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Proceedings

CFCC Working Group Meeting

CONTINUOUS FIBER CERAMIC COMPOSITE MATERIALS

Harrah's Lake Tahoe
Lake Tahoe, Nevada

October 6-7, 1997

Office of Industrial Technologies
Energy Efficiency and Renewable Energy
U.S. Department of Energy

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CFCC WORKING GROUP MEETING

AGENDA

AGENDA

CFCC WORKING GROUP MEETING - DAY 1
OCTOBER 6, 1997

Sunday, October 5, 1997

6:00-8:00 PM	Registration	
6:00-9:00 PM	"ASME Sub Task Group on Ceramic Pressure Equipment" Informal Meeting for All Those Persons Who Have New Text or Revisions of Sections	T. V. Narayanan Co-Chairman

Monday, October 6, 1997

7:45-8:30	Registration	
8:30-9:00	CFCC Program Manager	
9:00-9:30	Applications for Advanced Ceramics in Industries of the Future, United States Advanced Ceramics Association Study Summary	Doug Freitag/ Dave Richerson
9:30-10:00	Standards, Design Codes and Data Bases: The CFCC Program and Its Participation in ASTM, ISO, ASME and Military Handbook 17 Activities	Michael Jenkins
10:00-10:30	Break	
	Overview of CFCC Contractor Projects	
10:30-11:00	Babcock & Wilcox	Rich Goettler
11:00-11:30	Textron	Bill Darden
11:30-1:00	Lunch	
1:00-1:30	DuPont Lanxide Composites - DMO	Phil Craig
1:30-2:00	Dow Corning	Andy Szweda
2:00-2:30	General Electric	Krishan Luthra
2:30-3:00	Break	
3:00-3:30	DuPont Lanxide Composites - CVI	Phil Craig
3:30-4:00	AlliedSignal/Caterpillar	Mike Haselkorn
4:00-4:15	ORNL - Supporting Technologies Task Summary	Rick Lowden
4:15	VPI - Life-Prediction Overview	Ken Reifsnider
4:30	Hands-On Life-Prediction Demos and Reception	Ken Reifsnider Edgar Lara-Curzio

CFCC WORKING GROUP MEETING
DAY 2 - OCTOBER 7, 1997

MORNING SESSION

8:30	Workshop A - Application Specific Testing	Phil Craig Krishan Luthra
8:30-9:00	Case Study - Solar Turbines Combustor	Solar Turbines
9:00-9:15	Discussion	
9:15-9:45	Case Study - Hot Gas Filter for Chemical Industry	Charles Hall Dow Corning
9:45-10:00	Discussion	
10:00-10:30	Break	
10:30-10:45	Case Study - HSCT CMC Combuster	Dave Utah General Electric
10:45-11:00	Case Study - CFCC Shroud for Industrial Gas Turbines	Paul DiMascio General Electric
11:00-11:30	Discussion	
11:30-1:00	Lunch	

AFTERNOON SESSION

1:00-4:00	(See Following Page for Details on Afternoon Workshops)
5:00-9:00	"ASME Sub Task Group on Ceramic Pressure Equipment" Formal Meeting of the Sub Task Group Will Include Some Type of an Informal Dinner. For More Information, Contact T. V. Narayanan (201-535-2224)

Tuesday Afternoon Workshops

Two concurrent workshops, B: Materials Characterization and C: Life-Limiting Phenomena, will be held Tuesday afternoon, October 7, 1997. These are designed to be informal and discussion-oriented with extensive audience interaction. The workshops are intended to address critical issues within the CFCC community. They have been organized as a means of relating the work currently being conducted under Task 2 to relevant industrial interests and priorities.

Attendees should come prepared to discuss topics of interest and to participate in discussions. To facilitate the process, brief (5-15 min) and informal contributions are encouraged.

Workshop B: Materials Characterization

Facilitators: Karren More, Bill Ellingson, Andy Szweda

- Highlight several industry-related problems that have benefited from characterization effort
- Review characterization techniques available to industrial manufacturers and end-users and evaluate the benefits of doing both macro- and microstructural analysis of CFCCs
- Compile information that industrial R&D personnel are interested in acquiring using the various characterization methods; explore opportunities and mechanisms for doing characterization work

Schedule:

1:00	Review/highlight Task 2 efforts in support of materials and interface characterization
1:30	Participant input regarding issues of greatest importance to the materials and interface characterization effort - manufacturer/end-user perspectives
2:30	Break
3:00	General discussions on how to best address important characterization issues; prioritize industrial needs for characterization; how can we characterize CFCCs and elucidate life-limiting mechanisms

Workshop C: Life-Limiting Phenomena

Facilitators: Rick Lowden, Rich Goettler, Peter Tortorelli

- Examine different perspectives on life-limiting phenomena for CFCCs as viewed by manufacturers, end users, and R&D personnel
- Evaluate data and testing needs in support of key life-limiting processes and associated modeling

Schedule:

1:00	Task 2 efforts in support of life-limiting phenomena - approaches and issues
1:20	Participant input regarding particular perspectives on life-limiting phenomena for CFCCs: individual presentations and associated discussions
2:30	Break
3:00	General discussions on how to best address identified life-limiting phenomena in terms of • lab work • testing • modeling • database needs

CFCC WORKING GROUP MEETING

ATTENDEES' LIST

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CFCC WORKING GROUP MEETING

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LAKE TAHOE, NEVADA
OCTOBER 6-7, 1997

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CFCC WORKING GROUP MEETING

PRESENTATIONS

CFCC WORKING GROUP MEETING

OCTOBER 6-7, 1997
LAKE TAHOE, NEVADA

MERRILL SMITH
AND
DEBBIE HAUGHT

CFCC

Continuous Fiber Ceramic Composites

Developing processing methods for and fabrication of reliable, cost effective CFCCs for energy intensive industries

Department of Energy

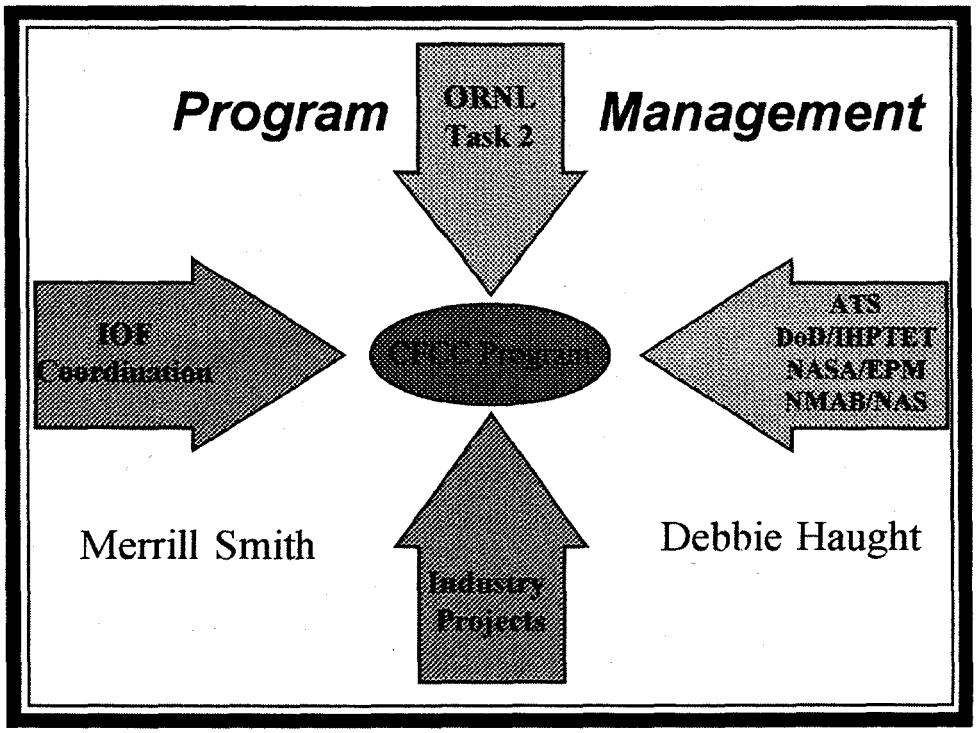
OIT

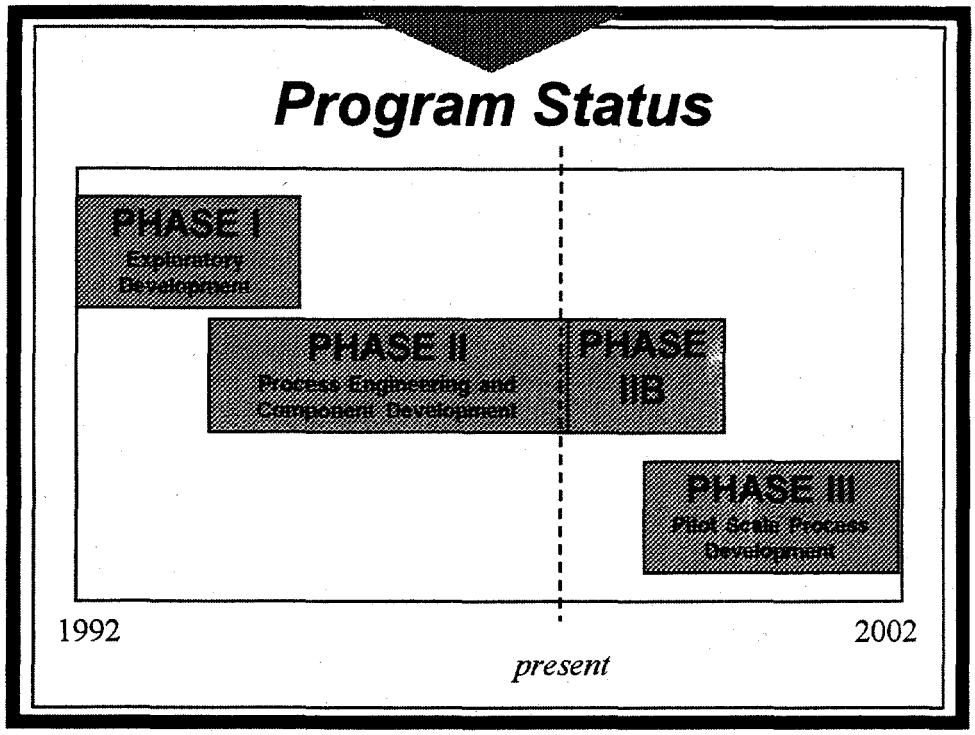
FY 1997 was a busy year

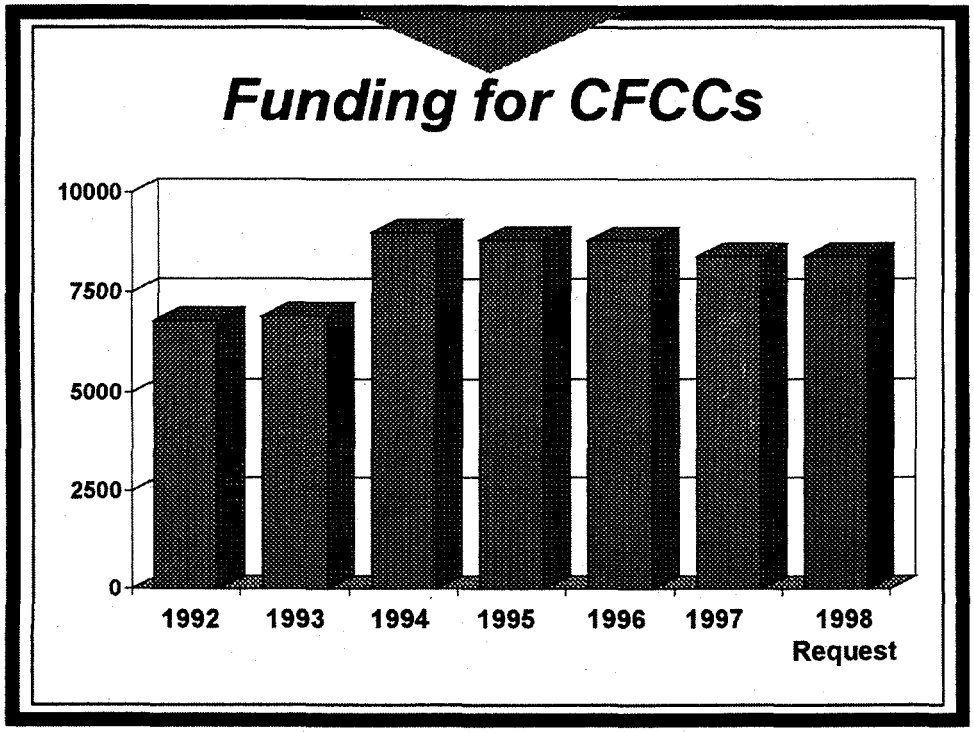
- ORNL Task 2 Review and Report
- Cocoa Beach Meeting (1/97)
  - CFCC Test Methods (open session)
  - Four CFCC Sessions (closed session)
- DOE 2nd Industrial Energy Efficiency Symposium & Exposition (2/97)
  - Awards for outstanding contributions to John Oleson (Dow Corning), Bill Long (ret. B&W) and TN Congressman Zack
- Industry Task 3 Project Review (7/97)

Program Changes

- Phase II Extension
- Additional HQ Program Management
  - Merrill Smith
  - Debbie Haught
- Amercom Cooperative Agreement ended (Amercom bought by Synterials)




Program Status
PHASE I
Exploratory Development
PHASE II
Process Engineering and Component Development
PHASE IIB
PHASE III
Pilot Scale Process Development
1992
2002
present


Funding for CFCCs
10000
7500
5000
2500
0
1992
1993
1994
1995
1996
1997
1998 Request

CFCC Participants

AlliedSignal
Dow Corning
Du Pont Lanxide
General Electric CRD
McDermott Technology
Textron Systems Div.
Oak Ridge National Lab
Advanced Ceramics Corp.
Ames Laboratory
Alzeta
Argonne National Lab
Caterpillar
Chevron
Cleveland State
Composite Optics
Fiber Materials Inc.
F.W. Schaefer
Foster Wheeler
GE Power Generation
Hauck Manufacturing
HyperTherm
IBIS Associates, Inc.
INEEL
Lanxide Corp.
LBL
Material Characterization Laboratory
Materials Research & Design
Materials Sciences Corp.
Northwestern Univ.
Ohio State U.
PNNL
Penn State U.
RPI
Solar Turbines
SNL
Stone & Webster
Sundstrand Fluid Handling
Synterials
Thermo Trex
TVA - Gallatin Station
U. of CA- Berkley
U. of Cincinnati
U. of Dayton Research
U. Hawaii
U. of Michigan
U. of Southern CA
U. of Washington
Virginia Tech
Williams International

CFCC Applications

Chemical Process Equip.
Combustor Liners
Containment Shell
Gas Turbine Shroud
Heat Exchanger Component
Hot Gas Filters
Interstage Seal Rings
Molten Metal Immersion Tube
Pipe Hangers
Radiant Porous Burners
Radiant Burner Tubes
Steam Reformer Components
Tube Shields
Turbine Tipshoe
Valve Guides
Vortex Finder

OIT / USACA Study

- Conduct a comprehensive study of how advanced ceramics can be targeted in the seven Industries of the Future (IOF) to improve energy efficiency and solve materials related problems
- On behalf of USACA, Doug Freitag of Bayside Materials Technology and Dave Richerson of Richerson & Associates are carrying out the study

USACA Study Overview

- Focus on advanced ceramics (monoliths, ceramic composites, coatings)
- Assess advanced ceramic materials technology base developed under government funding as it relates to IOF
- Survey IOF to identify advanced ceramics applications, opportunities, and barriers
- Share results of the survey with industry and solicit feedback
- Analyze data and provide recommendations
- Issue a final report

Advanced Ceramics have applications in IOF

	Glass	Refinery	Steel	Aluminum	Chemical	Forest Products	Metal Casting
Burners	X	X	X	X	X	X	X
High Temp. Insulation	X	X	X	X	X	X	X
Heat Recovery	X	X	X	X	X	X	X
Sensor Shields	X	X	X	X	X	X	X
Separation	X	X	X	X	X	X	X
Handling/Transport	X	X	X	X	X	X	X
Rollers	X		X	X	X	X	
Gas Injection	X		X	X			X
Gates/Valves	X	X	X	X	X	X	X
Fans/Stirrers	X	X	X	X	X	X	X
Molds/Tooling	X						X
Supports	X	X	X	X	X	X	X

CFCC

Accomplishments and Current Highlights

Materials & Process Improvements are Continuing

- Demonstrated 90% improvement in micro crack yield point at RT of Sylramic S200 with use of Sylramic SiC fiber (Dow Corning)
- Improved near net shape fabrication processes for valve guide blanks and reduced ID/OD machining stock by >20 % (AlliedSignal)
- Developed resin-derived interface C coating, with cost 1/10 of CVI (DuPont Lanxide)

Materials & Process Improvements are Continuing

- Completed 6600 hr stress rupture test which demonstrated reproducible low cycle fatigue behavior of DIMOX material (Du Pont Lanxide)
- Developed CFCC cost model for polymer impregnation and pyrolysis (PIP) process which suggests 25% reduction in manufacturing costs are possible (Dow Corning)

Materials & Process Improvements are Continuing

- Developed promising scheelite ($CaWO_4$) fiber coating for oxide-oxide composites (McDermott)
- Optimized fiber architecture for filament wound CFCC components. Ordered new 5-axis filament winder (Textron)
- Initiated efforts in low cost fiber reinforced thin sheet to produce CFCC's at $100's/lb (Du Pont Lanxide)

Task 2 - Understanding Key Material Factors that Limit Use of CFCCs

- Used electron microscopy along with mechanical property characterization to characterize effect of oxygen in BN interfaces for SiC/SiC composite systems.
  - Demonstrated O-contamination in BN degrades the mechanical reliability of these composites.

Task 2 - Understanding Key Material Factors that Limit Use of CFCCs

- A micromechanical model was developed to predict the oxidation-induced stress-rupture behavior of CFCCs.
- CFCC environmental effects test facilities at ORNL were established and initial experiments in inert gases, air, and air + water vapor have been conducted at elevated temperatures.

Task 2 - Developing Standard CFCC Test Methods

- Instrumental in formalizing 7 full-consensus standard test methods that have been adopted by in ASTM subcommittee C28.07 on Ceramic Matrix Composites
- Developed test to characterize the hoop strength of CFCC tubular components.

Environmental Durability of CFCCs Demonstrated

- 4000 hr exposure testing in direct-coal burning boiler at TVA Gallatin Station was completed for Nicalon/E-SiC tubes. The samples showed no evidence of corrosion or strength loss after exposure (1628 F avg) (DuPont Lanxide)
- Sylramic S200 coupons exposed in crude unit furnace for 8,424 hours still retained greater than 60% of original strength (Dow Corning)

Hot Gas Filters
McDermott

- Testing at Foster Wheeler 10 MW PFBC pilot scale facility in Karhula Finland has begun (67 hr testing to date)
- Proof of concept for CFCC IR radiant burner (based on TPV emitter technology) was demonstrated

Hot Gas Filters
ORNL / Dow Corning, Inc. CRADA

- Goal: Assess the use of high temperature filtration materials for use in the chemical industry's process streams
- Materials from 3M, Amercom, DuPont Lanxide, McDermott, Smart Ceramics, Techniweave, Pall Corporation, and Dow Corning have been tested for 24 hours in a series of fluidized bed reactor simulators in the furnace
- Rupture tests of ring samples is underway--as received (for baseline) and samples exposed in furnace

Diesel Valve Guides
(AlliedSignal/Caterpillar)

- Conducted simulated engine testing which demonstrated that CFCC valve guides caused less wear that both silicon nitride and cast iron guides
- Tested CFCC valve guides in Caterpillar gas-fired, diesel cycle engine for 224 hours

Radiant Burners

DuPont Lanxide/Alzeta

- Conducted 32,00 thermal cycle tests (2100F face temperature) on SiC/SiC radiant burner screens with no failure

Industrial Gas Turbine Components

- CFCC materials will allow turbines to operate at higher inlet temperatures and increase the power output by 2%. Applications include combustor liners, shrouds, rim seals, and tip shoes.

Participants:

- Dow Corning
- Du Pont Lanxide
- General Electric
- McDermott
- Textron
- Solar Turbines

- Solar turbines field test (1850 F TRIT) completed 1,000 hr on DuPont Lanxide CVI SiC liners

Industrial Gas Turbine Components

- DuPont Lanxide (CVI) combustor liner survived 100 hr test at Solar Turbines despite fabrication damage (delamination)
- GE developed new shroud design with reduced stress and better manufacturability
- Initiated coordinated effort between Virginia Tech, Solar Turbines and ORNL to predict the service life of combustor liners for gas turbine engines based on MRLife code.

Upcoming Gas Turbine Test Opportunities (Solar Turbines)

- 2000 hr field test (1850 F TRIT) - 11/97
- 4000 hr field test (2050 F TRIT) - 3Q '98
- Commercial Application at Malden Mills - 1999

Upcoming Meetings

- Advanced Turbine Systems Program Review
  - October 28 - 29, 1997 Morgantown, WV
- 22nd Annual Conference on Composites, Materials, & Structures (restricted sessions)
  - January 26-30, 1998 Cocoa Beach, FL
- CFCC Task 2 Review
  - March 1998, Oak Ridge, TN
- 3rd Industrial Energy Efficiency Symposium & Expo
  - February 1999, Washington, DC

Overview of Study on Applications for Advanced Ceramics in the Industries of the Future (IOF)

Study Sponsored by

Department of Energy
Office of Industrial Technologies

Study Conducted By

Doug Freitag, Bayside Materials Technology

And

Dave Richerson, Richerson and Associates

On Behalf Of The

United States Advanced Ceramics Association

USACA

Study Overview

- Focus on advanced ceramics (monoliths, ceramic composites, coatings)
- Assess advanced ceramic materials technology base developed under government funding as it relates to IOF
- Survey IOF to identify advanced ceramics applications, opportunities, and barriers
- Share results of the survey with industry and solicit feedback
- Analyze data and provide recommendations
- Issue a final report

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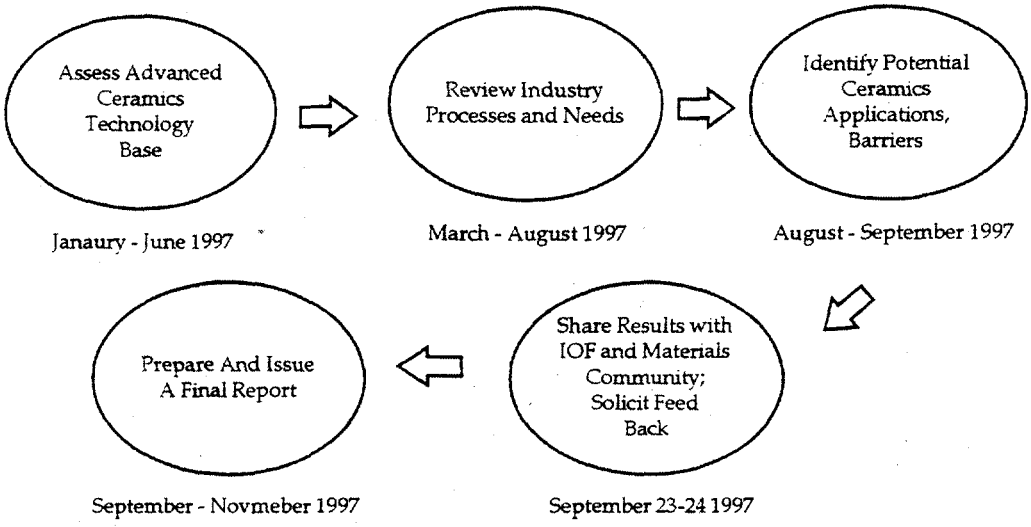
Study Timetable



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Key Drivers for the Substitution of Alternate Materials

- Energy Efficiency
- Environmental
- Life Cycle Cost
- Safety
- Productivity
- Maintenance
- Quality
- Dimensional Control
- New Products
- Recycling
- Product Development Cycle

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Barriers to Advanced Ceramic Materials Selection

- Metals Mentality
- Awareness
- Acquisition/Prototyping Cost
- Size Capability
- Repair
- Joining
- Codes
- NDE
- Database/Handbooks
- Design tools
- Lack of field data

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Applications within IOF where Advanced Ceramics Provide the Greatest Return on Investment

	Glass	Refinery	Steel	Aluminum	Chemical	Forest Products	Metal Casting
Burners	X	X	X	X	X	X	X
High Temp. Insulation	X	X	X	X	X	X	X
Heat Recovery	X	X	X	X	X	X	X
Sensor Shields	X	X	X	X	X	X	X
Separation	X	X	X	X	X	X	X
Handling/Transport	X	X	X	X	X	X	X
Rollers	X		X	X	X	X	
Gas Injection	X		X	X			X
Gates/Valves	X	X	X	X	X	X	X
Fans/Stirrers	X	X	X	X	X	X	X
Molds/Tooling	X						X
Supports	X	X	X	X	X	X	X

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Ceramic Materials Can Meet Cross-Cutting Needs of Industries of the Future

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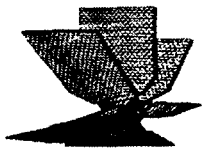
Monolithic Ceramics Are Working In Challenging Applications

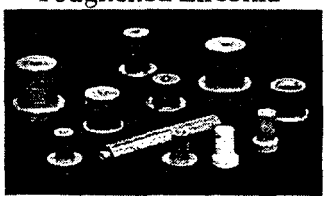
Silicon Nitride

Silicon Carbide

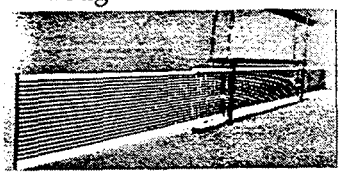
Aluminum Oxide



Toughened Zirconia



Toughened Alumina



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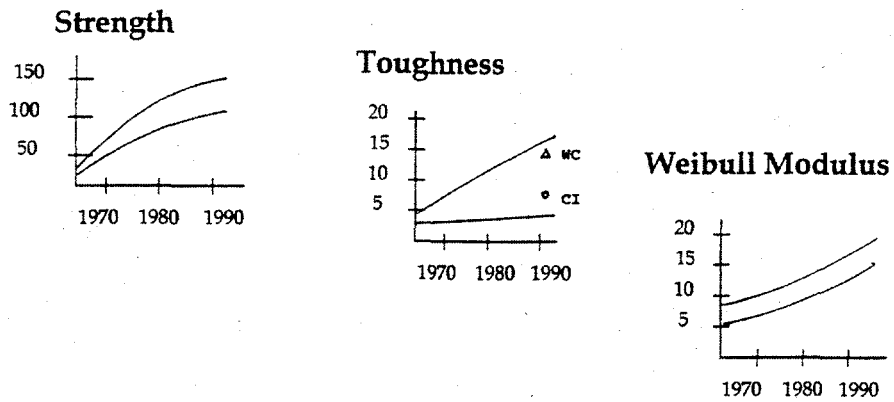
What is New & Different About 1997 Monolithic Ceramics



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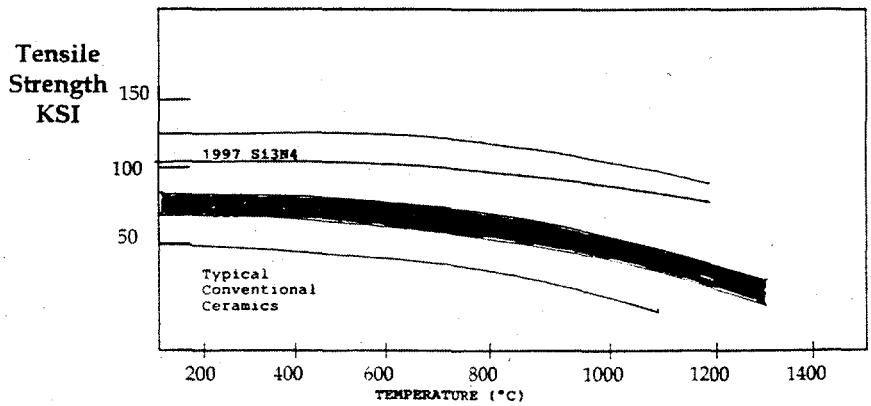
Silicon Nitride An Example of a New Generation of Ceramic Materials



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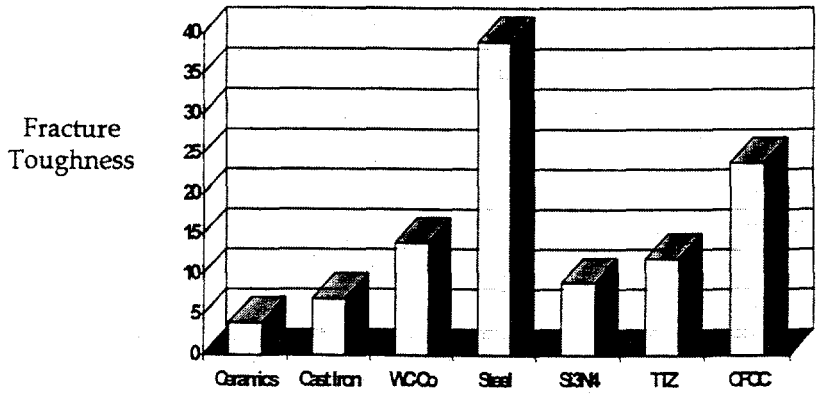
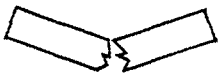
New Ceramics And Ceramic Matrix Composites Exhibit Increased Toughness



In Spite Of Improvements, Potential Users Still Cautious

Brittle Fracture



Careful Designs Required

NO Sharp Edge

YES Rounded Corners

Limited Database, Codes, and Standards

	A	B
Allowable Stress	?	?
Fatigue Limit	?	?
Stability at Temp.	?	?

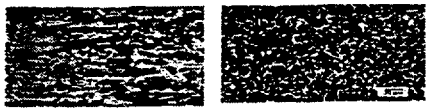
Price Premium

$

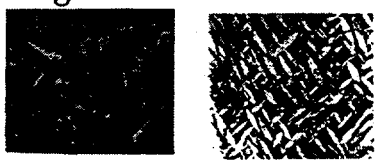
We Can Often Address These Concerns Through Engineered Materials

Continuous Fiber Ceramic Composites (CFCC)

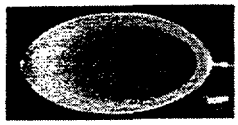
Ceramic-Reinforced Metals



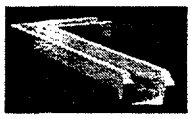
Engineered Ceramic Microstructures

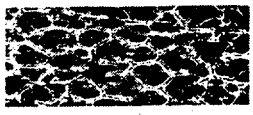


Ceramic-Coated Metals



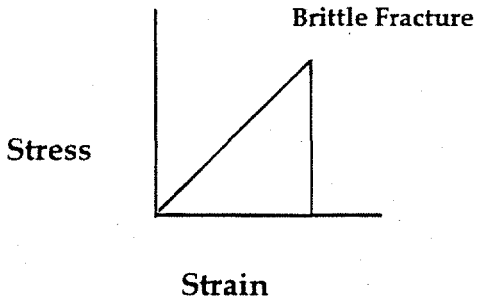
Advanced Refactories



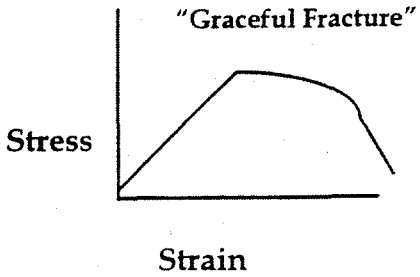


CFCC's Avoid Brittle Fracture

Monolithic Ceramics



Continuous Fiber Ceramic Composites



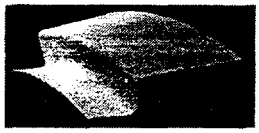
A Variety Of CFCC Materials Under Development

Chemical Vapor Infiltration

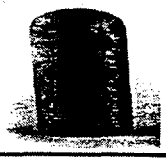
Melt Infiltration

Nitride-Bonded Silicon Carbide

SOL Infiltration



Directed Metal Oxidation



Pre-Ceramic Polymer Infiltration

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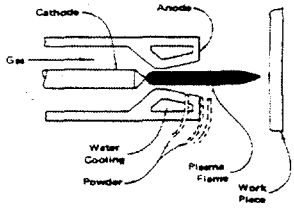
Coatings Can Combine Ceramics Benefits With Metals Structural Reliability

- Plasma Spray
- Physical Vapor Deposition
- Chemical Vapor Deposition
- Vapor Deposition Diamond
- Slurry or Sol Deposition



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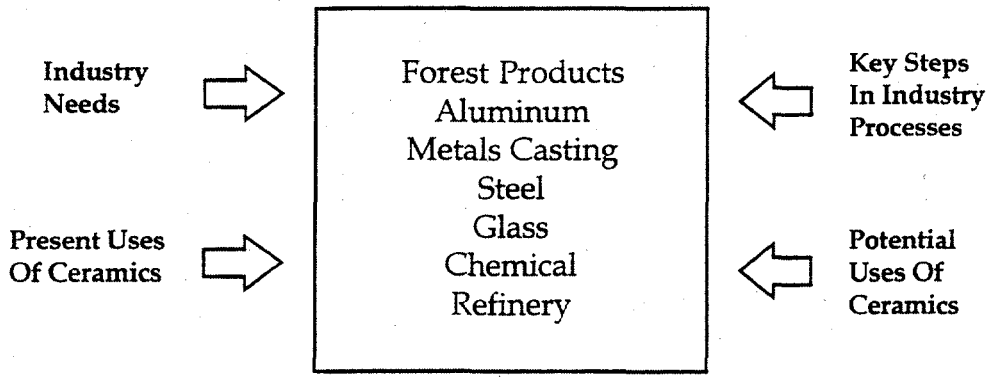
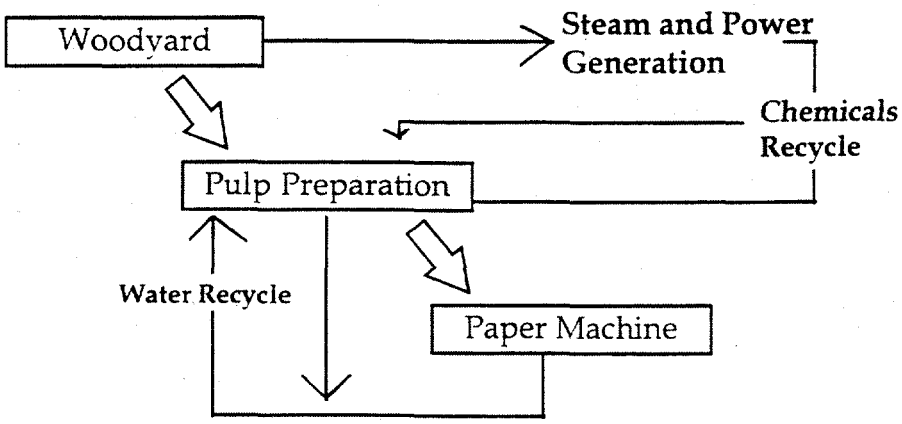
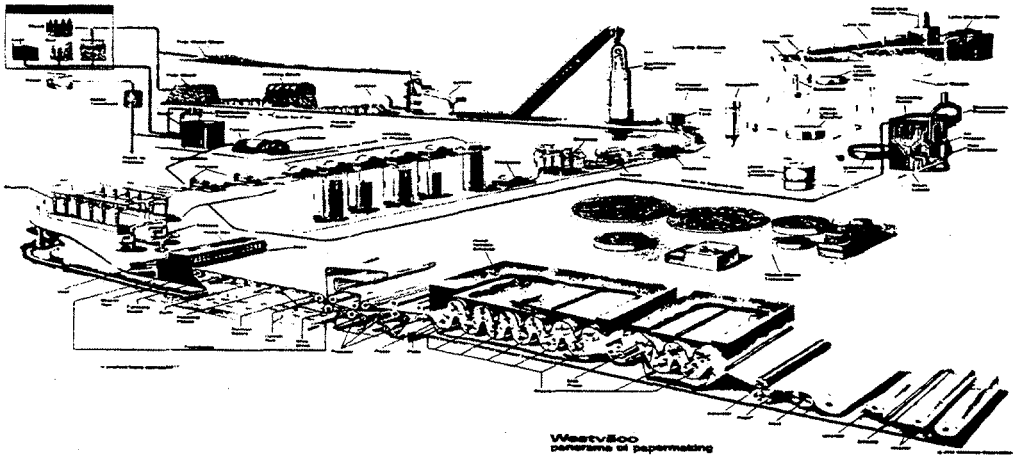
The USACA Study Shows That Ceramics Are Already Used and Can Provide Further Benefits To IOF



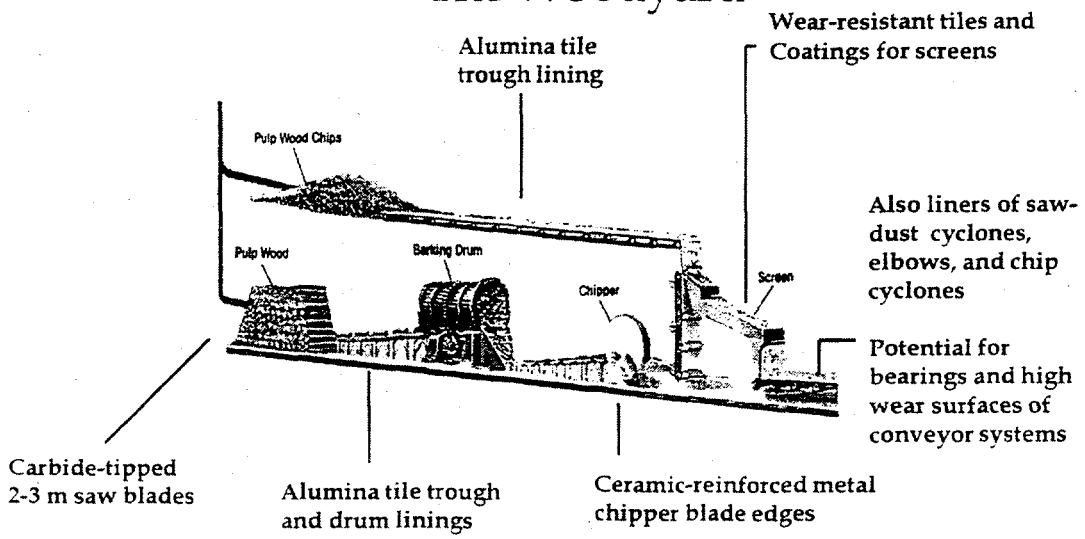
Important Segment Of Forest Products Is Papermaking



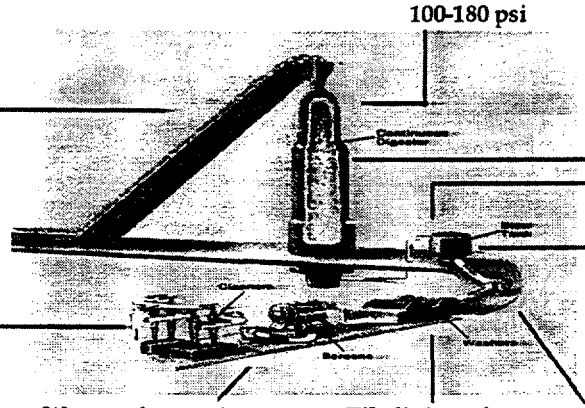
Each Process Stage Has Multiple Steps, Many of Which Utilize Or Could Utilize Ceramics



Current And Potential Uses of Ceramics In The Woodyard



Current And Potential Uses Of Ceramics In Pulp Digestion



20-80 m high
100-180 psi

Brick lining of sulfite mill digester

Troughs, conveyors

Also pump liners and seals

Lining of blow line

Barrels, shafts and cones of hydrocyclone cleaners

Blow tank target plate

Wear and corrosion resistant coatings

Tile lining of washer vats

Linings on surfaces of pipes

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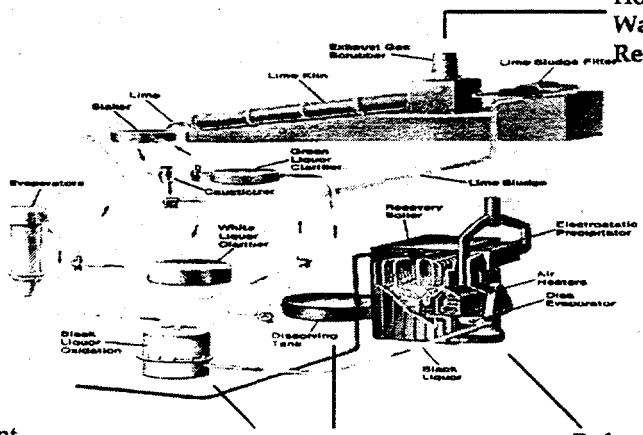
Current And Potential Uses Of Ceramics In Chemicals Recycling



Hot gas cleanup
Waste heat
Recovery

Also pump wear plates and seal faces

Also acid brick lined trench sewers

Also porous membranes for waste-water treatment

Tank linings

Refractories in recovery boiler

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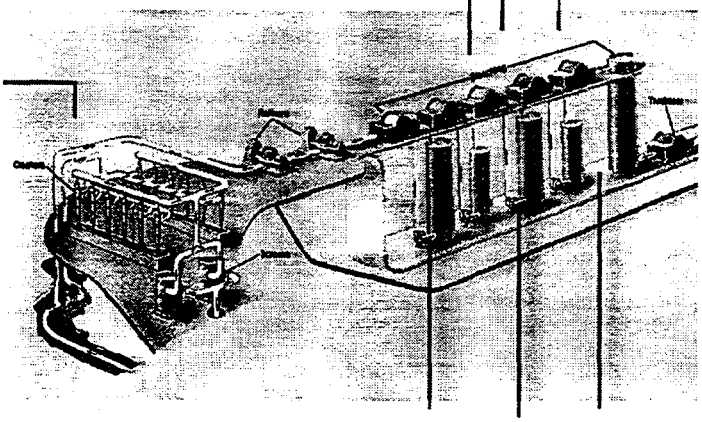
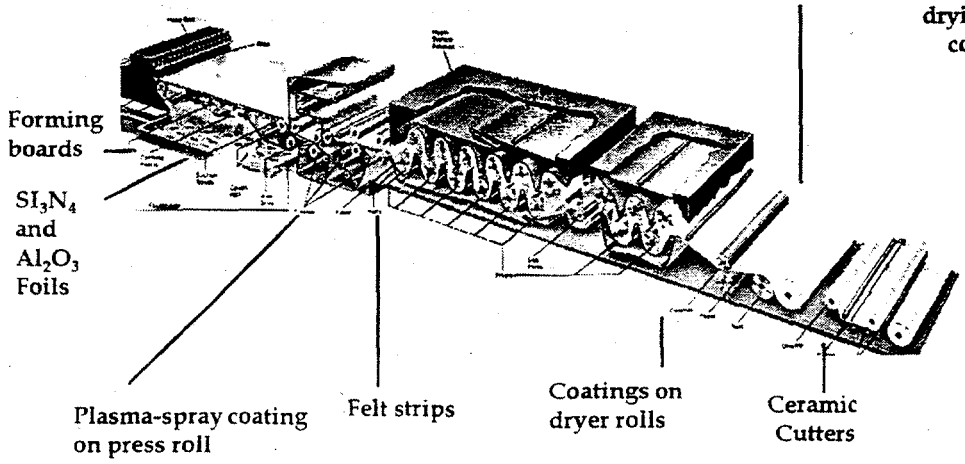
Current And Potential Uses Of Ceramics In Bleaching



Current And Potential Uses Of Ceramics In The Papermaking Machine



Aluminum Industry Involves Several Stages

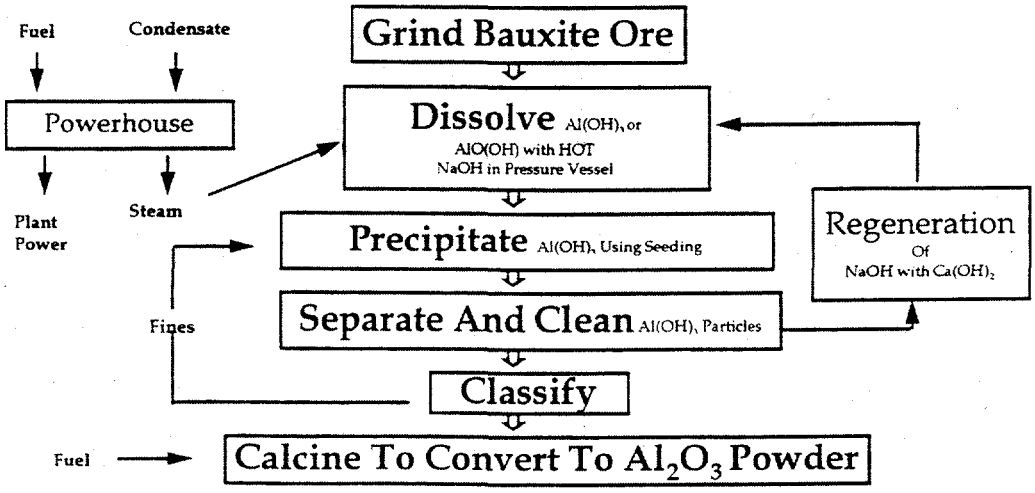
Extraction of Al_2O_3 from Bauxite Ore

Electrolytic Smelting of Aluminum Metal

Post-Smelting Processes

USACA

Aluminum Oxide Powder Is Extracted From Bauxite By The Bayer Process (Patented 1888)



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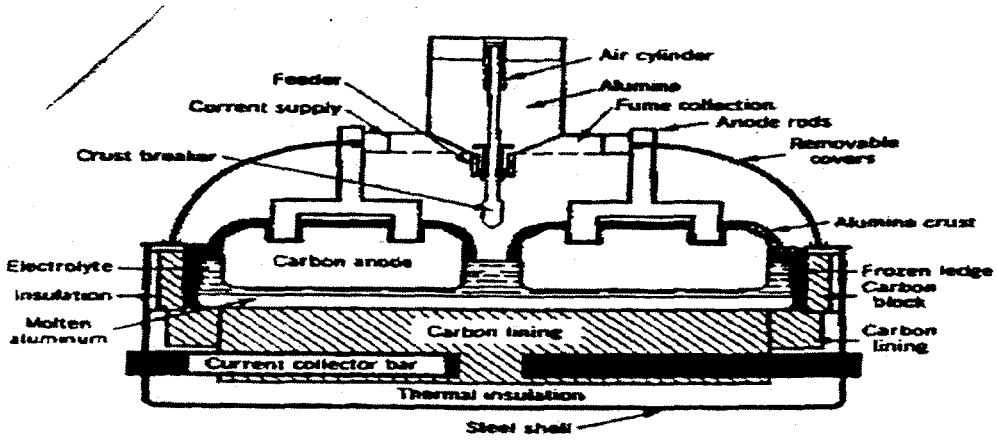
Smelting Is Energy Intensive

Electrical
- 6.419 kW.h/kg of Al
- 42.6% Efficiency

Thermal
- 970 degrees C
- 8.12 kW.h/kg of Al heat dissipation

Scale of Process
- Cell Size 9-16 m x 3-4 m x 1-1.3 m
- 250 - 1000 Cells/Plant
- ~ 130 Cells in series at 100-300 kA

Aluminum Metal Obtained By Electrolysis Of Alumina By The Hall-Héroult Process (1886)



Aluminum Requires Post-Smelting Processes

Siphoning From Hall-Héroult Cell
(99.6-99.9 % Pure Al)

↓

Refinement

↓

Alloying

↓

Degassing

↓

Filtration

↓

Casting Into Ingots,
Sheet or Extrusion Stock

USACA

Ceramic Materials Have Potential To Benefit Aluminum Industry

NEED/OPPORTUNITY	BENEFIT
Wetable, Inert Cathode	Save > 10% Electrical Energy
Inert, Non-Carbon Anode	Eliminate CO_2 Emissions, Decrease Energy
Improved Refractories	Longer Life, Lower Maintenance
Improved Castables	Easier Installation
In-Line Sensors	Better Process Control, Efficiency
Miscellaneous Liners	Reduced Wear, Corrosion; Thermal Control
Material Resistant to Molten Cryolite	Allow Hot Wall, Increase Efficiency

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Ceramics Can Enable Broadbased Benefits To Metals Casting

Increased Efficiency

Longer Life Between Maintenance Or Replacement

Pollution Reduction

Superior Product

USACA

Increased Efficiency

- Higher Temperature
- Alternate Processes
- Heat Retention and Recovery
- Increased Yield
- Decreased Machining Stock

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Longer Life Between Maintainence and Replacement

- Increased Corrosion Resistance
- Superior Wear Resistance
- Improved Thermal Shock, Thermal Cycle Resistance

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Pollution Reduction

- Decreased Fuel Use
- NOx Reduction
- Catalytic Processes
- Reduced Solid Waste

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Superior Products

- Decreased Inclusions
- Better Control of Chemistry
- Improved Dimensional Control

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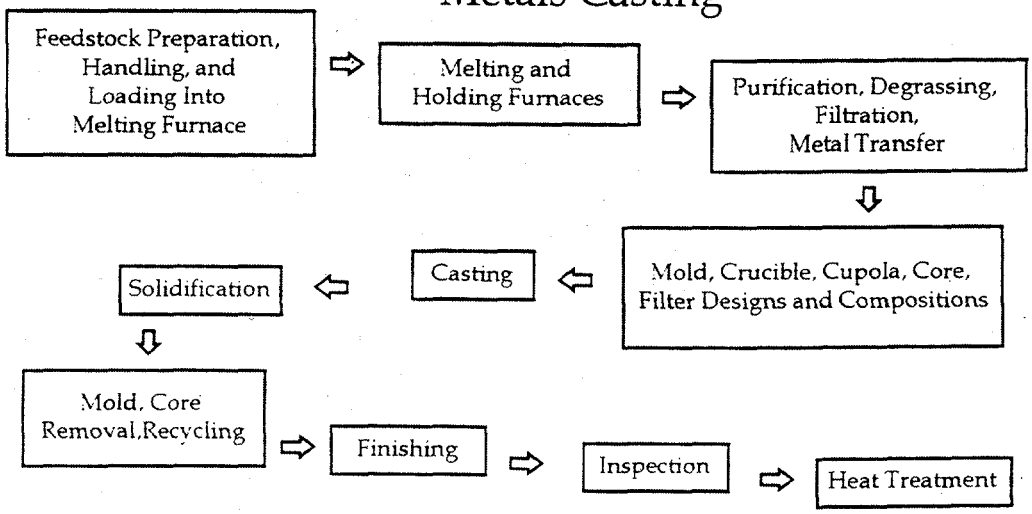
Ceramics Potentially Important In Each Step Of Metals Casting



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Ceramics For Metal Handling Prior To Casting

Feedstock Preparation, Handling, and Loading Into Melting Furnace ⇨ **Melting and Holding Furnaces** ⇨ **Purification, Degrassing, Filtration, Metal Transfer**

Tooling for scrap segregation

Rapid cutting of scrap by laser or waterjet

Wear resistant lining for chutes, conveyors and entrance to furnace

Burners

Furnace lining

Composition sensors (such as oxygen)

Trough

Heat exchanger

Above-metal circulation fan

Immersion heaters

Thermocouple protection or alternate temperature sensors

Sampling and analysis containers

Bubblers

Gas lances

Heat sources

Refactory lining, Troughs

Filters (bed, honeycomb, reticulated foam)

Ceramics For Tooling And Casting

Solidification ⇦ Casting ⇦ Mold, Crucible, Cupola, Core, Filter Designs and Compositions ⇦ Preparation of Mold, and other Hardware

Temperature measurement

Heat flow

Temperature measurement

Molten metal pumps

Mold and face coat

Crucible

Inertness

Core

Filter

Dimensional stability

Furnaces

Fixtures, tooling

Ceramics For Post-Casting Steps

Mold, Core Removal,Recycling	Finishing	Inspection	Heat Treatment
Sand blasting	Cutting and grinding wheels	Wear-resistant surfaces of measurement tools	Furnace lining
Recycling	Waterjet or laser cutting	Optical benches	Burners
	Abrasives (sand blasting, peening)	Ultrasonics	Fixtures
		Radiography	Circulation Fan
		Laser	Heat exchanger
			Pollution control
			Temperature control
			Bearings for Conveyors

Steel Industry

- Two Segments
  - Integrated Steel Mill
    - » Source of highest quality Steel
    - » 21 U.S. Operations remain
  - Mini-Mill
    - » Use scrap as feedstock
    - » 40% market share but growing
- Three primary operations
  - Melting
  - Refining and casting (70% of steel cost)
  - Forming and finishing
- End product cost and quality key drivers

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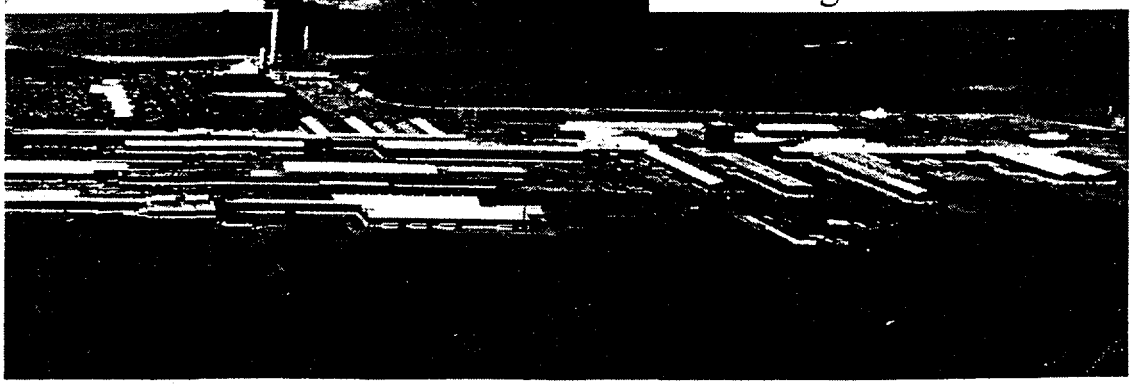
Mini-Mill

Steel Industry

Integrated Mill



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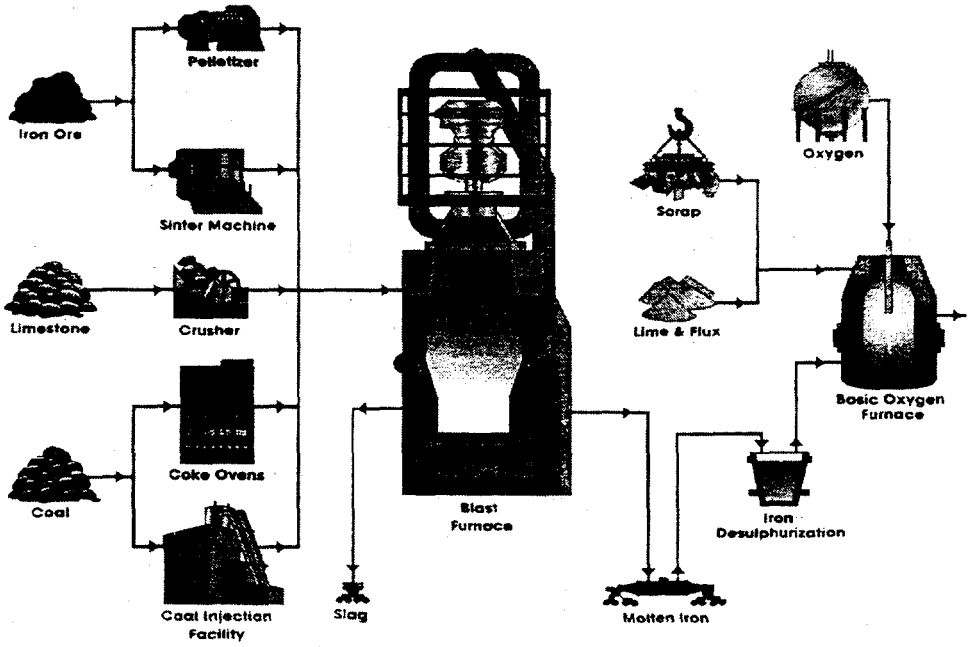
Integrated Steel Mill Melting Operation



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Integrated Steel Mill Melting Operation

- Current applications of ceramics
  - Refractory linings
  - Runners
  - Tap hole sleeves
  - Coke rolls

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Integrated Steel Mill Melting Operation

- Opportunities
  - Sensor shields (Basic Oxygen Firing temperature measurement)
  - Wear surfaces (coal injection, crusher, pelletizer, flue particle separators)
  - Tap hole gun nozzle
  - Improved refractories in coal injection area of blast furnace and desulphurization ladle.

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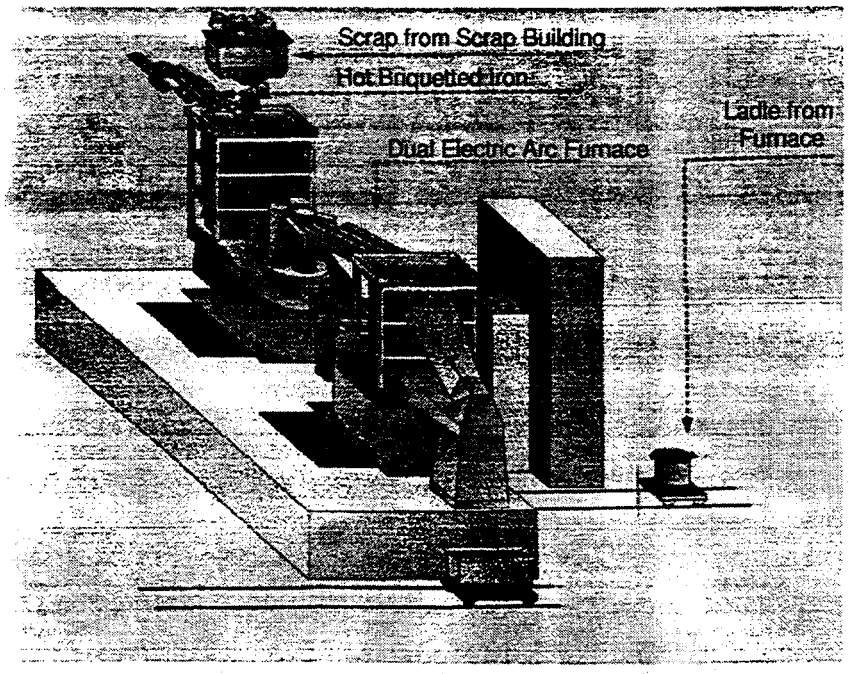
Mini-Steel Mill Melting Operation



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Mini-Steel Mill Melting Operation

- Current Applications of Ceramics
  - Tap hole sleeves
  - Runners
  - Refractory linings (MgO based)
  - Breast block
- Opportunities
  - Hot gas filtration
    - » PM2.5 (currently PM10)
    - » 400°F gas temperatures
    - » Highly corrosive fumes

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Mini-Steel Mill Melting Operation

- Opportunities (cont.)
  - Recuperator
    - » Current furnace efficiency 60%
    - » 2000°F, high alkalinity fumes
  - Below melt oxygen/carbon powder lances
    - » Currently consumable
  - Tap hole gun nozzle
    - » 2500-3000°F

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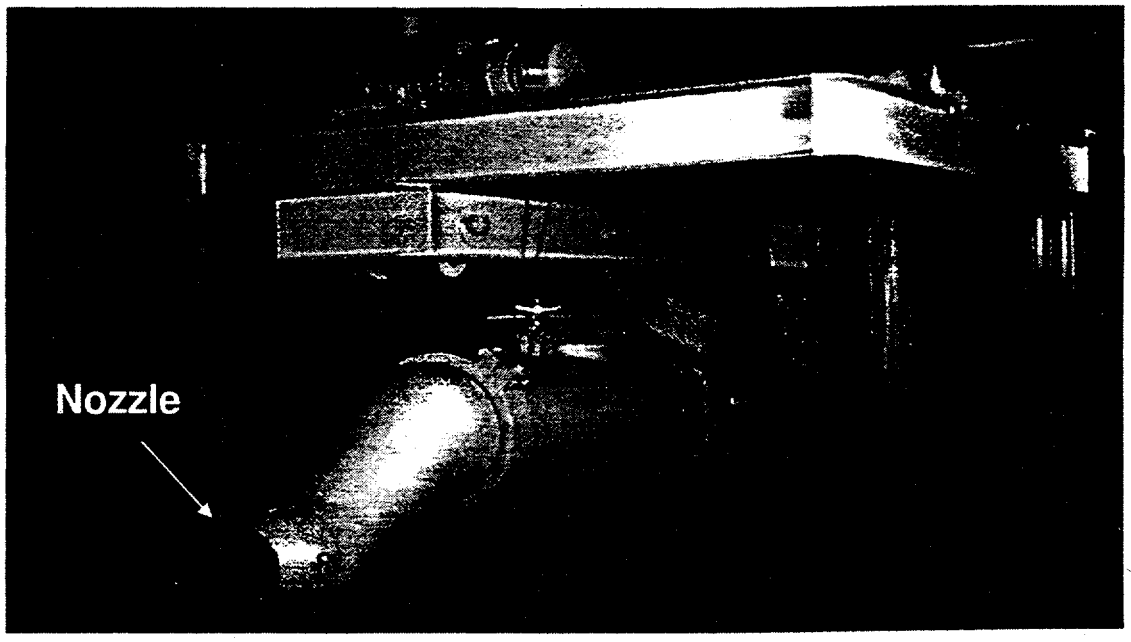
Tap Hole Gun



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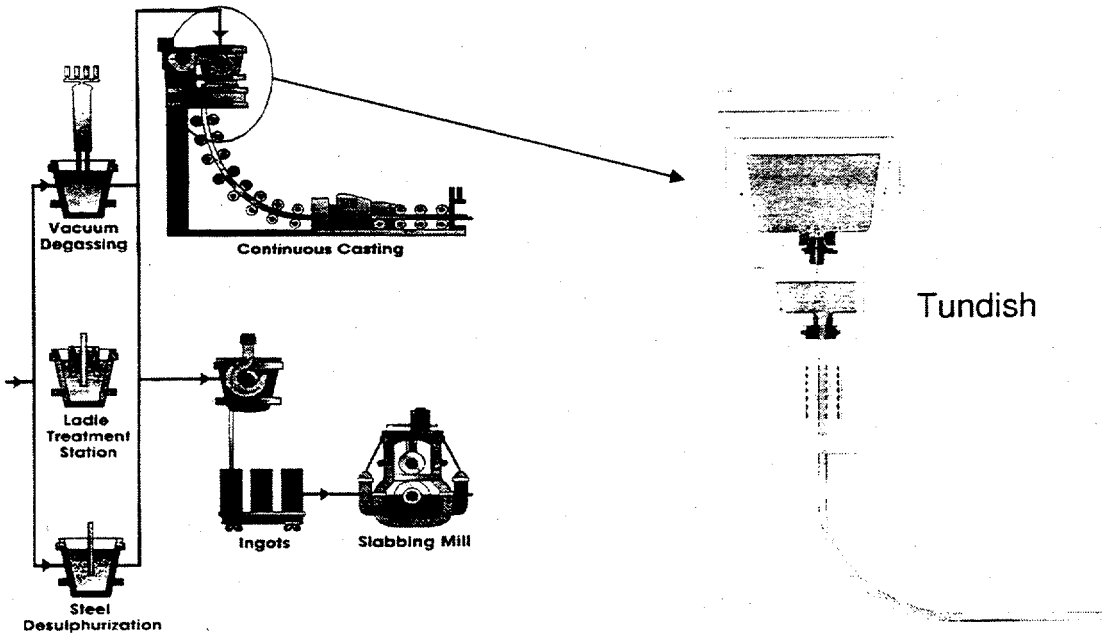
Refining and Casting Operations



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Refining and Casting Operations

- Current Applications of Ceramics
  - Refractory linings (Al_2O_3 based)
  - Ladle pour nozzles
  - Ladle impact pads
  - Tundish nozzle and socket block
  - Tap hole sleeves
  - Tundish dams/baffles
  - Brake ring (connects copper mold to tundish nozzle)

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Refining and Casting Operations

- Current Applications of Ceramics (cont.)
  - Stopper plugs
  - Ladle shroud
  - Ladle gate valves
  - Porous plugs
  - Metal filters
  - Coated water cooled lances
  - Burner nozzles

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Refining and Casting Operations

- Opportunities
  - Low erosion refractories for the ladle slag line
  - Low wear and erosion, high surface finish, tundish nozzle
    - » Poor surface finish results in plugging
    - » Limits tundish life and controls processing sequence
  - Higher flow porous ladle plugs
  - Uncooled, longer life lances and tuyeres
    - » Water cooling reduces efficiency
    - » Tuyeres burn back and make flow control difficult.
  - Ingot molds

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Refining and Casting Operations

- Opportunities (cont.)
  - Longer life brake ring
    - » BN looks promising but lacks toughness
    - » Size not readily available
  - Longer life tundish dams/baffles
    - » Current materials a source of contamination
    - » Provides uniform tundish temperature and flow during casting
  - Heat shields for slag and temperature sensors
  - Ladle recuperators
    - » 1800-2100 ° F
    - » Preheat air

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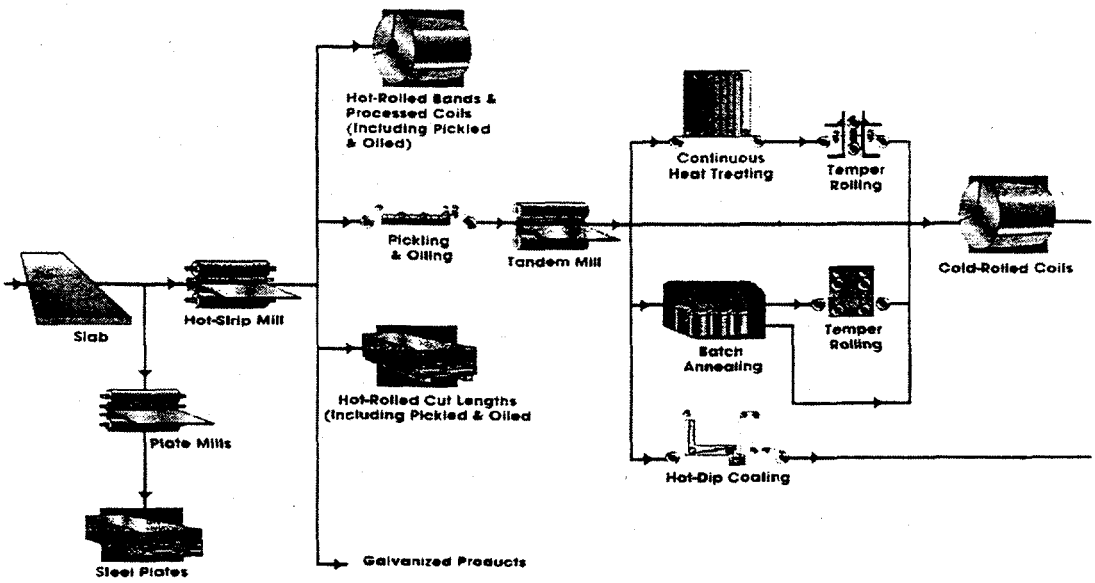
Forming and Finishing Operations



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Forming and Finishing Operations

- Current Applications of Ceramics
  - Descaling nozzles
  - Radiant burner tubes
  - Furnace furniture
  - Coated forming and conveying rolls
  - Fans
  - Corrosion resistant pump components for pickling
  - Refractory linings
  - Furnace heaters

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Forming and Finishing Operations

- Current Applications of Ceramics (cont.)
  - Forming dies
  - Wire Drawing guides/pulleys/cones
  - Burner nozzles
- Opportunities
  - Tougher radiant burner tubes
  - Pipe hangers
  - Improved finish rolls
    - » Low wear and corrosion, high surface finish

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Forming and Finishing Operations

- Opportunities (cont.)
  - Full ceramic fan and shaft
  - Chemical pump housings
  - Sensor shields for quality control
    - » Temperature
    - » Thickness
    - » Microstructure
  - Recuperators
    - » 40,000 potential sites
  - Bearings

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Forming and Finishing Operations

- Opportunities (cont.)
  - Uncooled conveyor components for use in continuous furnaces
  - Tougher, lightweight furnace furniture

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Glass Industry

- Four Segments
  - Container
  - Fiber
  - Flat
  - Specialty
- Specialty glass industry focus is higher temperature glasses and niche applications (60,000 products).
- Four primary operations
  - Batching
  - Forming
  - Melting & Refining
  - Annealing
- Cost, quality, and increased productivity key drivers for investment.
- Fused silica, graphite, platinum, and cooled metals widely used.

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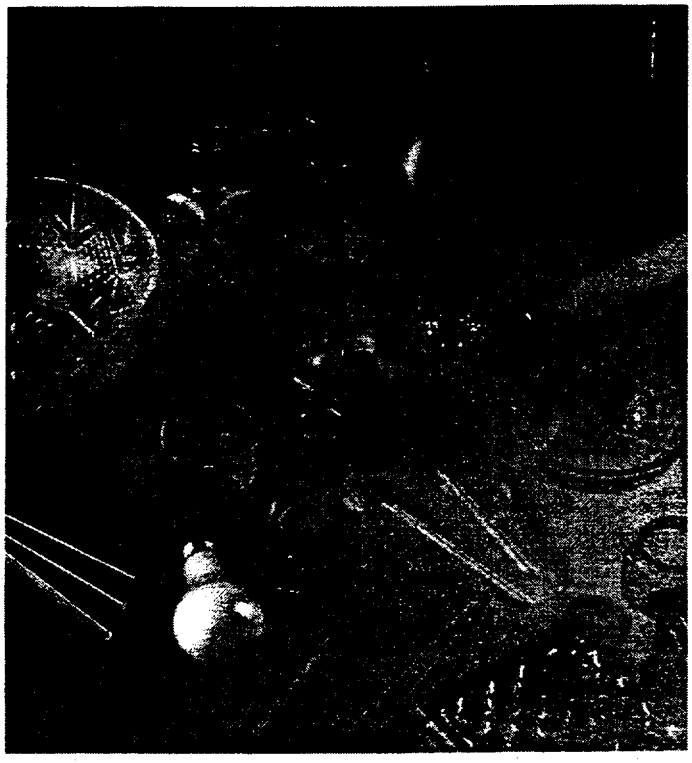
Glass Industry



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Batching

- Both raw materials and cullet.
- Contamination through ceramics (inclusions), metals (REDOX), and organics (REDOX)
- Current Applications of ceramics
  - Cullet pulverizer
  - Mixers
- Opportunities
  - Improved cullet pulverizer for reduced contamination
  - Batch preheaters
  - Sensors for contaminate detection

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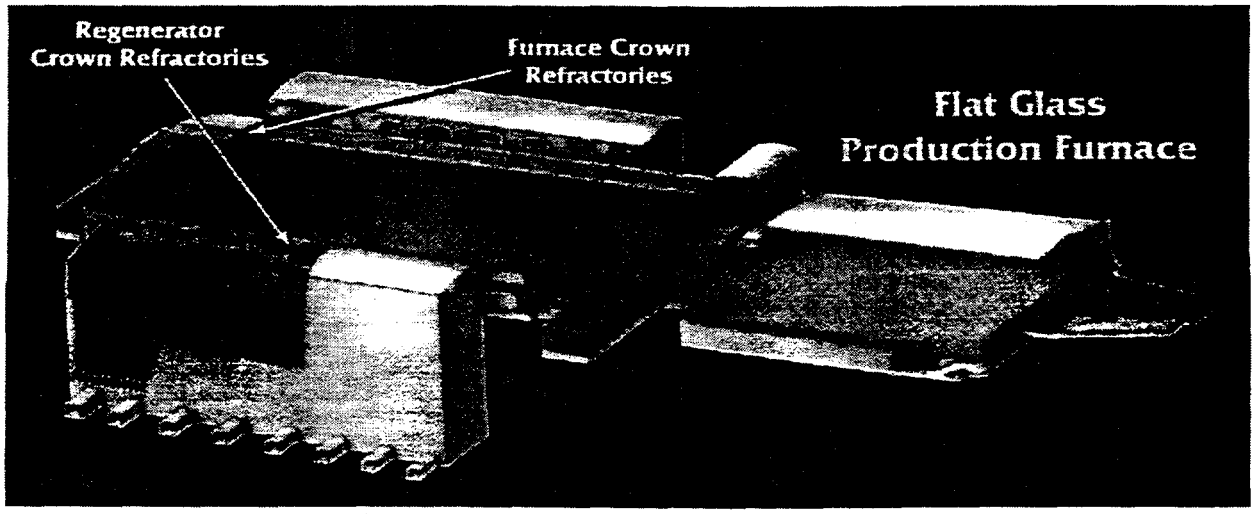
Melting & Refining Operations



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Melting & Refining Operations

- Over 500 glass melt furnaces in use
- Electric or combustion (predominate type)
- Current applications of ceramics
  - Refractories
  - Thermocouple shields (Al_2O_3)
  - Electrode blocks and holders
  - Gas injection tubes ($MoSi_2$)
  - Bag filters
  - Stopper rods

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Melting & Refining Operations

- Opportunities
  - Longer life, uncooled, lower cost stirrers
  - Refractories, sensors shields, bag filters, waste heat recovery, and burner nozzles for use with oxy-fuels
    - » 3100°F flue temperature, 60% moisture, high alkali
  - Higher temperature capable fan assembly for new combustion cycles
  - Uncooled heat exchanger in forehearth
  - Longer life burner orifices for conventional fuel fired furnace
    - » 1800-1900°F
  - Gas fired immersion boost heater
  - Uncooled camera sensor shield
  - Longer life stopper rods

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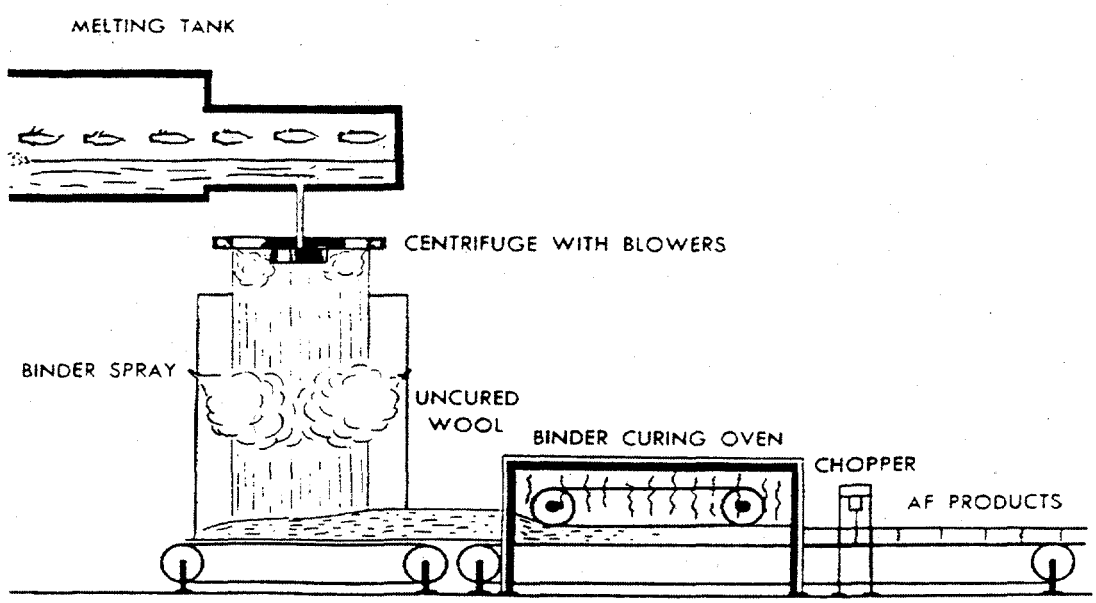
Fiber Forming Operation



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Fiber Forming Operation

- Two types
  - Wool
  - Continuous fiber
- Current Applications of Ceramics
  - Fiber handling guides
- Opportunities
  - Lower cost, longer life wire draw die
    - » 2,200°F, electric heated, platinum
  - Longer life spinner
    - » 2,000°F, 10,000-30,000 holes, high temperature base metal

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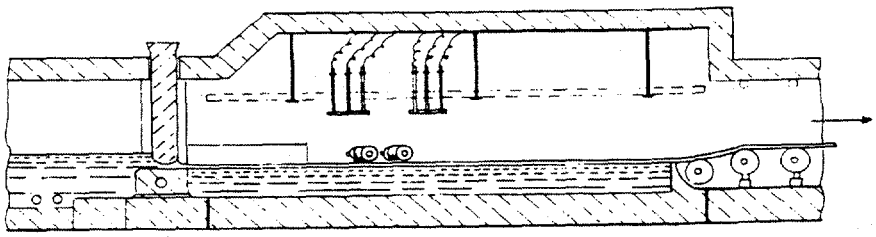
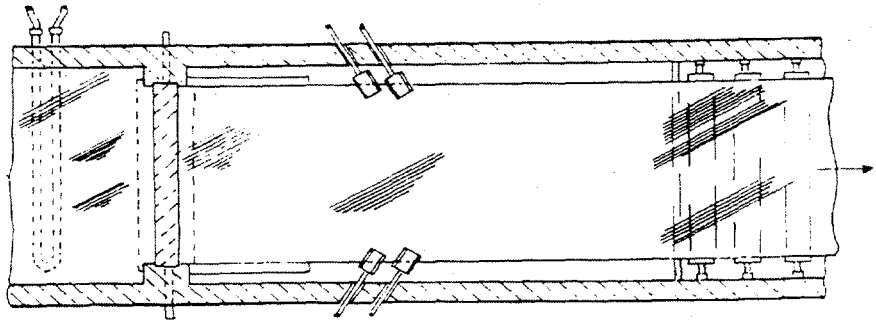
Flat Plate Forming Operation





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Flat Plate Forming Operation

- 2000°F operating temperature
- Current Applications of Ceramics
  - Tweel (SCFS)
  - Hot forming dies (fused silica)
  - Glo-bar heaters (SiC)
  - Forming rolls (fused silica)
- Opportunities
  - Sensor shields
  - Hot forming mold barrier
  - Longer life tweel

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Flat Plate Forming Operation

- Opportunities (cont.)
  - Abrasion resistant, lube free hot forming dies
  - Radiant burners
    - » reduce contamination caused by condensation on glo-bars
  - Hot handling equipment (mechanical drives)
  - Transport rolls

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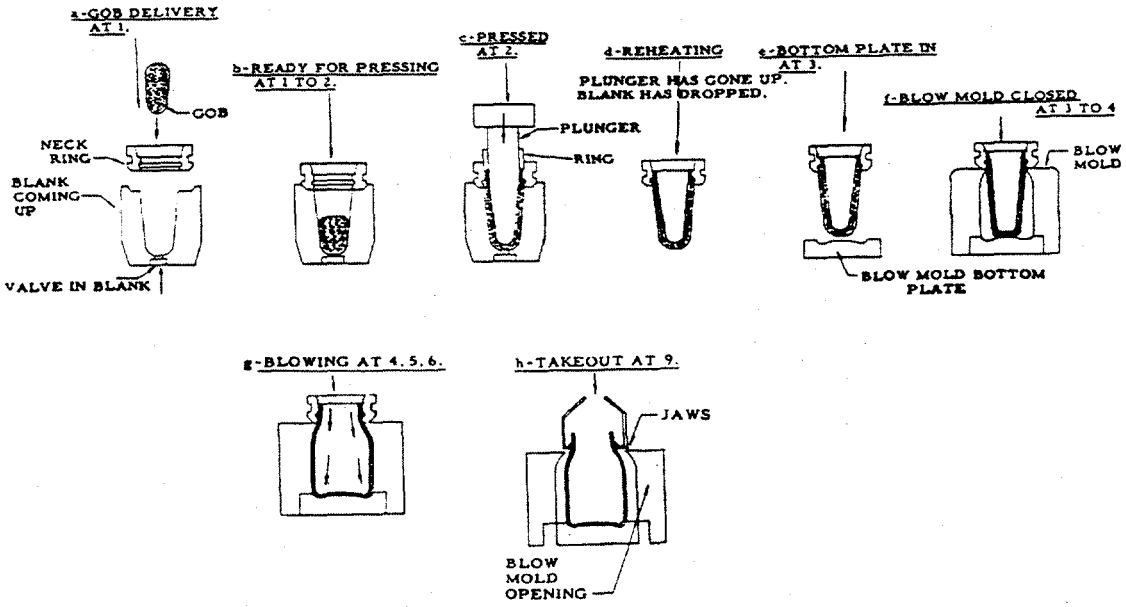
Container Forming Operation



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Container Forming Operation

- 2700°F glob temperature
- Current Applications of Ceramics
  - Mold coatings (TiC)
  - Setters (TiC)
  - Take out inserts (TiC
- Opportunities
  - Higher hot hardness mold materials
    - Reduce metal contamination
  - Materials with increased abrasion resistance for drive mechanisms
  - More durable conveyor guides, gob funnels, tong tips, blow head inserts
  - Sensor shield for gob temperature

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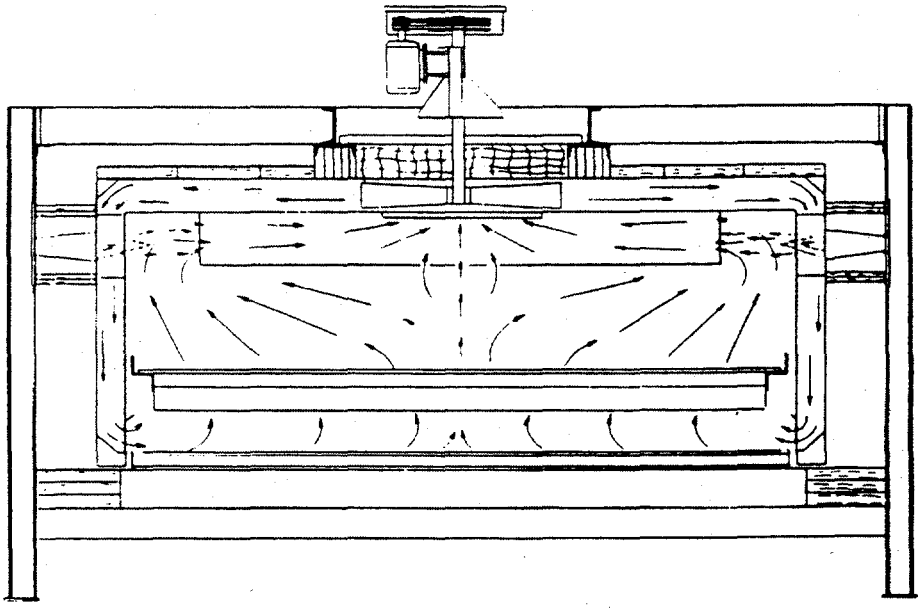
Annealing Operation



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Annealing Operation

- 400-500°F typical operating temperature
- Batch and continuous operation
- Current Applications of Ceramics
  - Transport rolls (SCFS or coated metal)
- Opportunities
  - Reduced roll damage
  - High temperature capable fan assembly
  - Conveyor materials
    - Uncooled, high abrasion resistance, oxidation resistance
  - Longer life trays
  - Longer life PAD and stacker materials

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Chemical Industry Overview

- Large, complex, diverse industry
- Over 70,000 products
- Materials Technology Institute serves as an entry point for new materials
- Wear and corrosion major problems
- Higher temperature desired to increase reaction rates
- High focus on safety
- Use of monolithic ceramics growing

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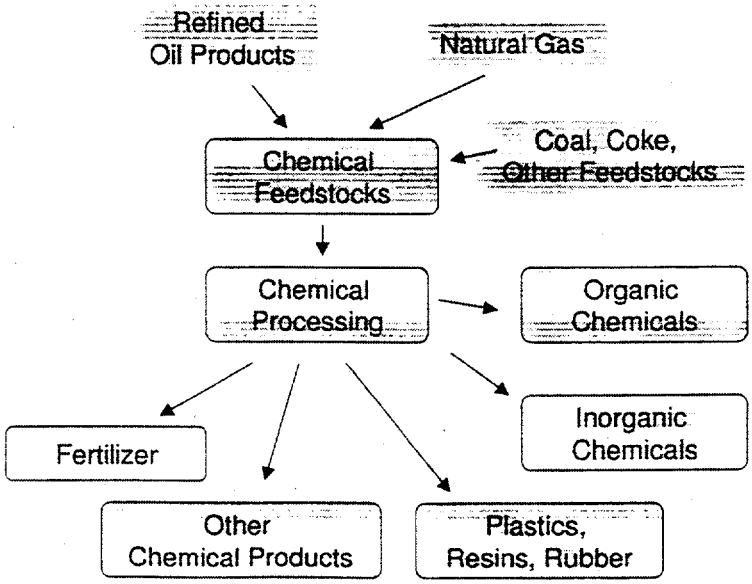
Chemical Industry Overview



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Ceramic Materials have Potential to Benefit Chemical Industry

Need/Opportunity	Benefit
Fluid Handling, Incinerator lining, Boiler tube sleeves	Extend life, lower maintenance
Dies/molds, Textile handling	Extend life, increase capacity and quality
Sensor shields	Extend life, lower maintenance, in-line control
Reactor vessel lining	Extend life, increase temperature
Furnace/Reformer tubes	Reduce fouling, increase temperature
Tube Sheets/Supports	Extend life, reduce weight and size, increase temp
Separation	Increase temperature, extend life, improve quality
Spray Nozzles, Rolls	Extend life, increase quality
Heat Exchangers	Extend life, increase efficiency
Cutters	Extend life, increase quality, reduce contaminatio

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Ceramic Materials are Currently in use in the Chemical Industry

Applications

- Fluid Handling
- Dies/molds
- Textile Handling
- Boiler Tube Sleeves
- Separation
- Spray Nozzles
- Heat Exchangers
- Rolls
- Cutters
- Refractories

Materials

- Silicon Carbide
- Alumina
- Silicon Nitride
- Boron Carbide
- Zirconia
- Tungsten Carbide
- Titanium Carbide

Limitations

- Cost
- Wear Resistance
- Size
- Toughness
- Reliability

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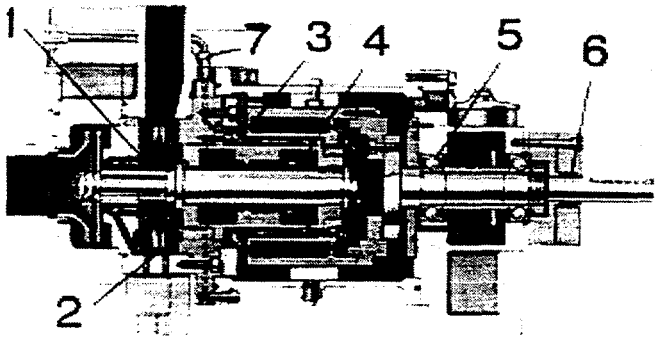
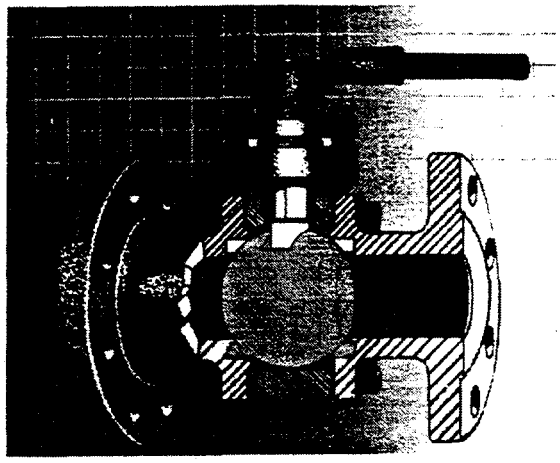
Fluid Handling offers a Growing Market Potential for Ceramics





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Fluid Handling offers a Growing Market Potential for Ceramics

- Pumps, valves, transfer pipes, fans
- Reliability a problem (corrosion, wear)
- Siliconized cast iron, Hastalloy or tantalum commonly used for pumps and valves.
- Ceramic coatings used in valves
- Plastic or refractory lined steel used for pipes
- Exotic metals, PMC, Ti alloy, and WC Coated steel used for fans
- Monolithics (SiC, Al_2O_3) being used for seals, bearings, shafts, and impellers but brittle
- Ceramic pump housings desired but remain costly due to complexity and brittle
- Pumps: 5″ - 14″ dia. impellers
- Valves: 2″ - 10″ dia. ball, 3′ - 5′ butterfly
- Fans: 2′ - 6′ dia.
- Pipes: up to 3′ x 100′ long

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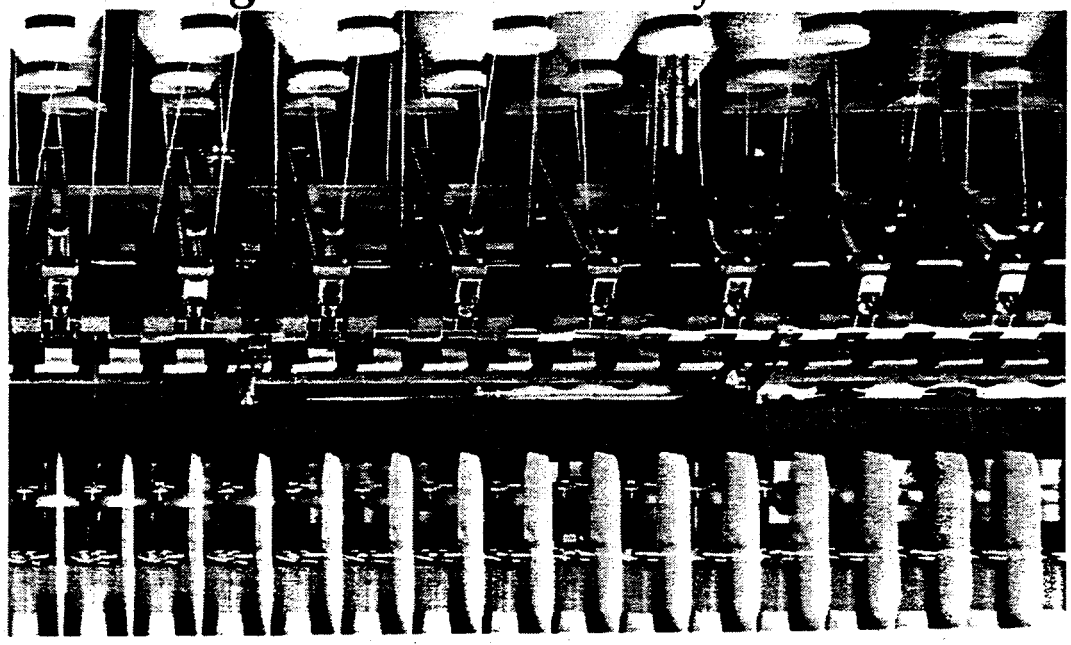
Finish Product Applications offer a Growing Market Potential for Ceramics



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Finish Product Applications offer a Growing Market Potential for Ceramics

- Used to further process chemicals into textiles, films, plastic goods, etc.
- High wear resistance for good dimensional stability, good surface finish, and increased production capacity
- High value added applications
- Large number of small parts (~1,000 per textile machine)
- Rolls: 12" dia. X 6' - 12' L, 20 per machine
- Al_2O_3 limited life for high wear, Si_3N_4 better but chips. TiC, WC coatings.
- Pore free coatings for rolls due to corrosion.
- Commonly used in open face machines. Catastrophic failure of concern.

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Refinery Industry Overview

- Transforms crude oil into various petroleum products
- Both separation and conversion processing operations
- Continuous operation
- Capital intensive with a large number of fluid/vapor handling equipment, sensors, heat exchangers, reaction vessels, and furnaces
- Corrosion, oxidation, wear, fouling and high temperatures a source of problems

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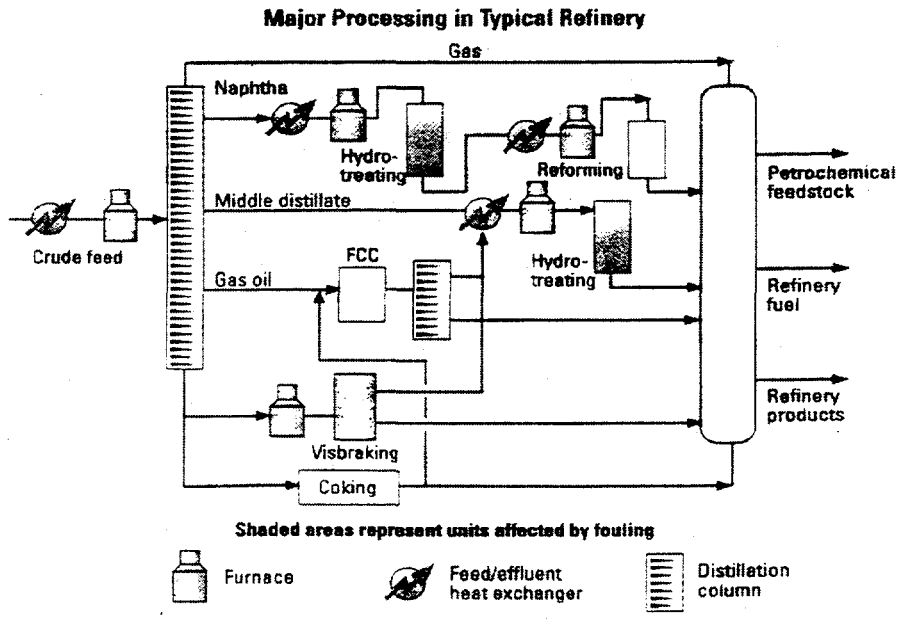
Refinery Industry Overview



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• Ceramic Materials have Potential to Benefit Refinery Industry

Need/Opportunity	Benefit
• Cyclones	• Reduce wear, corrosion, thermal degradation
• Sensor shields	• Increase reliability, end product quality
• Furnace/Reformer tubes, Heat Exchangers	• Reduce fouling, increase temperature
• Fluid Handling (pumps, valves)	• Reduce wear, corrosion
• Tube Sheets/Supports	• Reduce weight, corrosion
• Ceramic Membranes	• Increase temperature, efficiency, life
• Spray Nozzles	• Reduce wear

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Ceramic Materials are Currently in use in the Refinery Industry

Applications

- Spray Nozzles
- Valves
- Sensor Shields

Pump Seals/housings/bearings
Refractories

Materials

- Silicon Carbide
- Boron Carbide

Tungsten Carbide Cobalt

Limitations

- Cost
- Shape
- Size

Toughness
Strength

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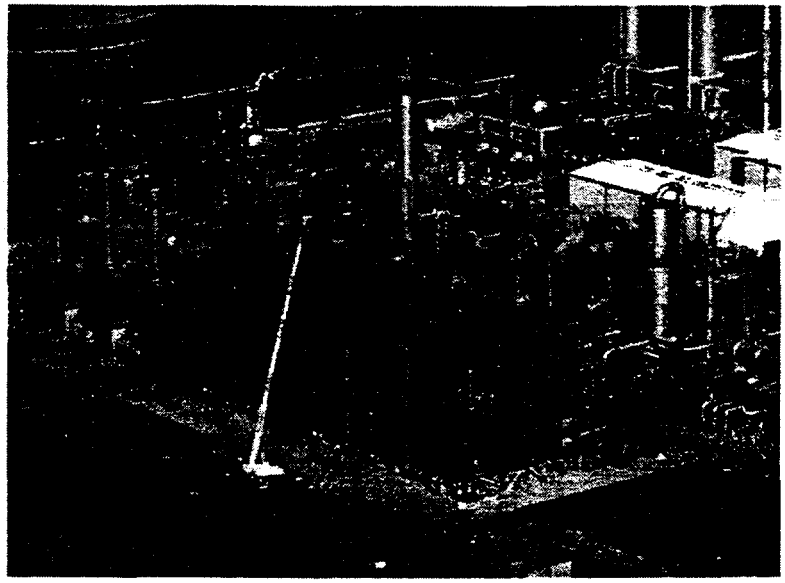
Ceramic Furnace/Reformer Tubes are a Potentially High Volume Market



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Ceramic Furnace/Reformer Tubes are a Potentially High Volume Market

- Ni, Cr steels currently used
- Higher temperature capability sought to increase efficiency and throughput
- Creep, corrosion, oxidation, erosion, and fouling a problem
- Defoul through additives or steam injection (20 - 100 day cycle)
- SiC shown to reduce fouling
- Monolithics brittle and low strength
- Ceramic coatings spall
- Requirements
  - 1"-8" Dia. X 10'-40'L
  - Impermeable to liquid or vapor
  - ASME codes
  - 1000°F - 2500°F/40-100 Psi
  - 180° bends
- Market size
  - > 100, 000 furnace tubes/plant
  - > 4,000 reformer tubes/plant x > 120 plants

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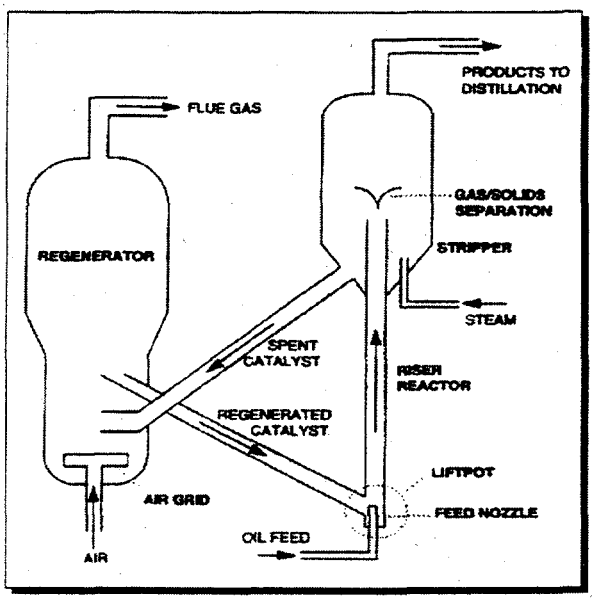
While Cyclones Provide an Opportunity for Ceramics, Shapes are Large and Complex



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While Cyclones Provide an Opportunity for Ceramics, Shapes are Large and Complex

- Fluid Catalytic Cracking (riser regenerator, separator, slide valves)
- 2,000 - 3,000 units in service
- High heat (1500°F), wear (powder catalyst), corrosion (sulfur) and oxidation.
- 1′ - 5′ dia. X 10′ - 20′ high
- Current strategy is to increase metal thickness to compensate for high wear
- Ceramic coatings commonly used due to large component size
- Monolithic ceramics brittle
- Refractory linings spall or mechanical failure

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Applications within IOF where Advanced Ceramics Provide the Greatest Return on Investment

	Glass	Refinery	Steel	Aluminum	Chemical	Forest Products	Metal Casting
Burners	X	X	X	X	X	X	X
High Temp. Insulation	X	X	X	X	X	X	X
Heat Recovery	X	X	X	X	X	X	X
Sensor Shields	X	X	X	X	X	X	X
Separation	X	X	X	X	X	X	X
Handling/Transport	X	X	X	X	X	X	X
Rollers	X		X	X	X	X	
Gas Injection	X		X	X			X
Gates/Valves	X	X	X	X	X	X	X
Fans/Stirrers	X	X	X	X	X	X	X
Molds/Tooling	X						X
Supports	X	X	X	X	X	X	X

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Summary/Conclusions

- Ceramic materials are currently being used in Industries of the Future.
- A new generation of advanced ceramic technologies are emerging as a result of DOE/Industry partnerships.
- This new generation of advanced ceramic technologies provide a significant opportunity to increase energy efficiency and solve environmental challenges in Industries of the Future.

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Preliminary Study Recommendations

- Expand efforts to make IOF industries aware of advanced ceramic technologies which have emerged as a result of DOE/Industry partnerships.
  - Establish a clearinghouse to serve as a conduit between the IOF companies and ceramics industry.
  - Perform materials audits of IOF companies to better identify opportunities for advanced ceramics insertion.
- Increase use of IOF field trials to demonstrate materials capability and accelerate technology transition
- Continue focus on advanced ceramics cost reduction and scale-up through manufacturing technology programs.
- Support the development of technologies which reduce barriers to system integration and maintenance of advanced ceramics.

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Design Codes and Data Bases: The CFCC Program and Its Involvement in ASTM, ISO, ASME and Military Handbook 17 Activities

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Lake Tahoe, Nevada - October 1997

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Outline

- *Background*
- *Code Activities*
  - *Military Handbook 17*
  - *ASME*
- *Standards Activities*
  - *ASTM - Standards Development & Organizational Structure*
  - *International Harmonization*
  - *ISO*
  - *CEN*
- *Validation of Standards*
- *Summary and Future Work*

Background

What are Standard Tests?

- *For the researcher: fundamental test methodologies and units of measure*
- *For the manufacturer and end-product user: Materials specifications and tests to meet requirements*
- *Standards are the "rules" and terms for information transfer between designers, manufacturers and end-product users*

Background

Why are Standard Tests Necessary?

- ***Industrial acceptance***

- ***Lack of standards in the establishment of engineering data bases will limit commercial diffusion of materials and components***

Background

What is a Design Code?

For the design engineer and the manufacturer, design codes are the guidelines, data bases, design "rules," inspection requirements, and qualification procedures to implement materials or concepts into various applications. In some cases, design codes are even legal requirements for certain applications.

Background

Why are Design Codes Necessary?

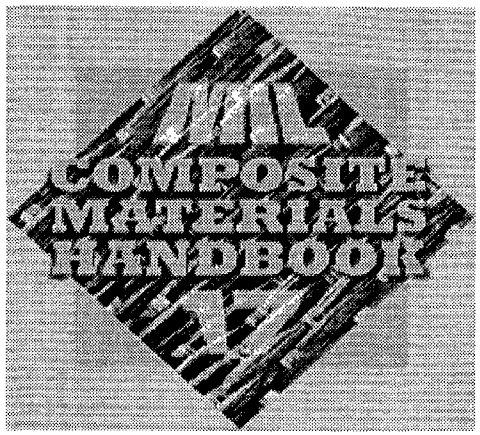
- *Industrial or vendor acceptance*
- *Lack of codes in the establishment of engineering data bases or accepted design procedures will limit commercial diffusion/acceptance of materials and components*
- *Legal Requirements*

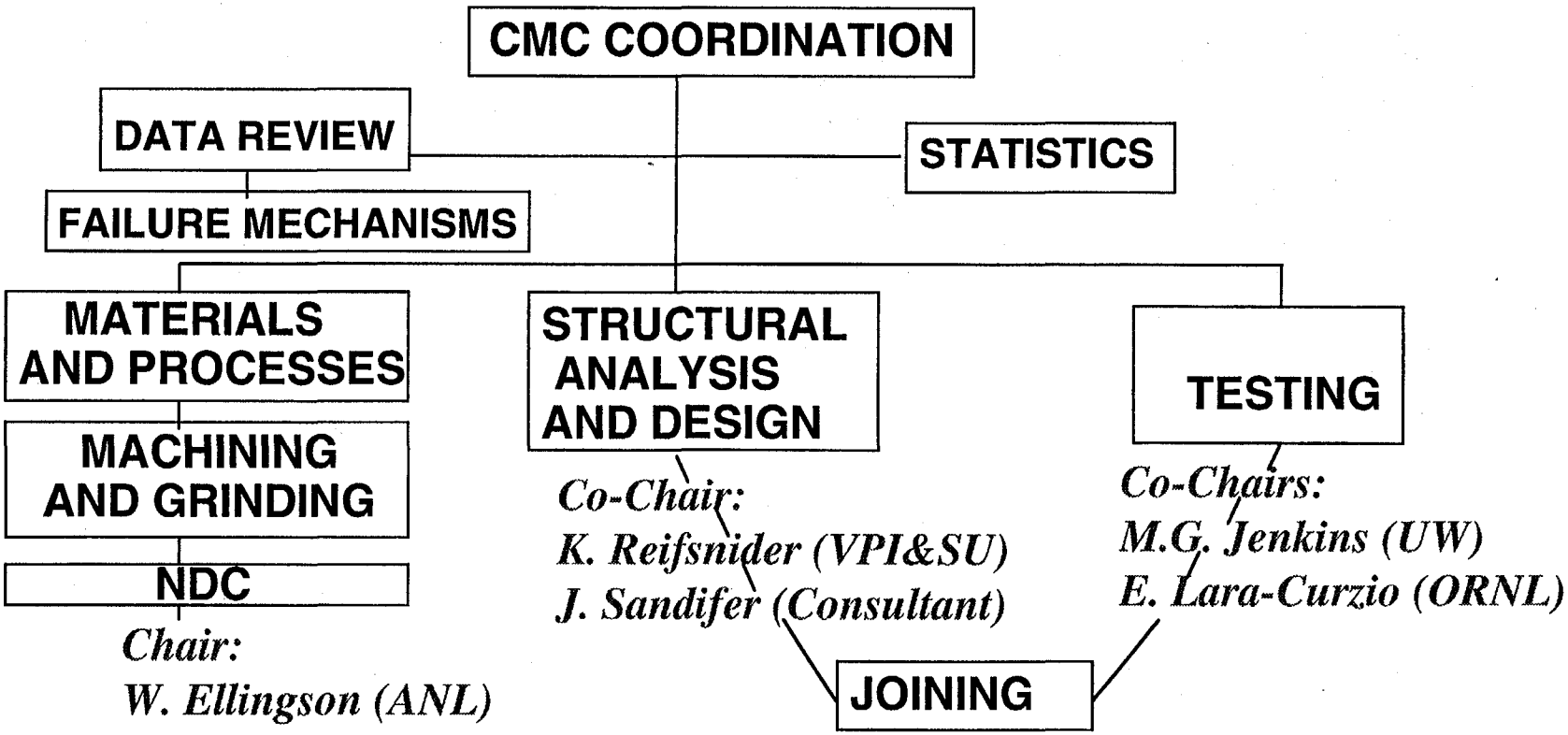
Code Activities

Mil-Hdbk-17

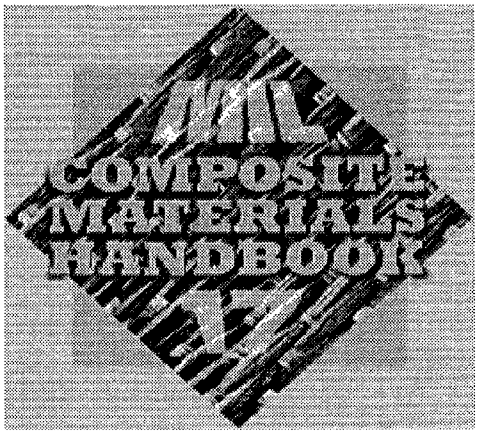
- *Guidelines, data bases, design methods for composites.*
- *PMC effort Initiated in 1959.*
  MMC effort initiated in 1993.
  CMC effort initiated in 1996.
- *CMC Leadership:*
  - *Chair - J.W. McCauley(U.S. Army Research Lab.)*
  - *Secretariat- J. Kibler (MSC through sub to Composites Optics, Inc.)*

Code Activities





Code Activities



Testing Working Group

Vision:

To be the primary and authoritative source for recommended/required methods for testing and characterization of CMC's and their constituent materials.

Code Activities

Testing Working Group

Goals:

- ***To identify appropriate existing consensus standard test methods for CMCs and their constituent materials.***
- ***To assist in the development of appropriate standard test methods for CMCs and their constituent materials.***

Code Activities

Testing Working Group

Objectives:

- ***Recommend appropriate existing standards for CMCs and their constituent materials for the Handbook.***
- ***Recommend appropriate new standards for CMCs and their constituent materials for the Handbook***

Code Activities

Testing Working Group

Examples of Existing Standards for CFCCs

Requirement	ASTM	CEN	ISO	HSR/EPM	PEC
Bulk CFCC Room Temp.					
Tension					
In-plane	C1275	ENV658-1	WG9	D-001-93	TS CMC01
Transverse	----	----	----	----	----
Flexure					
Bending	C1341	ENV658-3	----	D-003-93	TS CMC04
Shear	----	ENV658-5	----	----	----

Code Activities

Testing Working Group

Examples of Needs for Standards for CFCCs

Requirement	ASTM	CEN	ISO	HSR/EPM	PEC
Bulk CFCC High Temp. Control. Environ.					
Tension					
In-plane	----	----	----	----	----
Transverse	----	----	----	----	----
Flexure					
Bending	----	----	----	----	----
Shear	----	----	----	----	----

Code Activities

Testing Working Group

Acceptable Existing Standards Must be Selected and Values for Test Parameters be Chosen for "Qualifying" Materials.....For Example:

Tensile Test

In-plane tensile behaivour

R.T. ASTM C1275-95 - Test control: displacement?
Test rate: 0.005-0.05 mm/s (20-30 s)?
Specimen geometry: min. 25 mm gage length?
Number of Tests: 10?

H.T. ASTM C1359-97- Test control: displacement?
Test rate: 0.005-0.05 mm/s?
Specimen geometry: min. 25 mm gage length?
Number of Tests: 10?
Test Temperatures: ?

Code Activities

ASME (American Society of Mechanical Engineers)

- ***Boiler and Pressure Vessel Code currently does not allow CFCCs but is the legal requirement for pressure equipment in 48 states.***
- ***Task Group on Graphite and Ceramic Pressure Equipment was initiated February, 1995.***
- ***Leadership:***
  - ***Subtask Chair: T. V. Narayanan (Foster Wheeler)***
  - ***Lead on Section: M. G. Jenkins (U. of Washington)***

Code Activities

PROPOSED SECTION OF THE CODE FOR CFCCS

- GENERAL REQUIREMENTS
- MATERIAL REQUIREMENTS
- DESIGN REQUIREMENTS (includes reliability/probabilistic)
- FABRICATION REQUIREMENTS
- QUALIFICATIONS REQUIREMENTS
- PRESSURE RELIEF REQUIREMENTS
- RULES GOVERNING TESTING
- INSPECTION REQUIREMENTS
- MARKING AND STAMPING

ASTM Activities

Existant ASTM Standards (1997)

CFCC
Industrial Participants
"wish list"

- *Tension*
- *Shear*
- *Compression*
- *Flexure*
- *Thermal Fatigue*
- *Interfacial Shear*
- *Tensile Fatigue*
- *Thermal Shock*
- *Fiber Tensile Testing*
- *Creep/Stress Rupture*

Tension

Shear

Compression

Flexure

Tensile Fatigue

Creep/Stress Rupture

ASTM Activities

ASTM STANDARDS

C1275-95	***Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Ambient Temperatures***
C1292-95	***Standard Test Method for Shear Strength of Continuous Fiber- Reinforced Advanced Ceramics at Ambient Temperatures***
C1341-96	***Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics***
C1337-96	***Standard Test Method for Creep and Creep Rupture of Continuous Fiber- Reinforced Advanced Ceramics under Tensile Loading***

ASTM Activities

ASTM STANDARDS

C1358-97	*Standard Test Method for Monotonic Compressive Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Ambient Temperatures*
C1359-97	*Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Elevated Temperatures*
C1360-97	*Standard Practice for Constant-Amplitude, Axial, Tension-Tension Cyclic Fatigue of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures*
D3379-89	*Standard Test Method for Tensile Strength and Young's Modulus for High-Modulus Single Filament Materials*

Participation in ASTM

Committee C-28 Advanced Ceramics

C28.90
Executive Subcommittee

C28.93
Awards Subcommittee

C28.01
Properties and Performance

C28.02
Design and Evaluation

C28.05
Characterization and Processing

C28.07
Ceramic Matrix Composites

Chair: M. G. Jenkins
Secretary: E. Lara-Curzio

C28.91
Nomenclature

C28.94
ISO 206 TAG

Participation in ASTM

Sub Committee C-28.07 Ceramic Matrix Composites

Task Group C28.07.01
Tension (M. Jenkins-Leader)

Task Group C28.07.02
Compression (M. Jenkins-Leader)

Task Group C28.07.03
Creep(E. Lara-Curzio-Leader)

Task Group C28.07.04
Flexure (S. Gonczy-Leader)

Task Group C28.07.05
Shear(E. Lara-Curzio-Leader)

Task Group C28.07.06
Cyclic Fatigue (M. Jenkins-Leader)

Task Group C28.07.07
Fibres (J. Porter-Leader)

Task Group C28.07.08
Interfacial (E. Lara-Curzio-Leader)

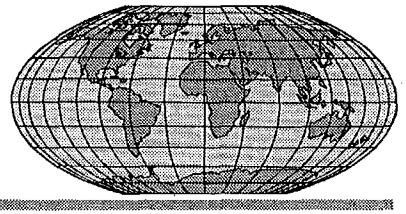
Task Group C28.07.09
Thermal (S. Gonczy-Leader)

Task Group C28.07.10
Environmental(P. Tortorelli?- Leader)

Task Group C28.07.11
TMF (M. Verilli?-Leader)

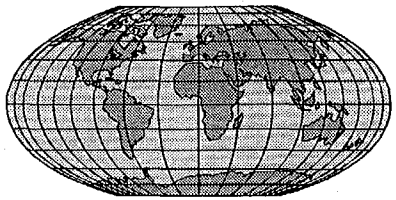
Task Group C28.07.01
Structural / Component (?-Leader)

International "Harmonization"



- *ASTM*
- *ISO (International Organization for Standardization)*
- *CEN TC-184 (Comité Européen de Normalisation)*
- *Japan Fine Ceramics Association*
- *JISC (Japanese Industrial Standards Committee)*

International "Harmonization"



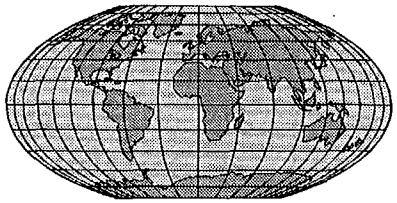
U. S. Technical Advisory Groups (ANSI)

Approval of Accreditation
U. S. TAG to ISO/TC 206 -
Fine (Advanced, Technical) Ceramics

The Executive Standards Council (ANSI) has approved the accreditation of the U. S. TAG to ISO/TC 206-Fine Ceramics, effective March 29, 1993.

ASTM will serve as TAG Administrator

International "Harmonization"



FIRST ISO STANDARD
FOR MECHANICAL BEHAVIOUR OF CFCCs

WG9 Draft:

Fine ceramics (Advanced ceramics, Advanced technical ceramics)
--Tensile (in-plane) Behaviour of Continuous Fibre-reinforced Ceramic Composites at Ambient Temperature

- ***This draft is part of stage 2 of the working group.***
- ***The U.S., through ANSI and ASTM, is the convener with M.G. Jenkins as chair of the working group.***
- ***Projected date of approval is 1999-2000 time frame.***

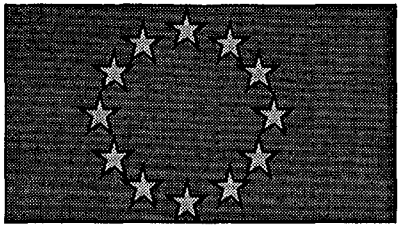
International "Harmonization"

FORMAL LIAISON BETWEEN

ASTM C28.07 AND CEN TC184 / SC1

- **ESTABLISHED SEPTEMBER 1996**
- **EXCHANGE OF DOCUMENTS**
- **COMMON EFFORTS FOR ISO STANDARDS**
- **COOPERATIVE ROUND-ROBIN EFFORTS**
- **JOINT TECHNICAL MEETINGS**

International "Harmonization"



CEN STANDARDS FOR MECHANICAL BEHAVIOUR

ENV 658-1 *Tensile Strength of Continuous Fibre Reinforced Ceramic Composites*

ENV 658-2 *Compressive Strength of Continuous Fibre Reinforced Ceramic Composites*

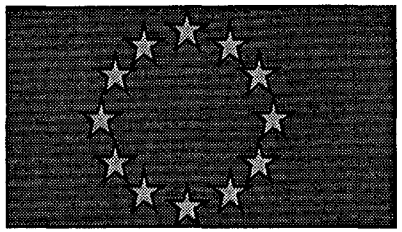
ENV 658-3 *Flexural Strength of Continuous Fibre Reinforced Ceramic Composites*

ENV 658-4 *Shear Strength (compression) of Continuous Fibre Reinforced Ceramic Composites*

ENV 658-5 *Shear Strength (3-point) of Continuous Fibre Reinforced Ceramic Composites*

ENV 658-6 *Shear Strength (double punch)of Continuous Fibre Reinforced Ceramic Composites*

International "Harmonization"



CEN STANDARDS FOR THERMAL PROPERTIES

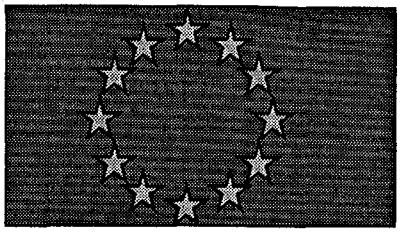
ENV 1159-1 ***Thermal Expansion of Continuous Fibre Reinforced Ceramic Composites***

ENV 1159-2 ***Thermal Diffusivity of Continuous Fibre Reinforced Ceramic Composites***

ENV 1159-3 ***Specific Heat of Continuous Fibre Reinforced Ceramic Composites***

ENV 1389 ***Density of Continuous Fibre Reinforced Ceramic Composites***

International "Harmonization"



CEN STANDARDS FOR FIBRE PROPERTIES

ENV 1007-1 ***Size Level of Fibres for Continuous Fibre Reinforced Ceramic Composites***

ENV 1007-2 ***Linear Mass of Fibres for Continuous Fibre Reinforced Ceramic Composites***

ENV 1007-3 ***Filament Diameter of Fibres for Continuous Fibre Reinforced Ceramic Composites***

ENV 1007-4 ***Filament Strength of Fibres for Continuous Fibre Reinforced Ceramic Composites***

Standards Validation

Goals of a recently-initiated round-robin

- ***Precision and bias for each of the national standards evaluated (ASTM-required)***
- ***Expanded, reliable data base for CFCCs (CFCC Program)***
- ***Statistical distributions of properties and performance of a single CFCC for a large statistical sample for design purposes (e.g., Mil-Hdbk-17)***
- ***Statistical distributions of properties and performance of a single CFCC for a large statistical sample for production purposes (CFCC Program)***

Standards Validation

What will be done in the round-robin ?

- ***Multiple-laboratory round-robin evaluation of three ASTM standards (C1275-95 for R.T. tensile behaviour, C1292-95 for interlaminar and in-plane shear strengths, and C1341-96 for flexural behaviour)***
- ***Commercially-available CFCC will be evaluated***
- ***One hundred of each specimen type (four hundred total) will be tested by up to eight laboratories for each of the three ASTM test standards***
- ***ASTM E691 will be used to determine inter and intra-laboratory statistics for determining precision and bias for each of the test standards***

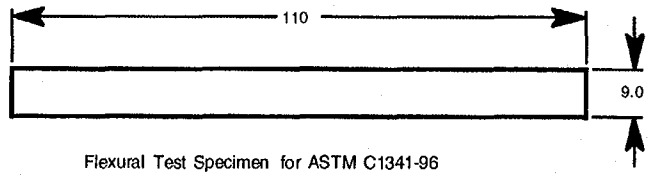
Standards Validation

Who will participate?

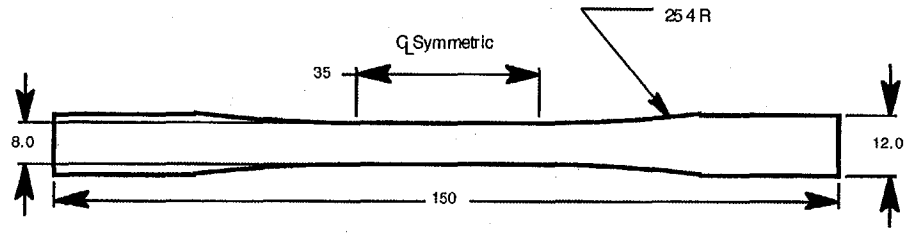
- ***Coordination: University of Washington (M. Jenkins) in collaboration with Gateway Materials Technology (S. Gonczy)***
- ***Academe: including Northwestern Univ., VPI&SU, UDRI, Univ. of Mich. and Univ. of Wash.***
- ***Industry: including Allied Signal, Ceramic Testing and Analysis, Cincinatti Test Lab, Dow Corning, DuPont Lanxide, GE, and Southern Research Inst.***
- ***Government: including NASA (Le), ORNL, and USAF (Wright Lab)***

Standards Validation

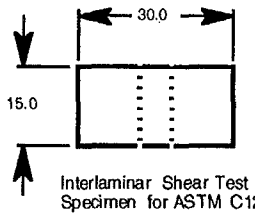
Test Specimens to be Used as Part of Round Robin



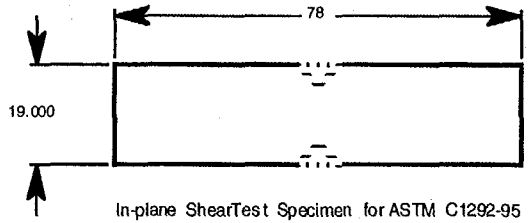
Flexural Test Specimen for ASTM C1341-96



Tensile Test Specimen for ASTM C1275-95



Interlaminar Shear Test Specimen for ASTM C1292-95



In-plane ShearTest Specimen for ASTM C1292-95

CFCC Round Robin Test Specimens
x=1, x.x=0.1, x.xx=0.01 mm

Summary and Future Work

- ***Mil-Hdbk-17 effort will establish a "qualified" data base and is utilizing standardization efforts, design methods, and processing lessons.***
- ***Draft of the section of ASME code for CFCC pressure equipment has been developed and is being refined***
- ***Round robin testing (tension, shear, and flexure) will be conducted to ascertain precision and bias, to provide input for data base development, and to foster cooperative exchanges among laboratories, agencies, and societies.***

Summary and Future Work

- ***Seven test methods have been standardized (ASTM)***
- ***Other draft standards are in ballot***
- ***ASTM Subcommittee C28.07 has 12 task groups which are extending standardization efforts into thermal, environmental, and structural areas***
- ***A draft ISO standard for tensile behaviour has been prepared based on ASTM C1275-95 and CEN ENV 658-1 --- U.S. is convener of this effort through ASTM and ANSI***
- ***Liaison has been established between ASTM and CEN for cooperative standardization work on CFCCs***

CFCC WORKING GROUP MEETING

OCTOBER 6-7, 1997
LAKE TAHOE, NEVADA

RICH GOETTLER
MCDERMOTT TECHNOLOGIES

CFCC Material Systems

Dense CFCC for structural applications

- Nextel™ 610 fiber (alumina)
- fugitive carbon fiber coating
- alumina (Al_2O_3) - YAG ($Y_3Al_5O_{12}$) matrix
- Nextel 720™ fiber (alumina mullite)
- scheelite ($CaWO_4$) fiber coating
- mullite-alumina matrix

Porous CFCC for filters and burners

- Nextel™ 610 fiber
- chopped fiber matrix (alumina and rare earth containing oxides)

CFCC Uses Evaluated by MTI

- Soot Blowers
- Tube Shields
- Turbine Combustor Liners
- Heat Exchanger Tubes (low and high pressure)
- Combustor Liners in Utility Boiler Burners
- Flame Stabilization Rings in Utility Boiler Burners
- Classified Components
- Hot Gas Filters (porous CFCC)
- TPV Power Generators (possible porous CFCC)
- Industrial Burners (porous CFCC)

McDERMOTT

M

Slurry Processing

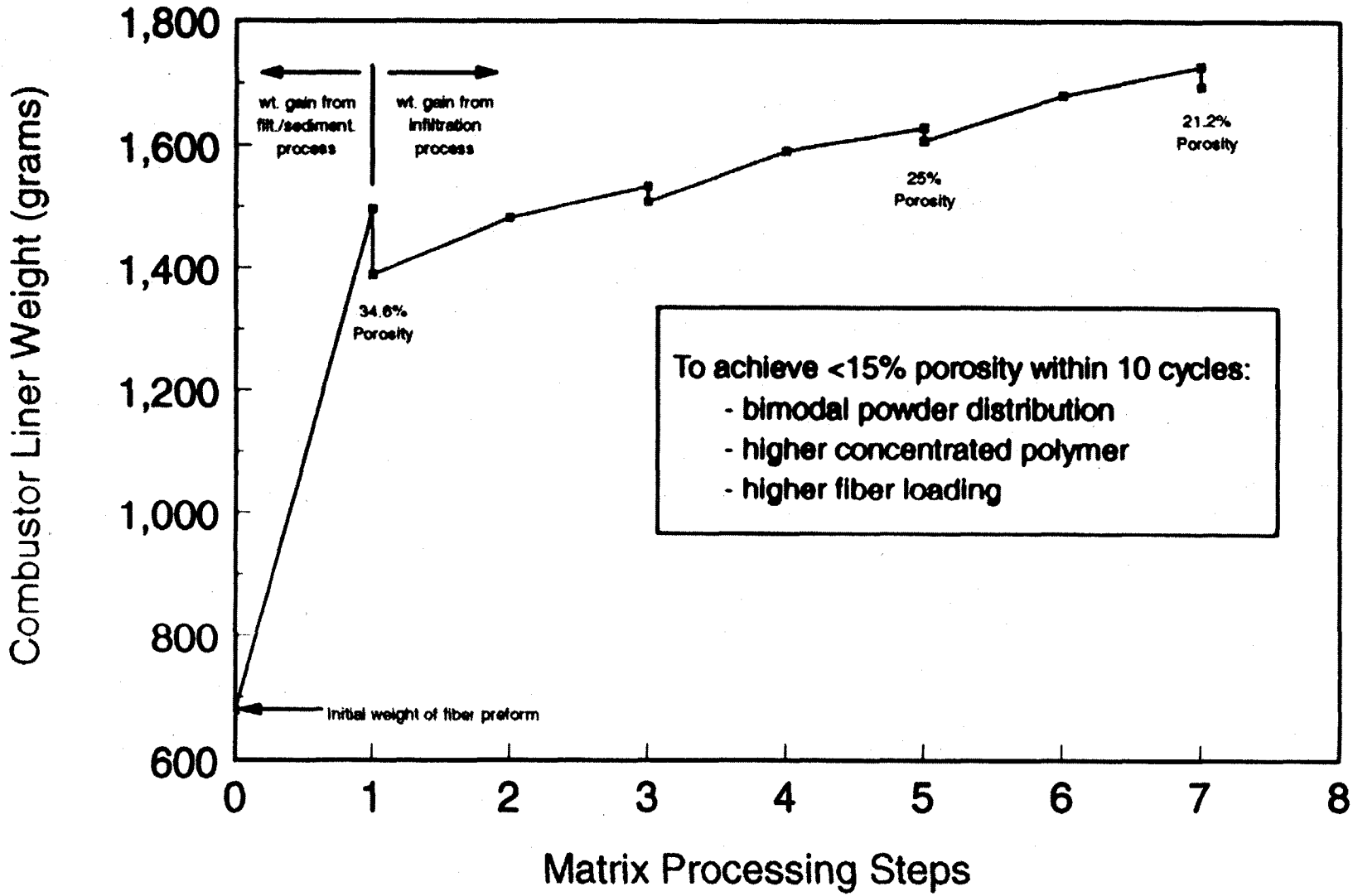
Infiltrations

to porosity <20%

Vacuum/Pressure

Infiltration

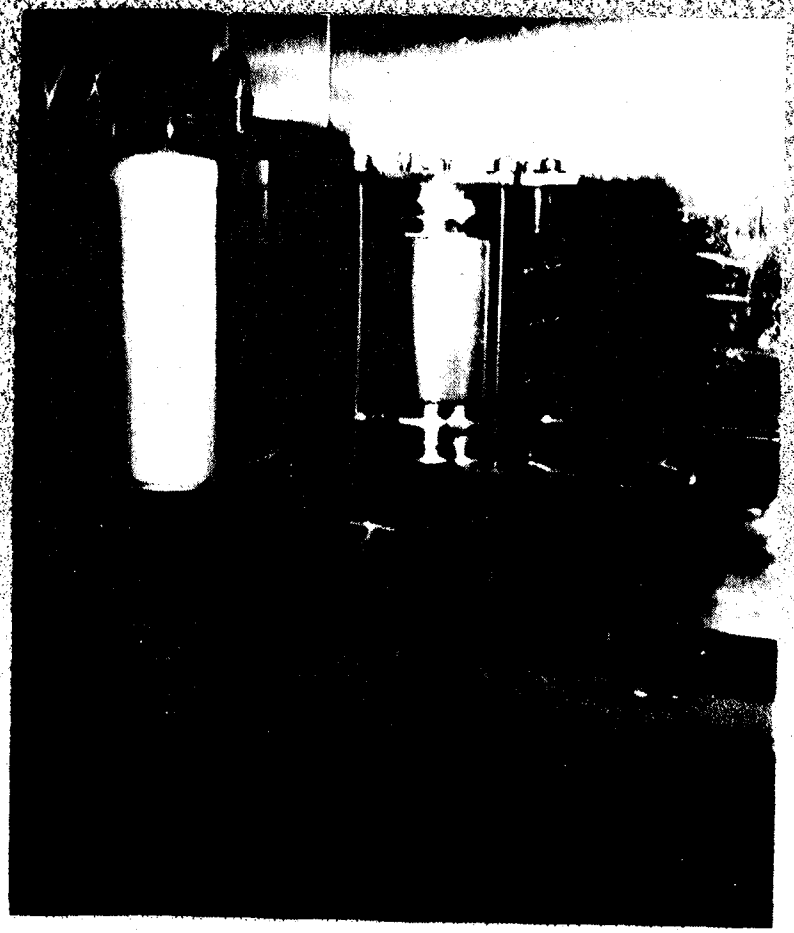
Processing History of Solar Turbine 3D Combustor Liner (fugitive interface)



lineproc

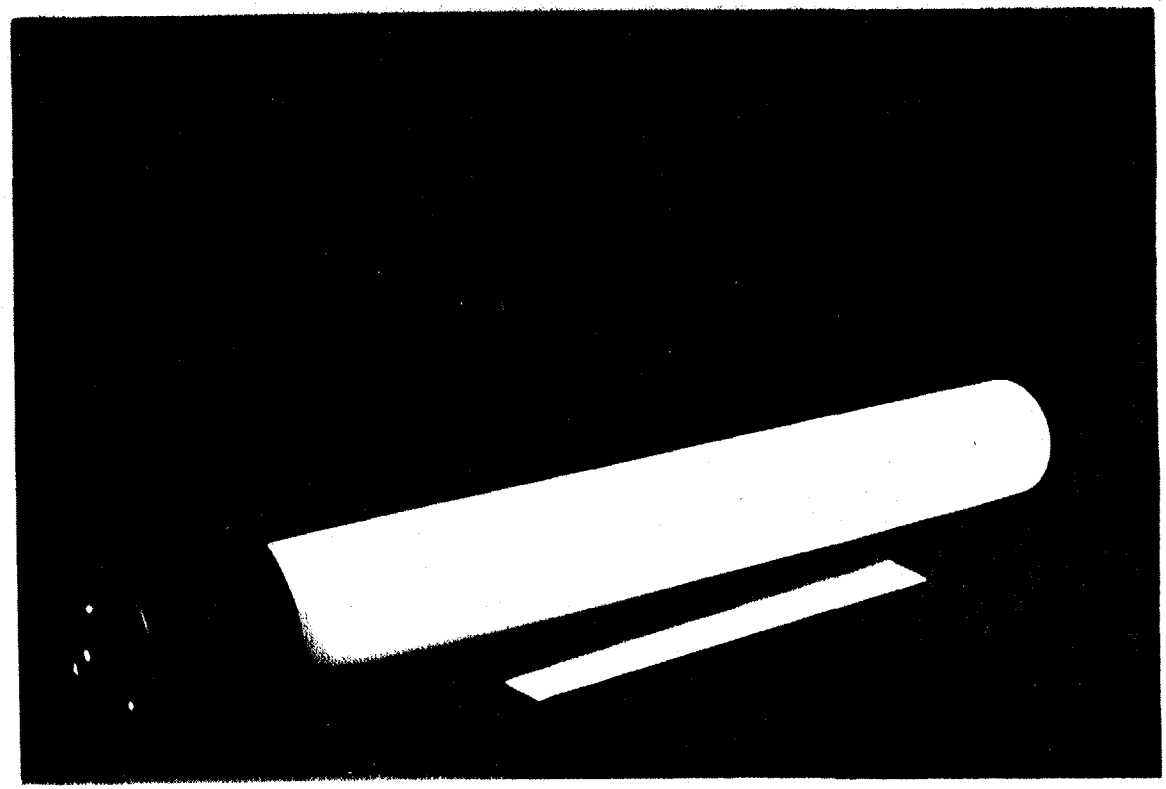
Typical Layup of CFCC Panels

Solar Liner Being Processed

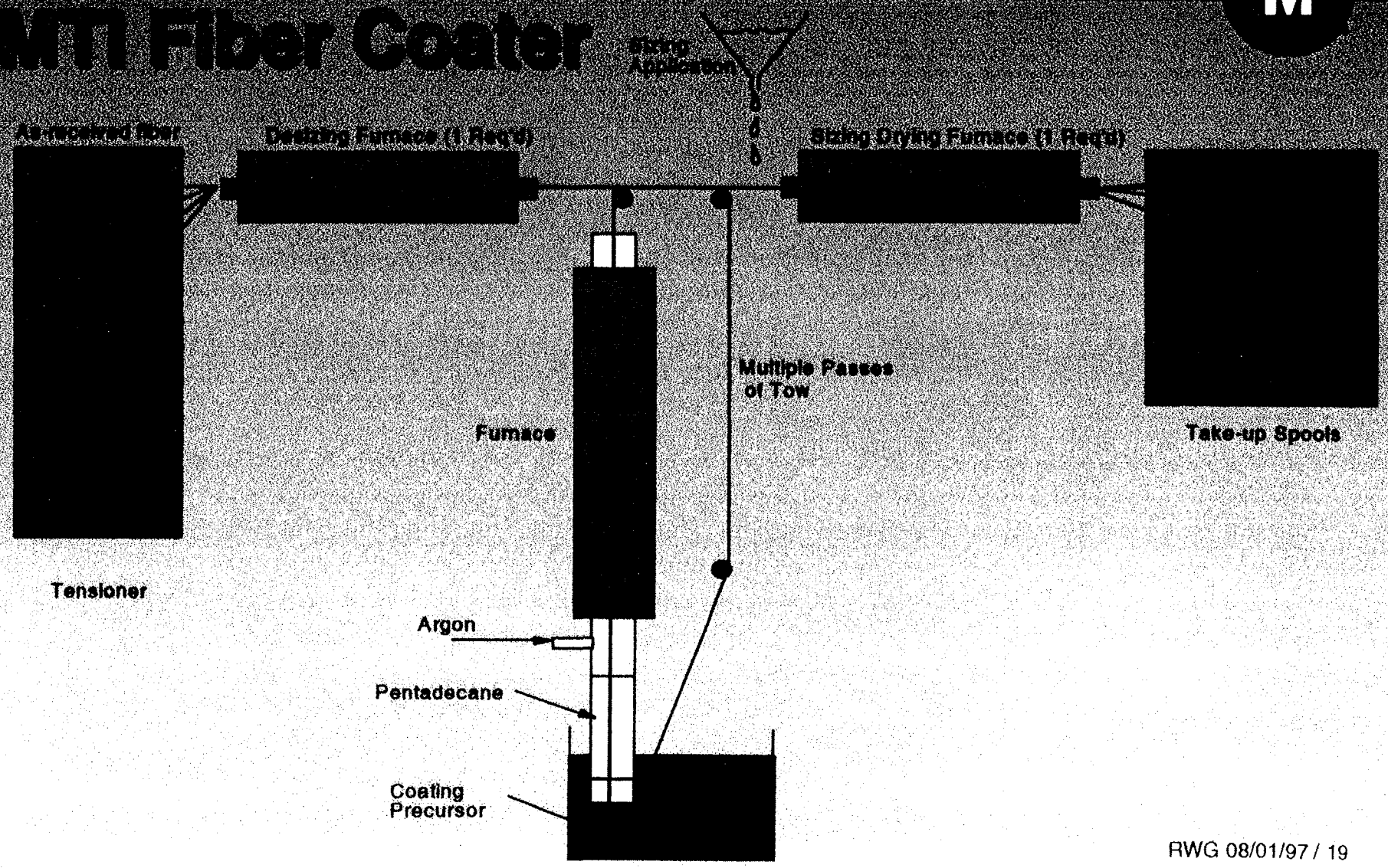


INITIALS DA/T/ES / 5

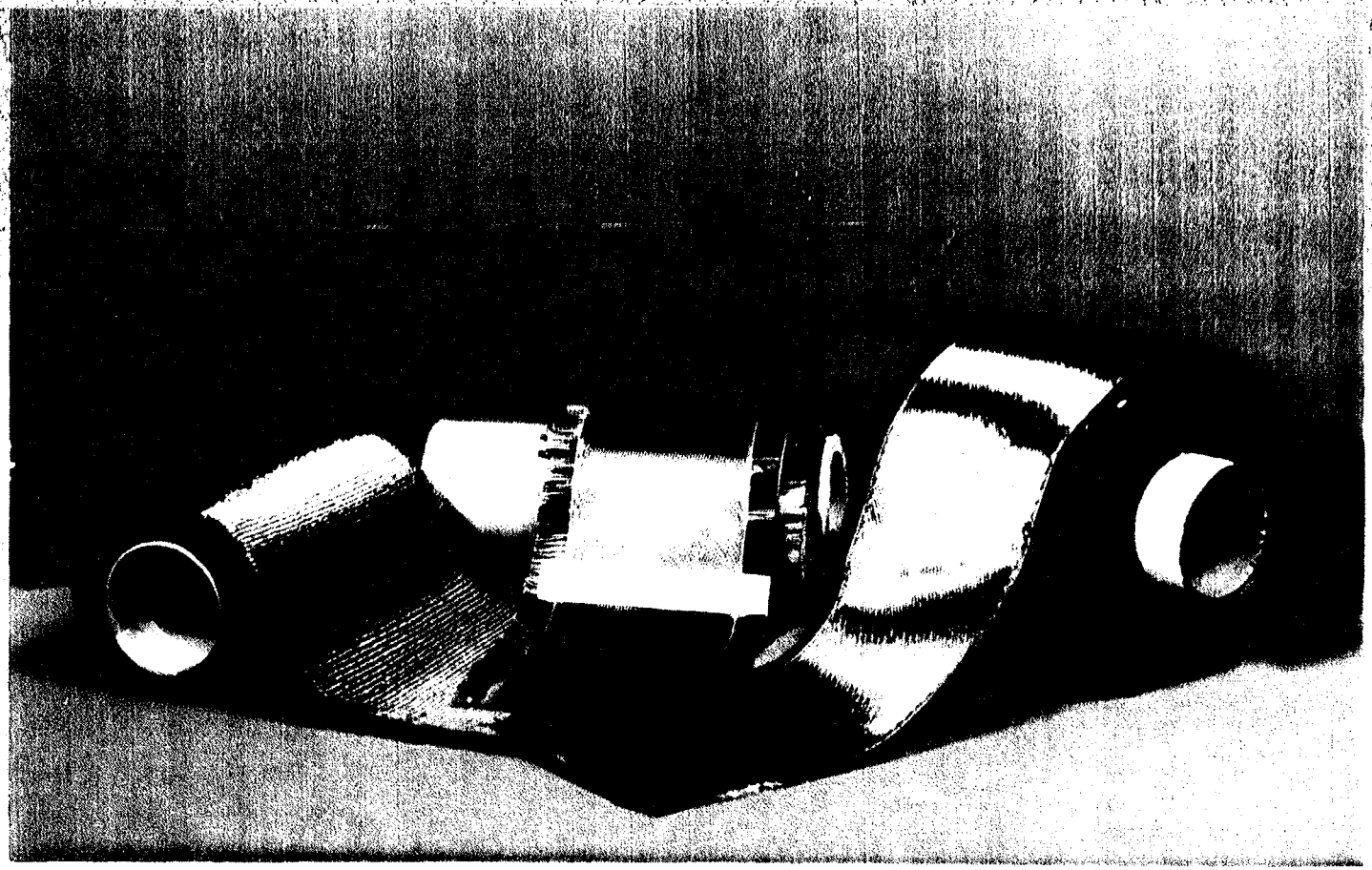
Filt./Sed. Tooling Demonstrated for Filament Wound Tubes



MTI Fiber Coater

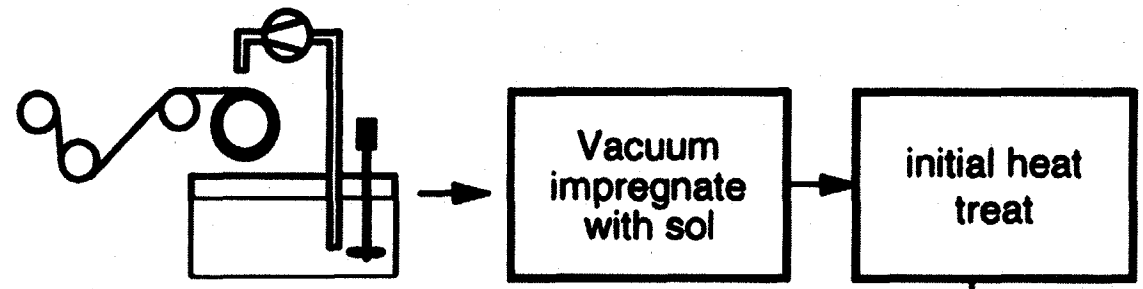


Nextel 610 and 720 Fabrics Woven with Carbon-Coated Fiber

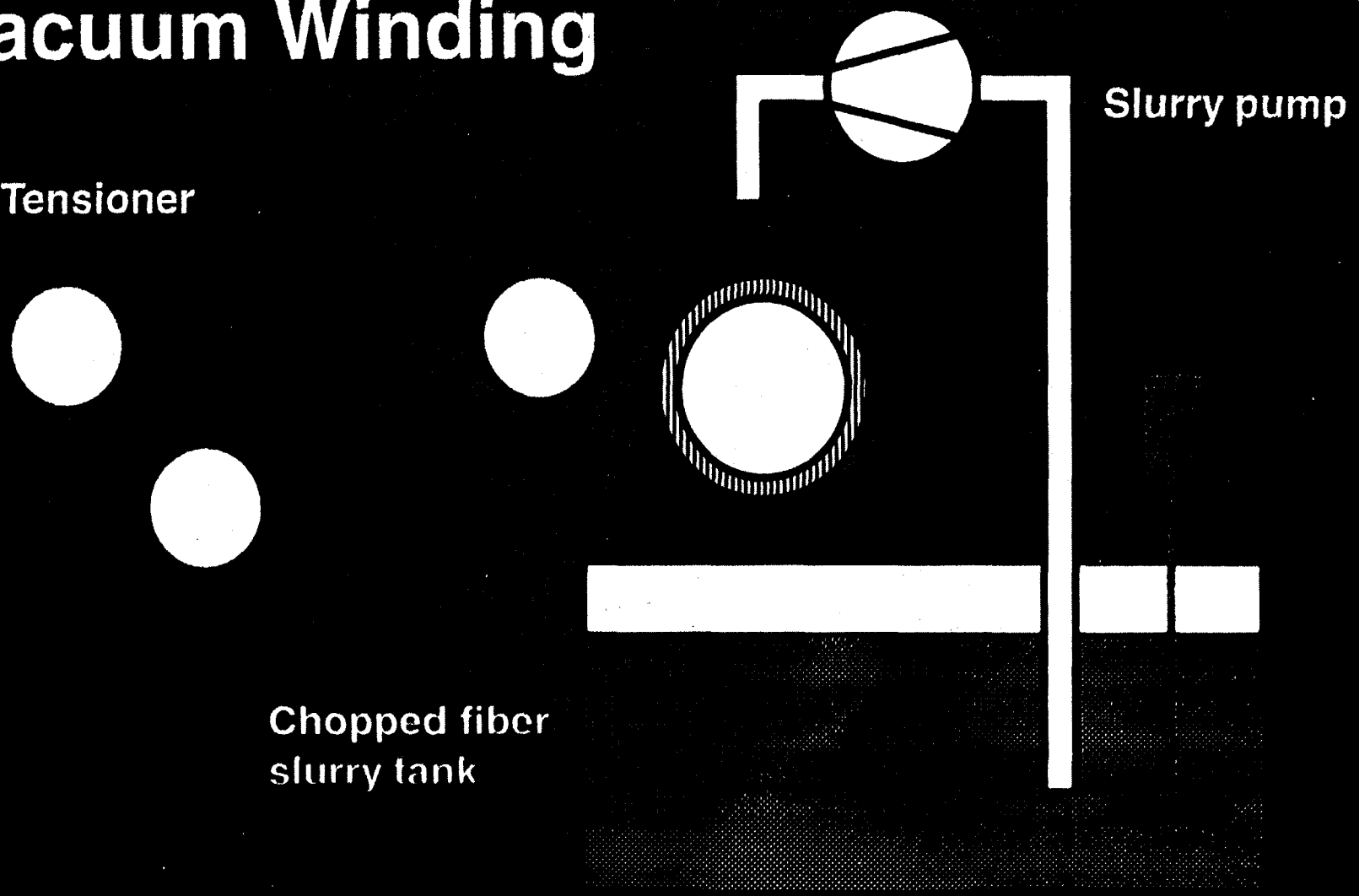


Filter Element Fabrication

Process Flow Diagram



Vacuum Winding

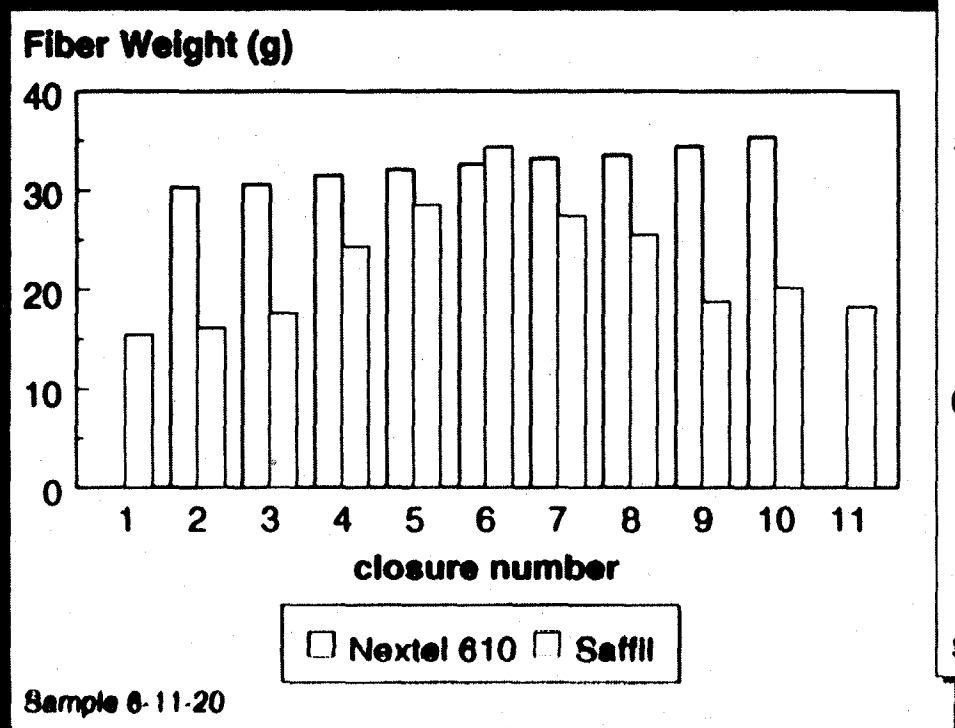


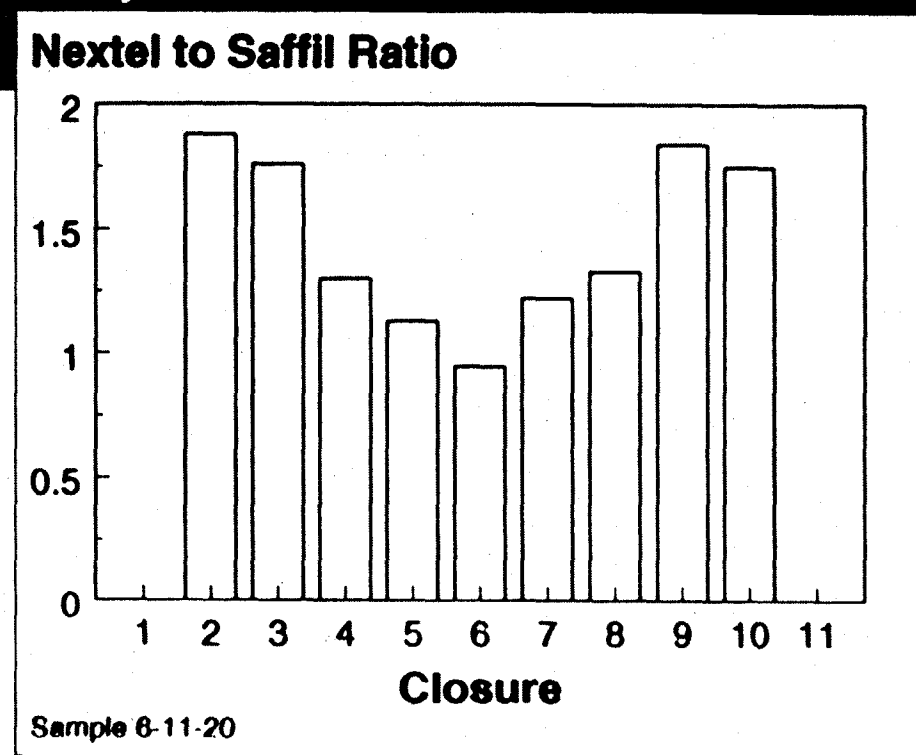
100.0 µ
15-1 0.0 19 084 671

Vacuum Winding

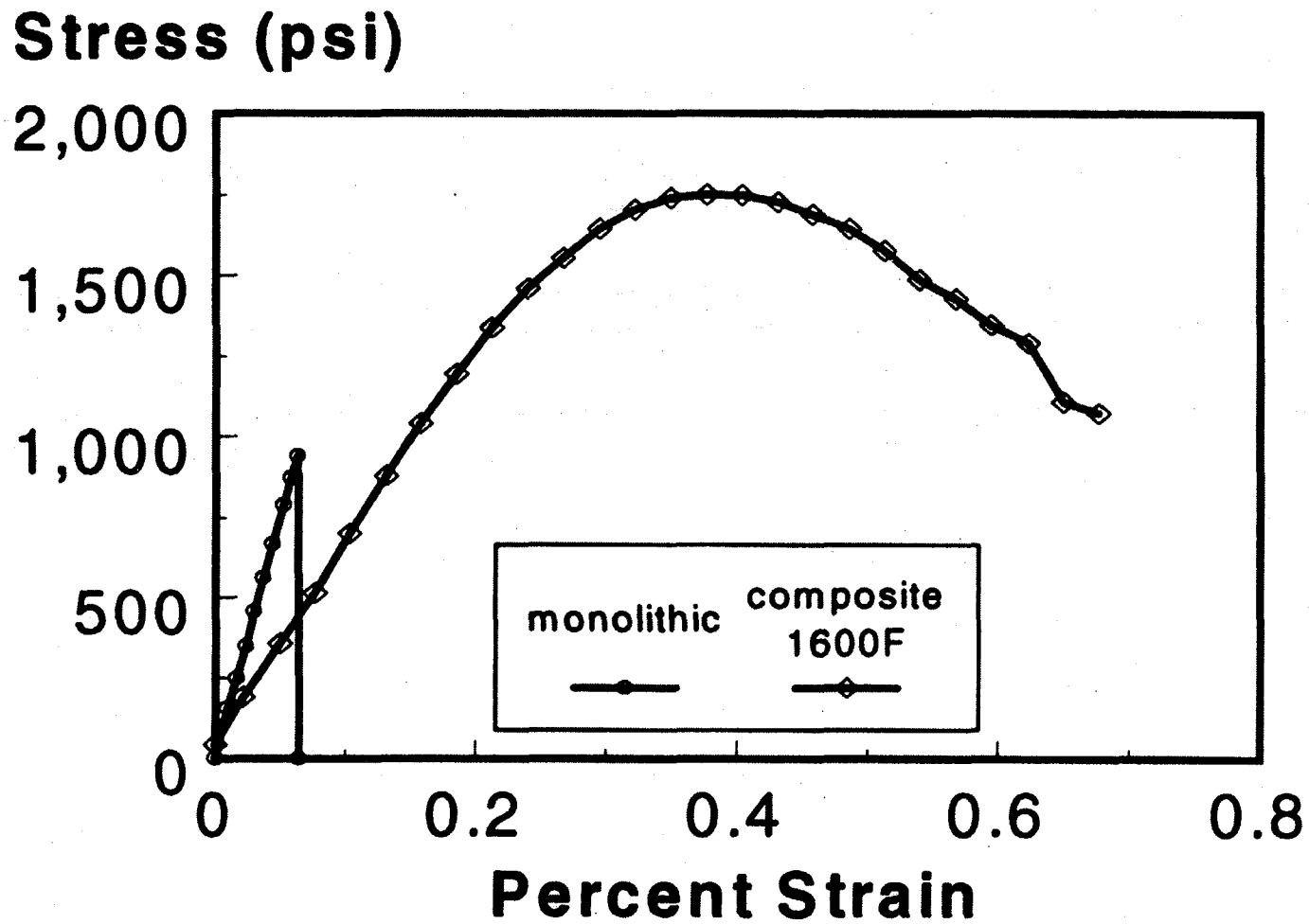
Controlling Fiber Distribution

Preliminary Data



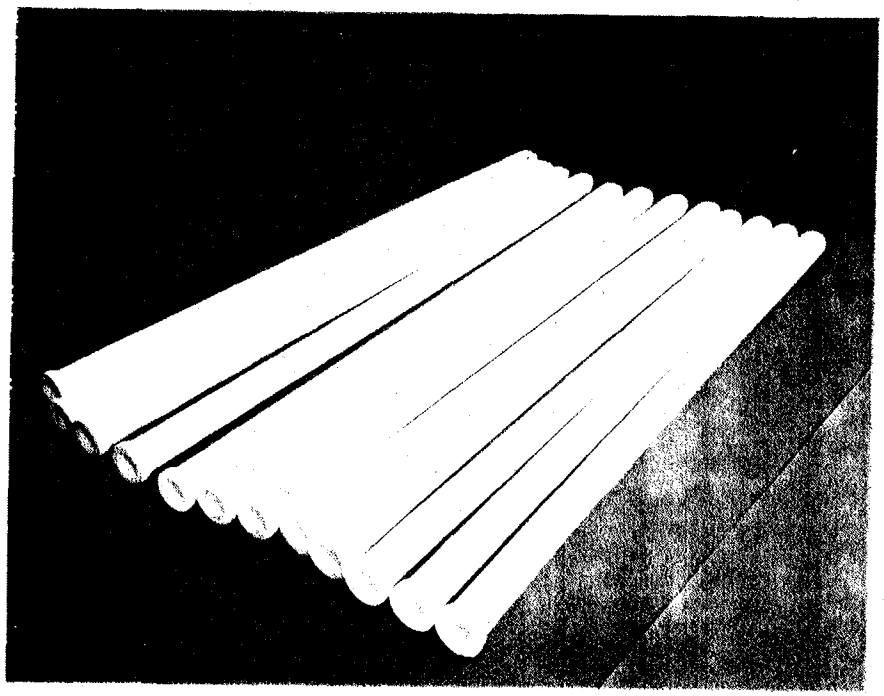


Advanced Hot Gas Filter Mechanical Properties



Fabrication of Full Scale Hot Gas Filters is Underway

12 filters delivered to Karhula, Finland for PFBC test



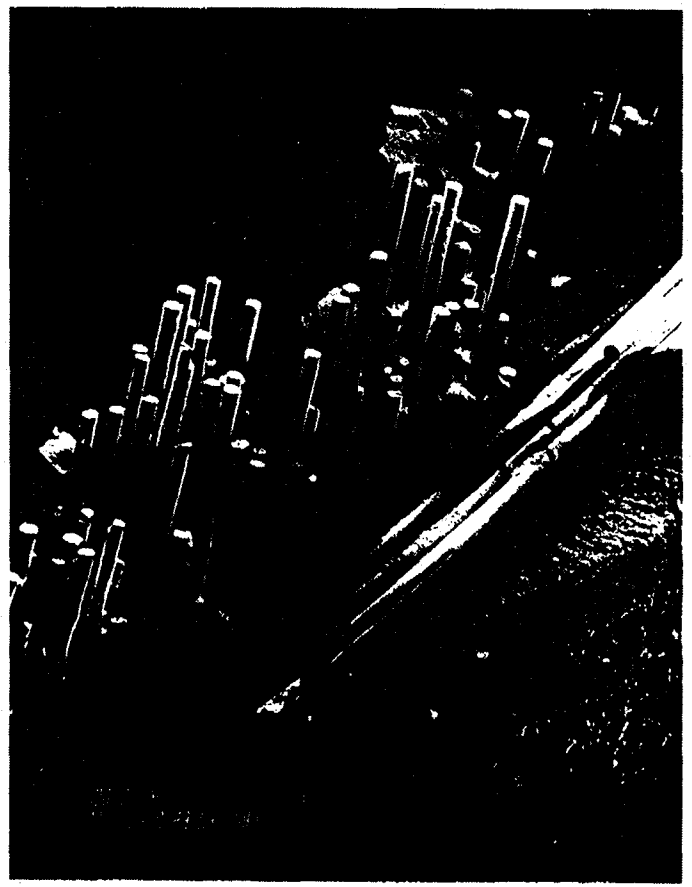
Current Field Testing

- Foster Wheeler 10 MW_{th} PFBC pilot scale facility, Karhula, Finland
  - Westinghouse Advanced Particle Filter System
    - 128 candle filters in 3 tiers
  - 6 MTI filter elements
  - Total run time to date: 67 hours
  - Planned total: 500 hours

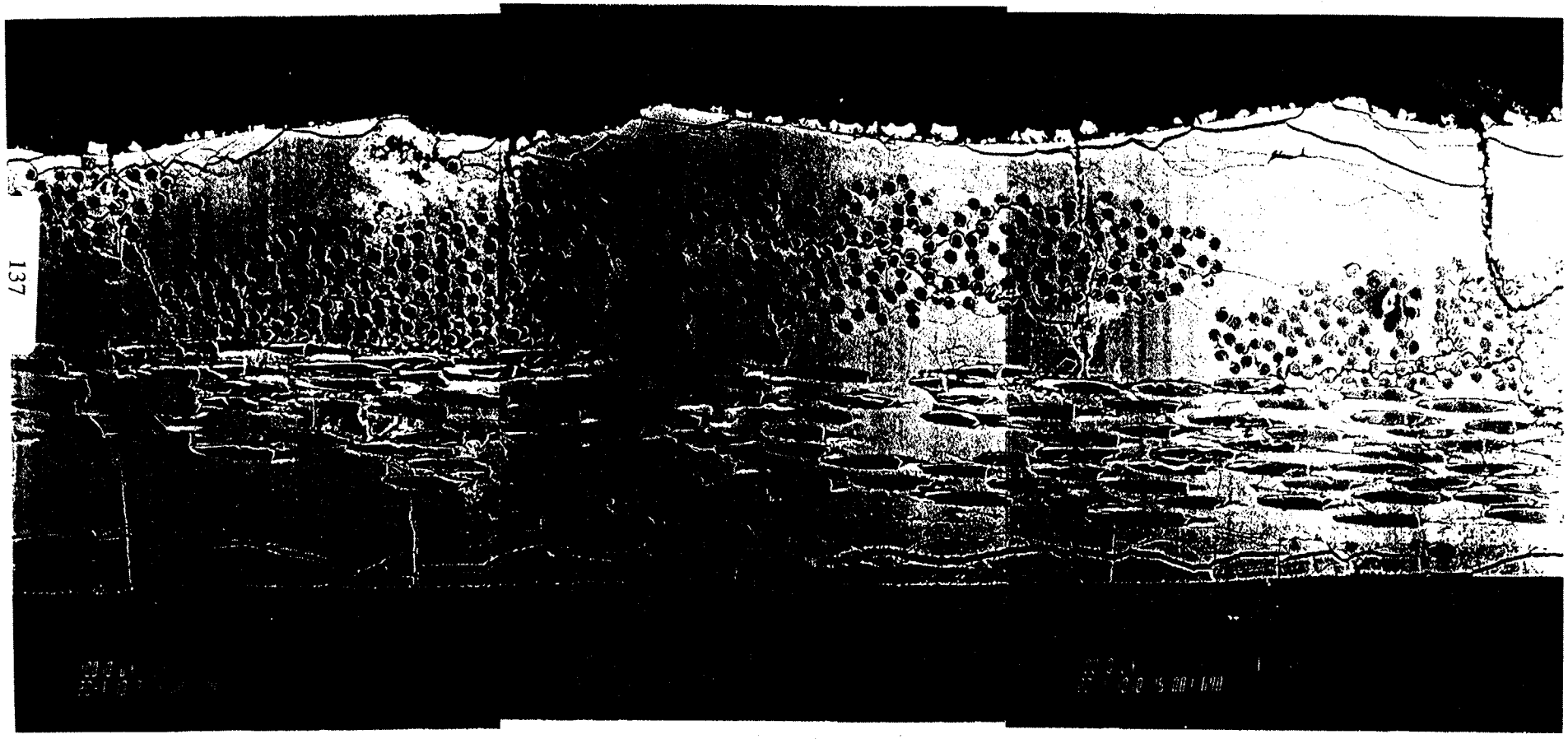
Future Field Testing

- Power Systems Development Facility, Wilsonville, AL
  - 4 MTI filters from the Karhula test
- Westinghouse High Temperature High Pressure Facility
  - 3 MTI filters from PSDF
- Successful completion of above will lead to bid for 3000 filter elements for Lakeland, FL PCFB facility
- Initiate fabrication and testing of Asahi Glass tube filter

Fracture Surface of Nextel 610/Alumina-Yttria CFCC



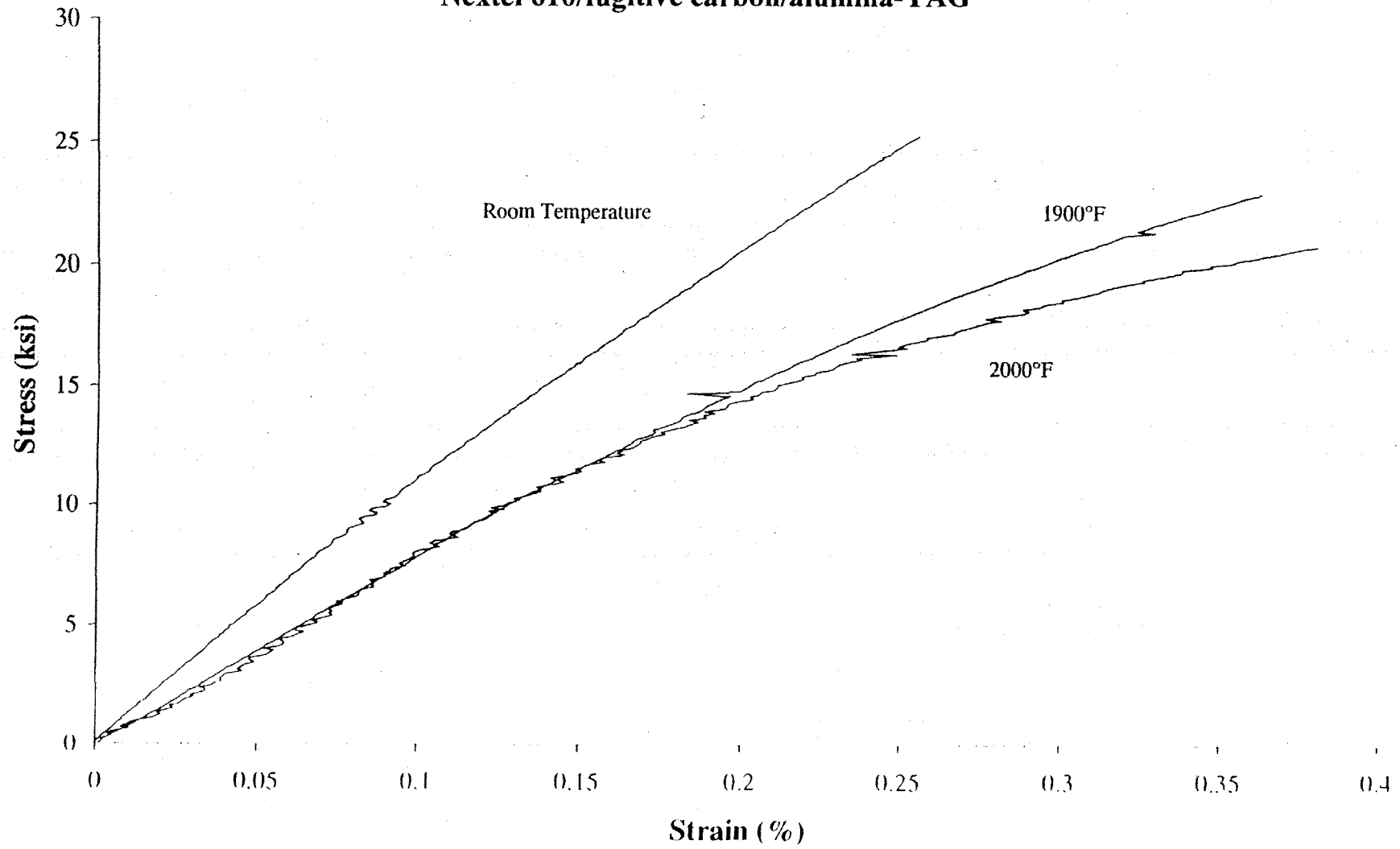
Microstructure of Nextel 610/Alumina-Yttria CFCC

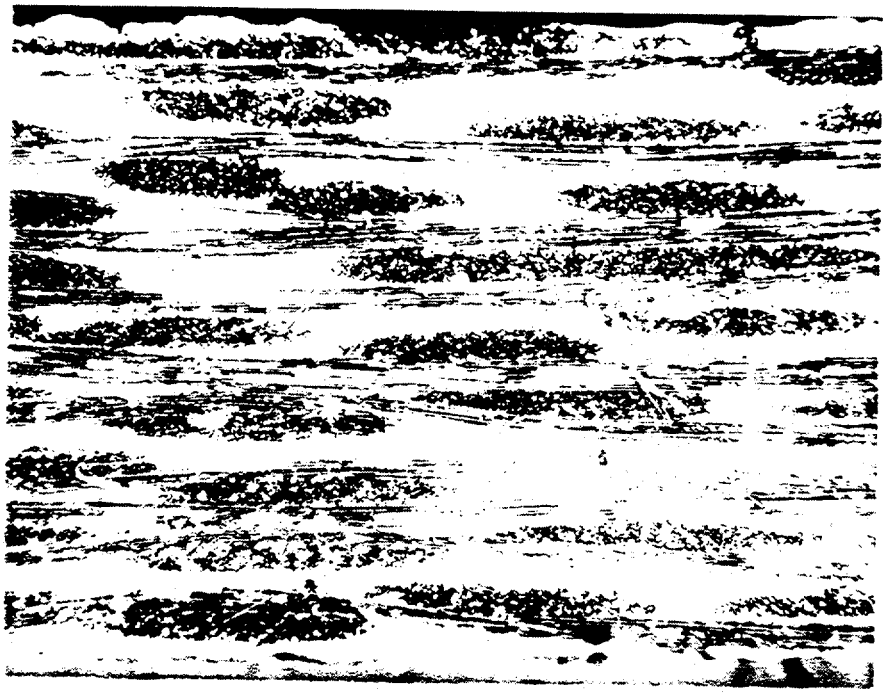


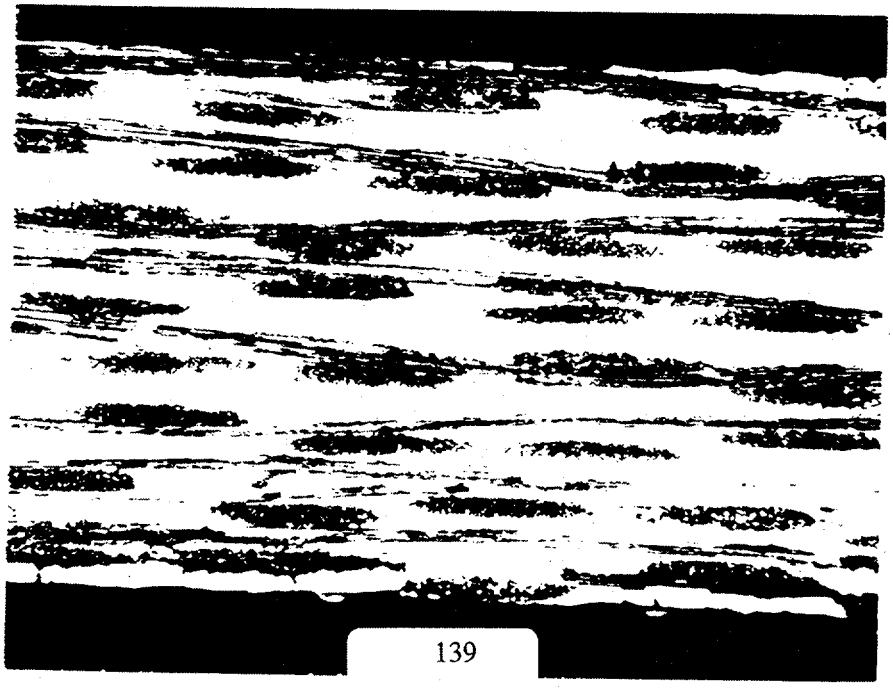
8-ply

Stress-Strain Curves at Various Temperatures

Nextel 610/fugitive carbon/alumina-YAG







Tungstates and Molybdates

Compound	Crystal Structure	Space Group	Melting Temperature (C)
$MgWO_4$	monoclinic	P2/a	1358
$CaWO_4$	tetragonal	$I4_1/a$	1580
$SrWO_4$	tetragonal	$I4_1/a$	1535
$BaWO_4$	tetragonal	$I4_1/a$	1475
$MgMoO_4$	monoclinic (low pressure) monoclinic (high pressure)	C2/m P2/c	1250
$CaMoO_4$	?	?	1445
$SrMoO_4$	tetragonal	$I4_1/a$	?
$BaMoO_4$	tetragonal	$I4_1/a$	?
$FeWO_4$	monoclinic	P2/c	?
$FeMoO_4$	monoclinic (high-temperature) monoclinic (low temperature)	P2/c C2/m	?
$MnWO_4$	monoclinic	P2/c	~1372
$MnMoO_4$	monoclinic	P2/c	~1130
$ZnWO_4$	monoclinic	P2/c	~1167
$ZnMoO_4$	monoclinic	P2/c	~1050
$PbWO_4$	tetragonal	$I4_1/a$	1123
$PbMoO_4$	tetragonal	$I4_1/a$	1063

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Tantalates and Niobates

Compound	Crystal Structure	Space Group	Melting Temperature (C)
(La, Ce, Pr)TaO_4	?	?	$LaTaO_4$, ~1930 $PrTaO_4$, ~1910
$Me^{3+}TaO_4$ (Y, Nd through La)	monoclinic (if crystallized below $T_{tetra-mono}$) monoclinic (after excursion to $T>T_{tetra-mono}$) tetragonal (high temperature)	P2/a I2 $I4_1/A$	$NdTaO_4$, ~1925
$Me^{3+}NbO_4$	monoclinic tetragonal (high temperature)	I2 (P2/a ??) $I4_1/A$	$LaNbO_4$, ~1620

$LaPO_4$ (monoclinic) - $P2_1/n$

YPO_4 (tetragonal) - $I4_4/amd$

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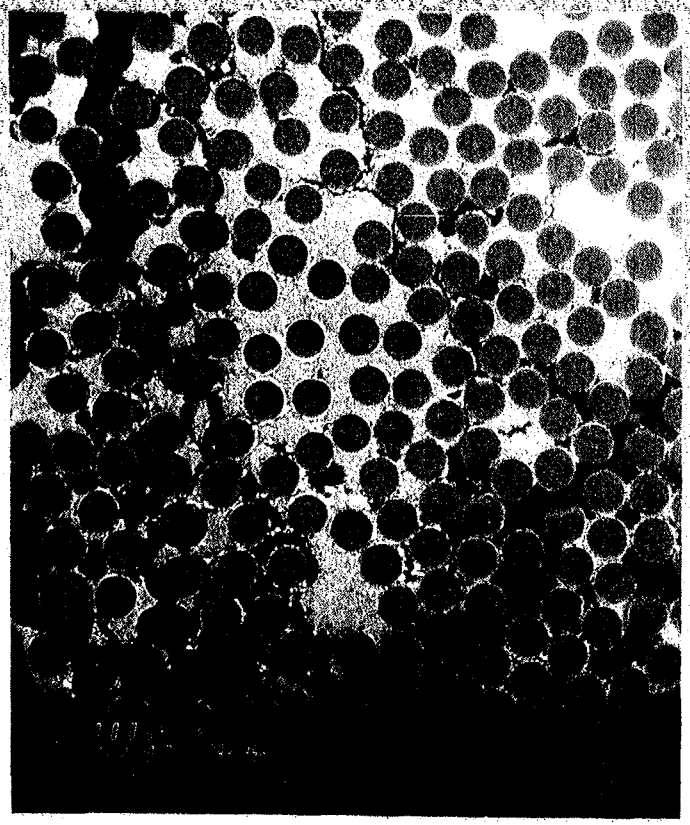
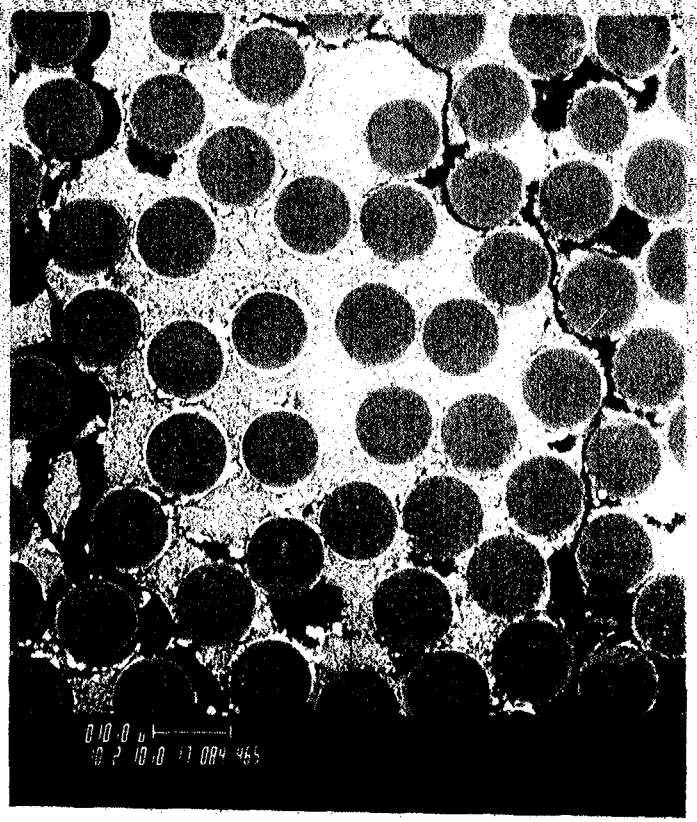
Interface Debonding Observed Near Composite Delamination





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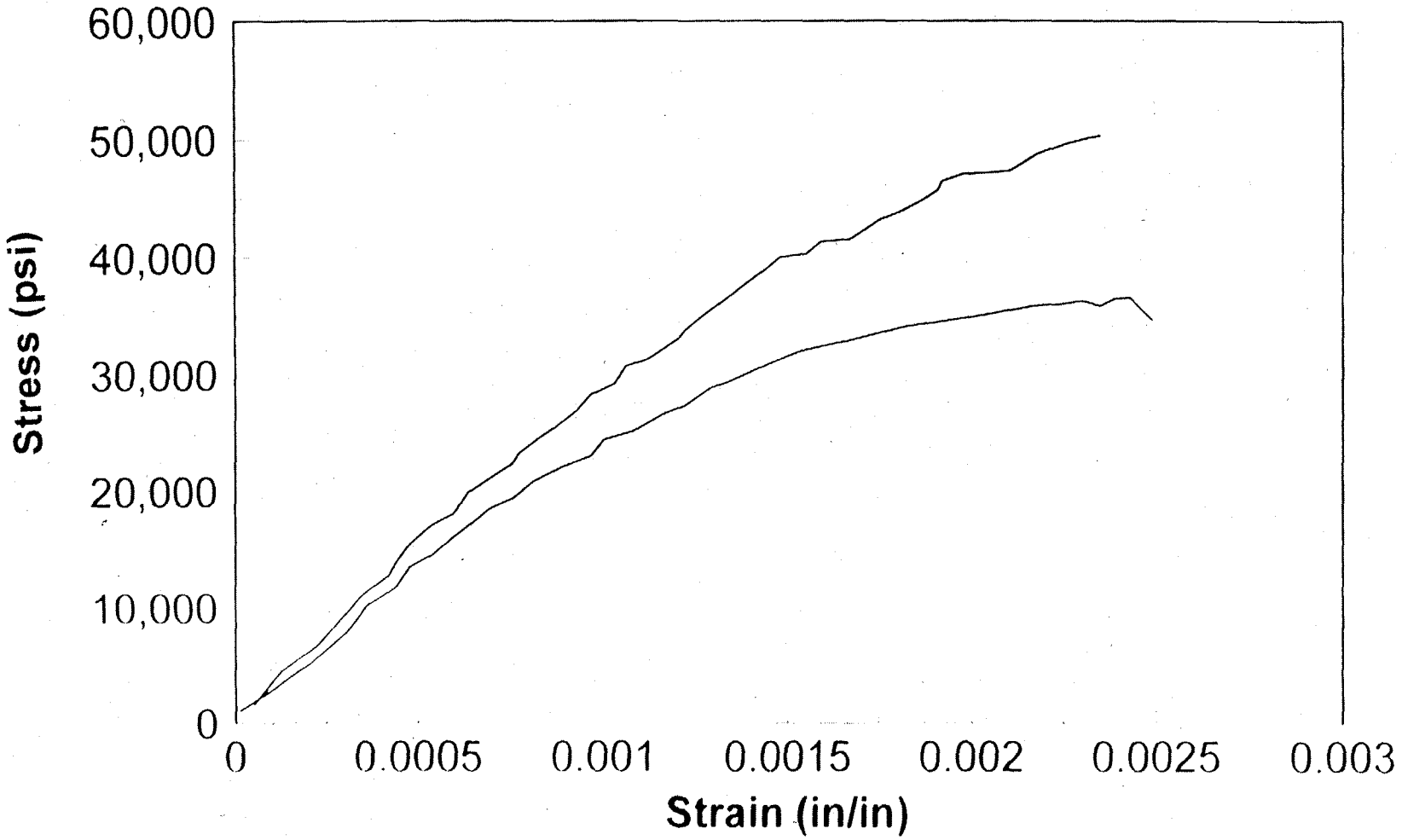
TEM of Nextel 610/Scheelite in Alumina-Scheelite Matrix

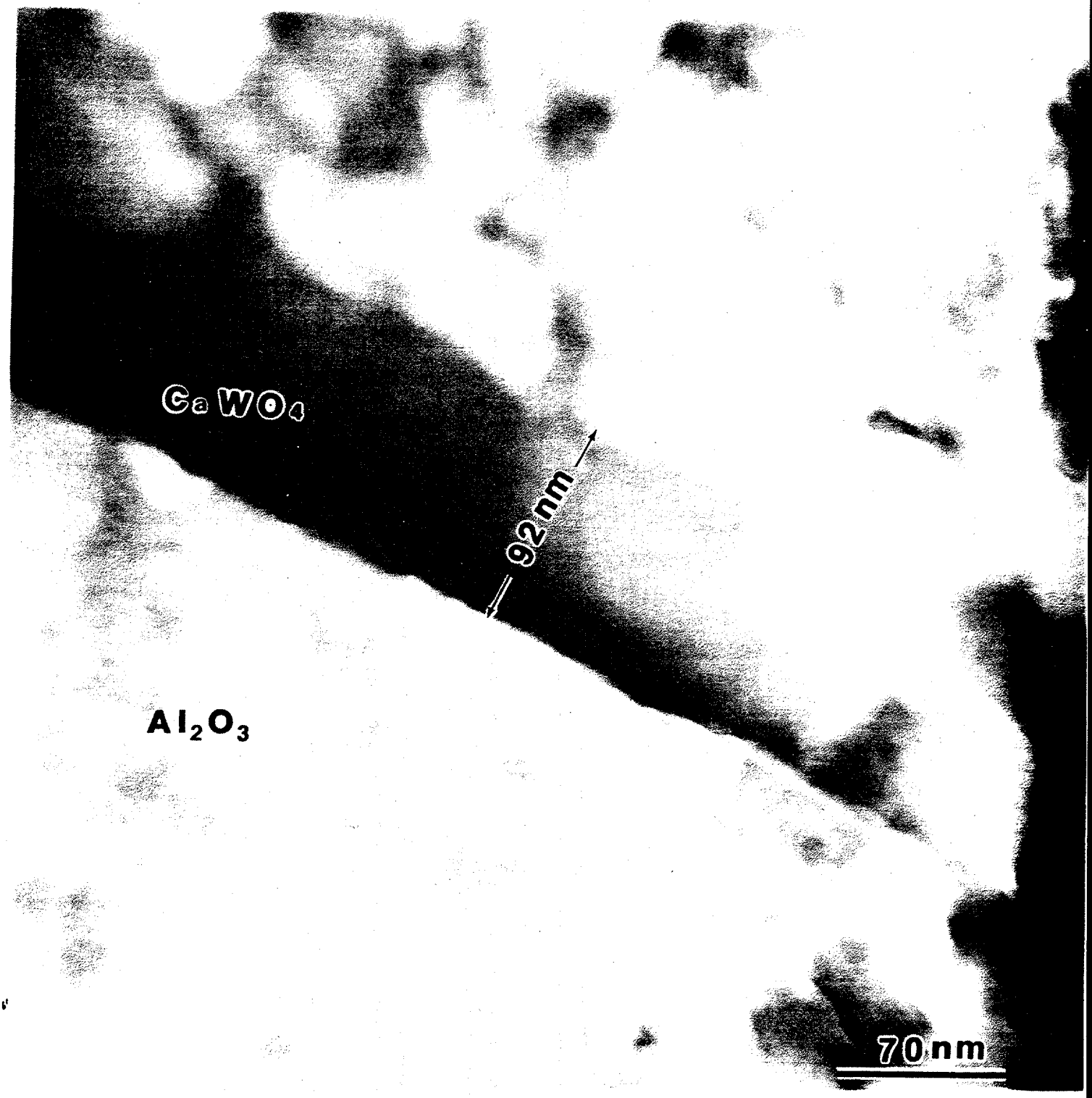
Unidirectional Nextel 610 CFCC with Scheelite Fiber Coating and Alumina-Scheelite Matrix

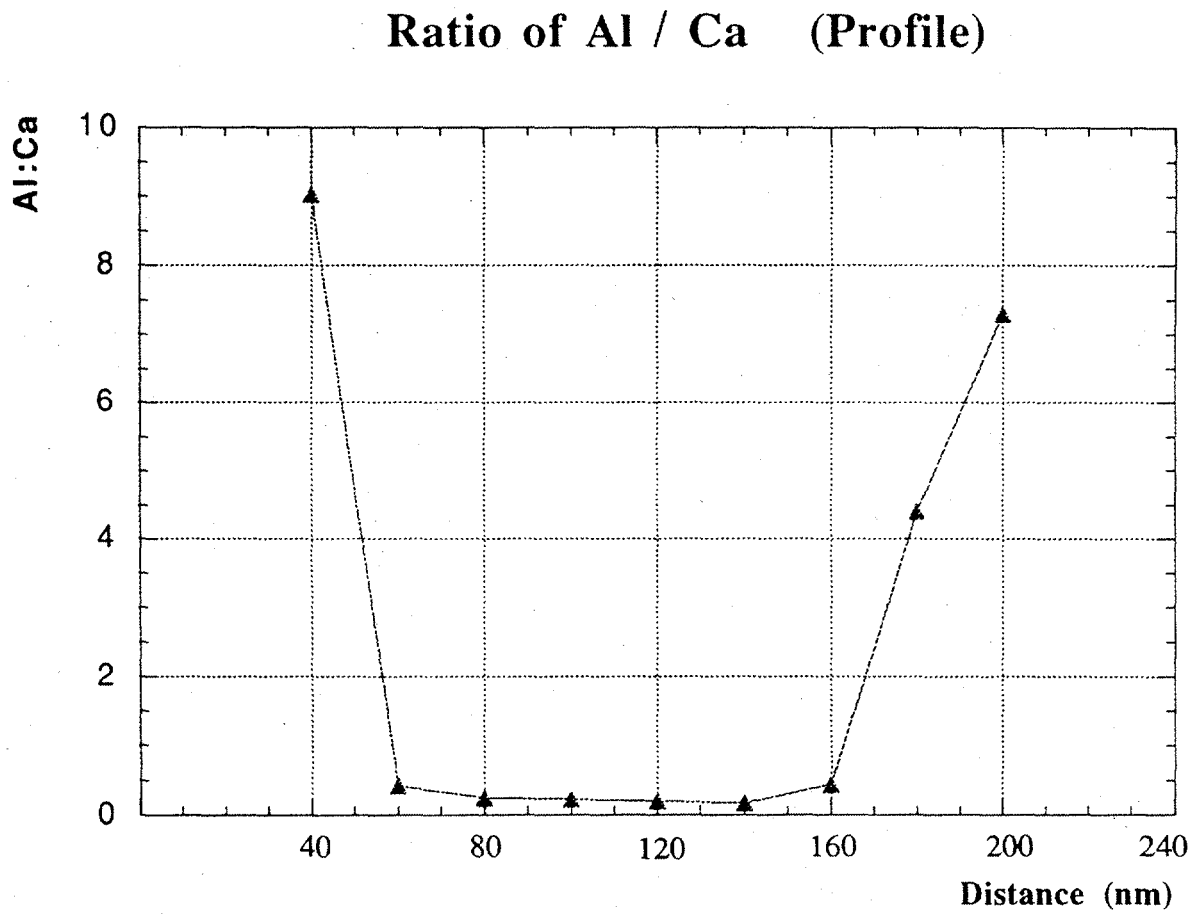
~20% residual porosity, room temperature tensile test

1355-69-CW1 & CW2 (850C)
Nextel 610, unidirectional
Scheelite Coating
Alumina/Scheelite matrix
20% porosity



100396


CaWO4
92 nm
Al2O3
70 nm


Ratio of Al / Ca (Profile)
Al:Ca
10
8
6
4
2
0
40
80
120
160
200
240
Distance (nm)

Composites Fabricated With Nextel 720

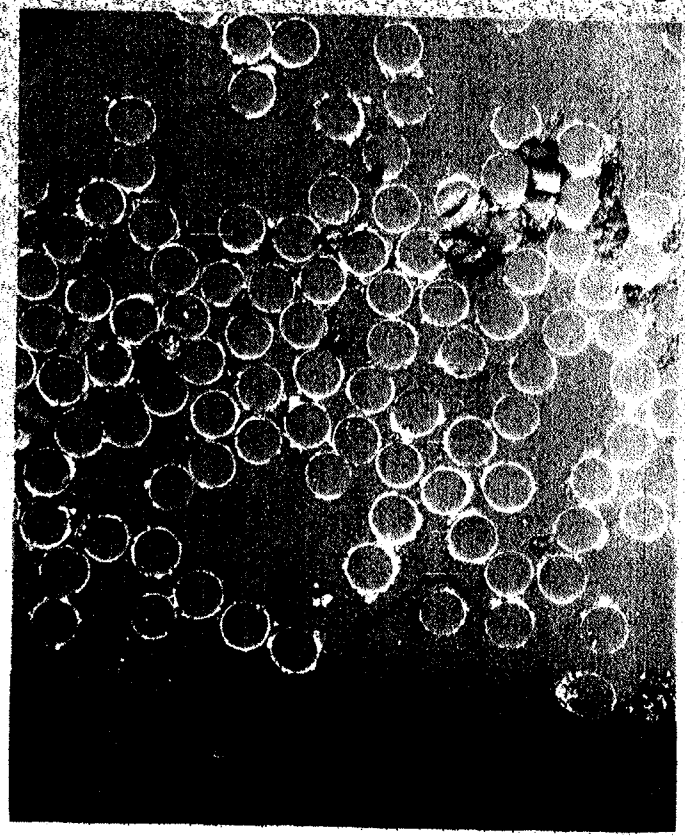
- Nextel 720/Scheelite in Alumina Matrix
  - 19.6% porosity
- Nextel 720/Scheelite in duplex Alumina-Scheelite matrix
  - 18.4% porosity

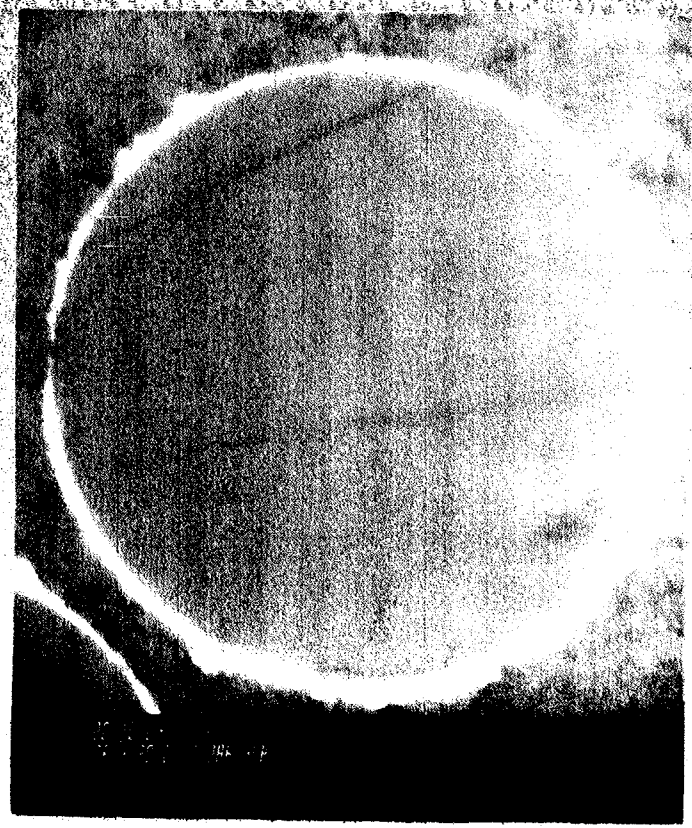
Nextel 720/Scheelite Composites Exhibit Low Strengths

- Alumina Matrix
  - fiber strength degradation from MTI alumina precursor
- Alumina-Scheelite matrix
  - decomposition of EDTA from scheelite precursor leaves char
  - scheelite coatings and matrix are reduced
  - likely fiber strength degradation from calcia reaction/diffusion

Nextel 720/$CaWO_4$/Alumina

- 19.6% residual porosity





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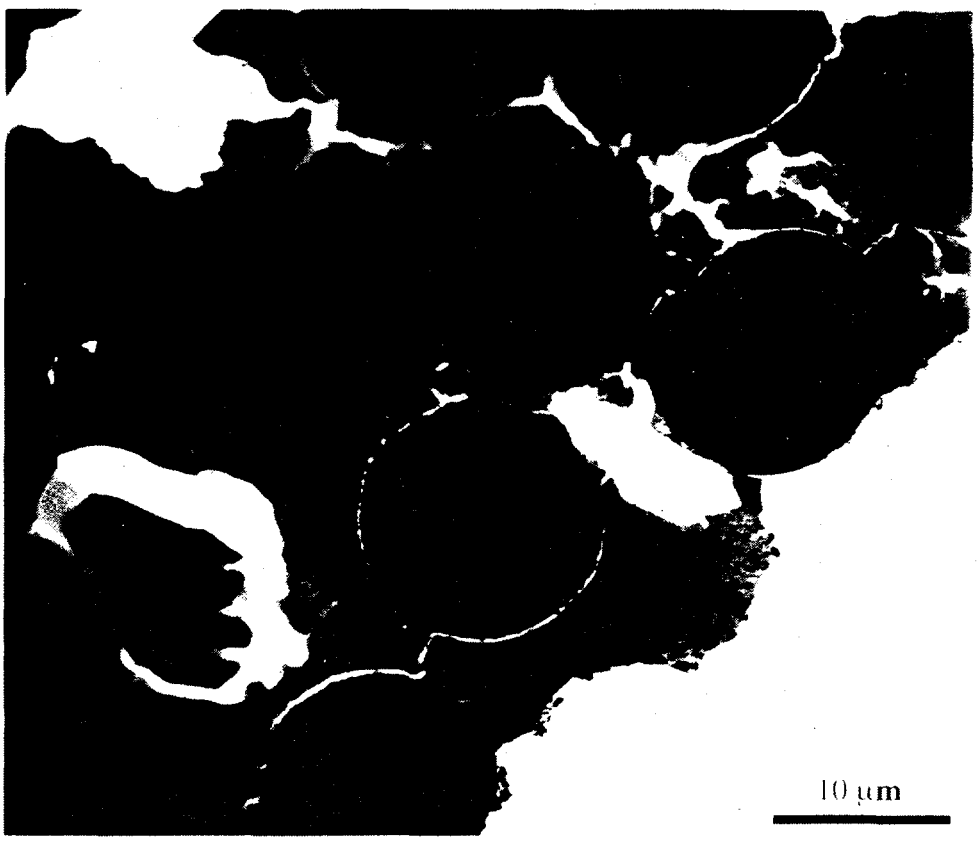
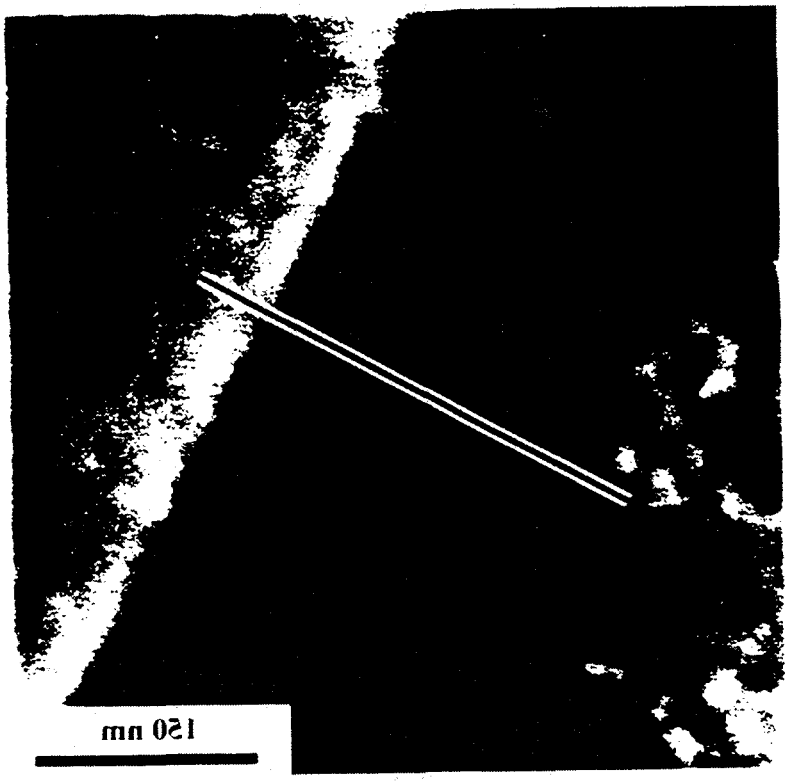
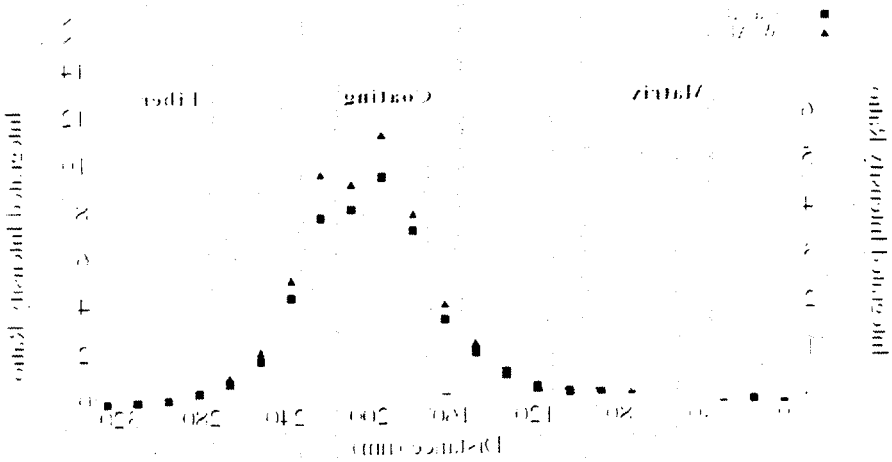
TEM Micrographs of N720/$CaWO_4$/Al_2O_3 Composite



EDS Profile
N720/$CaWO_4$/Al_2O_3





Debonding Along Nextel 720 - Scheelite Interfaces

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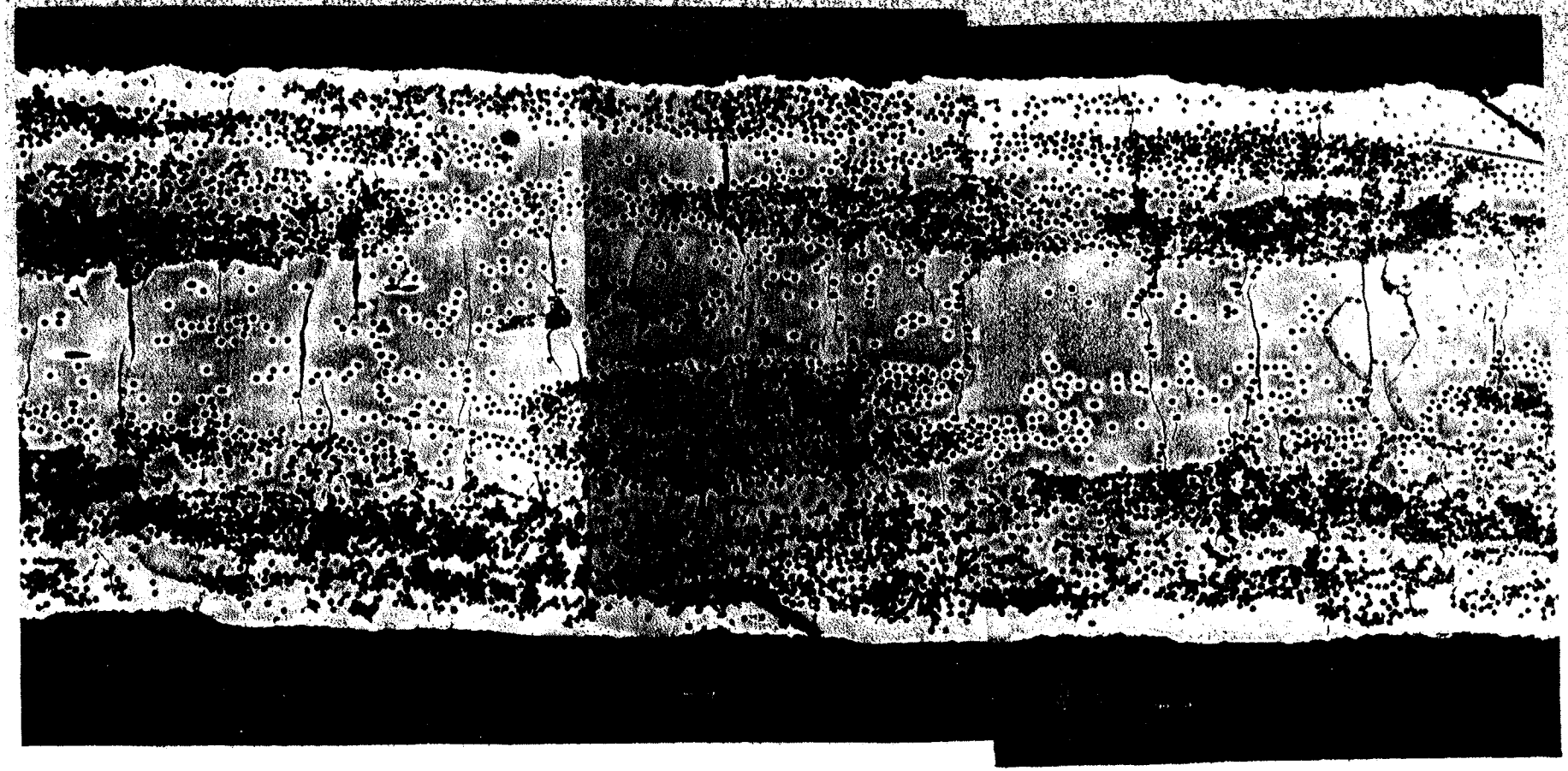
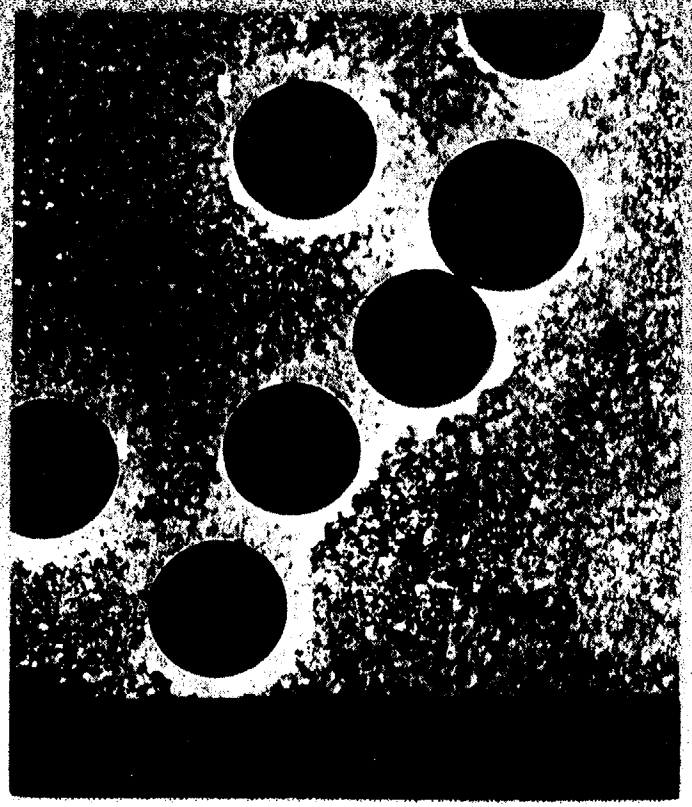
Localized Reduction of Scheelite

- effects appear worse in center of composite



Regions in Center of Composite



Scheelite Coatings on Nextel 720

- Heated at 1100°C for 1 hour

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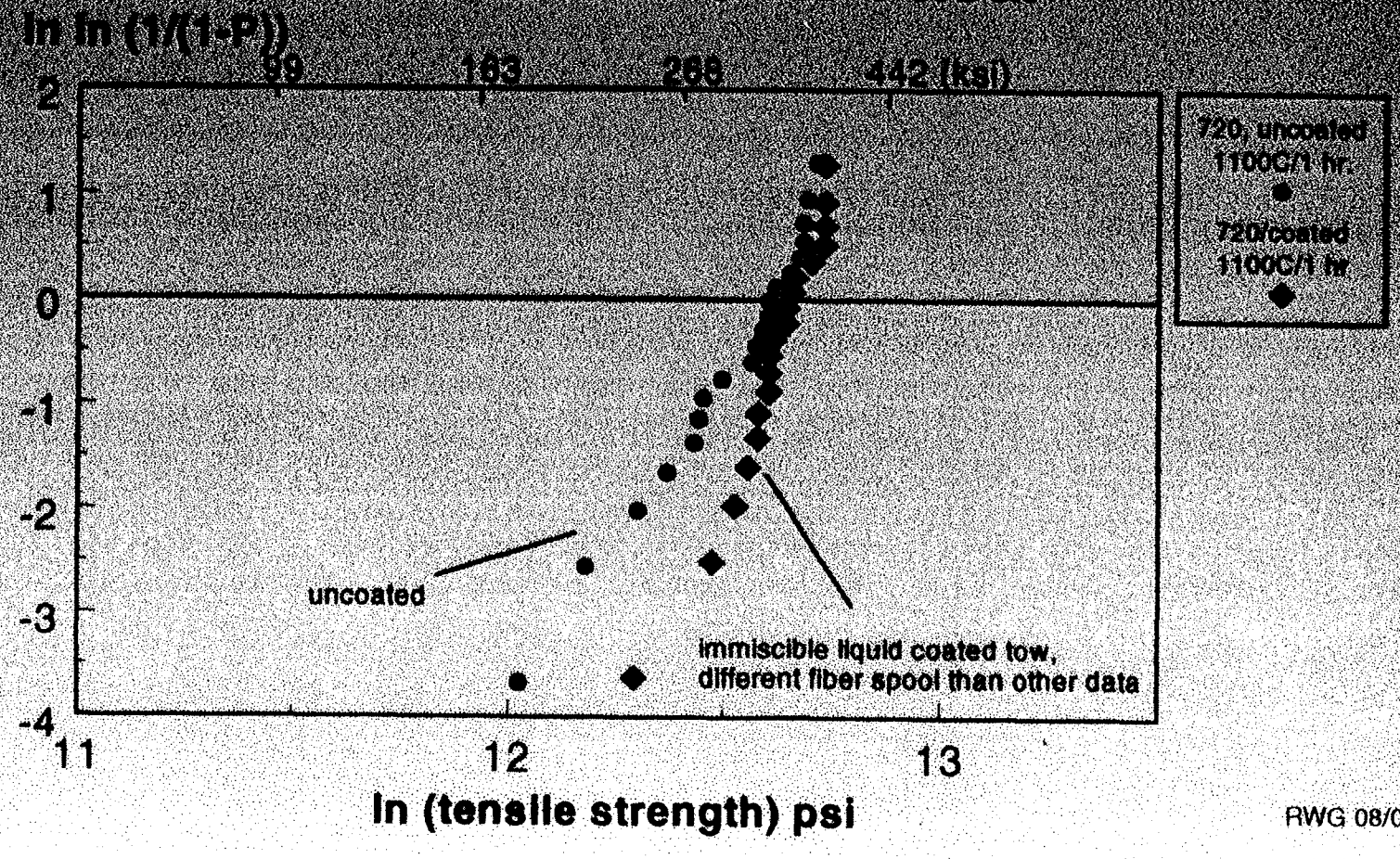
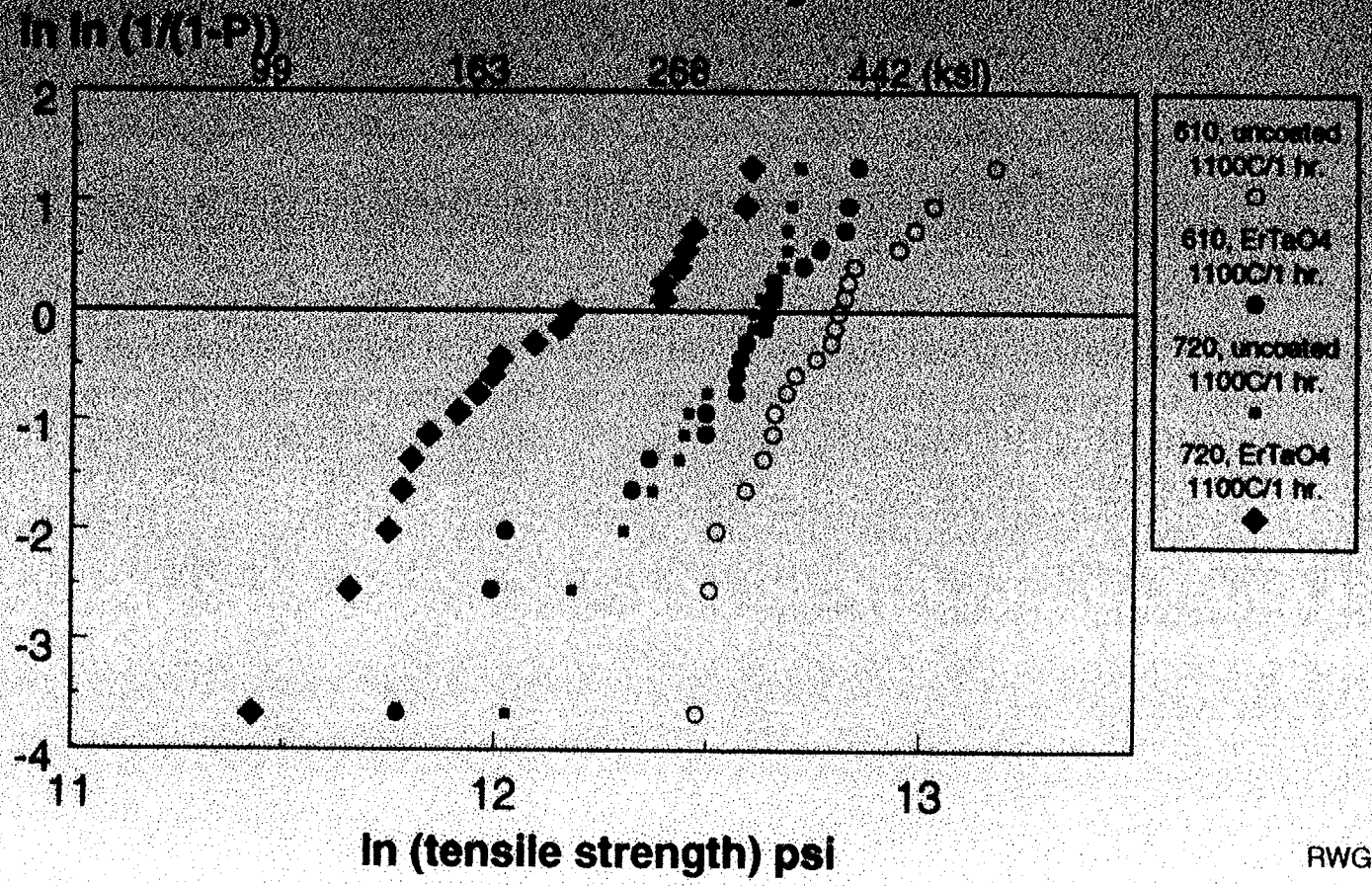
Nextel 720 is Stable with CaWO$_4$

- fibers heat treated at 1100°C for 1 hour

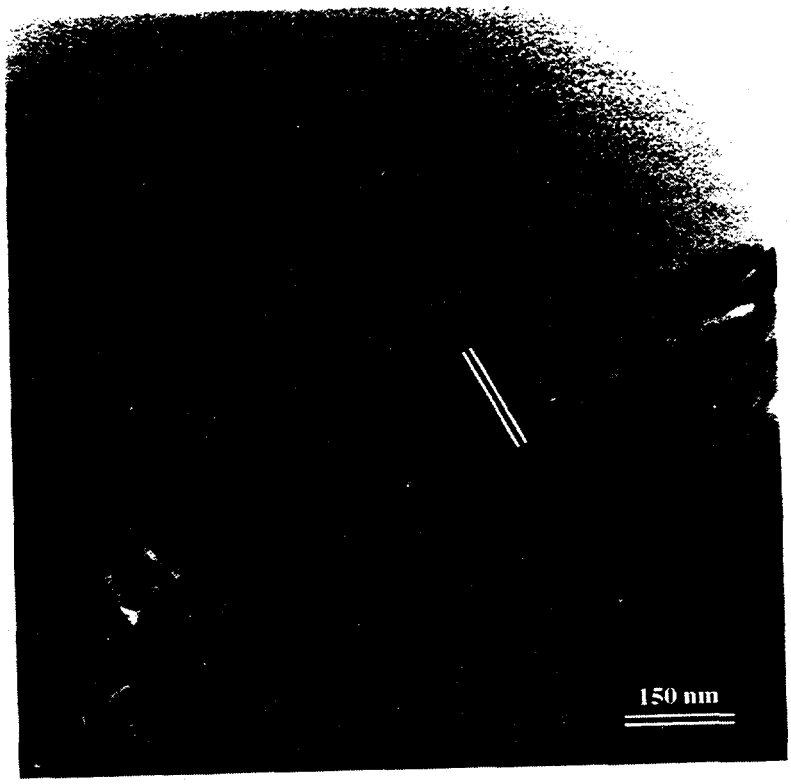


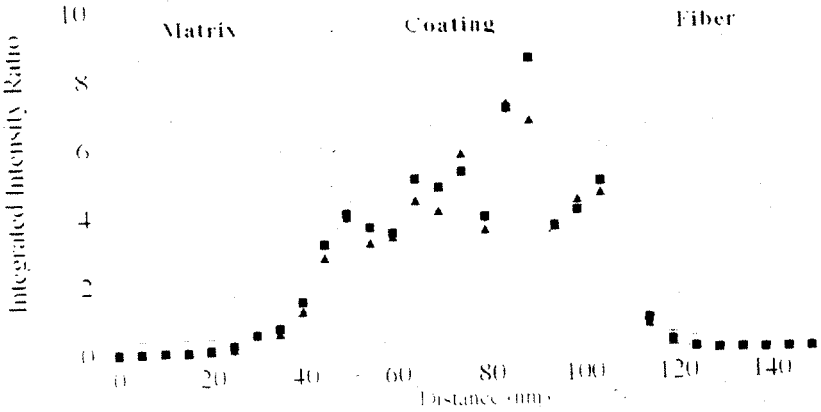
$ErTaO_4$ Degrades Fiber Strength

- may be some off-stoichiometry



EDS Profile
N610/$ErTaO_4$/Al_2O_3

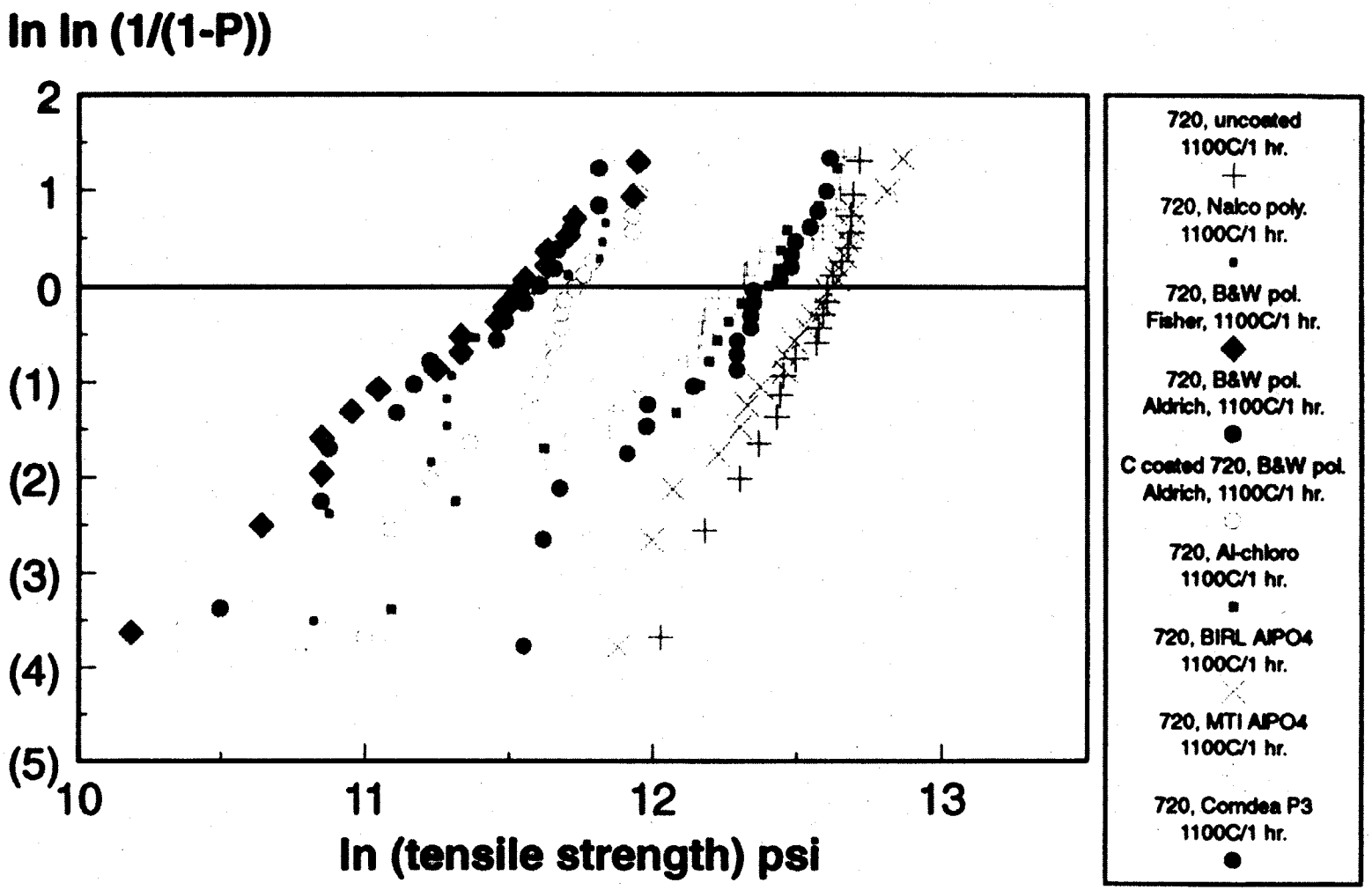




Matrix Processing Issues

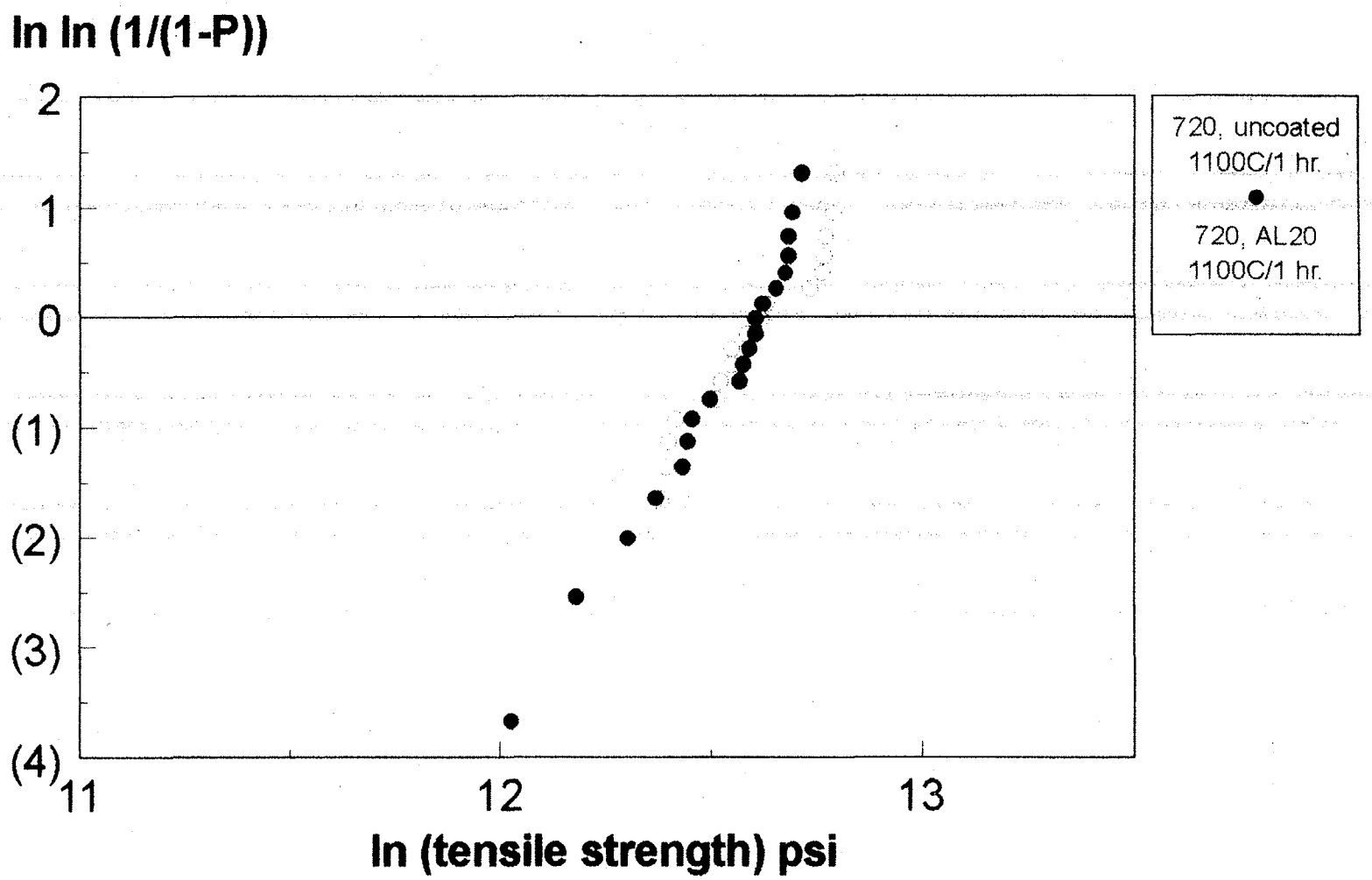
McDERMOTT M

- Alumina polymers degrade fiber strengths
  - MTI alumina polymer (derived from Al-nitrate)
  - Nalco polymer (derived from Al-chlorohydrate)
- Alumina salt solutions degrade fiber strengths
  - Al-chlorohydrate
  - boehmite sol with Al-acetate
- Boehmite sols do not degrade strength but poor infiltrant (skinning)
- Investigating $AlPO_4$
  - low density improves infiltration efficiency
  - polymorphic transformations



12 micron diameter fiber
Nalco polym. is exp. lot TX8918
fiber lot A0177-0274

720matr3



12 micron diameter fiber

720matr

McDERMOTT M

Status of Fiber Coatings

- Scheelite appears stable with Nextel 720 and 610 and debonds readily
- Strong composits still not demonstrated on very dense CFCC with Nextel 720
  - will work on more continuous/uniform coatings to protect fibers
  - modified matrix precursors to not degrade fiber strength
  - better match of matrix-to-fiber CTE
- Need more stable scheelite precursor solution
  - will work with BIRL/Northwestern Univ.
- YAG/fugitive-Carbon coatings on Nextel 610 to extend life of classified CFCCs

Solar Turbine Results With Early MTI CFCC Materials

McDERMOTT M

- Fabrication capabilities demonstrated
- CFCCs with Nextel 610 have inadequate temperature capability for 1200°C use liners
- Axial cracks during flame out conditions while under 10 hour rig test
- Improved fiber-matrix interface coatings needed to promote crack deflection and damage tolerance
- Need CFCC system utilizing Nextel 720 fibers

Turbine Activities

McDERMOTT M

Will supply 8" diameter by 8" long subscale liner to Solar Turbine for combustor rig testing

- Nextel 720/scheelite/alumina-mullite CFCC
- awaiting results from latest matrix improvements
  - 1200°C application

Currently fabricating test specimens for European Gas Turbine

- Nextel 610/fugitive/alumina-YAG
  - 900°C application

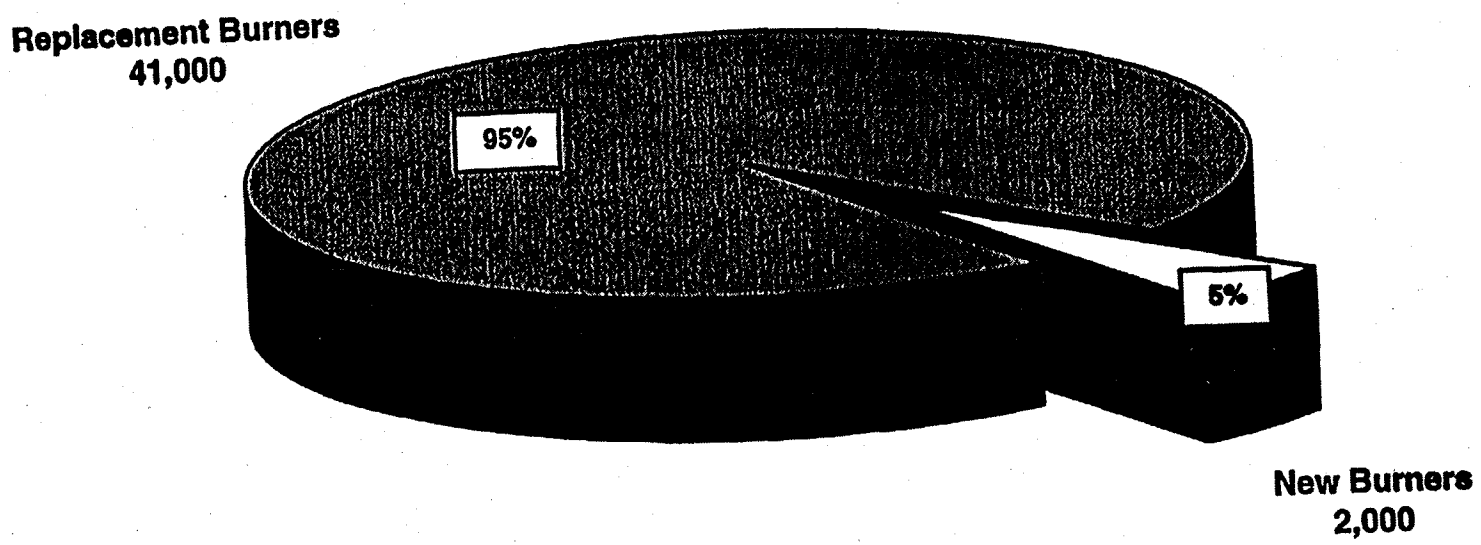
Porous Ceramic Fiber Elements for Gas-Fired Burners

Potential Applications

- Paint drying/curing
- Paper drying/profiling/coating
- Textile drying
- Thermoforming (?)
- Other industrial processes (?)

As evidenced by the high percentage of replacement units sold per year, the textiles industry is a mature IR market.

Annual Sales of IR Burners
- 1994 -

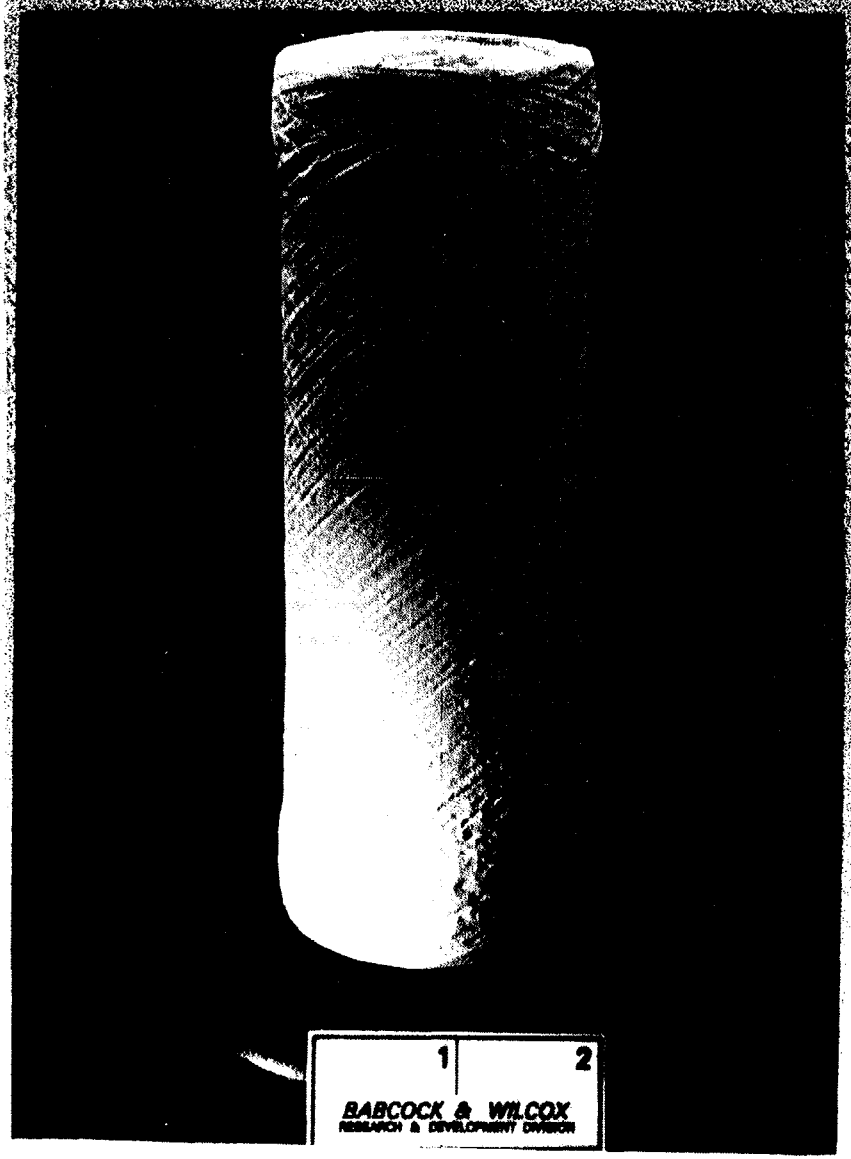


Total Annual Sales = 43,000

Rare Earth Oxide Broadband Emitters - Potential Advantages

- Improved heat transfer and product throughput
  - Radiation primarily in 1-3 micron wavelength range
- Improved burner efficiency
  - 10-20% reduction in fuel consumption
- Reduced product degradation and fire hazard
  - Lower product temperature
  - Rapid cooling of burner on shutdown

Mixed Rare Earth Oxide Emitter for High-Performance IR Burners



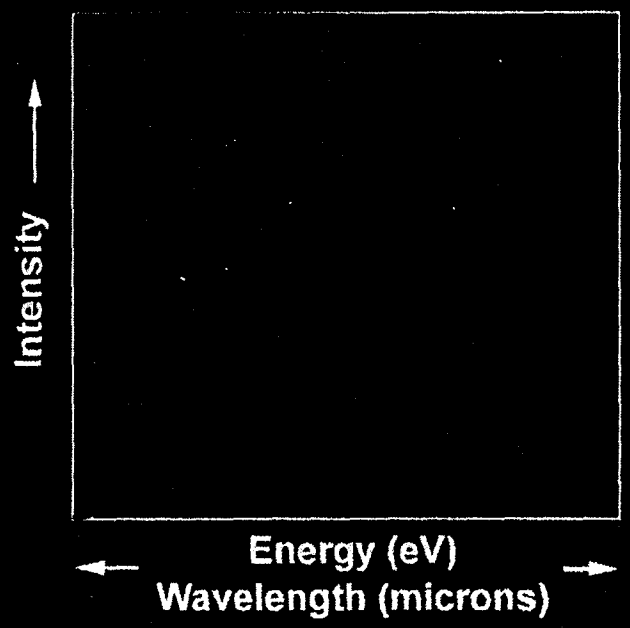
Erbia containing IR burner

TPV Power Generation

- Direct conversion of radiant energy from incandescent emitter to electricity using photovoltaic cells
- Analog to solar energy conversion
- Wide range of emitter heat sources
- Promising candidate for compact power sources and cogenerators

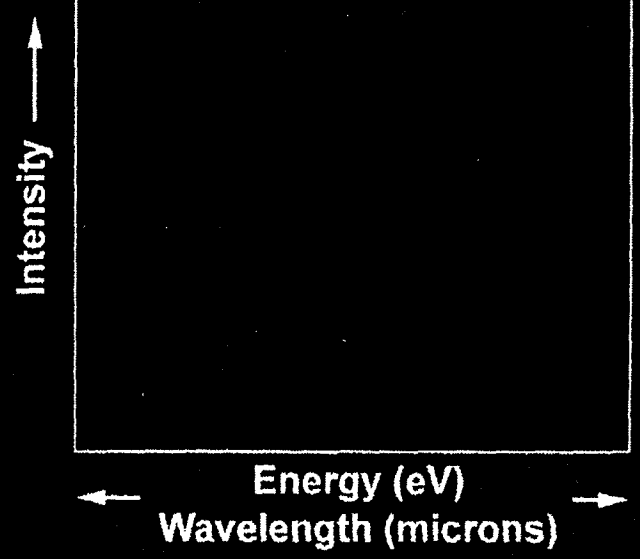
Classification of TPV Systems

Blackbody emitter



- Continuous spectrum
- Many emitter materials
- Significant "out-of-band" energy

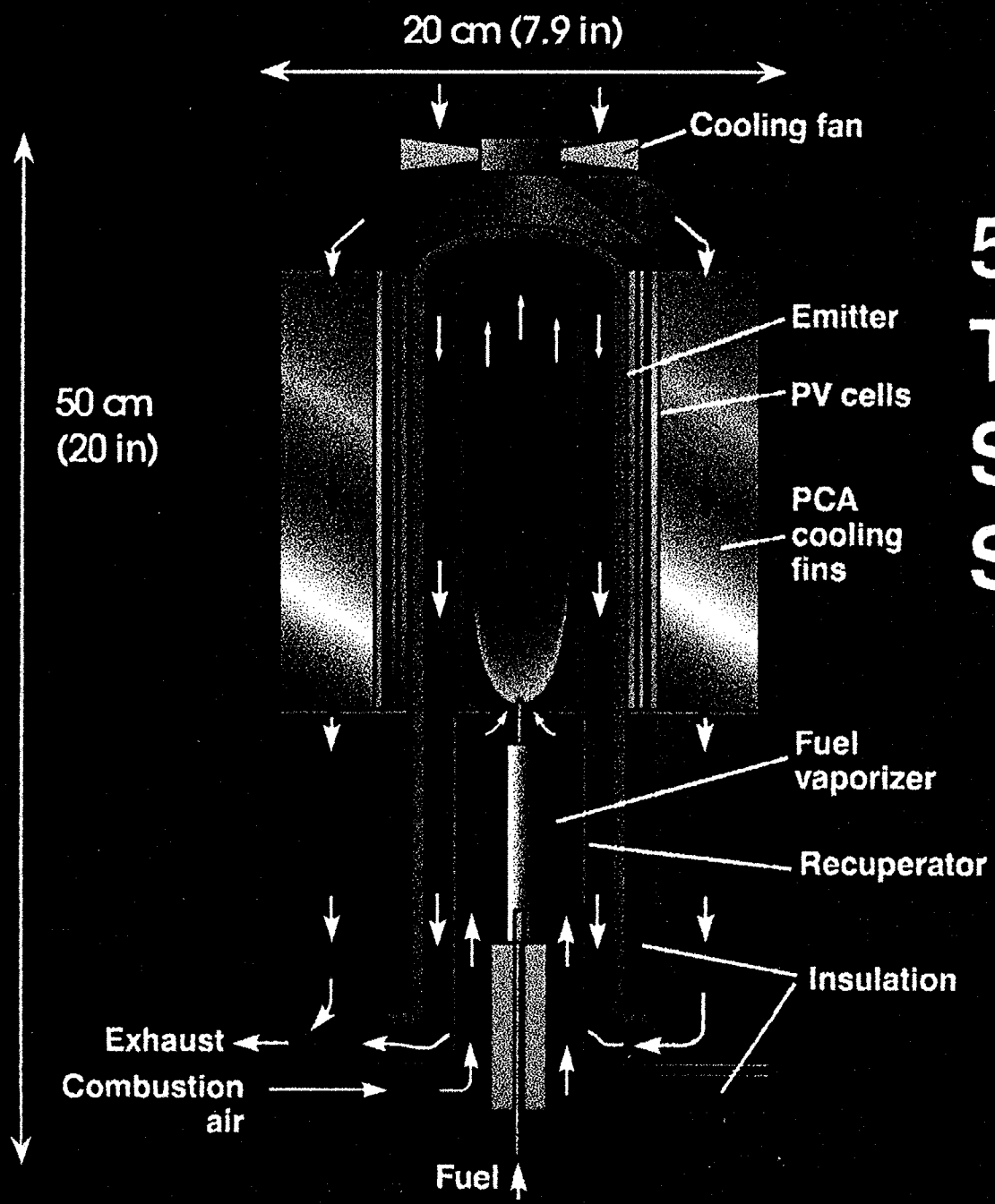
Selective emitter



- Discrete emission peaks
- Rare-earth oxides
- Greater percentage useful energy

EAB70918 10

500-W_e TPV Generator System Schematic



EAB70918 19

STATUS OF CLASSIFIED APPLICATION

- Creep rupture survival for 900 hours at conditions 100°C greater than use temperature (600 hour life is current design, would like to extend)

- Biggest test to date of CFCC to date in actual system
  - 20 CFCC parts, 200+ hours

- Next test beginning Sept. 22
  - 110 CFCC parts, 200+ hours planned

- Processing next round of 120+ parts for late Oct. test

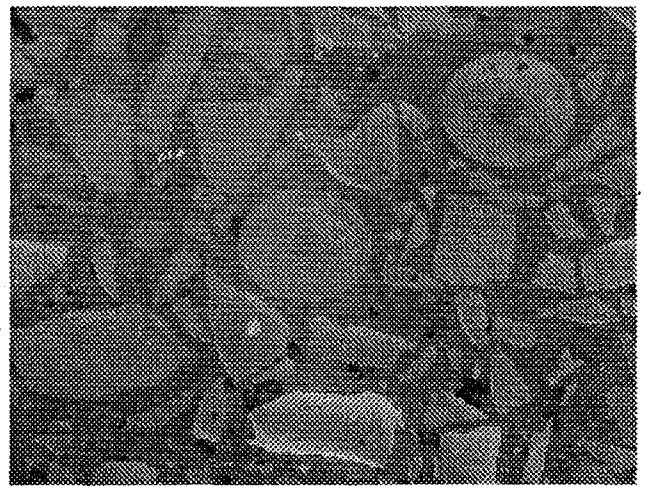
- Will continue to make ~120+ parts every 6 - 8 weeks

- Scale up to about double these quantities in spring 1998
  - actually about 20X on a weight basis

Continuous Fiber Ceramic Composites

— CFCC —

Working Group Meeting
October 6, 1997



Effort Presented is Performed Under
U.S. Department of Energy Cooperative Agreement
No. DE-FC02-92CE41001

Presented by: William S. Darden, Program Manager
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(978) 454-5632 fax
wdarden@systems.textron.com

PROGRAM OVERVIEW

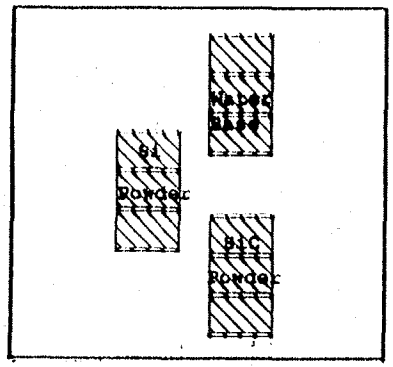
- Focus on tubular components for *industrial* applications
- Selected SCS-6 and SCS-9A reinforced NBSiC
- Open to SiC yarns when technically ready
- Slurry-based SiC & SN ceramic formulations utilizing wet filament winding and gas nitridation.
- TSC team includes:

Immersion Tubes			*Combustor*
F.W.Schaefer	**Hauck Mfg.(?)**	**TBD**	**Williams International**
Furnace Mfg.	Burner Mfg.	End-User	Stationary Turbine Mfg.

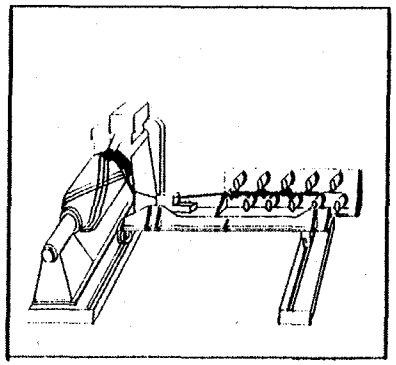
Materials Optimization/Evaluation

Composite Optics | MSC | IBIS | ThermoTrex

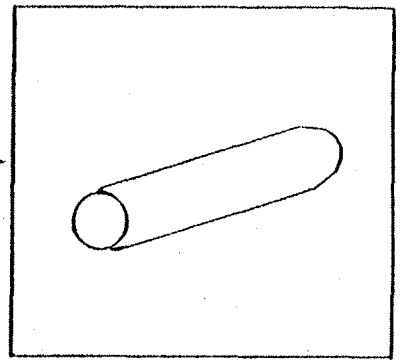
TYPICAL NB SiC CFCC PROCESS SCHEMATIC



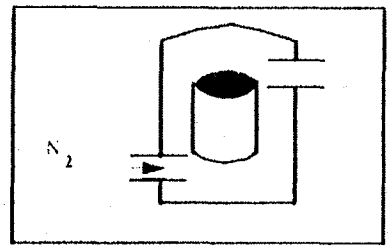
Powder Blending and Slip Formation
SiC + Si



Impregnate Fiber
and Filament Wind



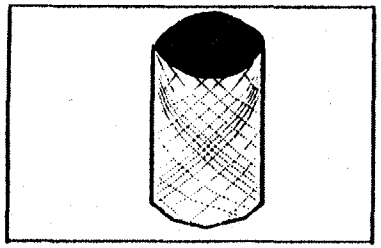
Tubular
Preform



Nitride Bond @ 1200 - 1400 C

Seal Surface *(if required)*

Rapid Densification
(if required)



Final CFCC
Tubular Component

Photos of Williams combustor and Doehler-Jarvis immersion tube

Phase 2 Objectives

- Transition to Prototype Components
  - Fabrication of Representative Parts
- Evaluate Fabrication Approaches
  - Refine Process Engineering
- **Test in Actual Service Environments**
  - **Testing by End-Users to Validate CFCC Benefits**
- **Demonstrate Scale-up Feasibility**
  - **Identify Major Cost Drivers**
  - **Automate Critical Process Steps**
- **Refine Economics**
  - **Document Economic Benefits to End Users**

Phase 2 Program Plan

- Task 3.2 Process Engineering
- **Task 3.3 Component Fabrication & Testing**
- **Task 3.4 Component Evaluation**
- Task 3.5 Joining

Process Engineering *(Task 3.2)*

- Material Property Data

ORNL: Thermal Diffusivity, Steam Corrosion, Creep Rupture
PNL: Fracture Toughness, Crack Bridging
UCinn: Thermal Shock
Williams Int'l: Tensile Strength, Modulus, LCF, CTE, Thermal Conductivity, Shear Strength, Flexural Strength
USC: Fiber/Matrix Interface Properties
COI: Consult & Test Planning

Process Engineering *(Task 3.2)*

- Rapid Densification (RD™) eliminated from consideration for these applications. An alternate surface sealing technique will be evaluated (ThermoTrex)
- 5-axis filament winding machine due for installation ~~Sept. 1997~~ ~~early Oct. 1997~~ ~~mid Oct. 1997~~ the week of October 20.
- Tooling modifications made for both applications

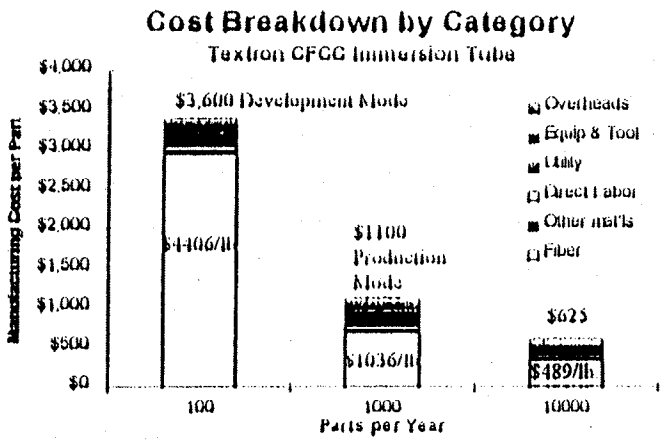
Photo of two mandrels here

Process Engineering *(Task 3.2)*

- IBIS Cost & Market Analysis
  - SiC fiber price vs. volume
  - Basic ceramic cost sensitivity
  - CFCC costs
    - assumptions
    - vs. volume
  - Conclusions

CFCC Immersion Tube Cost Breakdown

Cost Breakdown



IBIS Associates
Wellesley, Massachusetts

IBIS

Williams Int'l CFCC Combustor Liner

- Initial cost is a factor but performance benefits (higher T capability, lower CTE, longer life, reduced cooling) are more important.
- A new application *not* a replacement
- Total life cycle cost will be expected to be lower than metallic materials

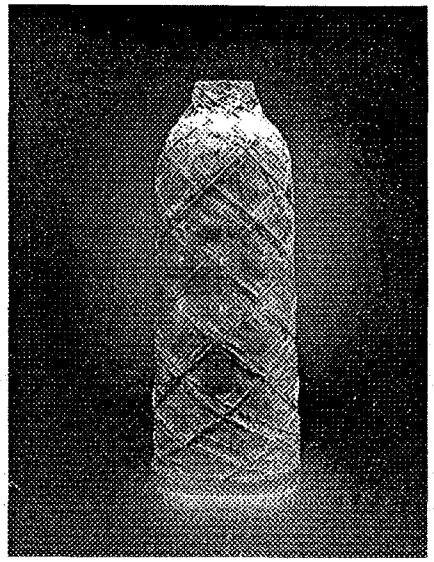
Component Fabrication and Testing
(Task 3.3)

- Immersion Burner Tubes
  - SCS-6/NBSiC
  - Closed-end Tube

- Status
  - ~25 parts so far have been produced using 3-segment plaster surfaced tooling
  - Tooling redesign and fabrication is completed; a demonstration tube is being made this month
  - Filament winding being transitioned to use new winder software
  - A new industrial (casting) partner is being "reeled" in

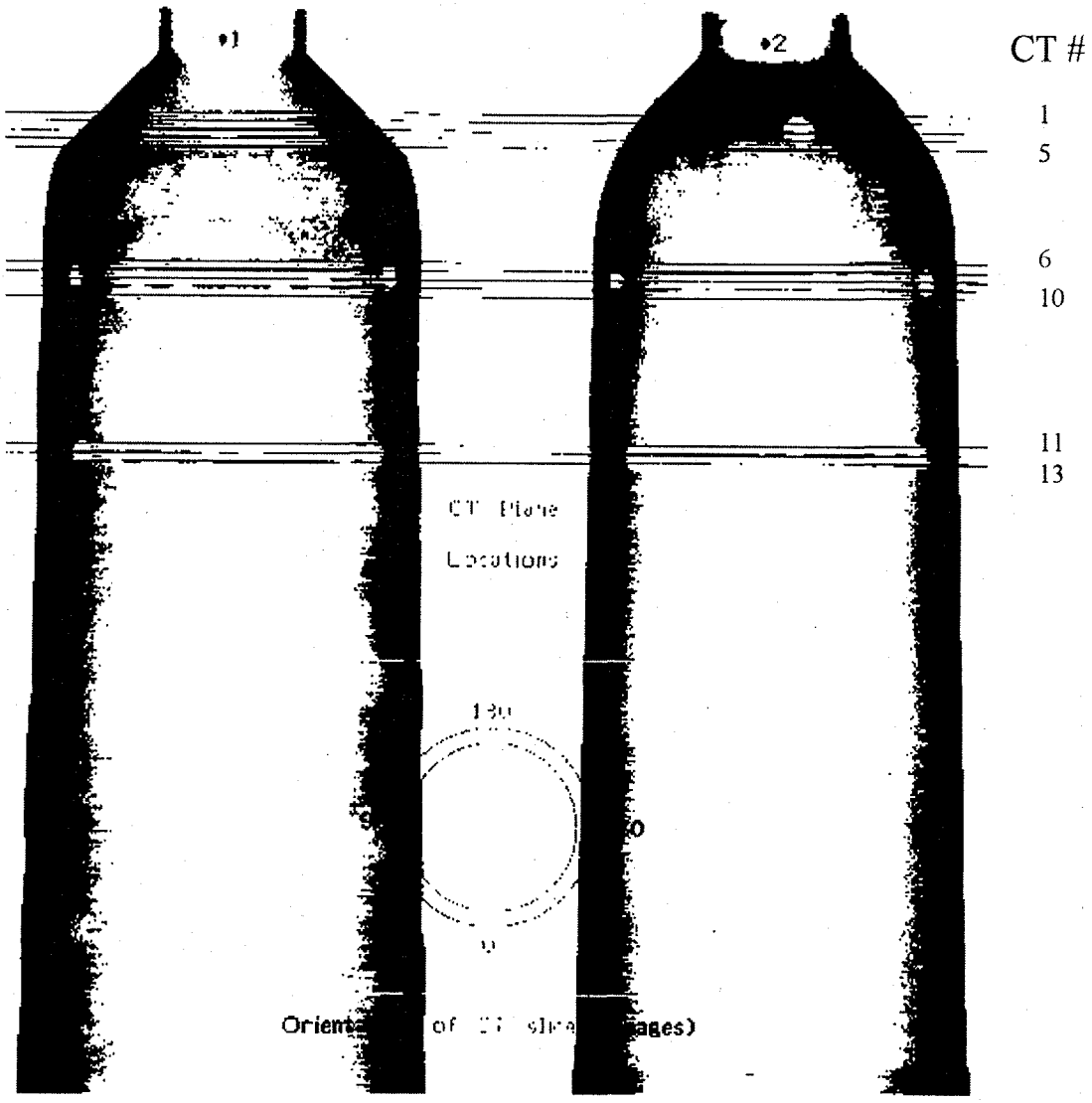
Component Fabrication and Testing

(Task 3.3)

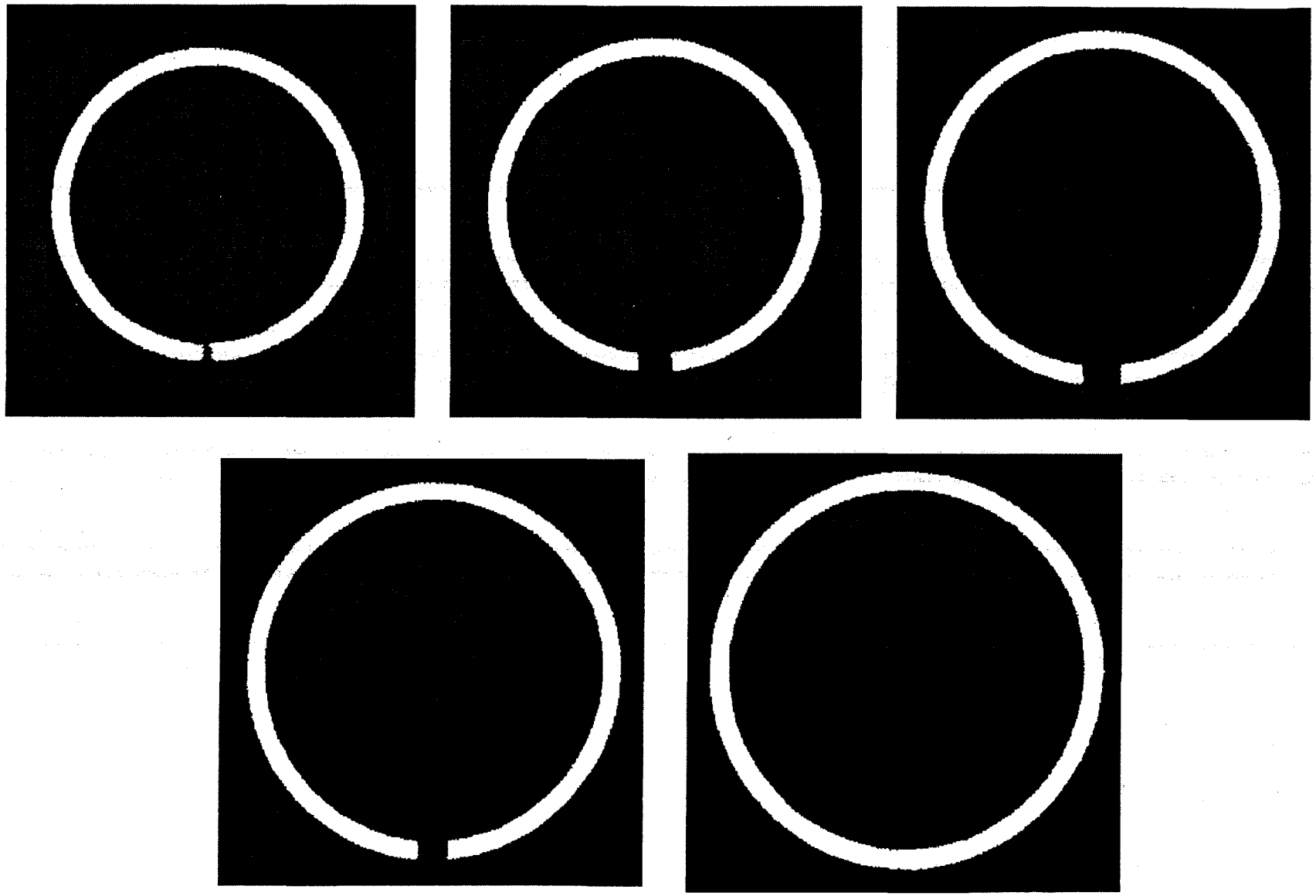


- Stationary Turbine Combustors
  - SCS-9A/NBSiC
  - 4" Dia. X 10.5" Long
  - Tapered + Bottle Neck Tube
  - +/- 45° Fiber Orientation
  -
- Status
  - ~15 full scale parts have been made.
  - Tooling has transitioned from 3-segment-to-2 piece mandrel
  - CFCC panels for design data testing are in-process
  - 2 second-generation combustors have been machined with a new hole pattern, NDT'd, and are to be burner tested for 100 hours at Williams (October 1997)

Computed Tomography Sections for Textron Liners



CT Slices for Liner # 1



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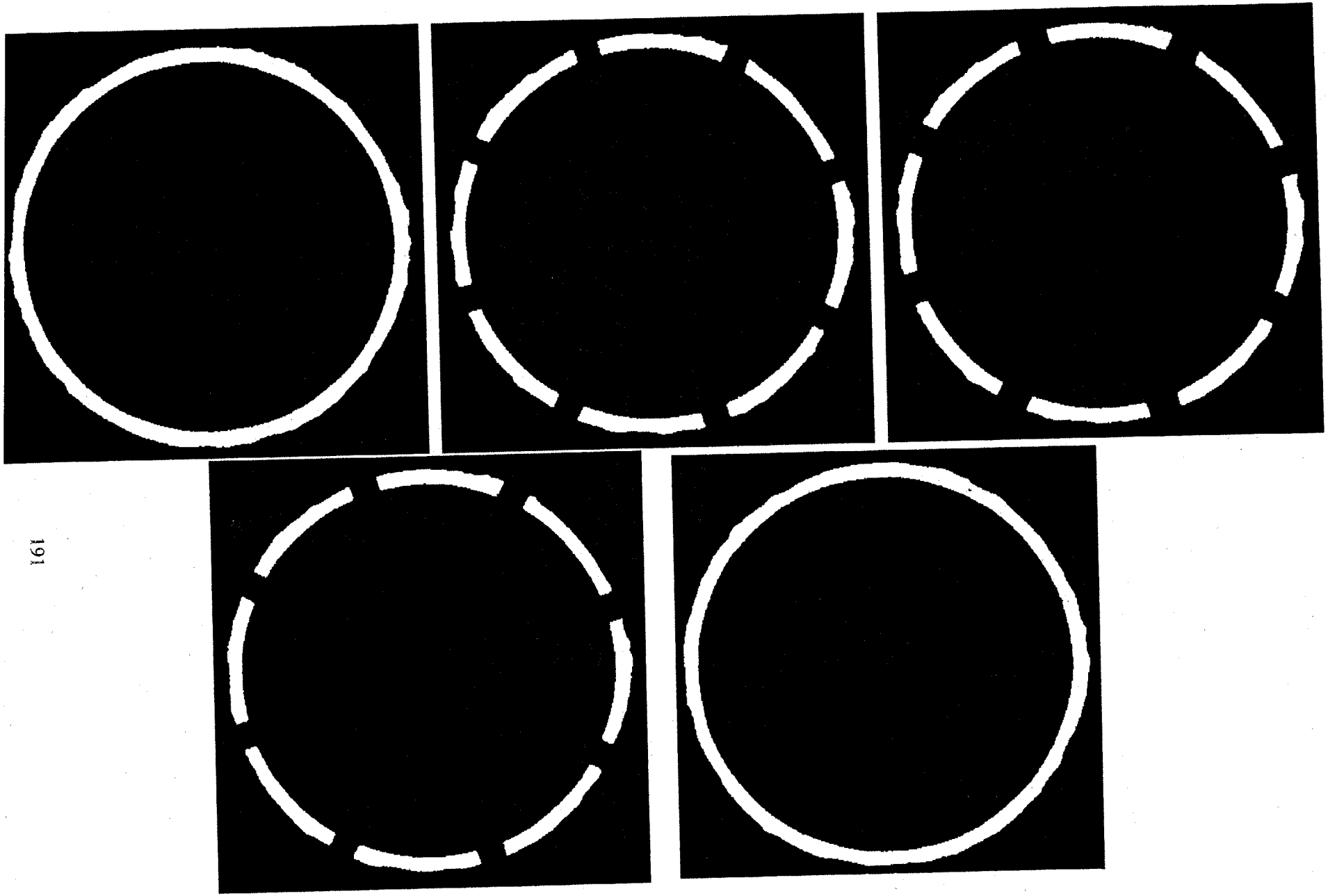

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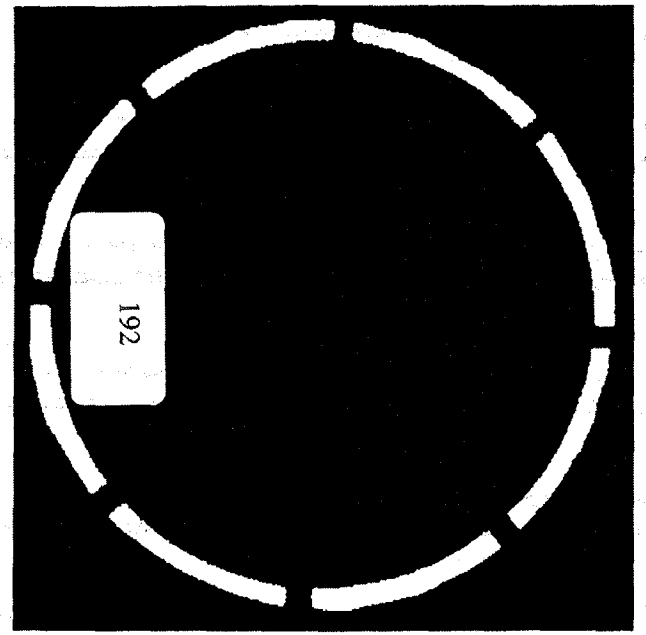

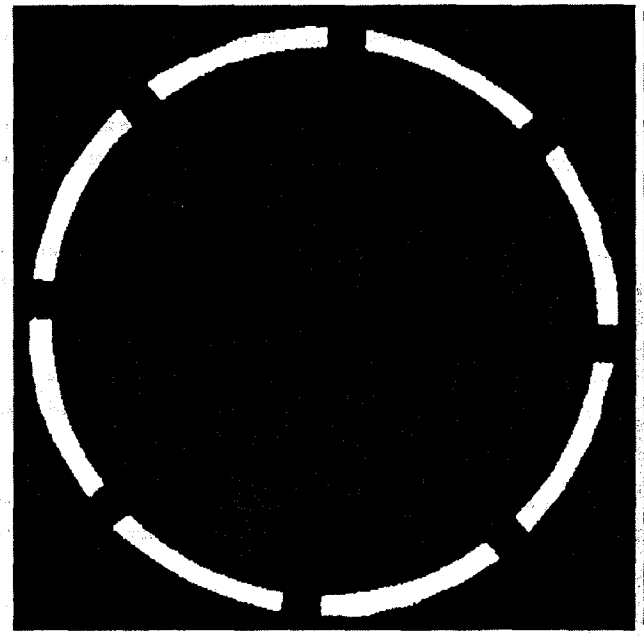

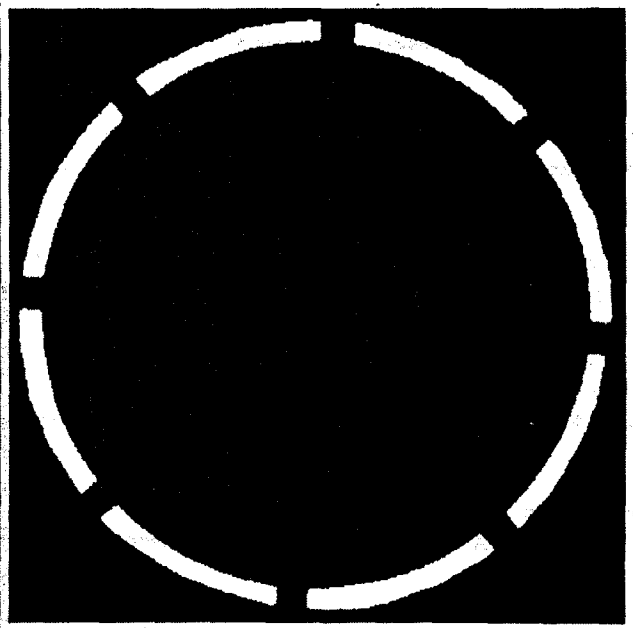

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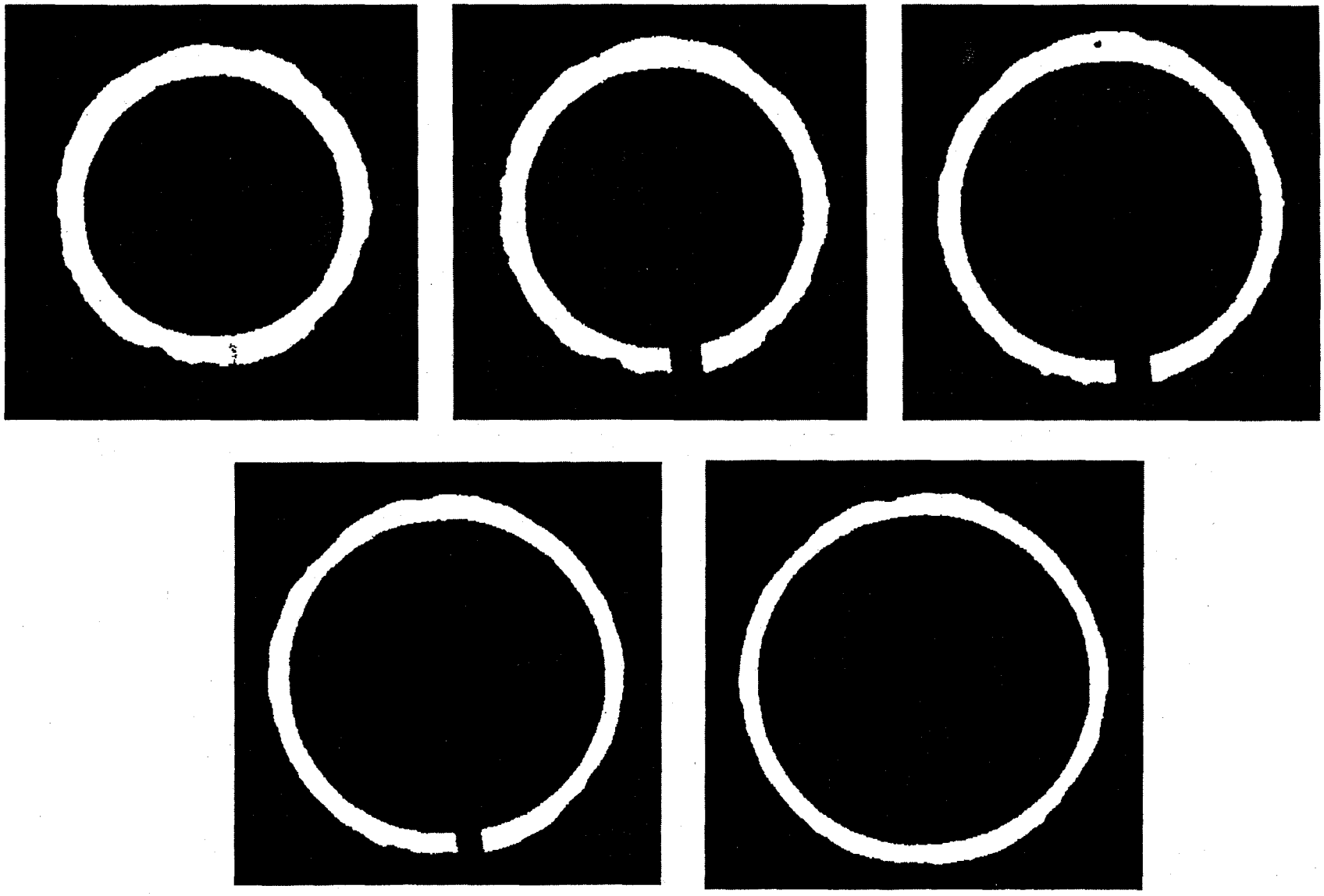

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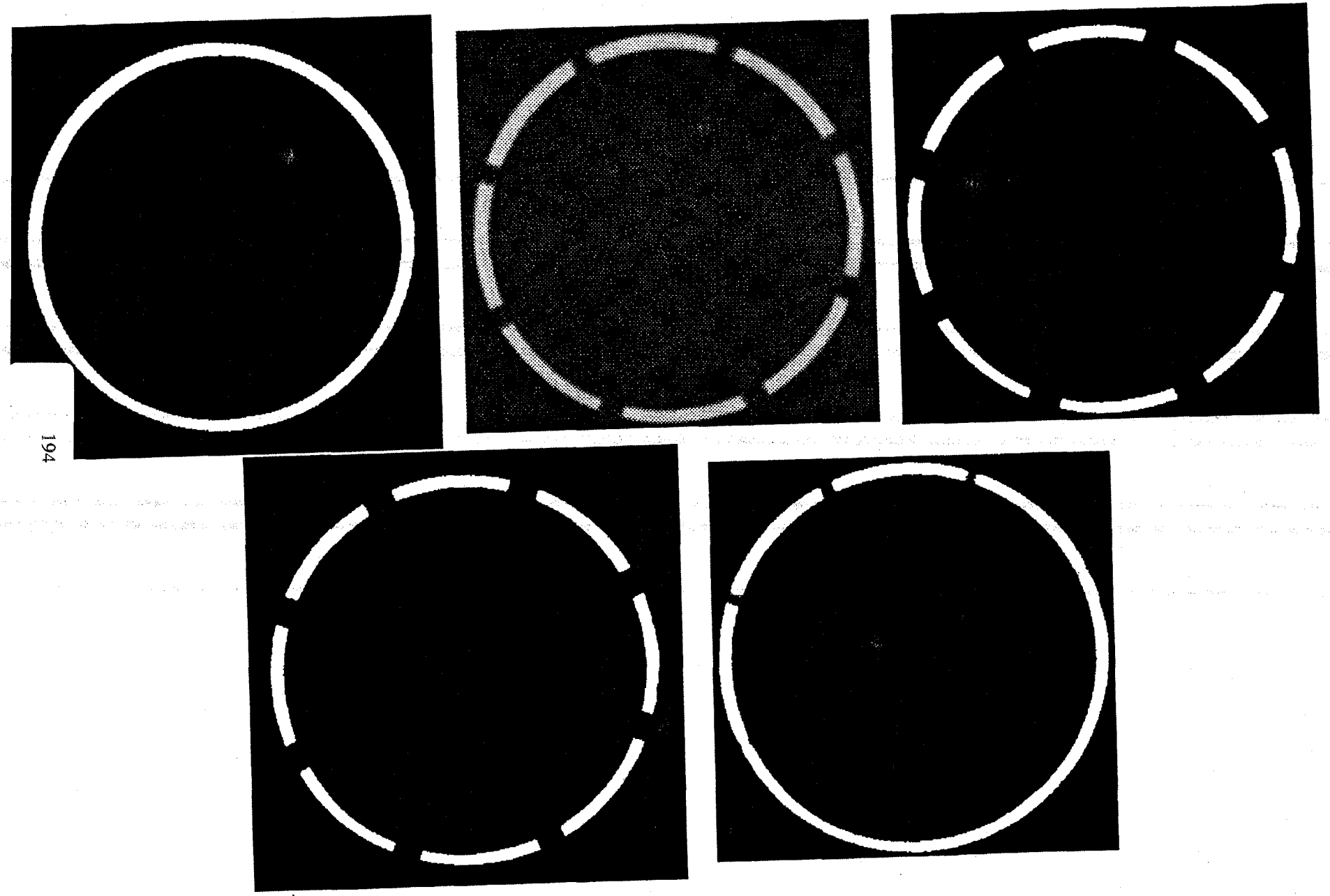

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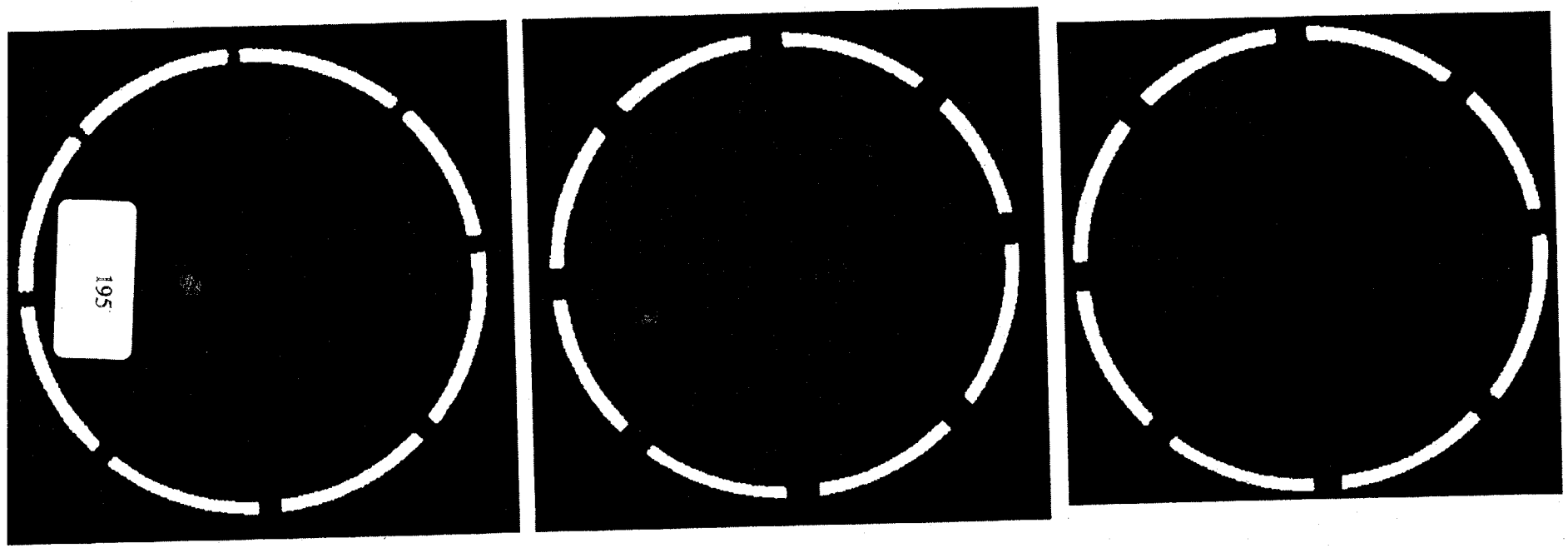

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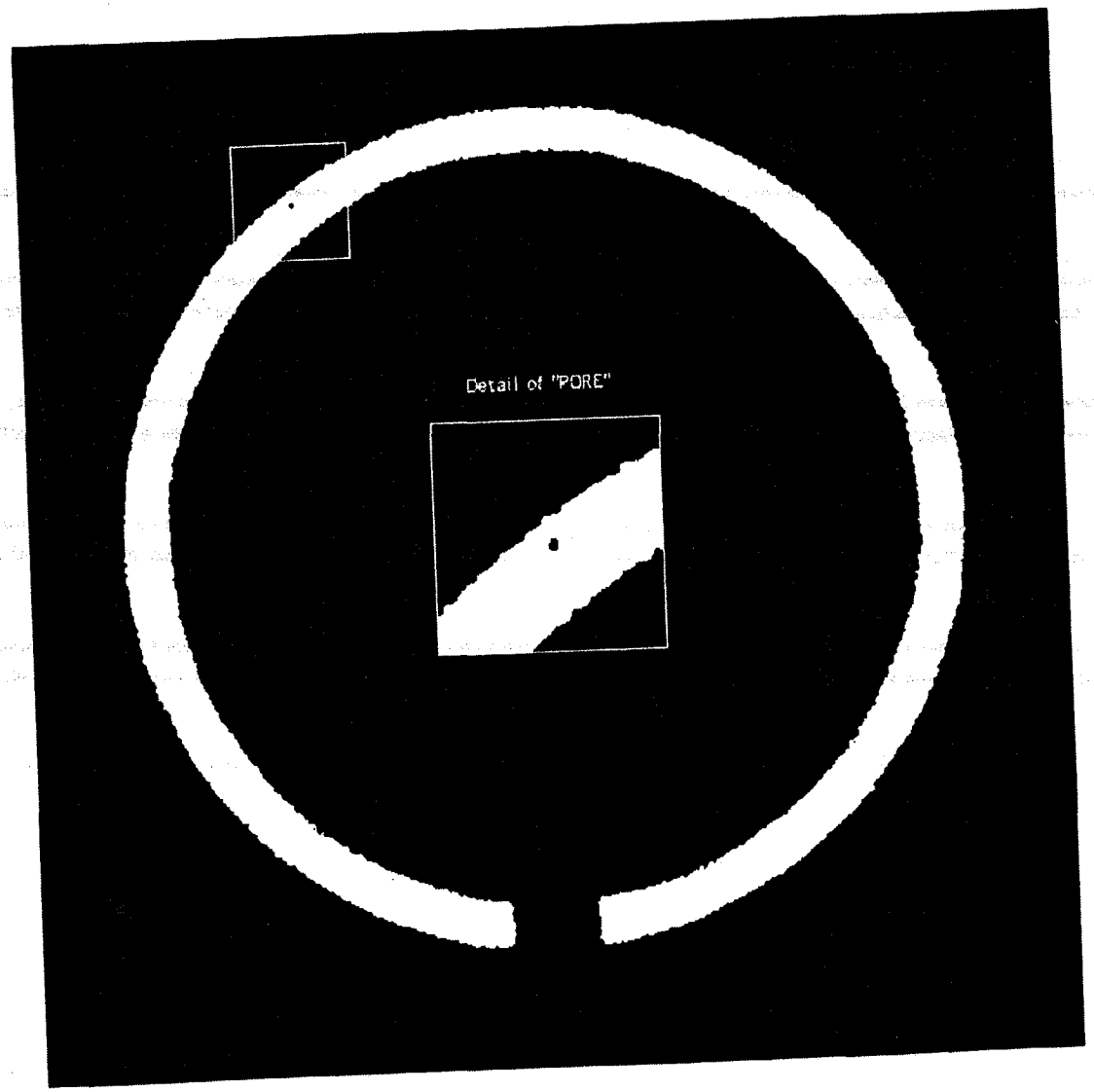

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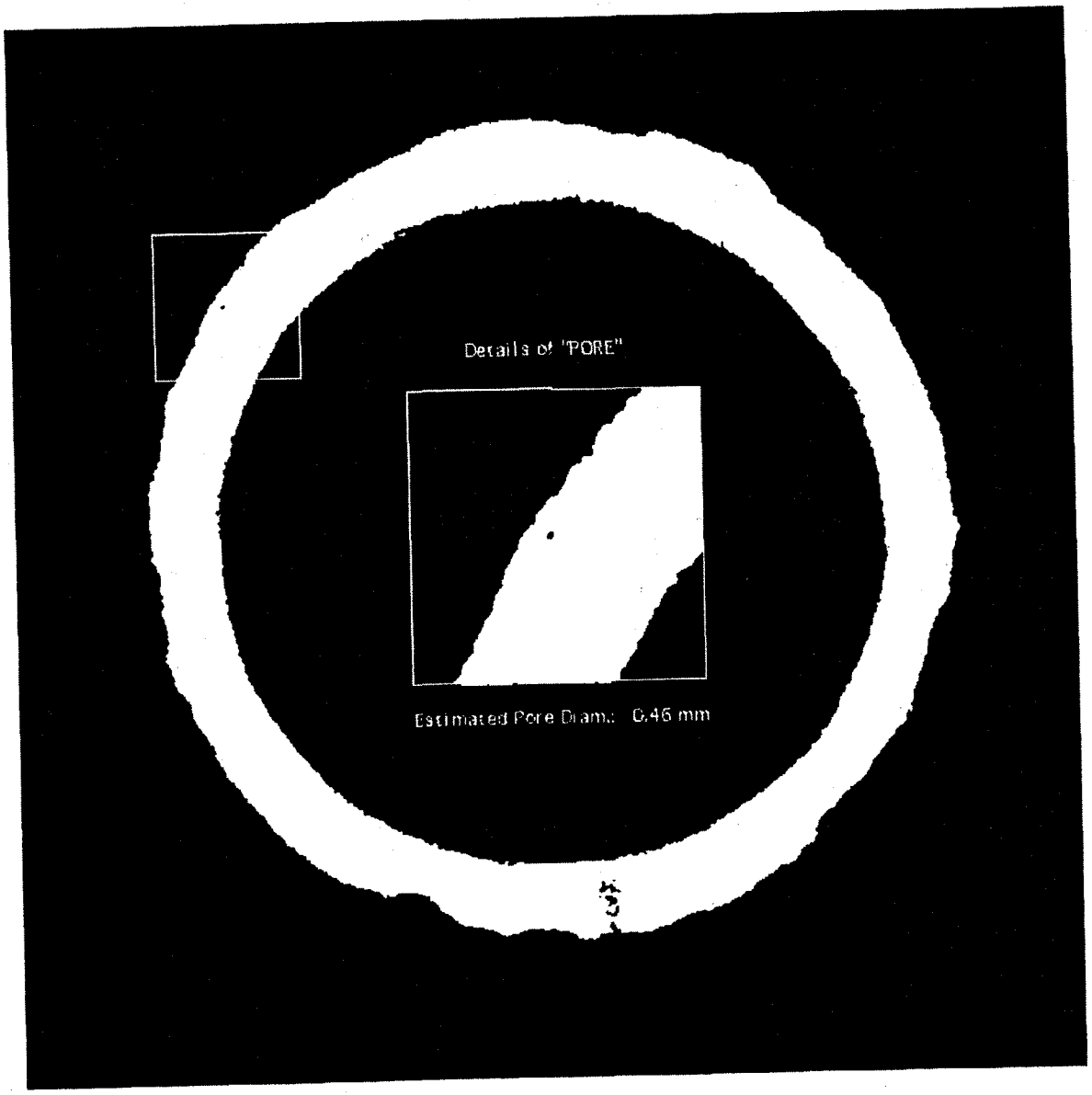

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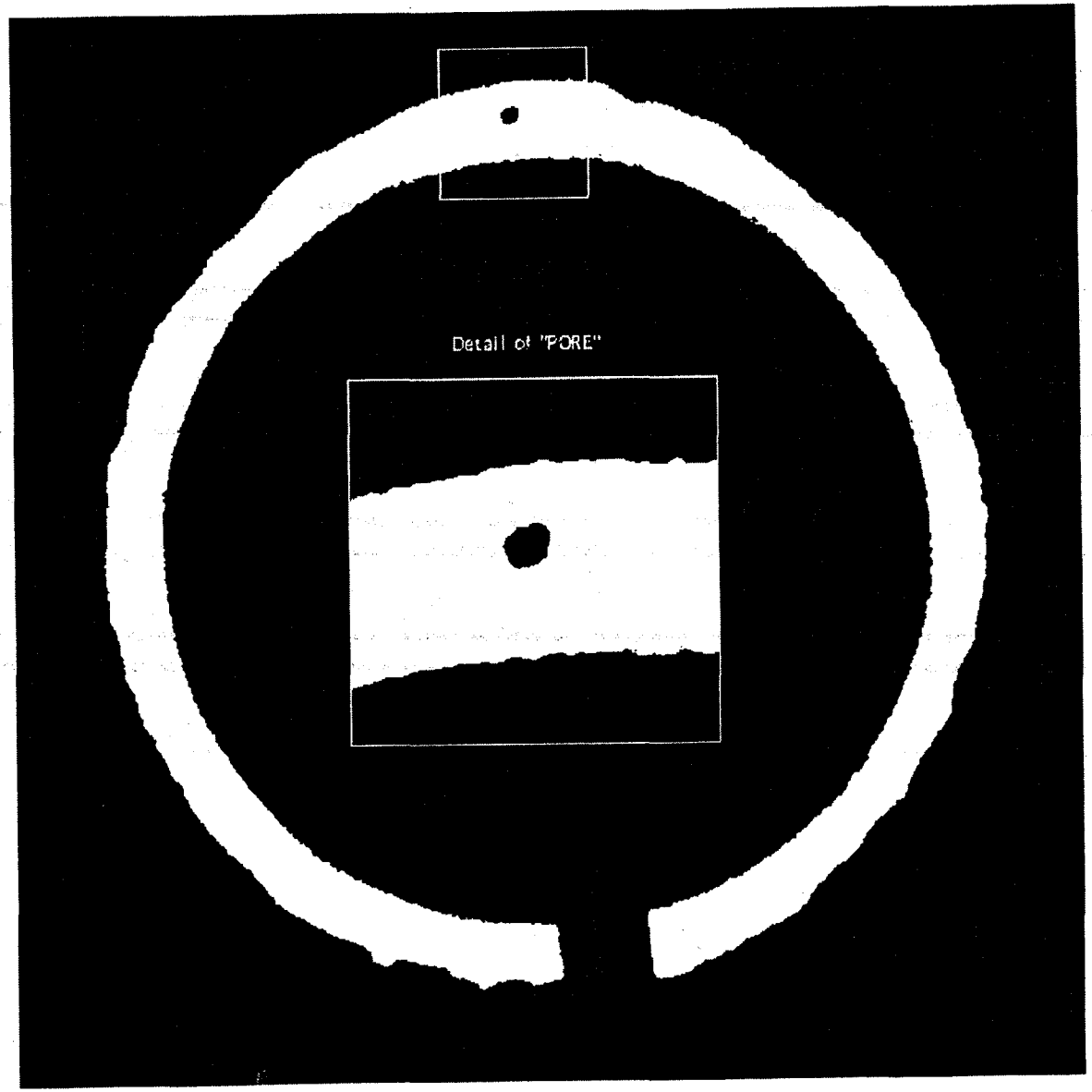
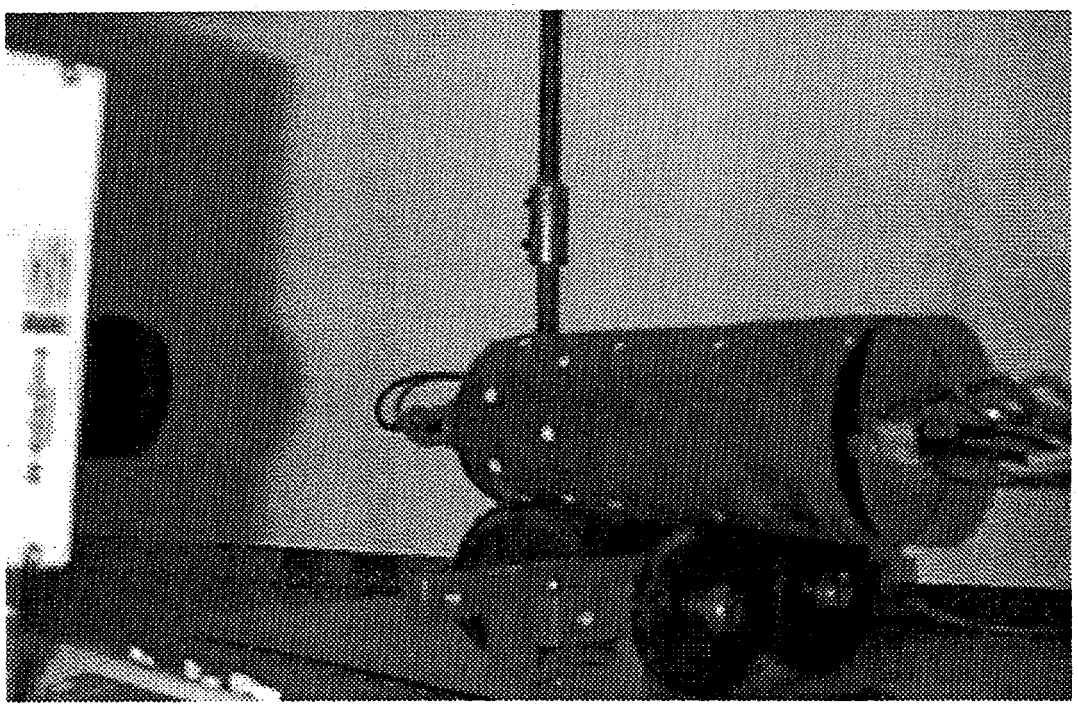
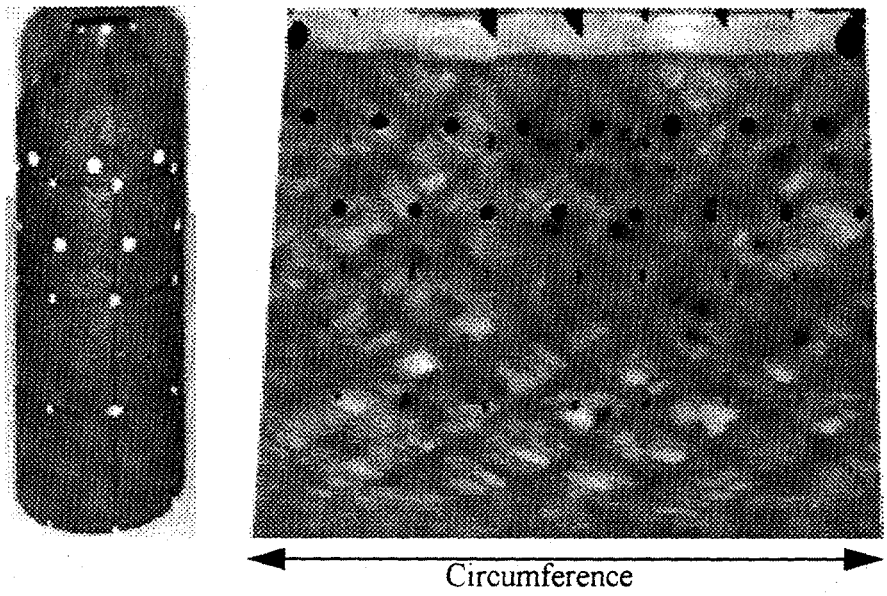


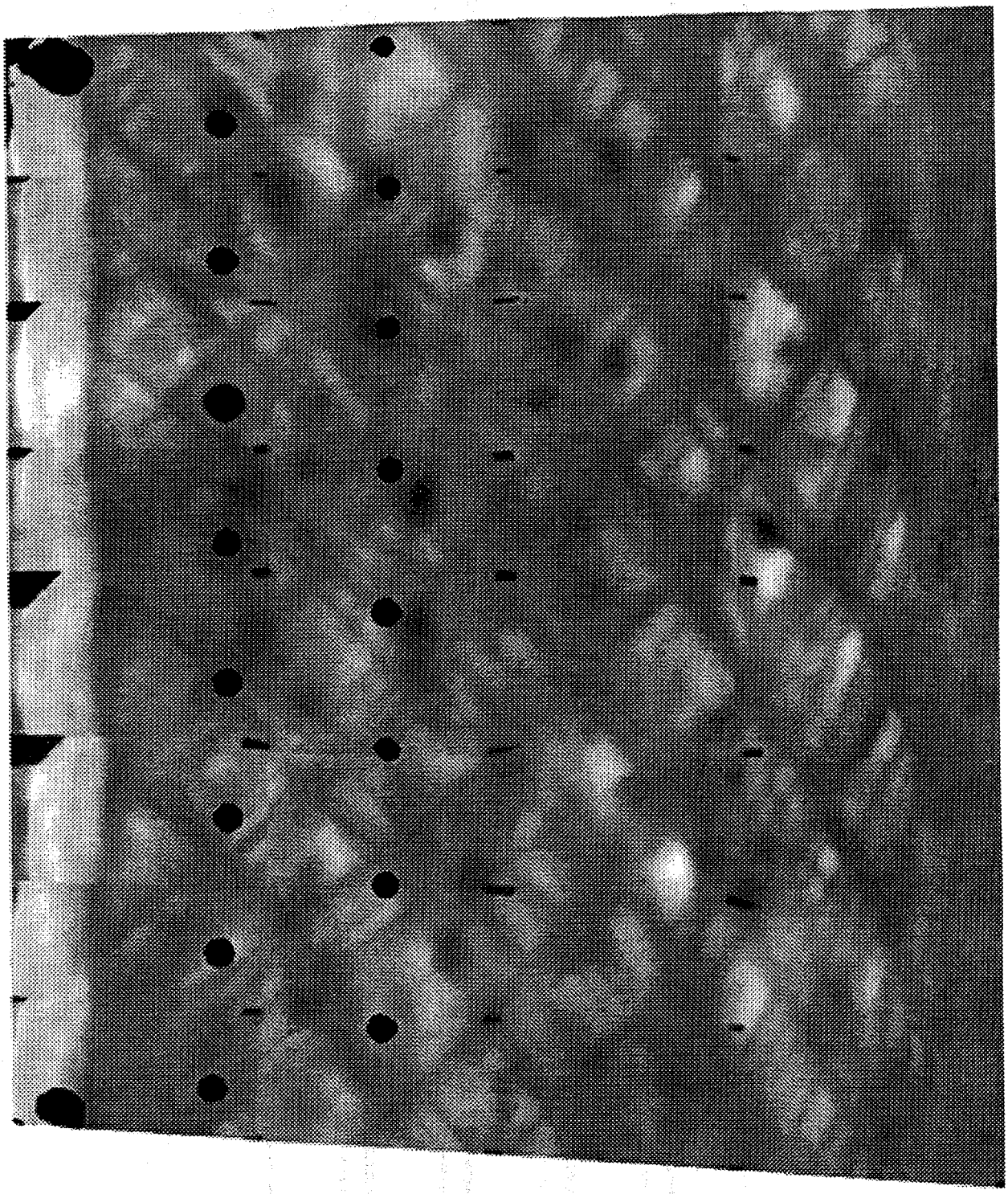
Thermal Imaging Test Setup



Orientation of Thermal Diffusivity Image

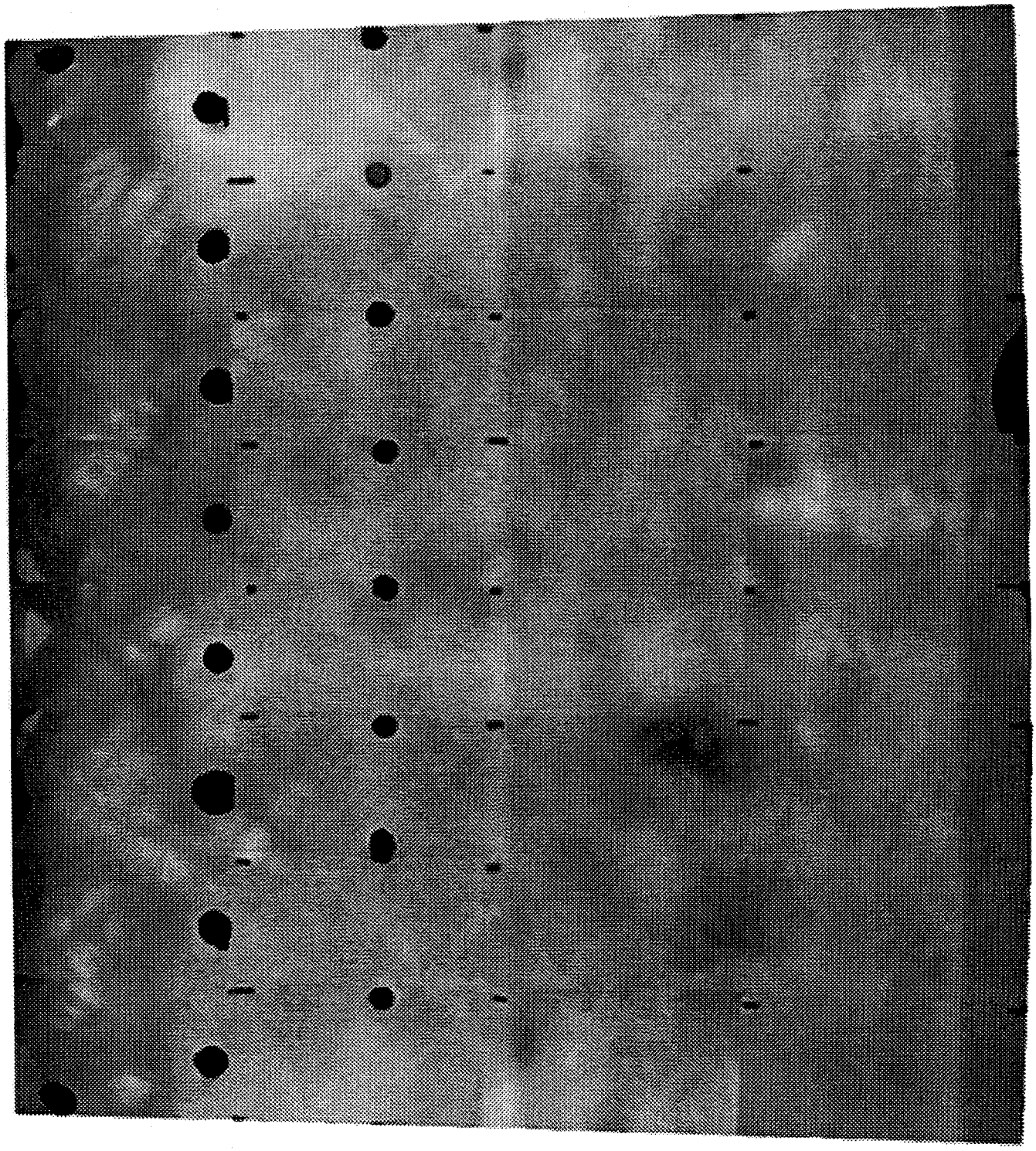


Thermal Diffusivity Image of Textron Liner # 1



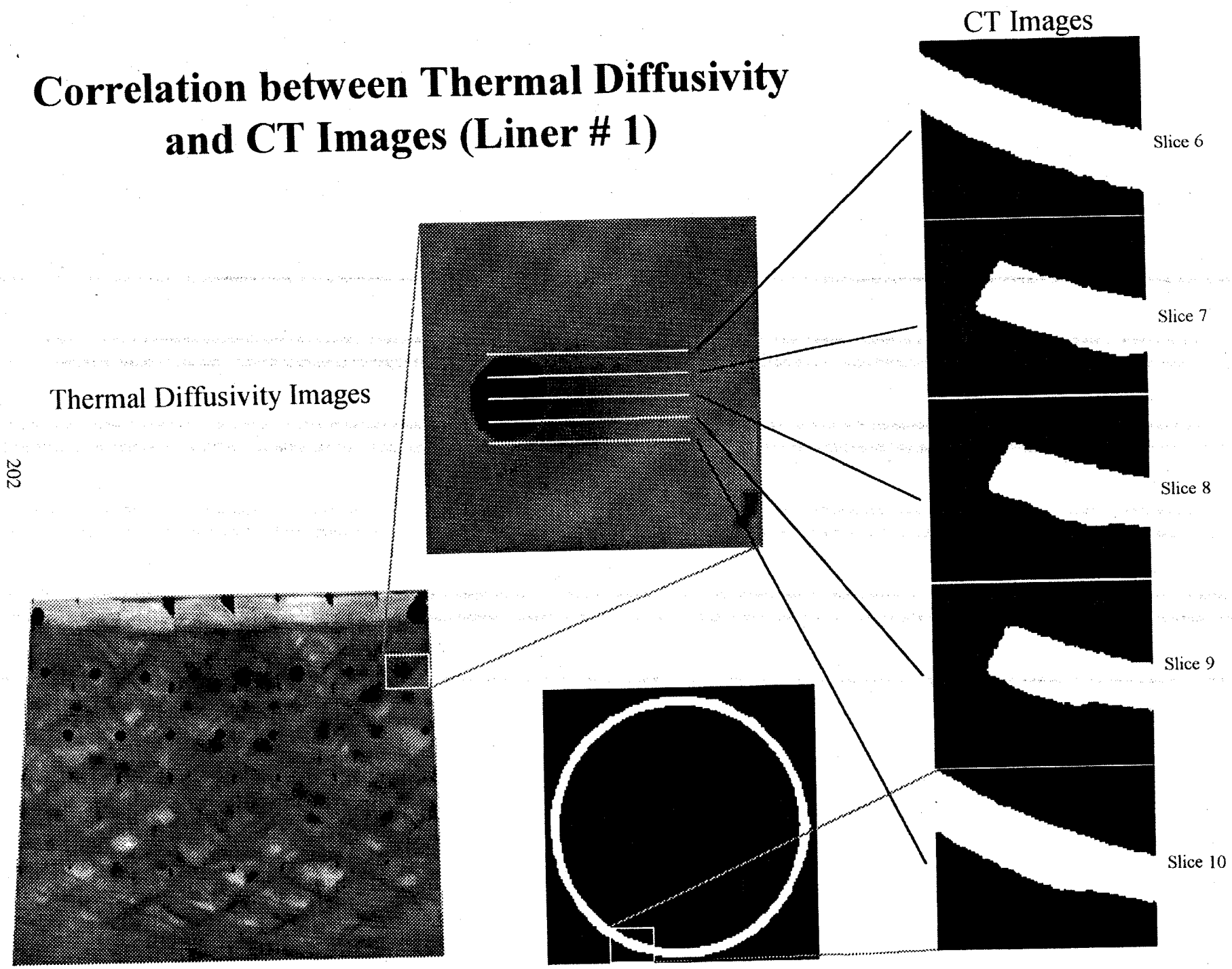
$1 \times 10^{-7}\ m^2/s$ Thermal Diffusivity $8.5 \times 10^{-7}\ m^2/s$

Thermal Diffusivity Image of Textron Liner # 2



1×10^{-7} m²/s Thermal Diffusivity 8.5×10^{-7} m²/s

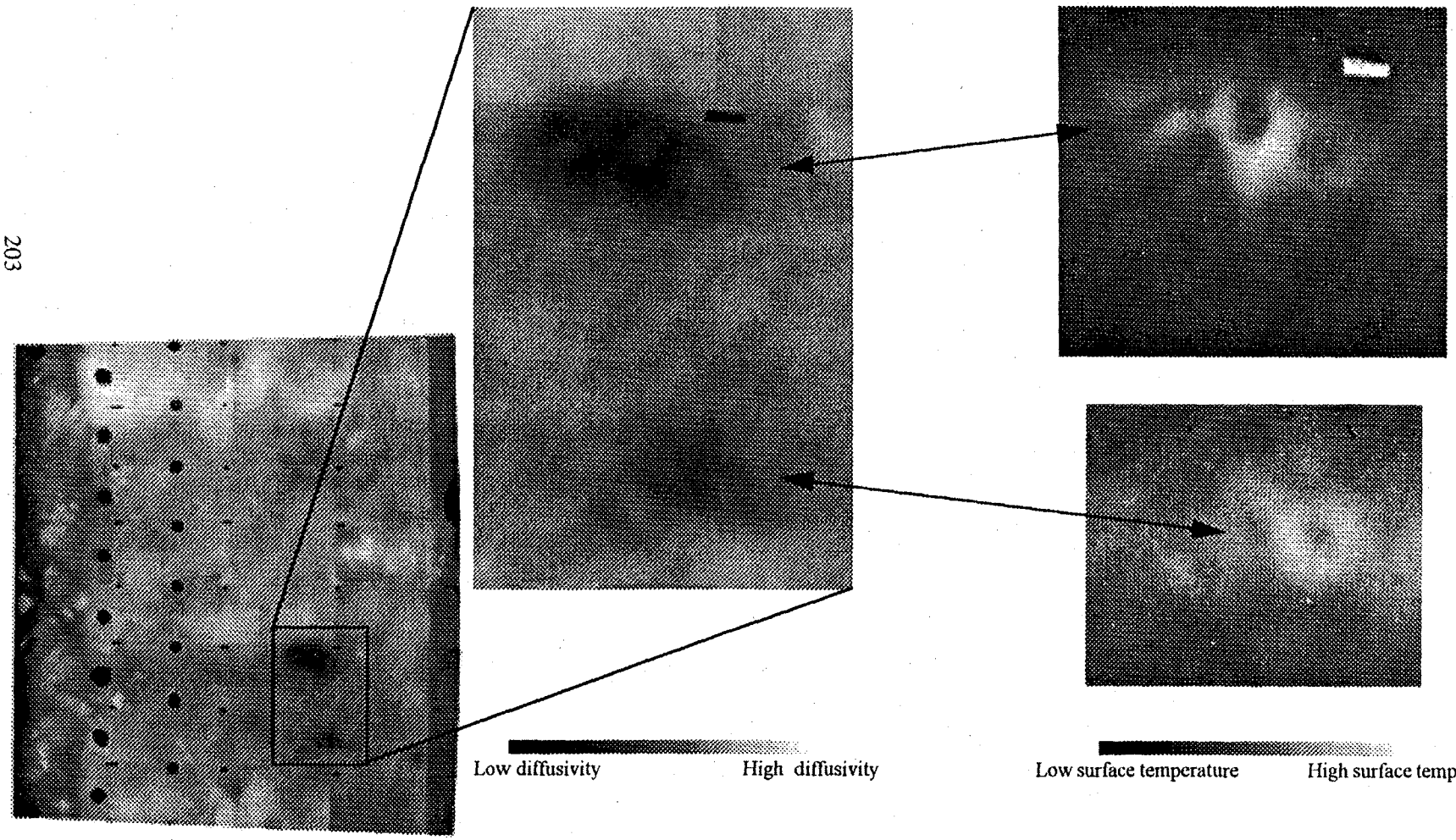
Correlation between Thermal Diffusivity and CT Images (Liner # 1)



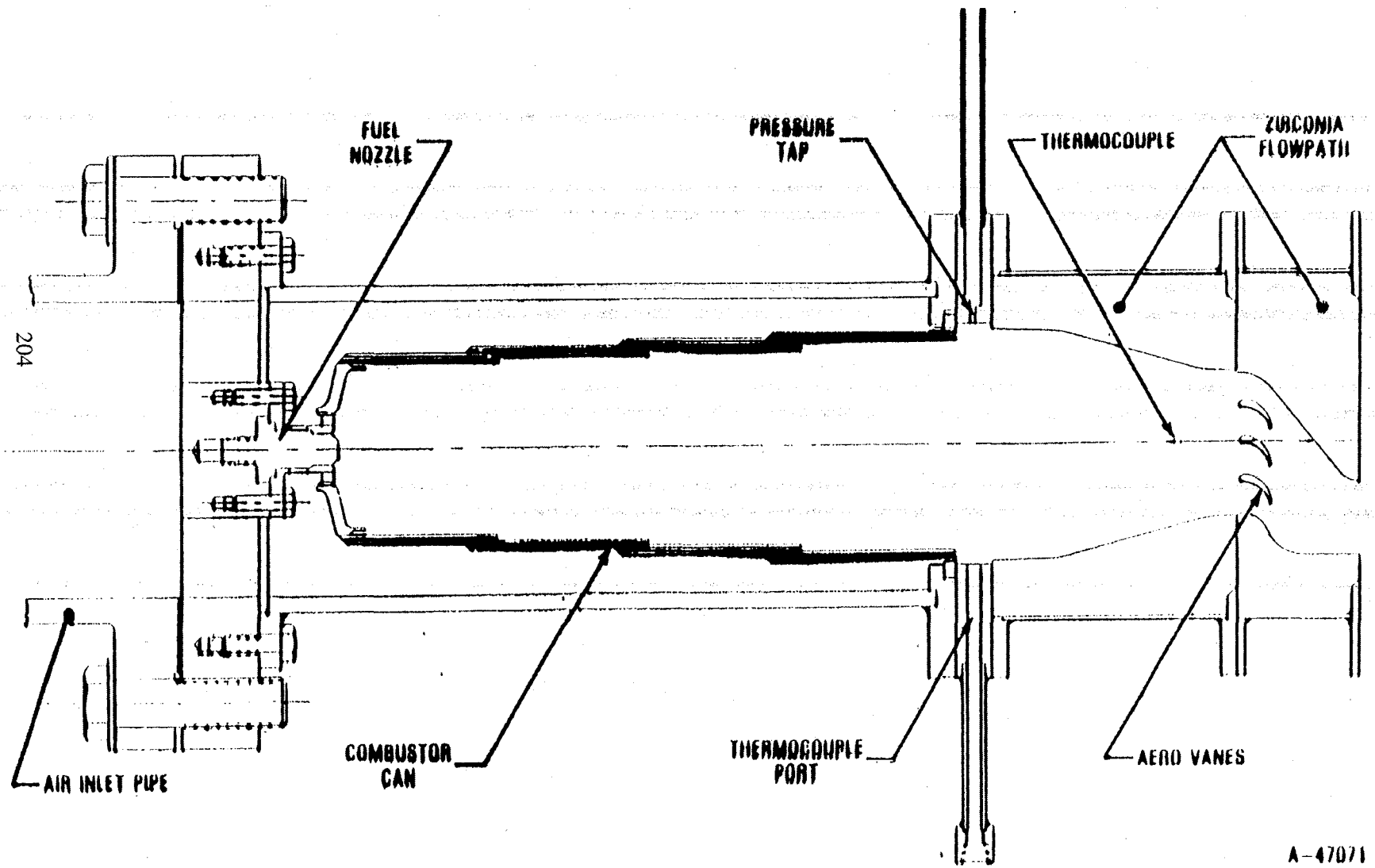
Correlation between Thermal Diffusivity Image and Front Thermal Flash Image (Liner # 2)

Thermal Diffusivity Image

Front Thermal Flash Image



WILLIAMS INTERNATIONAL HIGH VELOCITY COMBUSTOR RIG



Component Evaluation *(Task 3.4)*

- Microstructure
  - Demonstration immersion tube
- Physical, thermal, NDE and mechanical properties
  - Panel testing
  - After burner rig / casting furnace exposures
- Chemical Stability
  - confirm non-wetting in molten aluminum

Joining *(Task 3.5)*

- Sandia National Laboratory
  - Screening tests (wetting, reactivity, bonding)
  - Bond strength

- Ames Laboratory
  - Aysmmetric four point shear

Textron CFCCs : New & Improved

New: **Filament winder**
Industrial partner(s)
Property data
Personnel

Improved: **Tool designs**
Component designs
Winding capability

The future looks solid & the best is yet to come

DuPont Lanxide Composites Inc.
CFCC Program for
DMO Materials

Program Review

Oct 1997

Phillip A. Craig, Program Manager

Mr. Joe Mavec, DOE Program Manager

CFCC DMO Materials Program

- DMO generic for DIMOX™ directed metal oxidation process by Lanxide.
- Primary material to be studied is SiCf/Al2O3.
  - Ceramic Grade Nicalon™ silicon carbide fiber
  - Boron Nitride/Silicon Carbide dual interface
  - Alumina matrix with residual metal removed.
  - Except for scale-up, same recipe since 1989.
- Fiber Reinforced Thin Sheet Program
  - SiC or Alumina chopped fiber reinforced Alumina.
  - Intent is a system which is CTE matched.
  - Opportunity for CFCC performance at $100 / lb.

DuPont Lanxide Composites' DMO CFCC Program DE-FC02-92CE40994

CFCC Industrial Applications

- Foster Wheeler Development Corporation:
  - Very strong corporate committment at FWDC.
  - Petrochemical exposure testing.
    - » SiC/Alumina didn't perform well in coal ash exposures.
    - » Looks good in hot erosion applications.
  - Tube shield for heat exchanger tube life extension.
    - » Utilizing thin sheet ceramics materials.
- Heat Recuperators for Industries of the Future
  - Adaptation from HEV - Thin Sheet recuperator. Particulate reinforced thin sheet for lowest cost.
  - Chopped fiber materials in development to provide improved toughness.

CFCC Turbine Engine Applications

- General Electric Utility Turbine Engine Shroud
  - Comprehensive design, database, rig test program.
  - Planned use by huge power generation"utilities".
    - » Additional smaller engine application for near term being identified.
  - Engine product designers are part of the team.
- Solar Turbines' Industrial Turbine Engine Components
  - Corporate committment to evaluate many CFCC's.
  - Test Rig for exposure to combustion gases.
  - New designwork for tip shroud and other engine components.

Material Specific Successes

- Standardization of "product release" Specifications
  - CG Nicalon 6600 hour stress rupture test.
  - Reproducible L.C. Fatique behavior demonstrated.
- Fabrication limits established with consistent properties
  - 2mm to 10mm thickness; with 2:1 thickness variations.
- Hi-Nicalon™ fiber substitution Qualified.
  - 7700 hour stress rupture data.
  - Improved lifetimes due to fiber stability.
  - Adopted for use in GEPS Program.
- Joining techniques demonstrated
  - Brazing method for DMO-Superalloy joining to 800 C.
  - Ceraset™ preceramic polymer method for DMO-DMO joining to 1100 C.

DuPont Lanxide Composites' DMO CFCC Program DE-FC02-92CE40994

Interfaces - Specific Successes with Sandia National Lab

- Mark Allendorf and modeling team at Sandia.
- DLC Interface Coating Improvements
  - Process chemistry better understood w/ Sandia help.
  - Demonstrated improvements in uniformity
    - » Tray to Tray, Part to part, Within a Part
  - Comprehensive process understanding for BN :
    - » Shutoffs of power, process gasses, heat, pressure.
- Predictive model with Sandia Nat'l Lab
  - Correlations on coating performance for 50+ runs.
  - Providing daily use (PC-based) model for run set-up.
  - Supports other materials which can use BN/SiC dual interface system.
    - » CVI Enhanced SiC/SiC and Silicon MI systems.

Applications Specific Successes

- Solar Turbines Combustor
  - 8"dia x8" subelements used in combustor rig
  - 100 + hours @ 2150 F with no damage.
  - Leading to development of shroud for Solar engine.
- Validates use of DIMOX CFCC's for GEPS Shroud program:
  - Hi-Nicalon fiber will be used for higher stresses and longer lifetimes.
- Solar combustor and shroud work, and GE shroud program, are important to Low NOx system solutions.
- Commercial Spin-Offs
  - Successful programs and tests with many turbine engine customers, world wide.

Fiber Reinforced Thin Sheet Program

- Material and Process potentially solve several problems
  - Fast manufacturing cycle time
    - » Traditional preforming replaced with "paper" sheet technology; 24" x 100's feet in 1/2 day.
  - Very thin wall structures
    - » Application oriented to replace sheet metal.
  - Microcracking may be naturally solved
    - » Oxide/oxide/oxide system is CTE matched.
- Characterization work led by ORNL - H.T. Lin.
- Applications for:
  - Hybrid Vehicle heat recuperator for engines
  - Petrochemical applications
  - Excitement due to low cost alone.

DuPont Lanxide Composites' DMO CFCC Program DE-FC02-92CE40994

Materials Path Forward

- Improved SiCf/Alumina
  - Fiber modeling of mechanical trade-offs of modulus and CTE versus microcracking.
    - » Sylramic™, other fibers, may be more beneficial.
  - Support ORNL studies of fiber/interface oxidation
    - » Understand embrittlement of BN / CG Nicalon
    - » Need to understand CG system, as lowest cost fiber.
  - Aluminas should have slower oxidation mechanisms, and so if optimized will be improved materials over SiC/SiC:
- Understand & Characterize Hi-Nicalon/Alumina system
  - Quantify benefits vs. increased fiber cost.
  - Allow hotter post processing, for mullite coatings.
- Oxide/Oxide CFCC System
  - Need improved oxide interface and fibers come (Nex 720).

DuPont Lanxide Composites' DMO CFCC Program DE-FC02-92CE40994

DIMOX SiCf/Al2O3 Situation Today

- Commercially Sound Material
  - Performance and Cost follow Fiber selection.
  - T. E. applications are proceeding successfully:
    - » Serious commercial TE tip shroud application.
    - » Newest military flaps and seals displayed at Paris.
- Long Life Trials - Several 7000 hour datapoints.
- Manufacturing Cells Exist
  - Can be cost competitive with single crystal alloys.
- SiCf/Alumina part of DLC's Corporate Strategy
  - DLC is a manufacturing company.
  - DLC expects to produce several types of CMC's, and several families of materials (MMC, Polymers, etc).
  - SiCf/Alumina provides economic CFCC solutions.

Conclusions

- CFCC Program has clearly advanced the state of industrial readiness of DIMOX SICf/Alumina materials.
  - In material science,
  - In processability,
  - In reducing barriers to entry for industrial applications.
- Work planned in the original Phase 2 is not complete, and 1 to 2 years of effort are needed.
- Additional work to correct deficiencies in performance in chemical applications has been thought out.
- Continued work in DMO-CFCC Program provides positive assistance to applications and material science, with a strong commercial future.

DOW CORNING

Developments in PIP-Derived CFCCs

CFCC Program Review

Andy Szweda, Daniel Petrak, Ronald Boisvert
Dow Corning Corporation

Lake Tahoe, NV
October 6 - 7, 1997

1 10/28/97

Presentation Outline

- **PIP Materials & Process**
- **CFCC Applications**
- **SYLRAMIC™ SiC Fiber Reinforced CFCC**
- **Environmental Testing**

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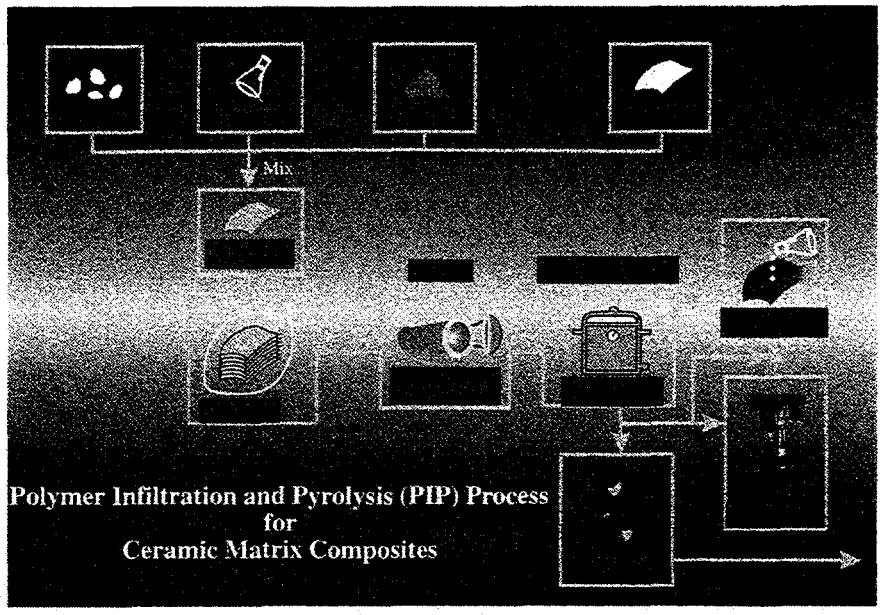

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SYLRAMIC™ Composites

- **SYLRAMIC™ S100 composite for low temperature applications**
  - **CG Nicalon 8 harness satin**
  - **Carbon interface coating**
- **SYLRAMIC™ S200 composite for moderate temperature applications**
  - **CG Nicalon 8 harness satin**
  - **Proprietary interface coating**
- **SYLRAMIC™ "S300" composite for high temperature applications**
  - **SYLRAMIC™ SiC Fiber**
  - **Advanced proprietary interface coating**

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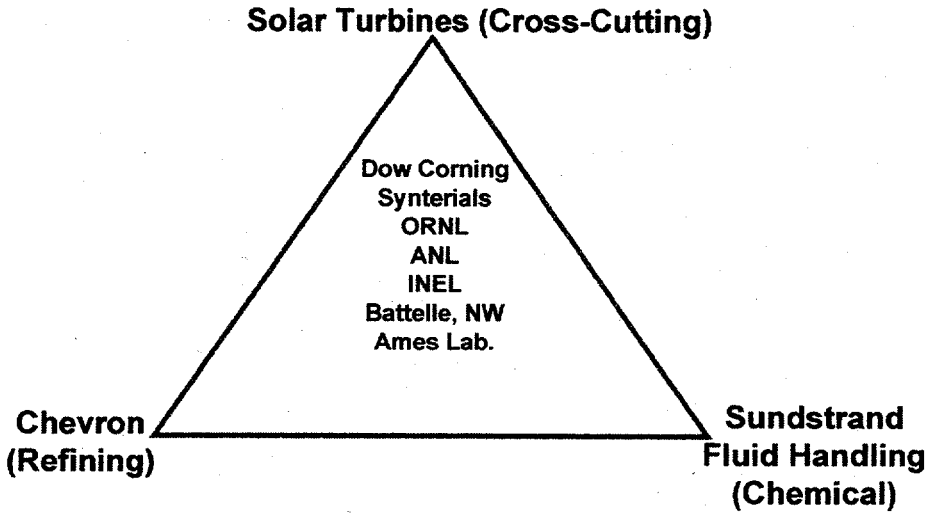
DOW CORNING

Dow Corning's CFCC Team



CFCC Applications

- **Solar Turbines (Cross-Cutting Technology)**
  - **Combustor Liner**
  - **Rim Seal**
  - **Turbine Tipshoe**
- **Sundstrand Fluid Handling/Aerospace (Chemical, Refinery, Pulp & Paper)**
  - **Containment Shell**
- **Chevron Research and Technology Company (Refinery)**
  - **Furnace Tube Hangers**

Requirements of Applications Being Pursued

Application	Temp	Environment	Life
Combustor Liner	2150°F	O_2, CO, H_2O, NOx	30,000 Hours
Rim Seal	1500°F	O_2, CO, H_2O	30,000 Hours
Tipshoe	2150°F	O_2, CO, H_2O	30,000 Hours
Furnace Tube Hanger	1800 - 2000°F	N_2, O_2, CO, H_2 CO_2, SO_2	25,000 Hours
Containment Shell for Chemical Pump	70 - 800°F	Organic and Inorganic Chemicals	30,000 Hours

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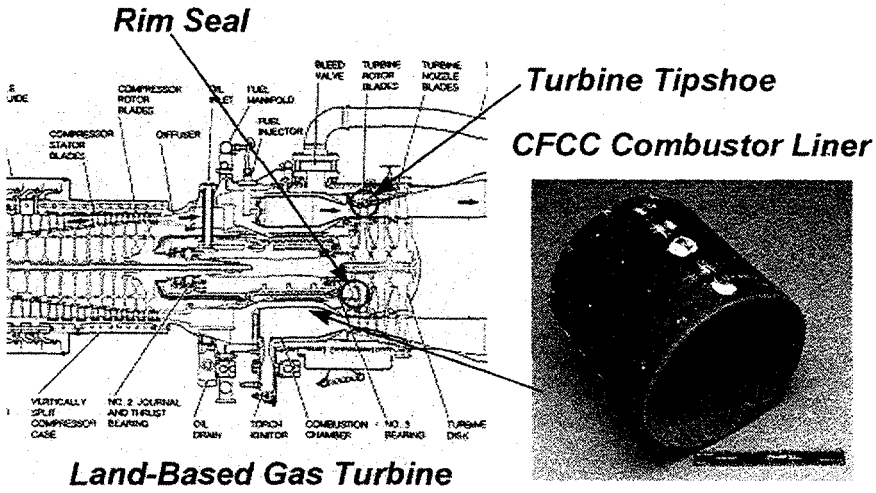
CFCC Components for Gas Turbines



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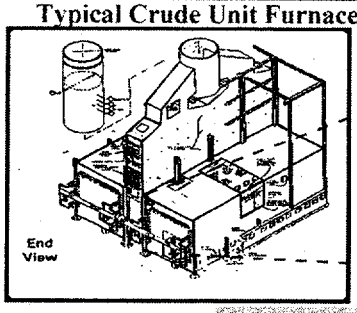
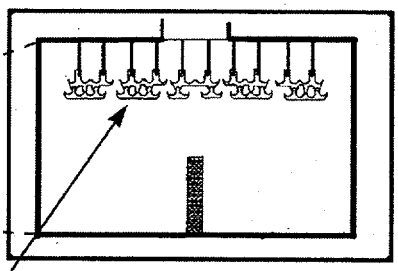
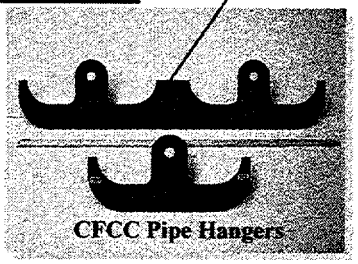
Furnace Pipe Hangers for Refinery Industries

Typical Crude Unit Furnace



Pipe Hanger Position in Furnace





CFCC Pipe Hangers

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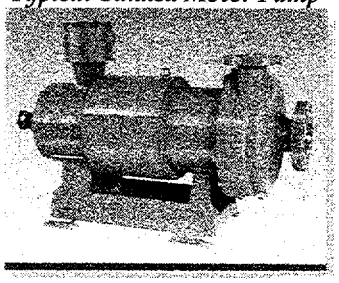
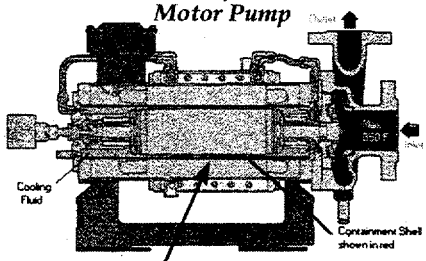
Containment Shell for Canned Motor Pump

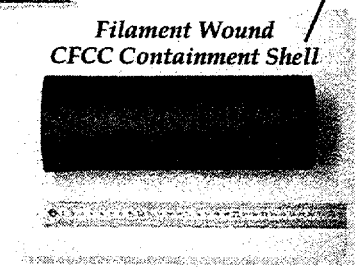
Typical Canned Motor Pump



Schematic of a Canned Motor Pump



Filament Wound CFCC Containment Shell



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SYLRAMIC™ SiC Fiber Reinforced CFCC

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Improved PIP Composite System

Material:

- **Reinforcement** - ***SYLRAMIC™* SiC Fiber, 5 HS weave**
- **Matrix** - **Polysilazane Derived SiNC**
  - **Si_3N_4/SiC Filler**
- **Interface** - **Boron Based**
- **Process** - **Polymer - Impregnation-Pyrolysis**
  - **Standard Temperature**
  - **High Temperature**

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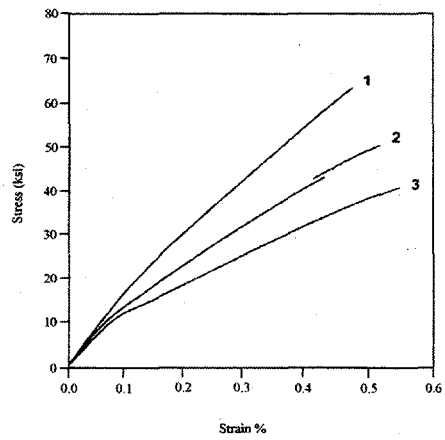
Comparison of Stress/Strain Curves for CMCs Fabricated with Various Fiber Reinforcements

1 - *SYLRAMIC™* SiC CMC
Si_3N_4 Filler/SiNC Matrix

2 - Hi-NICALON CMC
SiC Filler/SiNC Matrix

3 - CG NICALON CMC
SiC Filler/SiNC Matrix



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*SYLRAMIC™ SiC FiberReinforced CFCC Test Plan**

Tensile Strength	**- at RT, 815° C, 1200° C**
	- 815° C/1000h, 1200° C/1000h oxidation
Shear Strength	**- RT after 815° C/1000h,1200° C/1000h oxidation**
Tensile Fatigue	**- S/N curve 815° C, 1200° C**
Tensile Creep	**- 815° C, 1200° C**
Thermal Conductivity	**- RT to 1200° C**
Thermal Expansion	**- RT to 1200° C**

***Tests will be conducted for both high and STD temperature processed material**

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Tensile Properties of SYLRAMIC™ SiC Fiber Reinforced SYLRAMIC S200 CFCC

Process	Temperature (°C)	# of Specimens	Modulus (msi)	Prop. Limit (ksi)	Ultimate Strength (ksi)	Strain (%)
Low Temperature	20	2	21.9	18.6	52.5	0.34
	815	4	21.8	16.0	43.5	0.31
	1200	4	22.0	14.8	41.8	0.31
High Temperature	25	3	23.1	20.2	54.3	0.34
	815	4	22.0	18.0	48.0	0.35
	1200	4	22.0	13.5	40.8	0.31

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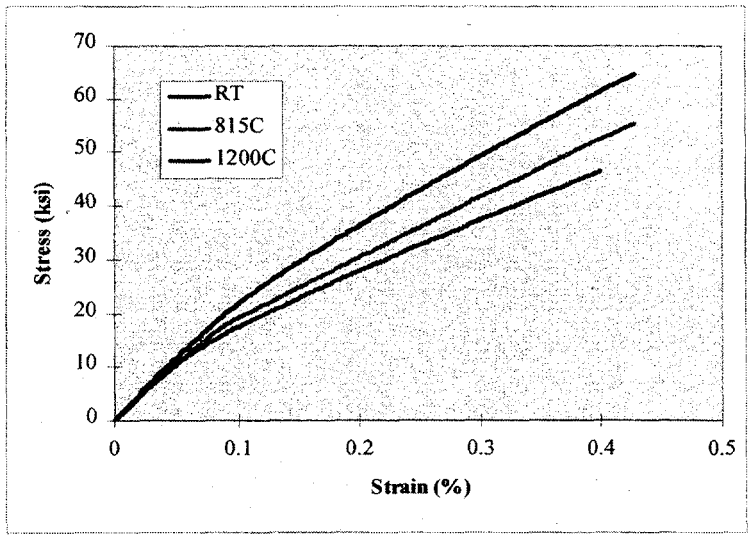
Tensile Properties of SYLRAMIC™ SiC Fiber Reinforced SYLRAMIC S200 CFCC



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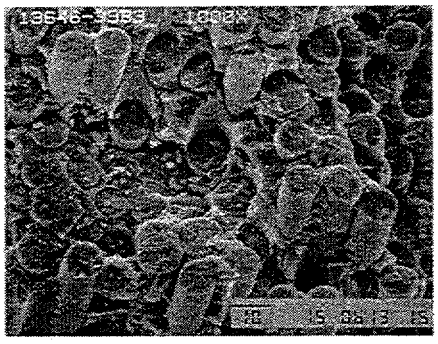
SEM Micrographs of Room Temperature Fracture Surface



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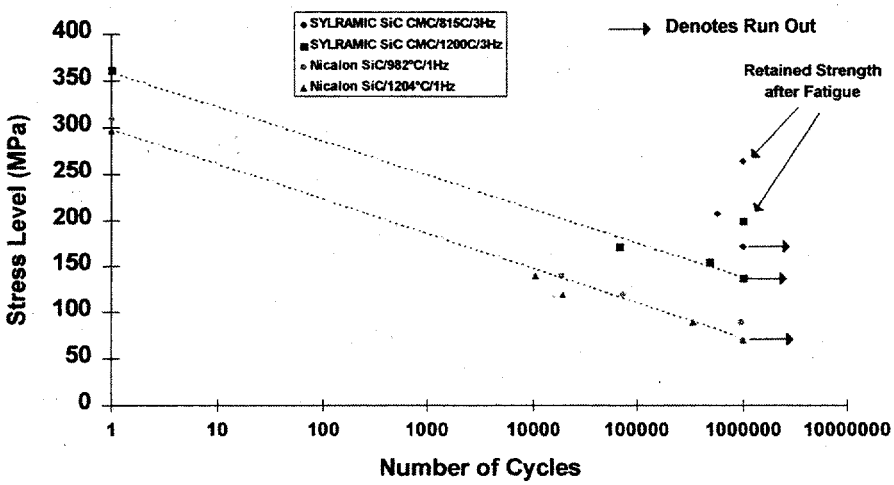
Comparison of Fatigue Data for SYLRAMIC™ SiC Fiber and Nicalon Fiber Reinforced CFCC



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Benefits of SYLRAMIC™ SiC Fiber in SYLRAMIC S200 CFCC

- **Enhanced thermal stability of entire system**
- **Can use higher processing temperature**
- **Higher proportional limit**
- **Environmental resistance**
- **Creep resistance**
- **Thermal conductivity**

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Environmental Testing

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Environmental Exposure of SYLRAMIC™ S200

Objective:

- **Evaluate composite performance under simulated combustor, dry air and inert conditions**

Applications:

- **Solar Turbine Combuster Liner/ Solar Turbines Seal**

Evaluation Temperatures:

- Combuster & Tipshoe - 2100°F
- Turbine Seal - 1500°F

Atmosphere:

- **Simulated combustion atmosphere:**

15 wt% Water	**13.75 wt% Oxygen**
4.75 wt% Carbon Dioxide	**66.5 wt% Nitrogen**

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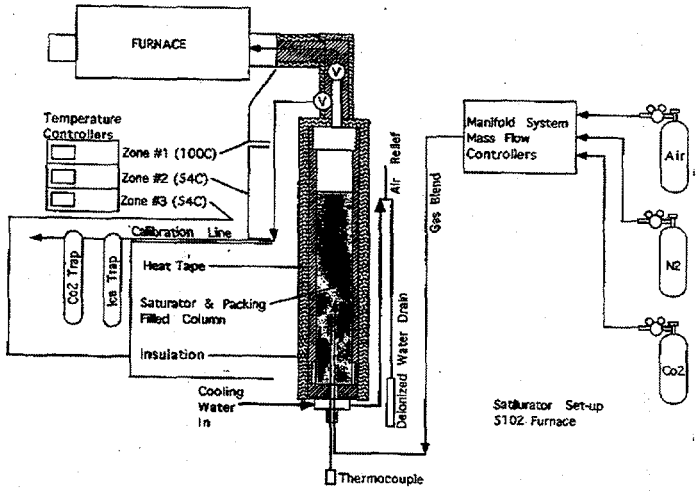
Environmental Exposure Test Matrix

Interlaminar Shear and Tensile Properties Evaluated

Environment	Temperature (F)	Static	Fatigued
Nitrogen	1500	Y	N
Nitrogen	2100	Y	N
Dry Air	1500	Y	Y
Dry Air	2100	Y	Y
Combustion	1500	Y	Y
Combustion	2100	Y	Y

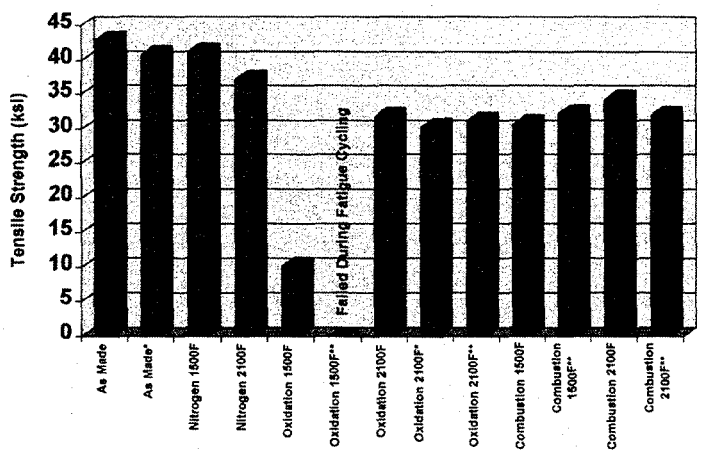
22 10/28/97

Environmental Exposure Test Equipment



23 10/28/97

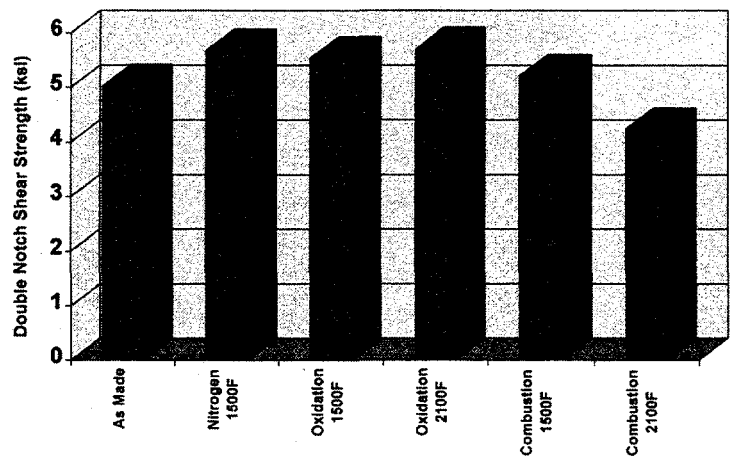
Effect of 1000 Hour Environmental Exposure on Tensile Properties



**** Fatigued to 11 ksi for a total of 4000 cycles prior to testing***
***** Fatigued to 11 ksi for 1000 cycles every 250 hours for a total of 4000 cycles prior to testing***

24 10/28/97

Effect of 1000 Hour Environmental Exposure on Shear Strength



25 10/28/97

Summary

- ***Mechanical properties of CG Nicalon CFCC stable at elevated temperatures in simulated combustion environments***
  - ***Low temperature oxidation embrittlement***
  - ***Improved performance in combustion environment***
  - ***Shear strength not effected by environments evaluated***

- ***Significant improvement demonstrated in thermo-mechanical properties of CFCC using SYLRAMIC™ SiC fiber***
  - ***Higher temperature processing***
  - ***90% improvement in microcrack yield point***
  - ***Improved low cycle fatigue performance***

26 10/28/97

Toughened Silcomp (SiC-Si) Composites for Gas Turbine Engine Applications

K.L. Luthra and G.S. Corman
General Electric Company
Schenectady, NY 12301

Principal Investigator: Krishan Luthra
Contract Monitor: Joe Mavec
DOE Contract No.: DE-FC02-92CE41000

GE Corporate Research and Development
GE Power Generation

Outline

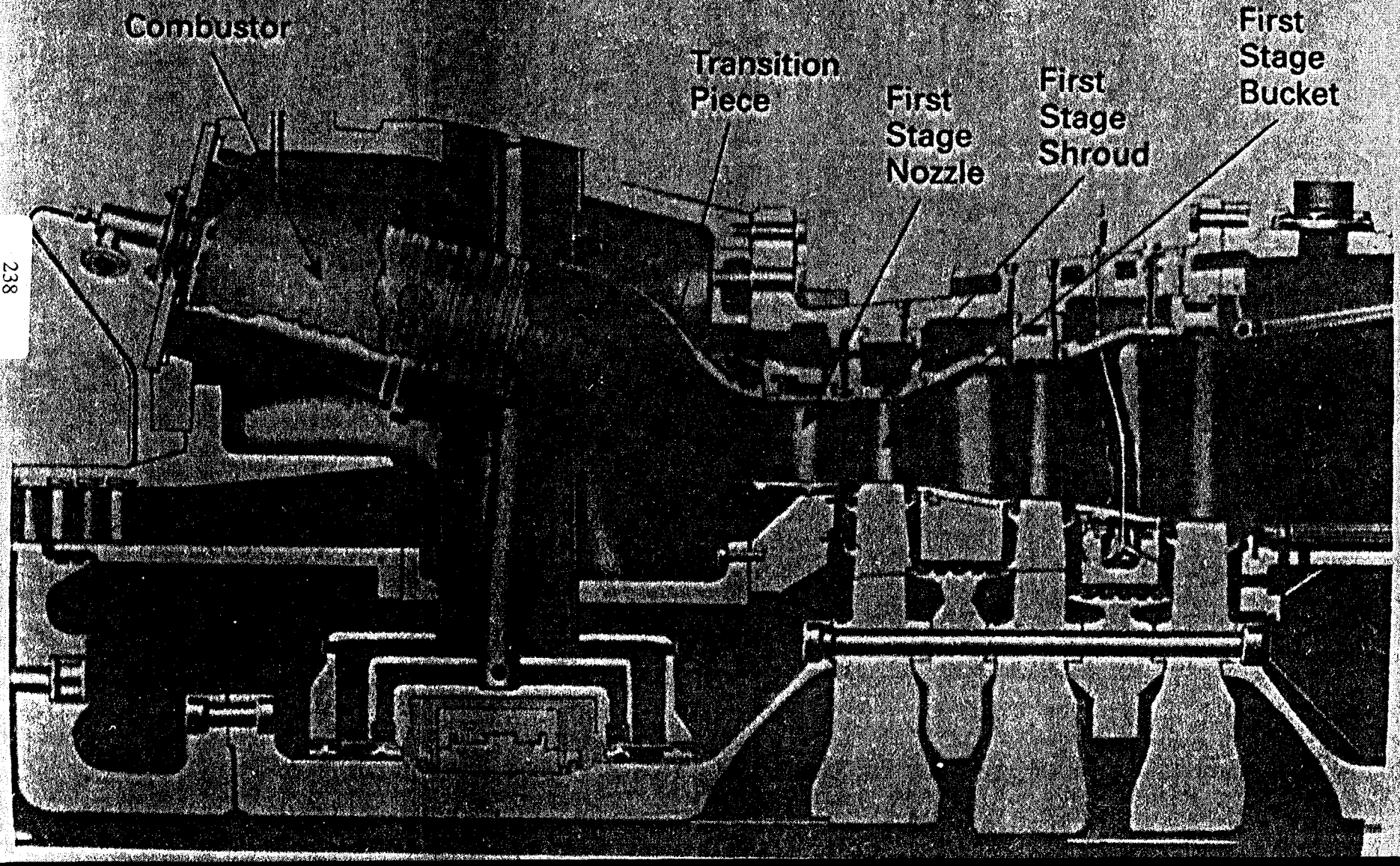
- Introduction
  - Objective
  - Material System
- Application
- Accomplishments
  - Material Properties
  - Component Design/Fabrication
- Rig Testing Plans
- Summary

Objectives

- Develop SiC fiber reinforced SiC-Si matrix composites prepared by melt infiltration for their use in hot sections of industrial gas turbines
  - focus on combustor and first stage shroud

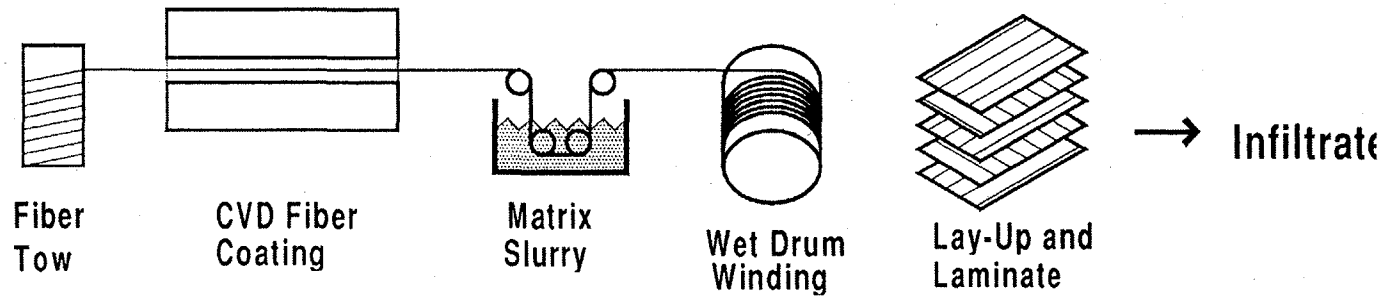
GE LARGE UTILITY GAS TURBINE

Hot Stage Components

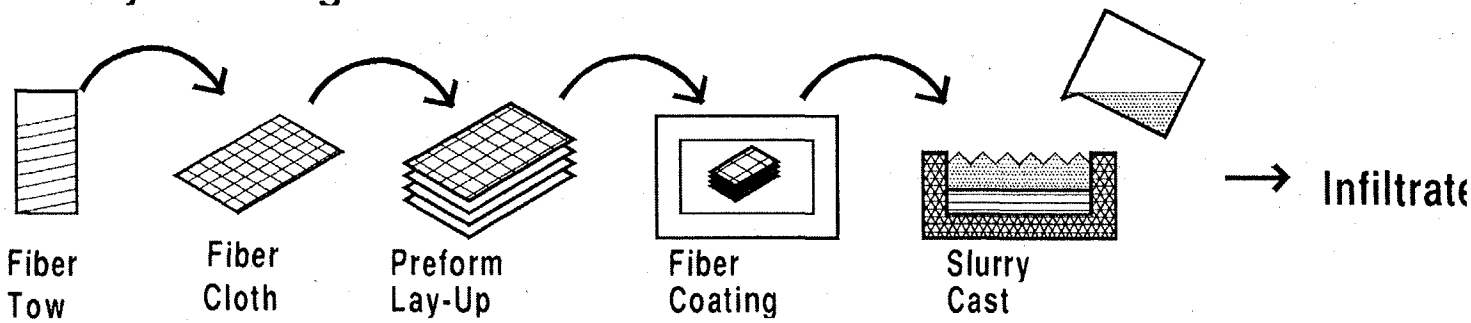


Fiber Reinforced SiC-Si Composites

Prepreg



Slurry Casting



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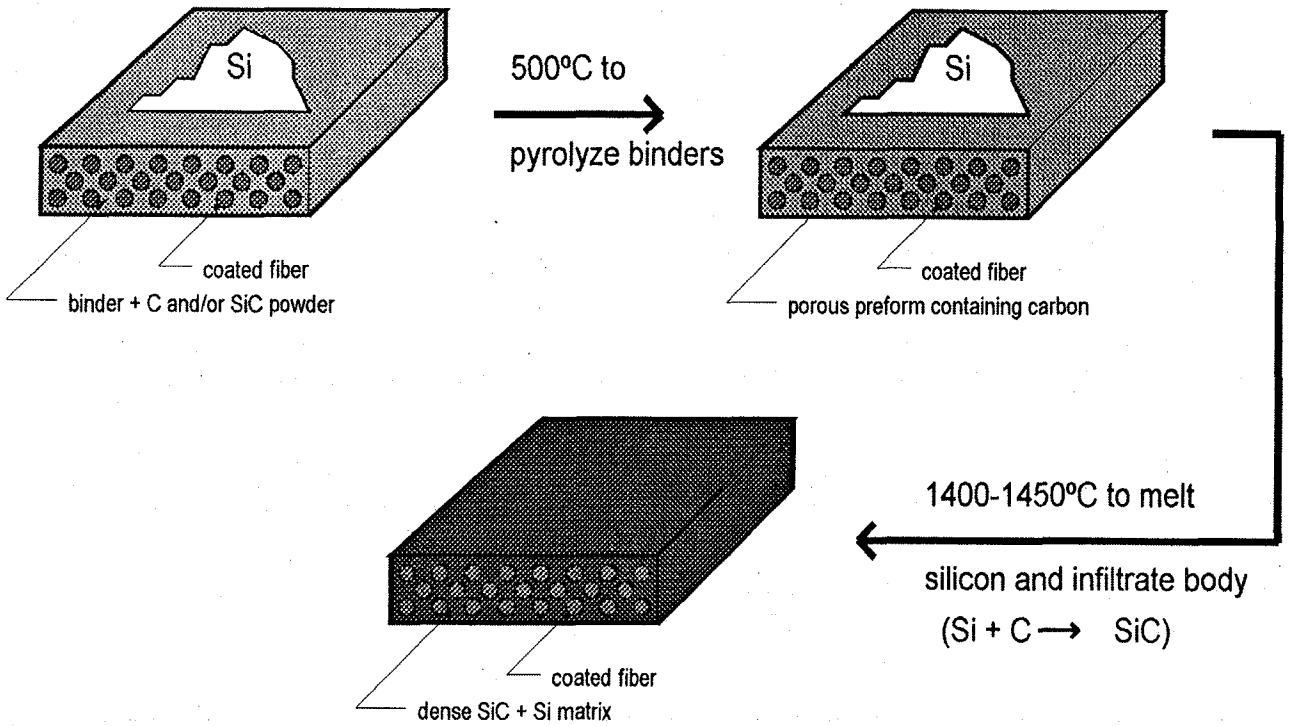
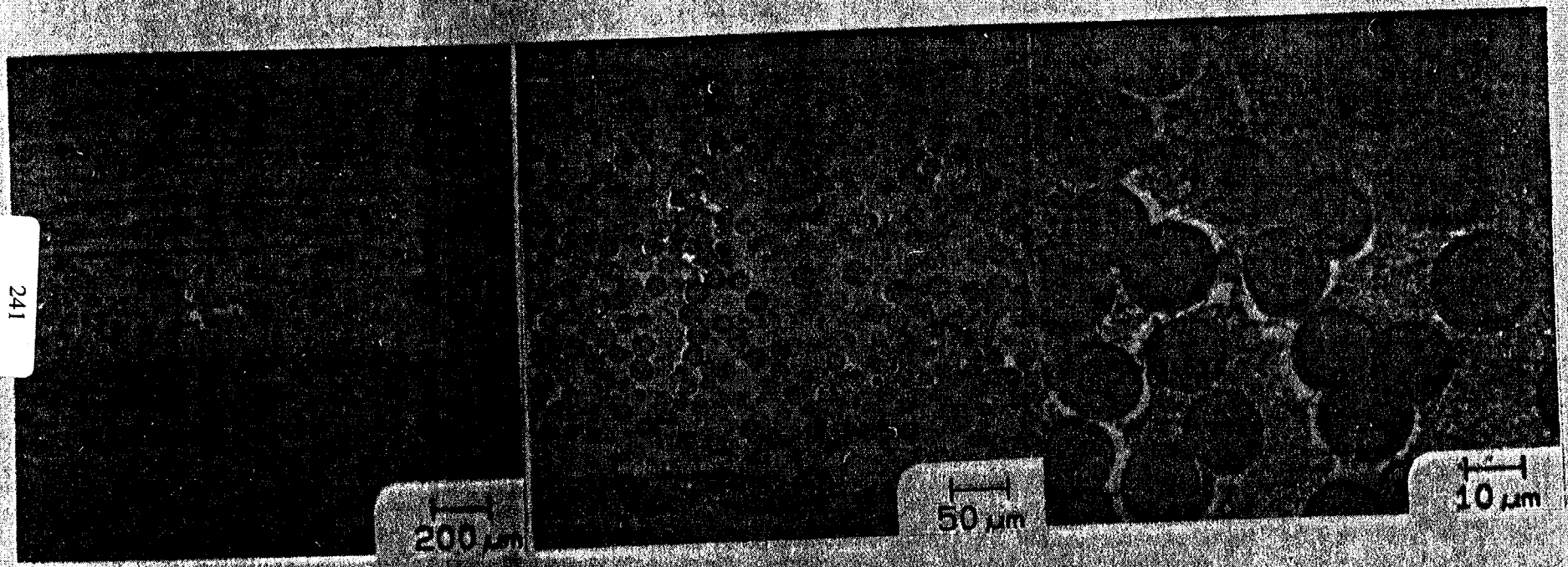
SiC Fiber Reinforced SiC-Si Composites by Melt Infiltration



Near-net, complex shape process yielding fully dense composites

Hi Nicalon Reinforced Composites

Advanced Ceramics Coatings



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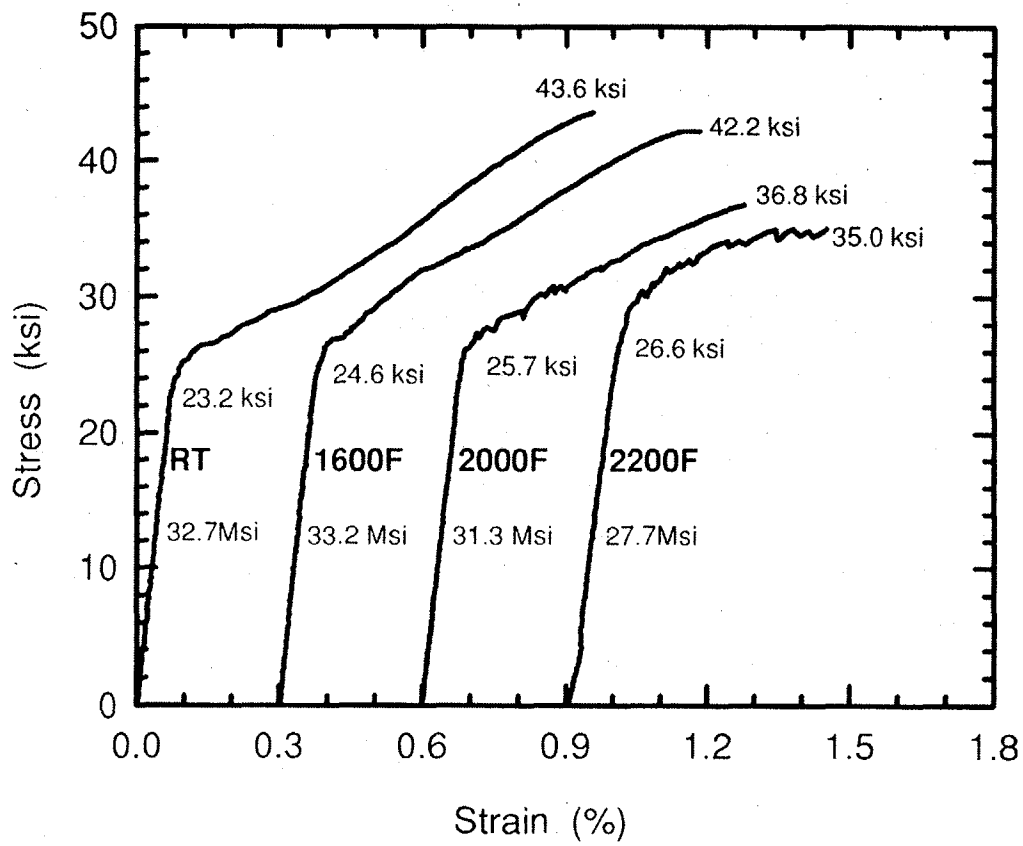
Recent CFCC Accomplishments

- Excellent mechanical properties observed on Hi-Nicalon fiber reinforced composites using fiber coatings from vendors
- Actively investigating oxidation issues following matrix cracking and foreign object damage issues
- Fabricated representative shrouds using SCS monofilaments and Hi Nicalon fibers
- Developed a new shroud design with reduced stresses and better manufacturability

Hi-Nicalon Fiber Reinforced Composites

- Tensile Behavior with Hi-Nicalon and Advanced Ceramics fiber coatings
  - Matrix cracking stress exceeds 20ksi (140MPa)
  - Matrix cracking stress and cracking strain increase with temperature
  - Composite modulus can be controlled by matrix composition
- Thermal stability demonstrated to 500 hours
  - 2000 hour exposure in process
- High thermal conductivity (exceeds 15 W/mK at temperatures up to 1200°C)
- High interlaminar properties
  - Interlaminar shear strength of 6.5 to 8.0 ksi at room temperature; 8.1 to 11.2 ksi at 1093ºC
  - Interlaminar tensile strength of 2.5 ksi at room temperature

Hi Nicalon Reinforced Composites



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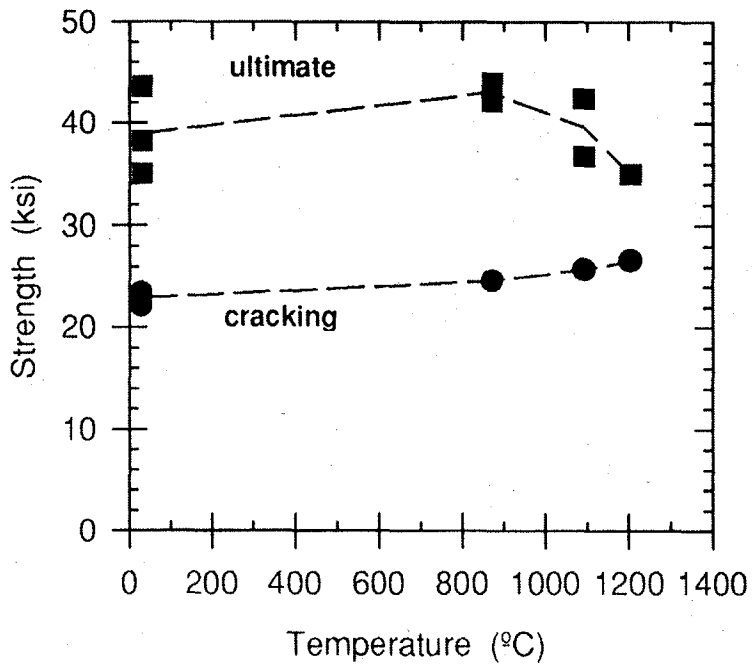
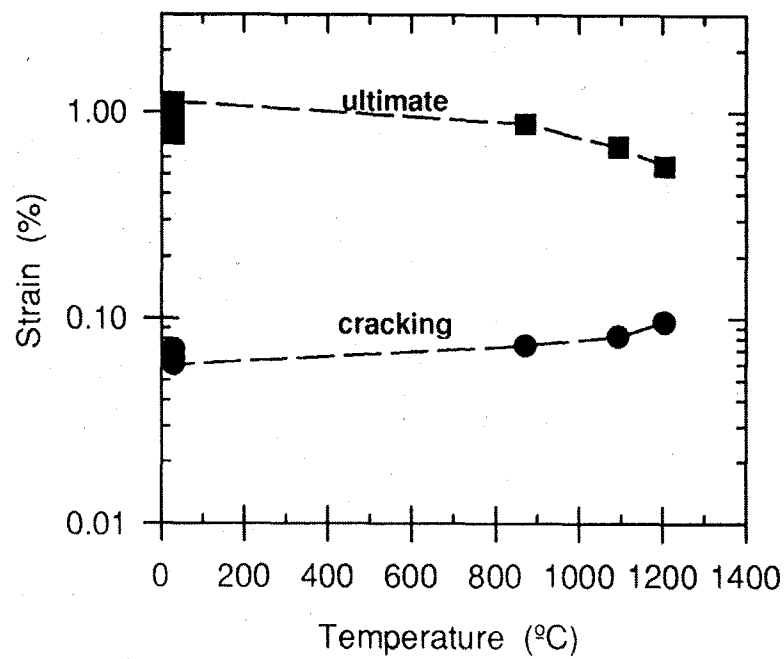
Hi Nicalon Reinforced Composites





~25 vol % 8-ply 0/90 composites

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Hi Nicalon Reinforced Composites

	RT		2000°F	
	Matrix A	Matrix B	Matrix A	Matrix B
Proportional Limit, ksi	23.2	16.1	25.7	19.5
%	0.071	0.073	0.082	0.094
Modulus, Msi	32.7	22.0	31.3	20.7

Modulus can be decreased
without decreasing proportional limit (design) strain

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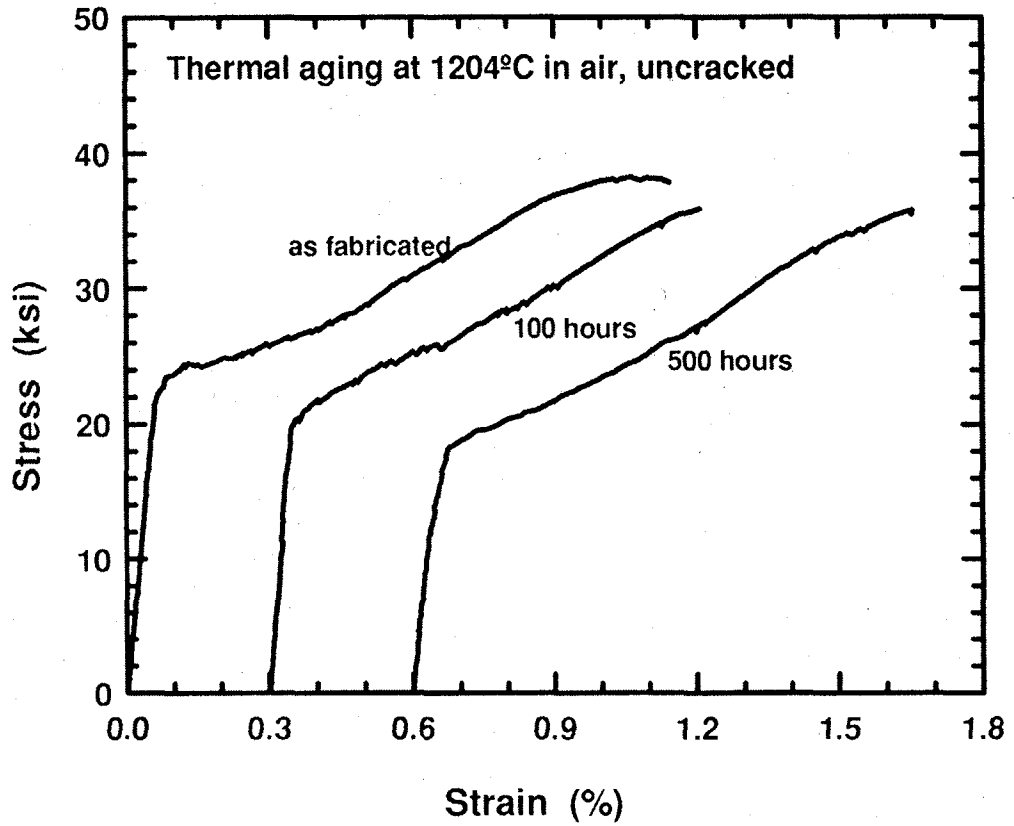
Thermal Stability



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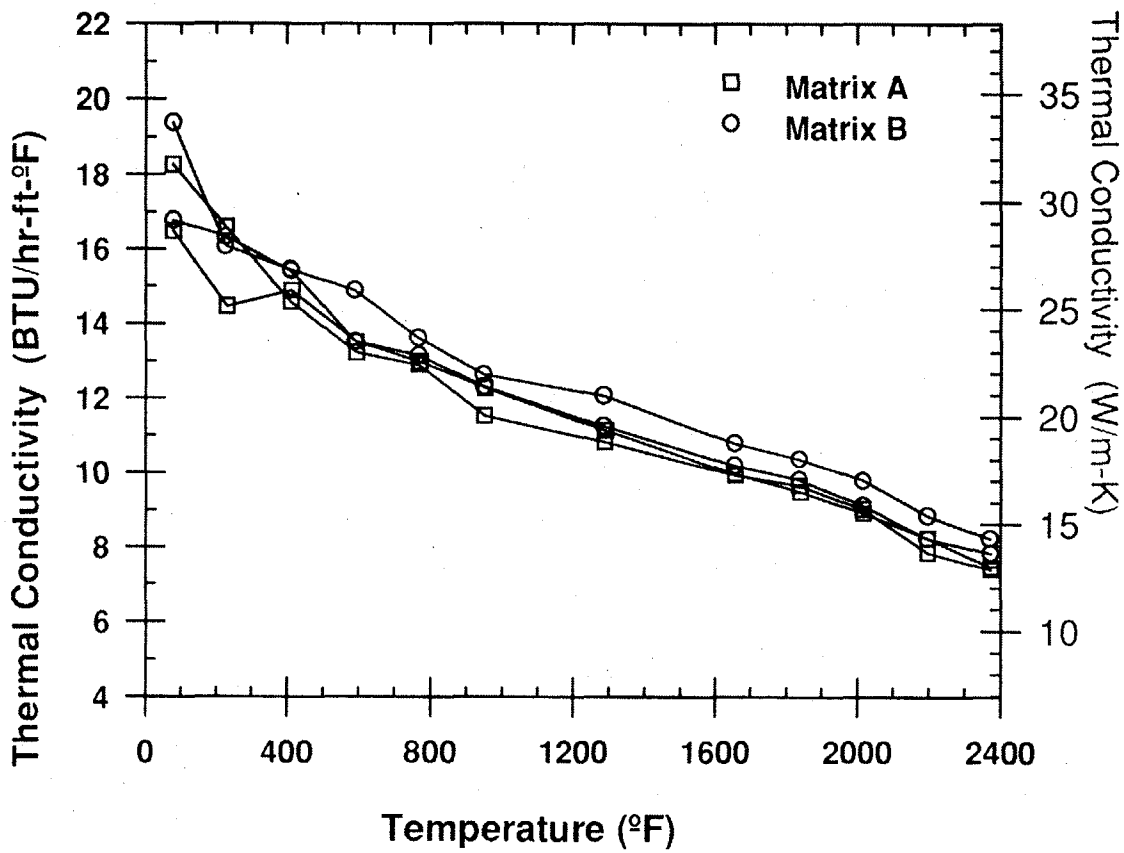
Thermal Conductivity



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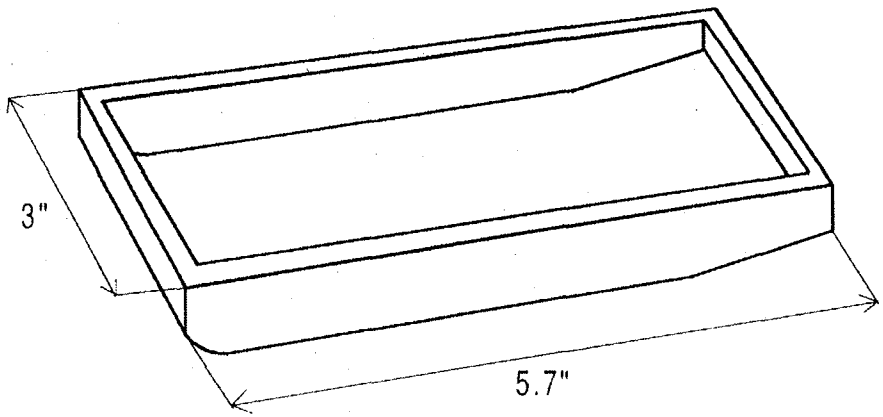
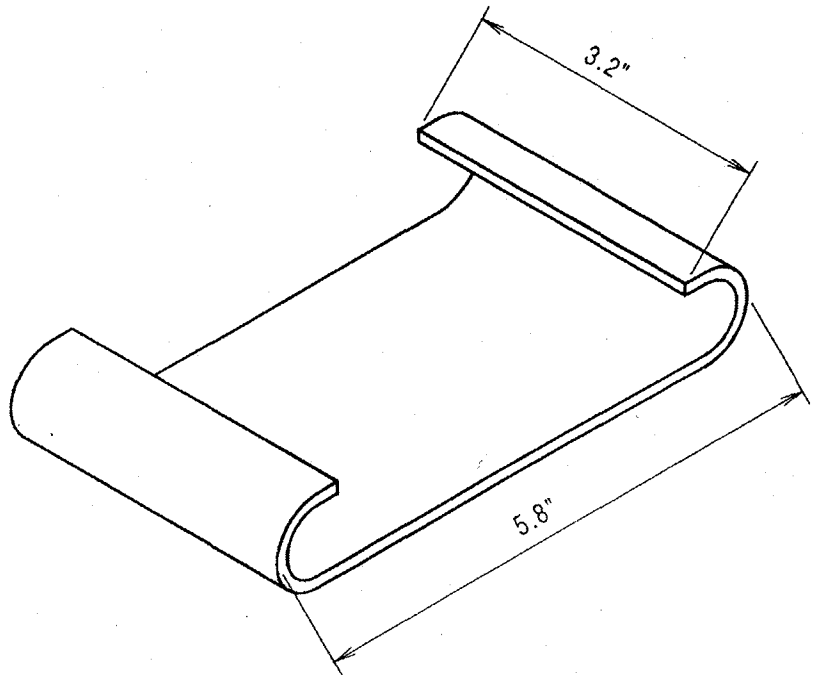
Shroud Design

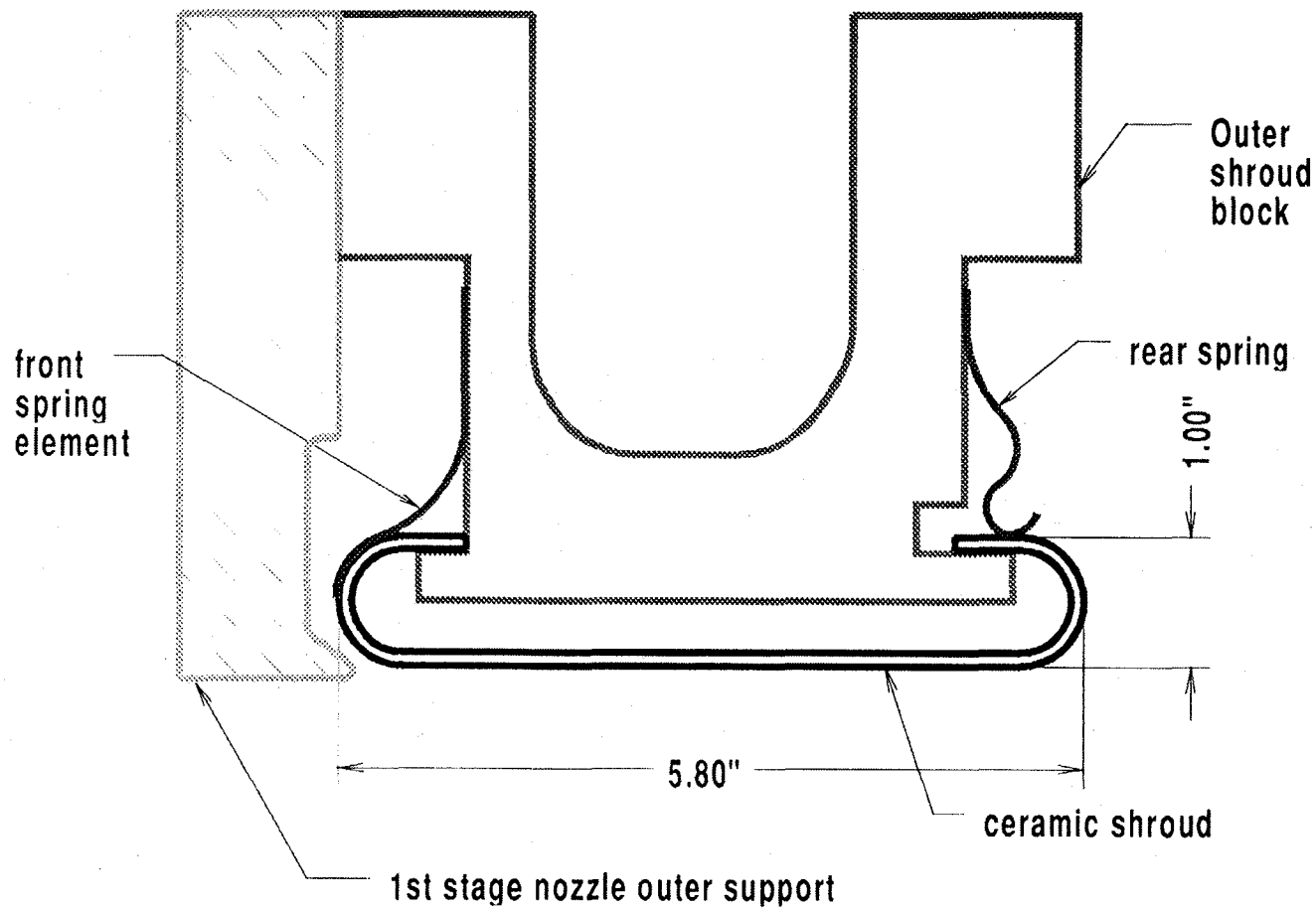


Phase 1 Design



Phase 2 Design

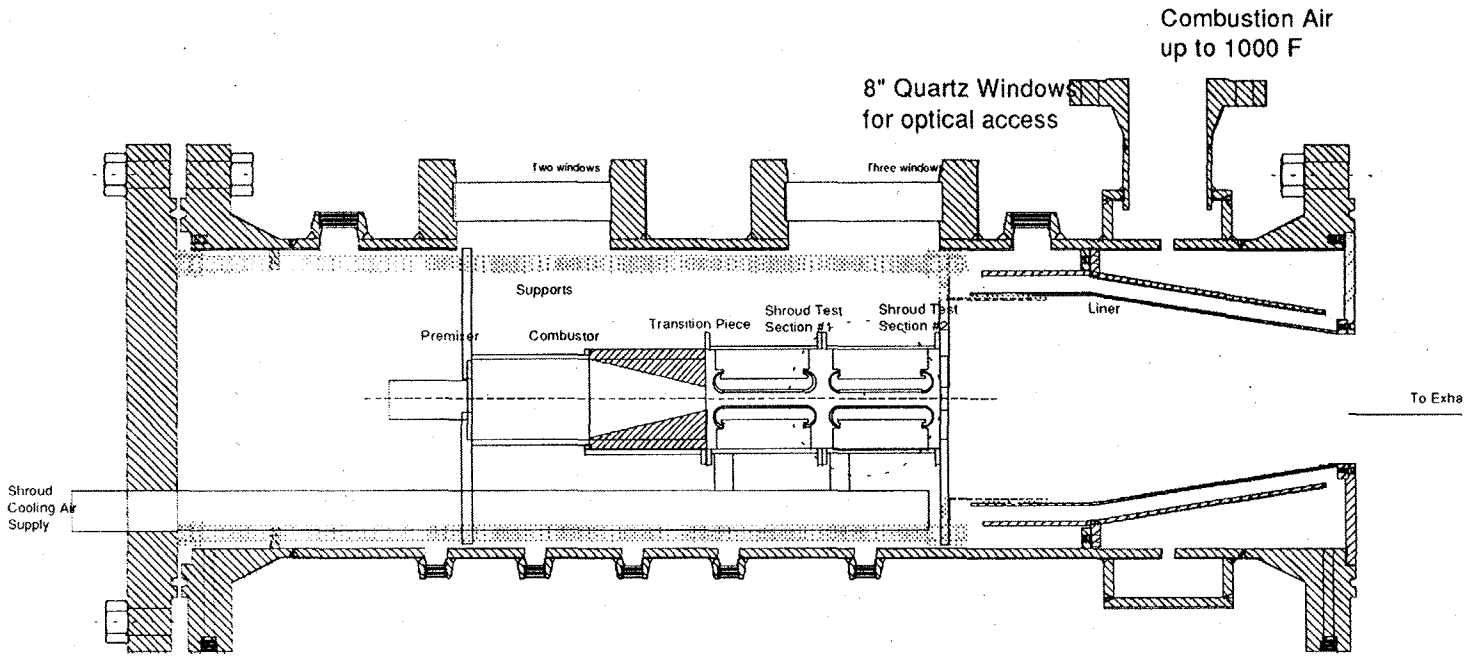
Shroud Design



Combustion Rig Testing

Layout of Ceramic Test Rig in 16" diameter vessel

- Two sets of windows permit viewing of combustor and shroud elements
- The test section is supported off the upstream flange assembly
- It is very easy to access the hardware for changes between tests



Tony Dean
August 1, 1997

GE Corporate Research and Development
GE Power Generation

Summary

- Toughened Silcomp (Melt Infiltration) composites are attractive candidates for industrial gas turbine applications
  - fully dense
  - high proportional limit
  - tailorable properties
  - high thermal conductivity
  - near net, complex shape capability
- Current program focus is on SiC Hi Nicalon reinforced composites
- Long life requirement, particularly after matrix cracking, is expected to be the biggest technical challenge
- Representative shroud & combustor component tests are being planned
- Phase II expected to be completed by December 1998

DuPont Lanxide Composites Inc.
CFCC Program for
CVI Materials

Program Review

Oct 1997

Phillip A. Craig, Program Manager

Ms. Jill Jonkouski - DOE Project Manager

CFCC CVI Materials Program

- Primary material studied is Enhanced SiCf/SiC
- "Enhanced SiC/SiC" is DLC's proprietary version of SiCf/SiC produced by CVI (Chemical Vapor Infiltration).
  - Ceramic Grade Nicalon™ silicon carbide fiber
  - Carbon, or Boron Nitride interface, applied by CVI
  - Silicon Carbide matrix by CVI.
  - DLC External Oxidation Protection coatings.
- Process licensed from SEP, France, by DuPont in1988, joined DuPont Lanxide Composites in 1992.
- E-SiC/SiC has been on a continual path of organized improvements for oxidation protection.

CFCC Industrial Applications

- Foster Wheeler Development Corporation: Tubes and Headers
  - Very strong corporate committment at FWDC.
  - Hot Air Heater and Steam Reformer identified as examples.
  - Coal Ash Exposure - the initial application test.
  - Penn State - Testing of tube permeability
- Alzeta Radiant Burner Screens
  - Single ply of 50% open area doubles radiant output with same fuel input.
  - Single unit tests now lead to field trials.
  - Commercial product expected in 12-18 months.

CFCC Turbine Engine Applications

- Solar Turbines' Industrial Turbine Engine Combustor
  - Corporate committment to evaluate many CFCC's.
  - Test Rig for exposure to combustion gases.
  - "Saturn" Engine Test to investigate retrofit Lox NOx opportunity.
  - Support "Centaur" Engine CSGT and ATS programs.
- General Electric Utility Turbine Engine Shroud
  - Shrouds for power generation - 30,000 hour life.
  - Pressurized rig being set up to test parts
  - Oversight by engine designers assures relevance.

Material Specific Successes

- Resin Derived Interface Proven
  - 1/10 th cost of CVI interface.
  - 2/3 tensile strength of CVI interface, maintains proportional limit.
  - Alzeta radiant burner screens show same and perhaps better thermal cycling performance
- Hi-Nicalon™ fiber substitution
  - Significantly longer lifetime, stronger, than CG Nicalon.
  - Adopted for use in Solar Centaur; and GE Shroud Programs.
  - Improved stability of interface provides longer lifetime.

NDE Specific Successes with Argonne Nat'l Lab

- Non Destructive Evaluation for Life Prediction
  - Observe delamination of Solar 8" liner from fabrication, through 100 hours of testing.
  - Developing life prediction with detail flaw mapping.
- Improved processing via NDE
  - Correlations of thermal diffusivity to density.
  - Improvements with reduced data scatter can seperate edge affects from bulk properties
  - Enables decisions for processing; which improves life; with density correlation to modulus and lifetime.
- Verified capability of DLC basic NDE capabilities.
  - DLC film x-ray and water coupled ultrasonics, not as fine resolution, basically correlate to detailed ANL flash thermography and air coupled ultrasonics.

Joining Specific Successes

- Joining work with silicon melt infiltration processes.
  - Work with INEL, Barry Rabin, has proven high strength joints at RT and HT.
  - Similar process as NASA Lewis, Jay Singh, has lower temp tooling requirements, will help in complex shapes.
  - Optimization, characterization, complex shapes, and size scale-up required, planned for 1998.
- Joining work with preceramic polymer
  - DLC has obtained 1100°C joints with Ceraset™
  - AMES Lab, work on atmospheric fabrication may help field joining.
- More detail in Wednesday talk by Patterson.

Application Specific Successes - Alzeta

- Alzeta Radiant Burner Screens
  - 2100° F face temperatures, natural gas. CFCC screens enables 100-130 kW/sq m heat flux.
  - 10,000 thermal cycles with CVI Interface - No failure
  - 32,000 thermal cycles with RDI Interface - No failure
  - Attachment options were investigated, optimized.
- New Scale-up Work started to optimize RDI process.
- New Field Testing
  - Materials test company
  - Glass manufacturer
- Industries of the Future will be a major thrust for additional applications.

CVI SiCf/SiC Situation Today

- Commercially Sound Material
  - Performance and Cost follow Fiber selection.
  - Turbine Engine and Industrial applications are proceeding successfully.
- Long Life Trials - Several 4000 hour datapoints.
- Manufacturing Reactors Exist
  - Volume of material processed increases, costs decrease.
- E- SiCf/SiC part of DLC's Corporate Strategy
  - DLC is a manufacturing company.
  - DLC expects to produce several types of CMC's, and several families of materials (MMC, CMC, Polymers, etc).
  - E-SiCf/SiC provides solutions over competitive std SiC/SiC. Product differentiation is key to success.

Solar Turbines - Specific Successes

- Solar Turbines Combustor
  - 8"dia x8" subelements used in combustor rig
  - 100 + hours testing to 2100° F
  - Liner had fabrication damage (delamination); and test demonstrated damage tolerance of material. (Preform method improved to eliminate flaws).
  - Leading to development of other combustors for Solar's ATS and newly developed engines.
- Solar investigating fuel burner and CFCC combustor match that allows Low NOx system retrofit for older engines.
- Materials work on Hi-Nicalon/E-SiC/SiC feeding CSGT program for Centaur engines.

Application Specific Successes
Foster Wheeler Development Corporation

- Foster Wheeler Coal Ash Exposures
  - Coal Ash Lab furnace at FWDC verifies 2100 F capability.
  - Pressurized tests at ORNL verifies E-SiC/SiC improved capability versus standard SiC/SiC.
  - Field Boiler Exposures (TVA Gallatin Station) verify 4000 hour durability (1750 F).
- Penn State Tube testing successful
  - Helium pressure to 700 psig at 2000 F successful.
  - Burst pressure of 2600 psi, good factor of safety.
- Vortex Finder Application
  - Wall of particle separator for fluidized bed combustor @1500-1800 F.
  - In field test at Rollins Plant (municiple solid waste) ,Chicago, IL

Program Path Forward

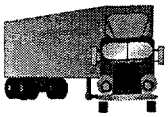
- Materials Work:
  - Process optimization for burner screen materials
    - » Resin derived interface scale-up
    - » Demonstrate other shapes of parts.
  - Continue work on coatings needs and benefits
    - » For coal ash long lifetime exposure
    - » For combustion atmospheres.
  - Continue application specific data on Hi-Nicalon CFCC's.
- Joining Work:
  - Demonstrate unifomrity in scale-up sizes to 8" dia.
  - Test for coal ash exposure resistance.
  - Develop design database.

Conclusions

- CFCC Program has clearly advanced the state of industrial readiness of Enhanced SICf/SiCmaterials.
  - In material science,
  - In processability,
  - In reducing barriers to entry for industrial applications.
- Most of the tasks planned in the original Phase 2 are complete.
- Work planned to meet the new goals of application specific testing will require 2-3 years of additional effort.
- Continued work in CVI-CFCC Program provides positive assistance to applications and material science, with a strong commercial future.

CATERPILLAR®

Self-Lubricating CFCCs For Diesel Engine Applications



M H Haselkorn
Caterpillar Inc.

Objectives of 3500 Natural Gas Engine Test

- Evaluate CFCC Valve Guide Attachment Method
- Determine If Engine Test Data matches Laboratory (Cameron-Plint) and Engine Rig Test (Battelle) Data

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CFCCs For Diesel Engine Valve Guides

- Silicon Nitride to Provide High Temperature Structural Support and Wear Resistance
- Carbon Fibers to Provide Solid Lubrication

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CFCC Valve Guide Attachment Measurement Results

- Some CFCC Valve Guide ODs were smaller than print specifications
- Some of the bores in cylinder head were larger than print specifications
- OD of Cast Iron Guides were oversized -- 0.2 mm above print <u>maximum</u> OD specification

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3500 Natural Gas Engine Test Results

- Test Stopped After 224 Hours because of a Facility Problem
- CFCC and Monolithic Silicon Nitride Guides Moved During Test
  - 7 CFCC Guides were pushed out 10-11 mm
  - 3 CFCC Guides were hammered into cylinder head
  - 1 Monolithic Silicon Nitride Guide was pushed out 10 mm

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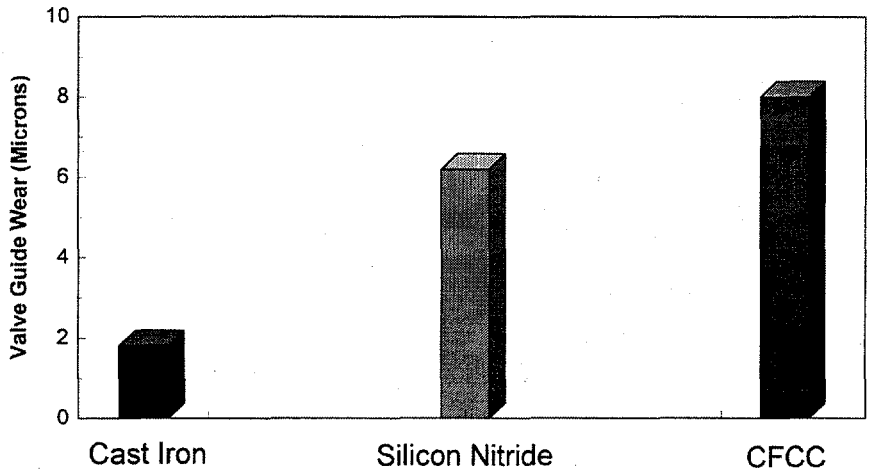
CFCC Valve Guide Attachment

- The following measurements were made to determine the reason for the valve guide movement
  - OD and ID of CFCC, silicon nitride, and cast iron guides
  - OD of valve stems,
  - Valve guide bores in cylinder head

CATERPILLAR®

3500 Engine Test 1 -- Valve Guide Wear After 224 Hours



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3500 Engine Test 1 Valve Guide Wear

- Wear Of CFCC Valve Guide Higher Than Cast Iron
  - Small amount of wear, difficult to measure accurately (difference 6 microns)
  - Break-in period longer for CFCC guide

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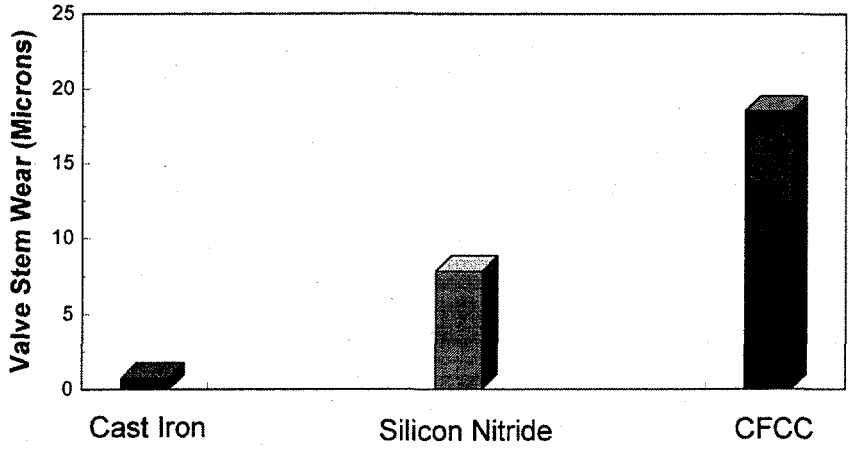
3500 Engine Test 1 -- Valve Stem Wear After 224 Hours



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3500 Engine Test 1 Valve Stem Wear

- Greater for CFCC Guide Than Cast Iron Guide
  - Short hour test with very little wear -- even on the stems which ran in CFCC guide
  - Length to Diameter Ratio of CFCC Guides make them more difficult to machine
  - Break-in period could be longer for the CFCC

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CFCC Valve Guide Attachment Conclusions

- Tolerance Stack-ups and Improper Machining of the CFCC ODs are the Reasons For the Valve Guides Moving

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CFCC Valve Guide Attachment - Corrective Action

- Measured all CFCC ODs and Cylinder head bores
- Matched Guide ODs to cylinder head bores to insure the proper press-fit
- After 8 hours no movement of CFCC valve guides

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3500 Natural Gas Engine Test 2

- Valve guides matched to cylinder head bores to ensure of proper press-fit
- Will Run for 500 hours
- 4 cylinders lubricated, 4 cylinder no lubrication (dry)
- Approximately 200 hours run

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Pre-NPI Process Requirements

- **Demonstrate Durability of CFCC Guides in Diesel Engine Environment (Performance, Life)**
  - Lifetime of Carbon Film
  - Guide and valve stem wear
- **Specify (Concurrent Preliminary Design)**
  - Dimensional and surface finish requirements
  - Mechanical properties of CFCC
  - Break-in procedure for CFCC guide (if required)

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Pre-NPI Process Requirements

- Identify and Demonstrate a cost-effective manufacturing process for producing reliable production quantities of CFCC guides
  - Manufacturing process development -- Allied Signal
  - Two-piece valve guide design

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Two-Piece CFCC Valve Guide Design

- Able to dry press component
  - high volume production process
- Reduced amount CFCC required
- ID machining smaller lengths

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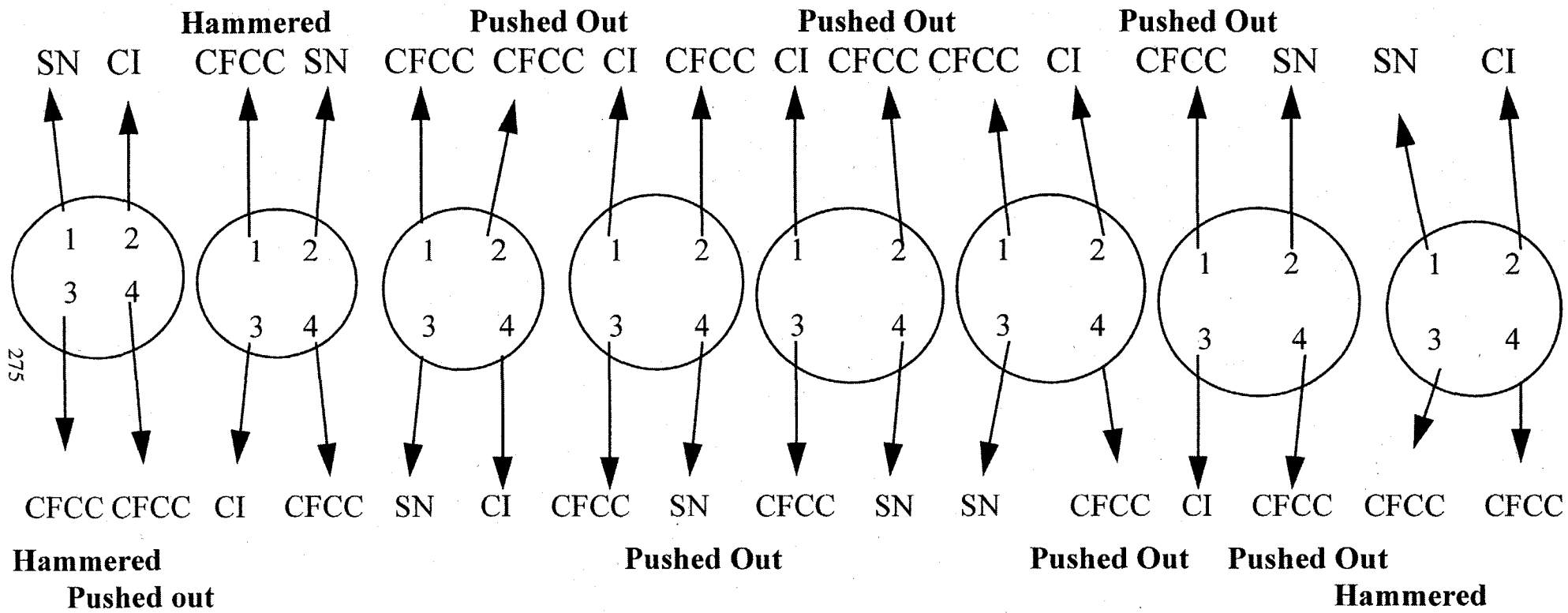
Valve Guide Configuration for 3508 CFCC Engine Test



SN = Monolithic Silicon Nitride guides

CFCC = CFCC Guides (10 volume Percent Carbon fibers)

CI = Cast iron guides (Baseline)

1,2,3,4 valve positions in cylinder

OVERVIEW OF THE CFCC PROGRAM'S SUPPORTING TECHNOLOGIES TASK

M. A. Karnitz, R. A. Lowden

Penny Humphreys, and S. A. North

Oak Ridge National Laboratory

and

M. Rawlins

DOE Oak Ridge Operations

Oak Ridge, Tennessee

THE SUPPORTING TECHNOLOGIES TASK PROVIDES ASSISTANCE IN MANY AREAS

- Administrative Functions
- Supporting Technologies R&D
- Industrial Interactions

ORNL PROVIDES ASSISTANCE TO THE DOE PROGRAM OFFICE, INDUSTRIAL PARTICIPANTS AND THE CERAMIC INDUSTRY

- Reporting & Communications
  - CFCC News
- Consultants and Program-Related Analyses
- Technical Coordination
  - CFCC Working Group Meetings
  - CFCC Steering Committee
- Coordination with Other Agencies and Organizations

TASK 2 EFFORTS PARALLEL THOSE OF THE INDUSTRIAL PROGRAMS

- Initial efforts focused on the more basic or generic aspects of CFCCs
- Techniques, facilities, and expertise are being utilized to assist the manufacturers in materials development and evaluation
- Current emphasis is on environmental effects and mechanical reliability
- As materials mature, and applications are better defined, the effects of in-service environments on performance and life will become priorities

COMPOSITE DESIGN HAS ADDRESSED THE DESIGN AND UTILIZATION OF COMPOSITES

Macromechanics - CLASS 2

Materials Science Corporation

MATERIALS CHARACTERIZATION IS PROVIDING DETAILED INFORMATION CONCERNING COMPOSITES, CONSTITUENTS, AND INTERACTIONS

Characterization -

Karren More, Oak Ridge National Laboratory

Interface Studies -

John Hellman, Pennsylvania State University

Sankar Sambasivan, Northwestern University

Rick Lowden, Oak Ridge National Laboratory

ACCEPTANCE OF, AND DESIGN WITH CFCCS WILL REQUIRE STANDARDIZED TEST METHODS

Thermomechanical Testing -

Michael Jenkins, University of Washington

Edgar Lara-Curzio, Oak Ridge National Laboratory

Non-Destructive Characterization

Bill Ellingson, Argonne National Laboratory

PERFORMANCE-RELATED PHENOMENA IS ADDRESSING APPLICATIONS-RELATED ISSUES

Performance Simulation -

Ken Reifsnider and Mike Pastor, Virginia Polytechnic Institute

Environmental Effects -

Pete Tortorelli, Oak Ridge National Laboratory

Mechanical Reliability -

H. T. Lin and Paul Becher, Oak Ridge National Laboratory

Thermal Shock -

Raj Singh, University of Cincinnati

WHAT ARE THE "PRODUCTS" OF THE SUPPORTING TECHNOLOGIES TASK?

- Computer Models and Codes
- Specialized Equipment and Facilities
- AdvancedTechniques and Procedures
- Data and Information
- Technical Reports and Publications
- Expertise and Experience
- Working Relationships: Teaming

THE DIRECTION OF TASK 2 CONTINUES TO BE "FLUID"

- Surveys of Industrial Needs and Requirements
- Steering Committee
- Working Group Meetings
- Program Management (DOE and ORNL)
- Task Review

HOWEVER, THE ORIGINAL FRAMEWORK HAS BEEN MAINTAINED TO PREVENT FRAGMENTATION, AND DILUTION OF EFFORT, AND TOTAL CHAOS

THE SUPPORTING TECHNOLOGIES TASK HAS SHIFTED EMPHASIS FROM GENERIC WORK TO DIRECT INDUSTRIAL SUPPORT

Current Industrial Interactions:

- AlliedSignal/Caterpillar
- Dow Corning
- DuPont Lanxide Composites
- General Electric
- McDermott Technology, Inc.
- Textron
- Hot Gas Filter Evaluation for the Chemical Industry

SUMMARY

- Supporting Technologies has evolved in response to the needs of the CFCC Program industrial participants
- Efforts are becoming less basic or generic and more "user" oriented
- Task 2 has many "products" that are available and ready for market, at a very reasonable price
- Techniques for the characterization and evaluation of CFCCs have been developed and optimized
- Facilities and equipment have been constructed
- Experience has yielded expertise

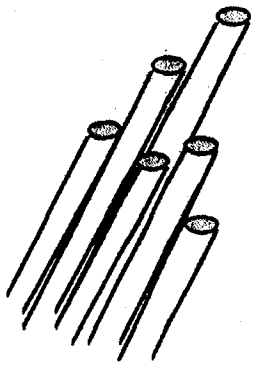
FUTURE

- Supporting Technologies will continue to assist the program office and serve the needs of the industrial participants
- Tasks will continue to become more "user" oriented, and industrial interactions expanded
- Supporting Technologies will become more involved in application-specific testing, joining, and other similar efforts
- Annual reviews will be held to gain input from industry and others from outside the Task

Virginia Tech

Life Prediction Methodologies for CFCC Components

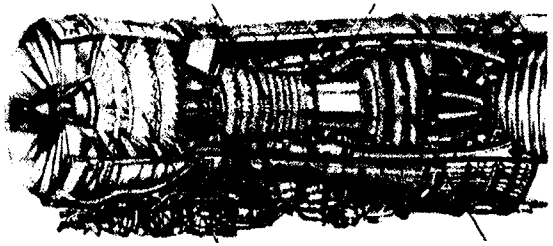
Materials Response Group
Virginia Tech



High Temperature Research Programs in the Materials Response Group at Virginia Tech

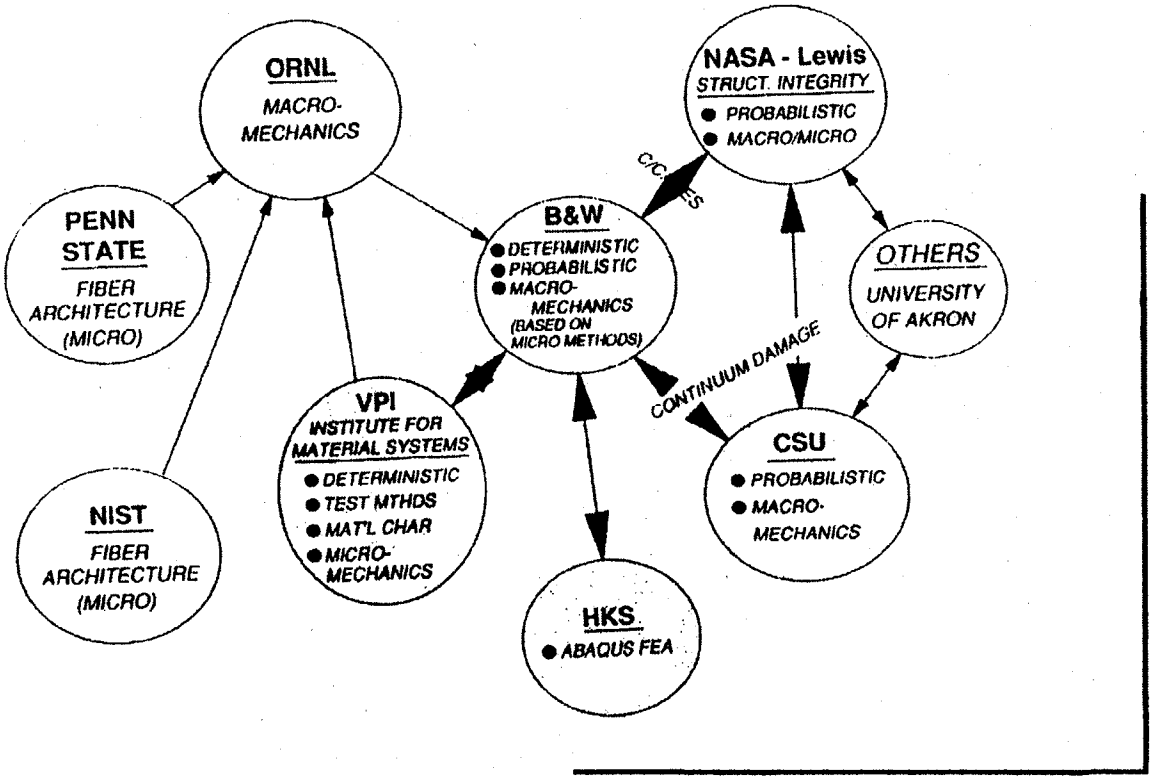
Principal thrusts / specialties:

- **Fatigue, fracture, stress rupture characterization of coupons & tubes / microanalysis of damage & failure modes; small-on-large dual frequency excitation**
- **Micromechanics of strength of composite systems / effect of temperature on strength; micromechanical stress rupture models**
- **3-D Simulation of fiber fracture in tension / effect of material scaling parameters, interphase regions, fiber-fiber interactions**
- **Durability and damage tolerance / prediction of remaining strength and life for combined fatigue, creep, stress rupture**
- **Combined thermal, mechanical, chemical design analysis / software development and integration**



High Temperature Research Programs in the Materials Response Group at Virginia Tech

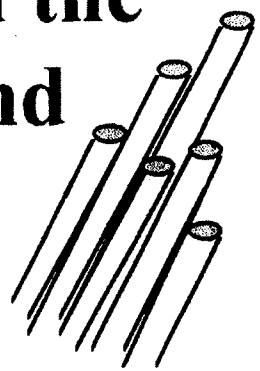
Some relationships:



Virginia Tech
Materials Response Group

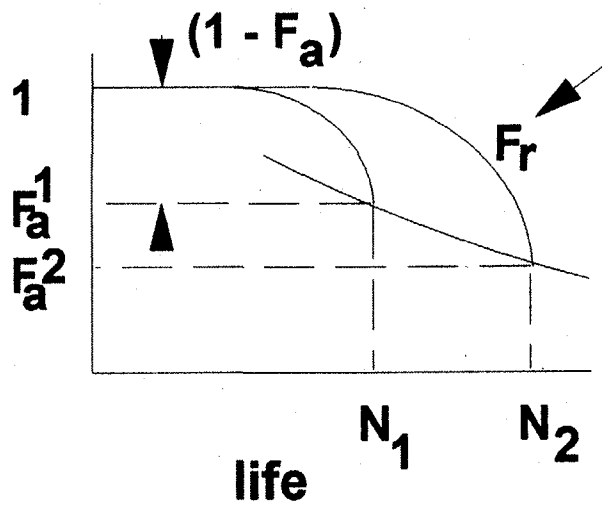
Life Prediction: Objectives -

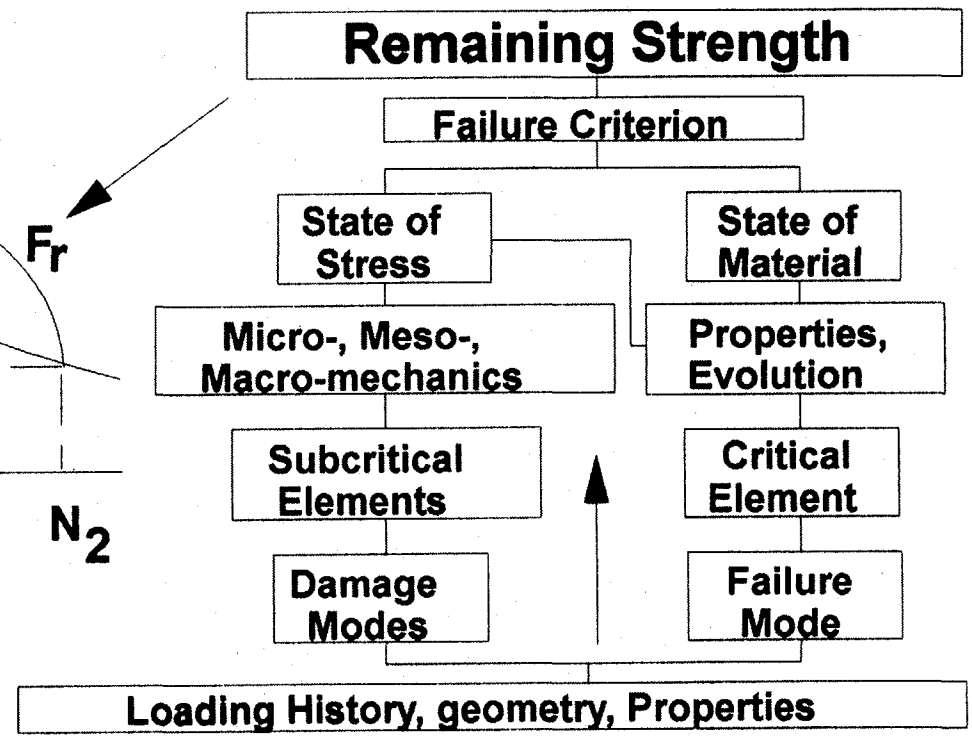
- ★ **To create a design tool that integrates and combines data and understanding from the CFCC program**
- ★ **To develop the capability to predict remaining strength and life for combined creep, stress rupture, and fatigue in the presence of mechanical, thermal, and chemical applied environments**



MRLife ™

MRLife Approach:

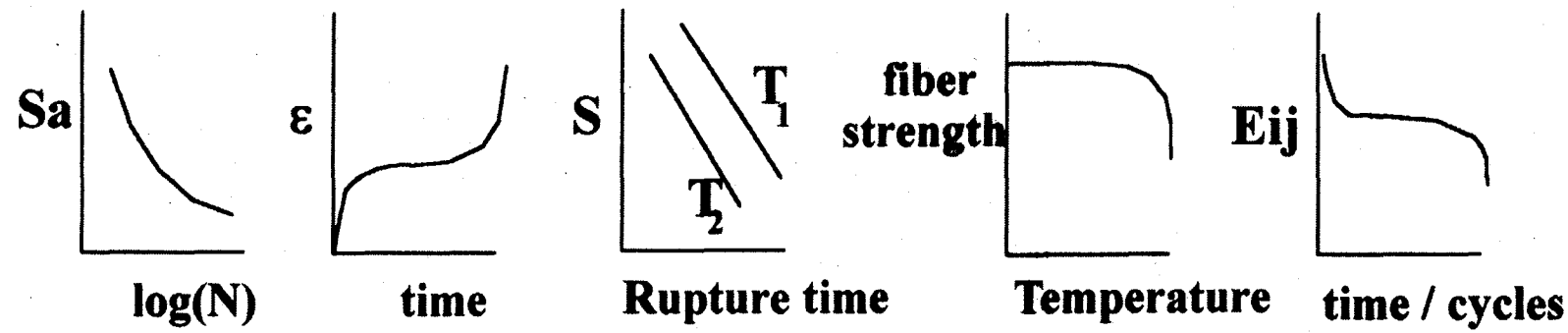




Materials Response Group
Virginia Tech

Material property inputs to the remaining strength equation:

Fatigue **Creep** **Stress Rupture** **Environments** **Micro-Damage**



$N(t)=N(\sigma/X(t))$ $E(t)\Rightarrow\sigma_{ij}(t)$ $X(t)$ $X(t)$ $G(t), \nu(t) \Rightarrow \sigma_{ij}(t)$

$$F_r(t)=1-\int_0^t \left(1-F_a\left(\frac{\sigma(t)_{ij}}{X(t)_{ij}}\right)\right)\cdot j\cdot\left(\frac{n}{N(t)}\right)^{j-1} d\left(\frac{n}{N(t)}\right)$$

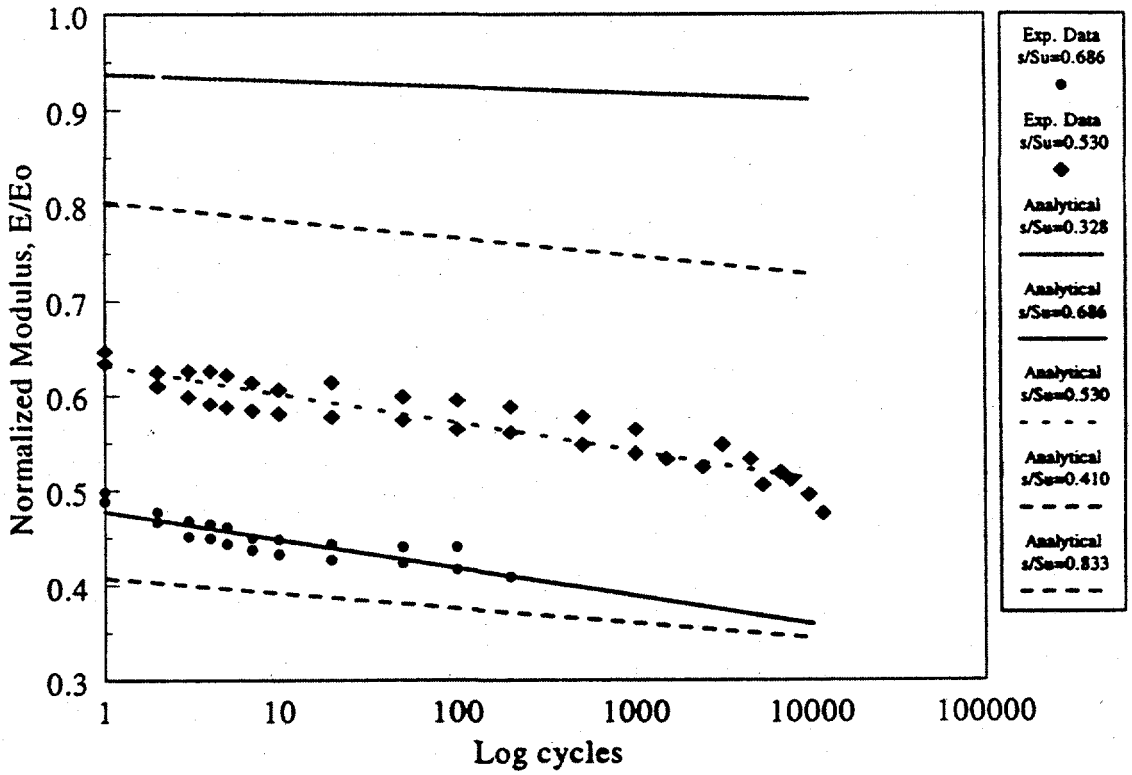


Figure 2: Stiffness Reduction of 2-D Woven Nicalon/[illegible]-SiC Composite at 1800°F

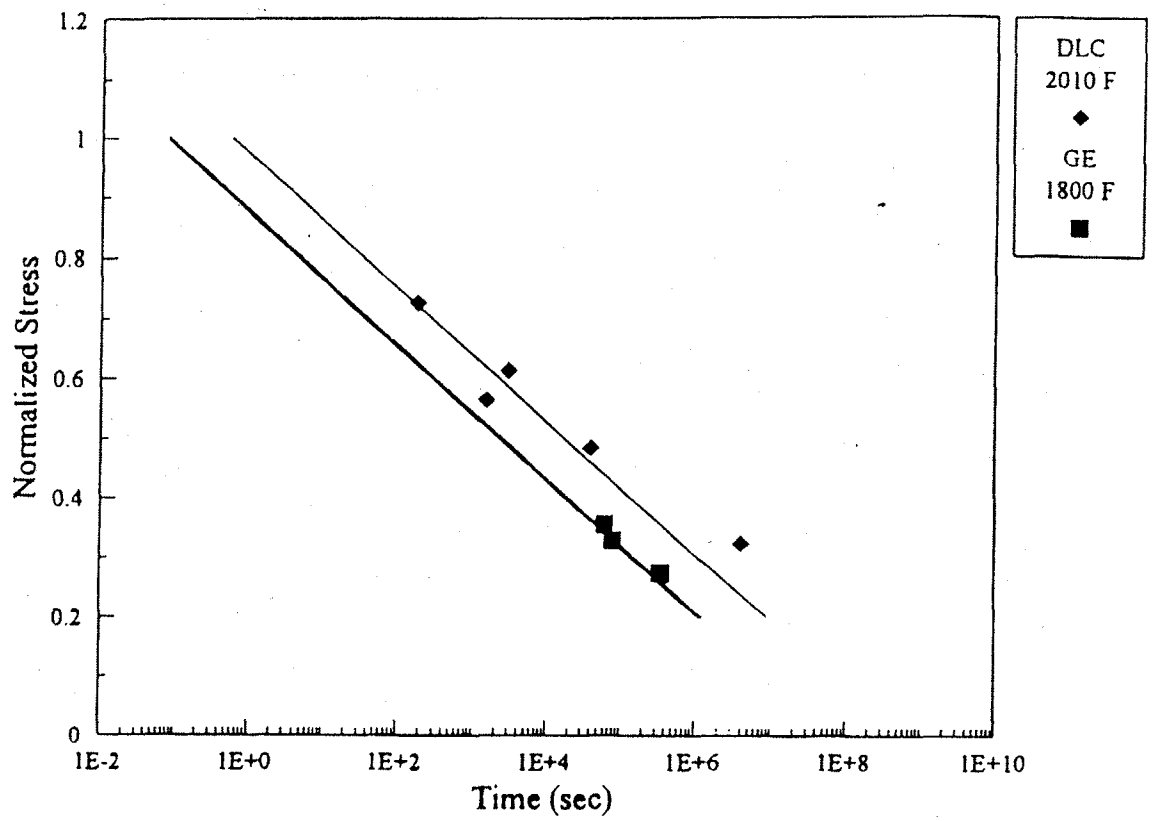


Figure 5.6:Stress Rupture data for 2-D woven Nicalon [redacted] laminates (adapted from ref. 92)

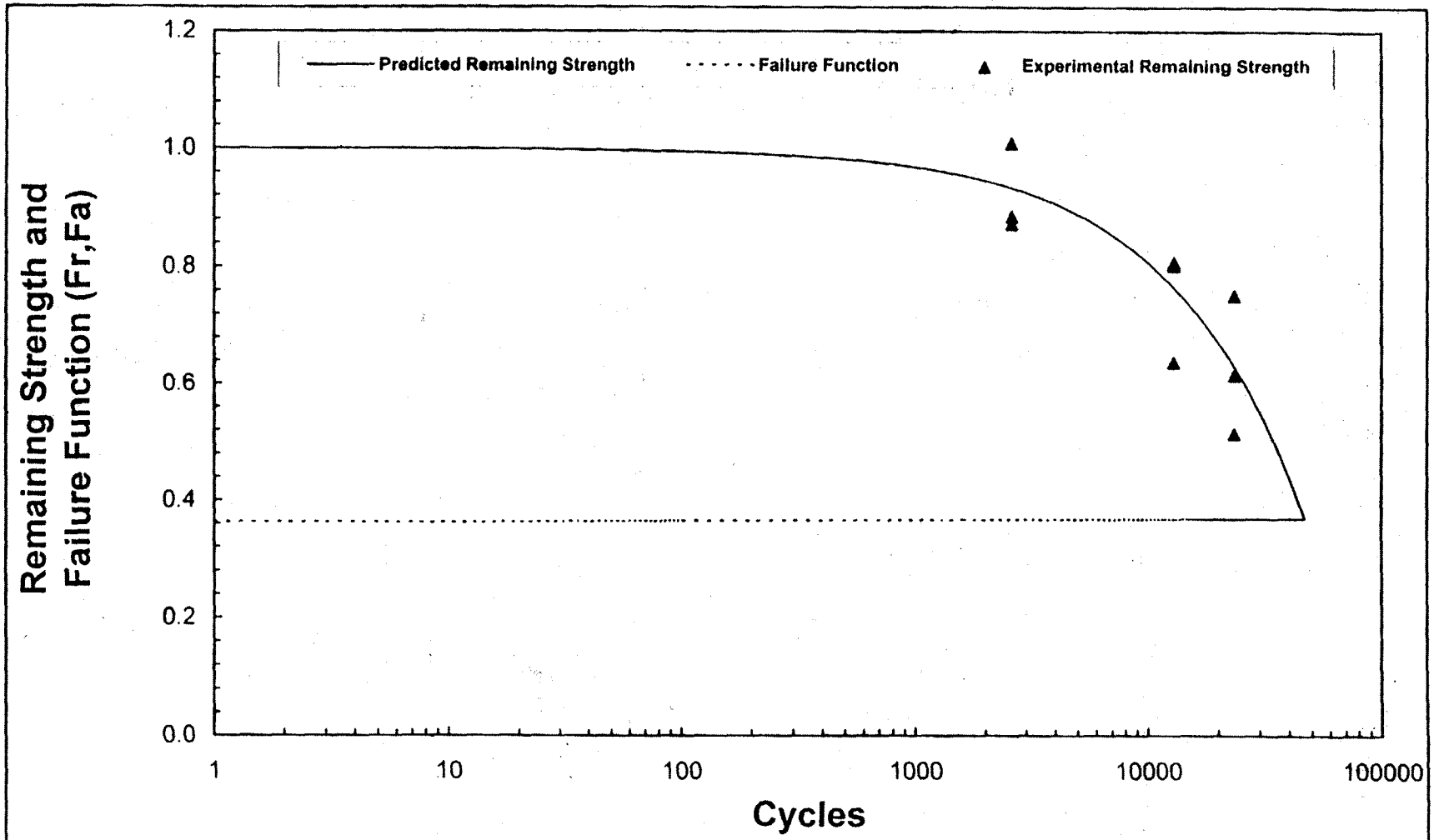


Figure 5.9: Comparison between experimental and predicted remaining strengths of $[(0,90)/(0,90)]_{2s}$ laminates for R=-1, σ_{max} = 13 Ksi, f=1 Hz at 1800 F under atm. air.

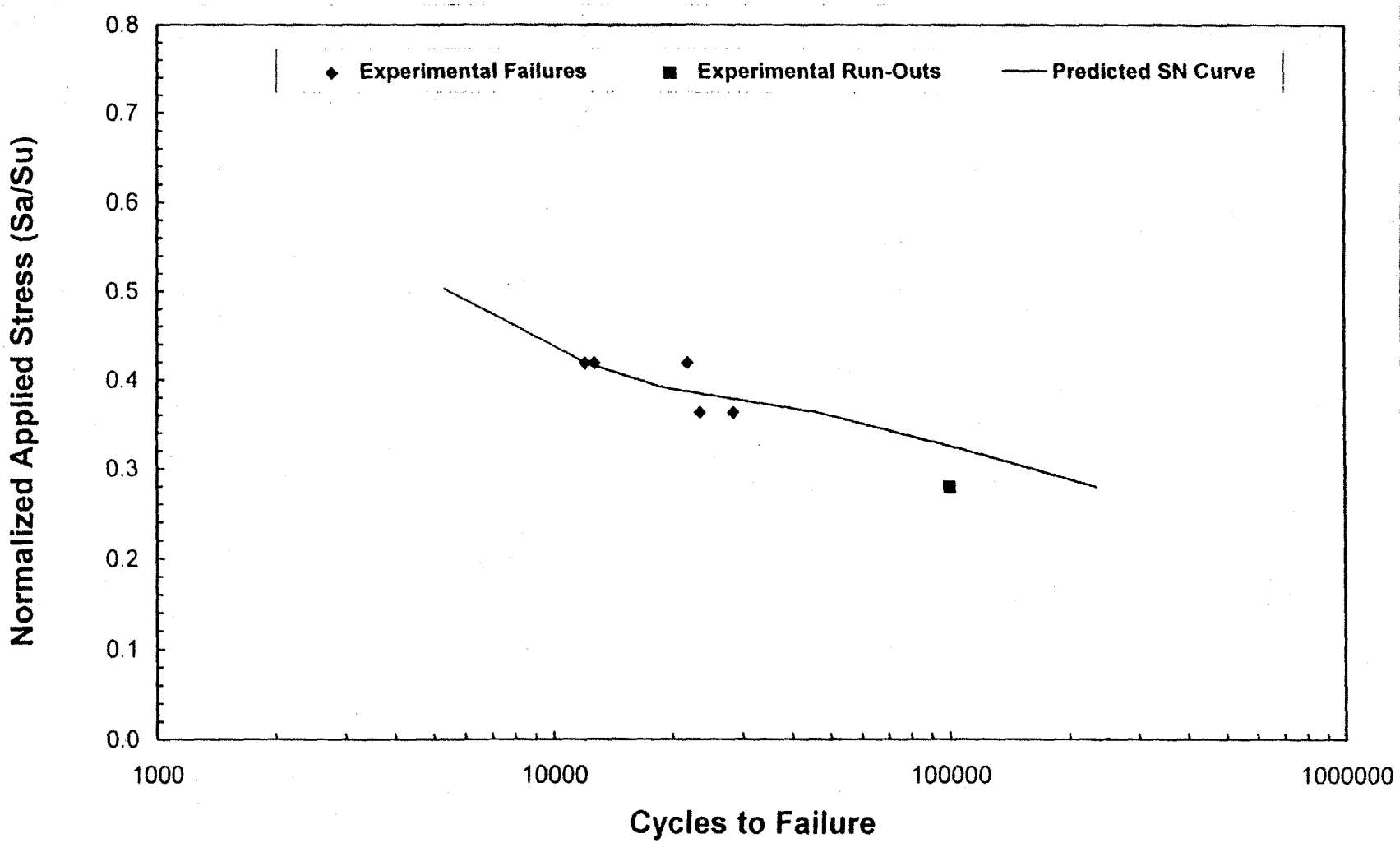
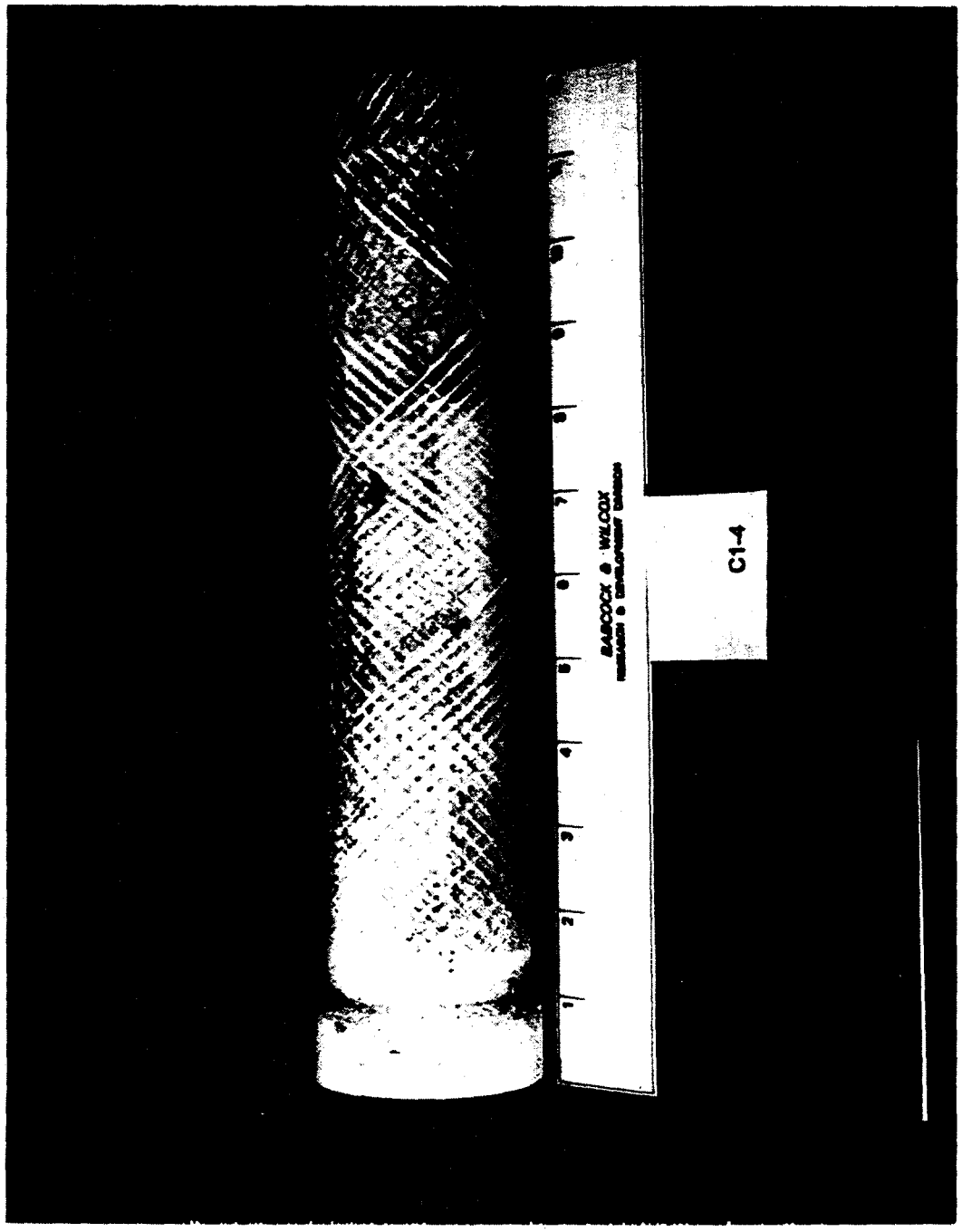


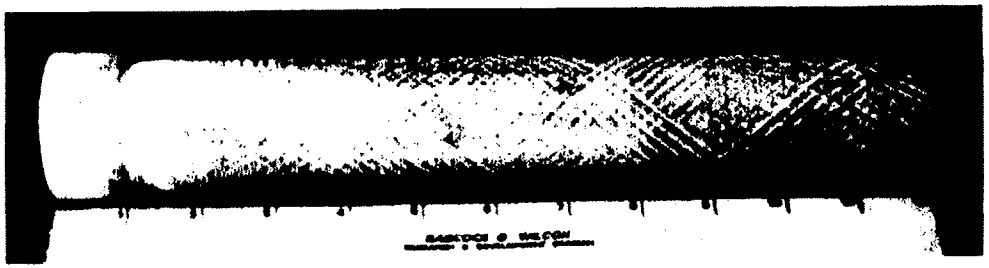
Figure 5.11: Comparison between experimental and predicted fatigue lives for $[(0,90)/(0,90)]_{2s}$ laminates with R=-1, f=1 Hz, at 1800 F under atm. air.


BABCOCK & WILCOX
C1-4

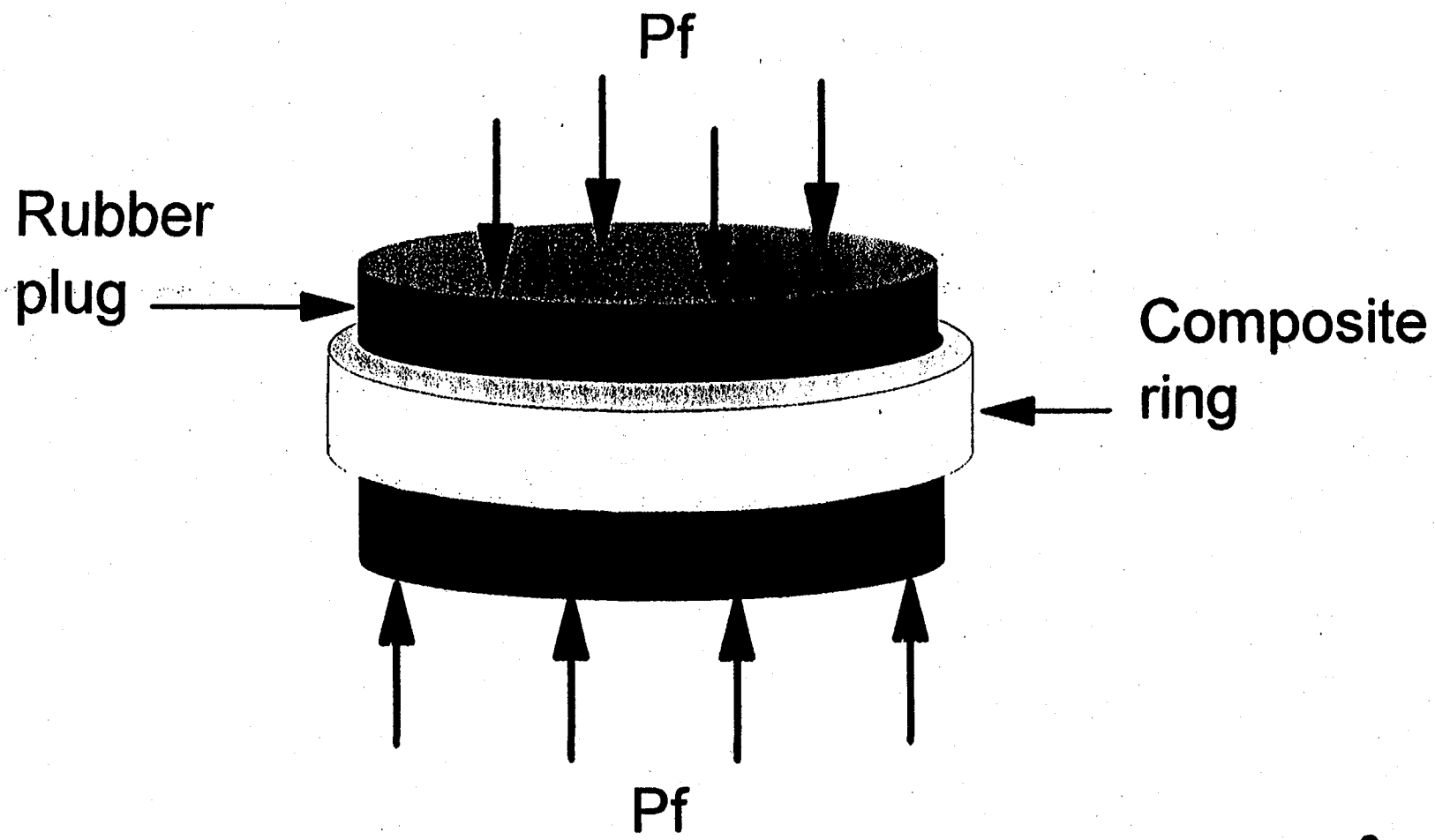
"*Mechanical Characterization and Durability Modeling*"

Task I - characterization of hot gas materials :

- |> Initial / remaining strength and life for unexposed / exposed tubes
- |> Damage and failure modes
- |> Special attention to cross-over points and Saffil matrix to support modeling of damage / failure processes

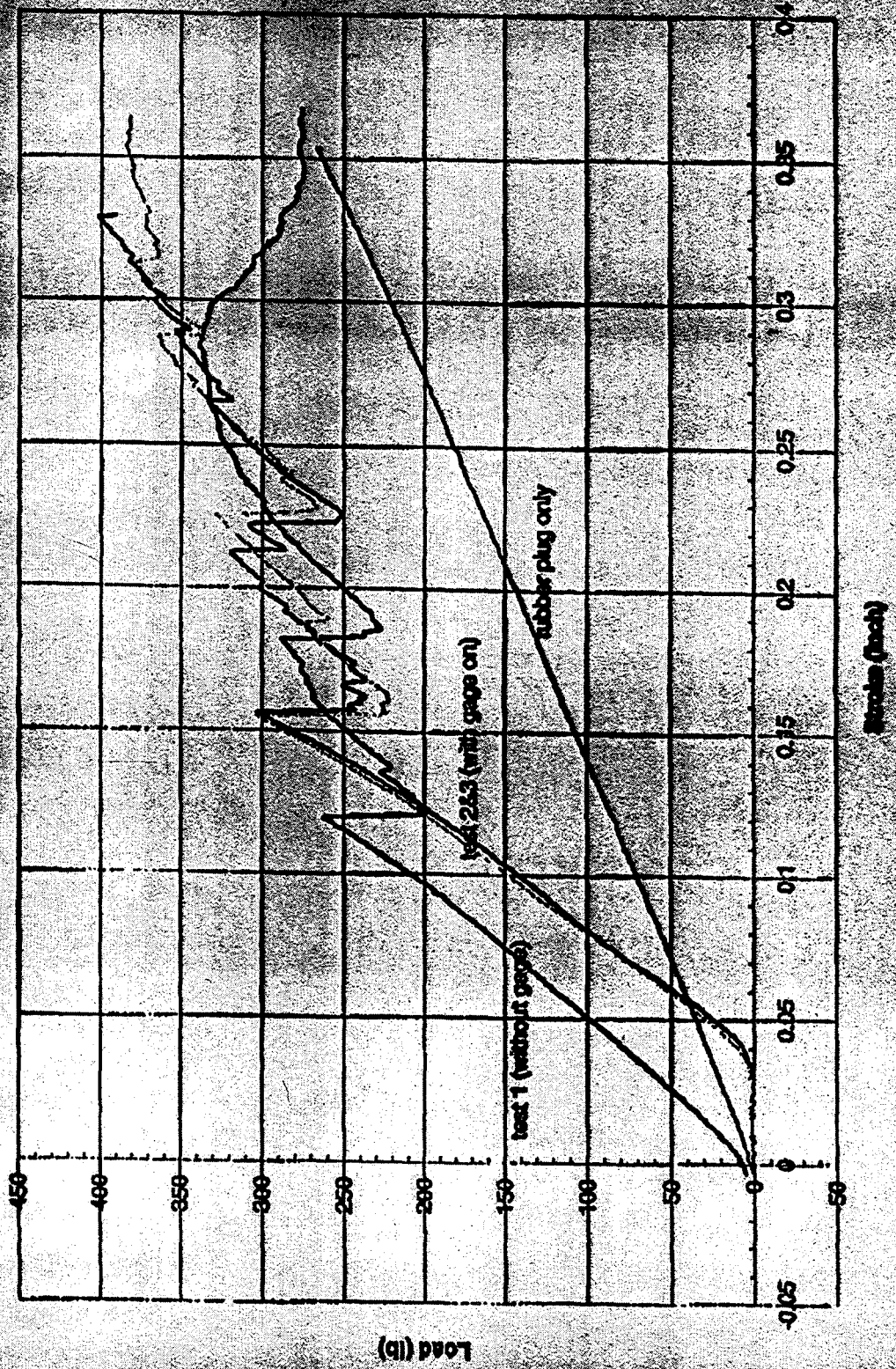


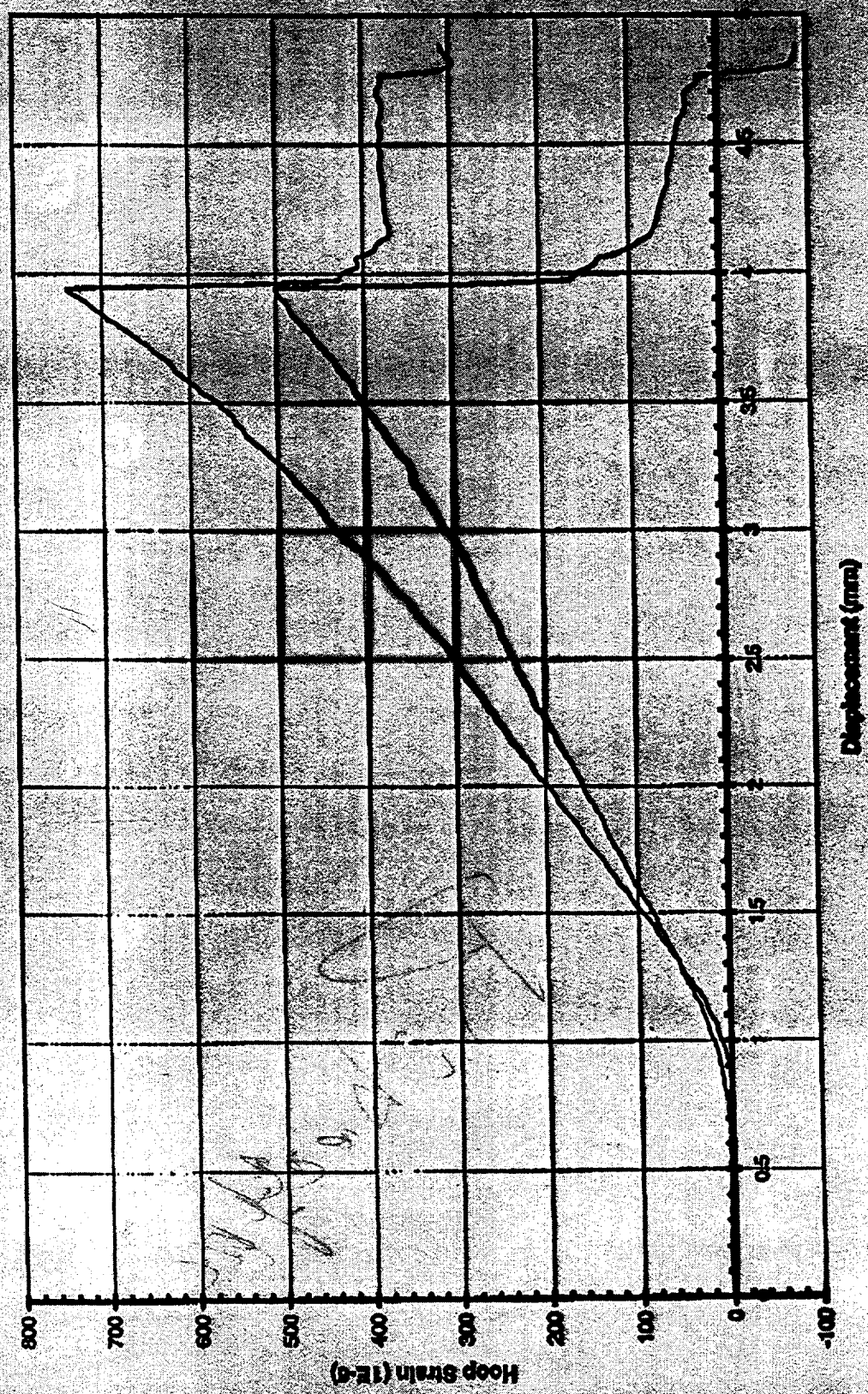
Measurement of composite strength:



$$\text{Radial_Pressure} = \frac{\upsilon}{1+\upsilon} \cdot \frac{Pf - Po}{\pi \cdot ri^2}$$

$$\text{Hoop_Stress} = p \cdot \frac{ro^2 + ri^2}{ro^2 - ri^2}$$


Load Vs. Displacement
Load (lb)
Displacement (inch)
test 1 (without gauge)
test 2&3 (with gauge on)
rubber plug only
450
400
350
300
250
200
150
100
50
0
50
-0.05
0
0.05
0.1
0.15
0.2
0.25
0.3
0.35
0.4



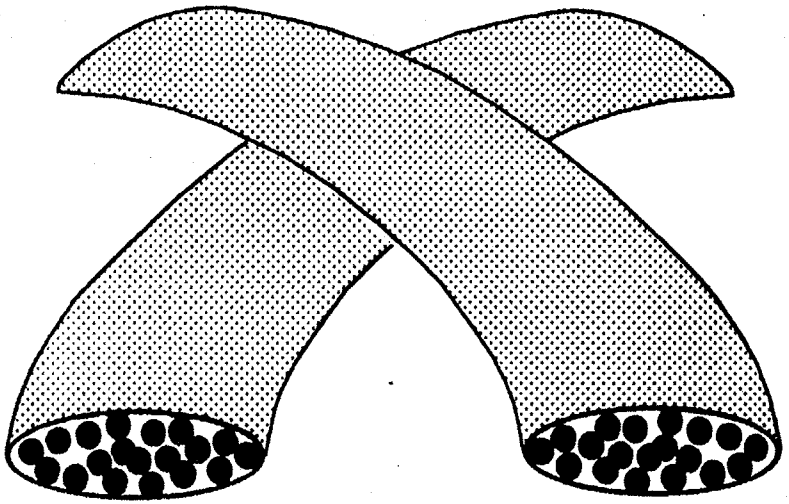
Hoop Strain vs. Displacement

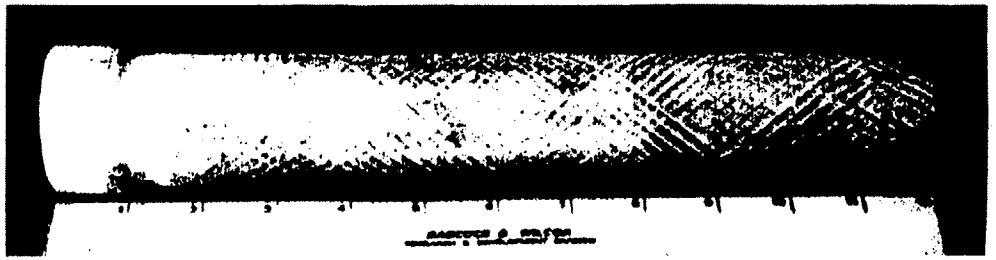
"*Mechanical Characterization and Durability Modeling*"

Task II - MRLife modeling; design support :

Ligature Analysis for Porous Media ("LAP" code?)

- |> Tows are ligatures with beam properties
- |> Crossover points are "bonded"
- |> Matrix / void properties appear in the bonding stiffness and strengths
- |> Tow-models could be micromechanical





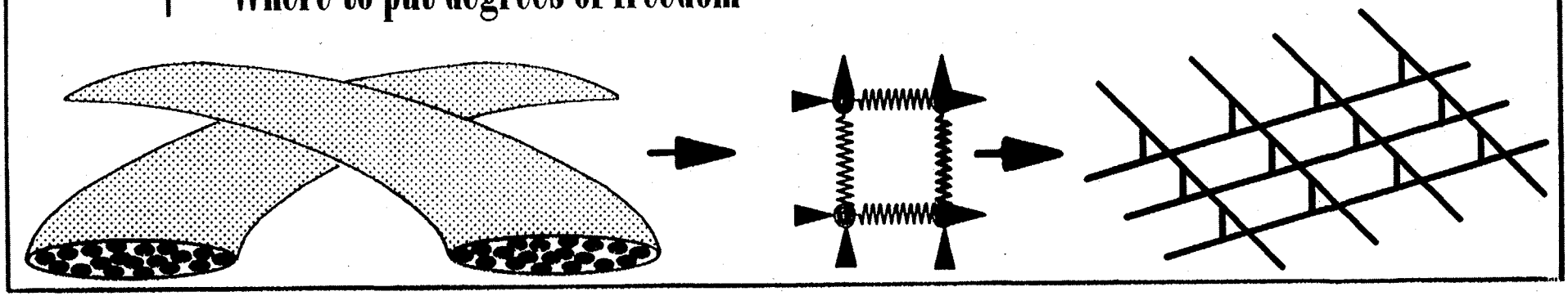
"*Mechanical Characterization and Durability Modeling*"

Ligature Analysis for Porous Media ("LAP") - Advantages / capabilities:

- |> "Anatomically correct" model for wound and woven microstructures
- |> Translates directly to macro-structural geometries
- |> Contains essentially all of the manufacturing parameters
- |> Can model local variations in properties (radial, spatial)

Ligature Analysis for Porous Media ("LAP") - Challenges:

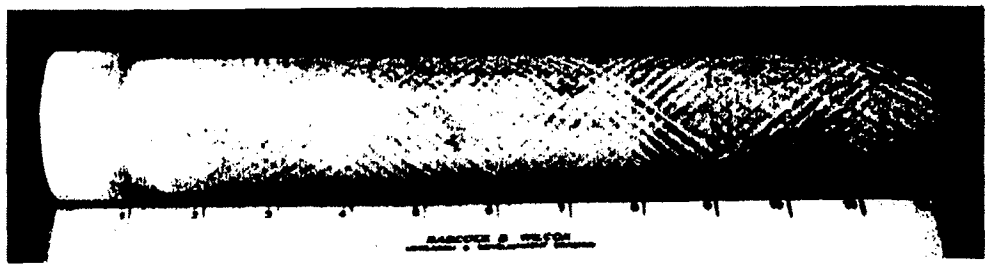
- |> How to measure thickness-direction properties
- |> Where to put degrees of freedom



"*Mechanical Characterization and Durability Modeling*"

Task II - MRLife modeling; design support :

|> We have constructed a new approach to the meso- and micro-modeling of porous materials that will enable us to predict global stiffness, strength, and life on the basis of constituent characteristics (including the void structure of the materials) and local stresses / strains. This unique enabling technology should provide a vital competitive edge to B&W and to their clients.

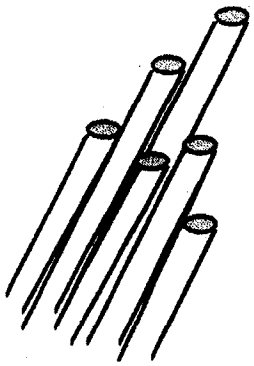


Virginia Tech

Materials Response Group

Current thrusts:

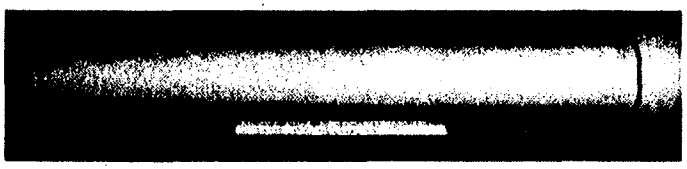
- ★ **Predict durability and damage tolerance of combustor liners as a function of material system parameters and operating environments**
- ★ **Create an integrated design tool by combining MRLife with ANSYS**

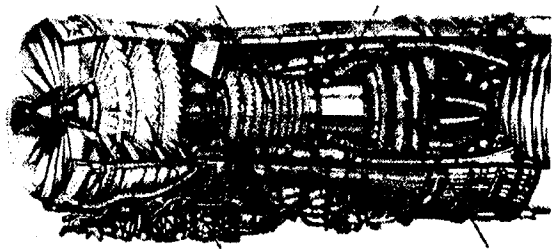


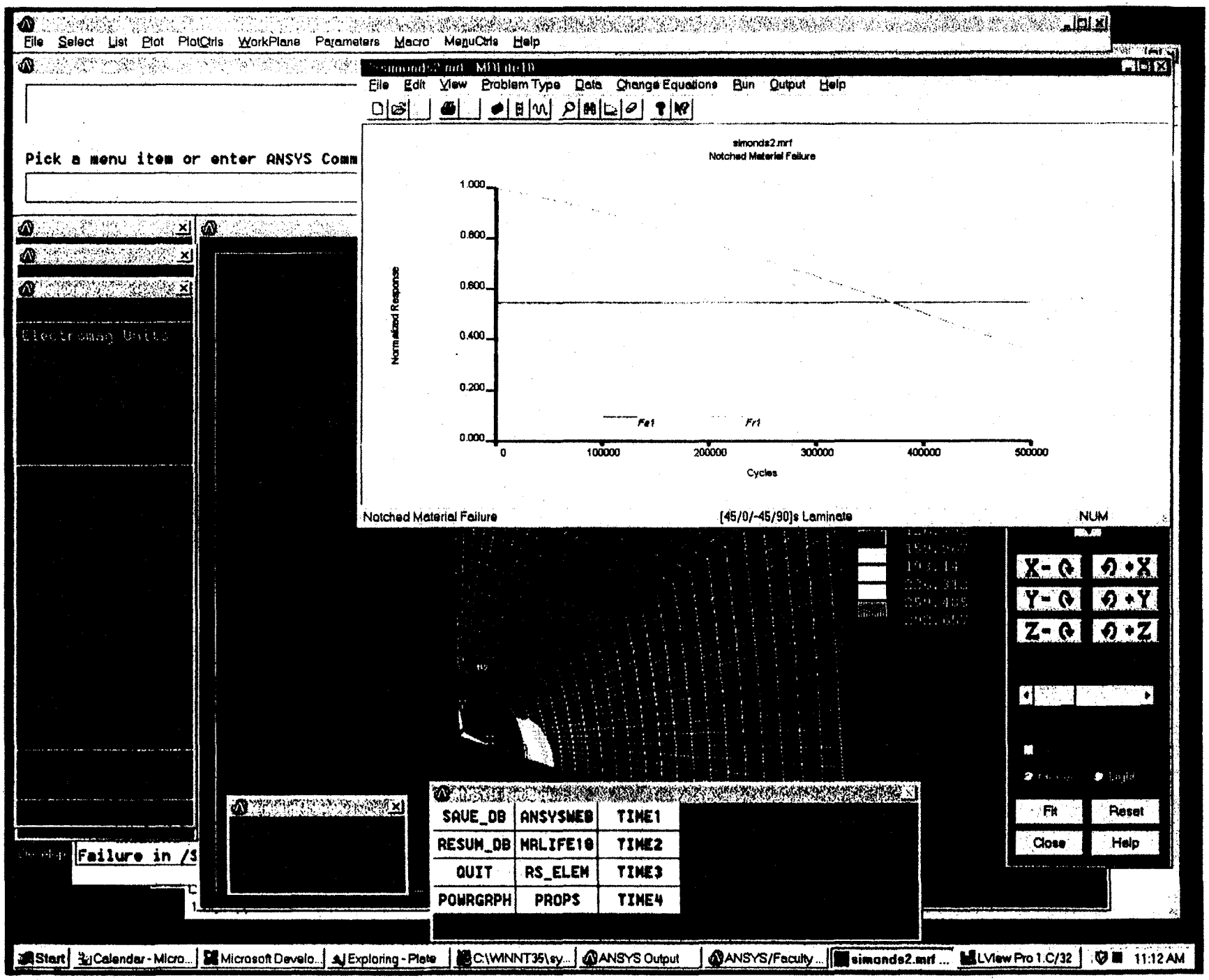
High Temperature Research Programs in the Materials Response Group at Virginia Tech

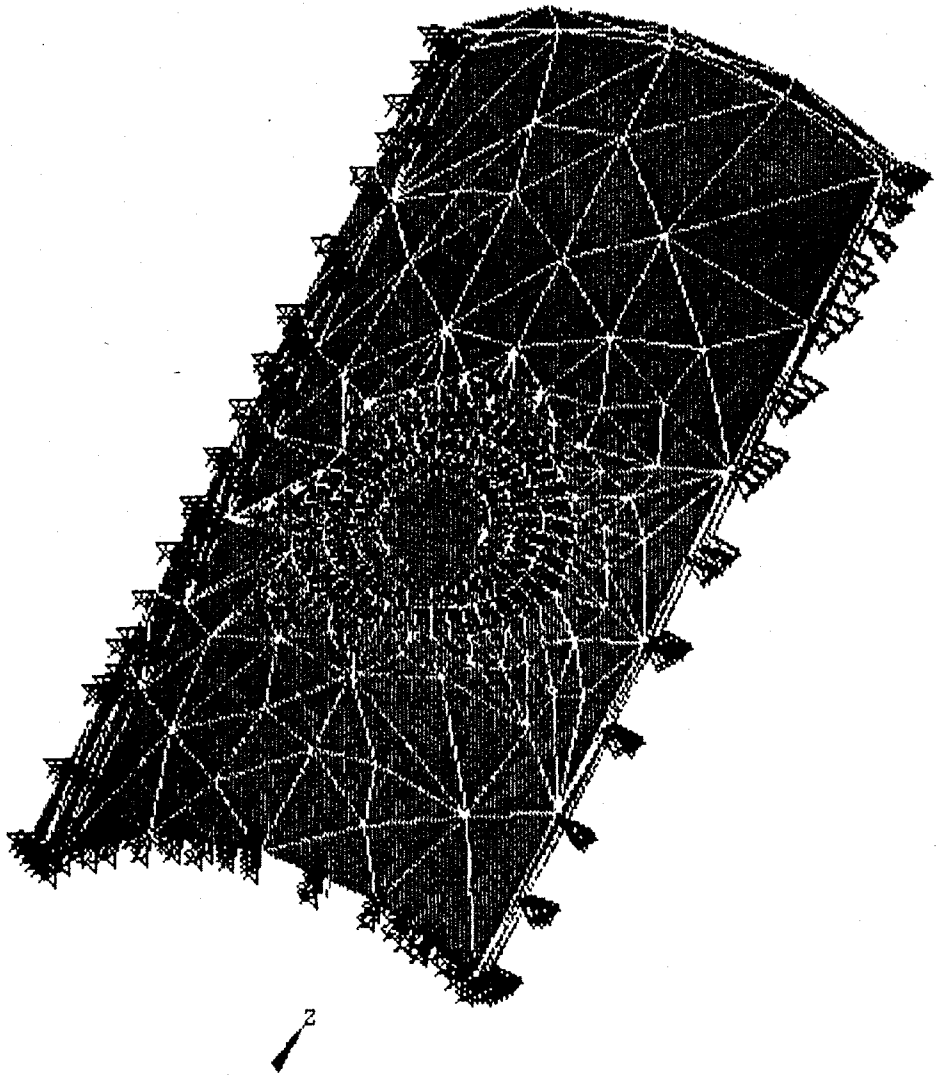
Combustor liners:

- **Began with a B&W prototyping effort; used MRLife / ABAQUS**
- **Have an informal team including Allison, Solar Turbines, Goodrich, Oak Ridge, Babcock and Wilcox, DuPont**
- **Recently initiated an effort to integrate MRLife with ANSYS for the purpose of achieving a complete life design of combustor liners**



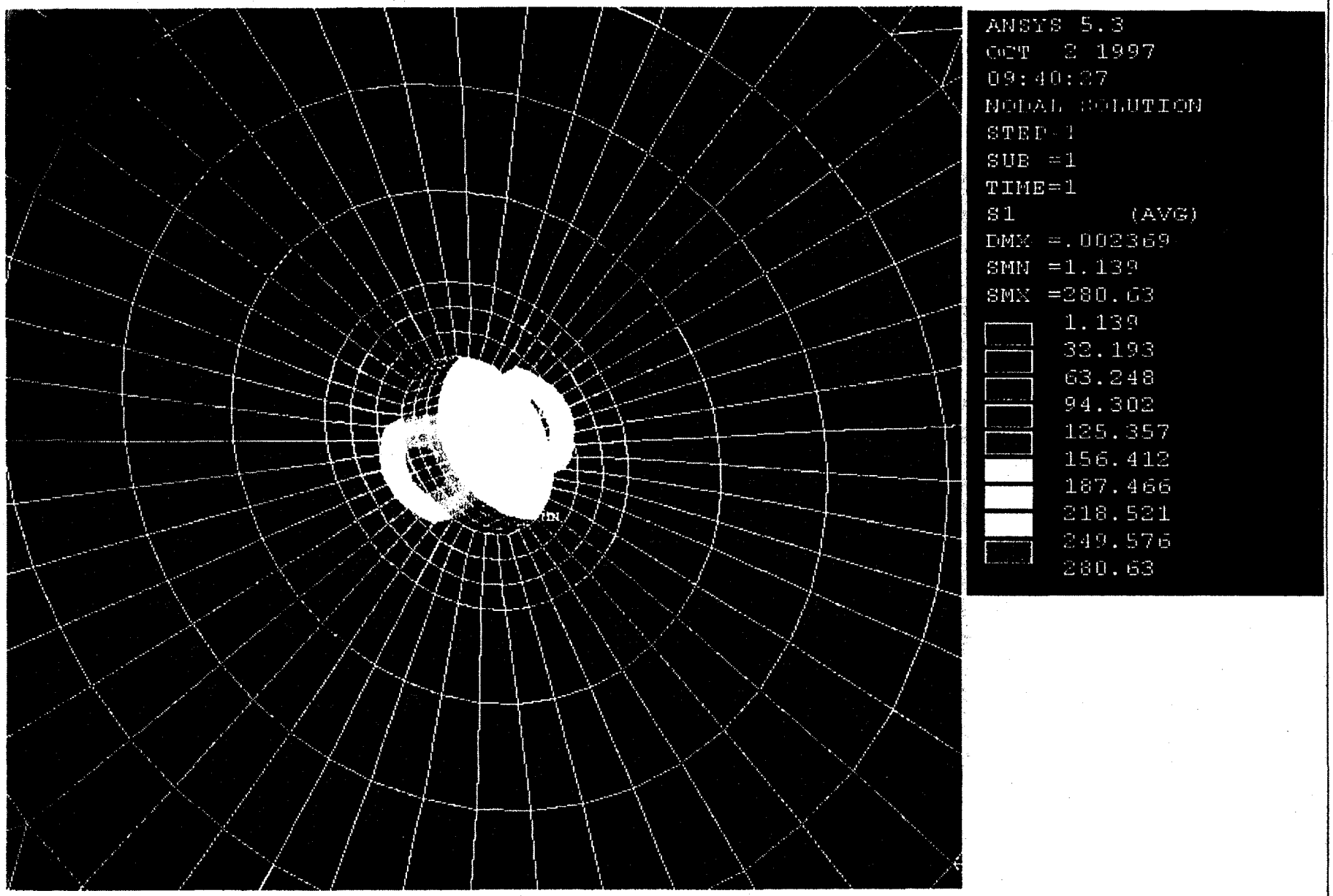



File Select List Plot PlotCtrls WorkPlane Parameters Macro MenuCtrls Help
File Edit View Problem Type Data Change Equations Run Output Help
Pick a menu item or enter ANSYS Comm
simonds2.mrf
Notched Material Failure
Normalized Response
1.000
0.800
0.600
0.400
0.200
0.000
Fa1
Fr1
0
100000
200000
300000
400000
500000
Cycles
Notched Material Failure
[45/0/-45/90]s Laminate
NUM
X-
+X
Y-
+Y
Z-
+Z
Fit
Reset
Close
Help
Failure in /S
SAVE_DB
ANSYSWEB
TIME1
RESUM_DB
MRLIFE10
TIME2
QUIT
RS_ELEM
TIME3
POWRGRPH
PROPS
TIME4
Start
Calendar - Micro...
Microsoft Develo...
Exploring - Plate
C:\WINNT35\sy...
ANSYS Output
ANSYS/Faculty ...
simonds2.mrf ...
LView Pro 1.C/32
11:12 AM

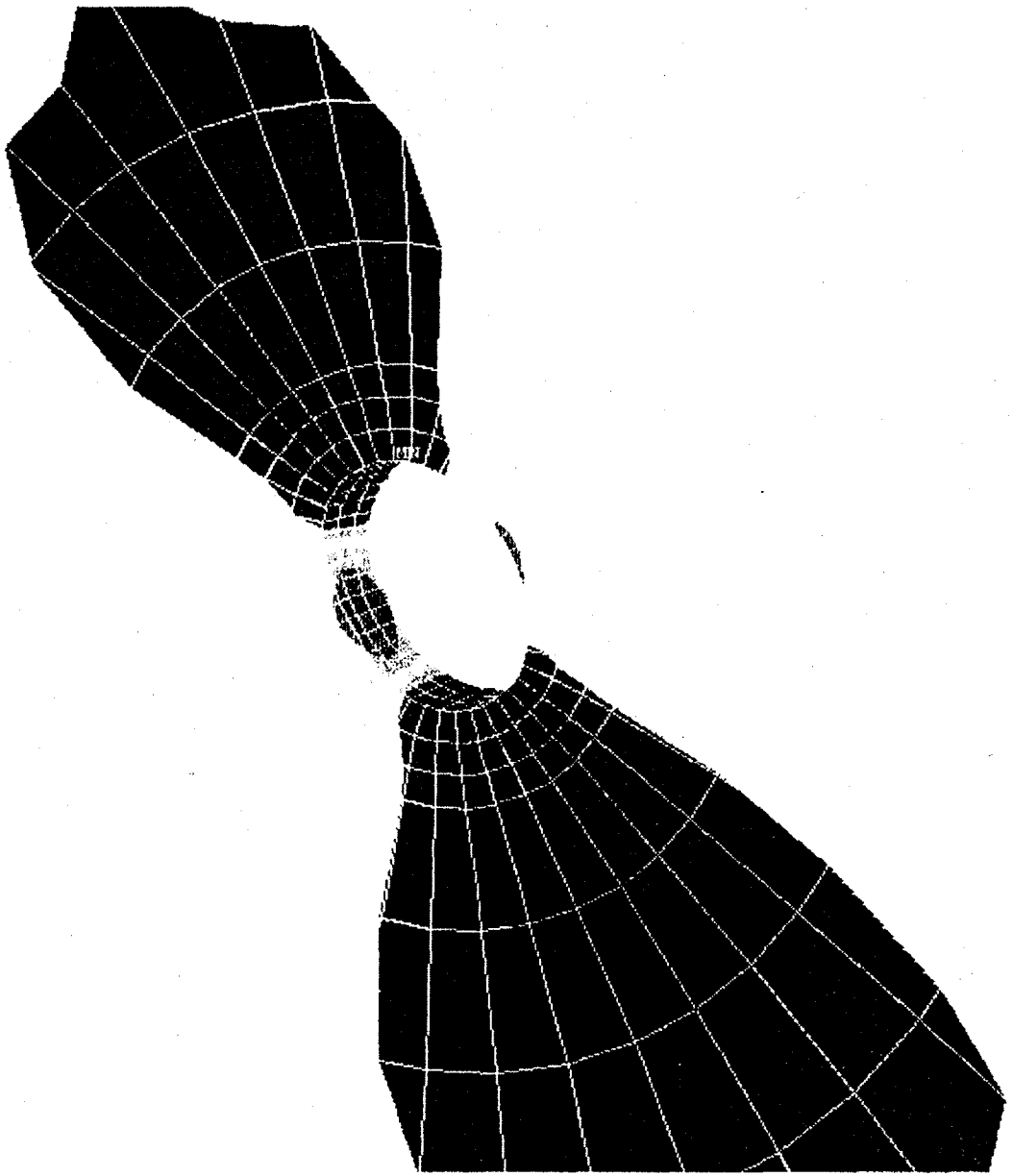


[redacted]

Loads and Boundary Conditions



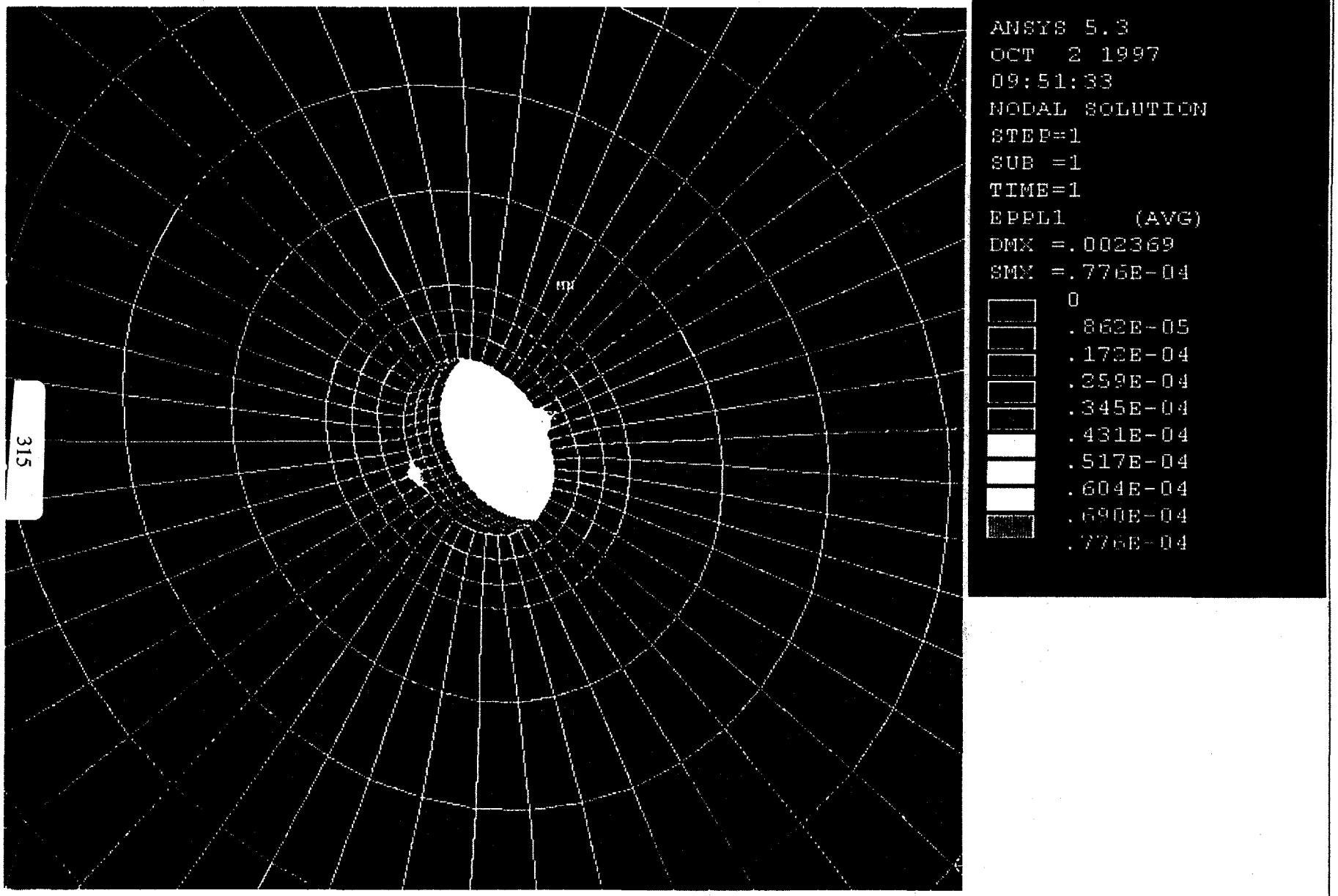
Initial Primary Principle Stress State



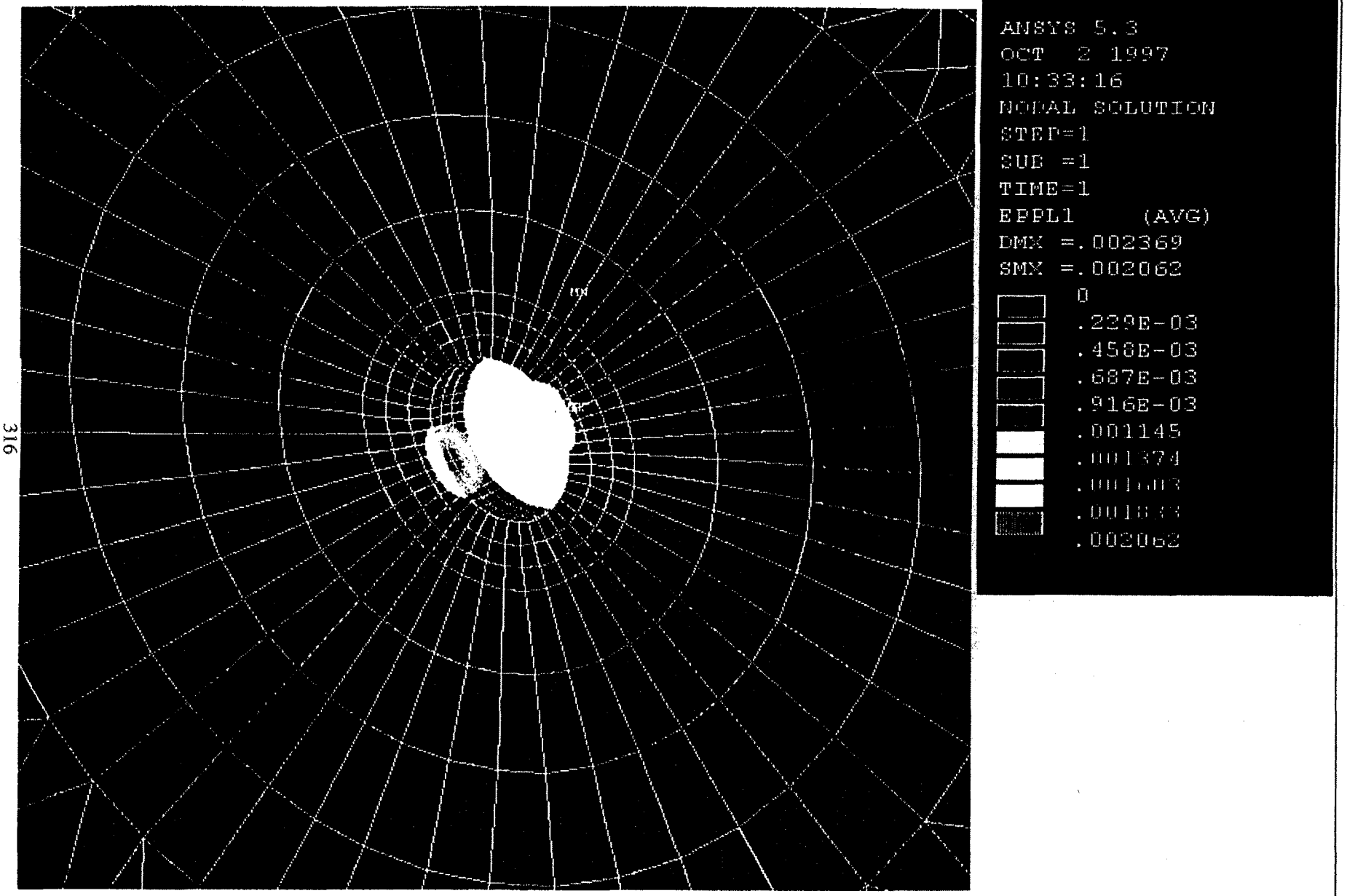
ANSYS 5.3
OCT 2 1997
10:17:50
NODAL SOLUTION
STEP=1
SUB =1
TIME=1
S1 (AVG)
DMX =.002377
SMN =-.708764
SMX =156.761

-.708764
16.788
34.284
51.781
69.278
86.774
104.271
121.767
139.264
156.761

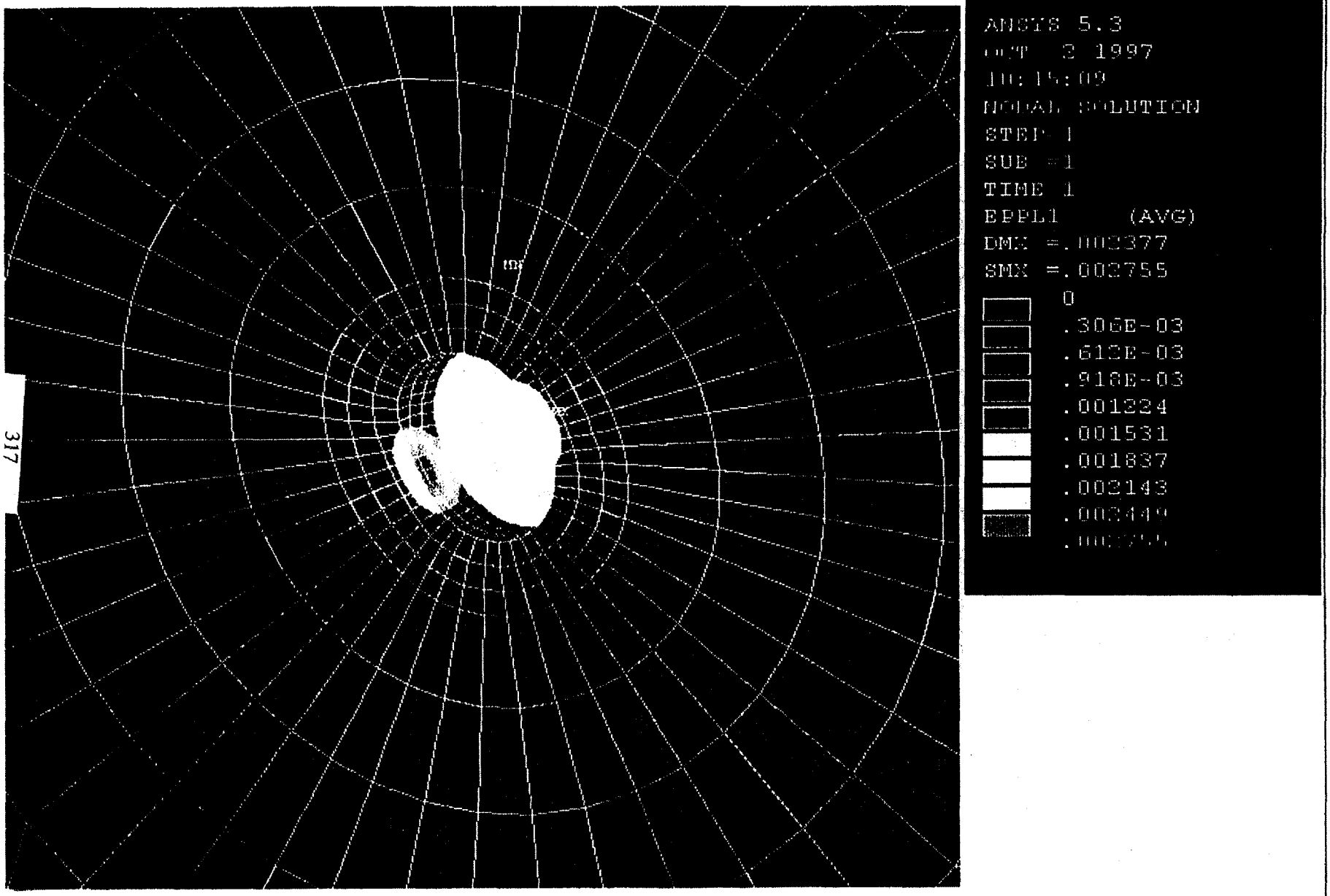
1st Principle Stress State at 100hrs



Strain Due to Damage in the 1st Principle Direction at 1hr



Strain Due to Damage in the 1st Principle Direction at 10hr



Strain Due to Damage in the 1st Principle Direction at 10hr

ENVIRONMENTAL TESTING OF CFCC'S IN COMBUSTION GAS ENVIRONMENTS

Krishan L. Luthra
GE Corporate Research and Development
Schenectady, NY 12301

October 7, 1997

Outline

- Environment
- Test Procedures
  - FOD, localized overstressing
- Results
- Summary (Issues)

Environment

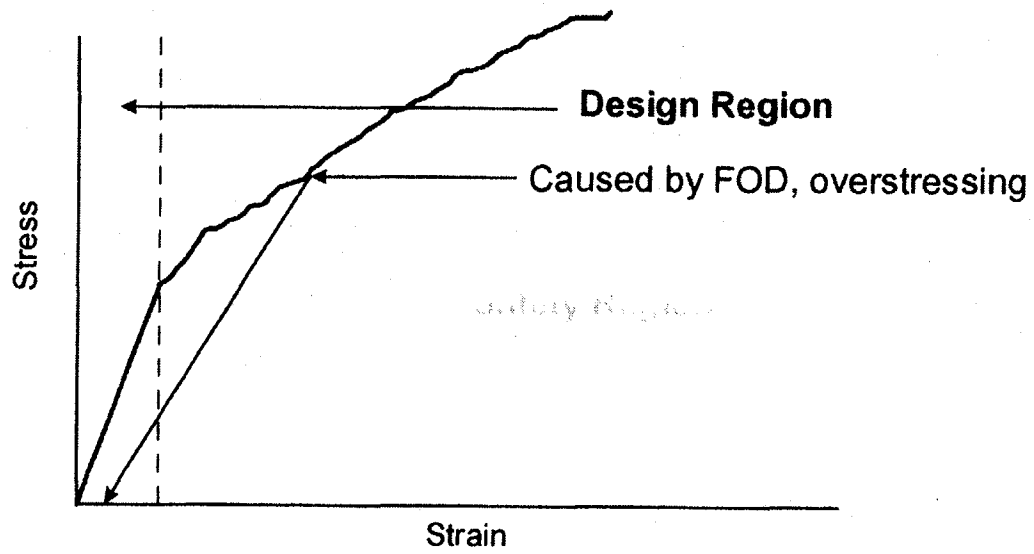
- Gas Chemistry
  - depends on air/fuel ratio and pressure

	GT at 12 atm		1 atm Tests	
	%	Partial Pressure, atm	Hot Corrosion	Oxidation
N_2	77.8	9.34	--	--
O_2	15.7	1.89	99.85	65
CO_2	3.5	0.42	--	--
H_2O	2.9	0.35	--	35
(SO_2+SO_3)	1.2×10^{-2}	1.5×10^{-3}	0.15	--

- Flow Conditions
  - mass transfer coefficient
  - influences volatilization of silica and boron oxide

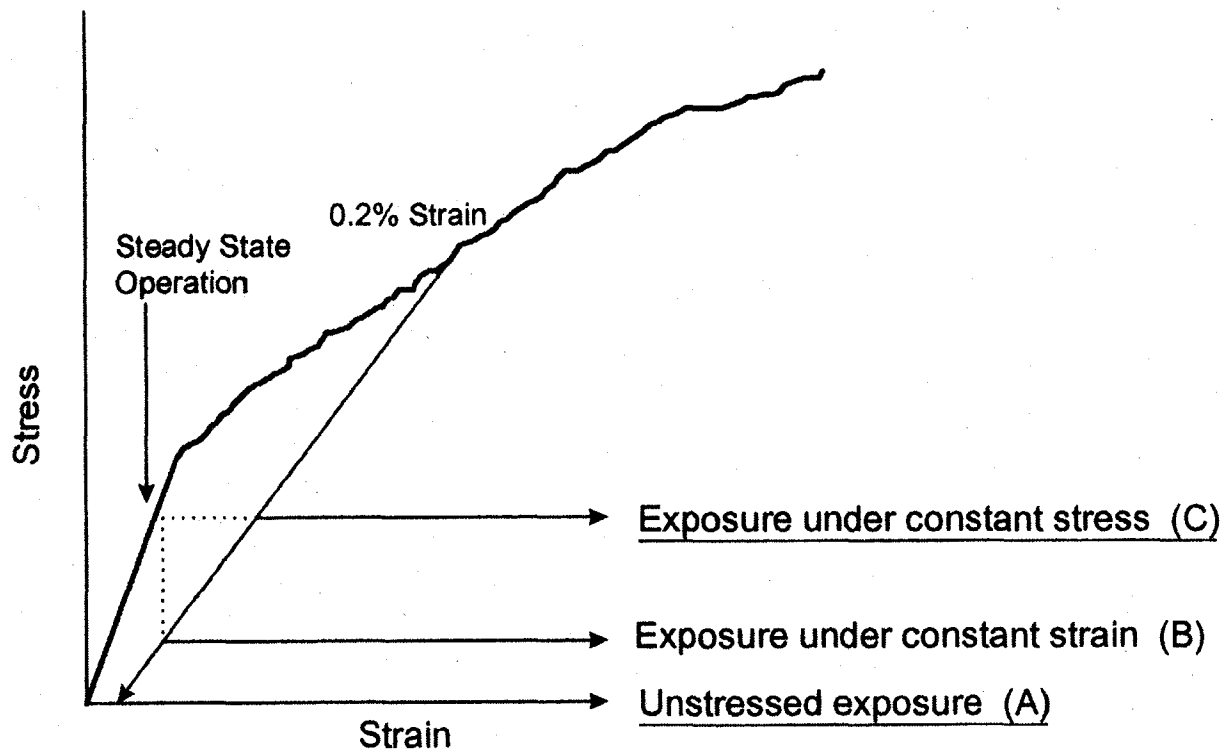
Testing

- Why do we need composite?
  - to avoid catastrophic failure caused by FOD, overstressing
- Design approach
  - design below proportional limit
  - unintentional exposure above the proportional limit (how many times?)



- Thermally loaded components
  - no mechanical load
  - combustor, shroud, heat exchanger
  - thermal strain induced stresses

Testing Approaches

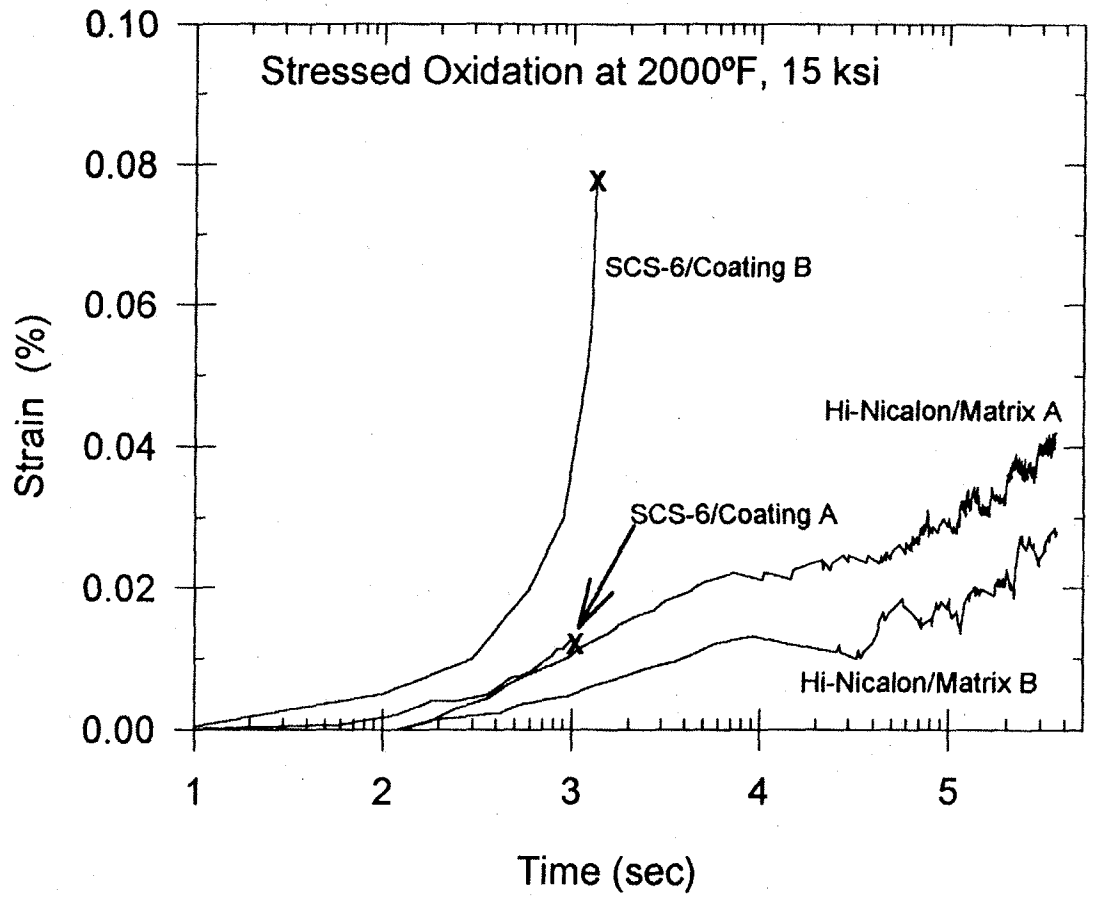


(D) Same as (B) except that the strain is cyclic (Hold Time Fatigue)

(E) Same as (C) except that the stress is cyclic (Hold Time Fatigue)

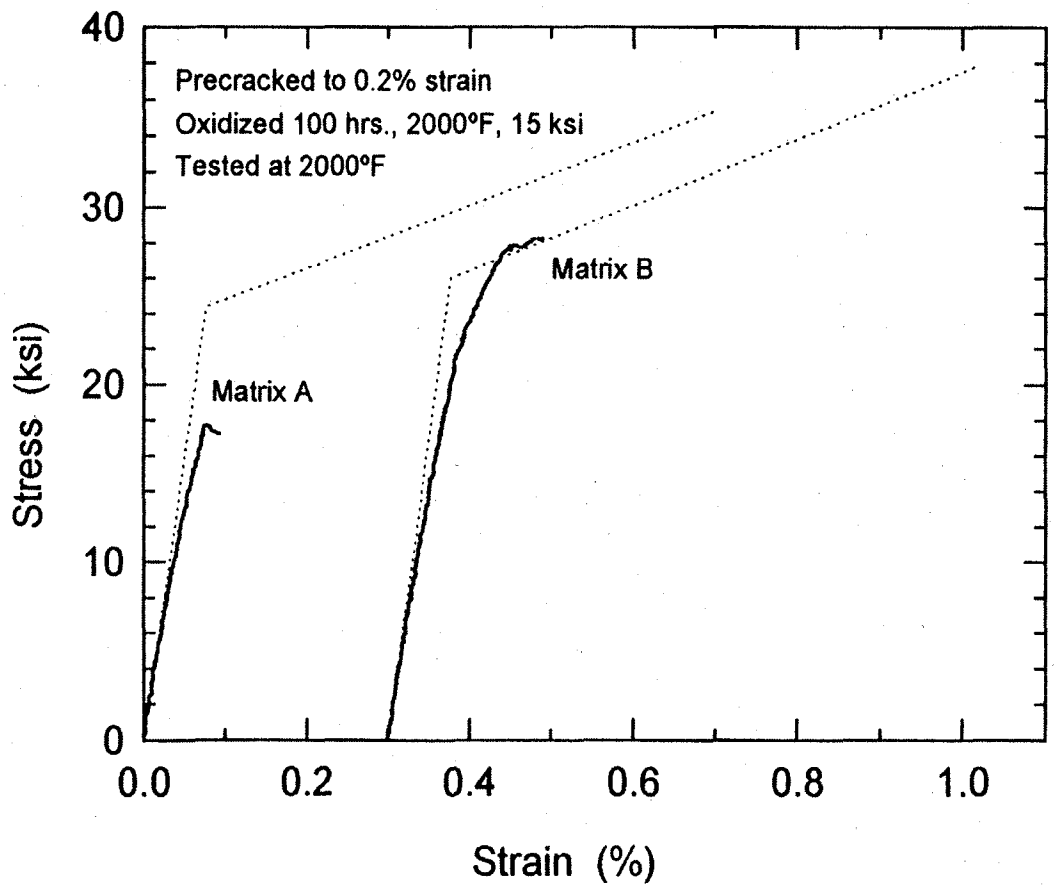
Effect of Matrix Cracking

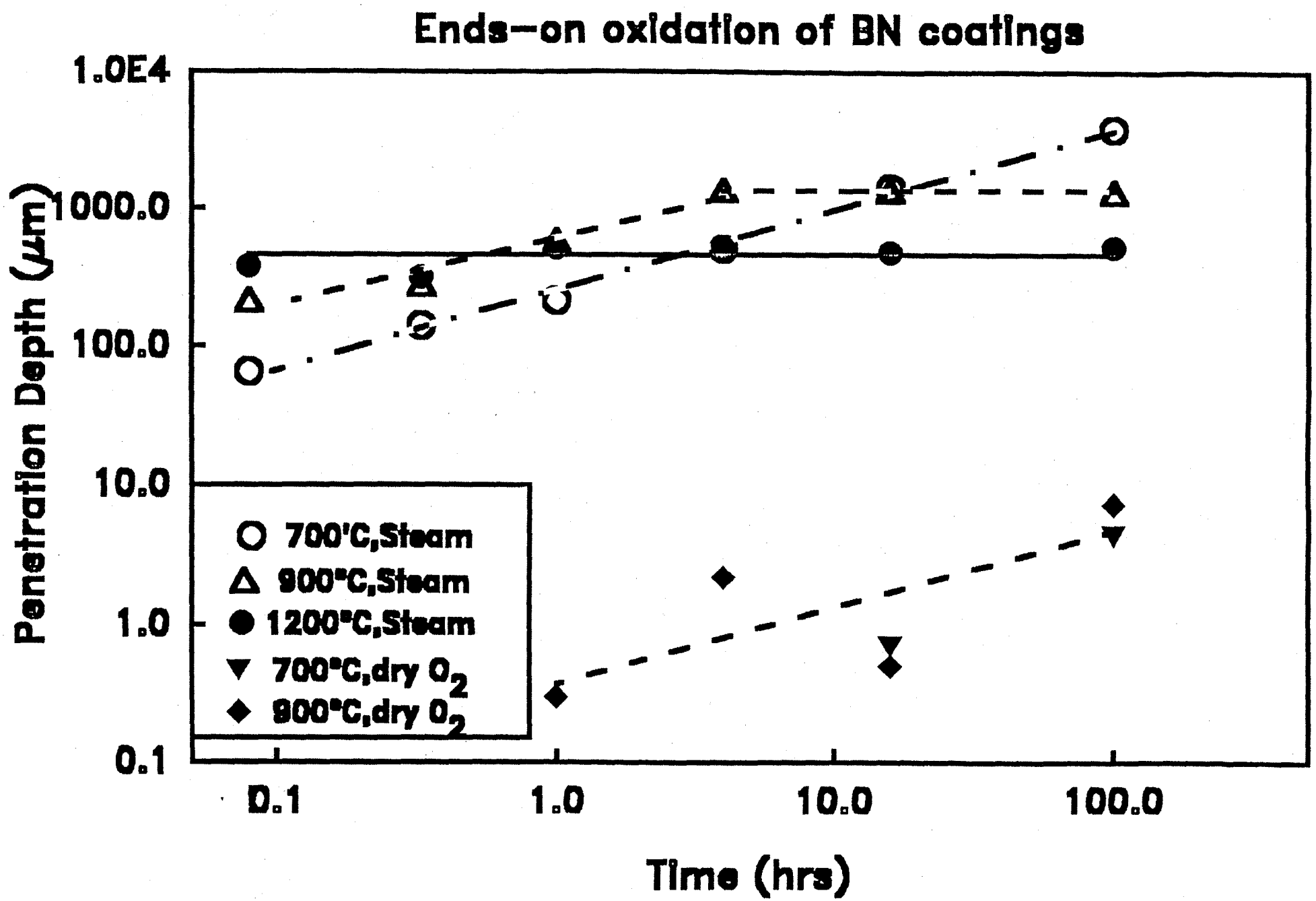
M.I. Composites



Effect of Matrix Cracking

M.I. Composites

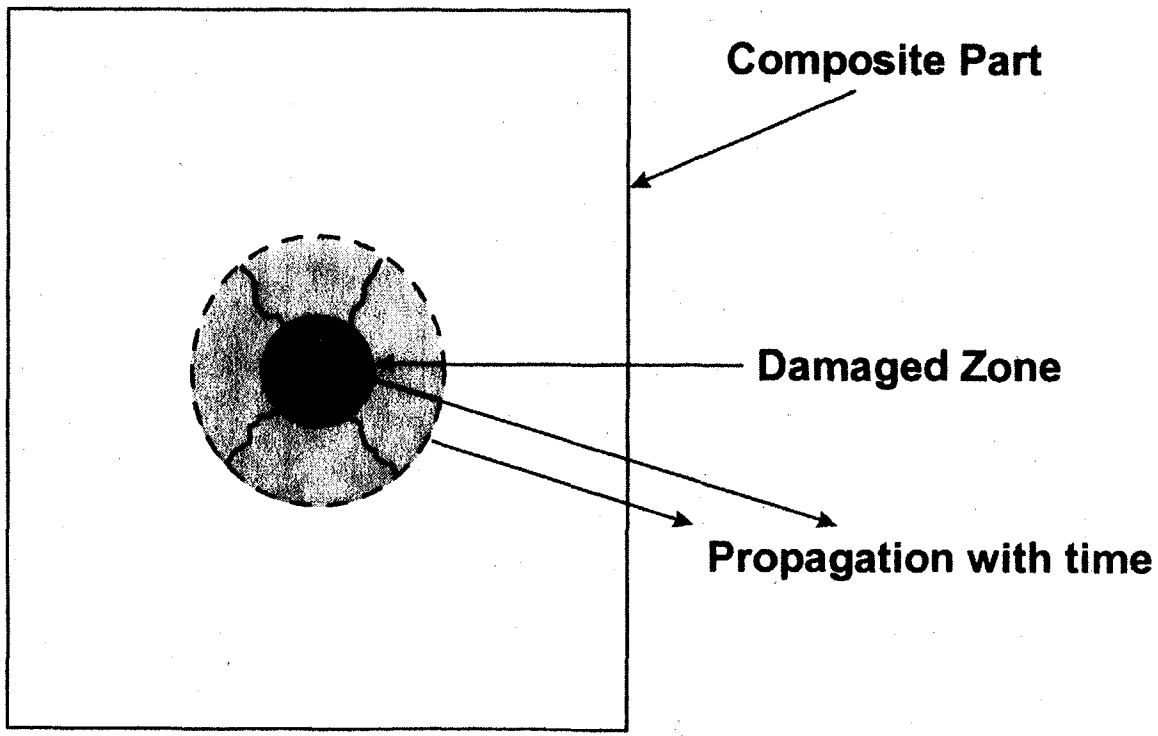



Ends-on oxidation of BN coatings
Penetration Depth (μm)
1.0E4
1000.0
100.0
10.0
1.0
0.1
0.1
1.0
10.0
100.0
Time (hrs)
700°C,Steam
900°C,Steam
1200°C,Steam
700°C,dry O2
900°C,dry O2

Summary

- Composite degradation by FOD, overstressing is a life limiting phenomenon

- Need to develop a standardized test for addressing FOD
  - test that measures the properties degradation (√)
  - test that measures the growth of the degraded zone



High Temperature Particle Filtration ORNL/DCC CRADA

Charlie Hall

Dow Corning Corporation

CRADA Objective

- Assess the feasibility of high temperature particulate filtration in the processes to produce dimthyldichlorosilane and to incinerate effluent gas from chlorosilane and chemical grade silicon production.

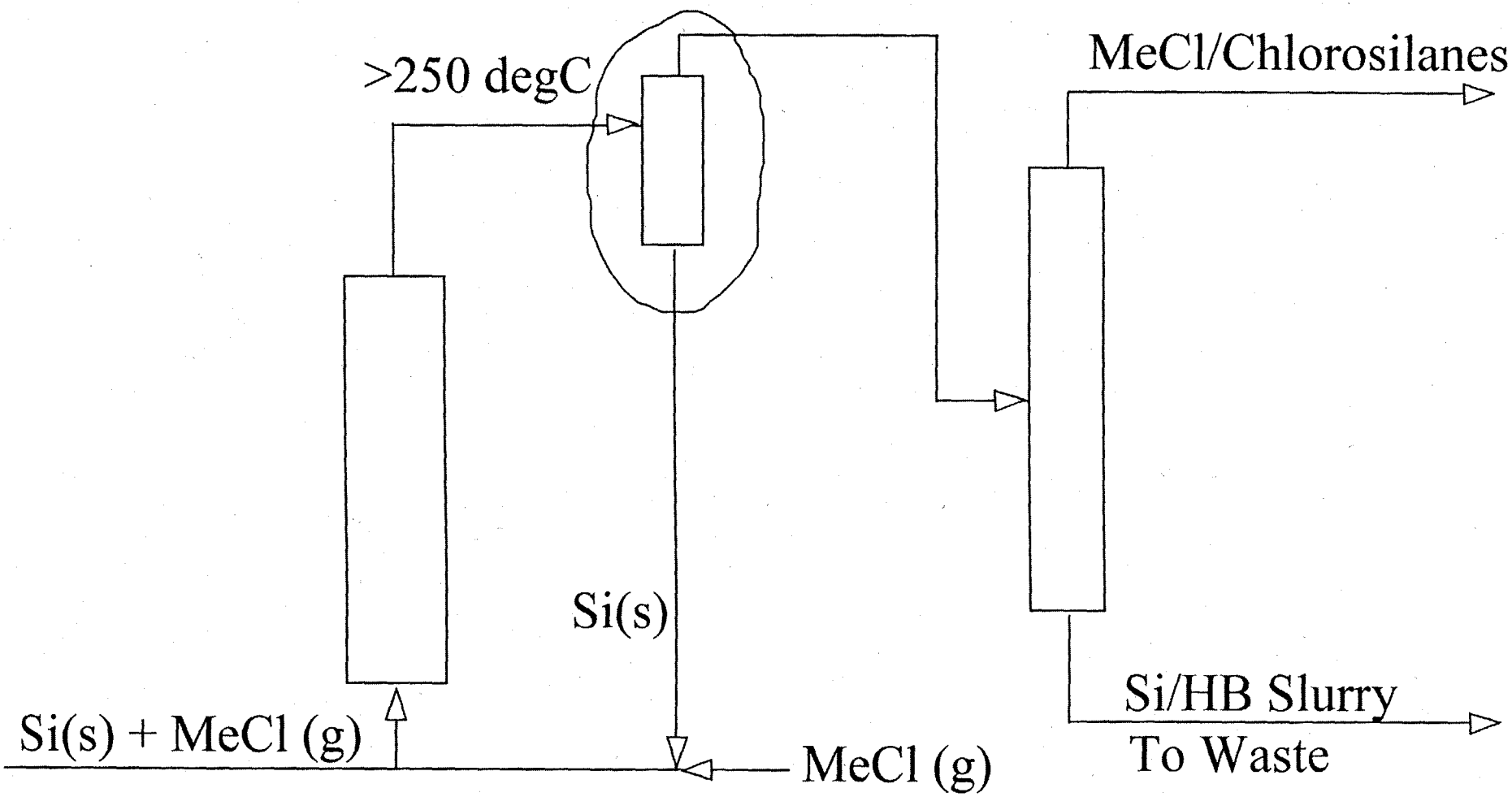
Dow Corning Carrollton Site Mission:

To be a high volume world class site, producing the highest quality methyl silicone products and intermediates in a safe, efficient, cost effective, and environmentally acceptable manner.

Silicon and Methyl Intermediates Mission:

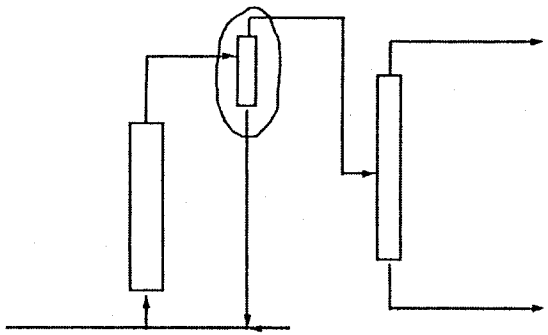
Identify and commercialize new technology to maintain Dow Corning's leadership in methyl intermediates production.

Fluidized Bed Reactor Process

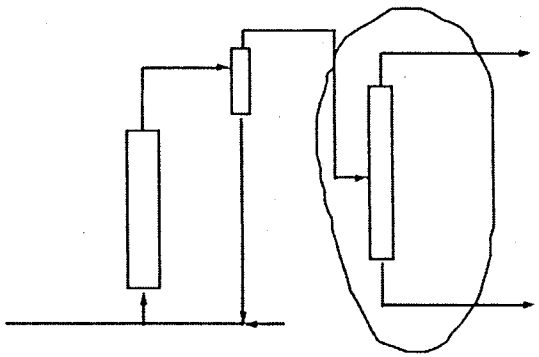


FBR Application

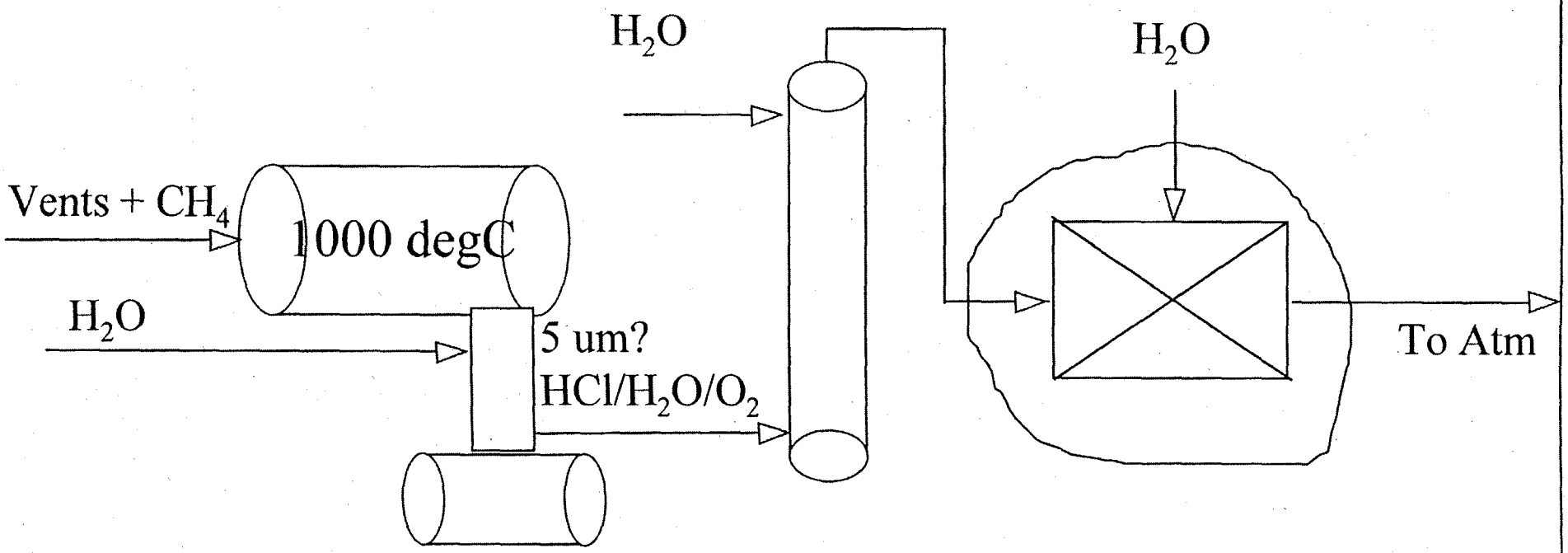
- Challenge
  - Reactive environment
    - localized high temperatures
    - cake formation time



- Benefits
  - Si Savings
  - Slurry Elimination
  - Separation Flexibility
    - heat recovery/integration
    - separation sequence

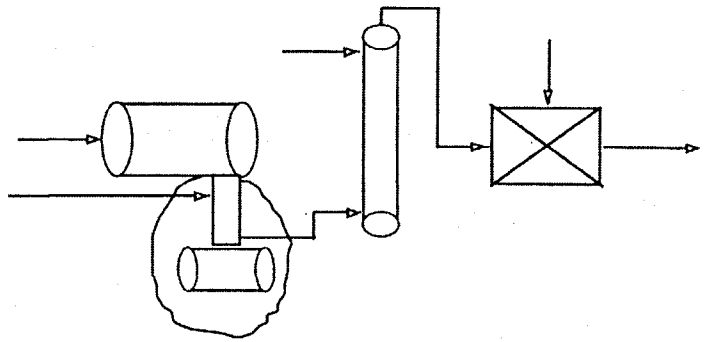


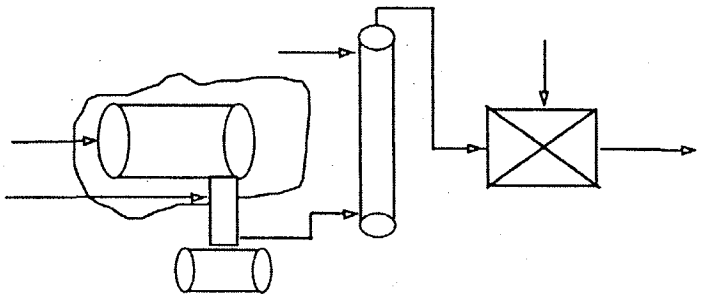
Thermal Oxidizer Process



Thermal Oxidizer Application

- Challenge
  - Particle size and characterization
  - Pressure Drop
- Benefits
  - Energy Recovery
  - Process Simplification





Benefits Summary

- FBR Process
  - >2 trillion Btu/yr nationally by 2010
  - 30 MM lb/yr waste reduction by 2010
  - Process flexibility (capital savings)
- Thermal Oxidizer Process
  - < 0.5 trillion Btu/yr nationally by 2010
  - Reduced hydraulic load to WWT
  - Prove technology for silicon smelters

Benefits Summary

- Silicon Smelters
  - > 10 trillion Btu/yr nationally by 2010
    - 280 MW capacity today
    - scaled at 5% growth
  - Increases attractiveness of fenceline plants

HSCT CMC COMBUSTOR

David Utah — GE Aircraft Engines
Gary Linsey — Pratt & Whitney
David Brewer — NASA LeRC

Presentation Outline

- Program History & Objectives
- Material Evaluations
  - Coupon Testing
  - Benchmark Testing
  - Rig Testing
  - Component Testing
- Summary

Program History

- NASA LeRC Contract Awarded Dec. 1991
  - GE Aircraft Engine & Pratt & Whitney
  - Integrated Technology Development Teams Established

- Components Evaluated
  - Combustor Programs (CMC's & Metal)
  - Exhaust Nozzle Program
  - Airfoils
  - Turbine Disks
  - Fan Containment

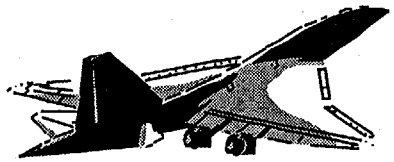
Enabling Propulsion Material (EPM)
CMC Combustor Program

Objectives

Develop and Demonstrate -

- CMC Material System, Design Concept, and Manufacturing Processes to Meet the HSCT Low Nox Combustor Requirements
  - Environmental (NOx < 5g/Kg)
  - Thermal and Structural
  - Economical
  - Durability (18,000 hours)
- Associated Structural and Life Prediction Tools
- Scaleable Component Manufacturing Capabilities That Meet HSCT Requirements
- Provide Sub-Components and Components for Performance And Durability Testing

Enabling Propulsion Material (EPM)
CMC Combustor Program



Material / Component Evaluations

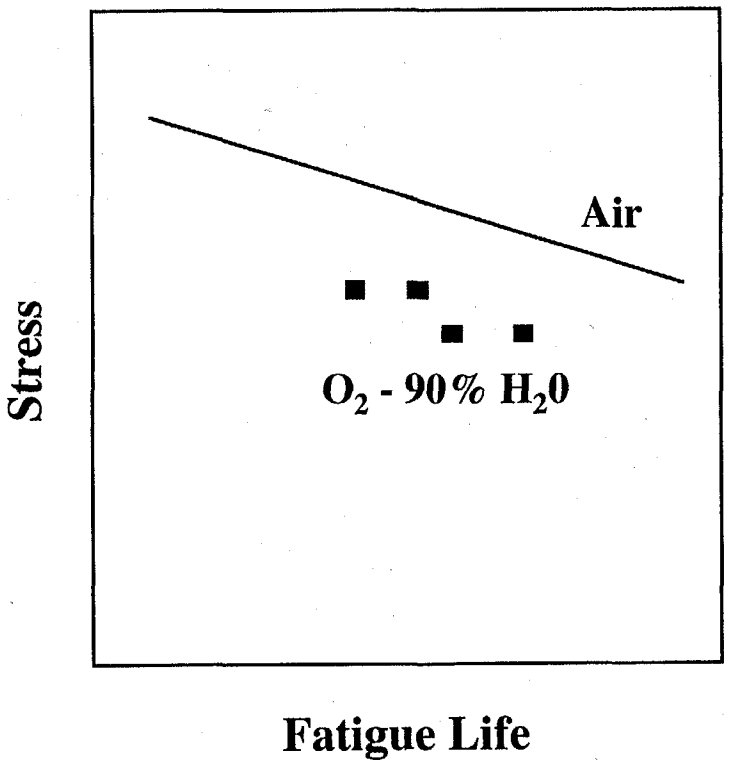
Coupon Testing → Benchmark Testing → Rig Testing → Components

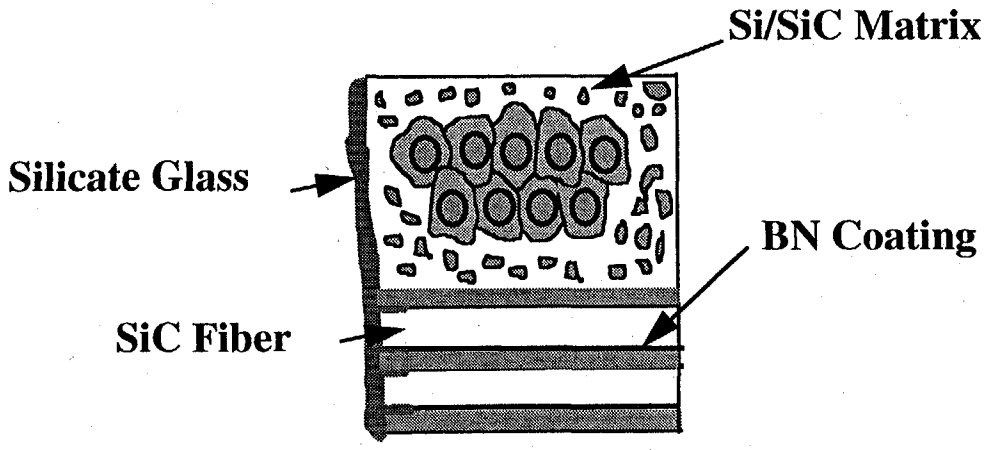
Coupon Testing

- Types of Testing
  - Thermal Expansion & Conductivity
  - Monotonic Tension & Compression
  - Interlaminar Tension & Shear
  - Creep / Stress Rupture
  - Fatigue
    - Low Cycle Fatigue
    - High Cycle Fatigue
    - Hold Time Fatigue (Air, O_2, O_2 + Water Vapor)

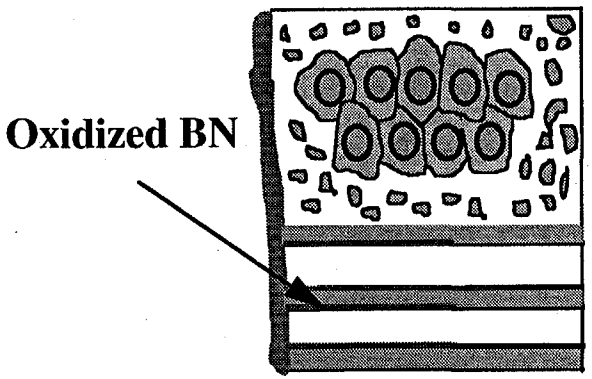
Enabling Propulsion Material (EPM)
CMC Combustor Program

Fatigue / Damage Mechanism in Air Versus Moisture





Air

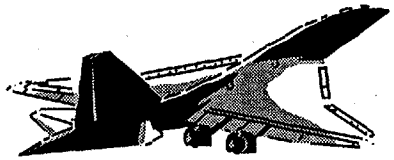


O_2 - 90% H_2O

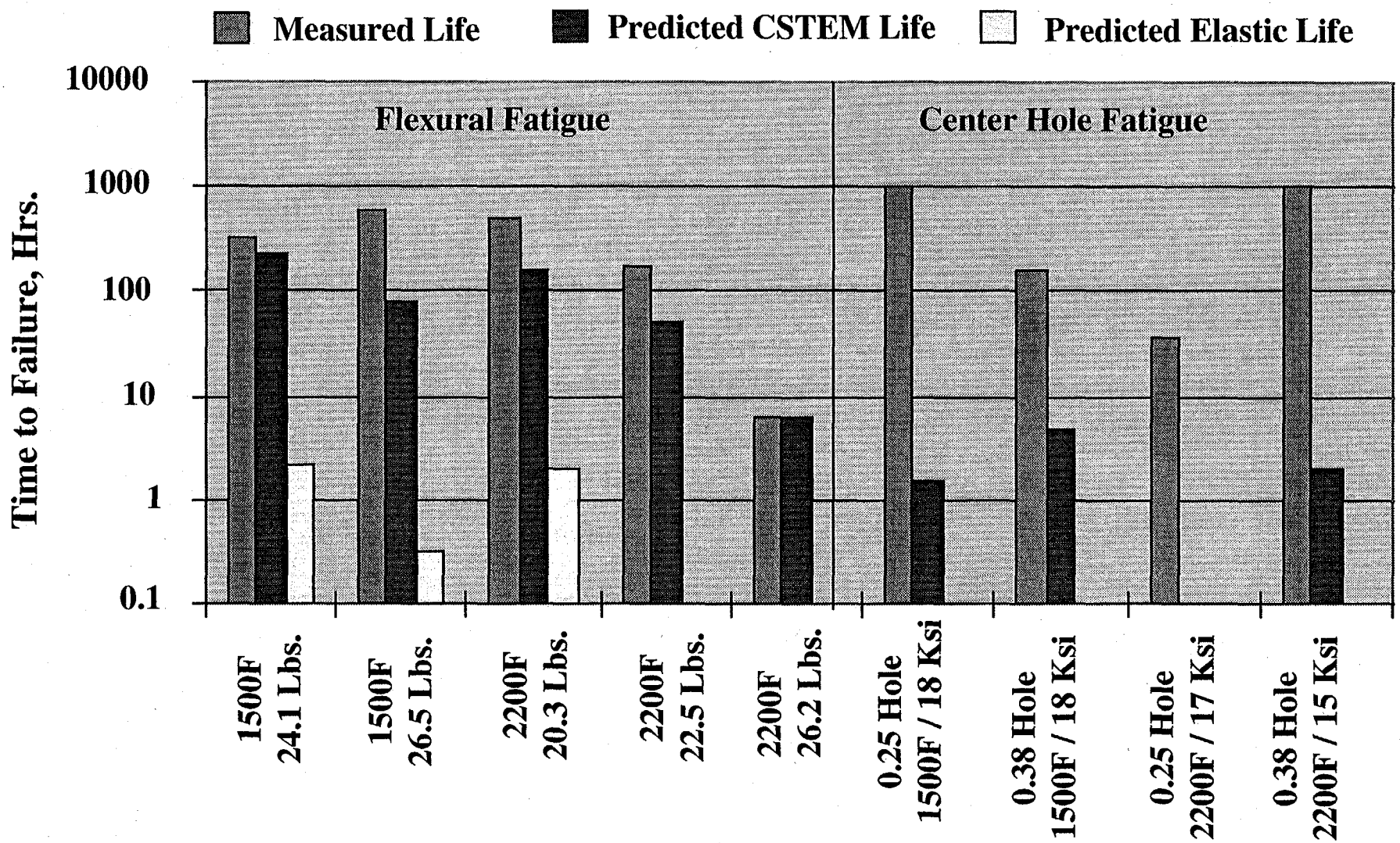
Benchmark Testing

- Evaluation of Life Prediction Techniques
- Two Simple Specimen Geometries Evaluated
  - Flexural Fatigue
  - Axial Fatigue with a Center Hole
- Stresses Predicted using Two Finite Element Codes
  - ANSYS (Elastic Analysis)
  - CSTEM
- Lives Calculated from Axial Fatigue Specimens

Enabling Propulsion Material (EPM)
CMC Combustor Program

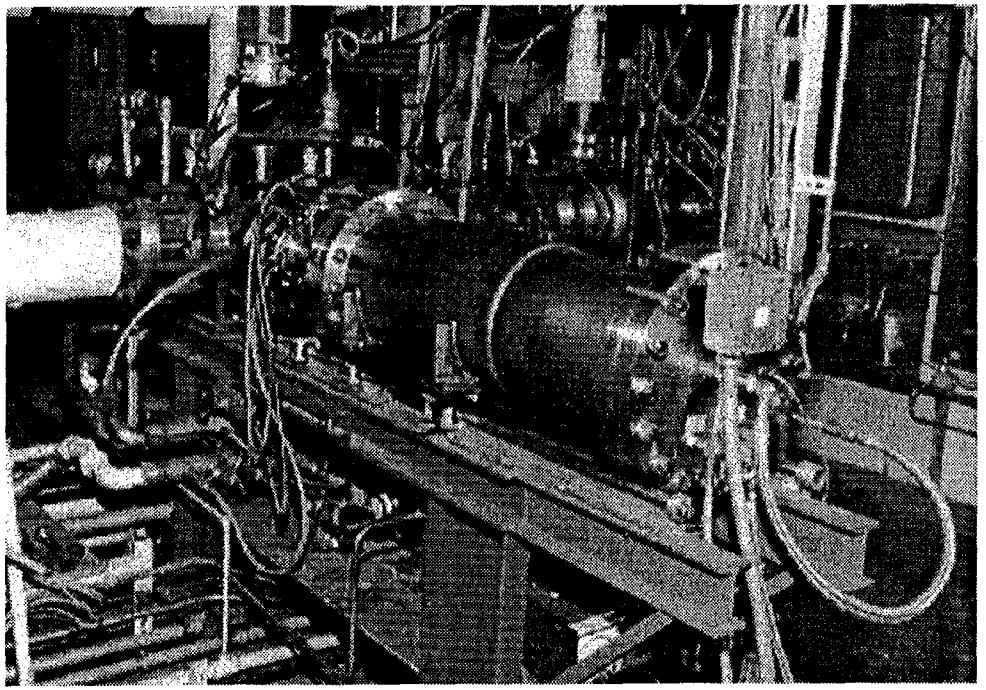


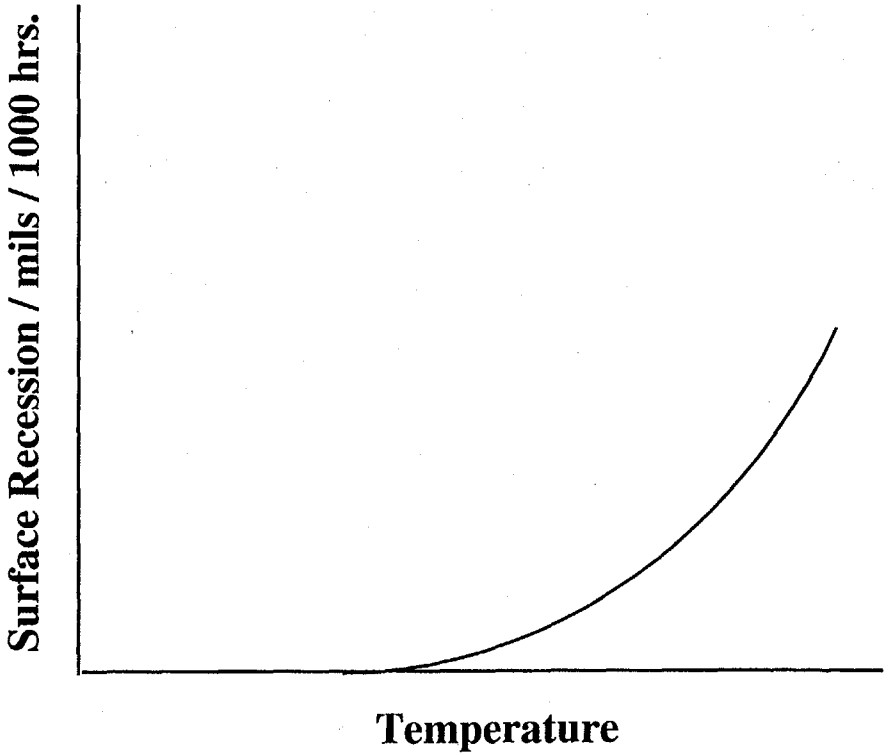
Life Predictions Results of Benchmark Test Specimens



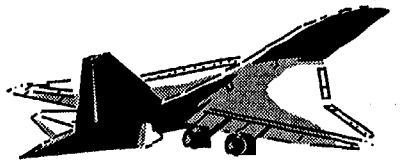
Enabling Propulsion Material (EPM)
CMC Combustor Program

High Pressure Burner Rig Testing

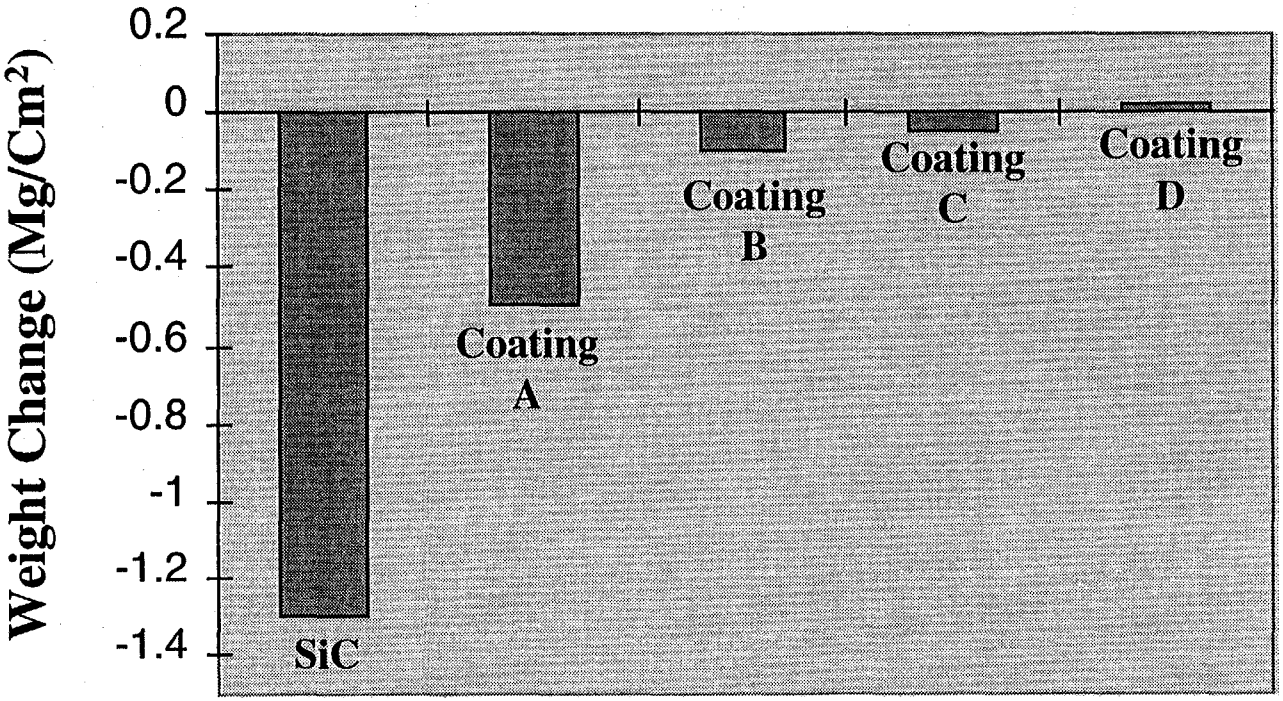




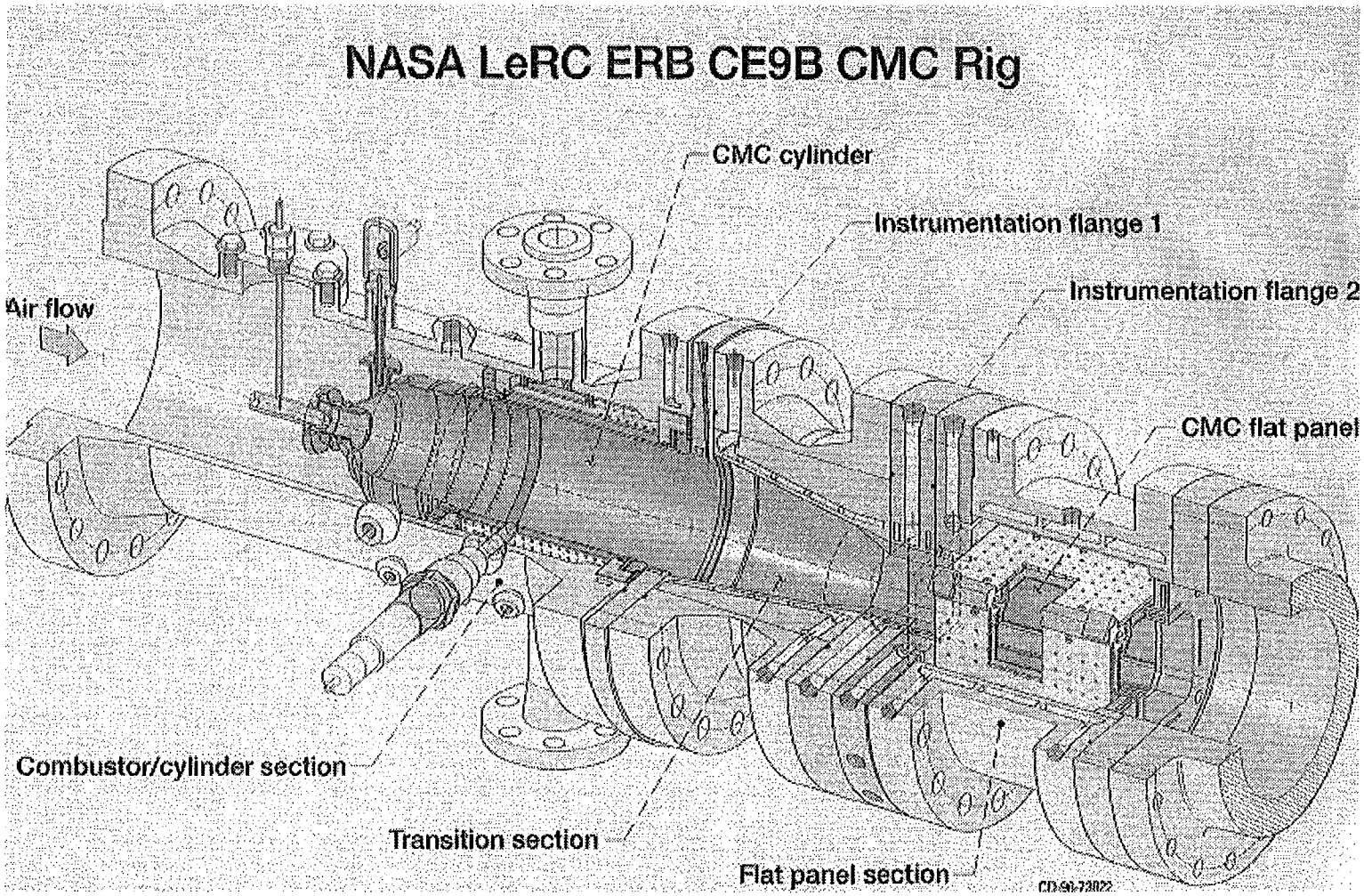
Enabling Propulsion Material (EPM)
CMC Combustor Program



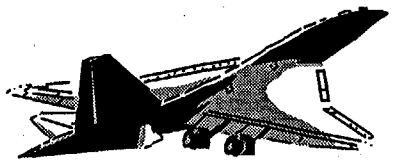
Environmental Barrier Coating Identified



Enabling Propulsion Material (EPM)
CMC Combustor Program



Enabling Propulsion Material (EPM)
CMC Combustor Program



Segment Test Summary

- 200 Hours of Rig Testing Completed

- Cylinder
  - Stresses and temperatures near expected operating conditions of engine
  - No cracking observed

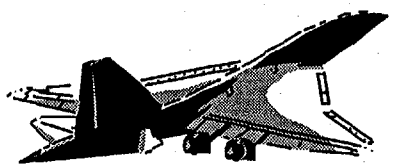
- Various Panels Evaluated
  - Materials / processes
  - Environmental barrier coatings
  - Delaminated panel
  - Notched panel

- NDE and Post-Test Evaluation Currently Underway

Component Testing

- Sector Test Program
  - Components are currently being fabricated
  - 60 degree arc
  - Testing scheduled for 3/98
- Annular Rig Program
  - Full annular / full size combustor
  - Preliminary design underway
  - Delivery scheduled for 6/00

Enabling Propulsion Material (EPM)
CMC Combustor Program



22nd Annual Conference on Composites

EPM CMC Combustor Session
- January 26 - 30 / Cocoa Beach
- Restricted Session
- 4 hour review

Topics
- Program Overview
- Component Design and Fabrication
- CMC Material Development
- Material Properties
- Micro-Structural Characterization
- Interface Development
- Environmental Barrier Coating
- CMC Modeling and Life Prediction
- Segment Testing

Enabling Propulsion Material (EPM)
CMC Combustor Program

Summary

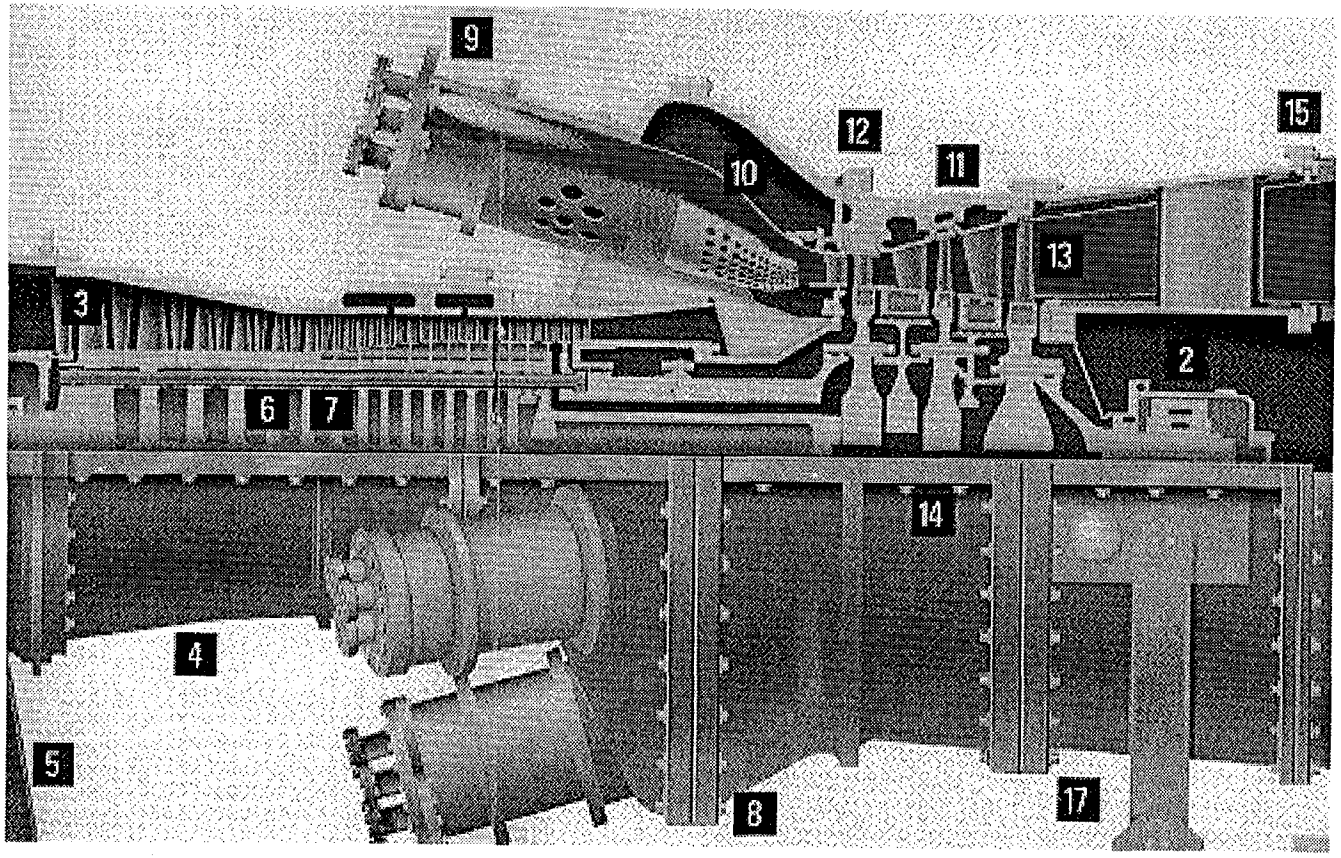
- Environmental Coupon Testing Needed for Combustor Applications
- Rig Testing Needed to Evaluate Surface Recession
- Additional Efforts Required on Life Prediction Techniques
- Component Testing Will Be Conducted to Evaluate EPM Material

Case Study - CFCC Shroud for Industrial Gas Turbines

October 7, 1997

Paul DiMascio
GE Power Generation Systems

Color Xsection of turbine



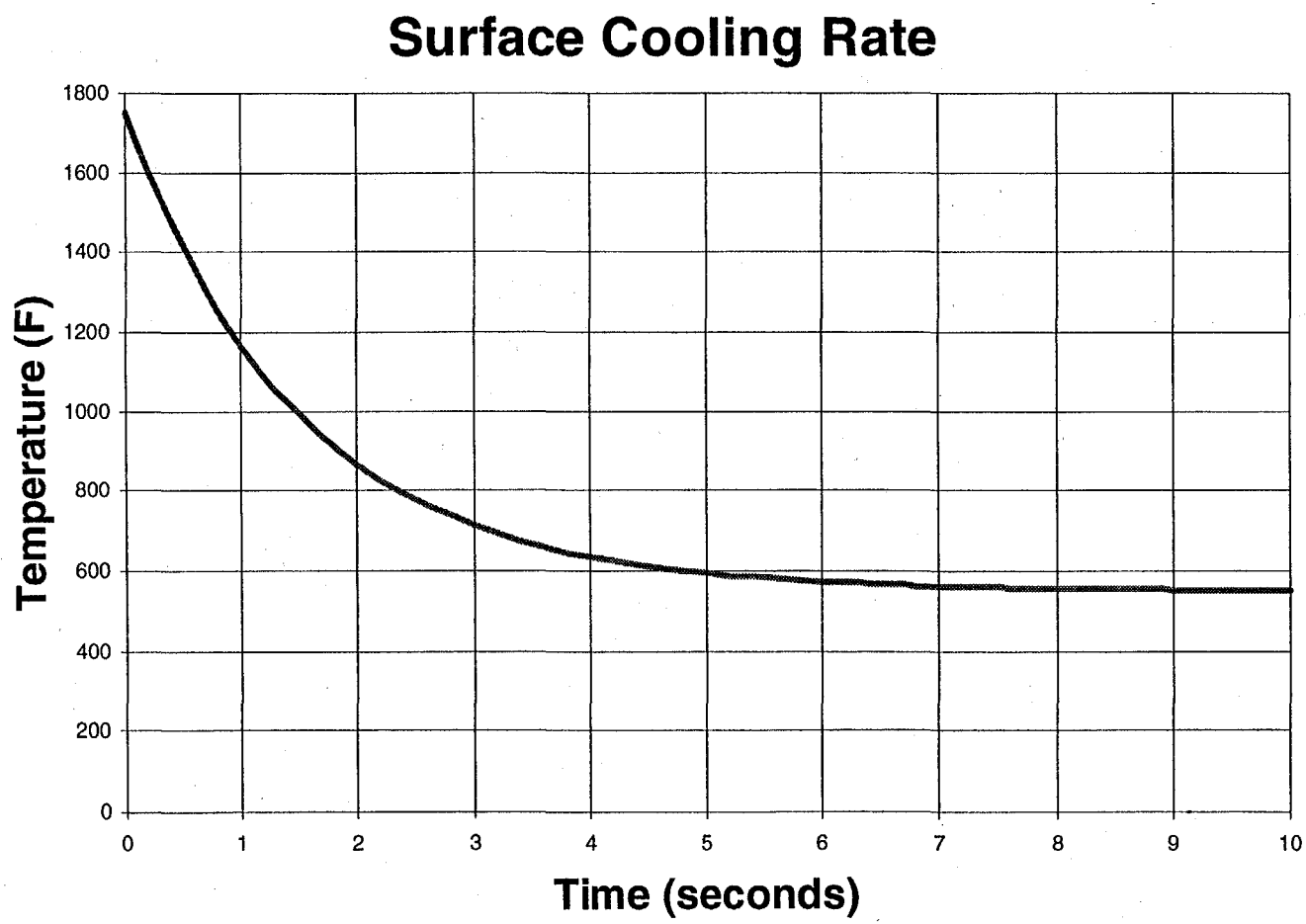
Environment

- Parts must last 24,000 to 72,000 hours
- Material data should cover most of that range.
- We are still testing metals that have been in use for years.
- A creep test running for 6 years just failed and others are still running.

Stationary and Rotating Parts

- Nozzles and Shrouds are primarily thermally loaded (low cycle fatigue).
  - Also have lesser aero, structural and vibratory loads.
- Rotating parts add centrifugal loading.
- Turbine trips, rapid load changes and foreign object damage (FOD)

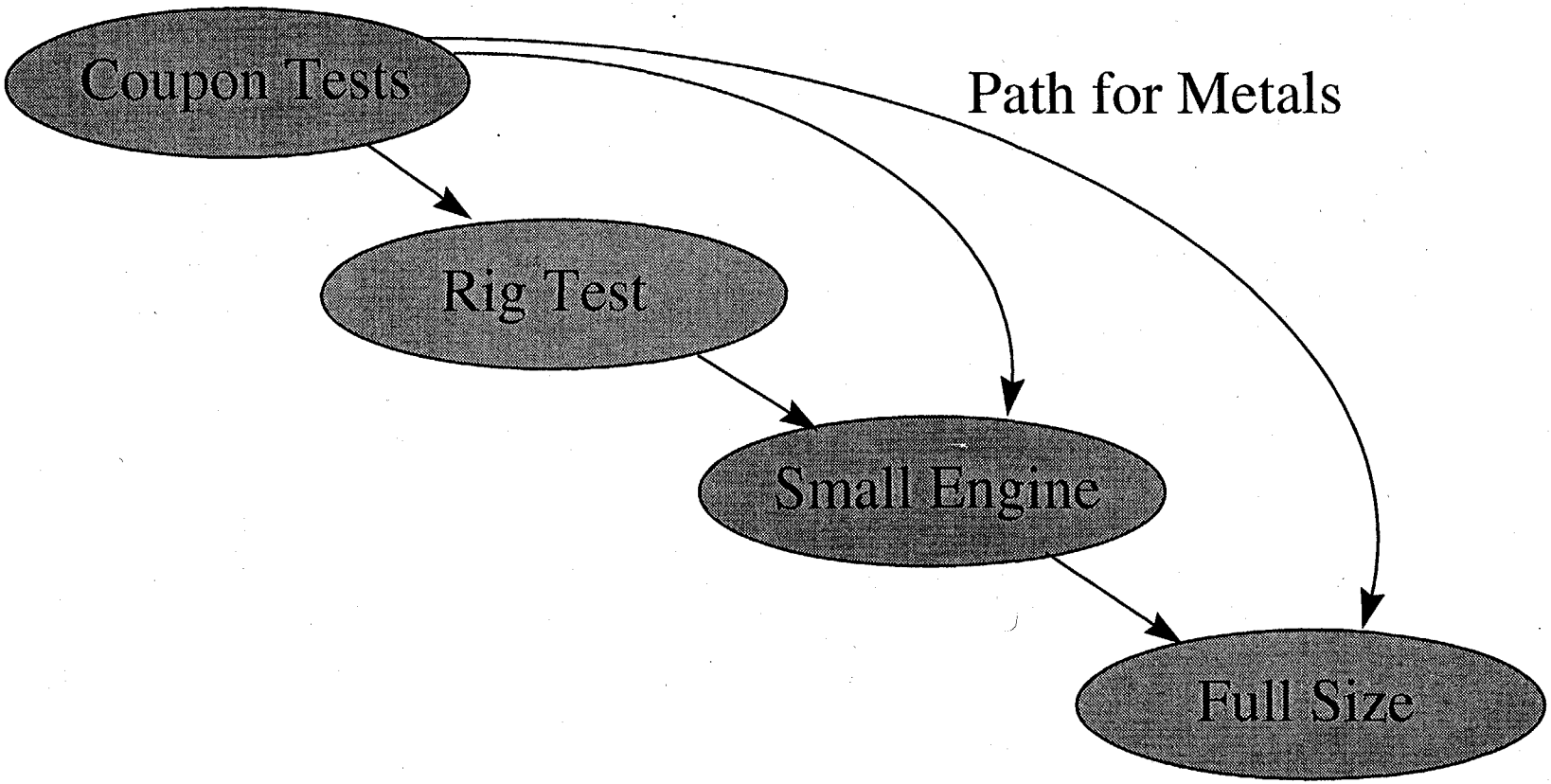
Turbine Trip



How Much Data?

- Physical properties - 12 specimens
- Creep - 60 - 200 specimens with lives out to 1/2 the design life
- Fatigue - 60 specimens with lives from 100 cycles to 10^7 cycles
- Oxidation/corrosion data to 24000 hours
- FCGR, Fracture Toughness

Path to Release New Material



Advantages of Combustion Rig

- Matches temperature and pressure exactly
- Creates same chemical environment (including corrosive atmosphere)
- Similar gas velocities and pressure fluctuations are possible
- Can duplicate thermal shock conditions

Disadvantages of Comb. Rig

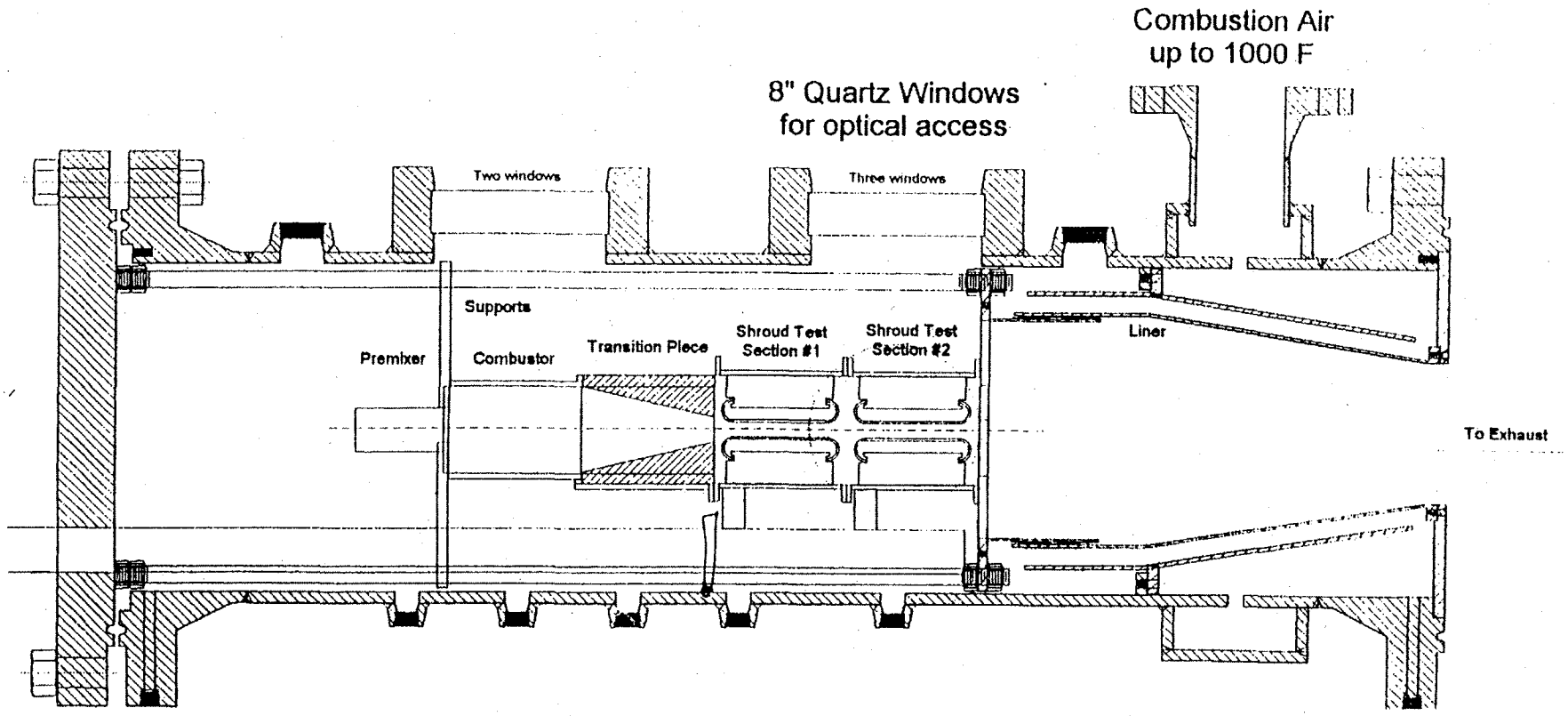
- No moving parts
- Velocity gradients don't match turbine
- Possibly different mechanical constraints
- Don't test many parts
- Expensive to run long duration tests

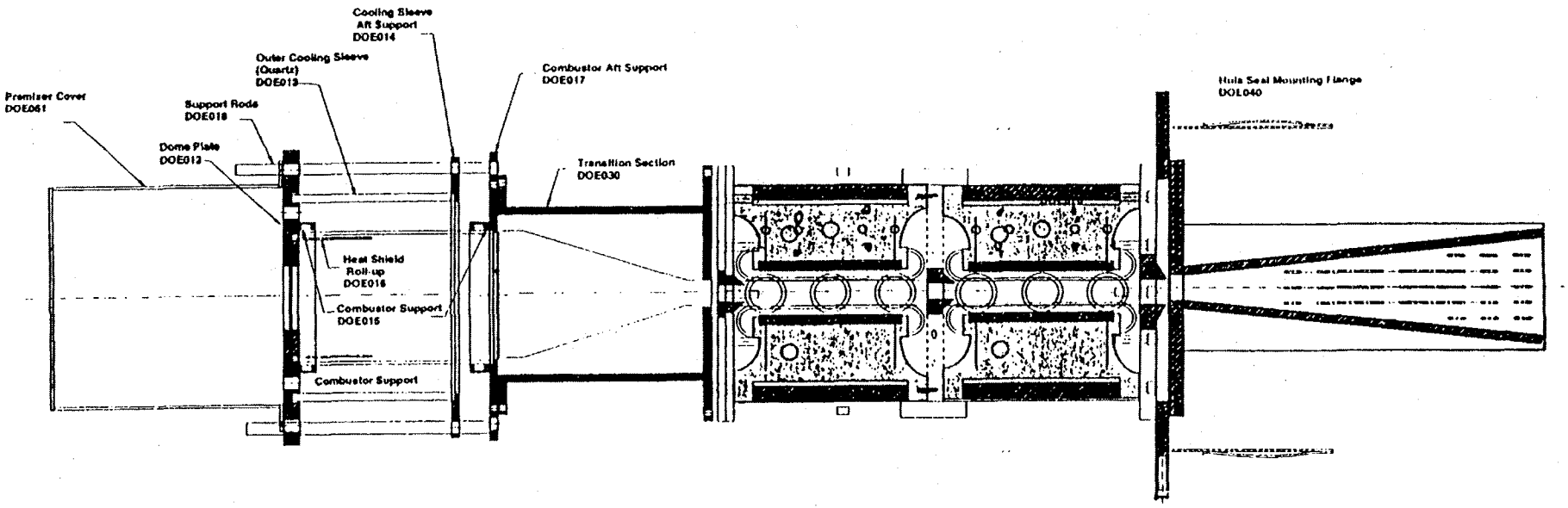
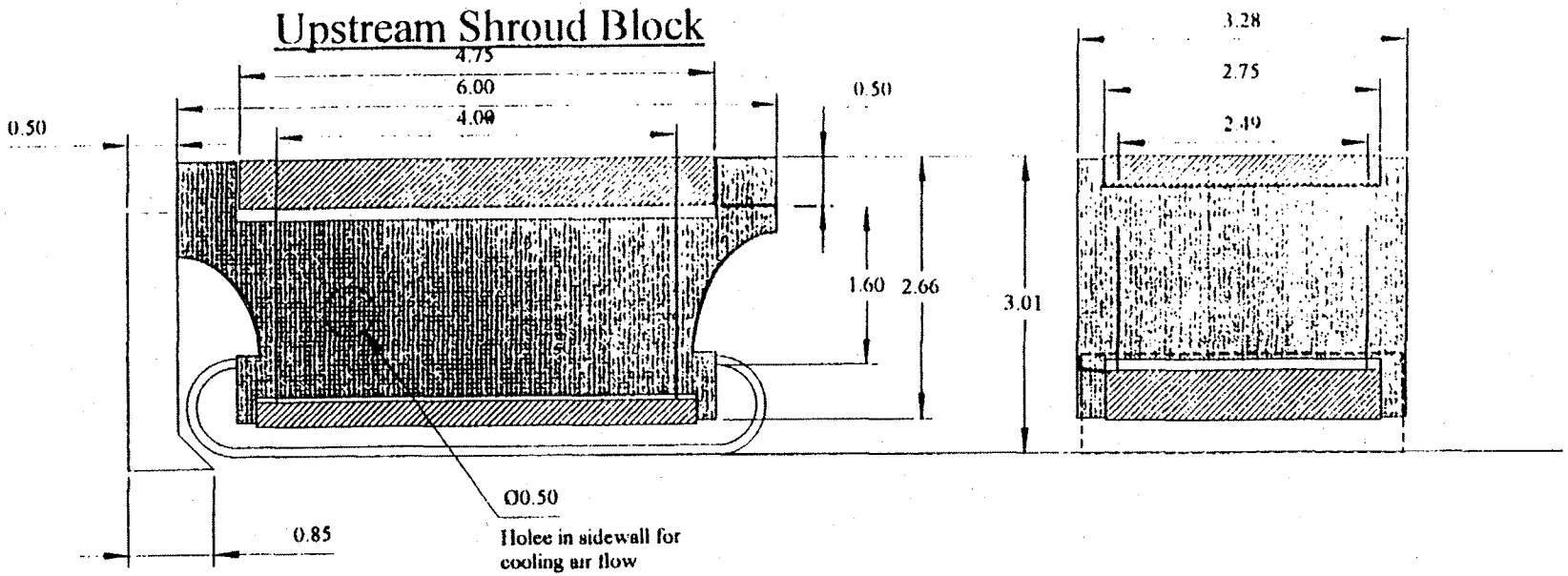
Goals of Rig Test

- Exceed the temperature and thermal gradients of target turbines
- Test thermal shock and pressure dynamics to levels greater than expected in turbines
- Run enough durability to convince designers to persue real engine test (200 hours of abusive temperature and cycles)

Layout of Ceramic Test Rig in 16" diameter vessel

- Two sets of windows permit viewing of combustor and shroud elements
- The test section is supported off the upstream flange assembly
- It is very easy to access the hardware for changes between tests




Cooling Sleeve
Aft Support
DOE014
Outer Cooling Sleeve
(Quartz)
DOE013
Premixer Cover
DOE061
Support Rods
DOE018
Dome Plate
DOE012
Combustor Aft Support
DOE017
Transition Section
DOE030
Hula Seal Mounting Flange
DOL040
Heat Shield
Roll-up
DOE016
Combustor Support
DOE015
Combustor Support

Upstream Shroud Block