Honeywell

Silicon Nitride Production Readiness Campaign

In Support of the

U.S. DOE Advanced Microturbine Systems Initiative

(Cooperative Agreement #DE-FC02-00CH11061)

Honeywell Engines, Systems & Services
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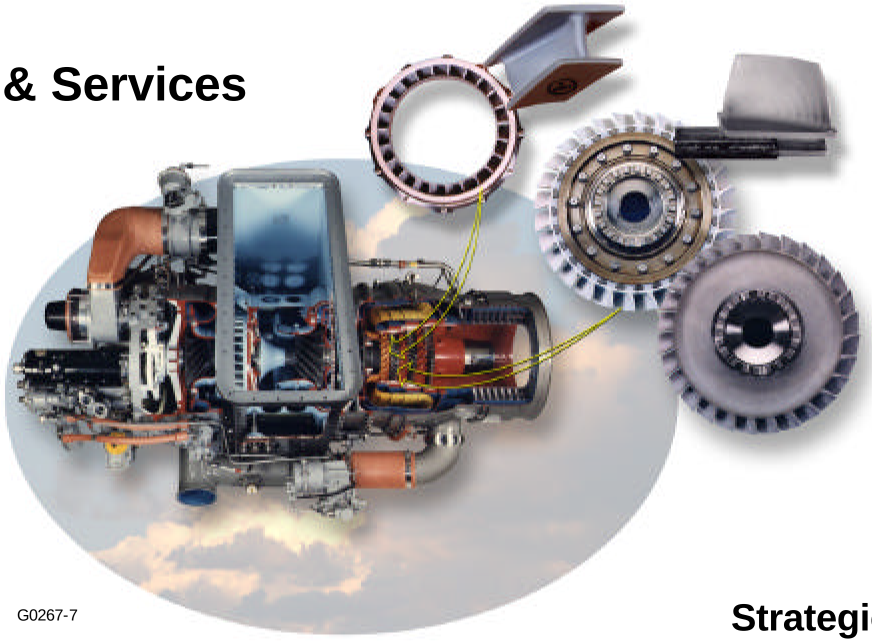

U.S. DOE EBC Workshop
November 6-7, 2002
Nashville, TN, USA

Advanced Technology

G0267-7

Strategic Technology

Core Team Members

- **Honeywell Engines, Systems & Services**
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  - **Lauri Strom, Materials & Process Engineering, Phoenix**
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  - **Dr. Chien-Wei Li, Advanced Materials R&D, Morristown**
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- **Fraunhofer Institute for Ceramic Technologies and Sintered Materials (IKTS), Dresden, Germany**
  - **Dr. Hagen Klemm, Silicon Nitride Materials Development**
- **H.C. Starck Ceramics, Selb, Germany**
  - **Dr. Gerhard Woetting, Materials R&D**
- **U.S. Department of Energy**
  - **Debbie Haught, Washington**
  - **Steve Waslo, Chicago**

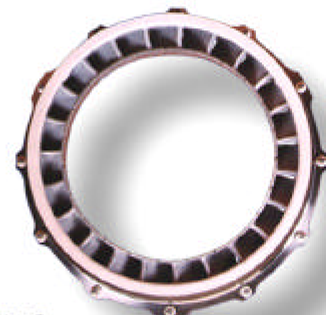
Agenda

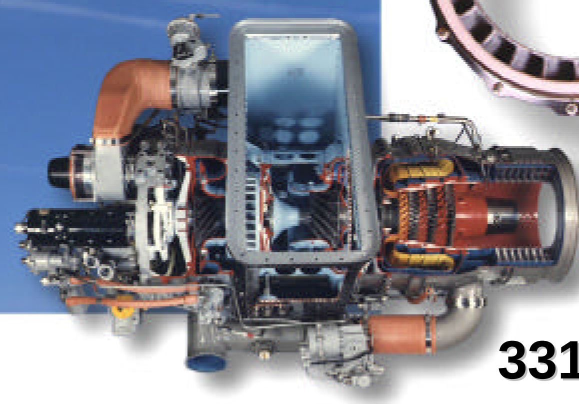
- Introduction
- Production Readiness Campaign Overview
- Generic Nozzle Ring Campaign I and II
- Forming Experiments
- Tool Design Methodology
- Gelcast AS950EXP Development
- Wrap-Up

Ceramic Nozzle Technology Being Proven Through Active Field Evaluation Program

- FAA certification in 3Q99
- Received OEM approval for field test
- Evaluation intended as technology demonstration, not as pre-production for this APU model
- Two-year field evaluation schedule
- First two units in commercial revenue service on Lufthansa Airbus A300-600 since 1Q 2000
- Third unit in commercial revenue service on Royal Jordanian Airbus A300-600 since 1Q 2002

Cumulative Time on Ceramic Nozzles as of Nov. 1, 2002:
10,000+ hours and
10,000+ cycles





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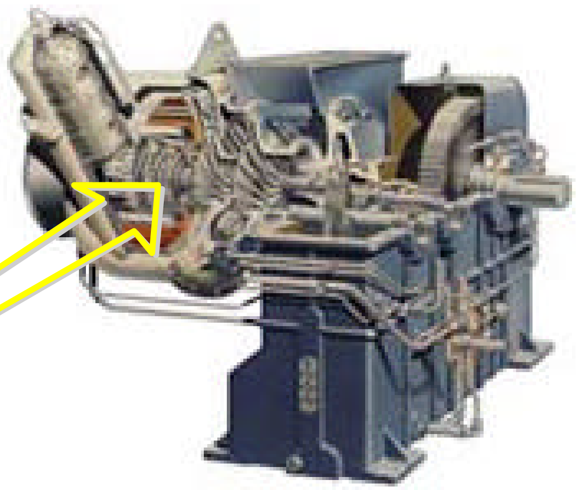
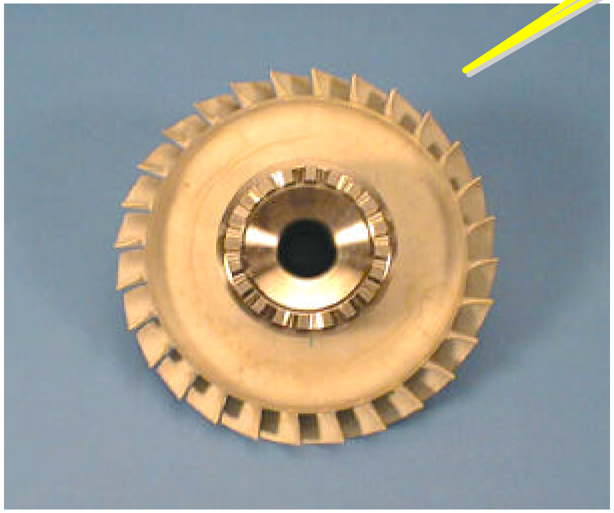
Ceramic Blisk Field Evaluation Initiated On Industrial Engine Platform

Field test goal:

- 10,000+ hours (2 year max) of continuous operation in Co-Gen application
- Installed in a Honeywell ASE8-800 at Questar Utilities Co. (Salt Lake City, UT)





Status:
- Field test started January 30, 2002
- Currently accumulated 6,100+ hours as of November 1, 2002.

Another Ceramic Industry-First for Honeywell

Principal Commercialization Barriers

- Inadequate oxidation protection technology
- Inadequate facilities available to screen candidate substrate material and novel coating systems prior to high risk engine tests
- Immature low-cost near-net-shape fabrication processes
- Insufficient publicly available material design database of latest ceramic material vintage
- Unproven tip rub resistance
- Lower impact resistance than single crystal alloys
- Immature Cooled Component Fabrication

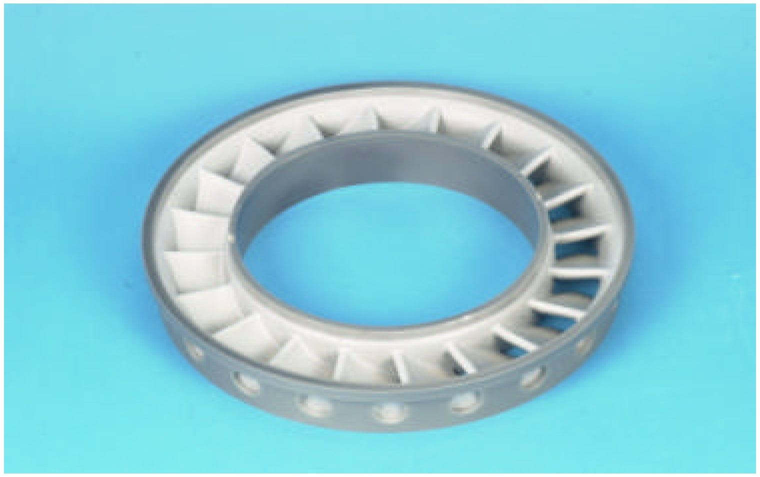
Honeywell's IR&D and Government-Funded Program Suite Addresses All Barriers And Enables Accelerated Commercialization of Ceramics in Small Gas Turbines

Motivation and Strategy

- **Original Honeywell AMS contract did not adequately address critical ceramic component commercialization requirements**
- **New program strategy was required to develop the infrastructure and materials & process engineering disciplines necessary to overcome those barriers, which currently prevent structural ceramic component commercialization in advanced heat engines**
- **Revised program plan provides approach to resolve each of these issues and follows a natural progression based on past DOE-funded efforts at HES&S**
- **Focus will be on very near-term ceramic component production capability for premium gas turbine applications such as advanced industrial microturbines for distributed power generation**
- **Effort will draw heavily on "lessons learned" from the problems and successes on previously completed ceramics development and demonstration programs**

Mature Low-Cost Near-Net-Shape Manufacturing Of Large Integral Ceramic Components

Nozzle Rings



Blisks

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Radial Turbine Wheels

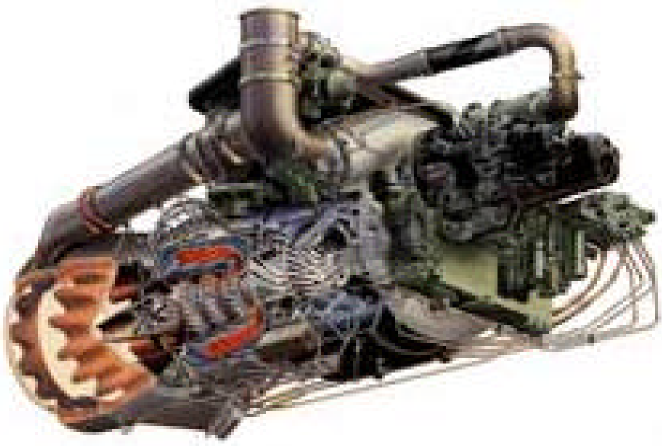
Work Breakdown Structure

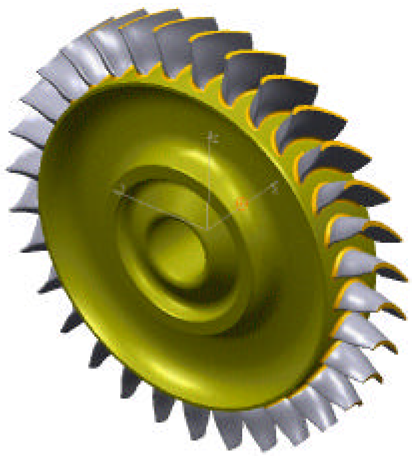
Honeywell ES&S
Advanced Microturbines Systems
Related Activities

- **DOE AMS Program Award 2002 & 2003**
  - **Process Maturation**
  - **Database Generation**
  - **Oxidation Protection**
  - **Project Management**
- **Honeywell IR&D & Potential Government Programs**
  - **Design Processes**
    2002
    Honeywell IR&D
  - **Oxidation Protection**
    2002 & 2003
    Honeywell IR&D
  - **Engine Testing**
    2004 Optional
    DOE AMS Program Phase
  - **Field Evaluation**
    2004 & 2005 Optional
    DOE AMS Program Phase
- **Micoturbine OEM Support** (Selected OEM Through Separate Subcontracts)
  - **Ceramic Component Design Consultation**
  - **Life Prediction**
  - **Process Development** (Radial Turbine Components)
  - **Parts Fabrication**
  - **Database Generation** (Required)
  - **Spin Pit Testing**

Honeywell IR&D Programs

- Sintering Cycle Optimization
- Alternative Manufacturing Approaches
- Next Generation Silicon Nitride Material Development
- Next Generation EBC Development
- Ceramic Hot Section Component Designs
- Design/Lifing Methodology Refinements
- Development Engine Testing
- Field Evaluation





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Honeywell Proprietary

Top Level Summary

- **Benefits to Selected Microturbine OEM**
  - **Delivery of high quality ceramic components**
  - **Mature low-cost near-net-shape fabrication process**
  - **Adequate oxidation protection**
  - **Joint development of publicly available material design database**
  - **Access to Honeywell ceramic gas turbine design & life prediction expertise**
- **Benefits to the Government**
  - **Protect past investment and build upon successes in ceramic gas turbine technology development**
  - **Participation of ORNL in key development activities**
    - **component characterization & database generation**
    - **oxidation protection system evaluation**
  - **UDRI (Mechanical Testing in Moist Environment)**
  - **Academia (Northwestern, Lehigh, UC Boulder) (EBC Fundamentals)**
  - **Argonne National Lab (NDE Characterization of EBCs)**

Production Readiness Development (Process Maturation)

Objective

- Develop a manufacturing process capable of producing silicon nitride turbine hardware that meets the customers requirements for quality, delivery and cost
- Develop a process that is robust to process noise
- Understand the capability of the process
- Production ready 331-500 APU nozzle rings and blisks by 2004

Success of Production Readiness Program is essential to future ceramic development at Honeywell

Process Targets and Specifications

- **Dimensional Control**
  - **Meets drawing requirements**
- **Mechanical Properties**
  - **Meets Honeywell Material Specification**
- **Defect Free**
  - **Meets drawing requirement, EMS53192 and EMS52309**
- **Cost**
  - **50-75% cost reduced over cooled metallic components**
- **On Time Delivery**
  - **95% on time delivery to customer request**

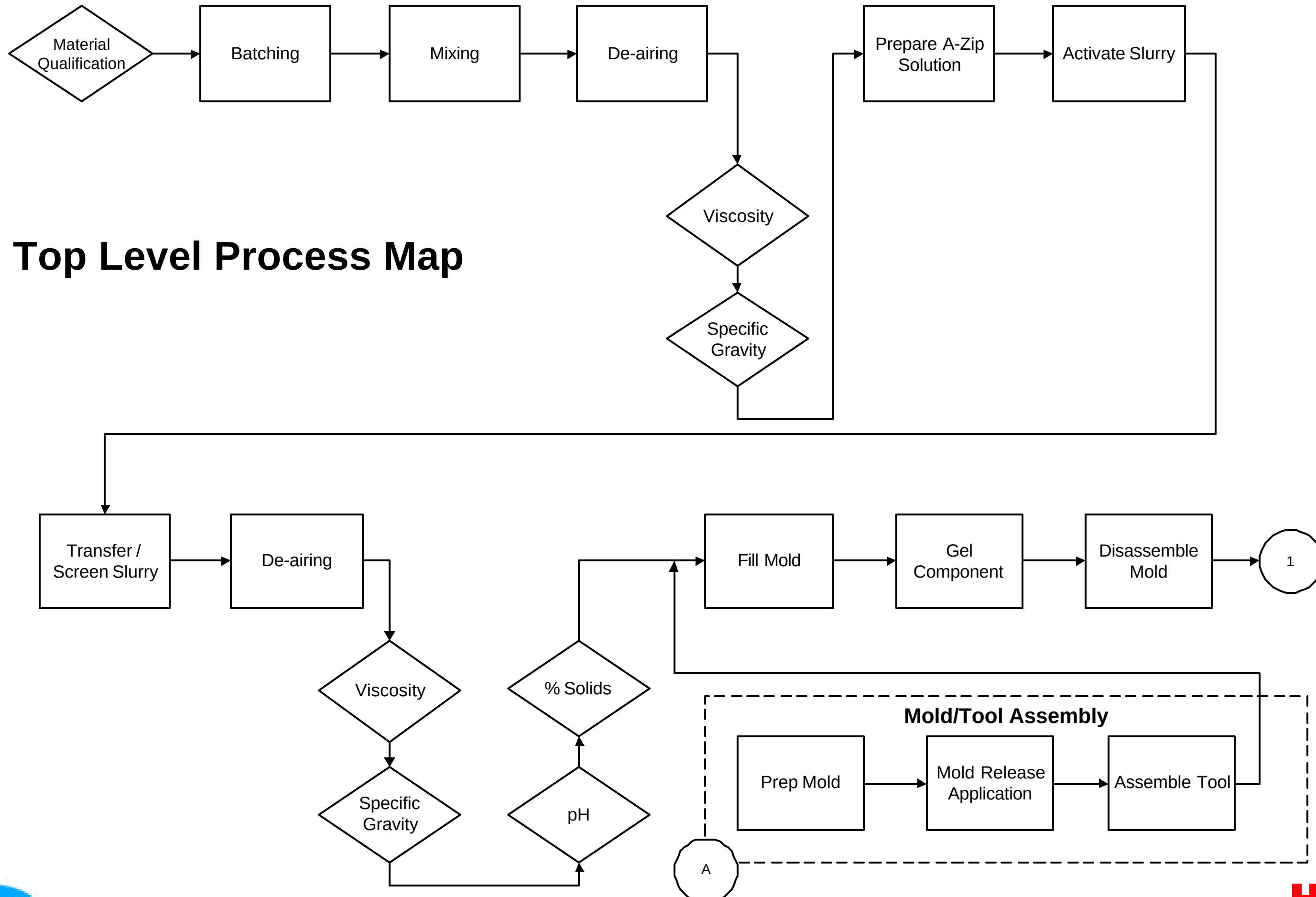
Six Sigma Will Enable Production Readiness

- **Six Sigma tools are being utilized to improve processes**
  - **Thought Process Maps**
  - **Process Mapping**
  - **Quality Function Deployment (QFD)**
  - **Fault Tree**
  - **FMEA - Failure Modes and Effects Analysis**
  - **Measurement System Analysis**
  - **Control Charts / Capability Analysis**
  - **DOE - Design of Experiments**

Gelcasting Process Development

- **Gelcasting process map including process inputs and outputs completed**

Top Level Process Map



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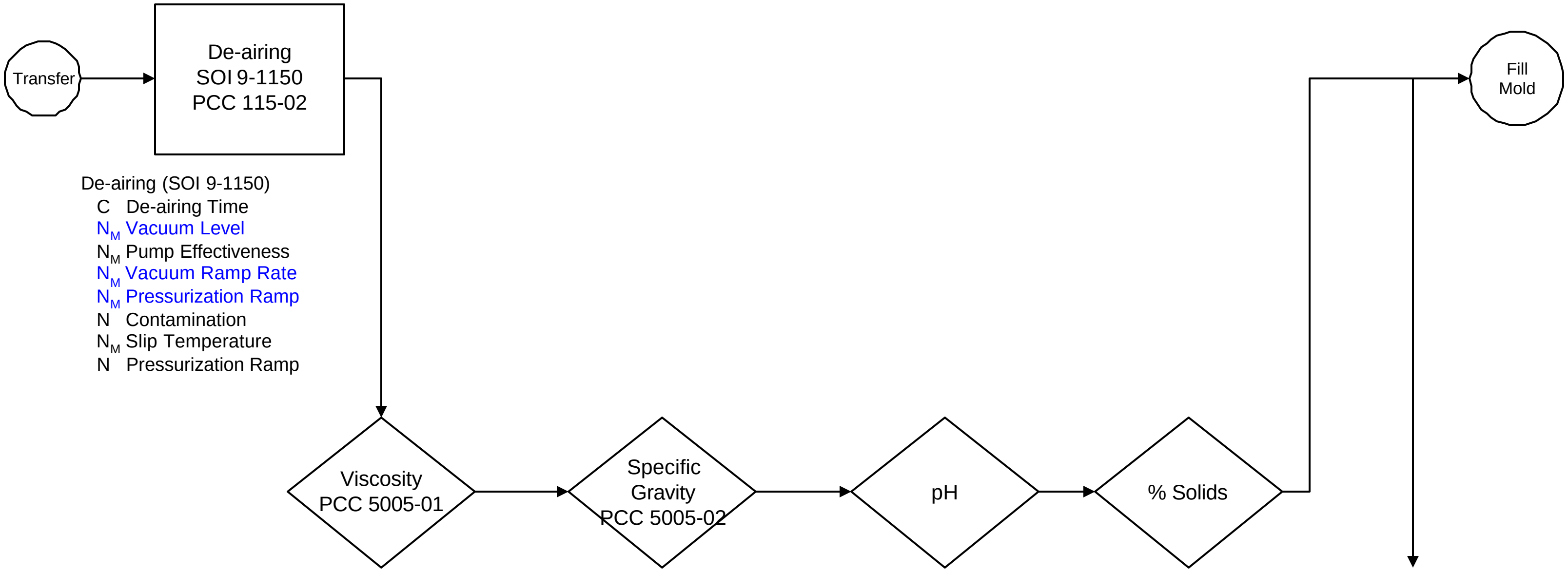
Detailed Process Maps Identified Process Inputs



De-airing (SOI 9-1150)
- C De-airing Time
- N_M Vacuum Level
- N_M Pump Effectiveness
- N_M Vacuum Ramp Rate
- N_M Pressurization Ramp
- N Contamination
- N_M Slip Temperature
- N Pressurization Ramp

Viscosity (PCC 5005-01)
- S Operator Technique
- C_S Spindle Size [#2]
- C_S Spindle Speed [30, 12, 3 rpm]
- C_S Container Size [150 ml Griffin beaker]
- C_S Depth of Immersion
- C_S Viscometer
- C_S Measurement Technique
- N Bubble Formation Under Spindle
- N_M Slip Temperature
- N_M Time Before Measuring
- N_M Measuring Time
- N_M Viscometer Accuracy

Specific Gravity (PCC 5005-02)
- S Operator Technique
- S Tared Balance
- S Measurement Flask [100.0 +/- 0.5 cc]
- C_S Measurement Technique
- N Entrapped Air
- N_M Time Elapsed Before Starting Test
- N Meniscus Profile Consistency

Fill Mold outputs
- I_N Activated Slip
- I_N Minimum Trapped Air
- I_N Sufficient Working Time
- I_N No Spot Gelation
- I_X Viscosity
- I_X Specific Gravity
- I_X % Solids
- I_X pH

KEY

X	Critical
C_X	Important Controllable
C_S	Controllable & Fixed
C	Controllable
S	Standard Operating Procedure
N_M	Noise , Measurable
N	Noise
I_X	Measured Output
I_M	Measurable Output
I_N	Non-Measurable Output

Generic Nozzle Ring Production Demonstration

- **Generic Nozzles Rings (GNRs) are being utilized to:**
  - **Understand the variation in AS800 baseline process**
  - **Understand how process inputs influence the process outputs**
  - **Understand the acceptable input levels to achieve the desired output**
  - **Gain production experience**

GNRs are being produced on a regular basis

Sub Scale (7 3/4 inch diameter)

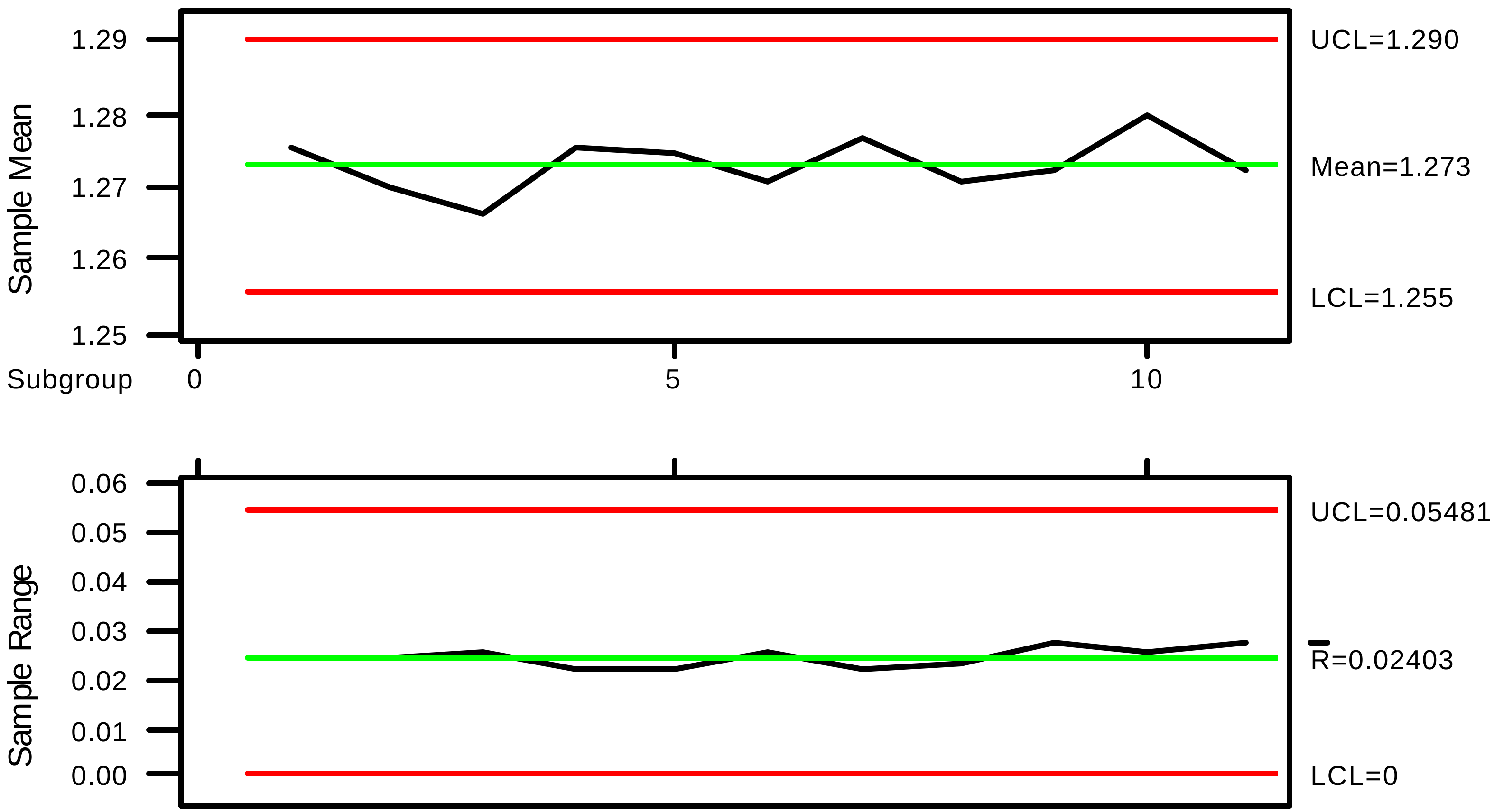
Full Scale (11 inch diameter)

Process inputs and outputs collected at each process step on every part

Dimensional data collected at five conditions

Variation Must Be Improved

GNR 1 Full Scale - Scale Factors on Diameters



Within Part Variation Must Be Improved

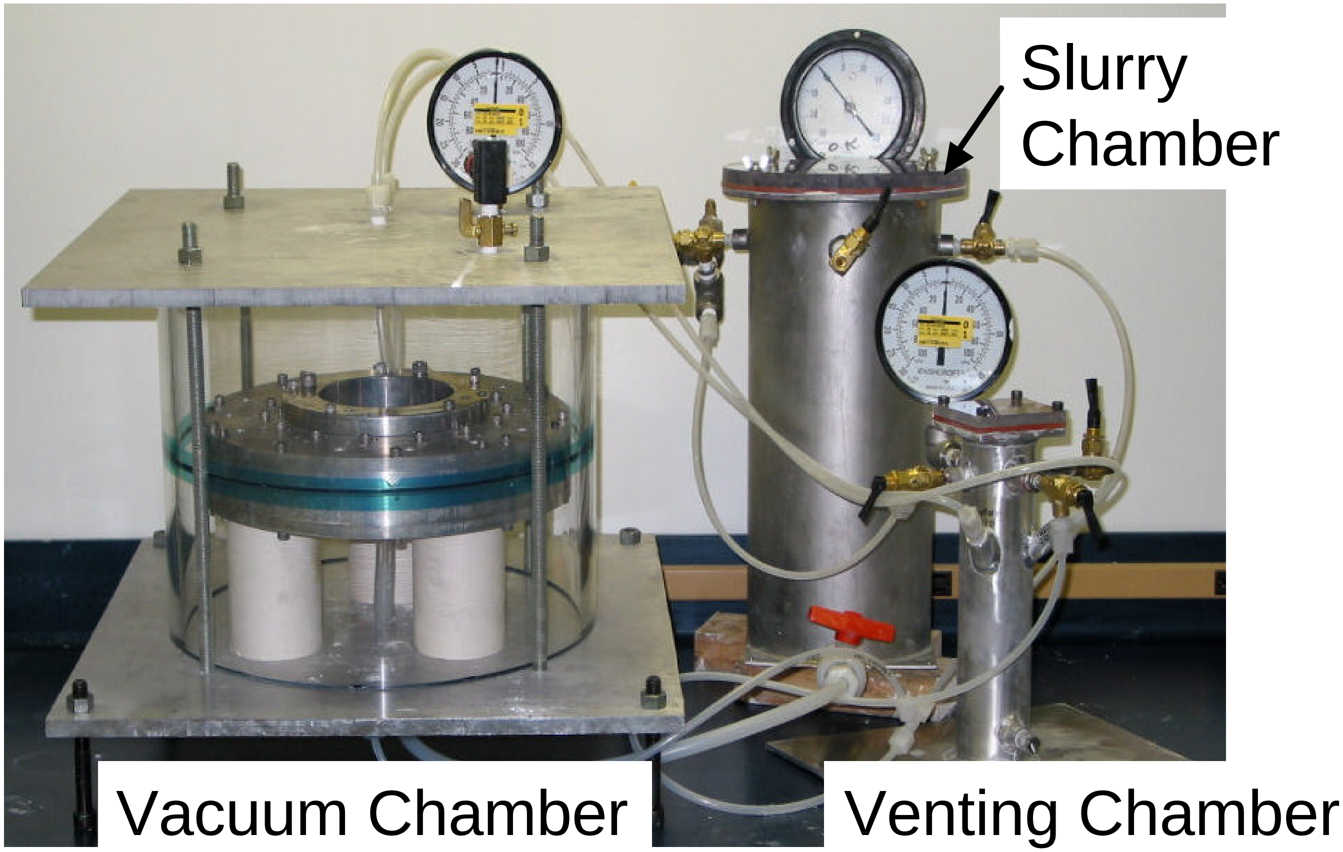
- GNR 1 established the baseline process
- The baseline process capability is not acceptable for production hardware
- Variation must be significantly reduced
- Changes to baseline process were identified for implementation in GNR 2

Post GNR 1 Screening Experiments and Evaluations

- **Determine if Vacuum Assisted Casting can be used to eliminate casting defects and reduce within part shrinkage uniformity**
- **Design an effective vacuum assisted apparatus for use with the gel casting process**
- **Determine effect on dimensions**
  - **Vacuum casting**
    - **flat vs tilted 4 degrees**
    - **center cone in vs center cone removed**
  - **Gravity casting**
    - **spin**
    - **vibration**
- **Green Density variation**
  - **Destructive analysis indicated a 0.5% between hub and shroud**
- **Dimensional change during drying**
  - **Real-time CMM analysis showed differential shrinkage rates between hub and shroud during early stages of drying process**

Vacuum Fill Utilized On Go-Forward Basis

- **Vacuum Assisted Slurry Transfer (VAST 1.0) - a process similar to vacuum transfer molding is utilized to address**
  - **Molding defects**
  - **Mold filling**



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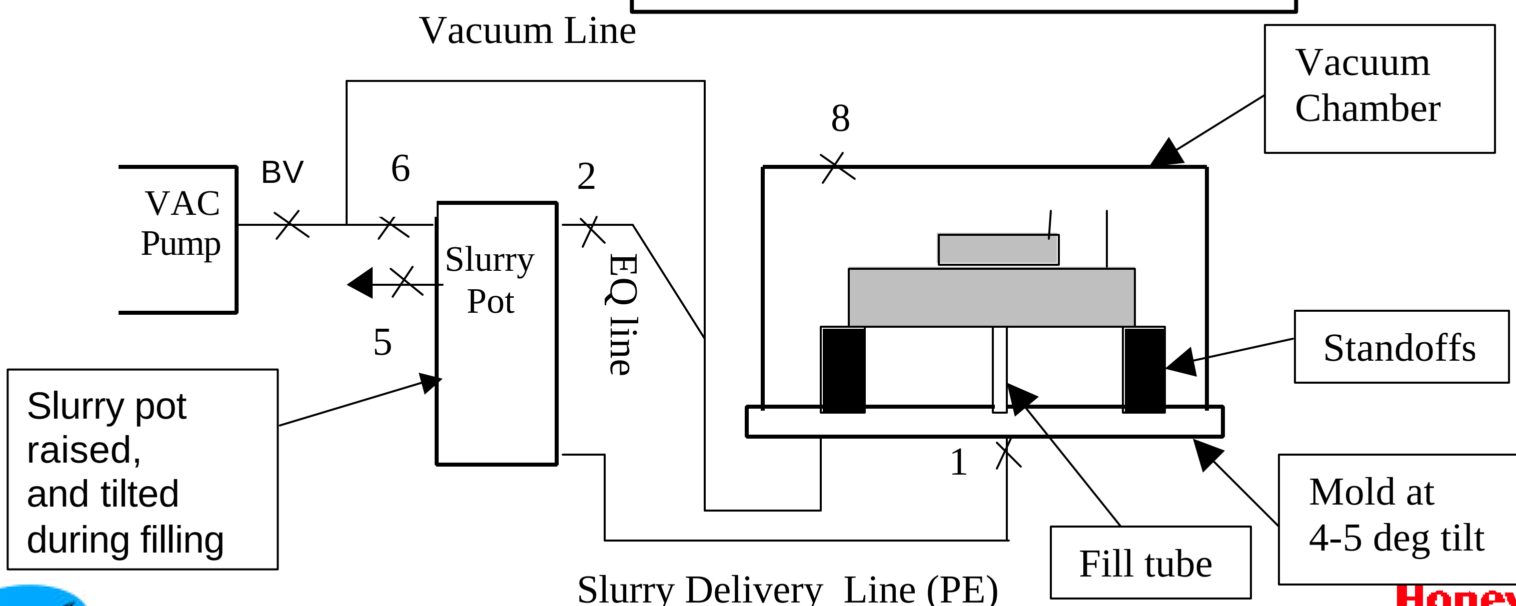
GNR 2 Has Incorporated Process Improvements

- **Vacuum casting to eliminate bubble defects**
- **Tool insert location accuracy**
- **Web thickness increased**
- **Hub and Shroud vent port size increased**
- **Improved drying cycle and better control of drying process**
- **Leave the tool center insert in place to prevent premature drying during demolding**

Schematic of VAST 2.0 system



General Risk Mitigation Activities

- **Screening Experiments**
  - **Modify current burnout cycle**
  - **Investigate sintering crucible material, payload volume, payload surface area**
  - **Settering to reduce drag**
  - **Improved control of drying environment and process**
- **Design of Experiments**
  - **Forming**
  - **Sintering and crystallization cycle optimization**
- **Various alternative fabrication approaches under consideration as a parallel path for risk mitigation**

Forming DOE

Standard Conditions

- Vacuum Cast
- Cast at a 4 degree tilt
- Improved Tools (positioning accuracy and venting)

Assumptions

- DOE - Full Factorial with four factors = 16 different runs + 8 duplicates
- Mill yields 3 sub scale GNR
- DOE required 8 days of casting
  - resulted in 24 sub scale GNR available from the DOE
- Casting order defined to maximize learning (slip aging effect)

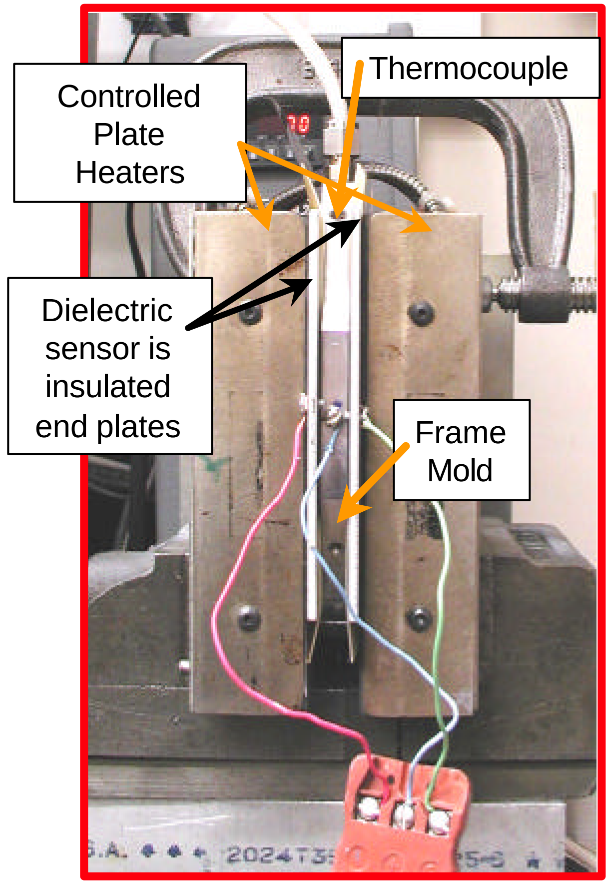
Forming DOE (cont.)

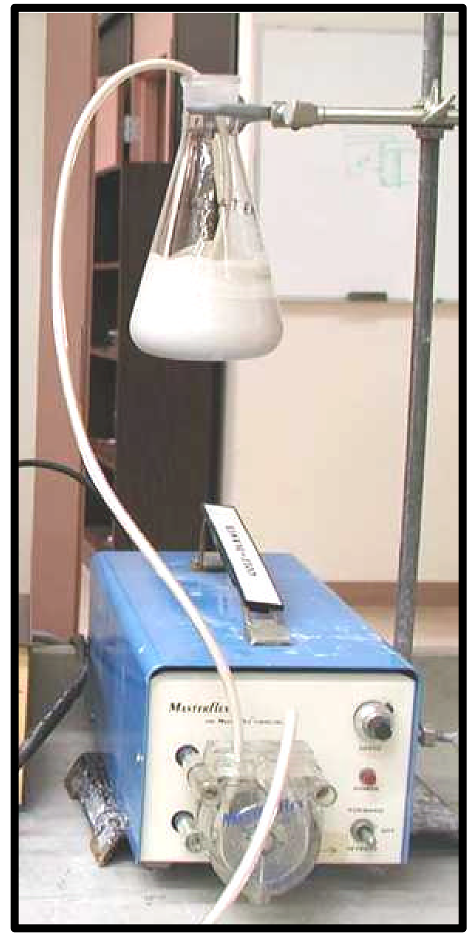
- Factors
  - Powder Lot (Viscosity, Surface Area)
  - Web Thickness (Flow Restrictions, Tooling Design)
  - Initiator Level (Gel Rigidity, Dimensional Control)
  - Pressure Differential (Flow Rate, Casting Defects)
- Response Variables
  - Shrinkage (i.e. scale factor)
  - Dimensions
  - Casting Defects
    - Number of pores/bubbles
    - Number of cracks
    - Location of defects
  - Slip Rheology (flow rate data a function of pressure)
  - Capture All “Green Sheet” Data

R&D Efforts to Gain Improved Fundamental Understanding of the Gelcasting Process

- **Simplified, instrumented mold has been designed and successfully operated**
- **Molding operations and monitoring has moved through several stages to the system currently in use**
  - **allows dielectric monitoring of gelation process**
- **Identification of an alternative compatible initiator has eliminated major source of variability**
  - **maintains slip stability and operates at lower concentration**
  - **minimizes gas evolution and reduces gelation temperature**
- **Production experience in Torrance with new initiator has been very favorable**
- **Further work with previous initiator has been discontinued**
- **Simulation of gelation process initiated**

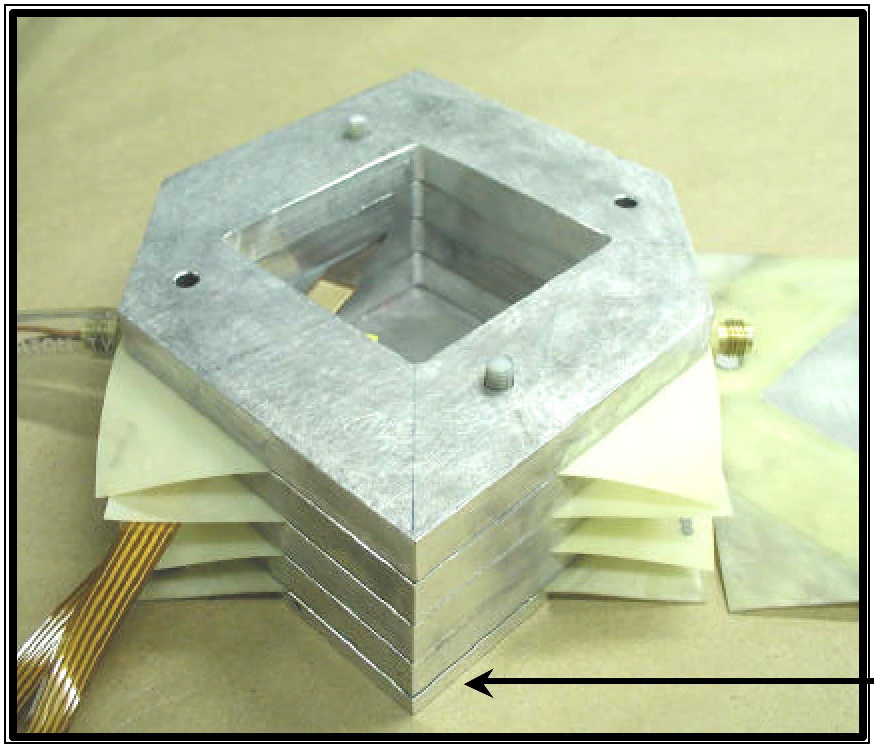
Simplified Instrumented Gelcasting Mold

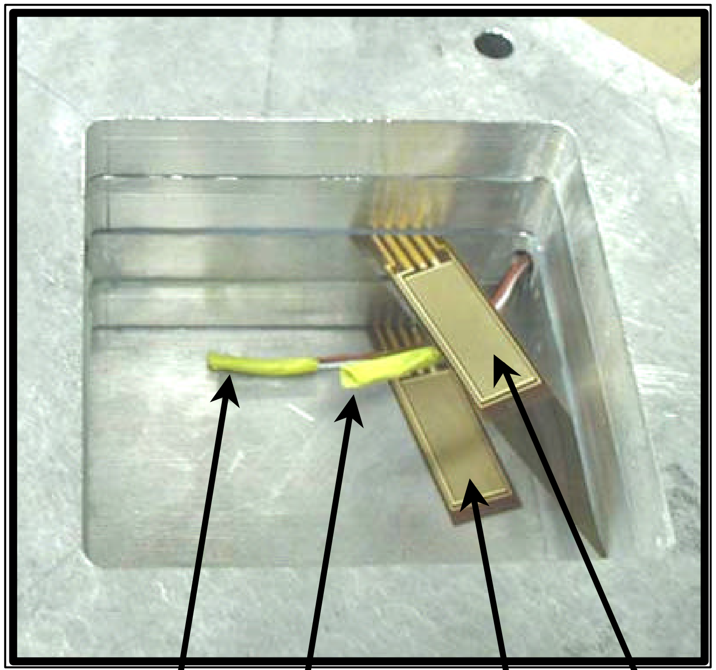




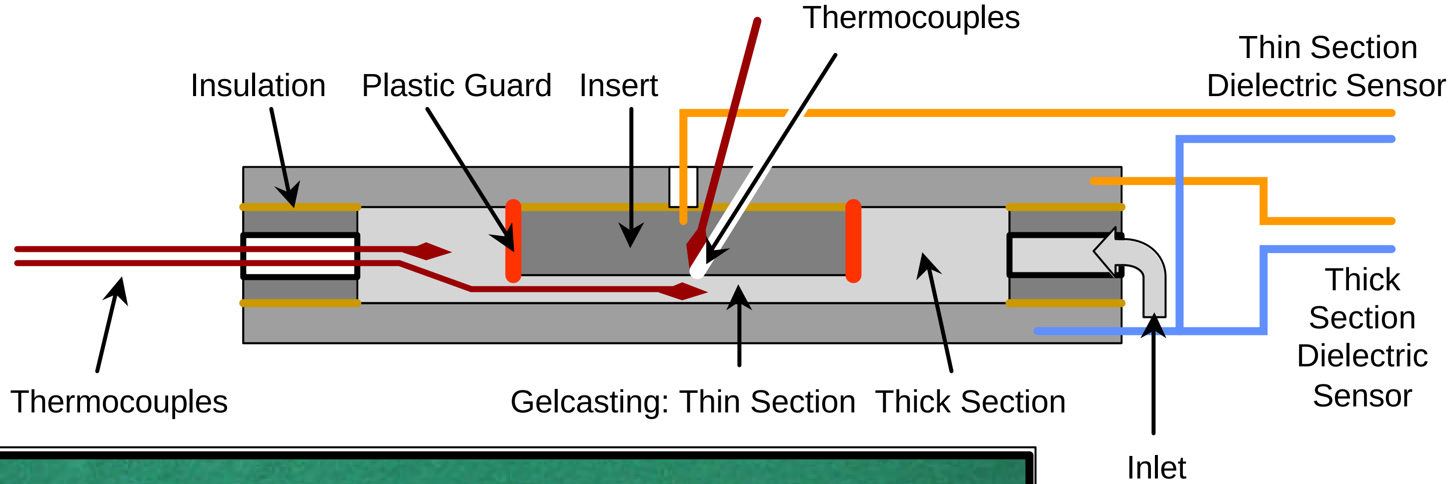
Thick Cross Section Gelcasting

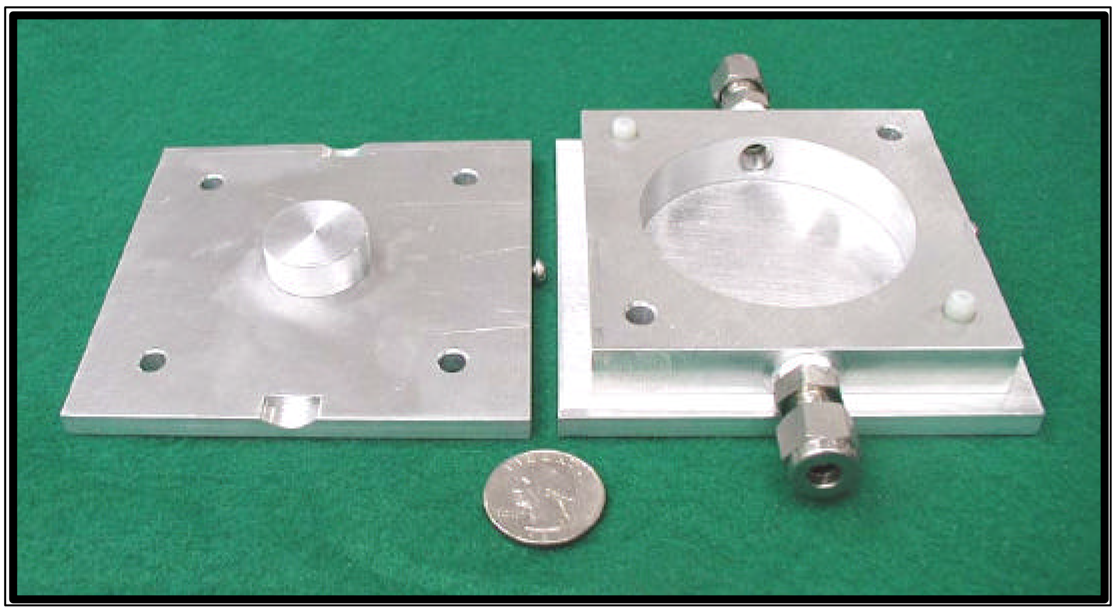
Thick Mold (57-mm)
with Gold plated IDEX Sensors
at Wall and Midplane

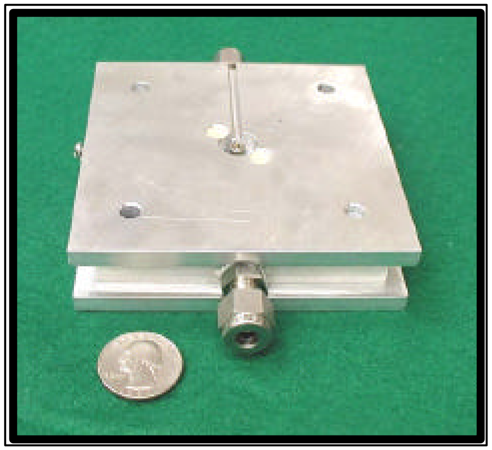




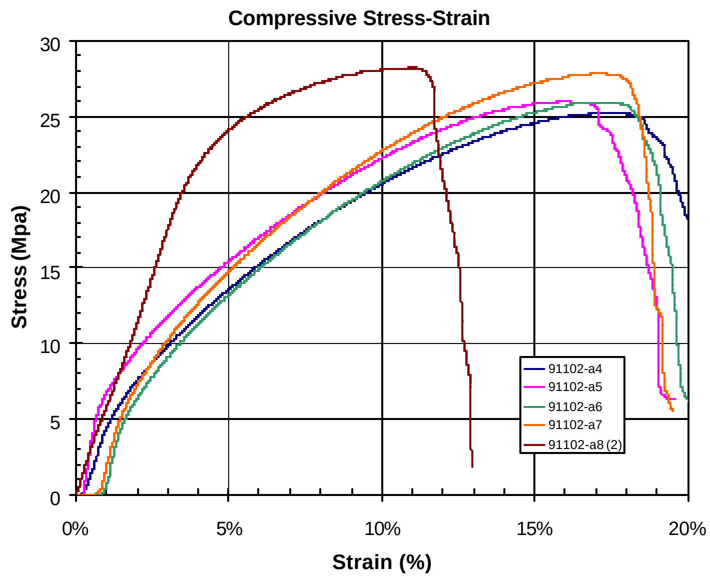
Combined Thin and Thick Cross Section Gelcasting

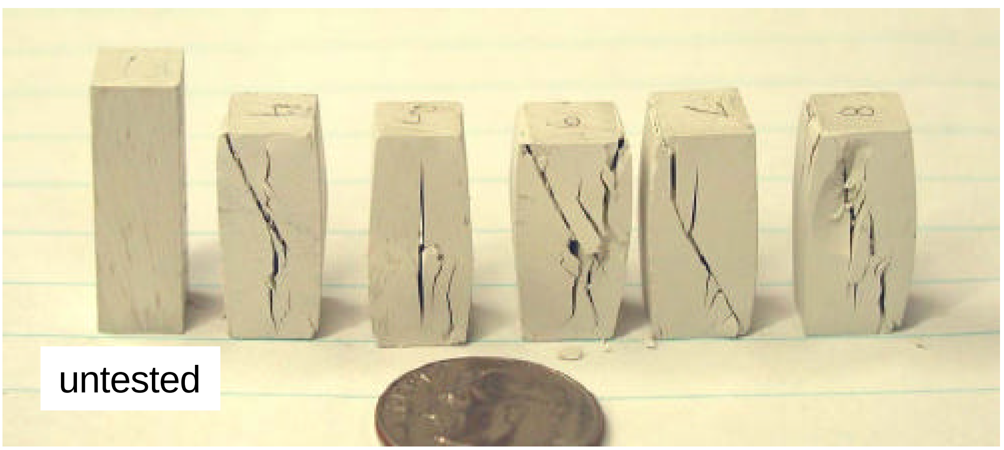


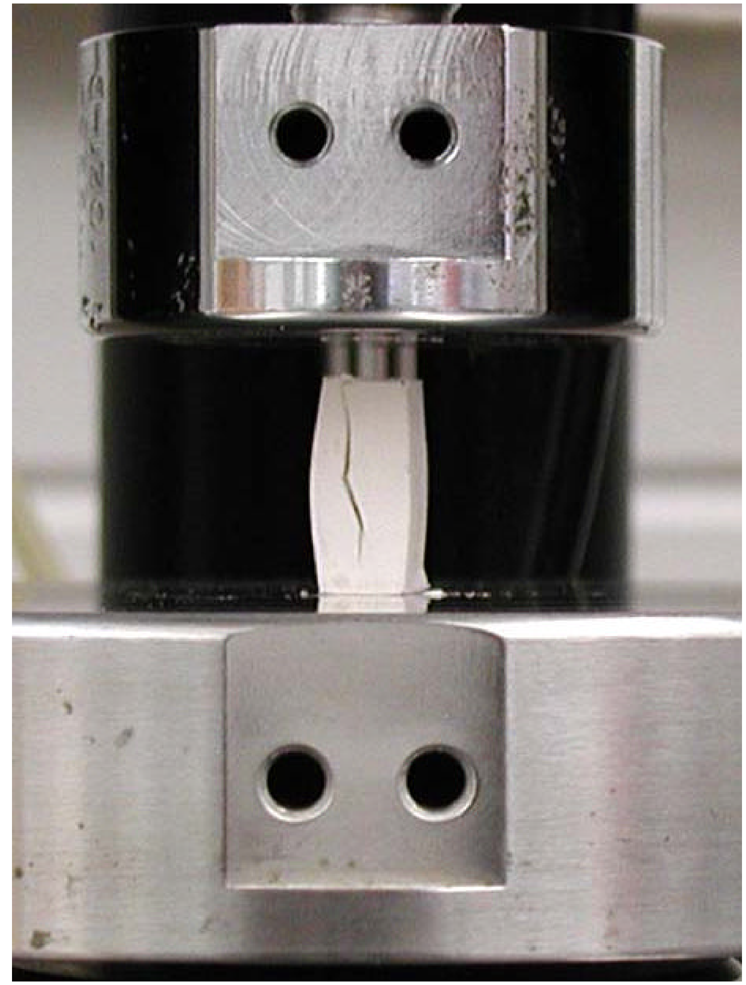




Compression Test of Gelled Samples







Tool Design Methodology Has Changed

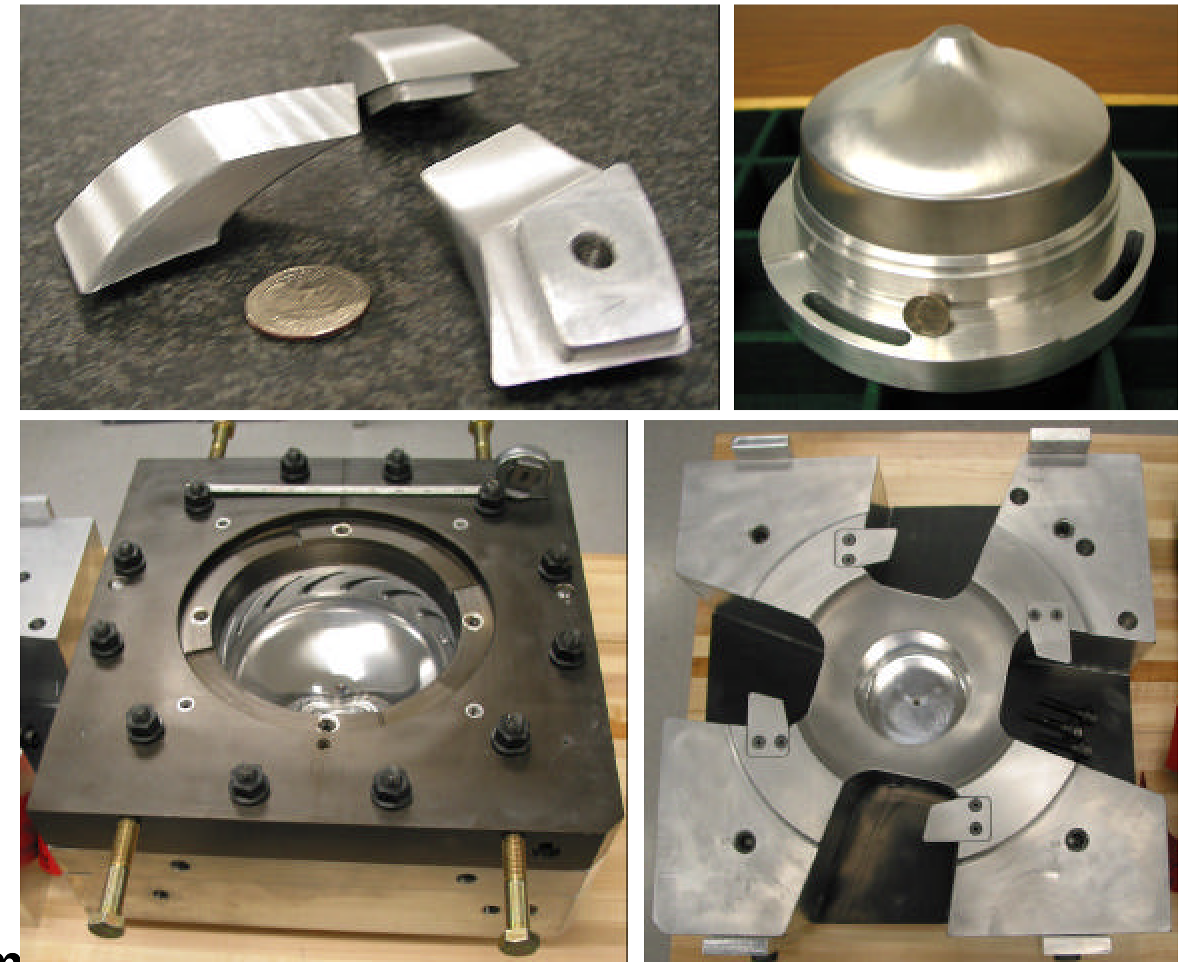
- Past practice
  - Tool design performed internally
  - Low cost tooling did not meet performance requirements
- New Practice
  - Specification of tooling performance requirements
  - Tool development performed by the tooling supplier
  - Payment milestones based on performance to established criteria
  - Final payment based on meeting performance specification

Tool Design and Integrity Are Critical for Tightly Toleranced Gelcast Components

Nozzle Ring Development Tool

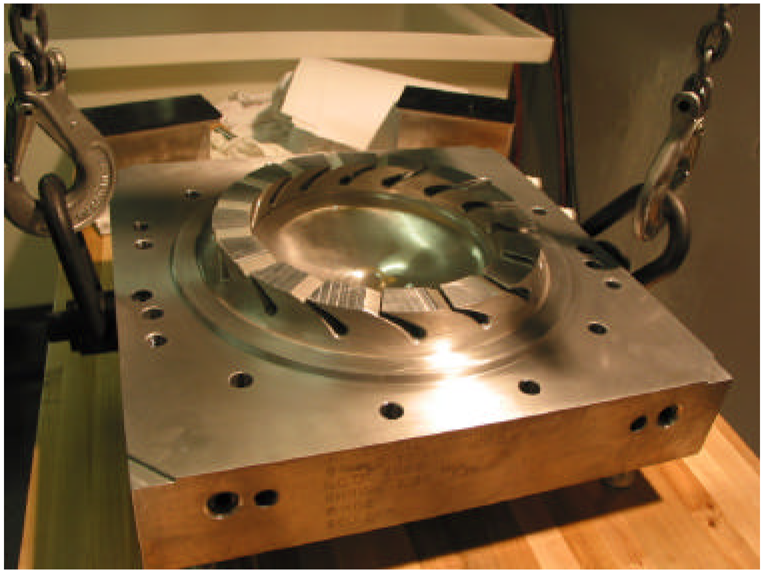
The new tool design process was used to design and fabricate new nozzle ring development tool

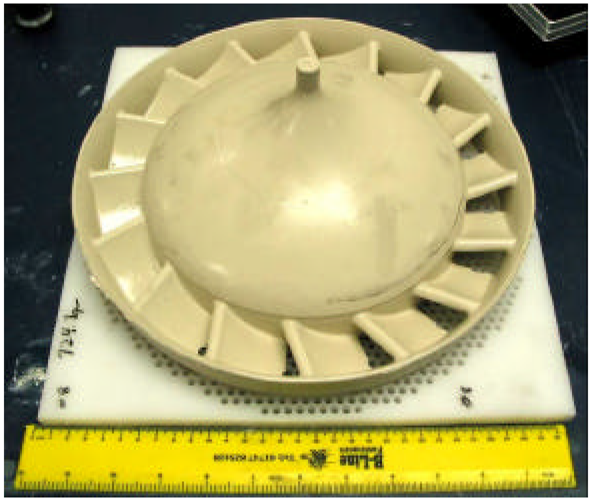
- Realistic nozzle geometry
- Design similar to production tool
  - fill
  - venting
  - heating and cooling
  - assembly and disassembly
- Compatible with vacuum casting process



Nozzle Ring Development Tool

Nozzle Ring Development Tool (cont.)





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AS950EXP Gelcast Process Refinement

- General Experimental Approach
  - Evaluate and optimize dispersant levels
  - Evaluate and optimize gelcast slurry characteristics
  - Demonstrate process by casting complex shape
  - Evaluate and optimize thermal processing
  - Prepare specimens for mechanical property verification
- Baseline process successfully gelcast complex shape
- Sintering cycle development needed to achieve full density
  - Gelcast AS950EXP material is < 90% dense when sintered in standard gelcast AS800 cycle.
    - Densification difficult due to low packing density (compared to slipcast), more refractory liquid phase, and high loading with SiC.
    - Sintering development activities at Honeywell Labs (Morristown, NJ), IKTS (Dresden, Germany), and Sandia National Labs (Albuquerque, NM) are being coordinated
  - Recent combination of burnout and sintering cycle modifications resulted in complete and robust densification of AS950EXP

Production Readiness Summary

- **Defect and Variation Reduction**
  - **Defects caused by fill method**
  - **Defects caused by tool design**
  - **Variability of scale factor**
    - **Within part**
    - **Part to part**
- **Distortion**
  - **Thermal environment**
  - **Drag / sintering fixturing**
  - **Wet part handling / fixturing**
- **Production processes**
  - **Process documentation**
  - **Process control**

Actions In Place To Address All Critical Issues