**ADVANCED MICROTURBINE SYSTEMS
PROGRAM
TECHNICAL FINAL REPORT**

**(COOPERATIVE AGREEMENT NO.
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FOREWORD

This Final Technical Report, prepared by Honeywell Engines, Systems & Services (Honeywell), Phoenix, AZ summarizes the activities completed by Honeywell and its suppliers under the Department of Energy (DOE)-sponsored Advanced Microturbine Systems (AMS) Program. Initial program activities under DOE Cooperative Agreement No. DE-FC02-00-CH11061 were performed by the former Honeywell Power Systems, Inc. (HPSI) division located in Albuquerque, NM. That division of Honeywell International, Inc. ceased business operations in September, 2001. Program activities for the descoped program were then transferred to Honeywell Engines, Systems & Services (HES&S) in Phoenix, AZ and completed under DOE Cooperative Agreement No. DE-FC26-00-CH11061.

This Final Report covers work performed by Honeywell International, Inc. divisions during the following reporting periods: Initial program – October 2000 through September 2001; Descoped program – February 2002 through April 2005.

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1. EXECUTIVE SUMMARY

1.1 Cooperative Agreement

Dept. of Energy (DOE) Cooperative Agreement DE-FC02-00-CH11061 was originally awarded to Honeywell International, Inc. – Honeywell Power Systems Inc. (HPSI) division located in Albuquerque, NM in October 2000 to conduct a program titled Advanced Microturbine Systems (AMS). The DOE Advanced Microturbines Systems Program was originally proposed as a five-year program to design and develop a high efficiency, low emissions, durable microturbine system. The period of performance was to be October 2000 through September 2005. Program efforts were underway, when one year into the program Honeywell sold the intellectual property of Honeywell Power Systems Inc. and HPSI ceased business operations. Honeywell made an internal decision to restructure the existing program due to the HPSI shutdown and submitted a formal request to DOE on September 24, 2001 to transfer the Cooperative Agreement to Honeywell Engines, Systems and Services (HES&S) in Phoenix, AZ in order to continue to offer support for DOE's Advanced Microturbine Program. Work continued on the descoped program under Cooperative Agreement No. DE-FC26-00-CH11061 and has been completed.

1.2 Initial Program and Descoped Program

To distinguish between the Advanced Microturbine Systems efforts, we refer to the efforts led by Honeywell Power Systems Inc. under Cooperative Agreement No. DE-FC02-00-CH11061 as the initial program, during the reporting period October 2000 through September 2001; and the subsequent efforts led by Honeywell Engines, Systems and Services under Cooperative Agreement No. DE-FC26-00-CH11061 as the descoped program, during the reporting period February 2002 through April 2005.

2. INITIAL PROGRAM TECHNICAL PROGRESS SUMMARY

2.1 Initial Program Objectives

During the initial AMS program efforts (reporting period October 2000 through September 2001), Honeywell Power Systems, Inc. (HPSI) located in Albuquerque, NM led a multi-disciplinary team consisting of multiple Honeywell business units, Precision Combustion, Inc., and the Dept. of Energy - Lawrence Berkeley National Laboratory (DOE-LBNL) to develop and demonstrate critical components, subsystems, and integrated systems that would achieve the program goals. The initial AMS program goals were as follows:

- High Efficiency – Overall thermal efficiency greater than 40 percent.
- Low Environmental Emissions – Nitrogen oxides (NO_x) emissions less than 7ppm and carbon monoxide (CO) and hydrocarbons (CH) emissions less than 25ppm.
- Multi-Fuel Options – Natural gas, liquid petroleum gas (LPG), diesel, kerosene, flare gas, and low BTU (biomass derived) fuels
- Improved Durability – 45,000 hrs. system service life and 11,000 hrs. before major overhaul.
- Economy – Cost less than \$500 per kW.

2.2 Initial Program Technical Progress

The initial AMS program kickoff meeting with DOE was held on December 7, 2000 at the HPSI Headquarters in Albuquerque, NM. Both the DOE Headquarters and Chicago Operations Program Managers and the Oak Ridge National Laboratory (DOE-ORNL) Materials Program Manager were in attendance, as well as the Core Team members and all other key business and technical program personnel. Honeywell reviewed the program organization, the technical outline of the program, reporting, lines of communication, and program budget. In addition, other related DOE DER programs were reviewed. A tour of the HPSI manufacturing and test facilities was conducted for the attendees.

HPSI then initiated a microturbine systems trade study. The first item accomplished was to determine the trade study goals. Next, the Large Integrated Turbine Group (LITG) Interface block diagram was completed. The interface block diagram is an important tool in defining each interface and controlling each interface within the Microturbine system. HPSI completed the systems preliminary block diagrams for a 1-1/2 spool Microturbine with a synchronous generator; a single-spool microturbine with a permanent magnet generator (PMG); a 1-1/2 spool microturbine with a PMG; a 1-1/2 spool microturbine with redundant synchronous generator(s); and a 1-1/2 spool microturbine with an inductive generator. HPSI then used these block diagrams as the basis for the trade studies.

Honeywell Engines, Systems and Services (HES&S) in Phoenix, AZ led Task I of the Trade Studies. Thermodynamic engine cycle models of non-recuperated, recuperated, and intercooled plus recuperated Brayton cycles for single-spool, 1-1/2-spool, and 2-spool engine configurations were developed. The model of the recuperated single-spool configuration was calibrated against existing performance data for the Honeywell Model Parallon75™ MicroTurbine. Appropriate scaling functions for all loss models (heat gates, pressure losses, mechanical losses, and component efficiencies) were developed and introduced into the cycle

analyses in order to allow parametric trade studies to be conducted for output power requirements in the range of interest from 150 to 500 kWe. Turbine inlet temperatures were varied between 1750°F and 2100°F. A parametric turbine-type selection logic (axial vs. radial turbine) based on aerodynamic performance, mechanical integrity, and manufacturability considerations and constraints was developed and introduced into the model. The logic also included preliminary component design algorithms, which were used to compute the basic dimensional parameters of the rotating aerodynamic components as well as to set the shaft speed.

A parametric cost model was developed which used these preliminary design parameters as well as the standard output of the cycle analyses to estimate fabrication cost in \$/kW for the given configuration(s). Preliminary parametric trade studies results were obtained for the recuperated as well as the intercooled plus recuperated single-spool and 1-1/2-spool configurations. Non-recuperated configurations were discarded, due to an inability to meet the specified performance objectives with reasonable turbine inlet temperatures and fabrication cost.

At the time of sale of the HPSI assets (see section 2.3) the trade study was not completed; therefore, no system configuration was downselected.

2.3 End of Initial Program and Transfer of Descoped Program

During the spring of 2001, Honeywell International, Inc. made a business decision to sell the intellectual property of HPSI, and that division then ceased business operations. Honeywell Engines, Systems and Services (HES&S) in Phoenix, AZ, working with the DOE Chicago Operations office then arranged to transfer the existing DOE Cooperative Agreement No. DE-FC02-00-CH11061 from HPSI to HES&S to continue Honeywell's support and work on the DOE Advanced Microturbine Systems program. Honeywell submitted the required official correspondence for the transfer request and an amendment was completed to make the necessary name change and revised Federal I.D. number, and to change the Project Director to Dr. Bjoern Schenk of HES&S. The descoped program efforts continued under DOE Cooperative Agreement No. DE-FC26-00-HC11061, beginning in February 2002.

To distinguish between the two Advanced Microturbine Systems efforts, we refer to the efforts led by the former Honeywell Power Systems Inc. division as the initial program (reporting period October 2000 through September 2001); and the subsequent efforts led by Honeywell Engines, Systems and Services as the descoped program (reporting period February 2002 through April 2005). Technical efforts completed under the descoped program are reported in the following sections.

3. DESCOPED PROGRAM TECHNICAL PROGRESS

3.1 Descoped Program Background

Under the original AMS proposal of October 2000 won by Honeywell Power Systems Inc. (HPSI) in Albuquerque NM, Honeywell Engines, Systems and Services (HES&S) in Phoenix AZ was one of the team members and the Honeywell Center of Excellence (COE) leading the turbine and compressor design efforts for the large microturbine. Honeywell Ceramic Components (HCC) in Torrance, CA was the team member and COE for ceramic component development. Following the decision by Honeywell International Inc. during the spring of 2001 to sell the intellectual property of HPSI, that division then ceased business operations. HES&S then proposed to DOE that the AMS program be continued as a descoped program under Task 2.15 titled “Ceramic Structure Design and Prototype Hardware” and Task 4.0, “Program Management and Reporting” in support of Task 2.15. The cooperative agreement was on suspension from November 2001 through February 2002. The so-called descoped program was then restarted under Cooperative Agreement No. DE-FC26-00-HC11061, covering the period of performance February 2002 through April 2005.

3.1.1 Descoped Program Statement of Work

The Statement of Work (SOW) for the descoped program covered the following activities, under Tasks 2.15 and 4.0 of the original proposal:

- Efforts were focused on manufacturing process issues involving the ceramic nozzle ring. Design of Experiments (D.O.E.s) were initiated to address forming of a net-shape component. Experiments were conducted to investigate tooling approaches, mold fill methods, and mold assembly and disassembly practices.
- Design Of Experiments (D.O.E.) activities were initiated to investigate the effects of heat treatment processes, including burnout, presintering, sintering, and crystallization on the component, achieving proper density and mechanical properties, correct crystalline phases, and controllable component distortion.
- Additionally, Honeywell initiated a process reproducibility task in which a ceramic generic nozzle ring design was processed on a continuous basis (multiple parts per week) to show the capability of the AS800 net-shape gelcasting process. This task was to continue throughout the descoped program effort, periodically incorporating process improvements as identified and demonstrated in the D.O.E.s.

3.2 Descoped Program Technical Progress Summary

3.2.1 Baseline Production Demonstration Using Generic Nozzle Ring

Gel casting was initially selected as the ceramic fabrication process for this program, due to promising capabilities to achieve a near net-shape, low-cost manufacturing process. Due to the extended time required for the design and fabrication of the tooling required to cast complex turbine nozzles capable of being used for engine testing, a simple component design, referred to as a generic nozzle ring (GNR), was initially used to conduct a baseline production demonstration effort. The purpose of this phase was to quantify the variation in the existing process with respect to top-level component requirements and to guide the process maturation efforts to be performed over the course of the program. Two GNR sizes were used: a full-scale GNR approximately 230 mm (9 inches) in diameter, and a sub-scale GNR, approximately 160 mm (6.3 inches) in diameter. The shape of these GNRs is shown schematically in Figure 1.

Figure 1. Generic Nozzle Ring Design.

3.2.1.1 GNR Casting Tooling

Relatively simple aluminum tooling was used for the initial GNR castings.

3.2.1.2 GNR Process Definition

A Process Map was completed which described the gelcasting process from raw material qualification through final inspection. Process inputs and outputs were identified and the inputs were classified as critical, controllable, standard operating procedure, or noise. The Process Map became a living document that was updated as the process was improved throughout this phase of the program.

The team also completed a component manufacturing Fault Tree and a Failure Modes and Effects Analysis (FMEA). The potential failure effects, failure modes, causes, and current controls were identified; the severity, occurrence, and detectability were scored; and a Risk Priority Number (RPN) was calculated. For high RPN causes, actions to reduce the occurrence or increase the detectability were identified and team members responsible for taking action were identified. The FMEA was reviewed and revised periodically and new failure modes and causes were added as they were identified. The RPN was revised as actions to reduce risk were completed and the probability of occurrence was decreased or the detectability was increased.

3.2.1.3 GNR Production Trial Campaign No. 1

The first round of GNR trial production (Campaign No. 1) was completed over a period of four weeks and shrinkage and dimensional conformance data was evaluated on 17 pairs of full-scale and sub-scale GNRs. For the baseline process, the within-part shrinkage variation ranged from 2.25 percent for the full-scale GNR, to 3.5 percent for the sub-scale GNR. Since this large within-part shrinkage variation needed to be eliminated prior to ordering engine component tooling, screening experiments and a D.O.E. were identified to determine both the cause of the shrinkage variation within the GNR and how to eliminate it.

The initial GNRs exhibited surface defects due to air bubbles trapped at the surface of the part during gelcasting. As a result, vacuum transfer molding was evaluated as a method of eliminating these air bubbles in the molded parts, and a bubble-free GNR was successfully molded using this approach. This forming technology was selected as the baseline molding method for GNR production during trial Campaign No. 2. A vacuum casting D.O.E. was conducted to evaluate the effect(s) of several factors, including fill port size, fill web size, and vacuum level. The response variables were shrinkage and casting defects.

3.2.1.4 GNR Production Trial Campaign No. 2

During GNR trial production Campaign No. 2, parts were cast using the vacuum-assisted casting process. Tool insert location accuracy was improved, the web thickness was increased, the hub and shroud vent port size was increased, and an improved drying cycle, having better control of the drying process, was implemented.

At the completion of Campaign No. 2, the full-scale and sub-scale GNRs were dimensionally inspected and scale factors were calculated. In order to justify the procurement of engine component tooling, the within-part scale factor must be uniform and controllable. The average scale factor(s) were calculated for the hub height, shroud height, hub inner diameter (ID) (upper and lower), and shroud outer diameter (OD) (upper and lower) dimensions. The upper dimension was measured near the top of the GNR and the lower dimension was measured near the bottom of the GNR. The diagram in Figure 2 shows the location(s) of the measurements.

Scale factors for the GNRs fabricated during trial Campaigns 1 and 2 are compared in Figure 3. The top graph shows the average of the six scale factor measurements for each GNR fabricated during the two Campaigns. The bottom graph shows the corresponding range of scale factors for each of the GNRs from the two Campaigns. The range chart shows the within-part variation of the scale factors. The average scale factors for all GNRs cast were fairly consistent, ranging from 1.282 to 1.286. The range chart showed a consistent range of approximately 0.059.

This means that the location that shrinks the most (the height) has a scale factor of 1.31 and the location that shrinks the least (the hub lower ID) has a scale factor of 1.25. This large difference in scale factor needed to be reduced unless a tool with variable scale factors could be built.

Figure 2. Location of Scale Factor Measurements on the Generic Nozzle Rings.

Figure 3. Campaign No. 1 and No. 2 Scale Factor Variation Within Parts.

3.2.1.5 Mechanical Properties Comparison

Mechanical properties were measured on test bars cut from the GNRs fabricated during Campaign No. 1 and the values were compared against historical data. In general, the properties were lower than the historical properties of AS800 gelcast material (generated using specimens cut from co-processed plates). The material properties from the GNR Campaign No. 1 specimens are summarized and compared against historical data in Table 1.

Table 1. GNR Campaign No. 1 Mechanical Properties Results.

AS800 Pedigree And Condition	Units	Average Strength	Weibull Modulus
GNR 1 Machined Strength	MPa	607	17
Historical Machined Strength	MPa	703	20
GNR 1 As-Processed Strength	MPa	483	13
Historical As-Processed Strength	MPa	614	13
GNR 1 Fracture Toughness	MPa*m ^{1/2}	7.9	---
Historical Fracture Toughness	MPa*m ^{1/2}	8.1	---

Mechanical properties were also measured on test bars cut from GNRs gelcast during the second GNR campaign. Campaign No. 2 properties were identical to Campaign No. 1 properties. Although there were many changes in the forming process between Campaign No. 1 and No. 2, there were no differences in the sintering cycle between the two campaigns. Fracture origins for machined test bars typically consisted of large grains for both Campaigns No. 1 and No. 2. Since the growth of large grains is primarily dependent on the sintering cycle, forming process changes would not be expected to have a significant effect on strength.

3.2.2 Development Nozzles

3.2.2.1 Nozzle Casting Tooling

The design and fabrication of the casting tooling for the engine component was conducted in parallel with the baseline production demonstration effort conducted on GNRs. A Failure Modes and Effects Analysis (FMEA) was performed on the tool design process and actions were taken to minimize risk. To evaluate the tool designers/fabricators that could be expected to meet the requirements of the gel casting process, a selection matrix was developed based on tooling experience and demonstrated capability. The highest-rated tool designers/fabricators were requested to quote on designing, fabricating, and proving the tools for an integral ceramic nozzle ring for engine testing. At the same time, the vendors were also asked to quote on design and fabrication of a tool to fabricate a gelcast ceramic integrally-bladed turbine disk (“blisk”). North Coast Tool was subsequently selected for design and fabrication of the nozzle tooling.

The greatest risks identified by the tooling FMEA were: (1) Minimizing corrosion and contamination of the tool material when exposed to the gelcasting slip mixture, and (2) Proper understanding of shrinkage, air entrapment, tool leakage, and tool sealing. Corrosion evaluation of potential tool materials was conducted by exposing material samples to the gelcasting slurry for multiple exposure cycles. A material system was selected that showed no indication of corrosion during the exposure testing.

Since the generic nozzle ring (GNR) design was not representative of the Honeywell Model 331-500 APU nozzle ring (the target engine application), a new tool, referred to as the nozzle development tool, was designed and fabricated. Although this tool was not classified as a production tool, it had airfoil cross sections and shroud thickness typical of the 331-500 APU components. In addition, the fill, venting, sealing, heating and cooling, and assembly and disassembly methods required for the development tool were also representative of a production tool. The approach to the tool design incorporated “best practices” based on Honeywell’s gelcasting expertise and North Coast Tool’s tool design expertise. A dedicated vacuum-assisted casting system was also designed and fabricated for use with the development tool. This system was different than the vacuum casting system used for the GNR tools, in that the development nozzle tool did not require a separate vacuum chamber. During tool fabrication, the tool assembly, casting, and disassembly process was documented and work instructions were created for use by the technicians who would be using the tool.

3.2.2.2 Nozzle Casting Process Capability

The initial ceramic nozzle development tool castings had areas of non-fill, areas where air bubbles accumulated, and a significant number of random air bubbles throughout the nozzle ring. Process improvements on slip de-airing, system evacuation, and filling and venting the tool eliminated many of the bubbles and non-fill areas. Additional tool venting improvements were identified to improve fill and reduce entrapped bubbles. Casting development trials were performed, the casting process was documented, and nozzle rings were produced at the rate of two per week. Figure 4 shows a typical development nozzle ring following disassembly of the tool after casting.

The development nozzle rings were dimensionally inspected both after drying and after sintering. An out-of-tolerance condition (OOT) was noted after drying, which worsened considerably after densification. Out-of-Roundness of the shroud after sintering ranged from 0.028 to 0.053 inch (0.71 to 1.35 mm), with values for the hub being somewhat better than the shroud, but overall unacceptable for engine test hardware. Airfoil profile data was collected on parts in the dry state and showed acceptable ranges of 0.004 to 0.010 inch (0.10 to 0.25 mm). However, data was not collected on the densified airfoils, as unexpected changes in the scale factor prevented the component measurement machine (CMM) program from accurately driving the probe for data collection. The scale factor range was improved compared to the generic nozzle rings (GNRs), but at 0.025 (compared to 0.059 for the GNRs), this failed to meet the goal of controlling the scale factor to three decimal places to ensure repeatability of component size within the part and from part-to-part.

G04-480-4

Figure 4. Gelcast Development Nozzle Ring After Disassembly of Tooling.

3.2.2.3 Gellation Improvement

Honeywell Laboratories (located in Morristown, NJ) focused on understanding and resolving some of the fundamental issues that impede commercialization of the gelcasting process. These efforts included: (1) Identifying compatible initiators that maintain slip stability and operate at lower concentrations to minimize gas evolution and reduce gelation temperatures; (2) Identifying mold surfaces and thermal controls to achieve uniform curing; (3) Eliminating voids by optimizing mold filling and sealing operations; and (4) Monitoring and simulating the gelcasting process. To this extent, a simplified, instrumented mold was designed, built, and successfully operated. This instrumented mold allowed dielectric monitoring of the gelation process. Evaluation of new initiator systems was also completed. One new initiator system was down-selected and implemented at Honeywell Ceramic Components (in Torrance, CA). Experience with the new initiator was very favorable in terms of eliminating a major source of variability in the gelation process. Further work with the previous initiator was discontinued.

3.2.3 Design Of Experiments (D.O.E.)

Several DOEs were conducted to improve process understanding as well as process control, and the DOEs and results are discussed in the following sections.

3.2.3.1 Forming D.O.E.

Two Forming D.O.E.s were executed in an attempt to understand how the key gelcasting process variables drive the response variables. Process variables investigated included casting pressures, gel time, and gel temperature. The response variables were scale factor variation and roundness. The D.O.E.s identified the following relationships:

- Key forming related factors were identified which led to establishing new levels which reduced scale factor variation in the dried part
- Scale factor range mean shifts were primarily being driven by powder lot characteristics
- Interactions between factors were identified and levels were set to minimize their effect(s).

3.2.3.2 Sintering D.O.E.

The Sintering D.O.E. was designed to explore the relationships between thermal parameters and dimensional control. Process variables evaluated included sintering temperature, sintering time, and ramp rates. The response variables were scale factor variation, roundness, and density. The experimental design maximized learning by incorporating several nested experiments into the primary D.O.E. The nested experiments evaluated the variables of burnout condition, part geometry, and sintering crucible type.

Factor interactions (including three-ways) dominated as main effects. The results suggested that scale factor variation was being driven by other factors that were not being evaluated, such as material composition, thermal gradients in the furnace or the part, or the crucible microenvironment. Over the experimental space explored, none of the D.O.E. factors or combination(s) of parameters examined resulted in significant distortion reduction as measured by scale factor range or roundness.

At this point, the sintering study moved to better understand exactly where in the sintering cycle that the densification occurs. This was accomplished by conducting partial sintering runs, consisting of sintering cycles terminated prematurely at different key times in the sintering cycle. The partial runs were conducted on both generic nozzle rings (GNRs) and on development nozzle rings. The within-part and part-to-part shrinkage was measured for each partial run to characterize sintering shrinkage.

Five temperature points, designated T1, T2, T3, T4, and T5 were selected for run termination. The experimental design also included the use of two different sintering crucible types and two different types of setters. The weight loss, density, and open porosity results for two different combinations of the experimental conditions are shown in Tables 2 and 3.

Table 2. Partial Sintering Run Results for GNR Using Type 1 Crucible and Type 1 Setter.

Temperature Termination Point	Weight Loss, %	Total Density, %	Open Porosity, %
T1	0.78	51.5	42.0
T2	0.98	65.7	28.5
T3	1.16	77.4	19.6
T4	1.48	89.0	0.1
T5	1.50	93.2	N/A

Table 3. Partial Sintering Run Results for GNR Using Type 2 Crucible and Type 1 Setter.

Temperature Termination Point	Weight Loss, %	Total Density, %	Open Porosity, %
T1	0.78	50.8	41.2
T2	0.97	61.6	31.1
T3	1.02	70.7	24.7
T4	1.26	81.7	17.0
T5	1.26	86.5	N/A

The results of the partial sintering runs showed that for this particular partial sintering cycle, the densification in crucible C2 lagged that in Crucible C1. However, full density (not shown) was attained in both crucible types in the complete sintering cycle.

The dimensions measured before and after sintering were used to determine scale factors for various dimensions of the parts. Figure 5 shows the results for the GNR densified in Crucible 1. The abbreviations used in this graph include those listed in Table 4.

Table 4. Abbreviations Used in Figure 5.

Abbreviation	Meaning
HLowSF	Scale Factor for Lower portion of Hub Diameter
HUpSF	Scale Factor for Upper portion of Hub Diameter
SLowSF	Scale Factor for Lower portion of Shroud Diameter
SupSF	Scale Factor for Upper portion of Shroud Diameter
SHtSF	Scale Factor for Shroud Height
HHtSF	Scale Factor for Hub Height

It is evident from the data in Figure 5 that each feature measured on the GNR exhibited a slightly different scale factor and that this difference became progressively larger throughout the sintering process. The scale factor for the shroud and hub height was always the greatest (maximum shrinkage), whereas the scale factor for the lower portion of the hub was always the least.

Figure 5. Scale Factor for Six GNR Dimensions as a Function of Termination Point in the Sintering Cycle.

3.2.4 Alternate Forming Methods

It became evident during the previously-discussed trials that the current gelcasting process was not capable of manufacturing ceramic engine components requiring high precision, accuracy, and good reproducibility. A variety of shortcomings of the baseline gelcasting approach were identified, including the following:

- Scale factor variation, both within the part and part-to-part
- Poor surface quality due to trapped bubbles or mold imperfections
- Difficult sinterability
- High costs of hard tooling (as there were indications that the abrasive nature of the slurry would result in low tool life).

The use of hard tooling also meant that any change in the casting density, such as might occur when changing powder lots, would automatically result in an out-of-tolerance part. To compensate, a knowledge base on how to change the solids loading to control the cast density needed to be developed. It was also judged that some of the scale factor variation was related to the sensitivity of gelcasting to drying shrinkage differences between thick and thin sections of the part.

As a result, it was decided to evaluate the process capability of alternative forming methods for manufacturing ceramic engine components. The following forming methods were selected for evaluation and comparison of scale factor uniformity and sintering distortion.

- Cold Isostatic Pressing (CIP) and pre-sinter machining
- Cold Isostatic Pressing (CIP) and green machining
- Slipcast and pre-sinter machining
- Gelcast net-shaped
- Gelcast and pre-sinter machining.

Cylindrical specimens (rings) similar in size to the sub-scale GNR outer shroud were used. The outer shroud diameter was approximately 160 mm (6.3 inches), the height was 50 mm (2 inches) and the wall thickness was 5 mm (0.2 inch). Four ring specimens of each material were evaluated, using the standard sintering cycle. The experiment was also designed to show any differences between sintering run and crucible type. First, two rings were sintered in a furnace run in two different crucibles (Type 1 and Type 2). A second sintering run was conducted on rings 3 and 4 using the same two crucibles (Type 1 and Type 2). This experiment gave the variation between crucible type and between sintering run.

The response variables included the following:

- Outside Diameter (OD) at the top, middle, and bottom of the ring
- Inside Diameter (ID) at the top, middle, and bottom of the ring
- Roundness, based on six diametrical locations
- Height
- True Position to OD mid position
- Scale factor for each measured feature
- Density and weight loss.

The following conclusions and observations were noted based on the results:

Scale Factor Range

- CIP < Slipcast << Gelcast
- Small amount of run-to-run variation
- Gelcast had the greatest part-to-part variation and CIP had the least

Roundness

- CIP and Slipcast << Gelcast
- Gelcast data showed more variation

True Position

- CIP < Slipcast << Gelcast
- Gelcast data showed more variation

Weight Loss

- Gelcast > Slipcast > CIP
- Type 1 Crucible > Type 2 Crucible

Density

- Type 1 Crucible > Type 2 Crucible

Although gelcasting allows for near net-shape, complex shape-making capability, it was determined that the scale factor variation for gelcasting was significantly higher than for the Cold Isostatic Pressing (CIP) or slip-cast processes. This large variation in scale factor, combined with the large size of the components required for the targeted engine application eliminated gelcasting as a viable forming approach for these components, as the required dimensional tolerances could never be achieved. Although the slipcast and pre-sinter machining process had been used successfully in the past to make individual engine quality blades and vanes, the casting of large billets required for integral nozzles and blisks components was always a low-yield process. As a result, the remainder of the program focused on the CIP forming process.

3.2.5 Cold Isostatic Pressing and Pre-Sinter Machining Process Development

The large variation in scale factor combined with the large size of the components required for the targeted engine application eliminated gelcasting as a viable forming approach. The alternate forming activity discussed earlier demonstrated that the scale factor range was minimized in CIPped material. This approach also ensured that dimensionally accurate parts went into the sintering cycle, and it offered an avenue for rapidly adjusting the machining process to compensate for non-uniform shrinkage or distortion during sintering as well as billet density changes related to raw material changes.

CIP pre-form machining can be accomplished immediately after the CIP process when the body still contains the CIP binder (i.e., green machining), or after binder burnout and presintering (i.e., pre-sinter machining). The current CIP binder system used by Honeywell Ceramic Components (HCC) was not developed for green machining and the resulting body is relatively fragile and susceptible to machining and handling damage. As a result, pre-sinter machining, an established production manufacturing process at HCC, was initially emphasized for the fabrication of integral nozzles and blisks. Although the initial emphasis was on pre-sinter machining, a D.O.E. was also planned to study improvements to the green machining process.

3.2.5.1 Pre-Sinter Machined Ceramic Nozzle Trials

CIPped nozzle blanks were pre-sinter machined into the engine nozzle component pre-form shape. Several airfoils of one of these nozzles are shown in Figure 6. Figure 7 shows the leading and trailing edge sides of a pre-sinter machined and densified, CIPped AS800 integral nozzle. This nozzle was final machined and inspected and found to be an engine quality piece of turbine hardware.

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Figure 6. Pre-Sinter Machined Airfoils in Integral Nozzle.

G04-480-7

(a) Leading Edges

(b) Trailing Edges

Figure 7. Engine Quality Pre-Sinter Machined AS800 Integral Nozzle.

3.2.5.2 Pre-Sinter Machined Ceramic Blisk Trials

Demonstration ceramic integrally-bladed turbine disks (“blisks”) – consisting of “paddle wheel” blisks with 12 simplified airfoils – were pre-sinter machined and densified for pre-sinter machining process development and dimensional conformance studies. A photograph of one of these parts after sintering is shown in Figure 8.

Figure 8. Pre-Sinter Machined Ceramic “Paddle Wheel” Demonstration Blisk After Densification.

3.2.6 Cold Isostatic Pressing and Green Machining Process Development

Achievement of an engine-quality AS800 integral nozzle via pre-sinter-machining of cold isostatic pressed (CIPped) stock material was a significant accomplishment. However, green machining of the same component offered the opportunity for significant reductions in cycle time and cost. As a result, the remainder of the program effort focused on the green machining and CIP approach. Tasks included determining the effect of solids loading and binder content on the spray-dried powder characteristics, as well as evaluating green machining parameters, sinterability, shrinkage uniformity/distortion, and mechanical properties.

3.2.6.1 Spray Drying Process Improvement

The initial process improvement efforts for the CIPped material focused on the spray-drying process used to make the granulated starting powder. A D.O.E. was conducted which examined the effect of four factors on the ceramic slurry feeding the spray dryer. The factors were related to milling profile, dispersant amounts, and binder additives. The outputs of the D.O.E. included slurry viscosity, tap density, and flow characteristics of the spray-dried powder, spray-dried powder yield, green density, sintered density, and strength properties. One of the four factors was shown to have a significant positive effect on the specified outputs.

A second D.O.E. was performed incorporating the results of the first D.O.E. with inputs from the CIP process. The D.O.E. outputs included slurry viscosity, tap density, and flow characteristics of the spray-dried powder, spray-dried powder yield, and green density. The resultant process yielded a CIPped body with roughly a 6-percent increase in green density over the baseline process, as shown in Figure 9. Sintering trials demonstrated that this body was easy to densify.

Figure 9. Summary of D.O.E. Results on CIP Process – Green Density of CIP Specimens.

It was demonstrated that AS800 spray-dried powder could be successfully prepared over a range of slurry properties, including solids loading and binder content. All of these spray-dried powders had acceptable tap densities and flow characteristics. As shown in Figure 10, a low solids loading (right photo in Figure 10) resulted in a finer spray-dried powder and a lower CIPped density, while a high solids loading (left photo) resulted in a coarser powder and a higher CIPped density. The powder choice for component fabrication depends on the machining characteristics of the CIPped billet, as well as the distortion and mechanical properties exhibited by the densified material.

Spray-dried AS800 powder was selected for use in the turbine component fabrication demonstration. The specific powder used was selected primarily on the basis of mechanical properties (discussed below), since some powders resulted in a high percentage of pore failures and lower strength in test bars machined from CIPped billets. It is expected that the powder selected will consistently result in average test bar strength in excess of 700 MPa, and a Weibull parameter in excess of 20.

G04-480-10A

Figure 10. Photomicrographic Comparison of Spray-Dried Material.

3.2.6.2 Green Machining Process Improvement

Although the current CIP binder system was not optimized for green machining, increasing the binder content by a factor of 3 to 4 demonstrated the capability for successful high-speed green machining. A D.O.E. was performed which compared the machineability of a green body with the standard amount of binder to that of a green material with a high binder content. A simple airfoil shape (shown in Figure 11) was used for this D.O.E. The D.O.E. evaluated the effects of spindle speed, feed rate, and scallop height on the machineability of the green material. The results showed that the higher binder content material could be machined significantly faster without chipping or loss of surface quality. No difference in tool wear could be detected from the results of this experiment.

G04-480-11

Figure 11. Simple Airfoil Shape Used for Green Machining D.O.E.

Using the higher binder content material, a baseline for high-speed green machining parameters was developed. As shown in Figure 12, a complex turbine rotor airfoil, representative of the engine component, was used for a D.O.E. which improved the green machining process. Input parameters to the D.O.E. included: tool material, tool geometry, feed rate, spindle speed, cooling, depth of cut, and tool path (direction of cut and step over distance). The D.O.E. output parameters included: surface quality, tool life, and cycle time. Based on this D.O.E., a green machining specification was developed.

Figure 12. Complex Turbine Rotor Airfoil Green Machined From CIPped Billet.

3.2.6.3 Distortion Ring Trials

All of the CIPped billets made from various spray-dried powders achieved complete densification in the standard sintering cycle used for AS800. Distortion rings, approximately 160 mm (6.3 inches) in diameter by 50 mm (2 inches) high with 5 mm (0.2 inch) wall thickness, were green machined and densified to evaluate distortion as measured by roundness and cylindricity. Figure 13 shows some of the roundness measurement results, in which the rings were measured at the bottom, top, and mid-span. In general, the roundness achieved in the green-machined CIPped rings demonstrated the potential to meet the requirements for turbine engine hardware, and was consistently superior to that achieved by any other forming method evaluated under this program.

G04-480-13A

Figure 13. Average and Range Roundness Data for Distortion Rings (OD/ID Inclusive).

3.2.6.4 Mechanical Properties Trials

This task was primarily focused on relating the spray-dried powder characteristics to the room temperature strength and failure origins determined on flexural test bars machined from dense, CIPped billets. The goal was to achieve a high-strength material (>700 MPa), with a high Weibull value (>20), which failed predominantly from large grains. The challenge was to minimize the pore size in the CIPped material so that pores were not strength limiting. Table 5 lists some of the screening data obtained for several combinations of spray-drying process (A, B, C, or D), solids loading (a, b, or c), and binder level (a or b).

Table 5. Strength Results Comparison for Several Spray-Drying Processes.

Spray Dry Process	Solids Loading	Binder Level	Room Temperature Fast Fracture, MPa	Weibull Value	Percentage Large Grain Failures
A	a	a	698	16	24
A	a	b	652	25	19
A	c	a	692	24	18
A	c	b	640	19	5
B	a	b	632	16	11
B	b	b	616	23	19
C	a	b	715	19	85
D	a	b	706	24	91

At this stage of the research, it appeared that spray-drying process C or D held the most promise for meeting the requirements, primarily due to the combination of high strength and high percentage of large grain failures.

3.2.7 Turbine Component Fabrication Demonstration

A green-machined turbine component demonstration was initiated for both an integral nozzle and an integrally-bladed turbine rotor (“blisk”). Tooling for CIPping the billets for green machining stock was procured, and a large quantity of spray-dried powder was stockpiled to support the component demonstration.

3.2.7.1 Nozzle Fabrication Demonstration

Figure 14 shows the green machined, AS800 integral nozzle that matches the requirements of the Honeywell Model 331-500 APU. Figure 15 shows the densified ceramic nozzle. Several integral nozzles have been fabricated and await final machining prior to engine testing.

**Figure 14. Green Machined Ceramic Integral Nozzle.
(Left) Leading Edges; (Right) Trailing Edges.**

Figure 15. Green Machined Integral Nozzle After Densification.

3.2.8 Ceramic Blisk Fabrication Demonstration

Figure 16 shows the green machined, AS800 ceramic blisk that matches the requirements of the Honeywell Model 331-500 APU. Figure 17 shows the densified blisk. Several integral blisks have been fabricated and await final machining prior to engine testing.

(a) Leading Edges

(b) Trailing Edges

Figure 16. Green Machined Blisk. (Left) Leading Edges; (Right) Trailing Edges.

G04-480-17

Figure 17. Green Machined Blisk After Densification.

4. CONCLUSIONS AND RECOMMENDATIONS

Under the Dept. of Energy Advanced Microturbine Systems Program, Honeywell Power Systems, Inc. (former division located in Albuquerque, NM) completed an advanced microturbine systems study in the initial program phase under Cooperative Agreement No. DE-FC02-00-CH11061 during the reporting period October 2000 through September 2001. In subsequent efforts led by Honeywell Engines, Systems and Services (located in Phoenix, AZ) under Cooperative Agreement No. DE-FC26-00-CH11061 during the reporting period February 2002 through April 2005, a manufacturing process for ceramic gas turbine engine components was demonstrated that was capable of meeting the dimensional and mechanical properties requirements and has the potential for meeting the cost targets for gas turbine applications.

The high aerodynamic performance that enables advanced microturbine systems requires exacting dimensional control of flowpath surfaces, especially in the turbine hot section. Although the gelcasting approach to fabricating ceramic hot section components has the potential to be a low-cost, near-net-shape manufacturing process, the gelcasting additives and tooling evaluated under this program were not shown to be capable of fabricating components meeting the required dimensional tolerances. As a result, an approach utilizing cold isostatic pressing (CIPping) and green machining was developed, and in a relatively short period of time was successfully used to fabricate engine-quality ceramic hardware. Although this approach involves multi-axis machining to achieve the basic component shape, it is anticipated that green machining can be performed at cutting speeds high enough to result in an economical process. Assuming that engine components made by this process will consistently meet the dimensional requirements, the next concern is adequate material properties. Mechanical property testing has demonstrated that a high-strength, high-Weibull material (Honeywell AS800) can be achieved by CIPping and the low occurrence of test bar failures initiating at pores in some formulations indicates that satisfactory properties can be achieved.

This program is now positioned to enter a material testing and component validation phase. The material formulation (binder, solids loading, spray-drying process, CIP parameters, etc.) needs to be fixed and material test billets and additional components need to be fabricated. Achievement of a material database for the resulting fixed process, along with successful rig and engine testing of trial components will justify the incorporation of ceramic components into a production engine for qualification tests.

At the present time, Honeywell intends to continue development under internal funding. Beginning in 2005, Honeywell will evaluate the ceramic integrally-bladed turbine disks (“blisks”) and nozzle rings produced in the DOE Microturbine program. We plan to spin test ten blisks to verify the predicted burst speed. If the actual burst speed is greater than the established blisk proof test speed, then a blisk that has passed the proof speed test and a ceramic nozzle will be subjected to a series of tests in a modified Honeywell production Model 331-500 Auxiliary Power Unit (APU). Tests will be conducted to validate the design, validate the mechanical loads, and validate the ceramic structures. An Accelerated Mission Cycle test and a 150-hour block operational test will also be completed. Honeywell will share the results of the internally-sponsored evaluations and any subsequent testing thereafter with the DOE Headquarters Program Manager. Honeywell will attend the 2005 DOE Annual Meeting.