.1.3.1 Objective/Approach

Failure analysis details the results of fractographic analyses of ceramic and metallic components that experienced unscheduled damage during rig/engine testing and evaluation. Fractographic analysis is one of the most powerful tools used in the failure analysis of engine or rig-tested components. A careful study of the general and detailed features of the topography of a fracture by visual assessment and scanning electron microscopic (SEM) examination with elemental X-ray analysis provides a wealth of information concerning the failure origin

and the failure mode(s). Analysis of hardware failures allows the separation of design features from material deficiencies, defects, or nonoptimum fabrication procedures and can suggest appropriate corrective measures.

3.1.3.2 Accomplishments/Results

Failure analysis of gasifier module C-6, Build 2 was conducted. This test of an all-ceramic gasifier turbine section consisted of a Kyocera SN253 Si_3N_4 gasifier rotor and a Kyocera SN252 Si_3N_4 scroll. Failure of the gasifier assembly was caused by impact damage resulting from melting of the metallic combustor dome.

3.1.3.3 Discussion

Hot Rig C-6, Build 2. The gasifier assembly C-6, composed of a Kyocera SN253 Si_3N_4 20-blade gasifier rotor and a Kyocera SN252 Si_3N_4 advanced concept scroll, was hot rig tested. The assembly failed while operating at 76% gasifier speed at a temperature of 1270°C (2320°F). The gasifier assembly had accumulated a total test time of 29.9 hr with a maximum rotor inlet temperature (RIT) of 1375°C (2507°F). Post-test inspection revealed the ceramic rotor and scroll had fractured. The rotor lost all of the airfoils while the hub and shaft sections were undamaged. The scroll body fractured into several large pieces.

Figure 3.1.3-1 shows the condition of the engine looking into the regenerator disk cavities. The scroll was fractured into several sections, with large pieces of the scroll still hanging onto the gasifier housing. There was ceramic debris observed at the bottom of the regenerator cavities. The thermal insulation layer on the gasifier housing had several large cracks as a result of the failure. Figure 3.1.3-2 is a close-up of the gasifier housing and ceramic rotor. The rotor lost all airfoils, with the hub and rotor shaft not showing any distress. The metallic combustor body (Haynes 230) was in good shape but part of the metallic dome had melted. Figure 3.1.3-3 shows that all the louvers except one were melted and lost. Two tabs on the outside were burned through. The side wall of the dome under the tab exhibiting the most thermal distress was also burned through. Figure 3.1.3-4 shows the as-torndown condition of the NGK MAS regenerator

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Figure 3.1.3-1. Condition of the engine looking into the regenerator disk cavities.

TE97-1007

Figure 3.1.3-2. Close-up of the gasifier housing and ceramic rotor.

TE97-1008

Figure 3.1.3-3. All the louvers except one were melted and lost.

TE97-1009

Figure 3.1.3-4. As-torndown condition of the NGK MAS regenerator disks.

disks. The left side disk exhibited a deep gouge mark with some cracking along the bond lines. Cracking probably originated at the opposite rim. The right side disk was in good shape.

Figures 3.1.3-5 and 3.1.3-6 show the reconstructed scroll. The scroll body broke into large pieces and all large pieces were recovered. The shroud and the middle sections broke into small pieces and could not be completely reassembled. Molten metal pieces splashed and adhered to the inside wall of the scroll inlet. Metallic deposits also were found further down the inside wall of the scroll. X-ray energy dispersive analysis (XEDA) indicated that the metallic deposits consisted of Rene 41 type material, from which the dome louver was fabricated. Figure 3.1.3-5 also shows the continuous circumferential rub mark on three adjacent large shroud pieces. There were no continuous rub marks on other smaller shroud pieces. XEDA indicated the continuous rub mark consisted of metallic Rene 41 type material.

Figure 3.1.3-5. Reconstructed scroll.

Figure 3.1.3-6. Reconstructed scroll.

There were four major continuous cracks observed on the scroll. Three originated at the three key slots. The longest one originated at the shroud (Figure 3.1.3-6). The exact origin could not be determined due to missing pieces around the origin. Apparently, one of the large molten metal pieces was caught between the rotor and shroud. The jolt might have fractured the scroll, knocked out all airfoils of the rotor, and initiated the fractures from the key slots.

Figure 3.1.3-7 shows the remains of the rotor. All of the airfoils were fractured and broken off, with only secondary fracture features remaining. The broken airfoils were fractured into small pieces. At the leading edge of one small piece of the airfoil, numerous foreign material deposits were found. The foreign materials were later identified by XEDA as either Haynes 230 or Rene 41 type materials, which were the materials used in the dome and louver, respectively.

Figure 3.1.3-7. Remains of the rotor.

3.2 CERAMIC COMPONENT PROCESS DEVELOPMENT AND FABRICATION

3.2.2 Schuller International

3.2.2.1 Objective/Approach

Efforts at Schuller International have been directed toward the development of an injection moldable insulation system addressing low cost, high volume production for automotive gas turbines. The primary approach has been modifying the insulation material system for improved injection molding properties while developing the molding process for both simple and complex engine components. Additional developmental items being addressed include evaluation and improvements to insulation/metal bondability, cyclic durability, and erosion resistance.

3.2.2.2 Accomplishments/Results

- An AGT-5 engine housing, gasifier housing, and combustor cover were successfully injection molded with the refined Schuller process and placed on test as a full engine installed in a test vehicle.
- Sixty hours of engine running were accumulated at Allison on the vehicle test bed engine and the engine was torn down for inspection of the insulation. The inspection revealed no degradation of the insulation surface or its bonding to the engine surfaces.
- Tooling was made to inject a hybrid engine housing with insulation to investigate feasibility of process translation to other complex engine shapes.
- Three hundred hours of durability testing were completed at Allison using the AGT-5 hot gasifier rig. The rig housing, gasifier housing, and combustor cover tested were insulated with Schuller injected insulation (SMIG 2.5)

Results of Rig Testing. After 300 cyclic durability hours at Allison, the engine components were disassembled and inspected. No erosion or cracking was evidenced on the combustor cover insulation. The gasifier housing showed some signs of cracking but remained in serviceable condition. The forward cavity of the engine housing had some debonding of the insulation from the interior surfaces but not sufficient enough in nature as to discontinue testing. The aft cavity showed considerable insulation erosion from the hot exhaust stream. The eroded insulation material was blown through the regenerators and led to frequent clogging of the Lamilloy® porous metal test combustor and dome. The cause of this erosion was determined to be the absence of a power turbine section removing temperature and velocity from the gas stream prior to its impinging on the insulation surfaces. A contributing factor was the absence of an exhaust diffuser in the aft cavity. A blast shield of Haynes Alloy 230 was fabricated and installed completely shielding the insulation surfaces from the exhaust stream. No significant combustor clogging was noted following this repair.

The hot rig housing and insulation remained in serviceable condition.

3.2.2.3 Discussion

Detailed discussions are given in the annual reports provided by Schuller which are included in Appendices A and B.

3.2.4 Corning, Incorporated

3.2.4.1 Objective/Approach

The objective of the regenerator activity with Corning, Inc., is the development of the materials and processing technology required for the fabrication of a reliable, low cost, high performance, one-piece extruded ceramic regenerator disk. The resulting disk must be capable of operating for 3500 hr in an automotive gas turbine environment at temperatures up to 1150°C (2102°F) at the regenerator inlet.

Two ceramic material systems are being developed for this effort, lithium-aluminosilicate (LAS) and magnesium-aluminosilicate (MAS). The extrusion die development approach has focused on the development of large diameter dies with a cell count of approximately 1100 rectangular cells/in.²

3.2.4.2 Accomplishments/Results

- Development of MAS and LAS regenerator core materials continued.
- Optimization of LAS material composition was completed.
- LAS and MAS material samples and disks fabricated and delivered to Allison for test and evaluation

3.2.4.3 Discussion

Summaries of all activities of Corning, Inc., in support of the program during 1995 and 1996 are given in the annual reports provided by Corning. These reports are included in Appendices C and D of this document.

* Lamilloy is a registered trademark of Allison Engine Company, Inc.

3.2.5 AlliedSignal Ceramic Components

3.2.5.1 Objective/Approach

A ceramic component development effort was conducted with AlliedSignal Ceramic Components (ASCC). The objective of the ASCC HVTE-TS activity is the demonstration of gelcasting as a viable production process for the manufacturing of ceramic components. The gasifier turbine rotor is the primary focus of the component development efforts. A second task was the development of an AGT-5 engine one-piece ceramic combustor. The material being used is AS800 Si₃N₄, a high temperature in-situ reinforced silicon nitride. The technical effort is focused on the two tasks of the fabrication of AS800 test specimens for Allison material characterization and the development and demonstration of process parameters for fabrication of gelcast axial rotors using reusable hard tooling.

3.2.5.2 Accomplishments/Results

- Material samples and AS800 silicon nitride rotors were provided to Allison for test and evaluation.
- Two two-piece AS800 silicon nitride combustors were provided to Allison for test and evaluation.

3.2.5.3 Discussion

Summaries of all activities of ASCC in support of the program during 1995 and 1996 are given in the annual reports provided by AlliedSignal. These reports are included as Appendixes E and F of this document.

3.2.6 Kyocera Industrial Ceramic

3.2.6.1 Objective/Approach

A development program with Kyocera Industrial Ceramics was conducted addressing development and fabrication of gasifier rotors and turbine scrolls.

3.2.6.2 Accomplishments/Results

- Five SN253 20-bladed gasifier rotors passed NDE and spin proof test screening and were delivered to Allison.
- Four rectangular cross-section scrolls were fabricated and delivered to Allison.
- Two circular cross-section scrolls were fabricated and delivered to Allison.

3.2.6.3 Discussion

Summaries of all activities of Kyocera Industrial Ceramics Corporation in support of the program during 1995 and 1996 are given in the annual reports provided by Kyocera. These reports are included in Appendixes G and H of this document.

4.0 COMPONENT RIG DEVELOPMENT AND TEST

4.1.2 Hot Gasifier Rig Development

4.1.2.1 Objective/Approach

The hot gasifier rig is under constant development to enhance the rig's capability to test ceramic gasifier components under reference powertrain design (RPD) conditions. A secondary objective was to complete setup and checkout of the AGT-5 hot gasifier rig transferred from the GM Technical Center to Allison Engine Company.

4.1.2.2 Accomplishments/Results

- Refurbishment of an Allison test facility for the AGT-5 hot gasifier and engine test programs was completed.
- Extensive checkout of the test facility including control, instrumentation, and data acquisition systems was completed using the all-metal AGT-5 hot gasifier rig.

4.1.2.3 Discussion

With the transfer of HVTE-TS program responsibilities to Allison, facilities were refurbished and set up to accommodate program testing requirements. Test hardware transferred to Allison included the AGT-5 test-bed engine and hot gasifier rig. Also transferred were related test equipment including a dynamometer, inlet air conditioner system, air ducting, oil lubrication system, engine controller, and temperature/pressure measurement instrumentation.

During 1995, the AGT-5 test hardware and auxiliary test equipment were installed in the test cell. Procurement, fabrication, and installation of test cell mechanical, electrical, and instrumentation systems were completed. The facility consists of two test-beds: one for the AGT-5 hot gasifier rig and one for the AGT-5 engine. Figure 4.1.2-1 shows the hot gasifier rig installed in Allison test cell 892. Figure 4.1.2-2 shows the engine installed in the test cell.

Figure 4.1.2-1. AGT-5 hot gasifier test rig.

Figure 4.1.2-2. AGT-5 engine test-bed.

Basic facility control and operation was facilitated by using a programmable logic controller (PLC). The PLC enables and controls cell operation including test-bed accessory systems, fuel delivery, and room air/exhaust systems. The PLC also incorporates provisions to enhance cell/test-bed safety. It monitors selected temperatures, speeds, and vibrations with respect to preset allowable limits and generates visual and audible limit violation alarms as required. Selected limit violations can also automatically initiate test-bed shutdown. Engine/gasifier rig digital controller hardware and software were supplied with the AGT-5 test hardware. New control logic was developed at Allison and a vendor selected to provide custom digital controller hardware and software.

The new controller provides for hot rig, regenerator rig, or engine automated control during startup, operation, and shutdown. The controller activates required accessories and engages the electrically driven starter system. During the start process, the controller verifies critical sensor signals are within proper range. After ignition and the gasifier rotor speed has achieved starting threshold, the controller disengages the starter and switches to automatic control mode after idle speed is achieved. During

normal control mode, the controller modulates fuel flow to maintain a prescribed run condition. It also monitors critical signals to ensure safe rig/engine operation. The controller performs a controlled system shutdown automatically when selected safety threshold levels are exceeded or upon command from an external controller.

A personal computer (PC) is interfaced to the digital controller permitting operator manual rig operation or preprogrammed automatic control of rig operation. During automatic operation, this PC commands the digital controller based on a defined durability or driving schedule.

The test cell incorporates instrumentation to measure rig speed, pressures, temperatures, vibration, air and fuel flow, etc. Real-time data acquisition and reduction are accomplished by three additional PCs. One of the PCs controls and acquires data from the instrumentation systems. The data are shared electronically with the remaining two PCs. The PCs convert measured data to engineering parameters, perform checks to ensure safe rig operation, log data to disk for permanent record, and provide real-time tabular and graphic display of rig operation, status, and performance.

The hot gasifier rig was fitted with a special sight tube with quartz window to permit observation of the gasifier turbine rotor exit during operation. This sight tube also incorporates an exhaust port (secondary exhaust) to allow diversion of hot gas flow from the rotor overboard of the rig. A high energy ignition system and compatible igniter were incorporated into the rig for reliable starting.

The AGT-5 hot gasifier rig test-bed is fully operational. The facility, instrumentation, control, and data acquisition systems have completed operational checkout. The AGT-5 engine test-bed installation is complete. Checkout of engine systems operation remains.

4.1.4 Regenerator Rig Development

Single Disk Hot Flow Performance Rig. The shakedown phase of the single disk hot flow performance rig is complete. This rig was designed and built for regenerator system development. Figure 4.1.4-1 is a photograph of the rig. This rig incorporates two independently controlled heating sources to allow variation of regenerator inlet air by an electric heater and gas side inlet air using a natural gas burner.

Extensive work was performed to develop a rig capable of handling the entire temperature and flow regime of the regenerator disk. An in-line

calibration of the two orifice systems provided a reference baseline measurement and utilizing the difference of two orifice meters during testing provided an accurate method to evaluate seal leakage. Careful attention was required to eliminate any possible nonrepresentative leak paths that could develop and introduce error. Minor modifications were made to the rig during the shakedown phase. One involved a rework to the rig piping just upstream of the ceramic disk located downstream of the burner unit. Due to thermal growth, the internal Hastelloy X liner impacted the ceramic disk causing failure that then sent fractured pieces downstream into the regenerator disk. A second configuration change was incorporated after a post-build investigation where it was determined that the housing inserts had buckled, thus restricting the full L-seal travel. The housing inserts were removed from the rig, straightened, and excess material removed to allow full seal travel.

The rig is capable of providing steady-state temperature and airflow conditions within the entire regenerator operating envelope. The maximum sustainable gas side inlet temperature obtained was 1800°F at a inlet mass flow rate of 0.350 lbm/sec at 433°F. A short excursion to a maximum temperature of 1916°F at a gas mass flow rate of 0.315 lbm/sec was performed to

TE97-984

Figure 4.1.4-1. Single disk hot flow regenerator rig.

investigate rig capabilities. Checkout and verification of the extensive temperature and pressure measurements used in the calculation of the disk thermal effectiveness and system pressure drop were performed. The shakedown phase of the rig is complete and the rig is ready for full-scale development and evaluation of the regenerator system.

4.2 COMPONENT RIG TESTING

4.2.3.4 Regenerator System Development and Test

4.2.3.4.1 Objective/Approach

The regenerator system development efforts conducted under this task were focused on component design, testing, and evaluation of candidate materials and systems for use in an advanced high temperature automotive turbine engine. The primary objective of the task was to develop an efficient and cost effective regenerator system compatible with high volume manufacturing.

Activities associated with the regenerator and seal system development were conducted in-house at Allison and with partners including Corning Incorporated and Argonne National Laboratory (ANL). Corning pursued regenerator disk material and fabrication process development. ANL provided support on regenerator seal development.

4.2.3.4.2 Accomplishments/Results

Substantial progress was made in all areas of the regenerator system development including:

- A 500-hr cyclic durability test of one-piece extruded MAS ceramic disks with regenerator inlet temperatures to 1750°F (954°C) was successfully completed.
- Regenerator seal material development, tribology studies, and testing were conducted at Argonne Labs.

4.2.3.4.3 Corning Regenerator Material and Process Development

Corning Incorporated is pursuing regenerator disk material and fabrication process development. Corning focus during this reporting period has been on regenerator disk overall cost reduction to levels acceptable for automotive use. Effort has concentrated materials development

and testing on their two most developed materials: magnesium-aluminosilicate (MAS) and lithium-aluminosilicate (LAS).

The relation between thermal expansion and strength of solid extruded rods as shown in Figure 4.2.3.4-1 provides a capsule representation of MAS and LAS material development. The low expansion of MAS is largely due to controlled microcracking. It is theorized that when the microcracks are highly stressed by cycling to elevated temperatures, they grow and result in strength loss. It was thought that MAS with reduced microcracks would result in a material with slightly higher expansion and higher strength than that of the MAS-2 point in Figure 4.2.3.4-1. This effect should result in a better retained strength after thermal cycling and shock. When evaluated using a slow thermal cycling test, MAS materials formulated in 1995 with less microcracking did show a significant improvement as demonstrated by less change in length after slow cycling tests. However, when evaluated using a rapid cycling thermal shock test, the retained strength was no better than MAS-2. Additional development of MAS materials was put on hold until after extruded disks with long time exposure in engine tests at Allison become available.

LAS materials work during 1995 and 1996 concentrated on reducing the material cost and firing shrinkage (which results in distortion and cracking). LAS is currently a glass-based material (MAS is 100% mineral-based) that involves more pre-extrusion processing and a higher extrusion

Figure 4.2.3.4-1. Corning MAS and LAS materials development.

pressure than MAS, resulting in higher processing cost. LAS-3 development involved adding mineral content to the extrusion batch to decrease cost, improve extrudability, and reduce firing shrinkage to acceptable levels. The LAS-4 material is a progression from LAS-3 obtained by optimizing sintering and firing cycles.

Early ceramic regenerator cores for gas turbine engines were fabricated by a tape wrapping process. This process is not cost effective for high-volume production. Independently of the regenerator effort, Corning has demonstrated that extrusion of MAS cellular structures for automotive catalytic converters is a feasible production process. Current extruded converter substrates have 400 to 500 cells/in.² (62 to 78 cells/cm²) with wall thickness to 0.0065 in. (0.165 mm). Thermal efficiency targets for regenerative gas turbine engines dictate high heat transfer surface area and low pressure drop for rotary regenerators. This translates to cell counts on the order of 1000 to 1500 cells/in.² (155 to 233 cells/cm²) and wall thickness of 0.004 to 0.005 in. (0.102 to 0.127 mm), respectively. Extending current die technologies based on the catalytic converter extrusions, Corning has provided Allison with regenerator cores having 1100 cells/in.² (171 cells/cm²) and 0.006 in. (0.152 mm) thick walls in MAS material. Novel die development work underway in support of Corning's catalyst business will provide strength improvements to the die, enhancing the capability for single piece extrusion of hybrid vehicle size cores.

4.2.3.4.4 Allison Regenerator Design and Fabrication

Regenerator disk design has concentrated on the hub area to reduce cost and to incorporate features made possible by extrusion. The latest design incorporates a recent Corning development, namely an extruded hub. Previous designs had used a cast hub that was separately fired and machined on its outside diameter. The hub was then joined to a separately fired, pre-machined matrix core in a third firing cycle using a cement that was matched to the thermal expansion of the individual parts. The extruded hub is pressed into a separately extruded disk

matrix while both are still in the "green" as-extruded state. The assembly then goes through a single firing with no pre-machining or cement joint required. Figure 4.2.3.4-2 shows a green extruded hub and one that has been installed and fired into a piece of disk matrix.

Previously, the outside diameter of the disk hub was driven by the matrix production process. The diameter of the wrapping mandrel or the extrusion die center support rod dictated the hub diameter. The center bearing was redesigned so that it and the disk hub would be covered by the narrower, constant width seal cross arm. This maximized the unblocked open frontal area of the disk available for heat transfer.

The hub redesign transferred all of the retention features for the center bearing from the disk hub to the more easily machineable metallic mounting spindle. The pressed-in hub is extruded with a single constant diameter through-hole that is finished machined after firing with one constant diameter boring operation.

The pressed-in extruded hub has undergone static-load testing successfully withstanding approximately eight times the maximum operational radial forces and forty times the maximum operational axial forces.

During 1996, a pair of MAS material extruded disks completed 500 hr of steady state durability testing at 1750°F (954°C) regenerator hot side inlet temperature. The small regenerator test rig (Figure 4.2.3.4-3) was operated at two conditions

TE98-501

Figure 4.2.3.4-2. Green extruded hub and extruded hub fired into disk matrix.

^{*} Day, J. P., 1996, "Automotive Gas Turbine Heat Exchanger Development," SAE 960085.

TE98-502

Figure 4.2.3.4-3. Small regenerator gasifier test rig.

of interest for regenerator testing: a high temperature/low pressure condition with a regenerator hot side inlet temperature of 1750°F (954°C), combined with an air side inlet pressure of 6.0 psig (41.3 kPa); and a low temperature/high pressure condition with a regenerator hot side inlet temperature of 1550°F (843°C) and air side inlet pressure of 32.5 psig (224.1 kPa). Air side inlet temperature was not measured during these tests. One of the disks is shown in Figure 4.2.3.4-4. These disks have also completed 50 hr of steady state testing at the low temperature/high pressure condition.

TE98-503

Figure 4.2.3.4-4. Extruded MAS disk after 500 hr durability test.

In addition to the full rotating disk testing, regenerator disk material samples have been exposed to simulated engine acceleration temperature spikes using a gas-fired cyclic thermal rig [1.5 x 2.5 x 3 in. (38.1 x 63.5 x 76.2 mm) sample size]. After testing, the regenerator material is analyzed to determine hot face strength loss.

The samples were exposed to cycles that represent expected operating transients including initial startup. The start-up accelerations were the most severe, with a rapid step from ambient to 1900°F (1039°C), held for several seconds, and followed by a rapid decrease to 1350°F (732°C). The rig cycled from 1350°F (732°C) to 2090°F (1143°C) [ten percent was added to the 1900°F (1039°C) peak to account for burner pattern] and back to 1350°F (732°C) every 15 seconds.

Samples of both MAS and LAS extruded material were evaluated for various numbers of temperature cycles. Figure 4.2.3.4-5 presents some results and shows how the samples are sliced at six axial stations through the disk from hot to cold face. Each slice is cut into three modulus of rupture (MOR) test bars and strength data obtained. All data shown is from samples tested to the 2090°F (1143°C) peak temperature. While this test indicates LAS is useful to 2090°F (1143°C), MAS retains almost no strength when cycled to this temperature. Testing with cycles to lower peak temperatures is underway to determine an acceptable use limit for MAS.

4.2.3.4.5 Argonne National Laboratory

A significant part of the total cost of the regenerator is attributable to the seal system. Allison has used high temperature carbon/graphite, and plasma sprayed coatings (primarily nickel oxide/ calcium fluoride applied over a metal substrate) for the regenerator disk primary seal. Most of the cost for these seal parts is incurred in the processing of these materials into finished parts. In addition to lower cost processing, better friction and wear performance are also goals.

ANL has evaluated over 45 candidate low temperature materials, including 6 different types of carbon/graphite, over 12 different types of carbon-carbon, 3 different types of advanced high temperature polyimides, and various other materials. These materials were subjected to a 7-hr

CYCLIC THERMAL RIG M.O.R. RESULTS

TE98-504

Figure 4.2.3.4-5. Modulus of rupture for disk samples exposed in the cyclic thermal rig.

screening test in which temperature was stepped up in four 55°C increments (99°F step) from starting to maximum temperature and then stepped back down while friction drive torque was measured. Most of these materials were unsuitable, being rejected for high wear, high friction, or both. The baseline for comparison for this test was Allison's standard carbon/graphite material which exhibits friction coefficients below 0.2, and wear below 4×10^{-8} in/hr (10^{-9} m/hr). One polyimide material, one carbon-carbon, and one alternate carbon/graphite material have shown friction and wear performance equal to or better than the baseline material. The carbon/graphite and polyimide are being subjected to longer duration testing (150+ hr) to better assess their capability.

The high temperature coatings work is directed at a replacement for nickel oxide/calcium fluoride. Again the goals were to reduce cost, friction, wear, and environmental impact. This work is being conducted on a high temperature friction and wear rig capable of temperatures to 1800°F (982°C). Plasma spray is currently being used to

apply the candidate wear face coatings to the wear buttons. High cell density MAS, LAS and AS disks both wrapped and extruded, have been supplied by Corning to support this testing. Several metal oxides in various combinations have been evaluated in this test rig. Coatings in which nickel oxide forms the major constituent with calcium fluoride, barium titanate, magnesium oxide, and strontium fluoride in various combinations as minor constituents have been tested. A second group consists of coatings that contain various mixtures of zinc oxide, tin oxide, calcium fluoride, strontium fluoride and magnesium oxide. The last group consists of modifications of commercially available coatings such as NASA's PS300 series coatings, and Tribolite. The most promising coating to date is a mixture of magnesium oxide, zinc oxide, and calcium fluoride that has exhibited friction coefficients as low as 0.2 at 1800°F (982°C).

5.0 PERFORMANCE AND ENDURANCE TESTING

5.1 COMBUSTION SYSTEM DEVELOPMENT

Although no efforts were directed solely to the development of a low emissions combustion system in 1996, a brief summary is provided on the performance of the diffusion flame combustor used to support the AGT-5 "all-ceramic" gasifier rig test. Some of the issues that arose during the endurance testing may also be relevant for the low emissions combustor and consequently are reported here. The combustor used to support this test was a standard Lamilloy diffusion flame combustor.

To provide more reliable ignition on Diesel 2, a high energy exciter was substituted for the automotive style ignition system. This system provided reliable ignition for the duration of the endurance test. A number of combustor components had reduced life in the 300 hr durability test. In each case the failure can be related to the high burner inlet temperature of the regenerative engine running at high turbine inlet temperatures. The high temperatures led to failures in three areas:

- The fuel nozzle tip began to oxidize.
- The Lamilloy on the combustor body delaminated forming a welt.
- The combustor dome was distorted due to high temperature and load induced by the combustor body.

The tip of the fuel nozzle oxidized significantly after 150 hr of testing and was beginning to curl. This condition gradually worsened until the nozzle was deemed unusable. A new supply of fuel nozzles was provided in which a material change had been made to Hastelloy X for the fuel nozzle shroud. New fuel nozzles did not show any oxidation.

The Lamilloy on the combustor body delaminated and formed a welt near the location of one of the primary holes after 180 hr of operation. A second combustor was used for the remaining portion of the endurance test without any delamination occurring.

Combustor domes sagged at the location of the support pin that holds the combustor body. Domes had to be replaced at intervals of 50 to 75 hr of operation. The cause for the distortion was determined to be the aerodynamic force of the combustor body on the support pin.

No low emissions combustion development is planned for 1997. The reduced life of the Lamilloy diffusion flame burner indicates the need for a more thermally resistant design. To address this, an effort is under way to design and proof test a ceramic diffusion flame combustor to provide a longer life combustor in support of further high temperature ceramic testing.

5.2 CERAMIC GASIFIER SECTION CYCLIC DURABILITY TEST

A major contractual milestone of the HVTE-TS program scheduled for 1996 was to complete a 300-hr cyclic durability test of an "all-ceramic" gasifier section. The "all-ceramic" gasifier section consisted of a ceramic inlet scroll assembly with integral ceramic rotor shroud and a ceramic gasifier rotor. The objective of the 300-hr test was to demonstrate durability performance of key technology components of the automotive gas turbine (AGT) including:

- Ceramic gasifier scroll assembly
- Ceramic gasifier turbine rotor
- Ceramic regenerator cores
- Regenerator cross arm hot seal materials
- Injection-molded insulation

A standard AGT-5 metal Lamilloy diffusion flame combustor was used during the 300-hr durability test since a proven ceramic combustor design was yet to be demonstrated.

The ceramic inlet scroll assembly was fabricated from SN252 silicon nitride by Kyocera Industrial Ceramics (Figure 5.2-1). The ceramic gasifier turbine is a 20-bladed axial flow rotor fabricated from SN253 silicon nitride by Kyocera (Figure 5.2-2). A newly designed mounting system positioned the ceramic scroll with respect to the assembly centerline using a crosskey system piloted from the gasifier rotor housing. This design allows the

TE97-985

Figure 5.2-1. SN252 silicon nitride inlet scroll by Kyocera.

TE97-986

Figure 5.2-2. SN253 silicon nitride gasifier rotor by Kyocera.

ceramic rotor and scroll to move as a single unit. Ceramic rotor stability and bearing life were enhanced with the addition of a patented Allison oil squeeze film damper design to both the compressor and turbine bearings to ensure proper damping and to limit oil flow requirements.

Extruded magnesium-aluminosilicate (MAS) ceramic pasted regenerator disks (Figure 5.2-3) were used with the AGT-5 hot gasifier rig. The cores were fabricated by pasting rectangular extruded MAS segments together and then machining the assembly to form a disk of the proper diameter.

Two different regenerator cross arm hot seal materials were used during hot gasifier rig testing: NiO/CaF₂ plasma sprayed onto an I-625 (Inconel 625 nickel alloy) substrate and I-112 (ZnO/SnO₂/CaF₂) plasma sprayed onto an I-625 substrate. Specific usage and performance are discussed below. The plasma sprayed cross arms are shown in Figure 5.2-4.

TE97-987

Figure 5.2-3. AGT-5 MAS pasted regenerator core.

TE97-988

Figure 5.2-4. AGT-5 regenerator hot seal plasma sprayed cross arms.

Hot rig components injection molded with insulation included the gasifier housing, AGT-5 engine housing, and combustor cover. These components are shown in Figure 5.2-5. Figure 5.2-6 is a close-up view of an injection-molded AGT-5 engine housing.

The ATTAP gas turbine engine durability cycle was established for automotive prime power application. The cycle has been periodically

Figure 5.2-5. Injection-molded insulation components for the AGT-5 hot gasifier rig.

Figure 5.2-6. AGT-5 engine housing with injection-molded insulation.

updated to better simulate real world vehicle use and to avoid premature failure of supporting metal hardware and insulation damage. In 1993, the durability cycle was again modified to simulate the 1992 RPD update on the Combined Federal Driving Cycle (CFDC). The revised cycle was 1 hr long while the CFDC was about 55 minutes. There were two starts per hour with one 10-minute soakback. The maximum rotor speed during the new cycle was 90%, corresponding to a rotor inlet temperature of 2100°F (1149°C). All subsequent ATTAP durability testing was based on the new cycle.

The HVTE-TS project emphasis shifted the automotive gas turbine engine environment from prime power to auxiliary power for hybrid vehicles also utilizing batteries and electric drive systems. The durability test schedule was again adjusted to simulate an accelerated life test based

on the cyclic operating modes of a typical hybrid vehicle turbine engine environment. The schedule was 1 hr in duration with one start/stop per cycle and included 20 accelerations and 24 decelerations. It also included a 9-minute soakback at the end. Execution of a complete schedule was one durability cycle. The maximum rotor speed of the cycle was 90% rated speed, corresponding to a rotor inlet temperature (RIT) of 2100°F (1149°C). Variations of this basic cyclic durability schedule (gasifier turbine rotor operation to 100% rated speed and RIT to 2500°F [1371°C]) were used during the test program in order to increase the severity of the ceramic component environment yet remain within the temperature limitations of rig hot section metallic components.

During test programs incorporating ceramic turbine scrolls, RIT is calculated rather than measured due to the difficulty of positioning thermocouples directly into the scroll. The equation for calculated RIT is based on a curve fit to temperature rise curves for diesel fuel. Inputs to the curve fit are combustor inlet temperature and combustor fuel/air ratio. Ceramic gasifier rotor proof testing (using a metallic inlet scroll with rotor inlet thermocouples installed) demonstrated excellent correlation between measured and calculated RIT.

Gasifier RIT control is accomplished by conditioning rig inlet air temperature, compressor bleed, exhaust cavity cooling air pressure, and secondary exhaust duct flow. The regenerator disk inlet temperature is a function of rotor exit temperature with exhaust cavity cooling air pressure and secondary exhaust duct flow levels providing auxiliary means to control disk inlet temperature.

In March 1996, a ceramic rotor proof test using a metallic turbine scroll was initiated using the AGT-5 hot rig. The purpose of the proof test was to verify the structural integrity of the ceramic rotor assembly. After proof testing, the ceramic rotor was incorporated into an "all-ceramic" gasifier power section assembly. The assembly was used to build up engine housing S/N 12 for durability testing. Table 5.2-I identifies the gasifier assembly ceramic parts used in this build. Engine housing S/N 12 incorporated hand layup insulation rather than injection-molded insulation. The gasifier housing and combustor cover were injection-molded with insulation. MAS ceramic pasted regenerator disks were used. Regenerator cross arm hot seal material (both left and right) was NiO/CaF₂ plasma sprayed onto an I-625 substrate.

First rig testing of an "all-ceramic" gasifier section was initiated in April 1996. Figure 5.2-7 shows rig performance during a 100% gasifier speed durability cycle with a RIT of 2507°F (1375°C). A total of 29.9 test hours was accumulated on the initial ceramic section build before a hardware failure occurred on May 14, 1996. The ceramic rotor and scroll were destroyed along with severe damage to other components. Failure analyses have concluded that sections of the metallic thermal variable geometry (TVG) combustor dome melted and deposited molten metal between the ceramic turbine rotor and its blade track causing failure of the rotor. The rotor failure then resulted in subsequent ceramic scroll assembly failure and damage to other components.

Table 5.2-I.
Gasifier section ceramic parts on test.

Gasifier rig	Gasifier build No.	Component	Serial No.	Supplier	Material
S/N 12	2	20-blade rotor	3-3	Kyocera	SN253 Si ₃ N ₄
S/N 12	2	Scroll	11-2	Kyocera	SN252 Si ₃ N ₄
S/N 14	3	20-blade rotor	4-2	Kyocera	SN253 Si ₃ N ₄
S/N 14	3	Scroll	34-2	Kyocera	SN252 Si ₃ N ₄

Figure 5.2-7. Ceramic durability rig operating to full rated speed and temperature.

The gasifier assembly was rebuilt with new ceramic components. It was installed in the S/N 14 engine housing, which had been refurbished with new injection-molded insulation. The buildup also included new ceramic MAS pasted regenerator disks (and seals) and new combustor components. Two different regenerator cross arm hot seal materials were used. The left cross arm was NiO/CaF₂ plasma sprayed onto an I-625 substrate. The right cross arm was I-112 (ZnO/SnO₂/CaF₂) plasma sprayed onto an I-625 substrate. This arrangement allowed simultaneous evaluation of two alternative cross arm coatings. A standard metallic AGT-5 Lamilloy combustor dome was used instead of the TVG dome. The gasifier section ceramic components along with the AGT-5 MAS regenerator disks and ceramic coated hot seal cross arms are shown in Figure 5.2-8.

Testing on the second buildup of the "all-ceramic" gasifier section was initiated in July 1996. The first durability cycle was completed after 1.5 hr of testing. The cycle was based on a 90% speed schedule and achieved a maximum RIT of 2143°F (1173°C) with a maximum regenerator inlet temperature of 1745 °F (952°C).

Rig performance at 12 hr into the durability test is shown in Figure 5.2-9. During execution of this 92% speed durability schedule, a maximum RIT of

2449°F (1343°C) was reached with a maximum regenerator inlet temperature of 1929°F (1054°C).

Figure 5.2-10 shows rig performance at 54 hr of testing. During execution of this 90% speed durability schedule, a maximum RIT of 2443°F (1339°C) was reached with a maximum regenerator inlet temperature of 1947°F (1064°C).

Rig performance at 150 hr into the durability test is shown in Figure 5.2-11. During execution of this 96% speed durability schedule, a maximum RIT of 2270°F (1243°C) was reached with a maximum regenerator inlet temperature of 1744°F (951°C).

Figure 5.2-12 is a photograph of the ceramic gasifier rotor at 100% rated gasifier rotor speed as viewed through the rig sight tube quartz window.

Scheduled rig inspections were conducted every 50 hr of testing. These inspections were primarily to evaluate gasifier section ceramic component condition including structural integrity and rotor blade tip clearance. No problems associated with the "all-ceramic" section were discovered during these inspections.

Figure 5.2-8. Hot gasifier rig ceramic components.

Figure 5.2-9. Ceramic durability rig performance (12 hr).

Figure 5.2-10. Ceramic durability rig performance (54 hr).

Figure 5.2-11. Ceramic durability rig performance (150 hr).

Figure 5.2-12. Ceramic gasifier rotor as viewed from sight tube.

A total of 14 nonscheduled rig inspections were made. These inspections were nonceramic component related. At 64 hr into the durability test, the left regenerator hot seal cross arm (NiO/CaF₂ plasma sprayed) was replaced with a cross arm having the I-112 (ZnO/SnO₂/CaF₂)

plasma sprayed coating. The original NiO/CaF₂ coated arm did not develop a full contact wear pattern and was judged to be a potential source of leakage. At 123 hr, a major repair of the engine housing injection-molded insulation was completed. Several areas on the left and right hot

side suffered insulation debonding from the metal housing and had broken away. The remaining insulation was trimmed and fixtures made to injection mold repair the affected areas. A part of the fixture was designed to be left in place to provide a blast shield from the high velocity rotor exhaust gases.

The 300-hr durability test of the "all-ceramic" gasifier section was successfully completed during November 1996. Rig performance at 300 hr of cyclic durability testing is shown in Figure 5.2-13. During execution of this 96% speed durability schedule, a maximum RIT of 2130°F (1165°C) was reached with a maximum regenerator inlet temperature of 1663°F (906°C).

Figure 5.2-14 shows rig speed, rotor inlet temperature, and regenerator disk inlet temperature maximum values for each run of the 300-hr durability test. A run is defined as a successful rig start to idle speed, manual or automatic durability cycle rig operation, and then rig shutdown. The majority of the runs prior to 215 test hours were executed using a durability schedule more severe than the base schedule (90%

rated gasifier rotor speed at RIT of 2100°F [1149°C]). After 215 test hours, rig operation was controlled to maintain RIT near the base schedule of 2100°F (1149°C).

Results of the 300-hr durability test can be summarized as follows:

- The ceramic turbine scroll assembly showed no evidence of physical or structural problems.
- The ceramic gasifier turbine rotor showed no evidence of physical or structural problems.
- The "all-ceramic" gasifier section completed the durability test without teardown.
- Maximum RIT of 2497°F (1369°C) was reached one time. RIT exceeded 2400°F (1316°C) 12 times. RIT was above 2100°F (1149°C) for almost 10 hr.
- A total of 392 successful ceramic rig starts were executed.

Figure 5.2-13. Ceramic durability rig performance (300 hr).

TE97-998

Figure 5.2-14. Maximum operating conditions reached throughout the 300-hr test of the "all ceramic" gasifier section.

- A total of 238 automatic 1-hr durability test schedules were executed with gasifier rotor speeds to 101%. The MAS ceramic regenerator disks had minimal surface breakdown and cracking. The maximum measured regenerator inlet temperature was 2001°F (1094°C).
- Cold side engine housing injection-molded insulation had minimal debonding from the metal housing. After repair, the hot side engine housing insulation completed over 177 hr of durability testing without additional problems.
- The I-112 (ZnO/SnO₂/CaF₂) coated regenerator right hot seal cross arm had minimal degradation in seal surface function. The left cross arm (NiO/CaF₂ plasma sprayed) was replaced with an I-112 coated cross arm at 64 hr testing and completed the remaining test hours.

Figure 5.2-15 shows the gasifier section components, regenerator disks, and hot seal cross arms after completing the 300-hr durability test. Teardown inspection and measurements showed all components to be in excellent condition. The ceramic inlet scroll assembly and ceramic gasifier rotor (Figure 5.2-16) were in excellent condition with microscopic inspection showing no evidence of any physical or structural damage.

Post-test inspection of other rig components revealed no major problems. The gasifier housing injection-molded insulation was cracked and debonded close to the hot section as shown in Figure 5.2-17. However, it remained in place and was fully operational throughout the 300-hr test. Figure 5.2-18 shows the regenerator disks after test. Only minimal surface breakdown and cracking were observed. The AGT-5 engine housing

Figure 5.2-15. AGT-5 hot gasifier rig components subjected to entire 300-hr cyclic durability test.

Figure 5.2-16. Ceramic gasifier rotor after 300-hr durability test.

Figure 5.2-17. Injection-molded insulation on gasifier housing after 300-hr durability test.

Figure 5.2-18. MAS pasted regenerator disks after 300-hr durability test.

injection-molded insulation after test is shown in Figure 5.2-19. The hot side injection-molded insulation was in excellent shape with only minor areas of delamination or debonding from the housing. Cold side insulation was also in excellent condition with only two areas of debonding from the housing. Both hot seal cross arms showed excellent wear patterns and durability. The cross arms are shown in Figure 5.2-20. The right cross arm is on the top in the photograph and was subject to 236 hr of testing. The left cross arm completed the 300-hr durability test. Figure 5.2-21 shows the injection-molded combustor cover after 300-hr of testing. The cover showed no signs of damage.

Prior to the 1996 HVTE-TS program, a total of 2321 test hours on ceramic rotors and 584 test hours on ceramic scrolls had been accumulated. During 1996, an additional 330.8 test hours were accumulated on both ceramic scrolls and rotors under the HVTE-TS program. Table 5.2-II summarizes this extensive ceramic component testing data base.

Figure 5.2-19. AGT-5 engine housing injection-molded insulation after 300-hr durability test.

Figure 5.2-20. Ceramic-coated hot seal cross arms after 300-hr durability test.

Table 5.2-II.
Gasifier section ceramic component data base.

Ceramic Rotor Data Base

Supplier	Rotor S/N	No. of blades	Rotor material	Hours on test		Peak speed %	Peak RIT °C (°F)
				1996 total	Cumulative total/component		
Kyocera	4-2	20	SN253	300.9	300.9	101	1369 (2497)
Kyocera	3-3	20	SN253	29.9	29.9	100	1375 (2507)
Norton	1538-75	20	NT154	0.0	35.5	90	1150 (2102)
Norton	1541-88	20	NT154	0.0	12.3	95	1150 (2102)
Norton	130X-2	20	NT164	0.0	62.6	95	1150 (2102)
Norton	122X-1	20	NT164	0.0	15.9	95	1200 (2192)
Kyocera	5K9	15	SN252	0.0	12.6	97	1327 (2421)
Kyocera	5K21	20	SN252	0.0	279.8	100	1380 (2515)
Norton	6F	20	NT154	0.0	12.0	100	1180 (2155)
Kyocera	5K27	20	SN252	0.0	0.5	100	1115 (2040)
Kyocera	5K23	20	SN252	0.0	13.1	100	1150 (2100)
Kyocera	5K13	15	SN252	0.0	22.4	100	1380 (2515)
Kyocera	5K12	15	SN252	0.0	229.0	101	1385 (2552)
Norton	5S	20	NT154	0.0	224.7	100	1350 (2460)
Kyocera	5K26	20	SN252	0.0	22.7	100	1408 (2566)
Kyocera	5K10	15	SN252	0.0	0.8	90	1277 (2331)
Carborundum	FX78527	15	a-SiC	0.0	0.0	40	N/A
Kyocera	5K22	20	SN252	0.0	1000.7	98	1395 (2543)
Kyocera	5K25	20	SN252	0.0	3.9	90	1160 (2120)
Kyocera	5K24	20	SN252	0.0	7.9	101	1150 (2102)
Kyocera	5K8	15	SN251	0.0	365.0	100	1150 (2102)
Cumulative Total				330.8	2652.2		

Ceramic Scroll Database

Supplier	Scroll type	Scroll S/N	Scroll material	Hours on test		Peak RIT °C (°F)
				1996 total	Cumulative total/component	
Kyocera	Advanced	34-2	SN252	300.9	300.9	1369 (2497)
Kyocera	Advanced	11-2	SN252	29.9	29.9	1375 (2507)
Kyocera	Standard	KX61234	SN252	0	0.3	1150 (2102)
Norton	Advanced	KX244-18	NT230	0	26.4	1150 (2102)
Norton	Advanced	KX61227	NT230	0	50.8	1150 (2102)
Norton	Advanced	KX61314	NT230	0	4.3	1150 (2102)
Norton	Advanced	KX61200	NT230	0	1.1	1327 (2420)
Kyocera	Advanced	KX59815	SN252	0	267.7	1380 (2515)
Norton	Standard	KX59008	NT230	0	1.5	1050 (1920)
Kyocera	Standard	S-2	SN252	0	13.5	1380 (2515)
Kyocera	Standard	0001-1	SN252	0	196.5	1385 (2525)
Norton	Standard	KX56721	NT230	0	8.9	1350 (2460)
Kyocera	Advanced	KX55599	SN252	0	1.1	930 (1706)
Kyocera	Advanced	KX55836	SN252	0	5.2	1406 (2563)
Kyocera	Advanced	S-1	SN252	0	7.2	1401 (2554)
Cumulative Total				330.8	915.3

Figure 5.2-21. Injection-molded insulation combustor cover after 300-hr durability test.

Appendix A

**1995 Annual Progress Report
Submitted by**

Schuller International

and Prepared for

Allison Engine Company, Inc.

under the

**Hybrid Vehicle Turbine Engine Technology Support Project
(HVTE-TS)**

Contract No. DE-AC02-96EE50453

Note: This appendix contains the annual progress report as submitted to Allison. The document has been edited only where necessary to delete information that is deemed to be proprietary in nature.

**HYBRID VEHICLE TURBINE ENGINE - TECHNOLOGY SUPPORT
(HVTE-TS)**

**ADVANCED TURBINE TECHNOLOGY APPLICATIONS PROJECT
(ATTAP)
NASA CONTRACT DEN3-336**

**ANNUAL PROGRESS REPORT
1995**

INSULATION FOR GAS TURBINE ENGINE

**ALLISON PO# H506058
PO# H416284**

SUBCONTRACTOR:

**MOUNTAIN TECHNICAL CENTER
SCHULLER INTERNATIONAL
BOX 625005
LITTLETON, COLORADO 80162-5005**

PREPARED BY:

PHILLIP C. MARTIN

JANUARY 1996

PREPARED FOR ALLISON ENGINE COMPANY

3.2.2 SCHULLER

Objective/Approach

Efforts at Schuller are aimed at developing an injection moldable insulation capable of low-cost high-volume production for automotive gas turbines. The approach is to modify the insulation material system for improved injection molding properties while developing the injection molding process for both simple and complex engine components. In addition to the development of the molding process, several other developmental items are being addressed including: insulation to metal hardware bond ability, cyclic durability and erosion resistance.

Accomplishments/Results

Molding System Development

- Developed next generation molding process using ringmesh molding forms and cloth lining.
- Modified molding system results in improved mold fill-out and improved surface finish with no "popcorning" as seen in prior injection moldings.
- Utilized the improved molding process to insulate AGT-5 gasifier housings and burner covers for engine builds at Allison.
- Fabricated molding tools for the AGT-5 rotor housing gasifier/burner cavity and the exhaust cavity.
- Utilized the improved molding process to insulate via injection molding, for the first time, AGT-5 rotor housings.
- Utilized the improved molding process to insulate gasifier housing and a hybrid combustor cover.
- Molded test samples for cyclic durability and thermal conductivity using the improved molding system.
- Investigated potentially more efficient fiber cleaning techniques.
- A small bag of wet insulation was frozen, thawed, and a bar sample was hand molded to evaluate the molding characteristics and drying shrinkage.
- Batches of insulation with longer length fibers were prepared to evaluate molding and drying shrinkage characteristics.

Cyclic Durability/Erosion Resistance Testing

- Exposed the insulation to 10500 cycles on the cyclic durability tester at exposure temperatures of >2600°F with no evidence of erosion, but some cracking of the surface of the insulation.
- The cyclic durability testing sample molded using the improved molding process was tested at 2300°F flame temperature at a flame impingement angle of ~45°. The sample has been exposed for >2700 cycles with no evidence of erosion or cracking.

- A mixture of the current insulation (70 percent by volume) and microporous insulation (30 percent by volume) was molded using the improved molding process. The dried insulation appears firm with a tough surface. Samples were molded for both cyclic durability testing and thermal conductivity measurements.
- The cyclic durability testing sample of the 70:30 mixture of turbine:microporous insulation, as noted above, was tested at 1800°F flame temperature with the flame normal to the insulation surface. The sample was exposed for >2350 cycles with no evidence of erosion or cracking.
- Samples of the 70:30 mixture were submitted for thermal conductivity testing in the high temperature calorimeter at hot face temperatures of 1200°F, 1800°F, and 2400°F. (Note: Previous testing of a 50:50 mixture showed significantly lower thermal conductivity when compared with the current insulation, but there was concern about the toughness of the insulation.)
- Exposed the molded turbine:microporous (50:50) sample on the durability/erosion test at 1800°F for 10,000 cycles with no evidence of cracking or erosion.
- Materials and techniques to patch damaged insulation are being evaluated on the cyclic durability test burner.

Materials Characterization

- Measured thermal conductivity of insulation molded using the improved molding system.
- Measured thermal conductivity of alternate insulation materials, including more thermally efficient microporous insulations, molded using the improved molding system.

Discussion

Molding System Development

Attempts to mold alternate insulation materials for internal Schuller projects resulted in the development of modifications to the injection molding system for the turbine engine insulation. This latest molding system development using the ringmesh and cloth lining eliminates the "popcorn" appearance of the previous molding system. The surface appearance of the insulation and the mold fill-out has been significantly improved. Several engine parts

have been successfully insulated and shipped to Allison for engine builds. Molding forms for the AGT-5 engine housing have been fabricated and AGT-5 engine housings have been insulated using the previous molding process and now the improved molding process. The engine housings have been shipped to Allison for engine builds. The latest molding system continues to be developed, including incorporation of the system for all parts including the complex piece engine housing.

A. gasifier housing was injection molded at Allison to demonstrate the current, improved molding process using a ringmesh and cloth lining system. This molding process has been designated Schuller Moldable Insulation Generation 2 or **SMIG2**. The previous molding process, sometimes including the terminology "popcorn" in reference to the surface appearance, has been designated **SMIG1**. Tooling has been fabricated to include the molding pattern as a permanent fixture in the molds. This tooling will be referred to as **SMIG3**. At this time, the most useful technique for molding system development is **SMIG2.5**, in which the ringmesh is attached to the mold with adhesive. The new molding system permits rapid evaluation of the molding

capabilities of current and alternate materials. Samples for cyclic durability/erosion resistance testing have been molded using this improved technique and have been tested on the cyclic durability test burner.

A small bag of wet insulation was frozen, thawed, and a bar sample was hand molded. The molding characteristics and drying shrinkage were unchanged when compared with the original, unfrozen material.

Cleaned fiber has been processed to yield longer length fibers. Batches of insulation with these longer length fibers have been prepared to evaluate molding and drying shrinkage characteristics. The insulation formulated using the longer length fibers had slightly less drying shrinkage and slightly lower density, but was somewhat less moldable. There was some evidence of molding flaws. The conclusion is that the level 600 processed fiber yields the best balance of molding and shrinkage.

Cyclic Durability/Erosion Resistance Testing

The burner system designed to expose the insulation to conditions expected in the operating engine has been used to proof test the insulation. The burner exposes the insulation to a high temperature gas flame with an impingement velocity of about 250 feet per second for 15 seconds every 60 seconds. The exposure is to last for 10,000 cycles or failure, whichever occurs first. The insulation, molded using the previous molding process, SMIG1, has been exposed for 10500 cycles at flame temperatures starting at 1800°F and going up to 2600°F with no evidence of erosion but with some cracking of the insulation surface. A second sample molded using the ringmesh and cloth lining molding process, SMIG2, has been exposed for 10500 cycles at flame temperatures >2600°F with peak temperatures of 2650-2700°F with no evidence of erosion but some evidence of cracking of the insulation surface.

The cyclic durability testing sample molded using the SMIG2 molding process was tested at 2300°F flame temperature at a flame impingement angle of ~45°. The sample was exposed for >2700 cycles with no evidence of erosion or cracking.

A mixture of the current insulation (70 percent by volume) and microporous insulation (30 percent by volume) was molded using the SMIG2.5 molding process. The dried insulation appears firm with a tough surface. Samples were molded for both cyclic durability testing and thermal conductivity measurements. The cyclic durability testing sample of the 70:30 mixture of turbine:microporous insulation was tested at 1800°F flame temperature with the flame normal to the insulation surface. The sample was exposed for ~3200 cycles with no evidence of erosion or cracking.

A sample of turbine:microporous, 15:85 by volume, insulation molded using the SMIG2.5 process has been tested on the cyclic durability test burner at >1800°F (peak temperatures ~2000°F) with flame impingement normal to the surface of the insulation. The sample has been exposed for 2000 cycles with no evidence of erosion. Minor surface cracks formed in the flame impingement area during the first hundred cycles but remained unchanged during subsequent cycles.

A sample of turbine:microporous, 30:70 by volume, insulation with added silicon carbide to increase radiation blocking, was molded using the SMIG2.5 process. The molded sample showed evidence of drying shrinkage and was not tested on the cyclic durability test burner.

Because the SMIG2.5 molding process has been shown to result in a tougher surface, a 50:50 SMIG2.5 sample was tested on the cyclic durability test burner at 1800°F with flame impingement normal to the surface of the insulation. The sample was exposed for 10000 cycles with no evidence of erosion or cracking. The surface of the insulation in the flame impingement area appeared to be harder to the touch than the surface outside the flame impingement area.

The following table summarizes the results of the flat sample cyclic durability testing performed to date.

Flat Sample Cyclic Durability Testing

Sample ID	Composition	Molding Technique	Flame Temp., °F	Cycles Exposed	After Exposure Density, pcf
6449-49	Turbine	Hand Molding SMIG 0	1800°F	10,500 No Failure	20.7
6449-58-1	Turbine	Simulated Injection "SMIG 1"	1800°F	10,500 No Failure	19.0
6449-75-5	Turbine	Injection SMIG 2	1800-2400°F	~5500 No Failure	24.5
6449-88-1	Turbine	Injection SMIG 2	2600-2700°F	10,500 Some cracks	21.8
6449-93-1	70/30 Turbine/ Microporous	Injection SMIG 2.5	1850-1875°F	3,192 No Failure	23.1
6449-93-3	50/50 Turbine/ Microporous	Injection SMIG 2.5	1850-1875°F	2075 No Failure	23.5
6449-94-1	Turbine	Injection SMIG 2	2300°F @ 45° angle	2705 No Failure	21.1
6449-97-1	Microporous	Injection SMIG 2.5	1800-2000°F	20 Cracking Failure	16.4
6449-93-4	30/70 Turbine/ Microporous	Injection SMIG 2.5	1850-1875°F	2020 No Failure	17.9
6449-120	50/50 Turbine/ Microporous	Injection SMIG 2.5	1850-1875°F	10000 No Failure	19.8

A flat sample was intentionally damaged before and after drying and then patched with wet insulation. This sample was mounted on the cyclic durability test burner and the high temperature integrity of the patched material tested. The patch of insulation covering a "before drying" hole blew out at 78 cycles. The test sample was removed from the test burner and repatched with wet insulation using a cloth and ringmesh compress to compact the wet insulation. This new patch was dried and the sample returned to the test burner. The test piece reached 2150 cycles. The repatched area remains intact. The patch of the screwdriver gouged dry insulation blew-out. The patch of the cork borer dry insulation loosened and nearly blew-out. The patch of the screwdriver gouged wet insulation remains intact but sounds hollow when finger tapped. The sample will be removed from the test burner and the damaged areas will be repatched using the cloth and ringmesh compress technique.

Materials Characterization

Samples of the 70:30 mixture were submitted for thermal conductivity testing in the high temperature calorimeter at hot face temperatures of 1200°F, 1800°F, and 2400°F. (Note: Previous testing of a 50:50 mixture showed significantly lower thermal conductivity when compared with the current insulation, but there was concern about the toughness of the insulation.) The results are included in the attached plot of thermal conductivity.

THERMAL CONDUCTIVITY TURBINE AND MICROPOROUS

Plans for Future Work

Molding System Development

Continued development of the improved molding system should allow the insulation to be applied to many of the insulated parts of the turbine engine including the engine housing. This will involve the fabrication of molds for the simple pieces, mold inserts for the complex piece, and mold lining systems for both. The insulated parts will be available for evaluation both in engines and in test rigs.

Cyclic Durability/Erosion Resistance Testing

The cyclic durability test burner provides the capability to evaluate the expected physical performance of the insulation at high temperatures. These performance characteristics include thermal shock resistance and erosion resistance to high temperature, high velocity gas flow. As changes are made to the insulation formulations or to the molding process, the performance of the insulation will be evaluated.

Appendix B

**1996 Annual Progress Report
Submitted by**

Schuller International

and Prepared for

Allison Engine Company, Inc.

under the

**Hybrid Vehicle Turbine Engine Technology Support Project
(HVTE-TS)**

Contract No. DE-AC02-96EE50453

Note: This appendix contains the annual progress report as submitted to Allison. The document has been edited only where necessary to delete information that is deemed to be proprietary in nature.

**HYBRID VEHICLE TURBINE ENGINE - TECHNOLOGY SUPPORT
(HVTE-TS)**

**ADVANCED TURBINE TECHNOLOGY APPLICATIONS PROJECT
(ATTAP)
NASA CONTRACT DEN3-336**

**ANNUAL PROGRESS REPORT
1996**

INSULATION FOR GAS TURBINE ENGINE

**ALLISON PO# H506058
PO# H416284**

SUBCONTRACTOR:

**MOUNTAIN TECHNICAL CENTER
SCHULLER INTERNATIONAL
BOX 625005
LITTLETON, COLORADO 80162-5005**

PREPARED BY:

PHILLIP C. MARTIN

APRIL 1997

PREPARED FOR ALLISON ENGINE COMPANY

3.2.2 SCHULLER

Objective/Approach

Efforts at Schuller are aimed at developing an injection moldable insulation capable of low-cost high-volume production for automotive gas turbines. The approach is to modify the insulation material system for improved injection molding properties while developing the injection molding process for both simple and complex engine components. In addition to the development of the molding process, several other developmental items are being addressed including: insulation to metal hardware bond ability, cyclic durability and erosion resistance.

Accomplishments/Results

Molding System Development

- A new methocellulose which replaced a product that is no longer available has been tested.
- Molding forms have been fabricated for the hybrid engine housing and insulation injection molding of the housing has been completed.
- Insulated an AGT-5 rotor housing with a lower density (~18 pcf) insulation formulation using the SMIG 2.5 molding process.
- Lower cost commercially available deshotted fibers from Thermal Ceramics have been evaluated for use in the insulation

Cyclic Durability/Erosion Resistance Testing

- Cyclic durability testing of patched insulation has been conducted.
- The lowest density (~17 pcf) formulation of the Thermal Ceramics deshotted CERACHROME fiber has been tested for cyclic durability.
- CERACHEM formulated turbine insulation (~18 pcf) has been tested for cyclic durability.

Materials Characterization

- Samples of CERACHROME formulated turbine insulation (~18 pcf) and CERACHEM formulated turbine insulation (~19 pcf) have been tested for thermal conductivity

Discussion

Molding System Development

A new methocellulose has been evaluated as a replacement for the previously used raw material, which is no longer available from the manufacturer. Minor changes in the insulation formulation have been tested to optimize the molding characteristics of the wet moldable insulation. This new material appears to provide performance similar to that of the old material. The new methocellulose will be used in future batches of insulation.

Lower density formulations of the turbine insulation have been achieved using deshotted CERACHROME and CERACHEM bulk fibers from Thermal Ceramics. These fibers are available commercially at lower overall cost than raw fiber which then must be further processed to remove shot. The cleaned fiber available from Thermal Ceramics has been evaluated for its utility in the insulation. Batches of insulation, with different levels of fiber processing performed at MTC, have been molded. The different levels of fiber processing required adjustments in the water to solids

ratio in each formulation in order to obtain moldable material. The different water to solids ratios yield dry densities ranging from ~17 pcf to ~25 pcf. The insulation resulting from all molded batches appears usable. The use of this fiber will result in significant cost and processing time reductions. Cyclic durability and thermal conductivity testing have been performed on the lower density formulations to determine the lowest usable density. Moldability of the insulation was excellent and drying shrinkage was acceptable.

Thermal Ceramics deshotted CERACHEM fiber has been evaluated for use in the turbine insulation. CERACHEM is less expensive than the presently used CERACHROME fiber. The normal service temperature limit (in fiber blanket applications) for CERACHEM is only slightly less than that for CERACHROME. In the turbine insulation form, the CERACHEM may perform as well as CERACHROME.

Cyclic Durability/Erosion Resistance Testing

A flat sample of molded insulation was intentionally damaged before and after drying and then patched with wet insulation. This sample was mounted on the cyclic durability test burner and the high temperature integrity of the patched material tested. The patch of insulation covering a "before drying" hole blew out at 78 cycles. The test sample was removed from the test burner and repatched with wet insulation using a cloth and ringmesh compress to compact the wet insulation. This new patch was dried and the sample returned to the test burner. The test piece reached 2150 cycles. The repatched area remains intact. The patch of the screwdriver gouged dry insulation blew-out. The patch of the cork borer dry insulation loosened and nearly blew-out. The patch of the screwdriver gouged wet insulation remains intact but sounds hollow when finger tapped. Patches applied using a ringmesh and cloth compress followed by "sealing" the edge of the patch with colloidal silica appear to function well. Molded, patched insulation has been tested on the cyclic durability test burner. Four patches remained in-place after exposure counts ranging from 5280 to 14960 cycles.

A piece of the lowest density (~17 pcf) formulation using Thermal Ceramics deshotted CERACHROME fiber has been tested for cyclic durability. The sample has been exposed to 10,300 cycles at >1800°F. There is no evidence of cracking or erosion. This sample is the best of all the samples tested to date. Since the cyclic durability sample exceeded 10,000 cycles without failure, additional samples have been molded and submitted for thermal conductivity testing.

A piece of low density (~18 pcf) formulation using Thermal Ceramics CERACHEM fiber has been tested for cyclic durability. The sample has been exposed to >12,000 cycles at >1800°F. There is no evidence of cracking or erosion. Since the cyclic durability sample exceeded 10,000 cycles without failure, additional samples have been molded and submitted for thermal conductivity testing.

Materials Characterization

Samples of the CERACHROME formulated turbine insulation (~18 pcf) have been tested for thermal conductivity. The thermal conductivity of this material is shown in the attached plot, along with previous test results for the turbine insulation at 23 pcf. The thermal conductivity is essentially the same at mean temperatures <1500°F. At higher mean temperatures the higher density may provide some additional radiation blocking and therefor slightly improved thermal conductivity. The insulation at 18 pcf will provide performance, equivalent to the performance of the insulation at 23 pcf, at the operating temperatures of the turbine engine.

Samples of the CERACHEM formulated turbine insulation at ~19 pcf have been tested for thermal conductivity. The results are shown in the attached plot which includes the above reported results for CERACHROME at ~18 pcf. The thermal conductivities of the CERACHEM and the CERACHROME samples are nearly identical over the entire mean temperature range of the test.

TURBINE INSULATION COMPARISON

Appendix C

**1995 Annual Progress Report
Submitted by**

Corning Incorporated

and Prepared for

Allison Engine Company, Inc.

under the

**Hybrid Vehicle Turbine Engine Technology Support Project
(HVTE-TS)**

Contract No. DE-AC02-96EE50453

Note: This appendix contains the annual progress report as submitted to Allison. The document has been edited only where necessary to delete information that is deemed to be proprietary in nature.

Corning Incorporated
Corning, New York 14831

**Extruded Gas Turbine
Regenerator Development**

1995
Annual Report

February 29, 1996

to-
Allison

from-
L.M. Holleran
Corning Incorporated

**Extruded Rotary Gas Turbine Heat Exchanger
Hybrid Vehicle Turbine Engine - Technology Support Program
Corning, Incorporated
By L.M. Holleran**

1. INTRODUCTION

The technical direction for Corning's extruded regenerator program through 1995 remained the development of a ceramic regenerator that would combine the excellent heat transfer and thermal durability properties of Corning's Cercor[®] product with the low cost and high volume of the extrusion process. The replacement of the ATTAP Program with the HVTE-TS (Hybrid Vehicle Turbine Engine - Technology Support) Program in 1994 had shifted the development focus from automotive prime mover units to auxiliary power units for hybrid electric drives⁽¹⁾. The specific objective of this program has been to establish both the materials and process (including the hardware) required to manufacture an extruded gas turbine regenerator capable of at least 1050°C operation. A proposal outlining a four year (1995 - 1998) HVTE-TS development program that would result in the demonstration of production feasibility was submitted to AMPS and approved with a contract for development through 1998 now in place.

In 1994, the two materials areas selected for further development evaluation (MAS and LAS) were identified along with the issues associated with each. The first LFA extrusions of high cell density (170 cells/sq cm) regenerator disk blanks - using the "pull-rod" die - were accomplished with several finished MAS-2 parts provided to Allison for engine rig testing. A new die fabrication process developed for Corning's Celcor[®] extrusion process was also selected as the primary direction for the next generation LFA and later VLFA (very large frontal area, ~30cm dia) dies.

Technical Program - 1995

The original scope of Corning's 1995 program was to establish lab capability to produce full sized disks through a new LFA die and do the critical material testing to determine if we can meet the basic performance requirements with the two remaining candidate materials - LAS and MAS. Specific strategy included:

- Work with Allison to define product performance and design requirements.
- Determine how to best batch the LAS compositions to minimize total cost.
- Determine what can be done to modify "standard" MAS compositions for extruded regenerator performance.
- Demonstrate in the lab we can extrude, dry and fire LFA size blanks of LAS and MAS batches.
- Build smaller test dies and run trial extrusions to select the best final design parameters for a second LFA die using the new die technology.
- Begin a process design and subsequent cost analysis for extruded regenerator fabrication.
- Supply extruded samples as requested by Allison Mobile Power Systems (AMPS).

2. MATERIALS

2.1 MAS Materials

While the MAS materials have the major advantages associated with over 20 years of automotive component production experience, early bench testing of the MAS-2 candidate at AMPS indicated that thermal cycling resulted in a significant loss of strength. Recognizing that the low thermal expansion of this candidate is due in large part to controlled micro-cracking, it was theorized that when these micro-cracks were highly stressed they could grow and result in strength loss. Thus to improve thermal cycle performance, a reduction in micro-cracking became the key materials development objective for MAS in 1995.

Because the current MAS material is the lowest expansion candidate in this system, the opportunity existed to trade off some expansion for a higher strength by decreasing micro-cracking. Once samples were available, testing would be done to measure any improvement in thermal cycle performance. Selected candidates would be further evaluated to determine if the higher original strength would sustain matrix mechanical integrity under conditions expected when full sized disks undergo engine rig testing (e.g. high thermal gradients).

A first series of batch compositions were designed using a computer model to predict resultant property data. These results demonstrated the degree of trade off of expansion for strength that was necessary and the range available in the current MAS system. A second approach to achieving an improved MAS composition was based on the discovery of a new composition area that was made as part of a separate Corning development program. A comparison of the relationship between expansion and strength, which is a good indicator of degree of micro-cracking, for these two approaches has been illustrated in Figure 1 (Experiment 8 are the original modifications; Experiment 9 are the new composition batches).

To measure if any improvement in thermal cycle performance had been obtained, rods of these new MAS materials were extruded and tested in the lab. The key test is a thermal cycle test consisting of a 30 minute soak at 1260°C followed by 30 minutes under forced air cooling at room temperature - the transfer between ambient and oven is accomplished in approximately 15 seconds. Data obtained from this experiment did show a significant improvement in performance had been achieved as measured as a lower change in length versus MAS-2 shown in Figure 2 for two of the better higher strength candidates.

As part of a separate internal program at Corning, thermal shock testing of extruded matrix samples has been underway. In this test, samples are mounted in the path of a high volume air stream with thermal shock "aging" accomplished over a wide range of temperatures. After cycling, samples were measured for strength. Using this technique, identical samples of MAS-2 and one of the new, higher strength candidates (strength 140% of MAS-2; expansion approximately 2X) were tested. Test results led to the conclusion that, although the new candidate material has higher initial strength, after thermal shock cycling, both samples retain about the same strength. It was also noted that the MAS-2 material was stronger than the higher strength candidate at temperatures in the range of use for the regenerator product.

Based on these test results, further material development in the MAS area was put on hold until MAS-2 has received full engine rig evaluation at AMPS. A number of LFA disks were provided for this purpose.

2.2 LAS Materials

A number of significant issues had been identified in 1994 for the LAS materials. These included the high material cost (glass batch), the high (15 to 20%) firing shrinkage, which resulted in distortion and cracking, and the problems encountered in earlier attempts to extrude this non-plastic batch. Several new directions in composition development were pursued that would address these areas.

Modification of the LAS-1, the original wrapped regenerator core material, started with changes in the nature of materials in the batch while maintaining the original oxide composition. The initial objective was to reduce firing shrinkage. With this approach shrinkages meeting target values of <10% were obtained but at the expense of low strength and high porosity.

A second series of modifications was made to develop a lower cost LAS batch. Lower cost raw materials were used for both the glass and in the formulation of the extrusion batch. The strength (>30 MPa), thermal expansion (<6 ppm/°C) and shrinkage targets were met but porosity remained higher than the <25% target. Additionally, targeted low firing shrinkage was only obtained with porosity levels greater than 35%.

A parallel effort to invent new, lower cost glass and mineral batches had also been pursued. A unique glass plus mineral composition area was identified with the invention of a unique glass composition. This material approach offered the opportunity to reduce batch cost to <20% of LAS-1, lower firing shrinkage to <10%, and provide improved processing. Several batches were designed and rod samples extruded to provide property data. Significant improvements in physical properties were later achieved with batch material modifications that yielded properties close to our target values with firing shrinkage measured at <10%. An extensive series of sintering experiments was next carried out on a selected composition, later designated as LAS-3, to develop an acceptable firing cycle (see Table 1 for property data).

Based on the property and test data as summarized in Table 1, LAS-3 was selected as an interim matrix material candidate for evaluation by AMPS. Materials development work in 1996 will focus on modifications of this batch candidate to improve durability and reduce thermal expansion. Further firing cycle development will also be done to improve thermal stability.

2.3 Material Candidate Testing

Other than standard physical property testing and analytical measurements (done to confirm the crystalline phases and oxides amounts present), lab testing consisted of setting up equipment and procedures and measurement of candidate thermal and chemical durability.

2.3.1 Thermal Durability

Two tests were run, Thermal Cycling (as described above) and Thermal Stability in which rod samples were held for up to five weeks at 1050°C. Thermal cycle test results have been summarized in Figure 2 for MAS and in Figure 3 for LAS. (Notes: The lower temperature data - 1150°C vs. 1260°C - was obtained to better reflect the maximum use temperature expected in service. LAS-3 is designated as candidate #80 in this and later figures and tables.) Thermal Stability test results for LAS candidates has been summarized in Figure 4.

It was concluded that the MAS-2 material has a significant increase in length with Thermal Cycling which is most likely due to the high degree of micro-cracking known to be present in the fired body. This lack of stability is a concern for thermal cycle failure and for disk failure under long term operational temperature gradients. The LAS-3 material showed a decrease in length on both tests although the total length change (Table 1) is close to the target values set for these tests. This decrease in length may be due to the continued sintering of the microstructure. As a result, additional sintering experiments will be run and the improvement in LAS-3 densification made an objective of further material development.

2.3.2 Chemical Durability

The objective of lab chemical durability testing in 1995 was to demonstrate the relative durability of the new candidate matrix batches to Celcor® wrapped core materials (Code 9455 and Code 9460). Initial testing was done on the wrapped materials to establish the efficacy of these tests and to serve as a standard for the evaluation of our new candidates. Testing of candidates started in late 1995. Priority was given to testing LAS-3, the selected LAS extrusion batch, as chemical durability was a major concern for this composition area. (MAS-2 testing is scheduled for 1996.)

Test procedures were setup to provide results similar to those obtained with testing done back in the mid seventies on Code 9455 and Code 9460 wrapped materials. After several test runs, a salt corrosion test was developed that measured the resultant change in length of samples after boiling in 3% NaNO_3 solution followed by a minimum 12 hr drying at 140°C and soaking at 800°C for two weeks. An unexpected result was obtained on the first set of LAS-3 samples as listed in Table 1, i.e. the length change of LAS-3 was significantly less than that obtained with Code 9455. (This testing will be repeated in 1996.)

In the seventies, the resistance of matrix materials to sulphur contamination had been tested by exposing samples at $\sim 660^\circ\text{F}$ to a reactive gas (SO_2 + air + moisture) that has passed over a catalyst (platinum). The sulphur vapor test, however, yielded results much less severe than expected and as a result a second test rig is being designed. A faster screening test for sulphur durability involving boiling in sulphuric acid - which had also been previously employed to test regenerator materials - was then set up and used.

In the sulphur boil test samples were boiled in four different concentrations of acid to yield four different reaction temperatures: 111°C , 127.2°C , 143.8°C and 174.2°C . Data analysis was based on the formation of ion exchanged phase and an Arrhenius plot is used to show the relationship of reaction rates to reaction temperature. Results have been included in Figure 4 for both 400 cell/in² LAS-3 matrix material and a set of >1000 cell/in² LAS-3 matrix samples and for Code 9455 extruded matrix (400 cell/in²). The Code 9455 wrapped matrix data was measured back in 1977. Conclusions: these results show a consistency in the reaction mechanism between the current test and the 1977 test; these results also show a consistency in the reaction mechanism between the UTE candidate and Code 9455; the LAS-3 appears to be 3 to 4X more reactive than Code 9455 in the current test; we have a good screening test for our extruded LAS candidate materials.

3. PROCESS TECHNOLOGY

3.1 Extrusion

Progress in the process area was highlighted in 1995 by the first successful extrusions of LAS batch compositions through the existing "pull-rod" die. Extrusion of a high glass content batch, designated as LAS-2, had been unsuccessful in 1994 trials. Modification of batch additives and rheology together with a redesign of the extrusion hardware were made to achieve acceptable results. (It should be noted that all extrusions through the pull-rod die require the use of low

viscosity batches. The resultant matrix materials have been characterized by a high number of distorted cell walls and a higher porosity than measured on other extruded samples.)

In December, the first LFA extrusion of LAS-3 was made and was considered successful. Major defect was poor skin quality.

Extrusions of MAS-2 were also completed in 1995 to provide LFA regenerator sample disks for AMPS evaluation.

3.2 Drying

To provide a base for process development in 1996, a lab rotary dielectric dryer was obtained and installed. LFA LAS-3 blanks up to 14" in length were successfully dried.

3.3 Firing

Once large green disk blanks were available, trials were made to determine if these disks could be successfully fired. Initial firings of the LAS-2 resulted in an acceptable level of distortion but significant cracking. A series of nine experimental firings were then made to examine a number of variables believed to effect cracking. A method to successfully fire these LAS-2 blanks was demonstrated.

Firing of LAS-3 blanks will begin in 1996. LFA MAS-2 blanks were fired without any significant problem.

3.4 Process Map

A detailed process mapping was initiated in late 1995. The results of this analysis will be to identify the potential final production process and any interim process staging (e.g. pilot line operation). As part of this study, the technical or engineering areas that must be addressed as part of the development process will be clarified.

4. DIE FABRICATION

As previously reported⁽¹⁾, a new die fabrication process has been developed for Corning's automotive substrate business. In the past year, it was a goal of this program to select the best alternative design, based on this new technology, for the scale up and building of an LFA die.

To determine the best design approach for the building of new LFA and VLFA regenerator extrusion dies, test die were fabricated of two selected alternatives. These test die were scaled to 10X of what would be the final design parameters and were sized to allow for the extrusion of an approximate 2.5" square matrix. A series of lab extrusions using standard Corning Celcor® batch material was made to evaluate material flow, pressure drop and knitting. Extrusions were made over a wide range of batch viscosity and extrusion conditions. Based on these results, a preferred design was chosen and a small extrusion die - known as a dielet - was designed. This dielet was designed to yield a finished matrix extrusion with >750 cells/cm² and <0.15 mm cell wall thickness.

Fabrication of the dielet was not completed in 1995 due to several delays. First, a delay in the approval of contract funding for the balance of 1995 resulted in a six week late start of the build. Later, changes in the design were required to reflect improvements based on experiments done on similar automotive substrate dies. Finally, a major problem was encountered in drilling the ~400 holes needed in the dielet. Although the hole drilling is a process that has been in use for years to produce extrusion die with older die technology, the first two die blocks drilled were unacceptable due to non uniformity of the holes. The decision was made in late 1995 to complete the dielet fabrication with the second block (~25 of the 400 holes were questionable) and attempt extrusion. A third block hole drilling was also started.

It had been originally planned to build the LFA regenerator die using this new technology in 1995 but this objective was also delayed into 1996 by HVTE-TS Program funding cuts. The preliminary design of the new LFA die was completed.

5. FINISHING

While many years of experience exist in the finishing LAS type materials for the wrapped regenerator disk product and Corning has some related experience with its MAS extruded product, the finishing of large extruded ceramic blanks represents a significant portion of the estimated total regenerator disk cost. The reduction in 1995 program funding and the high priority given to the

matrix material and die development resulted in minimum progress in finishing development. Demonstration of high volume, low cost finishing materials, processes and equipment must be accomplished to meet our 1998 HVTE-TS objectives.

One specific area that was pursued in 1995 - in conjunction with AMPS - was the development of a lower manufacturing cost regenerator hub. This hub concept would result in fewer finishing process steps and lower component cost. A hub design was made in conjunction with AMPS. A unique hub extrusion die was designed and fabricated and then used to extrude the new hub. Prototype hubs were extruded, dried and fired. After several trials, a process for insertion, sealing and co-firing of MAS hubs into the MAS-2 matrix was demonstrated. Preliminary samples were provided to AMPS for evaluation which will be done in 1996.

6. SAMPLES PROVIDED FOR EVALUATION

The following extruded regenerator samples were provided to AMPS for evaluation during 1995.

<u>Date</u>		
2/95	1	9.106"D MAS-2 Disk
	8	3" X 2.4" X 1.55" CTR Samples (MAS-2)
4/95	8	3" X 2.4" X 1.55" CTR Samples (LAS-2)
6/95	108	MAS-2 Seal Test Samples (to ANL)
7/95	2	LAS-2 6"D Seal Test Samples (to ANL)
	1	MAS-2 6"D Seal Test Sample (to ANL)
	1	Code 9455 Seal Test Sample (to ANL)
8/95	1	LAS-2 6"D Seal Test Samples (to ANL)
10/95	2	9.5"D MAS-2 Disks
12/95	4	3" X 2.4" X 1.55" CTR Samples (LAS-3)

7. PRODUCT TESTING

Other than the durability testing documented earlier in this report, and the standard QC testing done at Corning's Erwin Facility on finished disks, product performance testing was carried out on extruded matrix samples provided by Corning to AMPS and Argonne National Laboratory (ANL).

7.1 Erwin QC Testing

Quality Control testing is limited to the testing of leakage, pressure drop and expansion mismatch of matrix to hub.

7.1.1 Leakage

Leakage was measured using a standard test developed by Corning for wrapped regenerator disks. The range obtained with Corning's Cercor[®] product is 3 to 9 X 10⁻⁵ lb/sec/in² @20psi. Typical values obtained on the MAS-2 disks are listed in Table 1.

At AMPS' request, a comparison test was run on an MAS-2 disk using two different gasket widths. The following results were reported:

Standard Width (1.5")	Ave = 2.7, Range = 2.0 to 3.6 X 10 ⁻⁵
Narrow Width (0.35")	Ave = 4.4, Range = 2.4 to 5.6 X 10 ⁻⁵

7.1.2 Pressure Drop

Using another standard Cercor[®] test, the pressure drop at mid radius of finished disks is measured with a target of 9 or less inches of Kerosene. Results for MAS-2 disks were between 7 and 8".

7.1.3 Expansion Mismatch to HUB

The expansion mismatch of matrix material to the hub material was of particular concern for the MAS-2 disks as the hub used was of an LAS composition that was developed to match Code 9455 wrapped regenerator disks. The current specification for wrapped Cercor[®] disks is to be within +200 and -175ppm. Finished MAS-2 disks tested at +65 and -0ppm, well within the target range.

7.2 AMPS Product Testing

Testing at AMPS was done on small rectangular samples in their Cyclic Thermal Rig. In this test the samples are mounted in a hot gas flow and cycled over a range that simulates up to 10K of the most severe temperature cycles expected in service. Both MAS-2 and LAS-2 were tested. Results for MAS-2 have been included in Table 1. Neither candidate met the objective of a 200 psi retained hot face MOR. The low results for MAS-2 were discussed earlier; the low initial strength of LAS-2 was a cause of its poor performance (~40psi retained strength). Post mortem done on samples returned to Corning confirmed the MOR results obtained at AMPS.

Engine rig testing was started in 1995. Two MAS-2 LFA disks survived 140 of testing with no measurable degradation.

7.3 ANL Seal Testing

Seal wear test samples were provided to ANL. These were run against two different seal materials. Based on their initial test results, ANL concluded that acceptable performance was demonstrated by MAS-2 to 1600°F and by LAS-2 to 1200°F (vs. 1800°F for Code 9455).

A sample of LAS-2 that performed poorly in ANL's wear test was checked by x-ray diffraction and porosity measurement. The correct LAS fired phase was present but porosity was slightly higher than expected.

8. MANAGEMENT AND REPORTS

An SOW contract proposal for the HVTE-TS program to cover the years 1995 through 1998 was prepared and submitted to AMPS in February. This proposal was approved and is the basis for the current purchase order Corning is working under. Progress versus proposal schedule (as modified to reflect reduced funding) was tracked as summarized in Figure 5.

During 1995, a total of four face-to-face program reviews were held with AMPS, three at AMPS and one in Corning. In addition, several conference calls were made to include three with both ANL and AMPS.

Monthly and weekly written status reports were submitted.

At AMPS request, a paper⁽²⁾ was presented at the CCM meeting on 9/26/95 summarizing extruded regenerator program for HVTE-TS.

One U.S. patent⁽³⁾ was awarded to Corning for an LAS composition invention.

9. REFERENCES

1. "Extruded Rotary Gas Turbine Heat Exchanger, 1994 Annual Report", J.P. Day, 5/9/95.
2. "Extruded Regenerator Development", Paper presented at the Annual Automotive Technology Development Contractors' Coordination Meeting, 10/26/95.
3. U.S. Patent Number 5,403,787.

STATUS OF MATERIAL DEVELOPMENT

	<u>CODE 9455</u>		<u>MAS-2</u>	<u>LAS-3</u>	<u>TARGET</u>
STRENGTH					
RODS (MOR)		7000	4000	8800	
MATRIX (CRUSH)		4000	3600	-----	> 4400 psi
MATRIX (AXIAL,MOR)		1050	500	1300	
CTE					
RT - 800, ppm		0.4	0.33	0.6	< 0.5
POROSITY		<10%	23%	24%	< 25%
SHRINKAGE		15%	6 - 7%	7 - 9%	< 10%
THERMAL STABILITY @ 1050 / 5 WEEKS		- 125	----	- 390	< (300)ppm / 5 WEEKS
THERMAL CYCLE (100 CYCLES)	1260C 1150C	---- - 25	3800 ----	- 3000 - 400	< [300]ppm < [300]ppm
CYCLIC THERMAL (STRENGTH HOT FACE)	10000 1000	328 ---	< 25 < 25	---- ----	> 200 psi > 200 psi
WEAR TEST vs SEALS (OK USE TEMP F)		1800	1600	-----	TBD
LEAKAGE TEST		3 - 9	1.4 - 4	----	< 3 X 10-5
CHEM DURABILITY					
SALT (PPM, 1WK)		568	----	216	< [300]ppm
SULPHUR (BOIL TEST)		X	DISSOLVES	~4X	TBD

Table 1

Figure 1
MAS Composition - Trade Off Of CTE For MOR

Thermal Cycle Test (200 Cycles), MAS Candidates

Figure 2

Figure 3
Effect Of Composition And Thermal Cycling
Temperature On Dimensional Stability
(LAS Candidates)

Figure 4
Sulfuric Acid Boil Test Results.
Arrhenius Plot Of Formation Of Ion-Exchanged
Phase Vs. Reciprocal Temperature

Figure 5
HVTE-TS Extruded Gas Turbine Regenerator Program
Estimated Time Line 1995 - 1998 (Revised 9/20/95)

Appendix D

**1996 Annual Progress Report
Submitted by**

Corning Incorporated

and Prepared for

Allison Engine Company, Inc.

under the

**Hybrid Vehicle Turbine Engine Technology Support Project
(HVTE-TS)**

Contract No. DE-AC02-96EE50453

Note: This appendix contains the annual progress report as submitted to Allison. The document has been edited only where necessary to delete information that is deemed to be proprietary in nature.

**Extruded Gas Turbine
Regenerator Development**

**1996
Annual Report**

February 28, 1997

**To: Allison Mobile Power Systems
Allison Engine Company**

**From: L. M. Holleran
Corning Incorporated**

EXTRUDED ROTARY GAS TURBINE HEAT EXCHANGER HYBRID VEHICLE TURBINE ENGINE - TECHNOLOGY SUPPORT PROGRAM CORNING, INCORPORATED

1. INTRODUCTION

The objectives established for Corning's extruded regenerator program in 1996, under the Hybrid Vehicle Turbine Engine - Technology Support (HVTE-TS) Program, built upon the technical progress made in 1995⁽¹⁾. The program strategy remained the development of a ceramic regenerator for automotive gas turbines that would combine the excellent heat transfer and thermal durability properties of Corning's Celcor[®] product with the low cost and high volume of the extrusion process. The specific task of this program has been to establish both the materials and process (including the hardware) required to manufacture an extruded gas turbine regenerator capable of at least 1050°C operation. A proposal outlining a four year (1995 - 1998) HVTE-TS development program⁽²⁾ that would result in the demonstration of production feasibility was originally submitted to AMPS (Allison Mobile Power Systems) and approved with a contract for development that has been funded in both 1995 and 1996. (Corning is subcontracted to AMPS for this development work.)

The scope of Corning's 1995 program was to establish lab capability to produce full sized disks and do the critical material testing to determine if we can meet the basic performance requirements with the two remaining candidate materials, magnesium-alumina-silicate (MAS; cordierite) and lithium-alumina-silicate (LAS, beta-spodumene). Key accomplishments reported for 1995 included⁽²⁾:

- first LFA (large frontal area; e.g. >9"D) extrusion of an LAS batch (LAS-2)
- invention of a new LAS material family and the selection of the LAS-3 candidate for full matrix evaluation
- selection of MAS-2 as the primary MAS candidate for full engine rig evaluation
- successful 10X extrusion trials of alternatives and the selection of a design for a new LFA die
- first LFA extrusion and rotary drying of LAS-3 matrix batch
- design and demonstration of a low cost hub concept
- first 9.5"D finished disks supplied to AMPS (Allison Mobile Power Systems) for testing
- samples provided to ANL (Argonne National Laboratory) and first testing for seal compatibility.

The major development objectives established by Corning and AMPS for 1996 were:

- optimization of the LAS material composition and select the specific matrix candidate for LFA disk testing by AMPS
- develop the LFA lab process for LAS
- build a LFA extrusion die based on Corning's new technology (note this activity funded by Corning outside the HVTE-TS Contract; this was due, in part, to the reduction of contract funding).
- develop a lower cost hub technology (specifically the pressed-in-hub or PIH) and deliver LFA prototypes to AMPS
- fabricate and deliver the first LFA LAS regenerator disks to AMPS.

This report summarizes the 1996 results of Corning's extruded regenerator development program.

2. PROGRAM STATUS VERSUS OBJECTIVES - GENERAL

In 1996, funding for this program was significantly reduced for the second straight year. As a result, not all of the 1996 tasks as outlined in the original statement of work^(b) (SOW) were accomplished. To maintain acceptable progress on the development of LFA die technology, Corning made the decision to fund the LFA die task outside the HVTE-TS Contract. Available contract funds were then used for the material, process and finishing tasks.

This report documents the progress made in 1996 at Corning. A summary is outlined in Table 1. An updated timeline schedule is found in Figure 1. Key task status/accomplishments in 1996 included:

- the LAS-4 material candidate was developed and chosen for full evaluation by AMPS based on extensive lab evaluation
- because of cracking problems when fired, we were unable to deliver LFA LAS-4 disks to AMPS
- LAS matrix samples passed 3000 cycles on AMPS CTR test and were judged acceptable for seal compatibility by ANL
- rotary dielectric drying of LFA blanks was demonstrated
- a new, larger extrusion press was purchased which is expected to do a better job with the LFA sized extrusions (Corning funded outside HVTE-TS Contract)
- two dielets built and evaluated to select the best design for the new LFA die

- first LFA die fabrication trial failed and a second build was underway at the end of the year
- five LFA lab processing trials were run with good results
- four MAS-2 9.5"D disks were finished and demonstrated we could meet dimensional and surface finish requirements
- sufficient samples were provided to AMPS to keep their regenerator evaluation program on schedule.

3. MATERIALS

3.1 MAS

No material development activity was planned for the MAS materials in 1996 other than the continued evaluation of MAS-2 as a candidate for the extruded regenerator matrix material. Some follow-up testing for salt durability was done with results included in Table 2. MAS-2 was also used as the batch material for many LFA processing trials and for the development of the PIH as the LAS batch had not been selected. Samples of MAS-2 were provided to ANL to serve as a base extruded matrix sample for their seal development program.

3.2 LAS

A major milestone in the extruded regenerator matrix materials task was achieved in 1996 with the development of a primary LAS candidate - known as LAS-4. A backup candidate (lower CTE) was also identified. Composition candidates based on the originally selected glass-mineral batch (LAS-3)⁽¹⁾ were designed and evaluated with a primary objective to reduce the expansion of this material to ≤ 0.5 ppm at 1000°C while maintaining other key properties. Two phases of composition work were done. First a screening or mapping of the possible changes to LAS-3 were batched and tested. Based on these results, five new extrusion batches along with three repeat candidates were processed into rods and 1"D matrix samples and fired. Samples were then fired to three different peak temperatures using the sintering cycle developed in 1995 for LAS-3. These samples were then submitted for physical properties testing. With the completion of matrix physical property measurements, initial thermal cycling and chemical durability (see below), the LAS-4 candidate was shown to have met the original targets for its development, i.e. a thermal expansion < 5 ppm at 1000°C with no significant loss of strength or other key properties versus LAS-3 (see Table 3 below).

TABLE 3. Physical Property Data for LAS Candidates

Property	Target	Wrapped 9455	LAS-3	"LAS-4"	Backup	Remark
CTE (ppm)	≤ 0.5	~ 0.4	0.8	0.4-0.5	< 0.4	1000°C, cool
MOR (psi)	> 4400	7000	8800	7800	7000	rods
Porosity(%)	< 25	< 25	< 10	24	19-27	26-31

The range in properties listed was a result of the three different peak firing temperatures. The backup was chosen for its lower CTE, good MOR - but its higher porosity made it less attractive.

To obtain property data on a high cell density matrix, an extrusion of the LAS-4 candidate batch through one of the test dielets (see Section 4) was completed and samples fired. Physical property measurements were made on these matrix samples with the following results.

TABLE 4. LAS-4 High Cell Density Matrix Physical Properties

Axial MOR	1518 psi
CTE, 1000°C	4.8 ppm
Porosity	29%

Selection of an optimum extrusion batch and firing cycle may alter these numbers in the final version of LAS-4. Property data covering a number of later measurements is included in the summary table of candidate performance data (Table 2). By the end of 1996, green LFA blanks of LAS-4 had been successfully fabricated in the lab but attempts to fire the large disks had resulted in severe cracking.

A patent application was filed for the LAS-3/LAS-4 material invention.

3.3 Lab Testing

3.3.1 Chemical Durability

Both salt and sulphur durability were measured on the various MAS and LAS candidate materials.

Salt Durability

Length changes were measured on extruded matrix samples after salt exposure and high temperature aging to determine candidate material reaction. After initial length measurement, samples were chemically treated in a 3% solution of boiling NaNO_3 for 5 minutes. Samples were then dried at 140°C and their length re-measured. These samples were then dried at 800°C for 30 minutes and again re-measured. The samples were then placed back in a 800°C oven and left for one week (168 hours). They were removed and re-measured. The samples were place back in the oven at 800°C for an additional 168 hours heat treatment at 800°C. The change in length after thermal soaking for LAS-4 averaged 172 ppm after two weeks. Results compare favorably with those obtained on Code 9455 (599 ppm) and LAS-3 (216 - 320 ppm).

Sulphur Durability

A set of matrix candidates (400 cpsi) LAS-4 candidate and the backup candidate matrix samples were tested to determine their relative sulfuric acid durability as compared to earlier LAS materials. In this screening test samples were boiled in four different concentrations of acid to yield four different reaction temperatures: 111°C, 127.2°C, 143.8°C and 174.2°C. Data analysis was based on the formation of ion exchanged phase and an Arrhenius plot is used to show the relationship of reaction rates to reaction temperature.

Results have been plotted in Figure 2 along with data obtained earlier for LAS-3, Code 9455, and Code 9455 wrapped matrix data that was measured back in 1977. (Note: movement left is toward greater durability, right toward lower.)

Conclusions: these results showed a consistency in the reaction mechanism between the current test and all earlier tests; these results also show a consistency in the reaction mechanism between the LAS-4 candidate (and backup), Code 9455 and LAS-3. The LAS-4 (and backup) appears to be 3 to 4X more reactive than Code 9455 but is less reactive than LAS-3. The difference between the older 1970's data for Code 9455 and more recent testing is most likely due to differences in the testing itself - although the slopes of the lines are the same confirming a similar reaction mechanism.

3.3.2 Thermal Durability

A full Thermal Stability Test was completed on LAS-4 rod and matrix samples and the LAS-4 backup candidate (full test is 100 cycles, RT to 1150°C, hold 30 min, cool to RT, one cycle = 60 min). As with the LAS-3 material, there was continued shrinkage of the samples when cycled versus the expansion or sample length growth that had been measured in 1995 on MAS samples (expansion believed due to microcracking). Results have been plotted in Figure 3. Total shrinkage measured was greater than the target value of < 300 ppm and what had been measured earlier for LAS-3. Rod and high cell density matrix results were consistent. It was noted that a major portion of the shrinkage occurs during the first 20 cycles.

Effect of Sintering Temperature

LAS-4 rods fired to four different peak sintering temperatures were also tested. The four temperatures were the "standard" temperature used to fire LAS-3 matrix samples and +10°C, -20°C, and -35°C versus this standard. Shrinkage results have been plotted in Figure 4. The decrease in shrinkage with lower peak temperature was unexpected. While we have theorized a potential mechanism to explain this decrease, we have no hard data as yet.

Reduction in Shrinkage

A post firing process step was invented that was shown to significantly reduce the shrinkage that results from the Thermal Cycle Test. An experimental run that used this new treatment was made with the Thermal Cycle Test results plotted in Figure 4. This new firing cycle resulted in a decrease in the total shrinkage of both LAS-3 and LAS-4 of ~50% - thus meeting the target value of < 300 ppm. It was also noted that the difference in shrinkage between LAS-3 and LAS-4 was eliminated. Further testing to determine the possible negative impact of this process change must still be done in 1997 along with work to optimize this treatment.

4. LARGE FRONTAL AREA DIE FABRICATION

Excellent progress was made toward the fabrication of a new LFA die to replace the pull-rod die in use since 1994. When available, the new die is expected to provide a significantly wider process window for the extrusion process - specifically allowing for faster extrusion speeds and the use of stiffer batch materials. This in turn will result in improved blank skin quality and lower cell defects.

4.1 Test Dielets

Two test dielets were fabricated and trials run with each to demonstrate the design chosen for the new LFA die could be fabricated (at least in a small die) and the die would work - i.e. successfully extruded >1100 cpsi, ~6 mil wall matrix. Trials were run with the first dielet using MAS-2 and LAS-3 batch. The success of these trials allowed a decision to proceed with the fabrication of the new LFA die - a major program milestone for 1996. Samples extruded through this dielet demonstrated a 15% increase in MOR versus the high cell density matrix obtained through the pull-rod die.

A second dielet was fabricated to evaluate a number of improvements in the fabrication process with the objective of providing a die without the hole drilling defects that were present in the first dielet. Subsequent successful extrusion trials were also run on this dielet.

The first dielet was plated in the attempt to reduce the cell wall thickness of the fired matrix to ~5 mils. A trial extrusion was made which yielded an approximate 4.2 mil cell wall thickness. For a first trial of this wall thickness, excellent extrusion results were obtained with only a few distorted cells observed. This significantly increased confidence of extruding 5 mil wall product with the die design selected for this program. However, since the two attempts to plate die to achieve a targeted 5 mil wall thickness, yielded ~6 mil and ~4 mil walls, the plating process required for the fine cell geometries will require further development work in 1997 when the new LFA die is available.

4.2 LFA Die Fabrication

Internal Corning approval was obtained to fund the new LFA die outside the HVTE-TS contract as funding was not sufficient to support this task and keep other critical tasks on schedule. The die build began in February and, despite a number of minor setbacks, was on track for a September completion when it was destroyed during one of the final fabrication steps.

A series of fabrication trials with smaller dielets were then made that confirmed the cause of failure and demonstrated a modified process that avoided this problem. Additional Corning funding was obtained and a second die build started. At the end of 1996 this second LFA die was near completion with a projected first run date in February, 1997.

5. LFA PROCESS TECHNOLOGY

The objective of this task was to establish a repeatable lab scale process for fabrication of LFA, high cell density disks. Most of the extrusion process trials in 1996 were done with the MAS-2 batch as the candidate LAS material was still being defined and the use of an MAS batch allowed a much closer comparison to Corning's years of Celcor® production experience. Since the firing of MAS-2 had not presented a problem, firing studies focused on LAS-3 and then on the LAS-4 material when it was selected. Extrusion process work was then expanded to include this glass-mineral batch. Process steps that were addressed included: glass melting, mineral batch variation, extrusion, drying, and firing. All extrusion trials were done with the pull rod die. (Note: a wet blank is a sliced LFA sample before drying; a green blank is the same after drying; a fired blank is the same after drying; a disk is a finished LFA regenerator core with skin and hub).

5.1 Glass Melting (LAS)

No changes to the base glass used to formulate this material family were made in 1996. A scale up from 10 pound crucible melts to 200 pound melts was accomplished - with a total of three larger melts completed. Physical properties, oxide composition, and particle size (ground glass) were checked on each melt to insure compliance with target values.

5.2 Minerals (LAS)

Large lots of minerals were obtained from suppliers and checked for oxide content and particle size. Small rod extrusions were made of various combinations of mineral lots and glass melts. Rods were used to test the effect of mineral lot changes and different glass melts on two LAS candidates, one of which was LAS-3. Firing shrinkage was measured and physical property data - MOR, thermal expansion and porosity - was obtained. No significant difference in shrinkage or properties was measured as a result of using different batch mineral lots or melts.

5.3 Extrusion

A total of five LFA extrusion trials were made in 1996. Three using the MAS-2 batch and two done with LAS-4. Significant improvement in the quality of the blanks produced for both MAS-2 and LAS-4 batches was achieved by the development of a new wet log slicing technique, improved handling procedures, and better control of batch rheology. However, due to the tight process window afforded by the pull rod die, the difficulty maintaining a satisfactory batch rheology remained. Blanks made were characterized as having a higher than desired level of cell defects - attributed to poor flow of slightly stiff batches through the compound slot die or poor knitting if the batch were too soft. Soft batches also were harder to handle and resulted in distorted matrix structure. Sufficient MAS-2 and LAS-4 blanks were produced for drying and firing trials and to provide disk samples for AMPS. The quality of the MAS-2 blanks was as good as any made in past years.

A new, larger extrusion press was purchased by Corning in the fourth quarter and has been installed. This press will provide improved process control for future LFA trials. In conjunction with our new LFA die, this press will provide a significant improvement in our capability to extrude high cell density regenerator blanks.

5.4 Drying Trials

Concurrent with each extrusion run, drying experiments were run in the lab rotary dielectric dryer. Acceptable drying of both candidates was achieved although the LAS-4 batch required ~60% longer drying time to achieve an equivalent level of dryness vs. MAS-2. Changes in the drying process to reduce cracking on the bottom of the green blanks (blanks are dried with matrix oriented vertically in 12" to 18" lengths) were also developed.

5.5 Firing

The first attempts at firing LFA blanks of the LAS-3 candidate in early 1996 resulted in severe cracking. To meet task requirements for LFA samples, parallel experimental programs were followed to define a firing process for LAS-4 LFA green blanks. Objective of the short term program was to fire several blanks without cracking in order to provide finished disks to AMPS for testing in early 1997. The longer term program will focus on optimizing the fired properties and shrinkage control needed for consistent processing.

Short Term: Based on earlier firing trials of rods and small matrix samples, a peak sintering temperature of 20°C below the earlier LAS-3 standard cycle was chosen for LAS-4. Using the lower temperature, a gas firing trial with a new muffle setup yielded our first crack free LFA LAS-4 disk. This blank, however, exhibited a significant difference in shrinkage from top to bottom (the bottom being higher) believed due to the non uniformity of temperature during sintering. A second LFA blank was fired on the same cycle with a modified muffle to reduce the temperature gradient. This sample had only a small surface crack and the shrinkage difference was decreased by half. Based on these results, several good quality blanks were then fired in our larger lab gas kiln in an attempt to get a more uniform firing and good sample disks for AMPS. A number of

crack free LAS-4 blanks with improved shrinkage uniformity were thus obtained. Property data was measured on samples fired with these blanks and results fell within target limits. These blanks have been progressed to finishing but had not been shipped to AMPS at the end of 1996.

Long Term: A firing expert from our Celcor® engineering team was consulted. Based on his recommendation, green LAS-3 matrix samples were prepared and submitted to obtain shrinkage as a function of firing rate; MOR and elastic modulus of samples during actual firing; and DTA. MOR, CTE and shrinkage data obtained during the actual firing of LAS-3 matrix samples were used to calculate Thermal Shock Factors as a function of firing temperature using the formula below. (Results are plotted in Figure 6.)

$$\frac{\text{MOR} \times \text{E Mod}}{\text{CTE}} = \text{TSF}$$

A series of firings with thermocouples inserted in test LFA blanks to measure temperature gradients was planned as a next step. These experiments were delayed due to a kiln failure and a decision to switch to gas firing. New thermocouple and computer control equipment will be purchased by Corning in early 1997 and when available, used to make measurements which will be combined with TSF results to determine critical times in the firing cycle where cracking would most likely occur and thus facilitate the design of a new firing cycle.

Shrinkage and property measurements of green blanks as a function of peak firing temperature were made. Blanks were gas fired at four different temperatures to confirm the effect of peak firing temperature and that physical property targets have been achieved. Shrinkage results have been plotted in Figure 7. Conclusion: there is a significant change in firing shrinkage - or micro-structure densification - with peak firing temperature. Therefore, a thorough understanding of the LAS-4 firing process must be developed to achieve tight process control. Shrinkage variation within a disk was also measured. Physical property samples were prepared from the LFA blanks and submitted for measurement and will be reported in early 1997.

In another series of firing experiments, to determine where in the firing cycle cracking was occurring, LAS-4 blanks were fired up to various points in the firing cycle, the cycle stopped and the partially fired blank slowly cooled. An LAS-4 blank thus fired through the binder burnout stage of the cycle without cracking. The same blank was fired through the second major region of the firing cycle (after binder burnout) without cracking. This experiment will be completed in 1997.

A comparison of LAS-4 properties fired in gas vs. electric lab kilns was done with the only significant difference measured being a slightly lower porosity in the gas fired samples.

6. FINISHING

Blank finishing into extruded regenerator disks in 1996 focused on two areas: development of a low cost hub technology and the demonstration that existing manufacturing capability could be used to finish extruded blanks.

6.1 Hubs

The work begun in 1995 on the PIH (pressed-in-hub) was continued in 1996. The PIH development required the design and build of hub extrusion die, the successful extrusion of the hubs, insertion of the hubs into the core drilled matrix and, finally the co-firing (i.e. single firing step to sinter both matrix and hub and effect bonding). Progress in 1995 resulted in lab fabricated small matrix samples with PIH's in place.

PIH Process

Fabrication of dies for extrusion of the larger hub needed for PRD (pull rod die) blanks was completed. Larger hubs were successfully extruded. Subsequently, the first insertion of a PIH in the center of a full size blank (PRD) was completed and the blank fired. As insertion and firing trials continued with MAS-2, incomplete bonding between matrix and hub was observed and engineering process development was underway at the end of 1996 to improve the completeness of bonding.

After LAS hubs were successfully extruded, two initial trials to cofire LAS PIH's into LAS matrix were made. Although the bond between hub and matrix looked good, in one case the hub cracked, in the other the ID surface of the hub appeared to have partially melted. It was concluded that the firing shrinkage difference between matrix and hub along with the sensitivity of shrinkage (and sintering/melting) to peak temperature will require a more extensive development effort for LAS versus MAS.

PIH Testing

Using a test rig and procedure recommended by AMPS, an PIH was tested for strength by mounting the small round sample in a rig with a load applied to the circumference of the sample by a flexible belt. The sample supported by a fixed spindle through the hub. The hub tested was MAS-2 that had been co-fired into a piece of high cell density matrix MAS-2. The load was increased to a maximum of ~150 lbs with no damage noticeable to the hub or its surrounding matrix. No higher values were attempted as at this high load the belt had begun to crush the cell matrix on the periphery of the sample. The 150 lb load exceeds the target value provided by AMPS of 13 lbs by a factor of 10X. Additional testing was done by loading the hub in an attempt to "push out" the hub from the matrix. Testing to failure with the axial load applied to several pressed in hubs indicated that a load of > 1000 lbs would be required to cause failure. One sample survived a load of > 2300 lbs.

ID Surface Finish

An series of higher density hub materials and machining experiments were completed in 1996 to reduce the ID surface roughness of the PIH. A best effort machining trial of a high density candidate extruded hub (this material had a total porosity of 1.57% versus > 20% for MAS-2) was done with no significant improvement in the machined surface finish realized. With decision by AMPS that the as provided ID finish of the MAS PIH was satisfactory, this development activity was stopped.

6.2 Disk Finishing

A finishing trial of six LFA MAS-2 disks was completed (cast hubs) to determine if Corning's existing manufacturing capability could be used to finish extruded disks to AMPS preliminary specification. A complete QC of four disks was done. With the exception of a higher than specification number of cell defects (as expected), all requirements for surface finish and part dimensions were met. In addition, the QC test results for leakage and pressure drop as summarized in Table 5 below (specification numbers are standard for 1400 cell wrapped Code 9455 disks) were acceptable.

TABLE 5. Quality Control Testing of MAS-2 LFA Disks

<u>Test</u>	<u>Range</u>	<u>Specification</u>
Leakage (lbs/sec/in ² , X10 ⁻⁵)	1.5 - 2.1	< 15*
Pressure Drop (inches kerosene)	7.8 - 8.4	< 9

*Note: target spec for HVTE-TS was set at 3 or less.

7. SAMPLES

The following samples were provided to AMPS for evaluation in 1996.

Cyclic Thermal Rig (3"X2.4"X1.55"): 6 MAS-2, 4 LAS-3, 6 LAS-4
Engine Rig (9.5"DX2.4"): 2 MAS-2
Vibration Test (9.5"DX2"): 2 MAS-2
ANL Seal Test (6"DX3"): 2 MAS-2, 1 LAS-3.

8. SAMPLE TESTING RESULTS

Samples provided to AMPS were tested on their engine rig and in their Cyclic Thermal Rig Test. The most significant results reported in 1996: the successful completion of >500 hours of steady state (1750°F) by two 9.106"D MAS-2 disks; and both LAS-3 and LAS-4 CTR samples passed the hot face retained strength target (>200 psi, MOR) after 3,000 cycles to 2090°F. In addition, while the MAS-2 failed the CTR test after only 100 cycles, an MAS-2 sample did meet the target retained strength after 3,000 cycles to a lower peak temperature (1700°F) - more representative of a "metal cycle" turbine operation. A second LAS-3 sample was tested to 10K cycles but was found to be cracked when cut up after testing (to obtain the MOR bars). A post mortem of this sample was done as described below. Testing of LAS-4 (high temperature "ceramic cycle") and MAS-2 (lower temperature) will be extended to 10k cycles in early 1997.

9. SAMPLE POST MORTEM

A post mortem was done on an LAS-3 CTR sample that had been found to be cracked after 10,000 cycles on AMPS Cyclic Thermal Rig Test. Although sample sizes available for testing (MOR, CTE) were smaller than "standard", test results led to the following conclusions:

- No change in sintered material (X-ray, Porosity).
- Strong evidence that the mechanism of strength loss is associated with the development of micro-cracking. Based on hysteresis in CTE curves and an >60% reduction in strength with the hot face more micro-cracked than the cold face.
- Higher than expected retained strength at hot face (MOR = 155 psi).

A detailed summary of the post mortem results was published in a separate report.

10. MANAGEMENT AND REPORTS

During 1996, three Corning Team visits to AMPS were made to review program status. In addition, the Corning Team participated in several reviews of the ANL seal development program both at AMPS and at ANL. In addition to these face-to-face meetings, frequent conference calls were conducted on an as need basis.

Weekly, monthly and an annual written reports were provided to AMPS.

Paul Day and Bob GrosJean attended the PNGV Gas Turbine Summit Meeting in Washington in May 9 at which Paul Day presented a brief update on extruded regenerator development and the need for commercial justification of the investment required to scale up to production.

Lou Holleran participated in a PNGV Manufacturing Workshop in Detroit (August).

Lou Holleran and Bob GrosJean attended the annual CCM in Detroit (October).

11. REFERENCES

- (1) Extruded Gas Turbine Regenerator Development, 1995 Annual Report, dated: 2/29/96.
- (2) HVTE-TS Extruded Gas Turbine Heat Exchanger Program 1995-1998, SOW Proposal submitted to AMPS on 2/27/95.

TABLE 1: Summary of Extruded Regenerator Development Program-1996

<u>TASK AREA</u>	<u>1996 OBJECTIVES</u>	<u>ACCOMPLISHMENTS</u>
MATERIALS	MAS: PROVIDE SAMPLES FOR AMPS & ANL TESTING	SEE BELOW
	LAS: OPTIMIZE LAS-3 AND SELECT LAS-4 BASED ON LAB EVALUATION	OVER 30 CANDIDATES EVALUATED & TESTED W/ LAS-4 SELECTED 8/96 BASE SINTERING CYCLE TO OPTIMIZE PROPERTIES CHOSEN
PROCESS & MATERIAL DEVELOPMENT	IMPROVE LAB LFA PROCESS CAPABILITY	ROTARY DIELECTRIC DRYER UPGRADED AND CYCLE DEVELOPED IMPROVED WET HANDLING AND CUTTING RAN FIVE LFA EXTRUSION AND DRYING TRIALS (MAS-2, LAS-3, LAS-4) FIRING TRIALS OF MAS-2 IN PRODUCTION KILNS NEW EXTRUSION PRESS PURCHASED FOR LAB INSTALLATION 12/96

TASK AREA**1996 OBJECTIVES****ACCOMPLISHMENTS**

DIE FABRICATION

BUILD THE LFA DIE BY 3D QTR

TWO DIELETS BUILT AND RUN TO
DEMONSTRATE DESIGN AND
FABRICATION PROCESSCORNING DECISION TO FUND
OUTSIDE HVTE-TS PROGRAM1ST BUILD FAILED 9/96 - ADDITIONAL
FUNDING FOR 2ND BUILD OBTAINED
NOW UNDERWAY W/ PREVIEW 1/97

SAMPLES

DELIVER SAMPLES FOR TESTING TO
INCLUDE: MAS-2 FOR AMPS/ANL TEST
AND FIRST LFA LAS DISK SAMPLES6 MAS-2, 4 LAS-3, 6 LAS-4 CTR
2 LFA MAS-2 ENGINE RIG (9.5"D)
2 LFA MAS-2 SHOCK/VIBRATION
2 MAS-2, 1 LAS-3 ANL SEAL (6"D)

FINISHING & ASSEMBLY

DEVELOP PIH TO ALLOW DELIVERY OF
LFA DISKS BY 12/96PROCESS TO PROVIDE HIGH
STRENGTH BOND DEMONSTRATED
BUT STILL HAVE INCOMPLETE SEAL
TWO PROTOTYPES READY FOR
VIBRATION TESTINGDEMONSTRATE WE CAN MEET FINAL PRODUCT
FINISHING QUALITYFOUR MAS-2 BLANKS FINISHED IN
PRODUCTION FACILITY AND MET
DIMENSIONAL AND SURFACE FINISH
REQUIREMENTS - CELL DEFECTS
STILL AN ISSUE W/ PRD

Table 2: Summary of Matrix Candidate Data, December 1996

	MAS-2	Code 9455	LAS-3	LAS-4	Target Values
Strength					
Rods (MOR)	4000	7000	8800	7362 - 8451	7500
Matrix (Crush)	3600	4000	-	-	6700
Matrix (Axial MOR)	500 - 890	1050	1300	1518	1100
Matrix (Tang. MOR)	342 - 290	848	706	-	600
Thermal Expansion					
RT - 1000°C (ppm)	0.58	0.4	0.74	4.5 - 5.5	<0.5
Microcracking	Yes	None	None	None	None
Porosity	30%	<10%	26%	20% - 30%	TBD
Shrinkage	6 -7%	15%	7 - 9%	7.5 - 8.5%	<10%
Thermal Stability Test					
1050°C / 5 Weeks (ppm)	-	-125	-390	-	<300
Thermal Cycle Test					
1150°C - RT (100 cycles)	>3500	-25	-400	-475 (-262) ()=Treated	<300
Cyclic Thermal Rig Test (2090°F)					
Residual Hot Face Strength (MOR)					
3 K cycles (psi)	< 25	-	483	371	>200
10 K cycles (psi)	< 25	328	155	-	>200
Seal Test (@ ANL)					
100 Hrs (1600 - 1800°F)	0.6	0.35	0.63	-	<0.3
Coeff. of Friction					
Leakage Test	1.5 - 2.5 x 10 ⁻⁵	3 - 9 x 10 ⁻⁵	-	-	<3 X 10 ⁻⁵
Chemical Durability					
Salt (ppm / 1 week)	-8 - 67	537 - 605	382 - 406	181 - 218	<300
Sulfur (Boil Test)	Dissolves	X	3-4X	3-4X	<300

Figure 2: Sulfuric Acid Test Results

Figure 3

**THERMAL CYCLING TEST
(EFFECT OF SINTERING TEMPERATURE ON LAS-4)**

Figure 4

Figure 5

Figure 7

Appendix E

**1995 Annual Progress Report
Submitted by**

AlliedSignal Ceramic Components

and Prepared for

Allison Engine Company, Inc.

under the

**Hybrid Vehicle Turbine Engine Technology Support Project
(HVTE-TS)**

Contract No. DE-AC02-96EE50453

Note: This appendix contains the annual progress report as submitted to Allison. The document has been edited only where necessary to delete information that is deemed to be proprietary in nature.

HYBRID VEHICLE TURBINE ENGINE - TECHNOLOGY SUPPORT PROJECT

ANNUAL PROGRESS REPORT

COVERING PERIOD DECEMBER 1994 THROUGH DECEMBER 1995

SUBCONTRACT NO. H416285 (PRIME CONTRACT NO. DEN3-336)

"DEVELOPMENT OF A PRODUCTION VIABLE MANUFACTURING PROCESS FOR AS800
SILICON NITRIDE COMPONENTS"

AlliedSignal Ceramic Components
TOR - 1/5 - 1 - 27000
2525 West 190th St.
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February 26, 1995

"PREPARED FOR ALLISON ENGINE COMPANY"

I. INTRODUCTION

The objective of AlliedSignal Ceramic Components (CC) on the Hybrid Vehicle Turbine Engine - Technology Support (HVTE-TS) program is to demonstrate gelcasting as a viable production process for manufacturing gasifier turbine wheels, and other components, using AS800 high temperature in-situ reinforced microstructure silicon nitride. The four-year program started in 1994. The primary goal of the program is the demonstration of a production process which meets all customer requirements of cost, schedule, quantity, and quality.

The first year effort focused on providing AS800 silicon nitride test specimens to Allison for property evaluation/validation and on procuring a mold tool for gelcasting AS800 AGT-5 design turbine wheels. Slipcast AS800 specimens (machined and as-processed) and gelcast AS800 specimens (machined) were delivered to Allison in the first year. A re-usable, metal mold tool was designed and fabricated for gelcasting AGT-5 wheels, and the first wheels were gelcast in the mold.

Gelcasting of AS800 AGT-5 turbine wheels continued through the second year. A second task that was started in 1995, development of the gelcast AS800 combustor, continued the prototype work that was initiated under the NIST Advanced Technology Program. The 1995 delivery milestones were delayed due to processing issues. Gelcast AS800 test specimens (as-processed) and engine quality turbine wheels remain to be delivered to Allison in 1996.

II. TECHNICAL PROGRESS SUMMARY

Gelcast AS800 Test Specimens:

Material evaluation was to be completed during the first year of the program. Mil-standard specimens of slipcast AS800 and of gelcast AS800 were to be delivered to Allison for flexure tests. The slipcast AS800 specimens (3mm x 4mm x 50mm bars, 50 machined, 50 as-processed) were delivered in 12/94 and 1/95. The gelcast AS800 specimens (50 machined) were delivered in 6/95. The flexure strength of the machined gelcast bars was measured at CC on a small number of test bars from the same lots. The strength was comparable to slipcast bars at room

temperature (108 ksi gelcast, 100 ksi slipcast) and was lower at 2500°F (58 ksi gelcast, 76 ksi slipcast).

The delivery of as-processed specimens has been delayed due to tooling and densification issues. Plates for the machined test specimens were gelcast in tooling that was originally used for slipcasting; it provided poor as-cast surfaces of the gelcast material. New tools were made for gelcast AS800 plates which will provide high-quality as-cast surfaces. The as-processed gelcast AS800 test specimens will be delivered when the densification issues have been resolved. A description of densification process development follows in the Task 1 section of this report.

Task 1 - Fabrication of Gelcast AS800 AGT-5 Wheels Using Re-usable Tooling

The objective of this task is to deliver five gelcast AS800 AGT-5 wheels for engine testing at Allison. The wheel delivery date was postponed from 9/95 to 11/95, and from 11/95 until 1996. While the AGT-5 mold was being fabricated, experimental castings were performed with existing mold tooling, that was originally used to injection-mold an impulse-type axial wheel (referred to as the "generic wheel" in this report). The activities on Task 1 during 1995 were mold design and fabrication, forming, and densification.

AGT-5 Mold Fabrication Activities:

Mold Masters of Cleveland, Ohio was selected to design and construct the AGT-5 wheel mold tool. The mold was designed to allow evaluation of several filling methods. The design allows pouring gel slip through either shaft while spinning the mold, or by pumping/pressurizing the gel slip into the mold. Tight fit of the mold sections was specified because the AS800 gel slip is a low viscosity fluid. The conceptual tool design drawing was reviewed and approved at CC in May.

The delivery of the mold tool was later than scheduled due to extensive design work, and to delay in determining the scaling factor. An electronic surface model of the AGT-5 wheel was required to complete the mold design. The model was constructed from the blade book and prints supplied by Allison. The model took longer to complete than estimated. Blade sections were not available to define the area where the blades' leading edges blend into the hub. The combined efforts of Mold Masters, Precision Measurement Laboratories (PML), and CC were required to complete the electronic surface model of the wheel and to finish details of the mold design, including the blade/fillet/hub blend and the location of parting lines for the mold sections. A large scale (3X) laminated object manufacture (LOM) model of a wheel section, similar to a stereolithography model, was sent to Allison for inspection. Per Allison's review, Mold Masters was given approval to complete fabrication of the tool. Mold Masters negotiated a price increase and new delivery date of the tool due to the design effort. Mold Masters delivered the AGT-5 gelcast mold tool to CC on 8/30/95.

Details of the mold cavity can be seen in Figure 1, with mold sections pulled away from the hub. The partially assembled tool is shown in Figure 2. Assembly of the mold is completed by placing a clamping ring around the closed mold sections (thereby sealing the blade tips), inverting the top plate onto the clamped mold sections, and bolting the top plate to the base plate.

Forming:

While the AGT-5 mold was being designed and fabricated, the generic wheel mold was used to develop gelcasting by the spinning technique. AS800 gel slip was poured into the mold as it rotated on a turntable. Results from the generic wheel casting experiments were used to determine features for the AGT-5 mold that would facilitate successful casting. An example of one of the first generic wheel castings is shown in Figure 3 (cast in 1994). It represents nearly the worst result, with many air bubbles and broken blades. The wheel shown in Figure 4 illustrates that improvements were made in the gelcast spinning technique during the first months of 1995. The HVTE-TS Program funds were used for gelcasting experiments of the generic wheel through February. Wheels were gelcast in March, using NIST Advanced Technology Program funds, in the metal generic wheel mold and a dipped wax pattern mold. While improvements in casting technique were made, none of the gelcast wheels were defect-free.

Defects in the generic wheel were attributed to deficiencies in the mold tool, which had been designed for wax injection molding. Typical defects included: blades broken from the hub, voids in the hub rim and blade tips, and cracks in the hub and/or blades. The slip leaked from the mold during casting, which contributed to the voids in the casting and probably weakened the blades. In contrast, the dipped wax pattern did not leak slip during casting and contained fewer defects. The casting experience in the generic mold indicated that the AGT-5 gelcasting mold must contain low-viscosity slip without leaking, and that the capability for multiple filling methods should be incorporated in the design.

The first AS800 AGT-5 wheel was gelcast in the mold on 8/31/95 by the spin cast technique. Filling of the mold was satisfactory. However, the mold was not leak tight which led to defects in the component. Two blades cracked, probably due to weakness from leakage and/or stress applied during mold disassembly. One blade developed a small crack and the other blade cracked off completely. All other blades were completely filled and remained attached to the hub. One shaft was cracked while removing the wheel from the mold due to stress applied during mold disassembly. The first gelcast AS800 AGT-5 wheel is shown in Figure 5. Completely filled blades, hub, and shaft are illustrated in this view of the wheel. A view of the other hub, seen in Figure 6, again shows complete fill of the blades along the trailing edge. The broken blade and shaft can be seen in this view of the wheel. Modification of the mold and/or a change in procedure for disassembly of the mold would be required to avoid cracking of shafts and blades.

Alternative filling techniques were used during the last months of 1995. The mold was filled from the top plate and the bottom plate, by spinning and/or by plunging the slip into the mold. Twenty four AGT-5 wheels were gelcast in all. A good example of spin casting is illustrated by the wheel shown in Figures 7 and 8. This wheel was gelcast by pouring slip through the larger shaft as the mold was spun on a tilted platform. Similar results were obtained by filling from the smaller shaft while spinning. The spin-cast technique produced the best results, but none of the wheels were defect-free. Attempts at plunge filling resulted in severe slip leakage. The plunge filled wheels had numerous voids, and the leaked slip interfered with releasing the part from the mold and caused cracking of blades and shafts. Mold Masters was called to correct the slip containment deficiency.

Mold Masters visited CC, observed de-molding of a gelcast wheel, and suggested some changes in the de-molding procedure which should eliminate cracked hubs and blades. Mold Masters proposed setting up a transfer-press arrangement for the AGT-5 wheel mold. The transfer-press set-up applies a clamping pressure to the mold to eliminate slip leakage, and applies pressure to the slip via a plunger to force air out of the mold chamber. The transfer-press method was demonstrated at Mold Masters in November using a low-viscosity plaster mix to simulate the

AS800 gel slip. Four plaster wheels were cast. Plaster did not leak from the mold during casting. Air entrapment was markedly reduced by the third and fourth castings after venting modifications were made to the tool. The results warranted a trial at CC using AS800 gelcast material. The transfer press was shipped to CC at the end of December. A representative from Mold Masters was scheduled for set-up of the transfer press and training in its operation during January 1996.

Densification:

Gelcast AS800 requires a higher temperature sintering cycle than slipcast AS800. Crucibles used for sintering AS800 are coated on the interior surfaces to provide the proper atmosphere for sintering. A block of gelcast AS800 was first densified at CC in a small, laboratory scale crucible in the fall of 1994. Full densification of gelcast AS800 was repeated in that crucible. During 1995, attempts to densify larger, complex-shaped components in larger capacity crucibles were not uniformly successful. The larger crucibles had been used for slipcast AS800 densification, and the higher temperature of gelcast AS800 sintering caused deterioration of these crucibles. New alternative crucible materials and coatings were used to sinter large components, but the results were still inconsistent. The small, lab scale crucible's coating also began to deteriorate, and sintering results became inconsistent for the small gelcast AS800 blocks.

Of the twenty four gelcast AGT-5 wheels, seven were fully processed. Six were scrapped after casting with no further processing. Eleven wheels were processed through drying or binder burnout and will be used in sintering cycle development runs. The seven fully processed wheels demonstrate good as-sintered surfaces. One wheel hub (blades and shafts had cracked) reached full density and two others were very close to full density. All wheels were at least 90% dense.

Efforts to determine the correct combination of sintering parameters continued. Experiments were planned in November for selected crucible/crucible coating combinations in standard and experimental sintering cycles. Orders were placed for the crucibles, one arrived at CC in December, the remaining crucibles will arrive by February 1996. The densification group began an analysis and design of experiments for the sintering cycle parameters and sintering crucible materials. The experiments for sintering cycle development are scheduled to begin in January 1996.

Task 2 -Development and Demonstration of AS800 Combustor and Scroll Fabrication

The NIST Advanced Technology Program supported early stages of combustor development in 1994 and early 1995. Six inch long, 2 inch diameter, 0.1875 inch thick tubes were gelcast using all metal tooling. The next shape to be gelcast were 6 inch long, 0.1875 inch thick tubes with diameters necking down from 2.5 inches to 1.75 inches. All-metal tooling was first used for this shape; later gelcasting was done using a wax insert in metal housing. The combustor diameter necks down at either end, so dissolvable wax inserts will be required to form the interior wall.

The next stage of combustor development was supported by the HVTE-TS program and with Ceramic Components' internal funding in 1995. Prototype tooling was designed in September for gelcasting components which closely resemble the actual combustor size and shape. Two tools were made: a dipping tool to make the wax inserts, and a metal housing tool. The prototype tools, purchased with CC internal funds, were received in November.

Seven combustor prototypes were gelcast in November and December. The first combustor lacked uniform wall thickness because the wax insert shifted position during casting, and it cracked early in the drying process. The second combustor broke while being removed from the mold. The sections of the broken combustor are shown in Figure 9. The third casting, shown in

Figure 10, appears defect-free and it survived all but the last segment of the drying cycle. A CC internal milestone for December, delivery of a combustor prototype, was delayed. The drying, binder extraction, and sintering cycles will require further development for the combustor in 1996.

Task 3 - Program Management and Documentation

The original 1994 funds were used to support the program activities through September 1995 as indicated on the 1994 milestone chart shown in Figure 11. Progress and spending were behind schedule, and the milestone chart was revised accordingly. The chart shows the most recent work plan (modified 6/95) for the 1994 funds. Funds for 1995 activities were released in August. Labor hours for fabrication of wheels were divided between Task 2 of the 1994 purchase order and Task 1 of the 1995 purchase order on the HVTE-TS Program until September, when the 1994 funds were depleted. All work on wheel fabrication after September was charged to Task 1 the 1995 HVTE-TS Program. The milestone chart for 1995 is shown in Figure 12.

It became apparent in October that the approaching milestone, delivery of five AGT-5 wheels, would not be met on time. A meeting was held at CC with Allison, during which progress on the program was presented and the delay in milestones was discussed. Planned activities were described for improving the AS800 gel slip preparation, forming, and densification processes. These activities are supported by the HVTE-TS, ARPA/AMP, and NASA AITP programs as well as CC internal programs. A plan with re-scheduled milestones, based on progress in the above development activities, will be proposed in early 1996. All further work to meet test specimen fabrication and delivery obligations will be charged to AlliedSignal Ceramic Components internal funding.

Figure 1. Mold sections slide inward to the hub to form the wheel mold cavity.

Figure 2. A ring (not shown) holds the sections in place and seals the blade tips. The top plate (right) completes the mold cavity when inverted onto the lower assembly (left). The top plate is bolted to the base plate to complete the mol assembly.

Figure 3. First generic wheel castings demonstrate the worst case with numerous voids and cracked blades.

Figure 4. Quality of generic wheels improved as casting technique was developed in 1995.

Figure 5. Completely filled blades, hub, and shaft are shown in this view of the first gelcast AGT-5 wheel.

Figure 6. Blade and shaft cracked when the first AGT-5 wheel was removed from the mold.

AlliedSignal 104898-1

Figure 7. AGT-5 wheel was gelcast by pouring slip through large shaft while mold rotated on a tilted turntable.

Figure 8. Blades and shafts remain attached to the wheel. Defects consist of small voids.

AlliedSignal 105089-1

Figure 9. Gelcast AS800 combustor prototype cracked during dc-molding.

Figure 10. Gelcast AS800 combustor prototype was successfully de-molded.

Figure 11. 1994 work plan was modified. Funding from 1994 supported tasks through September.

Figure 12. 1995 work plan included delivery of five AGT-5 wheels. Re-scheduled milestones will be proposed in early 1996.

Appendix F

**1996 Annual Progress Report
Submitted by**

AlliedSignal Ceramic Components

and Prepared for

Allison Engine Company, Inc.

under the

**Hybrid Vehicle Turbine Engine Technology Support Project
(HVTE-TS)**

Contract No. DE-AC02-96EE50453

Note: This appendix contains the annual progress report as submitted to Allison. The document has been edited only where necessary to delete information that is deemed to be proprietary in nature.

HYBRID VEHICLE TURBINE ENGINE - TECHNOLOGY SUPPORT PROJECT

ANNUAL PROGRESS REPORT
COVERING PERIOD JANUARY THROUGH DECEMBER 1996

SUBCONTRACT NO. H506060 (PRIME CONTRACT NO. DEN3-336)
"DEVELOPMENT OF A PRODUCTION VIABLE MANUFACTURING PROCESS FOR AS800
SILICON NITRIDE COMPONENTS"

AlliedSignal Ceramic Components
TOR - 1/5 - 1 - 27000
2525 West 190th St.
Torrance, CA 90504

February 17, 1997

"PREPARED FOR ALLISON ENGINE COMPANY"

I. INTRODUCTION

The objective of AlliedSignal Ceramic Components (CC) on the Hybrid Vehicle Turbine Engine - Technology Support (HVTE-TS) program is to demonstrate gelcasting as a viable production process for manufacturing ceramic turbine engine components, using AS800 high temperature in-situ reinforced microstructure silicon nitride. The four-year program started in 1994. The primary goal of the program is the demonstration of a production process which meets all customer requirements of cost, schedule, quantity, and quality.

The first year effort focused on providing AS800 silicon nitride test specimens to Allison for property evaluation/validation and on procuring a mold tool for gelcasting AS800 AGT-5 turbine wheels. Slip cast AS800 specimens (machined and as-processed) and gelcast AS800 specimens (machined) were delivered to Allison in the first year. As-sintered test specimens remain to be delivered for property evaluation at Allison. A re-usable, metal mold tool was designed and fabricated for gelcasting AGT-5 wheels in the first year, and the first wheels were gelcast in the mold.

Three tasks, active during the second year, continued during the third year of the HVTE-TS Program. The delivery of five engine-quality wheels was a milestone for Task 1 in 1995. The delivery was delayed due to process development issues. Task 1 continued in 1996 with the gelcasting of AS800 AGT-5 turbine wheels utilizing the reusable mold. Under Task 2, a gelcasting mold was fabricated for prototype hybrid vehicle combustors in 1995. Task 2 continued in 1996. A fabrication process was developed for the prototype combustor, and a mold was fabricated for the hybrid vehicle combustor. A combustor was fabricated and machined to demonstrate the fabrication process. Task 5 continued through 1996, consisting of program management and documentation.

II. TECHNICAL PROGRESS SUMMARY

Task 1 - Fabrication of Gelcast AS800 AGT-5 Wheels Using Re-usable Tooling

The objective of this task is to deliver five gelcast AS800 AGT-5 wheels for engine testing at Allison. The wheel delivery date has been postponed several times from the original 9/95 date into 1997. The primary reason for delay is casting defects. Task 1 focused on reducing defects during 1996. Alternative forming techniques were investigated, a new analysis tool was used on the process, and the AGT-5 mold was modified.

Most of the AGT-5 gelcast wheels, from the start of the program to the beginning of 1996, had been formed by spin casting. In spin casting, the mold spins on a turntable as it is filled with AS800 gel slip. Spin casting has been demonstrated as a viable wheel forming technique in other molds, but no defect-free gelcast wheels had been produced by spin casting in the AGT-5 mold. The cause of most defects was attributed to leakage in the mold. Mold Masters, the AGT-5 mold fabricator, recommended the use of a transfer press to eliminate slip leakage. The transfer press method has been used effectively to form components from other ceramic slip systems. Clamping pressure is applied to the mold, and a plunger applies pressure to the ceramic slip to fill the mold cavity. However, the transfer press experiments conducted at CC in early 1996 gave poor results. The clamping pressure proved ineffective for containing AS800 gel slip in the AGT-5 mold. The AS800 gel slip, which contains water, sub-micron particle size powders, and polymerization chemicals, was forced into the spaces between mold sections during transfer press casting. The AS800 gel slip acted as an adhesive in the tight spaces between mold sections, making mold disassembly very difficult and thereby increasing the likelihood of cracked blades. Furthermore, voids were not eliminated in any of the castings. Seven wheels were gelcast in the transfer press. The results from these experiments implied that pressure-fill casting would require a significant developmental effort. Therefore, the focus of wheel forming development returned to spin casting.

During the next couple of months of spin casting, results proved unpredictable and unsatisfactory. Voids were found in various locations (i.e., the hub, shafts, or blades) on all of the wheels cast by the spinning method during that period. Two of the wheels had all blades and both shafts attached, but all other wheels had cracked blades and/or cracked shafts. The exact causes for the defects were not well understood. In order to determine the root causes of defects, the forming team began using a new approach to analyze the gelcasting process. The team began using a quality tool called "Operational Excellence," which was rolled out in 1995 at AlliedSignal. The goals of Operational Excellence are to reduce variation and to eliminate defects in a given process. The course of action taken was to: 1) map every detail of the gelcast wheel forming process, 2) conduct a failure modes and effects analysis (FMEA), 3) perform a measurement systems evaluation (MSE), and 4) conduct a design of experiments (DOE).

The AGT-5 process was mapped in detail. The process map provided a launching point for the FMEA. The FMEA was applied to each step in the spin-casting process. All known inputs to the process were listed, and the possible failure effects were rated for severity, occurrence, and detectability (an MSE was performed). Upon compiling the ratings, the process inputs with the highest number were identified as those most likely to have a great impact on the casting quality. The highest ratings for the AGT-5 gelcasting process inputs were those which 1) control the strength of the gelled part, and 2) could affect the occurrence of voids. Experiments were designed accordingly.

The first DOE was conducted to optimize the gel strength, using a new gelation initiator, in order to consistently produce castings with both shafts and all blades attached. The new gel initiator, developed at ORNL, is thermally activated. In contrast, the original initiator system was chemically activated, and the gelation reaction started immediately upon mixing at room temperature (after filling, mold was placed in an oven to accelerate gelation). Thermally activated initiation allows better control of the working time, gel structure, and part strength. For this DOE, heating and cooling of the mold was accomplished via the mold's water channels, using a digitally controlled water recirculator, for more precise control and rapid response. The experimental matrix was designed to examine the effects of the following process inputs: 1) heating rate, 2) hold temperature, 3) time at hold temperature, 4) cooling rate, and 5) temperature at disassembly during gelcasting. The measured output was the strength of the casting, and observed outputs were part release and part integrity. From the results of the first DOE, a combination of process inputs was determined which resulted in crack-free hubs, good part release, and a strong gel structure. To complement the optimized process inputs, a method of mold disassembly was devised by which the alignment of mold sections is maintained which contributed to the quality of gelcast wheels. Misalignment of mold sections during disassembly had been a contributing cause of cracked shafts and blades.

The second DOE conducted in 1996, again based on the FMEA, was to minimize voids in the AGT-5 wheels. The process inputs: 1) spin table speed, and 2) the angle of the turntable, were varied. The observed outputs were the quantity and location of voids in the blades, hubs and shafts. The DOE results indicated that speed, by itself, did not have a large effect on the quality of the casting. However, speed interacted with tilt angle to increase or reduce the number of voids. The best results were a compromise of the two inputs' effects; defects were minimized from a combination of high speed rotation and low tilt angle. However, no combination of casting inputs was determined which did, or potentially would, produce defect-free castings using the original wheel mold configuration. The forming team concluded that modifications to the mold or fabrication of a new mold would be necessary to cast a defect-free AGT-5 wheel.

A gelcasting procedure capable of producing engine-quality AGT-5 castings would have had to be demonstrated by mid-August in order to deliver turbine wheels for testing by the end of 1996. The CC internal milestone of 8/15/96, "Cast Defect Free AGT-5 Wheel," was missed. The casting team had conducted AGT-5 gelcasting experiments which provided CC with a thorough understanding of the casting parameters. The casting parameters for the AS800 gel slip system were optimized, but an engine-quality casting had not been produced using the original mold configuration. Cracked blades were still a fairly common defect, but crack-free wheels had been produced with the AGT-5 mold and the general frequency of cracking had been substantially reduced. The most consistent defect was voids. Voids had occurred in every AGT-5 wheel casting to date. Voids were always present in critical areas such as the hub rim, making the part unacceptable for engine testing. CC proposed modifying the mold, which was approved by Allison. The particular benefit of the modification was that air entrapment on the hub rim would be reduced or eliminated; the downside is that additional machining of the gelcast wheels will be required.

Originally, the mold formed net-shape hub faces on the AGT-5 wheel. Modification of the mold eliminated a frequent site of air entrapment - namely, the outer rim of the concave-shaped hub face which meets the airfoils' trailing edges. That hub section of the mold was re-machined to form a convex surface on one hub face; also, the shaft diameter was enlarged. This configuration had proven moderately successful under the DARPA/AMP Program for gelcasting turbine wheels. The wheel shown in Figure 1 was gelcast in the modified AGT-5 mold. In all, ten wheels were gelcast in the re-worked mold, using the spin casting method, by the end of 1996. A distinct improvement was noted in the quality of the castings, but none were defect free. Each casting was visually inspected and of the ten wheels, two have the potential for testing. Voids in those

two wheels are not located in high stress regions, and it appears likely that the minor defects can be hand finished to facilitate proof testing.

While the AGT-5 was being gelcast in the modified mold by spin casting, a set of vacuum gelcasting experiments were conducted using another turbine wheel mold under the DARPA Program. The DARPA turbine wheels usually contain small voids, similar to the AGT-5, but the voids are generally located in areas which will be machined, such as at the blade tips. The first two DARPA vacuum gelcasting experiments resulted in a wheel with very few voids, so three AGT-5 wheels were gelcast using the vacuum method. The voids were somewhat smaller than usual in the AGT-5, but were more numerous. The vacuum gelcasting method will be developed further with the DARPA wheel under the DARPA Program. When results improve, vacuum gelcasting will be tried again with the AGT-5 mold. Meanwhile, AGT-5 gelcasting will resume in 1997 using the spin cast method.

Task 2 -Development and Demonstration of AS800 Combustor and Scroll Fabrication

Combustor development continued in early 1996 using the prototype mold that was fabricated in the fall of 1995. Gelcast combustor prototypes were used to develop effective drying and sintering cycles. The first prototypes cracked severely during the drying cycle. Three potential sources of the cracks were: 1) thin areas in the combustor due to tooling/alignment issues, 2) an overly aggressive drying cycle, and 3) an "inhibition layer" on the inner wall of the combustor, where the wax insert interferes with the polymerization of the gel slip.

The tool was modified to correct alignment and wall thickness issues, but cracking still occurred during drying using the standard cycle. Experimental drying cycles were used in the humidity dryers. Reductions in cracking resulted from the experimental cycles, but none of the dried combustors were crack-free. These findings pointed to the inhibition layer as a primary cause of cracking. The inhibition layer on the interior of the prototype combustors penetrated approximately 0.020 inches into the wall thickness. As experiments to develop an effective drying cycle for combustors continued, casting methods which would reduce or eliminate the inhibition layer were developed.

The slip formula was changed in order to observe whether the inhibition layer was reduced. Three monomer ratio levels were used. The inhibition layer thickness was expected to decrease with an increase in the monomer ratio, but an undesirable effect from this change was that the strength of gelled material decreased with an increase in the monomer ratio. Further, the potential advantage was not realized. Inhibition layers did not vary between the three monomer ratio levels, so use of the standard monomer ratio was resumed. The most effective method found to prevent an inhibition layer was to apply a barrier coating to the wax core before gelcasting. Wax cores were coated with spray paint, which prevented the interaction between the wax core and the gel slip. The first combustor that was gelcast with the spray-painted core was fully dried, crack-free, using an extended drying cycle. The extended drying cycle was repeated on a second combustor with success, then a third combustor was dried, crack-free, in a shorter drying cycle. By the middle of the year combustor prototypes could be gelcast, dried, have the binder burned out, and be sintered with no ill effects. A prototype combustor was delivered to Allison in order to demonstrate the prototype fabrication process.

Allison released a revised combustor print, and tooling for the deliverable combustor was designed and fabricated. The casting, drying, and binder extraction procedures translated directly from the prototype combustors to the newly designed, deliverable combustors. The sintering procedure had to be altered to accommodate the new design. The first combustor blank to be densified rested directly on the floor of the crucible during sintering, and friction caused distortion

of the combustor inlet area. The subsequent combustor blanks were sintered using a setter plate, which greatly reduced distortion.

Processing was on schedule to meet the August delivery milestone of two combustors, but a delay was announced for the delivery of machined combustors from Ellis Ceramics. The form grinding tools (for the inlet and outlet areas) took longer to fabricate than Ellis expected. Meanwhile, Ellis fabricated fixture tooling and began drilling experiments. Ellis used a core drill on one of the prototype combustor blanks and reported excellent results, which allayed concerns that severe chipping would occur upon break-through of the drill.

The first machined AS800 combustor was received from Ellis Ceramics on October 2, and was examined on that day at CC during the program review with Allison. The combustor is shown in Figure 2. A number of machining defects were observed. Among the defects noted were: some chipping on the exterior and a great deal of chipping on the interior in the region of the igniter and dilution holes, lack of edge break on the i.d. of the combustor inlet, lack of blending in transitions between machined and as-sintered surfaces, and dimensions out of tolerance. A spall layer (perhaps a thin inhibition layer) was observed on the combustor's interior. The spall layer contributed to the chipping that was noted around the drilled holes. CC discussed the machining defects with Ellis Ceramics to determine whether their procedures could be satisfactorily improved. Ellis agreed with all of CC's and Allison's observations. Ellis stated that their machining procedures could be improved, based on this initial experience, and expressed confidence in their ability to meet the requirements. Two new blanks were shipped to Ellis to be machined into deliverable combustors.

To reduce chipping around the igniter, dilution, and drain holes, Ellis ordered a low melting temperature, low expansion metal alloy to fill the combustor blanks for dampening during the drilling operation. One combustor blank was scrapped after grinding, and before drilling, due to machining errors; the outlet was machined out of round and the flange could not be formed at the inlet because too much stock had been machined away. Grinding operations on the remaining combustor blank were completed within tolerance, but the drilling operation was interrupted after breaking one drill bit and nearly consuming the one remaining drill bit. The drilling problems resulted from the use of potting metal in the combustor for its dampening effect. The dampening effect adversely affected the ultrasonic drilling action, so the use of potting metal to prevent chipping was abandoned. An alternative, two-step plan was made for producing chip-free holes. Undersized holes will be drilled, and the holes will be opened to their specified diameter using ream tools. Ellis received the new drill / ream tools during December, but did not complete the machining of one combustor as expected. Ellis scheduled completion of the combustor for January 1997.

Task 3 - Program Management and Documentation

Progress and spending remained behind schedule during 1996. The schedule and the spending charts for the year are attached. CC was advised by Allison in the third quarter that additional funds for 1996 would not be available as planned, and that 1996 funds would need to be used through 3/97. A brief overview of the program was presented to Allison in October. Also, a revised work schedule for delivery of the AGT-5 wheels, and a spending plan for the remaining 1996 funds was discussed. The estimated cost to complete tasks, based on a work schedule to deliver five AGT-5 wheels in 3/97, indicated that the remaining funds would be used by the end of 1996 if the rate of spending continued at the same pace. However, as no additional funds would be available before 3/97, the work was scaled-down to deliver the combustor in progress and as many wheels as possible in 1997 with the remaining funds.

Figure 1. The modified AGT-5 mold produces castings with an enlarged shaft and a convex surface on one hub face.

AlliedSignal

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Figure 2. The first fully machined, gelcast AS800 combustor demonstrates the state of the current fabrication process.

AlliedSignal Ceramic Components
Allison Hybrid Vehicle Turbine Engine - Technology Support
Proposed 1996 Work Plan Revision

Project: HVTE-TS
 March 25, 1996

Task

Progress

Milestone

Summary

Rolled Up Task

Rolled Up Milestone

Rolled Up Progress

Internal Milestone

Rolled Up Internal Milestone

AlliedSignal Ceramic Components
Allison Hybrid Vehicle Turbine Engine - Technology Support
Proposed 1996 Work Plan Revision

Project: HVTE-TS
 March 25, 1996

Task		Rolled Up Milestone	
Progress		Rolled Up Progress	
Milestone		Internal Milestone	
Summary		Rolled Up Internal Milestone	
Rolled Up Task			

Appendix G

**1995 Annual Progress Report
Submitted by**

Kyocera Industrial Ceramics Corp.

and Prepared for

Allison Engine Company, Inc.

under the

**Hybrid Vehicle Turbine Engine Technology Support Project
(HVTE-TS)**

Contract No. DE-AC02-96EE50453

Note: This appendix contains the annual progress report as submitted to Allison. The document has been edited only where necessary to delete information that is deemed to be proprietary in nature.

KYOCERA INDUSTRIAL CERAMICS CORP.

Objective/Approach

Kyocera Industrial Ceramics Corporation's (KICC) support of the Technology Support Program has a multi-year plan addressing near, intermediate, and long term needs. The near term needs are the fabrication of AGT-5 engine components to support engine testing. This need will continue throughout the Program. This activity has primarily utilized technology transferred from Kyocera's facilities in Japan. Near term efforts also include extending the technology to meet the component delivery needs of the Hybrid Vehicle Propulsion Program. Intermediate plans focus on developing new and/or improved processes for both advanced component designs, such as integral nozzle assemblies, and processes capable of producing large quantities of parts at low cost. The longest term program goal will be to demonstrate a moderate size production run of selected components.

Delivery of the components needed for the AGT-5 test bed is one of the near term objectives. Both gasifier rotors and turbine scrolls are being fabricated. Although Kyocera had previously provided both components fabricated from SN252, the aim of this initial effort is to provide the rotor from the newer

SN253 material. The rotor is being fabricated by solid slip casting, whereas the scroll is fabricated by drain casting. Two scroll configurations are being worked on; rectangular and circular cross section designs. These descriptions refer to the shape of the flow path cross section. The rectangular scroll is the design originally fabricated by Kyocera for Allison during the ATTAP project. Although a circular cross section geometry is more desirable for aerodynamic performance, the rectangular configuration is being processed to expedite delivery by utilizing existing mold tooling. In parallel with the fabrication of the rectangular scroll, KICC is also developing the processes needed to fabricate scrolls of the circular configuration. Initially, the circular scroll process development is focusing on the scroll designed for the Hybrid engine. Subsequently, a circular cross-section scroll for the AGT-5 test bed will also be fabricated.

All batches of scrolls and rotors are screened for acceptable MOR at room temperature and 1350°C, as well as flexural stress rupture. In-process and final inspection of all components includes radiographic, fluorescent penetrant, and coordinate measuring machine inspections. Additionally, rotors are screened by ultrasonic inspection and proof spin testing.

Accomplishments

- Three (of five required) SN253 20 bladed AGT-5 rotors have passed NDE and spin proof test requirements, and have been delivered.
- One (of three required) rectangular scroll has been delivered.
- Polycarbonate scroll patterns have successfully been used in the mold fabrication process, shortening the tool development process.
- Rectangular scroll casting/sintering processes are being applied to the circular scroll designs.

Discussion

AGT-5 Rotor fabrication progress

To accomplish this quickly, traditional slip casting and the previous mold tooling are being utilized. Rotors are cast to near net shape to provide as-processed blades and minimal machining of the hub and shaft surfaces. Figure 1 illustrates the rotor after sintering.

Only tip grinding is performed on the blades. Rotors are spin tested by KICC prior to the rotor-to-shaft joining, performed by Allison. To accomplish the proof testing, and still deliver an unblemished part, KICC is utilizing specially designed balancing and spin testing tooling which allows proof testing to be performed without removing any balancing stock from the ceramic part.

Figure 1, AGT-5 20 bladed rotor in the as-cast and sintered condition.

Four lots of rotors have been processed and MOR bars cut from rotor hubs have exhibited strengths above both the in-house room temperature and elevated temperature specifications, as shown in Figure 2.

Figure 2, Flexural MOR strengths for co-processed SN253 rotors.

Subsequent spin proof testing of the rotors has also confirmed the integrity of all of the completed rotors. Figure 3 illustrates a rotor captured at the 80,000 rpm proof speed by high speed electronic recording. The recording system is also configured to record rotor burst

occurrences, although no AGT-5 rotor bursts have occurred in proof testing activities.

Figure 3, Rotor S/N 5-5 captured at the 80,000 rpm proof speed.

Three rotors have been completed and delivered to Allison. Although two additional rotors have been completed, and passed proof testing, NDE indication in the rotor hub precluded their delivery. Two additional rotors are currently being processed for future delivery.

AGT-5 Rectangular cross section scroll fabrication

The AGT-5 rectangular cross section scroll is being fabricated in one piece by drain casting from the SN252 composition. Since this part had previously been fabricated by Kyocera in Japan during the ATTAP program, existing plaster mold designs were utilized.

The desire for thin, uniform, void free walls in this complex shape presents the initial processing challenge. Prevention of air entrapment during the casting process is the primary processing challenge, as the complex shape provides many

locations for bubbles to lodge. Additionally, the thin walls are susceptible to distortion during sintering. Finally, the overall complexity of the part, in conjunction with the need for close tolerance at the gas inlet, outlet and support features, present grinding set-up and dimensional inspection challenges. These challenges are being addressed by using a well controlled and understood slip, multiple piece plaster molds, iteratively improved casting and draining techniques, and special fixtures for supporting the part in sintering and final grinding. Only gas inlet, outlet, and mounting surfaces of the part are ground. No proof testing is performed on this part due to the difficulty in simulating the thermally induced stress states which occur in use. A successfully cast part is illustrated in Figure 4.

Figure 4, Cast, sintered, and ground AGT-5 rectangular scroll.

Processing results from 8 casting lots have confirmed the attainment of the thin, uniform wall thickness from lot to lot. The data in Figure 5 summarize wall thickness measurements taken from locations throughout multiple green castings. Wall thickness variations within a

part are less than the variation shown here for lot-to-lot.

Figure 5, Uniform wall thickness has been achieved for multiple lots of scrolls.

Modulus of rupture bars cut from co-processed plates have been used to screen scrolls prior to shipment. Certification data for 3 lots are summarized in Figure 6.

Figure 6, MOR Certification data for SN252 scrolls.

Circular cross section scroll process development

The rectangular scroll fabrication process is being extended to circular cross section design. Circular scroll designs are available for both the hybrid engine and for the AGT-5 test bed. With Allison's support, KICC

has utilized polycarbonate mold patterns fabricated by solid free form processing. Patterns were provided to KICC by Allison based on component design data and shrinkage factors. The patterns, illustrated in Figure 7, were used to fabricate molds for both component designs, and provided a significant reduction in design-to-mold fabrication effort.

Figure 7, Polycarbonate scroll pattern for a) Hybrid engine and b) AGT-5 engine.

The new mold fabrication approach and the rectangular scroll drain casting techniques were applied initially to the circular scroll for the hybrid engine. Similar processing, materials and certification as those for the rectangular design are being utilized. The process has been modified for the component specific shape, i.e. mold and fixture modifications. The same processing techniques will also be applied to the circular scroll for the AGT-5, and scroll process development will continue to be performed to further improve process yield and control.

Appendix H

**1996 Annual Progress Report
Submitted by**

Kyocera Industrial Ceramics Corp.

and Prepared for

Allison Engine Company, Inc.

under the

**Hybrid Vehicle Turbine Engine Technology Support Project
(HVTE-TS)**

Contract No. DE-AC02-96EE50453

Note: This appendix contains the annual progress report as submitted to Allison. The document has been edited only where necessary to delete information that is deemed to be proprietary in nature.

KYOCERA INDUSTRIAL CERAMICS CORP.

1996 Progress Report

Objective/Approach

Kyocera Industrial Ceramics Corporation's (KICC) 1996 support of the Technology Support Program has focused on:

- delivery of AGT-5 gasifier turbine rotors and turbine scrolls, and
- development and demonstration of manufacturing technologies applicable to low cost, high volume production.

The manufacturing technologies being addressed include:

- developing a fabrication capability for circular cross section turbine scrolls,
- demonstrating a ceramic-to-ceramic joining method applicable to integral nozzle assemblies,
- applying Kyocera's Hybrid Molding process to turbine rotors

Accomplishments

Deliveries:

- Two SN253 20 bladed AGT-5 gasifier rotors passed NDE and spin proof test screening, and were delivered. These deliveries completed the Allison's requests for 5 parts. (These parts were subsequently successfully rig tested by Allison)
- Three rectangular cross section scrolls were delivered, bringing the total number of delivered parts to four. These deliveries

completed Allison's request. (These parts were subsequently successfully rig tested by Allison).

Process Development:

- Rectangular scroll casting and sintering processes were successfully applied to the circular scroll designs, and two circular cross section AGT-5 scrolls were delivered (3 requested)
- A Hybrid Molding die was obtained for the AGT-5 gasifier rotor, and forming iterations were initiated.

Discussion

AGT-5 Gasifier Rotor Fabrication

Although Kyocera had previously provided SN252 AGT-5 gasifier rotors fabricated in Japan under the previous AGT program, the aim of this effort was to provide rotors from Kyocera's newer SN253 material. To expedite deliveries, rotors were fabricated by solid slip casting, followed by densification and diamond grinding. Rotors were cast to near net shape to provide as-processed blades, and minimal machining of the hub and shaft surfaces. In 1995 KICC successfully fabricated three rotors, and qualified them through co-processed material

evaluation, NDE, and spin proof testing to 80,000 rpm.

In 1996, KICC completed two more rotors, fulfilling Allison's requested quantities. Figure 1 illustrates an as-sintered rotor.

Figure 1, Slip cast AGT-5 20 bladed rotor in the as-sintered condition.

AGT-5 Rectangular Cross Section Scroll Fabrication

AGT-5 rectangular cross section scrolls were fabricated in one piece by drain casting from the SN252 composition. With the exception of the gas inlet, outlet, and mounting surfaces, the majority of the part surfaces remain in the as-fired condition. No proof testing is performed on this part due to the difficulty in simulating the in-use thermally induced stress states. Parts are qualified via certification of co-processed test material, and FPI and radiographic inspections.

Initial fabrication efforts in 1995 resulted in two significant challenges: air entrapment voids entrapment, and non uniform wall thickness. During 1995, wall

thickness uniformity was achieved but voids continued to limit good processing yield. In 1996, mold fill and mold draining procedures were improvement eliminated most of the void occurrences. This improvement allowed the completion and delivery of three parts, completing Allison's delivery request. An example of a completed rectangular cross section scroll is shown in Figure 2.

Figure 2, Completed AGT-5 rectangular scroll.

Circular Cross Section Scroll Process Development

The drain casting process used to make the rectangular scrolls was adapted to provide scrolls with circular cross section design. The initial design available for process development was the scroll for Allison's hybrid vehicle propulsion engine. This component was the subject of development efforts throughout 1995, during which time the occurrence of voids plagued component completion. During 1996, the circular scroll component design for the AGT-5 engine also became available, and process development continued on both

scroll designs. Both fabrication efforts utilized polycarbonate mold patterns fabricated by solid free form processing. Patterns were provided to KICC by Allison, as shown in Figure 3.

a) AGT5 Scroll Pattern

b) Hybrid Engine Scroll Pattern

Figure 3, Scroll polycarbonate mold patterns

Throughout 1996, drain casting procedures were iteratively improved to achieve pore free components. Application of the improvements developed for the rectangular cross section scroll also provided improved results for the circular design scrolls. Consequently, scrolls of both designs were successfully cast and sintered, as

shown in Figure 4. Only the AGT-5 scrolls were carried through final grinding. Two completed scrolls of the AGT-5 configuration were delivered to Allison for rig testing.

a)

b)

Figure 4, Cast and sintered circular cross section scrolls: a) AGT-5 design, and b) Hybrid design

Ceramic to Ceramic Joining

The objective of this effort is to develop and demonstrate joining techniques which would be

applicable to the fabrication of an integral nozzle assembly. Kyocera is evaluating two methods to achieve this joint: 1) Joining materials in the "green" state, and 2) bonding materials after sintering. Both

methods would initially be evaluated by joining simple cylinders which could be machined into MOR bars for subsequent strength evaluation. If successful, the initial step would be followed by the joining of parts simulating the assembly of a nozzle assembly.

KICC elected to join hybrid molded material because this forming approach would be suitable for the production of the complex subelements of a nozzle assembly. Hybrid Molded cylinders of SN252 material were fabricated, with a portion of the lot processed through sintering, while the remaining cylinders were kept in the as-formed (green) condition. Both green and sintered cylinders were subsequently processed through joining processes. The process involved the application of Hybrid Molding slip between the two cylinders to be joined. The "assembly" was then processed through standard thermal processes to achieve the desired bond. During 1996, cylinders were joined, but low strengths were achieved, due to air entrapment in the joint. An improvement to the joining method to minimize the voids is to be evaluated in 1997.

Hybrid Molded Gasifier Rotor Development

The objective of this effort is to apply the Hybrid Molding process to the fabrication of SN281 axial rotors. Hybrid Molding is a proven process for high volume production, and SN281 is Kyocera's newer material for high temperature gas turbine applications. The task requires the development of the forming, drying

and dewaxing parameters to achieve complete mold fill without defects. This optimization is accomplished through die design and fabrication, and the performance of multiple molding cycle iterations. The task was initiated in 1996.

During this reporting period, KICC performed the initial die design, procured the die, and initiated forming cycle iterations. Because SN281 is a new composition to Hybrid Molding, with different viscosity characteristics than prior Hybrid Molded material, several iterations were required to determine the slip preparation and mold filling parameters needed to achieve complete mold fill. These efforts were successful, as illustrated in Figure 5. However, subsequent drying and dewaxing of the rotors revealed the presence of cracking in some of the rotor hubs. Thus, additional forming and drying cycle iterations are required to improve process yield. These iterations are planned to occur in 1997.

Figure 5, Hybrid Molded AGT-5 rotor processed through partial densification

Appendix I

Glossary/Abbreviations/Acronym List

GLOSSARY/ABBREVIATIONS/ACRONYMS LIST

°C	degrees Celcius
°F	degrees Fahrenheit
AGT	automotive gas turbine/Advanced Gas Turbine
AMPS	Allison Mobile Power Systems
ANL	Argonne National Laboratory
APU	auxiliary power unit
AS	aluminum silicate
AS800	ASCC silicon nitride
ASCC	AlliedSignal Ceramic Components
ATTAP	Advanced Turbine Technology Applications Project
BU	build up
CAD	computer-aided design
CCM	Contractors Coordination Meeting
CFDC	combined federal driving cycle
CIP	cold isostatic pressing
CO	carbon monoxide
CTAHE	Ceramic Technology for Advanced Heat Engines
CTE	coefficient of thermal expansion
CTR	cyclic thermal rig
DARPA	Defense Advanced Research Projects Agency
DoE	Department of Energy
DOE	design of experiments
E	Young's modulus
FOD	foreign object damage
FPL	fiber processing level
GM	General Motors Corporation
GTE	GTE Laboratories Inc.
HC	hydrocarbon
HEV	Hybrid Electric Vehicle
HIP	hot isostatic pressing
hp	horsepower
hr	hour
HTML	High Temperature Materials Laboratory
HVTE-TS	Hybrid Vehicle Turbine Engine - Technology Support
Hybrid	Hybrid Vehicle Propulsion System Program
I.D.	inside diameter
K _{IC}	fracture toughness
KICC	Kyocera Industrial Ceramics Corporation
ksi	kilopounds per square inch
LAS	lithium-aluminosilicate
LFA	large frontal area
lb/in. ²	pounds per square inch
lb/sec	pounds per seconds
LeRC	NASA Lewis Research Center (LeRC)

GLOSSARY/ABBREVIATIONS/ACRONYMS LIST (cont)

LFA	large frontal area
m	meter
MAS	magnesium-aluminosilicate
MIL-STD	military standard
MOR	modulus of rupture
MPa	megaPascals
mpg	miles per gallon
N1	gasifier speed
NAC	Norton Advanced Ceramics
NASA	National Aeronautics and Space Administration
NDE	nondestructive evaluation
No.	number
NOx	oxides of nitrogen
NT154	Norton silicon nitride
NT164	Norton silicon nitride
NT230	Norton siliconized silicon carbide
O.D.	outside diameter
ORNL	Oak Ridge National Laboratory
PC	personal computer
PIH	pressed-in-hub
PLC	programmable logic controller
P/N	part number
POS	probability of survival
PRD	pull rod die
psi	pounds per square inch
psid	pounds per square inch delta (pressure differential)
RIT	rotor inlet temperature
RPD	reference powertrain design
rpm	revolutions per minute
S/N	serial number
SEM	scanning electron microscopic
Si ₃ N ₄	silicon nitride
SiC	silicon carbide
SMIG	Schuller Moldable Insulation Generation
SN252	Kyocera silicon nitride
SOW	statement of work
temp	temperature
TIT	turbine inlet temperature
TVG	thermal variable geometry
UHC	unburned hydrocarbons
ULEV	ultra low emissions vehicle standard
VLFA	very large frontal area
WBS	work breakdown structure
XEDA	X-ray energy dispersive analysis