CONF-8910245--Summs.

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**Program
Review
Meeting**

Summary Report

ADVANCED HEAT EXCHANGERS

July 1990

Ramada Renaissance Hotel
Herndon, Virginia
October 11-12, 1989

U.S. Department of Energy
Office of Industrial Programs

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Office of Industrial Programs
Washington, DC 20585

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INTRODUCTION

On a semiannual basis a review meeting of the U.S. Department of Energy's Advanced Heat Exchangers Program is held with the objective of reviewing ongoing and recently completed project activities. During the meeting, personnel from industrial contractors and National Laboratories who have been funded to develop advanced heat exchanger technology by the U.S. Department of Energy, Office of Industrial Programs present technical aspects of their projects. The projects generally deal with mid to high temperature waste heat recuperation and other subject areas of heat exchange. Each presentation is followed by a discussion period during which the presenters field questions from the meeting attendees.

This report documents the most recent meeting, held October 11-12, 1989. It is composed primarily of a series of short sections addressing the individual projects. Each section includes presentation slides of the project and a summary of questions and answers that followed. An appendix of meeting attendees is also included.

Advanced Heat Exchangers Program Review

October 11 & 12, 1989
Ramada Renaissance Hotel
Herndon, Virginia

AGENDA

Wednesday
October 11, 1989

8:00 a.m.	Registration Coffee & Rolls	
9:00 a.m.	Introduction & Program Overview	Scott Richlen *DOE*
9:15 a.m.	Development of Advanced Composite Ceramic Heat Exchanger System	Don Hindman *Babcock & Wilcox*
9:45 a.m.	Composite Heat Exchanger Materials Support	Irv Federer *ORNL*
10:00 a.m.	High Pressure Heat Exchange System (HiPHES) Overview	Gary Peterson *DOE-ID*
10:10 a.m.	Preliminary Design of a High Pressure, High-Temperature Ceramic Air Heat Exchanger	Bruce Harkins *Solar Turbines*
10:40 a.m.	Break	
11:00 a.m.	HiPHES Development of a Convective Steam Reformer	Joe Williams *Stone & Webster*
11:30 a.m.	High Pressure Heat Exchanger	Bill Parks *Babcock & Wilcox*
12:00	Luncheon	Grand Ballroom
1:30 p.m.	Current and Planned HiPHES Materials Support	Irv Federer *ORNL*
1:45 p.m.	ORNL Overview of Materials Support Work	Mike Karnitz *ORNL*
2:00 p.m.	Methodology Studies for Continuous-Fiber Ceramic-Matrix Composite	Mike Karnitz *ORNL*
2:20 p.m.	Gelcasting of Monolithic Ceramics and Chopped Fiber Reinforced Composites	Albert Young *ORNL*
2:40 p.m.	Break	
3:00 p.m.	Scale Up of a Process for Manufacture of Beta Silcon Carbide Powder	Peter Shaffer *Advanced Refractory Technologies*

AGENDA (Continued)

Wednesday
October 11, 1989 (cont'd)

3:30 p.m.	Development of a Manufacturers Capability to Fabricate Whisker-Toughened Ceramic Tubes	Mike Karnitz *ORNL*
3:45 p.m.	Biological Effects of Silcon Carbide Whiskers	Neil Johnson *Inhalation Toxicology Research Institute*
4:30 p.m.	Closing Remarks	Scott Richlen *DOE*
4:45 p.m.	Adjourn	

Thursday
October 12, 1989

8:00 a.m.	Coffee & Rolls	
8:30 a.m.	Enhanced Shell-and-Tube Heat Exchangers for Industry	Art Bergles *Rensselaer Polytechnic Institute*
9:00 a.m.	Enhanced Tubes for Steam Condensers	Ralph Webb *Pennsylvania State University*
9:30 a.m.	Assessment of Strength Limiting Flaws in Heat Exchanger Components	John Bower *Babcock & Wilcox*
10:15 a.m.	INEL Support for Assessment of Strength Limiting Flaws in Ceramic Heat Exchanger Components	Walt Reuter *INEL*
10:45 a.m.	Break	
11:00 a.m.	Fouling Probe Overview	Gary Peterson *DOE-ID*
11:15 a.m.	Diagnostic Tests in a Flue Gas Stream - Fouling Probe	Manohar Sohal *INEL*
11:30 a.m.	Corrosion Probe Testing and Development	Irv Federer *ORNL*
11:45 a.m.	Concluding Remarks	Scott Richlen *DOE*
12:00	Adjourn	

PROGRAM OVERVIEW

Scott L. Richlen
DOE

PROGRAM OVERVIEW

PRESENTATION OUTLINE

TITLE

Its been about two years since the last Advanced Heat Exchangers Program review. The purpose of this presentation is to overview the program to bring the meeting attendees up to date.

PROGRAM TIME LINE

The various R&D topics addressed over the past years are shown on this slide. Note that the R&D covers a wide span of interests. During the meeting, current projects associated with these R&D topics will be reviewed. A bibliography of reports from completed projects is available from the Program Office. Reports can be ordered from NTIS.

TWO YEARS AGO...

Status of the Program at the last review.

TODAY...

A substantial portion of the Advanced Heat Exchanger Program's research deals with developing various support technologies for high temperature (ceramic) recuperators.

TODAY...

Two recently opened areas of research are related to process heat exchange. These are the High Pressure Heat Exchange Systems (HiPHES) projects and the projects to test enhanced tube surfaces for boilers and condenser applications.

TOMORROW...

The Program will place increased emphasis on R&D of heat transfer and design of heat exchangers while lessening the emphasis on materials research.

REVIEW - FIRST DAY

Both the Advanced Heat Exchanger and the HiPHES projects presentations will be followed by presentations covering the materials research being conducted in support of each of these project areas. The ORNL Materials Research not directly tied to individual projects will then be reviewed followed by a discussion of research on the potential health hazard of ceramic whiskers.

REVIEW - SECOND DAY

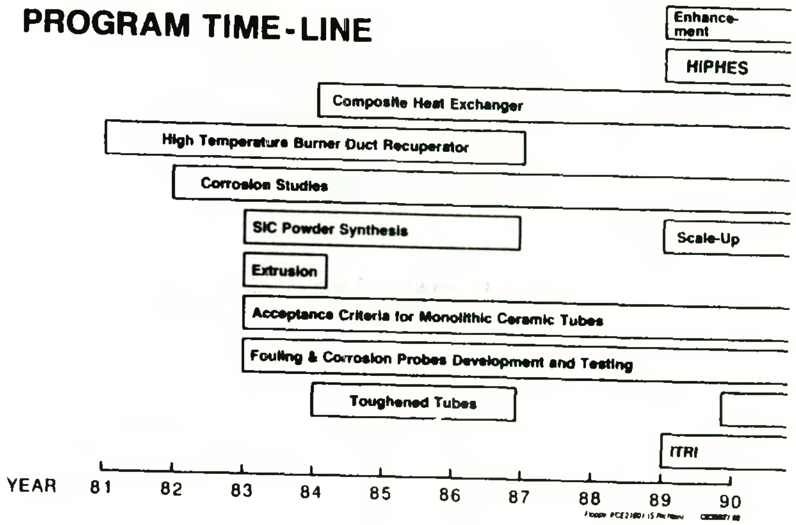
A discussion of the university tests of enhanced tube performance will be followed by presentations of the R&D results from the studies of strength limiting flaws. The final presentations of the meeting will cover the development and testing of fouling and corrosion probes.

ADVANCED HEAT EXCHANGERS

PROGRAM REVIEW

OCTOBER 11 & 12, 1989

PROGRAM TIME-LINE



TWO YEARS AGO...

- **Completed or Completing HTBDRs, SiC Powder Synthesis, Toughened Tubes, Fouling Probe Design and Lab Tests, HIPHES Assessment**
- **In the Middle of Ph II SLF, Ph II Composite Hx, Corrosion Studies**
- **Contemplating Enhancement Research, New Wet-Forming Process, Advanced Corrosion Probe, HIPHES**

TODAY...

- **High Temperature Recuperator**
  - Continued Corrosion Studies
  - Ph III SLF
  - Completion of Powder Synthesis Scale-Up
  - Continuation of Gel-Casting
  - Initiation of Toughened Tube Scale-Up

TODAY...

- **Process Heat Exchange**
  - \- Continuation of HiPHES
  - \- Completion of Enhancement
- **Fouling & Corrosion Probes**
  - \- Continued Testing and Development

TOMORROW...

- **Heat Transfer**
- **HIPHES**
- **Recuperators**
- **Fouling**
- **New Designs**

REVIEW - FIRST DAY

- **Advanced Heat Exchanger**
- **HIPHES**
- **ORNL Materials Research**
- **ITRI Whisker Study**

REVIEW - SECOND DAY

- **Enhancement**
- **Strength Limiting Flaws**
- **Fouling and Corrosion Probes**

DEVELOPMENT OF ADVANCED COMPOSITE CERAMIC HEAT EXCHANGER SYSTEM

Don L. Hindman
Babcock & Wilcox

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGERS PROGRAM
PROJECT DESCRIPTION

TITLE: Development of an Advanced Composite Ceramic Heat Exchanger System

COOPERATIVE AGREEMENT NO.: DE-FC07-89ID12868, through DOE-ID

CONTRACTOR: Babcock and Wilcox (B&W)
Box 11165
Lynchburg, VA 24506-1165

PRINCIPAL INVESTIGATOR: Don L. Hindman, 804-522-5825

OBJECTIVE: Develop a ceramic fiber composite heat exchanger for efficient and economic recovery of heat from industrial corrosive and fouling gas streams.

TECHNICAL APPROACH: Select host site and conduct energy audit. Refine the preliminary design of the prototype heat exchanger developed in Phase 2. Verify composite material performance by laboratory corrosion tests and by exposure to site flue gases. Verify integrity of tube-tube sheet seals in laboratory tests simulating pressures and temperatures anticipated in actual use. Fabricate several full scale composite tubes for use in the prototype heat exchanger and characterize their density, porosity, and permeability. Finalize design to meet host site conditions. Construct and install a prototype heat exchanger at the host site facility. Operate the prototype in a field test over a period of approximately six months. Analyze thermal efficiencies, materials performance, and economics.

SYSTEM DESCRIPTION: The preliminary design of the heat exchanger includes the following major items: ceramic composite outer tubes closed on one end, ceramic inner tubes open on both ends, metallic tubesheet, and compliant ceramic seals.

The outer tubes will contain DuPont PRD 166 continuous ceramic fiber in a matrix of zirconia or alumina. (Both types of matrix material have provided high strength, corrosion resistant composites in laboratory tests.) The outer tubes will have a circular cross section and will be suspended vertically from the tubesheet. The inner tubes will be mounted inside the outer tubes in a bayonet style configuration.

The tubesheet will be air-cooled and will be protected from the corrosive flue gases by a ceramic insulating barrier material. Seals will be fabricated from a low density ceramic containing discontinous fiber. They will also be protected by the insulating barrier material from the flue gases.

The prototype heat exchanger will be integrated into the flue of a hazardous waste incinerator at the DuPont Experimental Station in Wilmington, DE. It will be installed in a slipstream configuration and will be connected to the flue through isolation valves which will allow the heat exchanger to be taken off-line without shutting down the incinerator. After passing through the heat exchanger flue gases will be passed through a quench system. They will then be returned to the process downstream from a spray dryer which is used to remove particulates. The goal is to return the gases to the process in a state identical to that of flue gases passing through the spray dryer. To minimize impact on normal incinerator operations the recovered heat will be dumped to atmosphere.

CURRENT STATUS (10/89): Work on four major tasks which are to be completed prior to a decision point in March 1990 is continuing according to schedule. These four major tasks are: selection of a host site application and signing of a host site agreement, performance of a market study, economic analysis of the host site installation, and verification of materials performance prior to fabrication of the prototype.

ACCOMPLISHMENTS: An agreement to use a DuPont hazardous waste incinerator as host site for the field test has been obtained. Host site operating parameters have been quantified for use in designing the heat exchanger.

Composite samples have been fabricated and are presently being tested for corrosion resistance in lab tests at ORNL. Other samples are being fabricated for testing in the flue of the host site prior to construction of the heat exchanger.

Preparations for lab tests of the integrity of seals of a full scale tube assembly at temperature are underway. Several full size composite tubes are being fabricated to demonstrate the feasibility of scaling up the manufacturing process.

FUTURE WORK: Complete tests of composite samples in lab tests and in flue at host site. Complete tests of tube-tube sheet seal integrity. Complete market study and signing of formal host site agreement. Conduct energy audit of host site and adapt preliminary heat exchanger design to host site conditions. Demonstrate feasibility of manufacturing full scale composite tubes.

SUMMARY: Work toward development of a ceramic fiber composite heat exchanger is proceeding according to schedule. A field test of a prototype unit will be performed in the third year of the present program phase.

BIBLIOGRAPHY:

1. Research and Development of a Ceramic Fiber Composite Heat Exchanger, Phase 1 Final Report, DOE/ID/12536-1, Babcock and Wilcox Company, Lynchburg, VA, July 1985, (ITAR Distribution Restricted).

2. DeBellis, C. L. Kneidel, K. C., "Thermal and Fluid Design of a High Temperature Ceramic Fiber Composite Heat Exchanger," *24th National Heat Transfer Conference and Exhibition, Pittsburgh, PA, August 9-12, 1987.*

3. Research and Development of a Ceramic Fiber Composite Heat Exchanger, Phase 2 Final Report, DOE/ID/12536-2, Babcock and Wilcox Company, Lynchburg, VA, September 1989, (ITAR Distribution Restricted).

ADVANCED HEAT EXCHANGER PROJECT
(AHX)

PROGRAM GOAL

- DEVELOP AND FIELD TEST A CERAMIC COMPOSITE BASED HEAT EXCHANGER FOR USE IN CORROSIVE WASTE HEAT RECOVERY

PROGRAM PARTICIPANTS

DOE OFFICE OF INDUSTRIAL PROGRAMS
BABCOCK & WILCOX COMPANY
E. I. DUPONT de NEMOURS AND COMPANY

ADVANCED HEAT EXCHANGER PROJECT

PHASE 1
IDENTIFY CRITICAL NEEDS

PHASE 2
RESOLVE CRITICAL NEEDS

PHASE 3
FIELD TEST PROTOTYPE HEAT EXCHANGER

PHASE 1

PAPER STUDY WHICH IDENTIFIED CRITICAL NEEDS

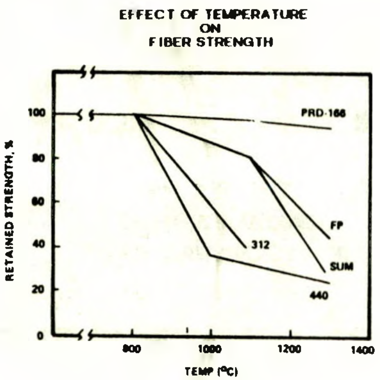
- MATERIALS DEVELOPMENT
- PREFORM DEVELOPMENT
- JOINING AND SEALING

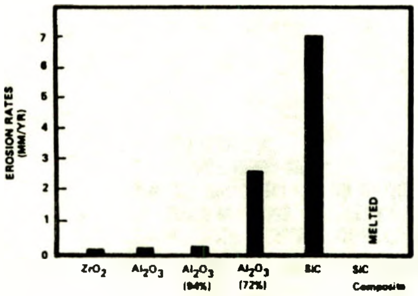
PRELIMINARY DESIGN OF A LOW PRESSURE RECUPERATOR

PHASE 2

RESOLUTION OF CRITICAL NEEDS IDENTIFIED IN PHASE 1

- OXIDE/OXIDE CERAMIC COMPOSITE SYSTEMS
- FILAMENT WINDING OF TUBULAR PREFORMS
- SOL-GEL PROCESSING FOR MATRIX DENSIFICATION
- COMPLIANT FIBER SEALS
- DESIGN UPDATED

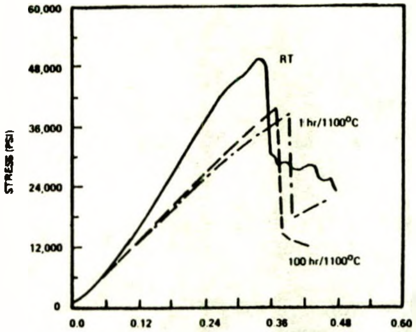

EFFECT OF TEMPERATURE
ON
FIBER STRENGTH



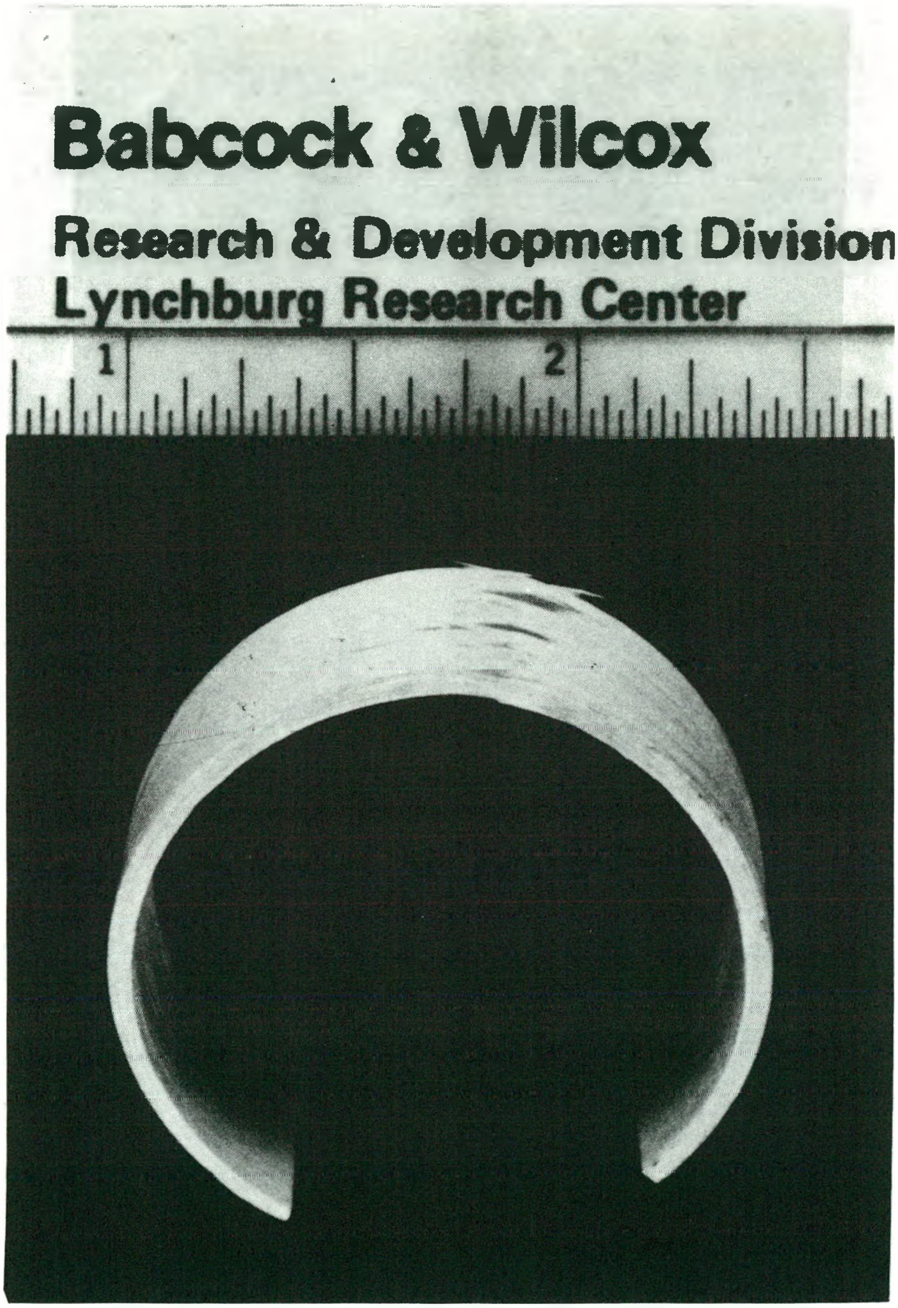
Relative Erosion Rates for Simple Oxides
Compared to Silicon Carbide Materials

COMPOSITE PROPERTIES

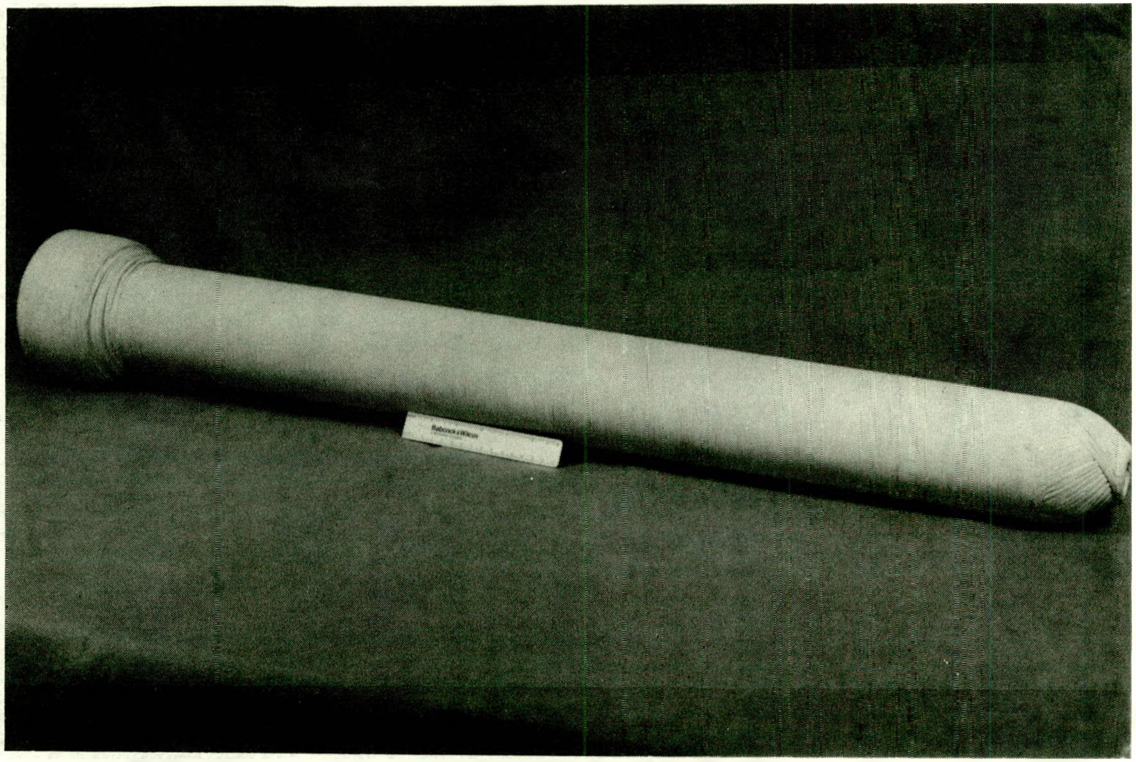
SYSTEM	C-RING STRENGTH (KSI) RT	HT
PRD166/ZIRCONIA	46-53	30-35 (1100C)
PRD166/ALUMINA	17-24	20-25 (1000C)
SUMITOMO/ZIRCONIA	23	19 (1000C)
SUMITOMO/ALUMINA	33	24 (1000C)



Stress-Strain Curve of a Ceramic Composite Showing
Non-Brittle Fracture Behavior



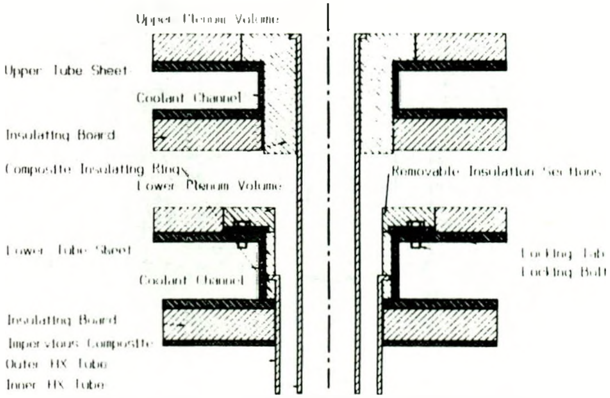
TYPICAL C-RING FRACTURE SPECIMEN



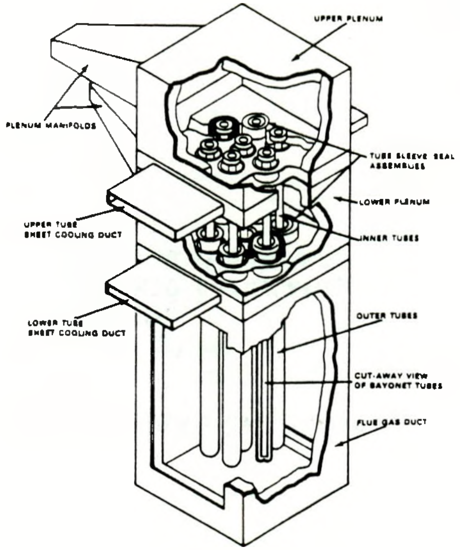
FULL SCALE CERAMIC COMPOSITE TUBE

CORROSION RESISTANCE

TEST	RESULT
Na_2CO_3	TESTED 600 HOURS AT 2100F vs. 48 HOURS FOR SiC
REFUSE	TESTED 1040 HOURS AT 1600F - NO CORROSION
REFUSE	TESTED 1030 HOURS AT 2150F - MINOR REACTION



TOP ACCESS HEAT EXCHANGER CROSS SECTION



PROTOTYPE HEAT EXCHANGER
CUTAWAY VIEW

PHASE 3 - TEN MONTH ACTION PLAN

- * MARKET SURVEY
- * HOST SITE
- * ECONOMIC JUSTIFICATION
- * TECHNICAL PERFORMANCE

HEAT EXCHANGER MARKET STUDY

OBJECTIVE

ASSESS OPPORTUNITIES FOR CERAMIC HEAT EXCHANGERS
IN THE CHEMICAL PROCESS INDUSTRIES

INCLUDE

HAZARDOUS WASTE
LOW BTU CONTAMINATION CLEAN-UP
PRODUCTION PROCESSES SUCH AS H_2SO_4, $TiCl_4$

THRUST OF STUDY

MARKET POTENTIAL IN 1990-2000
IDENTIFY TECHNOLOGY ADVANTAGES/LIMITS/COSTS
DETERMINE VALUE IN USE
ESTIMATE MARKET SEGMENTS AND VOLUME

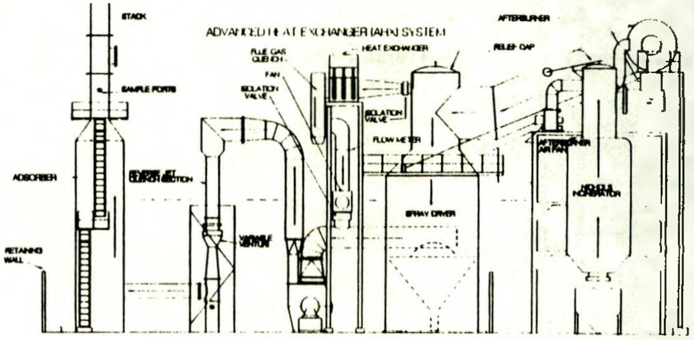
PROPOSED HOST SITE

DUPONT HAZARDOUS WASTE INCINERATOR

MODIFIED NICHOLS MONOHEARTH INCINERATOR
500 TONS WASTE PER YEAR
ALL TYPES WASTE EXCEPT PCB'S AND DIOXIN PRECURSORS

THE SITE OFFERS

GOOD TEST OF COMPOSITE MATERIALS PERFORMANCE
EASE OF INSPECTION/REPAIR OF COMPONENTS
TEST UNDER RESEARCH CONDITIONS



PROPOSED ADVANCED CERAMIC HEAT EXCHANGER INSTALLATION
IN DuPont HAZARDOUS WASTE INCINERATOR

ECONOMIC JUSTIFICATION

ASSESS ECONOMIC IMPACT ON THE HOST PROCESS VIA PROCESS IMPROVEMENTS OR ENERGY SAVINGS

ESTABLISH PROJECTED COST OF FULL SCALE UNIT

ESTABLISH ACCEPTABLE ROI

TECHNICAL PERFORMANCE

SCALE-UP OF TUBES

SITE FLUE TEST

PROCESSING IMPROVEMENTS

LONG TERM MATERIALS RESPONSE

PHASE 3 FIELD TEST

HOST SITE SELECTION/AGREEMENT
FINAL DESIGN
PROCUREMENT/PRODUCTION
INSTALLATION
SHAKE-DOWN
OPERATION
ASSESSMENT AND FINAL ECONOMICS

FIELD TEST DESCRIPTION

SIX MONTH DURATION
USE PRESENT OPERATING SCHEDULES AND PROCEDURES
PERIODIC INSPECTION OF COMPONENTS
POSSIBLE SHORT PERIODS OF INTENSIVE MEASUREMENTS
POST EXAMINATION OF COMPONENTS

FIELD TEST SCHEDULE

HOST SITE SELECTION	8/89
QUANTIFY OPERATING PARAMETERS	9/89
PRETESTING OF SAMPLES	2/90
SIGNED HOST SITE AGREEMENT	2/90
FINAL DESIGN	6/90
ENERGY AUDIT OF HOST SITE	3/91
INSTALLATION OF AHX	7/91
SHAKEDOWN	8/91
PERFORMANCE TESTING	2/92

SUMMARY

- HOST SITE HAS BEEN SELECTED
- PRELIMINARY DESIGN MODIFICATIONS UNDERWAY
- MARKET STUDY UNDERWAY
- MATERIALS TESTING IN THE LAB UNDERWAY
- MATERIALS TESTING AT THE HOST SITE TO START
- FIELD TEST SCHEDULED FOR LATE 1991

DISCUSSION

Development of Advanced Composite Ceramic Heat Exchanger System

Don L. Hindman

Q: What is the anticipated cost of the system?

A: The ceramic tubes are the major cost item right now due to the cost of the fiber. It is an experimental product and costs approximately $300/lb. As production increases the price is expected to decrease, following the same trend as carbon fiber has.

Q: Where would such an exchanger be used within a sulfuric acid plant?

A: We expect that it would be used in the spent acid process to preheat combustion air. DuPont has looked at this and they have some ideas, which they consider proprietary, on how to install a heat exchanger. We don't see a high temperature application in the sulfur burning process.

Q: You're showing that the heat being recovered by the recuperative heat exchanger is actually being used in the stack. Did you consider taking the recuperative heat and using it for the air going to incineration, which would be what you do in a practical situation? Going to the stack, the heat is completely lost.

A: B&W considered that, and it is still an option. The operators of the incinerator at DuPont are very much interested in making sure that we don't interfere with their normal operations. There are several places that the heat could be used at the incinerator.

Comment: The overall plan is to use the heat for preheating combustion air in the afterburner and in the incinerator. But at this point we don't want to disturb the system. We also don't want to get into all the costs of putting in high-temperature burners to handle the hot air.

Q: Yttria stabilized zirconia is used in the tube?

A: Yes, although in some applications an alumina matrix may be used.

Comment: The stabilized zirconia is usually 20 percent; partially stabilized zirconia is usually somewhere between 6 and 8, 6 and 10 percent.

A: The zirconia contains about 10 mole percent yttria.

COMPOSITE HEAT EXCHANGER MATERIALS SUPPORT

J. I. Federer
ORNL

U. S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGER PROGRAM
TASK DESCRIPTION

TITLE: Composite Heat Exchanger Materials Support
WORK PROPOSAL NUMBER: CEED009
CONTRACTOR: Oak Ridge National Laboratory

PRINCIPAL INVESTIGATORS: R. A. Bradley, 615/574-6094 and J. I. Federer, 615/574-5131

OBJECTIVE: Evaluate the thermal stability and corrosion behavior of ceramic composites fabricated by Babcock & Wilcox Company.

TECHNICAL APPROACH: Subject ceramic composite specimens to thermal aging in air followed by thermal aging of duplicate specimens in a corrosive atmosphere in laboratory furnaces. Determine dimensional stability from changes in diameter and length. Use conventional techniques such as ceramography, X-ray diffraction, and microanalysis to investigate corrosion reactions and causes of dimensional changes, if any.

SYSTEM DESCRIPTION: Ceramic composites consist of continuous fiber hoop- or helical-wrapped in a ceramic matrix. Tubular specimens are supported in furnace-heated alumina tubes for thermal aging in air and in a corrosive atmosphere.

CURRENT STATUS (9/89): Testing of composites fabricated by Babcock & Wilcox under Phase II was completed in early FY 1989. Seven of 12 specimens to be tested under Phase III have been received. Pretest measurements of specimens and preparation of test facilities are in progress.

ACCOMPLISHMENTS: Fourteen ceramic composite specimens were tested at a maximum temperature of 1160°C for times up to 640 h in an oxidizing atmosphere containing Na_2CO_3 vapor.

CONCLUSIONS: The results of the corrosion test indicated that PRD 166 fiber is more corrosion-resistant than Sumitomo fiber, that a ZrO_2 matrix is more dimensionally stable than an Al_2O_3 matrix, and that helical-wrapped fiber provides significant dimensional stability to composites.

BIBLIOGRAPHY:

1. J. I. Federer and W. P. Parks, Jr., "Laboratory Corrosion Testing of Babcock & Wilcox Ceramic Composites," letter report to G. R. Peterson, November 14, 1988.

COMPOSITE HEAT EXCHANGER MATERIALS SUPPORT

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METALS AND CERAMICS DIVISION
OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

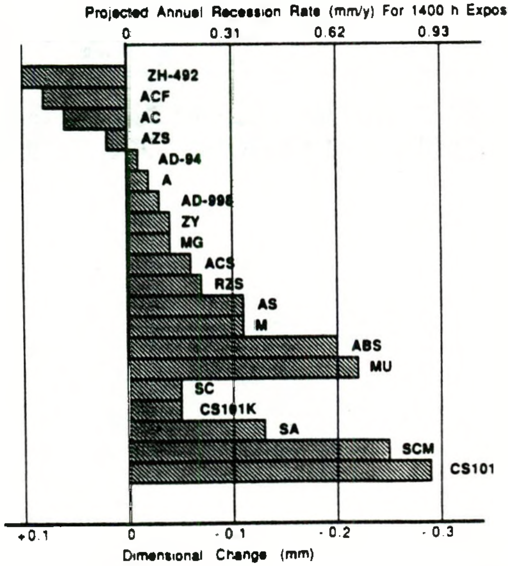
U. S. DEPARTMENT OF ENERGY
OFFICE OF INDUSTRIAL PROGRAMS
DIVISION OF WASTE ENERGY REDUCTION
WASTE ENERGY RECOVERY BRANCH

ADVANCED HEAT EXCHANGERS PROGRAM REVIEW
HERNDON, VIRGINIA
OCTOBER 11 AND 12, 1989

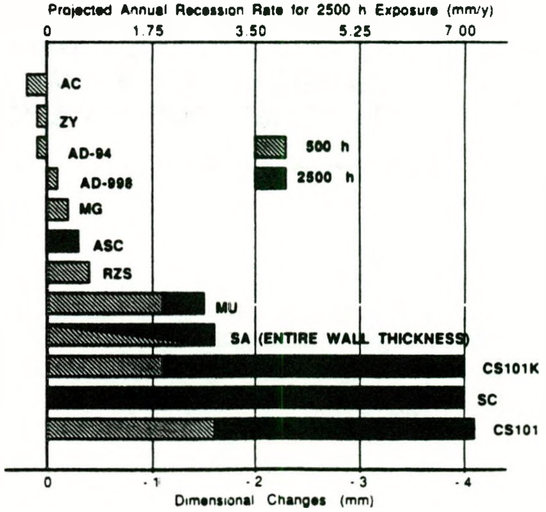
THE CORROSION BEHAVIOR OF MATRIX MATERIALS WAS INVESTIGATED IN INDUSTRIAL FURNACE EXPOSURE TESTS

Furnace	Time (h)	Temperature (°C)	Number of Specimens	
			Installed	Recovered
Aluminum remelt No. 1	500	750-1250	23	12
Aluminum remelt No. 1	2500	750-1250	25	10
Aluminum remelt No. 2	9700	750-1200	28	22
Steel soaking pit	1400	1150-1175	23	23

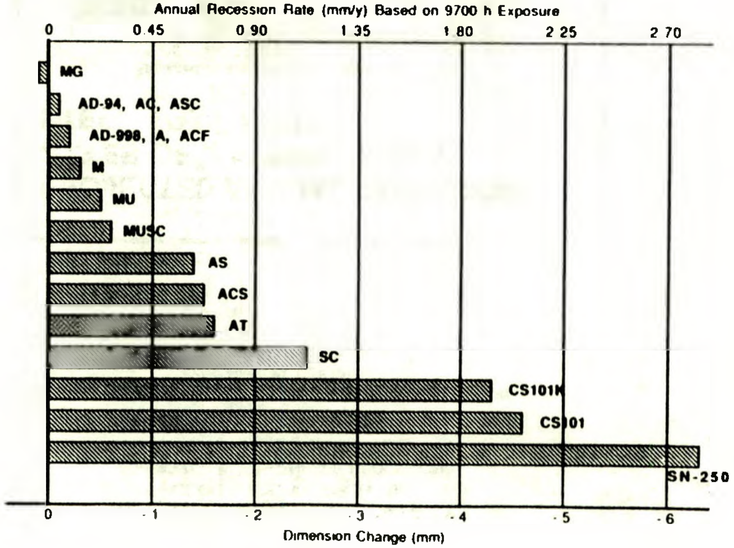
PROJECTED ANNUAL RECESSION RATES OF MATERIALS IN A STEEL SOAKING PIT



PROJECTED ANNUAL RECESSION RATES OF MATERIALS IN ALUMINUM REMELT FURNACE NO. 1



ANNUAL RECESSION RATE OF MATERIALS IN ALUMINUM REMELT FURNACE NO. 2

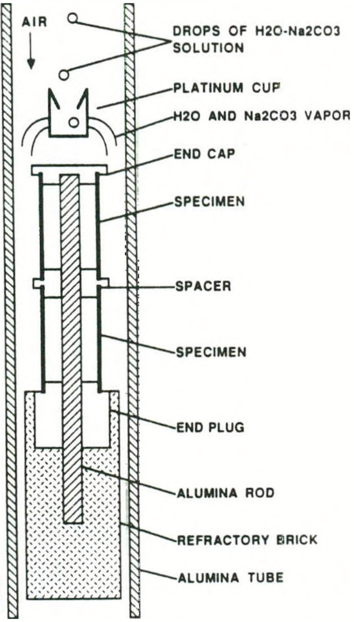


CERAMIC COMPOSITES WERE FABRICATED FOR TESTING WITH Al2O3 AND ZrO2 AS MATRIX MATERIALS

Composite	Continuous Fiber[a]	Matrix	Coating
1A	PRD166	ZrO2	None
2B	PRD166	ZrO2	SnO2
3A	PRD166	ZrO2	ZrO2
4A	PRD166	ZrO2	Al2O3
12	PRD166	ZrO2	None
13	PRD166	ZrO2	SnO2
5A	PRD166	Al2O3	None
6B	PRD166	Al2O3	SnO2
7A	Sumitomo	ZrO2	None
8A	Sumitomo	ZrO2	ZrO2
9A	Sumitomo	Al2O3	None
10A	Sumitomo	Al2O3	SnO2
11A	Sumitomo	Al2O3	ZrO2
F111B	Sumitomo	Al2O3	None

[a] All composites were hoop-wrapped except 12 and 13, which contained both hoop- and helical-wrapped fibers

CERAMIC COMPOSITES WERE CORROSION TESTED IN LABORATORY FURNACES



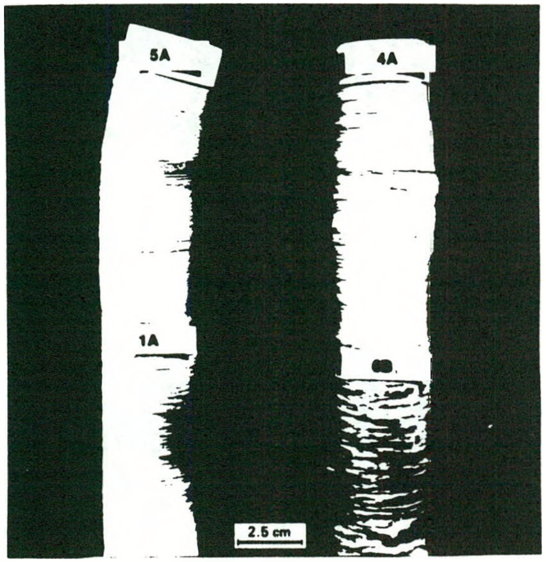
TEMPERATURE
1160°C max.

FLOW RATE
8000 cm3/min

DURATION
UP TO 640 h

NOT TO SCALE

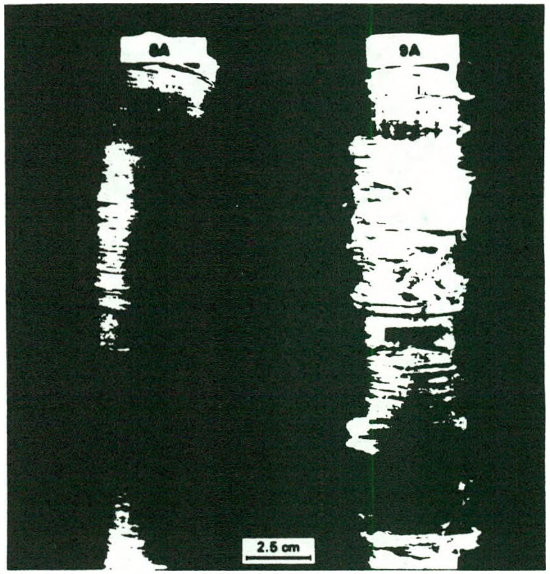
CERAMIC COMPOSITES WITH PRD166 FIBER AFTER THE CORROSION TEST



MATRIX MATERIALS

1A AND 4A	ZrO_2
5A AND 6B	Al_2O_3

CERAMIC COMPOSITES WITH SUMITOMO FIBER AFTER THE CORROSION TEST



MATRIX MATERIAL

7A AND 8A	ZrO_2
9A AND F111B	Al_2O_3

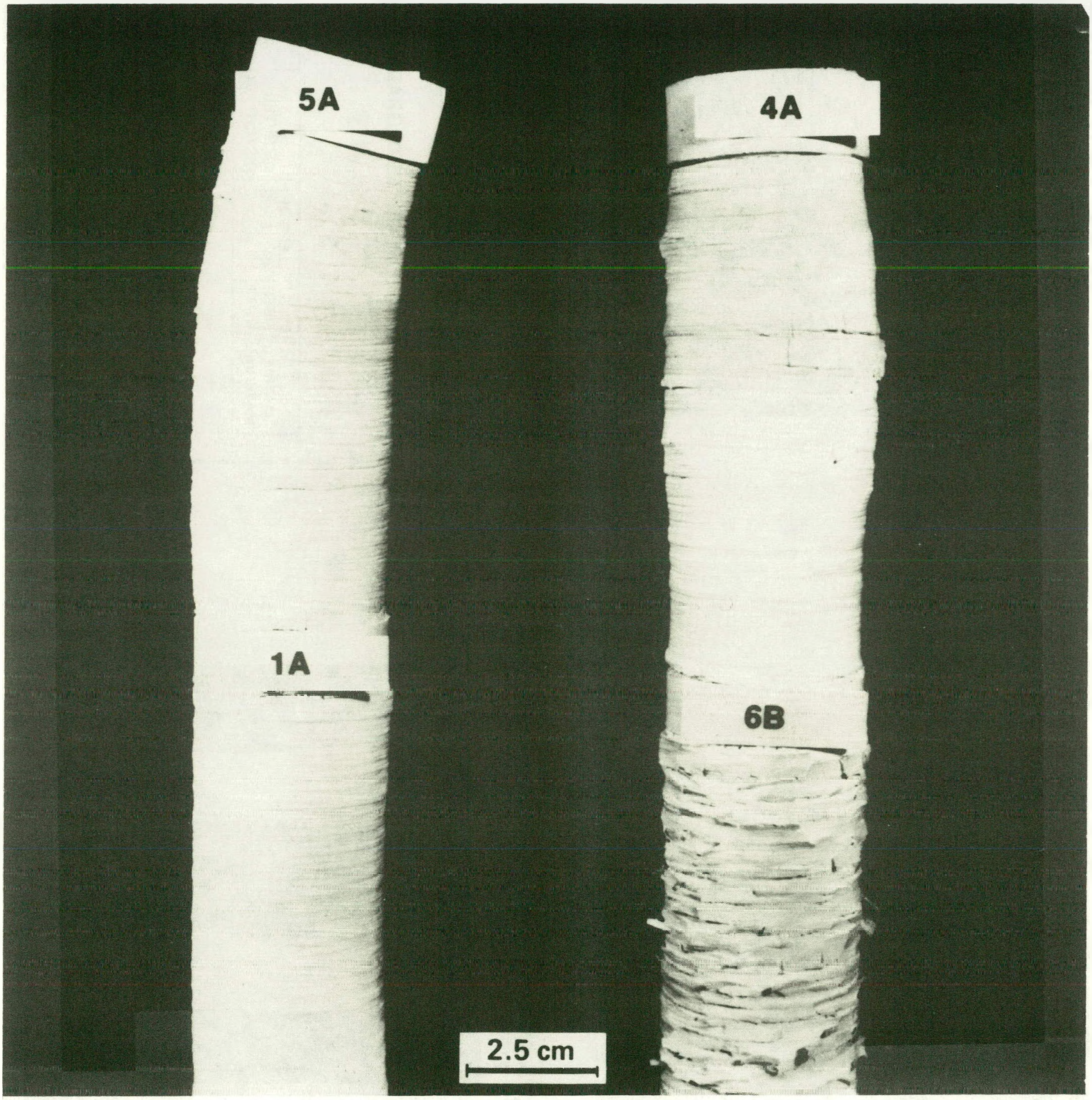
CERAMIC COMPOSITES DEGRADED BY DIFFERENT MODES

MATRIX MATERIAL	FIBER WRAP	LENGTH INCREASE (%)	DIAMETER DISTORTION	EXFOLIATION
ZrO_2	HOOP	1.7-19.7	YES	2 OF 6
ZrO_2	HELICAL	0	NO	NO
Al_2O_3	HOOP	0-18.6	YES	5 OF 6

Na IN FIBER

PRD166	DETECTABLE
SUMITOMO	SUBSTANTIAL

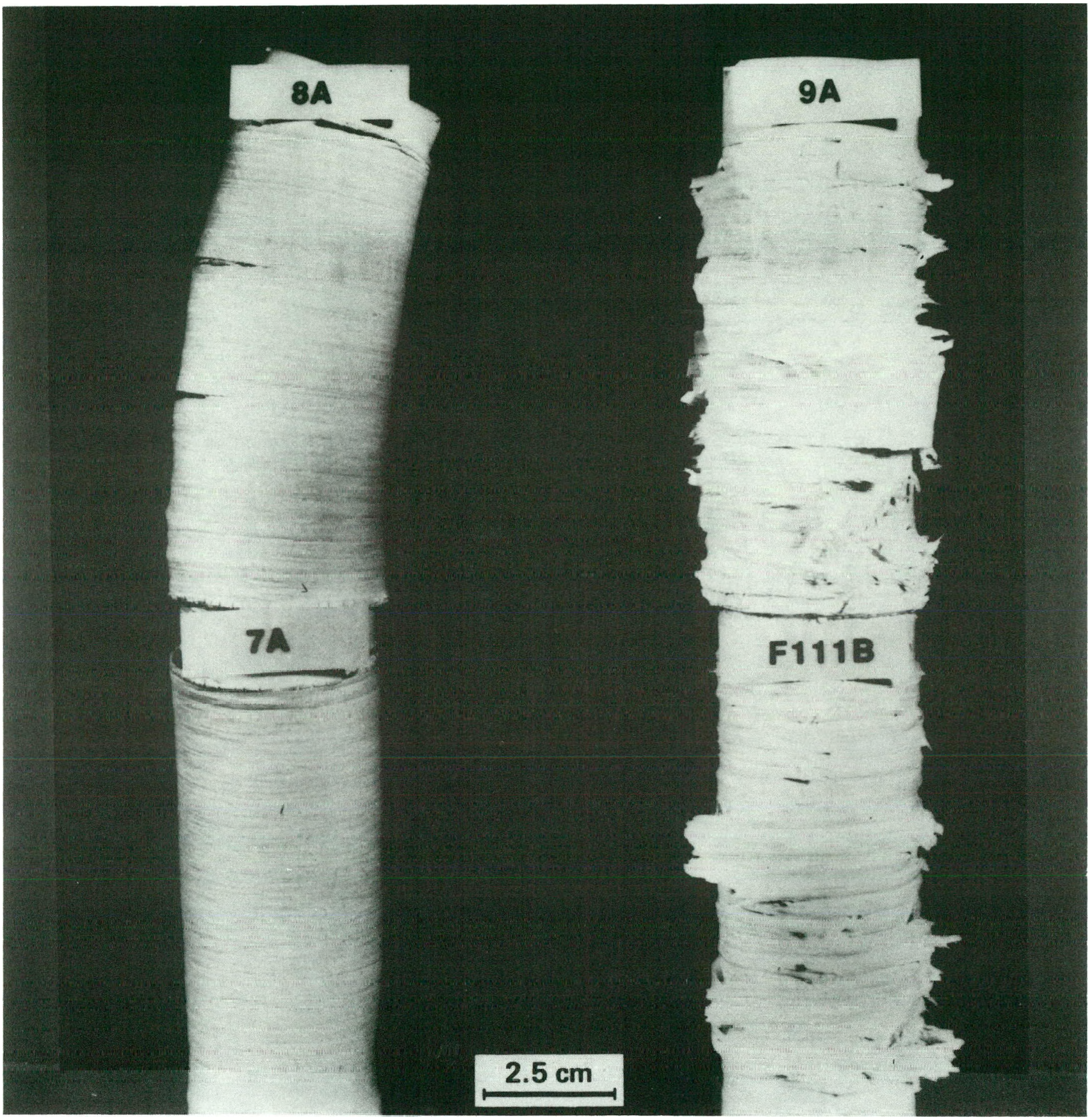
CERAMIC COMPOSITES WITH PRD166 FIBER AFTER THE CORROSION TEST



MATRIX MATERIALS

1A AND 4A	**ZrO2**
5A AND 6B	**Al2O3**

CERAMIC COMPOSITES WITH SUMITOMO FIBER AFTER THE CORROSION TEST



MATRIX MATERIAL

7A AND 8A	ZrO2
9A AND F111B	Al2O3

DISCUSSION

Composite Heat Exchanger Materials Support

J. I. Federer

Q: Why was the sodium carbonate used as the corrosive medium?

A: In other tests we have found that it forms a very corrosive atmosphere for silicon carbide and for oxide ceramics that contain silica. The intent of this test was to determine if the matrix material could protect the silica-containing fiber from the corrosive environment. It was not intended to duplicate an industrial atmosphere, which is very difficult to reproduce in a laboratory furnace.

Q: Have you done any post-exposure testing on mechanical properties? Not only the extent of strength retention, but also whether you're retaining the original toughness?

A: No, we have not. In the original tests that I described, most of the specimens were not suitable for strength testing after the corrosion test. A few were, but I allowed the specimens to be exposed to air for a period of several weeks until Babcock & Wilcox personnel could look at the specimens. The sodium carbonate tended to take up water, and although they possibly could have been dried and used, we were afraid that something had happened that would influence the results. So, we did not attempt any mechanical property testing of those samples, although that would be desirable in the future.

Q: The samples I'd be particularly interested in were the ones that were in the test that were thermally cycled. I'm wondering if in the future, these particular types of samples might be subjected to some pre- and post-testing of mechanical properties?

A: If the specimens survive in reasonably good condition, we can perform some mechanical testing. But testing of composite materials is not as straightforward as testing monoliths. We need to develop standard specimens and methods of load applications so that the results can be reproduced by others in the laboratory.

Comment: We've cycled specimens in air without the corrosive environments and found no reduction in strength. We've used about 25 cycles up to 1100°C. In this test and in others, although we haven't measured actual strengths, we have found a retention of the non-brittle failure in some of the samples, whereas others were brittle. Every sample tested was different with respect to coatings, fibers, and matrices, so general conclusions could not be made. Zirconia was generally the most stable material in all the corrosion tests.

Q: As a follow up to your comment about leaving the specimens in the laboratory environment and apparent degradation due to the interaction of the corrosion with water -- is that not likely to pose a serious problem in actual operation?

A: It's possible that it does pose a problem, but it's one that we did not investigate thoroughly. In some industrial furnaces, the temperature may not decrease to room temperature, so water evaporation would still occur. In an extended shutdown, however, the system would get cool enough for condensation and water absorption to occur. Hydrated compounds might then form. These compounds would deydrate when the system was reheated, but we do not know if hydration-dehydration would cause a problem.

Q: Have you made any calculations of the thermal stresses in these oxide materials that occur at the maximum temperatures?

A: We've done some calculations based on typical high temperatures, without corrosion as a variable. For a bayonet-style heat exchanger tube experiencing transient conditions, the thermal stress was 2,000 to 4,000 psi, very low. There's much controversy on the design tensile strength for ceramic composites. Basically, we are considering a 10 to 1 ratio between strength and applied stress, which seems pretty safe. Further work on the whole ceramic composite community is needed to answer your question better. Back to the issue of hydration -- the problem is related to whether sodium hydroxide, sodium alumina-silicate, or some hydrated compound would form. There aren't many applications where pure sodium is present, so the amount of compounds actually forming would have to be evaluated in each case. These composites do have a residual porosity, which may be a problem. We're doing work to reduce their porosity, but they are still slightly permeable. It's an application dependent issue.

HIGH PRESSURE HEAT EXCHANGE SYSTEM (HiPHES) OVERVIEW

Gary R. Peterson
DOE-ID

High Pressure Heat Exchange System (HiPHES) Overview

Gary R. Peterson, P.E.
Project Manager
U.S. Department of Energy
Idaho Operations Office
Idaho Falls, Idaho

Three HiPHES Projects Are Currently Concluding Phase I

1. Brayton Cycle Cogeneration System on a Municipal Waste Incinerator - Babcock & Wilcox
2. Brayton Cycle Cogeneration System on a Hazardous Waste Incinerator
3. Convective Steam Reformer for Methane or Ammonia Production

Phase II Will Resolve Key R&D Issues

PRELIMINARY DESIGN OF HIGH PRESSURE, HIGH-TEMPERATURE CERAMIC AIR HEAT EXCHANGER

Bruce L. Harkins
Solar Turbines

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGERS PROGRAMS
PROJECT DESCRIPTION

TITLE: PRELIMINARY DESIGN OF A HIGH PRESSURE, HIGH-TEMPERATURE CERAMIC AIR HEAT EXCHANGER.

COOPERATIVE AGREEMENT NO.: DE-FC07-88ID12799, Through DOE-ID.

CONTRACTOR: Solar Turbines Incorporated
P.O. Box 85376
San Diego CA. 92138-5376

PRINCIPAL INVESTIGATOR: Bruce Harkins, 619-544-5398

OBJECTIVE: Develop a preliminary design for a High-Pressure Heat Exchanger System (HiPHES) to economically recover heat from high-temperature corrosive gas streams and utilize the recovered heat energy to operate an indirectly heated gas turbine generator set. The operational goals for the heat exchanger were to heat compressed air, 150 psia at 600°F to a temperature of 1650°F from a gas stream with temperature in excess of 2300°F.

TECHNICAL APPROACH: The design strategy was to consider three competing concepts, approved by DOE, and evaluate each concept based on mechanical analysis, material, material joining, functionality, reliability, manufacturability and cost.

SYSTEM DESCRIPTION: The HiPHES system being developed by Solar will be incorporated into a hazardous waste incinerator located in Houston, Texas. The two principal components that compose the system are the heat exchanger and the indirectly heated gas turbine.

The heat exchanger is based on a modular design so it could be readily adapted to other processes by modifying the module size or increasing the number of modules. The modular design is based on a bayonet style design (tube within a tube). Air enters the module through an upper section or header and travels down an inner tube inside the ceramic tube. The air exits the inner tube into the annulus formed by the inner and outer ceramic tube, travels up the annulus while being heated, and exits into the lower header of the module. The air is delivered to and from the modules by refractory lined manifolds.

The preliminary heat exchanger design developed for the HiPHES system uses a combination of ceramics (monolithic and composites) and metallic materials. Each material is used to its maximum advantage. Ceramics (monolithics and composites) are used for headers and heat transfer surfaces where the need for high temperature, corrosive resistances and high reliability are required. Metallic materials have lower costs and are used for components not exposed to hostile environments.

The indirectly heated gas turbine is based on a Solar's Centaur model industrial gas turbine. In the conventional gas turbine cycle, atmospheric air is initially compressed, heated by the combustor and finally, the air is expanded through the power turbine to perform work. The indirectly heated gas turbine is basically the same as the conventional gas turbine, except the heat energy for the cycle is obtained with the HiPHES heat exchanger.

The preliminary design of the HiPHES system was based on the proposed demonstration site at a hazardous waste facility. The use of a HiPHES system at a hazardous facility would give a significant improvement in the overall energy utilization. Depending on the hazardous waste, the stack gas temperatures generally range from 2100°F to 2400°F. The hazardous waste disposal cycle uses a rotary kiln and afterburner to achieve the required temperatures and resident time set by the Environment Protection Agency. Hot corrosive gas (1800°F) is fed from the rotary kiln to the afterburner where additional heat is added to achieve required temperatures (2300°F). The hot corrosive gas is then fed to the HiPHES heat exchanger and thermal energy is extracted. The flue gas temperature is reduced to approximately 1300°F at the last pass of the heat exchanger. The reduced temperature flue gas is then directed through the gas clean up equipment. The energy saving based on the demonstration HiPHES system would be 312,000 MMBtu/yr, which translates into 2.2 million gallon/yr of No. 2 diesel fuel or 306 million cuft/yr of natural gas/yr.

CURRENT STATUS (9/87): The project has been completed, and the final report is currently being reviewed by The Department of Energy.

ACCOMPLISHMENTS: A preliminary design was developed which uses a combination of monolithic and composite ceramics and metallic materials. Ceramics are used where the need for high temperature, corrosive resistance and reliability are required. Metallic materials are used for components not exposed to hostile environments to realize lower costs. The design overcomes critical problems normally encountered in the joining of ceramics to themselves or metals. The joining technique is a proprietary process developed at Solar. Analytical calculations predict the heat exchanger design will heat 150 psia air at 600°F to 1650°F with a flue gas pressure drop of 1 in. of water with an air side pressure drop of 7 psi.

FUTURE WORK: Phase II - Technology development of critical items identified in Phase I. The objective of Phase II will be to complete a detailed design of the heat exchanger with an end goal of testing a prototype module.

CONCLUSIONS: A ceramic heat exchanger can be designed and manufactured economically; however, many critical technologies must be investigated before ceramic heat exchangers become widely used components. The primary critical issues are:

- Fouling and corrosion
- Fabrication methods
- NDE and lifetime predictions
- Costs
- Ceramic coatings

7130n:BMD:seb

PROGRAM
REVIEW
HiPHES

A DOE FUNDED PROGRAM FOR THE DEVELOPMENT
OF HIGH-PRESSURE HEAT EXCHANGE SYSTEMS

HiPHES
PROGRAM

Phase I	- Develop a preliminary HiPHES design. - Evaluate economic feasibility. - Identify key research/development problems.
Phase II	- Resolve research/development problems. - Develop the HiPHES detailed-estimate design.
Phase III	- Fabricate hardware. - Field test prototype HiPHES design.

BACKGROUND
APPLICATIONS

GENERAL CONCEPT	ENERGY SAVINGS QUADS	COST SAVING MILLONS $
Power generation from low pressure exhaust	0.464	1,472
Steam reforming of natural gas	0.022	344
Heat transfer to or from hot liquid flows	0.074	131
Recover heat from corrosive waste gases	0.068	162
	0.628	2,109

BACKGROUND
PROBLEMS

- High cost of advanced materials
- Loss of strength at high temperatures for metallic components.
- Complex joining designs between ceramic to ceramic or ceramic to metal.
- Unpredictable, catastophic structural failure of stressed ceramic components.
- Complex designs to overcome differential expansion rates between materials.

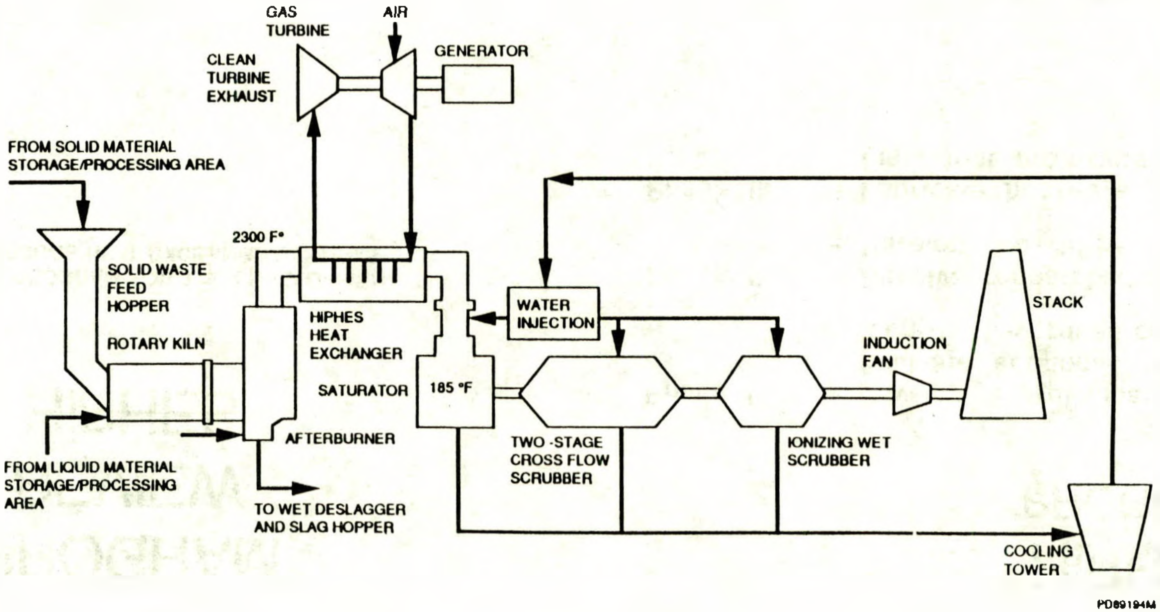
HiPHES
PHASE I

SOLAR'S OBJECTIVE

- Develop advanced air heater beyond state-of-the-art based on composite ceramics producing high pressure, high temperature air feeding an indirectly fired gas turbine.
- Obtain significant energy savings with attractive economics.
- Use exhaust gases from hazardous waste incinerator as the heating source for the air heater.
- Improve performance of Hazardous waste process.

DESIGN
CHALLENGES

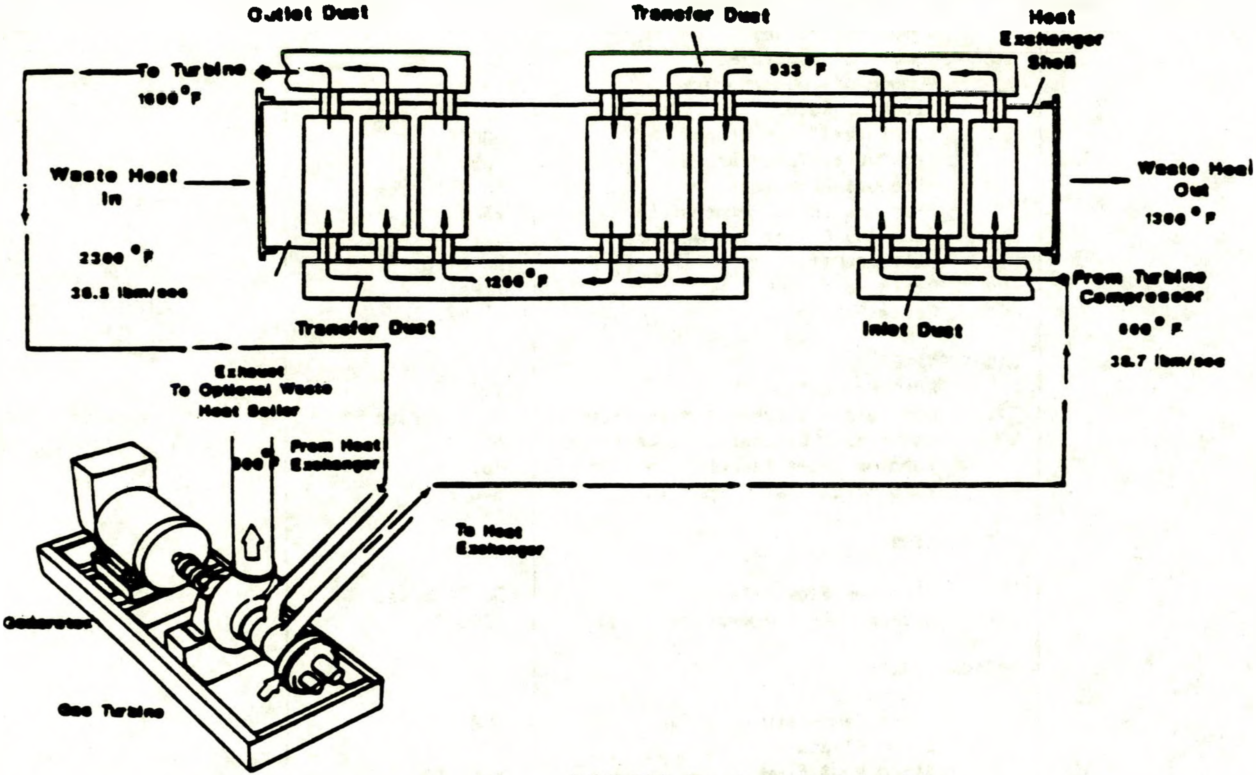
- **High Thermal Expansion difference**
- **Joining of non-similar materials**
- **Stress Analysis**
- **Assembly**
- **Maintenance and Repair**



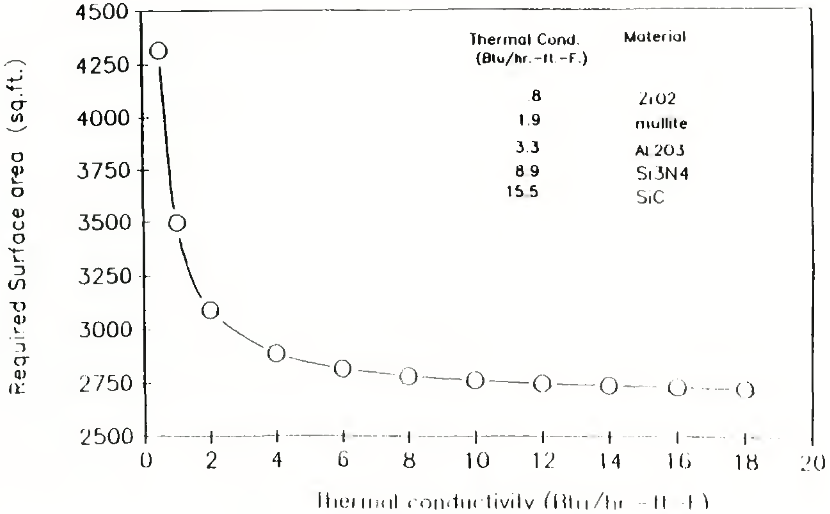
Hazard Waste Incinerator System

HiPHES Component Specification

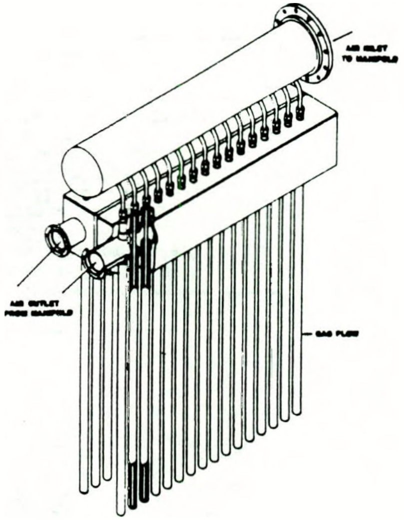
CERAMIC HEAT EXCHANGER	
Number of Passes	3
Number of Module/Pass	54
Number of Tubes/Module	16
Air Flow Rate	38.7 lb/sec
Air Inlet Temperature	600°F
Air Outlet Temperature	1650°F
Flue Gas Flow Rate	36.7 lb/sec
Flue Gas Inlet Temperature	2300°F
Flue Gas Outlet Temperature	1270°F
Heat Transferred	34.0 10^6 Btu/hr
Delta Pair	7 Psi
Delta P Flue	1 INWC
GAS TURBINE	
Pressure Ratio	9.3
Compressor Discharge Mass Flow	38.7 lb/sec
Compressor Discharge Temperature	600°F
Turbine Inlet Temperature	1650°F
Turbine Exhaust Temperature	840°F
INCINERATOR	
Flue Gas Flow Rate	36.5 lb/sec
Afterburner Temperature	2300°F
OPTIONAL HRSG	
Steam Temperature	752°F
Steam Pressure	650 Psia
Steam Mass Flow	4.75 lb/sec
SYSTEM OUTPUT	
HiPHES	2.5 MW
Optional HRSG	1.4 MW
Total	3.9 MW electrical

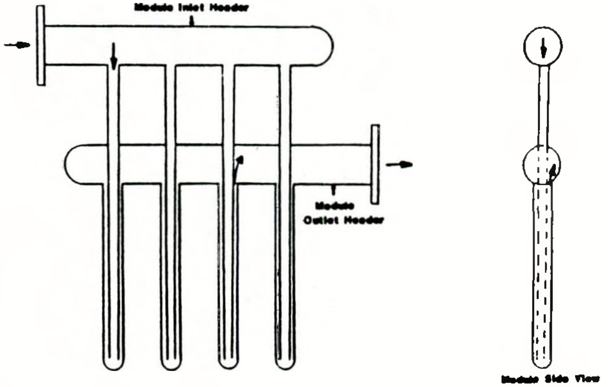


HiPHES System Schematic

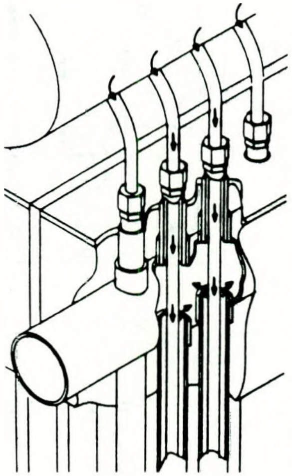


REQUIRED SURFACE AREA AS A FUNCTION OF THERMAL CONDUCTIVITY

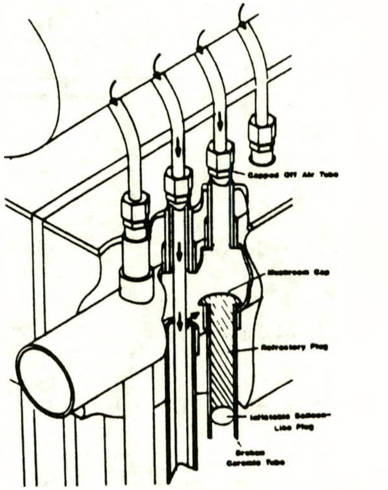




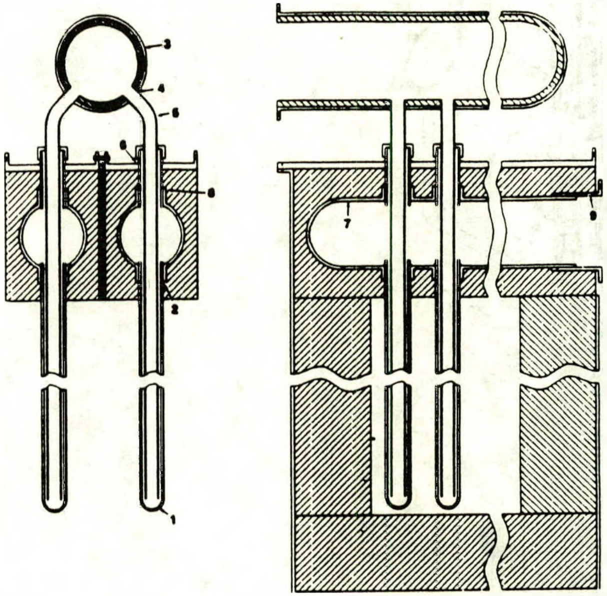
Heat Exchanger Module Schematic



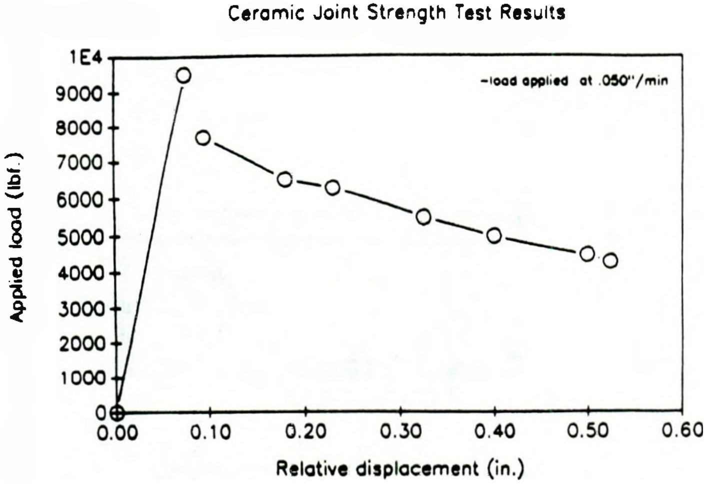
Normal Module Configuration

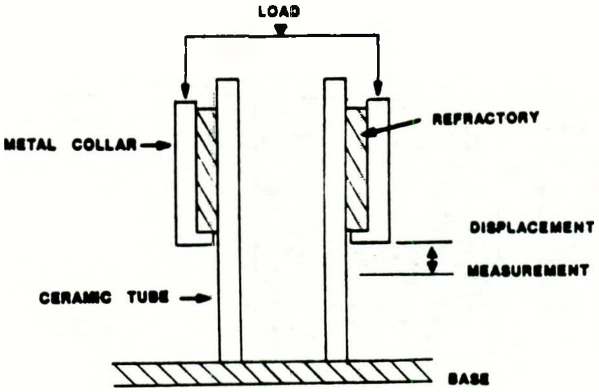


Module with Repaired Tube



Description	Design
1) Gas Side Tube	Monolithic Ceramics 2" OD x .1875" Wall
2) Joint	Monolithic Ceramic to Composite Ceramic, see Figure 2.6.1
3) Air Header	Metal SS 310 Pipe x .25" Wall
4) Joint	Welded Joint SS 310 to SS 310
5) Air Tube	Metal SS 310 1.125" x .065"
6) Seal	Metal Tube Through Metal Exhaust Duct, see Figure 2.6.2
7) Gas Header	Ceramic Composite 5" ID x .25" Wall
8) Joint	Metal SS 310 to Composite Ceramic, see Figure 2.6.3
9) Joint	Metal to Composite Ceramic





CERAMIC JOINT TEST

FIXTURE SCHEMATIC

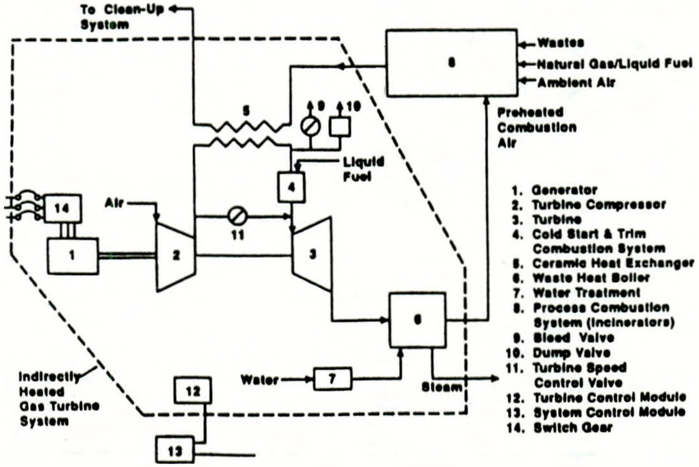
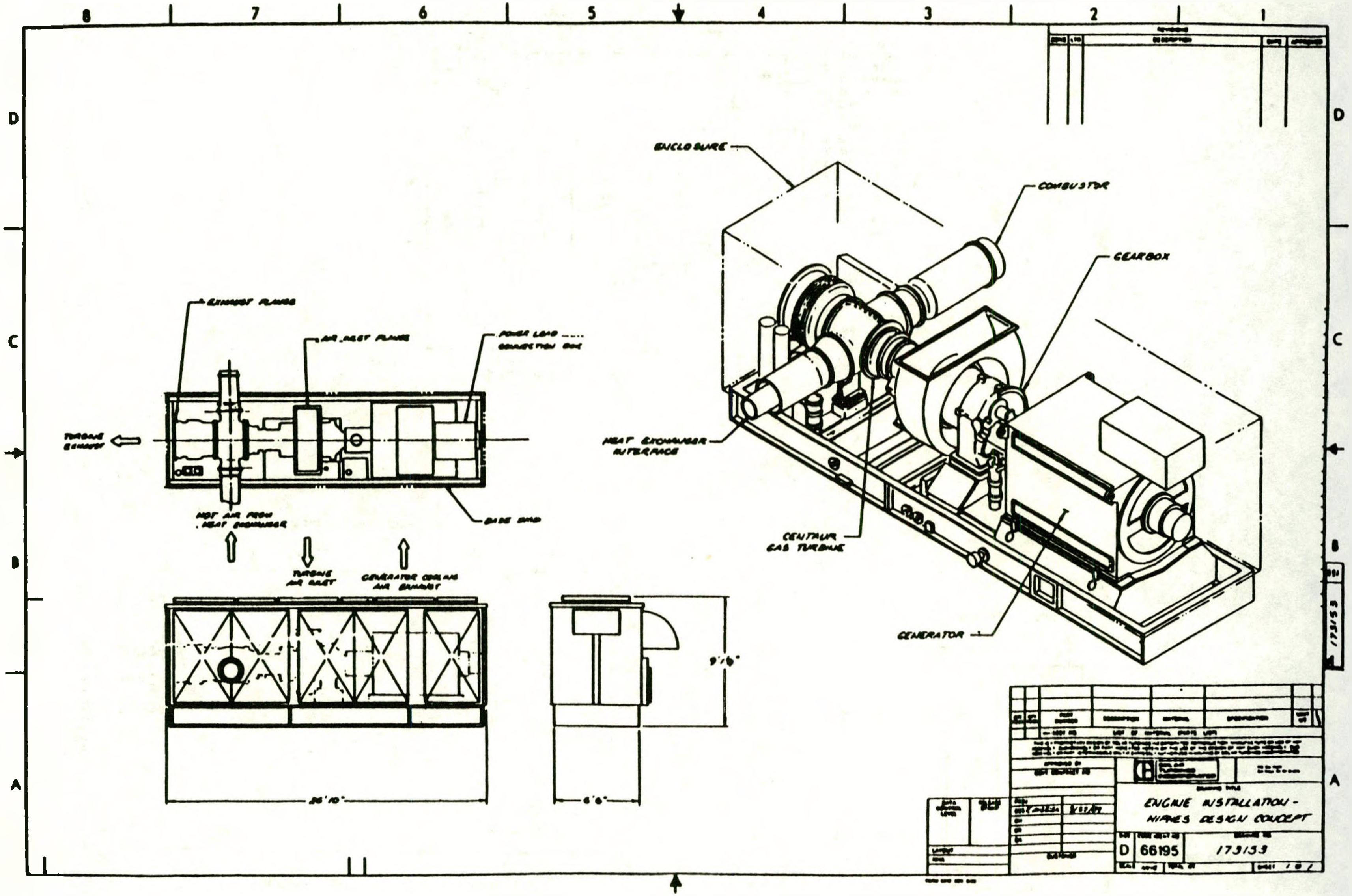


Figure 1. Indirect Heated Gas Turbine Schematic

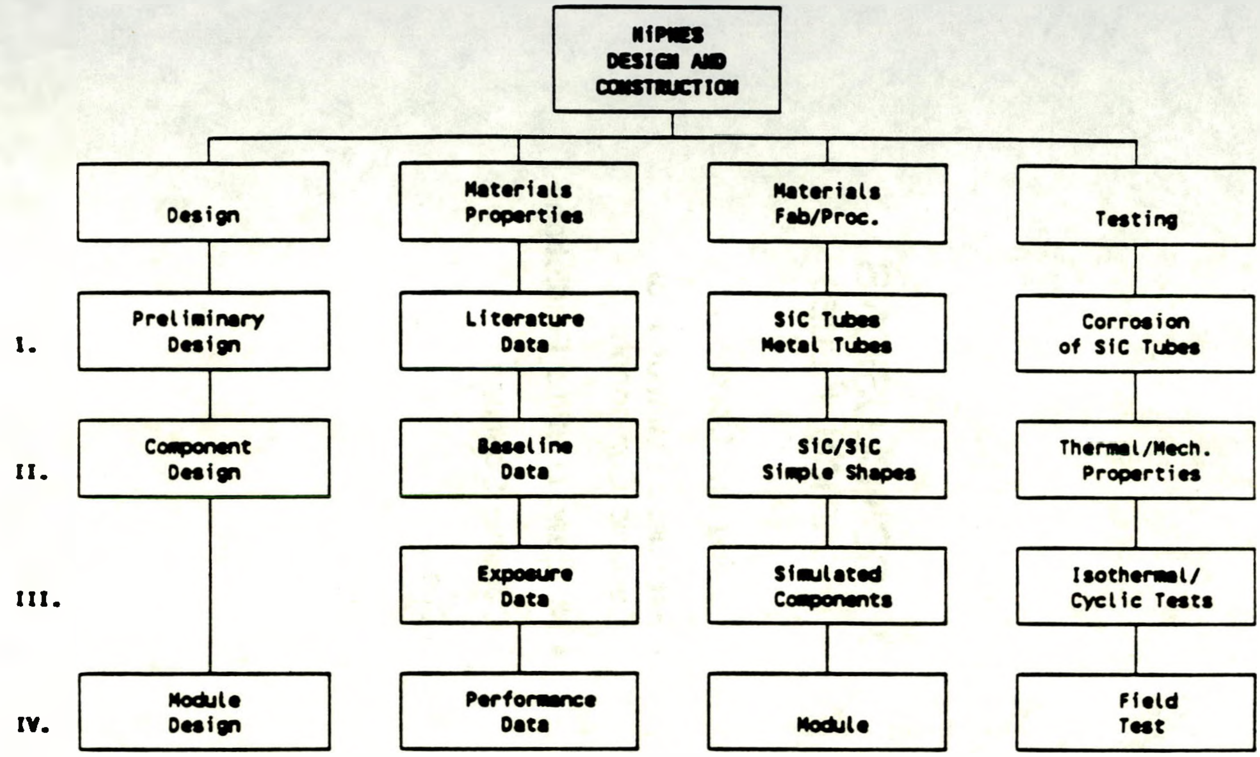
8
7
6
5
4
3
2
1
D
C
B
A
ENCLOSURE
COMBUSTOR
GEARBOX
HEAT EXCHANGER INTERFACE
CENTAUR GAS TURBINE
GENERATOR
EXHAUST FLANGE
AIR INLET FLANGE
POWER LEAD CONNECTION BOX
TURBINE EXHAUST
BASE SKID
HOT AIR FROM HEAT EXCHANGER
TURBINE AIR INLET
GENERATOR COOLING AIR EXHAUST
9'6"
25'10"
6'6"
ENGINE INSTALLATION - HIPHES DESIGN CONCEPT
D
66195
173153
173153

CONCLUSIONS

- Developed State-of-the-Art Heat Exchanger
  - Technically Feasible
  - Economically Attractive
- Innovative Design, Solutions to
  - Joining of Ceramics
  - Assembly Method
  - Maintenance/Repair

CONCLUSIONS
CRITICAL ISSUES

- Fouling and Corrosion
- Fabrication Methods for Ceramic Components
- NDE and Lifetime Predictions
- Ceramic Coatings
- Costs



Flowchart of Test Plan

Materials Property Testing

Test	SiC Tubes	Composite		Refractory Shell
		Fiber	Composite	
Density/Porosity	X	X	X	X
Dimensions		X	X	
* Visual Texture	X	X	X	X
* Tensile Strength	X	X	X	X
* Compressive Strength			X	X
* Interply Tensile Strength (Lam.)			X	
* In-Plane Shear Strength			X	
* Interply Shear Strength (Lam.)			X	
* Flexure Strength (MOR)			X	X
* C-Ring/O-Ring Strength	X		X	
* Burst Strength	X		X	
* Crack Propagation Stress vs. Crack Length Orien.	X		X	X
* Fracture Toughness	X		X	X
* Weibull Modulus	X		X	X
Elastic Modulus	X	X	X	X
Poisson's Ratio	X	X	X	X
Thermal Expansion	X		X	X
Thermal Conductivity	X		X	X
Thermal Shock Resistance	X		X	X
Corrosion Rate	X		X	X
NDE	X		X	X

* At RT and 2300°F before and after exposure high temperature testing at lower T for composites.

DISCUSSION

Preliminary Design of High Pressure High Temperature Ceramic Air Heat Exchanger

Bruce L. Harkins

Q: When you're talking about heat transfer through ceramics, have you considered aluminum nitride?

A: Not at this point. But in Phase II we will be reviewing numerous material systems.

Comment: We have had some very outstanding and surprising results with aluminum nitride. We had parts that were put in caustic for a hundred hours at 1000°C and nothing happened. If you look at the chemistry of aluminum nitride and alumina you would figure that something would happen. It might be worth considering.

A: Yes. You are right, it might be worth considering aluminum nitride or alumina as a material to investigate.

Comment: Right now it's fairly new material. It can be fabricated by any of a variety of methods -- extruded, injection molded, the normal ceramic technology. But it is showing some remarkable corrosion resistance.

Q: On those glass seals with which you say had 24 hours without failure -- do you have any change in pressure across the joints?

A: No, there is not a pressure change across those joints.

Q: With regard to cost, can you offer a couple of comments about the future and what energy cost framework you would need to make this viable?

A: We are currently estimating the cost of the heat exchanger. For a total system not including installation, the cost is about 2.5 million. But because this is a new technology the cost should go down, depending on how the development goes. The major part of that cost is the ceramics used in the heat exchanger. Three quarters of the total cost is ceramic material cost. Assembly costs are very minimal.

Q: I'd like to hear about some of the dimensions for the tubes used in the heat exchangers in terms of lengths, diameters, and spacings.

A: The heat transfer surfaces themselves, the ceramic tubes, are 2 inches in diameter, .1875 inches in wall thickness. The inner tubes are 1.125 inches in diameter and .065 inches in wall thickness. The headed diameters are 5 inches internal with a 1/8-inch wall thickness.

Q: Two years ago, at the inception of the HiPHES program, there was initial concern about the ability of monolithic ceramics in general to perform under this temperature and pressure regime. What's been the experience so far in the limited tests you've done? What is the current thinking of using monolithics in this environment?

A: Currently, they are continuing with the monolithic tubes for the heat transfer surface. As I mentioned, we have 24,000 hours of experience with the monolithic tubes on a recuperator. We also performed a 3,200 hour test at a hazardous waste incinerator. These were not pressurized tubes. We will be running some more tests. In fact tomorrow I'll be installing tubes at the hazardous waste incincerator again to measure wall recession rates. Down the road, on the start of Phase 2, we will be looking at developing a small pressurized rig to install in the intended host site and continue investigating the material. But to date, we've had good luck with the monolithic ceramic tubes.

HiPHES DEVELOPMENT OF A CONVECTIVE STEAM REFORMER

Joseph J. Williams
Stone & Webster

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGERS PROGRAM
PROJECT DESCRIPTION

TITLE: DEVELOPMENT OF HIGH PRESSURE HEAT EXCHANGE SYSTEM (HiPHES)

COOPERATIVE AGREEMENT NO: DE-FC07-88ID12797

CONTRACTOR: Stone & Webster Engineering Corporation
245 Summer Street
Boston, MA 02107

PRINCIPAL INVESTIGATOR: Joseph J. Williams, (617) 589-7197

OBJECTIVE: Develop an advanced high-pressure heat exchanger utilizing ceramic tubes for a convective steam/methane reformer integrated with a gas turbine cycle. The HiPHES steam/methane reformer is a convective type, catalytic reactor utilizing multiple pairs of a tube-within-a-tube configuration. The use of ceramic tubes, will allow reaction temperatures higher than the current state-of-the-art metal tube outlet temperatures of 1600 F. Higher reaction temperatures increase feedstock conversion and reduce energy requirements, compared to currently available reformers of the radiant-box type. Therefore, the goal of the HiPHES convective reformer is to operate at process temperatures in excess of 1900 F with a tube side pressure of 300 psia and a shell side pressure of approximately 120 psia.

TECHNICAL APPROACH: The project effort is divided into three phases. Phase I analyzes the technical and economic merits of the proposed HiPHES, develops a HiPHES preliminary design, identifies key research and development (R&D) problems, and plans their resolution. Phase II, if funded, will conduct the research and development tasks addressing those key problems identified in Phase I, and will provide the detailed-estimate/design for the Phase II field test unit. Phase III, if funded, will complete the design, fabrication, field testing, and evaluation of the prototype HiPHES.

SYSTEM DESCRIPTION: The proposed HiPHES ceramic steam/methane reformer converts methane (i.e. natural gas) and steam into a synthesis gas (H_2, CO, CO_2, H_2O, and CH_4) at 300 psia and 1900 F. Synthesis gas is used as a feedstock for the production of several higher-value chemicals such as methanol and hydrogen. In Phase I the economics of a 1,000 TPD methanol plant using a HiPHES ceramic steam/methane reformer were analyzed. In the methanol plant, the cooled synthesis gas is compressed to the methanol reactor pressure of 1195 psia, heated against methanol reactor effluent, and fed to the methanol reactor.

The methanol reactor is a catalyst-filled tubular type reactor which generates saturated 400 psia steam on the shell side to absorb the heat of reaction. In the reactor, 2 moles of hydrogen and 1 mole of carbon monoxide react to form 1 mole of methanol. The conversion of H_2 and CO per pass is not complete, and therefore a portion of the reactor effluent is recompressed with the fresh synthesis gas feed and recycled to the reactor inlet. Hydrogen-rich purge streams from the reactor effluent are used as fuel gas to the reformer and gas turbine.

The proposed HiPHES ceramic reformer is a convective reformer which uses the sensible heat of hot combustion gases to supply the heat of reaction. In the Tube-Within-a-Tube (TWIT) design some of the heat of reaction is also supplied by cooling the hot synthesis gas (in the inner tube) against the reactants (in the annulus). To improve the convective heat transfer to the reactants, the combustion gases are under pressure and flow at high velocity. In the proposed HiPHES reformer the compressed combustion air is supplied by a gas turbine cycle. A portion of the gas turbine air is sent to the reformer via a booster compressor and a like amount of combustion gases from the reformer are returned to the gas turbine combustor to replace the attempering air.

CURRENT STATUS (10/89): Phase I has been completed and a draft Phase I report and Phase II Scope of Work (SOW) have been submitted to DOE for comments.

ACCOMPLISHMENT: Phase I results included:

1) Determined the benefits of using ceramic materials in a steam/methane reformer. These benefits include improved methanol yields (higher lb methanol product/lb methane feed), and reduced energy requirements as compared to radiant reforming.

2) Received notification that ASME Section VIII-1 requirements can be met for the use of ceramic tubes within a metal pressure vessel. The metal shell must be designed for the higher pressure inside the ceramic tubes.

3) Identified up to 4 ceramic materials which appear to withstand chemical attack by the synthesis gas. A survey of available materials and the required material properties was conducted by Dr. John Haggerty of MIT. Also, ORNL conducted two sets of tests, one at 100 hours and another at 500 hours, which examined the effects of temperature and chemical attack on eight candidate materials. These tests identified the 4 ceramic materials which may be suitable for reforming tube applications.

4) Developed a ceramic HiPHES reformer design. Several alternative designs were also examined.

5) Completed a market survey which indicates that the potential market for methanol and the proposed HiPHES reformer is very promising.

FUTURE WORK: Proposed Phase II work will include testing of tubes, ceramic/ceramic joints and ceramic/metal joints at reforming temperatures and pressure and in a simulated synthesis gas atmosphere. Creep and fracture toughness tests are also planned. A detailed process design and estimate for a test unit at a host site will also be completed in Phase II. Phase III will include engineering, construction, and operation of the test unit.

CONCLUSIONS: The use of ceramic tubes in a convective steam/methane reformer has the potential to reduce feedstock requirements and fuel consumption when compared to radiant reforming. Several ceramic materials have been identified which are expected to withstand reforming conditions. However, before this technology can be commercialized effective ceramic tubes and joints must be proven. This will be the main focus of Phase II.

HiPHES DEVELOPMENT OF A CONVECTIVE STEAM REFORMER

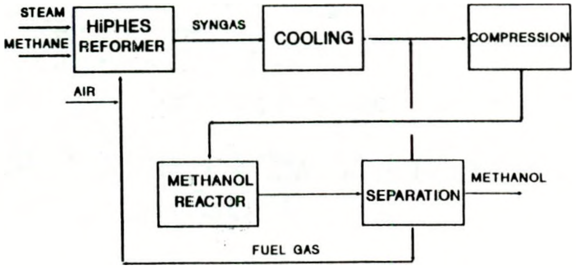
STONE & WEBSTER ENGINEERING CORPORATION

October 11, 1989

Project Description:

"To develop an advanced ceramic tubed convective steam/methane reformer integrated with a gas turbine cycle."

HiPHES FLOW CHART



MAJOR TASKS

PHASE I

- Identify Candidate Ceramic Materials
- Compare With Conventional Radiant Reforming
- Process Design & Cost Estimate for a 1,000 TPD Methanol Plant
- Market Survey
- Identify Areas for Further R & D

HISTORY -
STONE & WEBSTER POWER REFORMER

- Convective Reforming With Integrated Gas Turbine Cycle
  - Metal Tubes
  - Conventional Reaction Temp (1600 F)
- In-House Design Work and Industry Sponsored Studies
- Originated in Mid 1970's
- Mechanical Design Patents

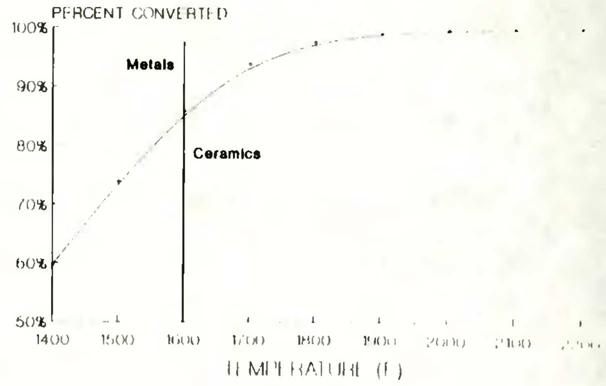
HISTORY -
ADVANTAGES OF POWER REFORMING

- Compact Design
  - Smaller Plot Area
- Lower Capital Cost
- Weight Reduction

WHY HiPHES?

- Higher Temperatures for Processing
- Greater Process Efficiency
  - Reduced Raw Materials Use
  - Reduced Energy Requirements
- Capital Cost Reduction Potential
- Plot Area Reduction
- Applications
  - Methanol & Other Liquid Fuels
  - Hydrogen/Ammonia Production
  - Other Processes

CONVERSION VS TEMPERATURE



400 psia [illegible]

HiPHES PROGRAM GOALS

- Identify Ceramic Materials Suitable for Reforming Service
- Determine Benefits of Using Ceramics for Reforming
- Identify Potential Market for HiPHES
- Demonstrate Steam/Methane Reforming in a Ceramic Tubed Reactor
- Reduce Cost of Producing Ceramic Tubes

PHASE I RESULTS

- Use of Ceramics Improves Methanol Yields
- Use of Ceramics Reduces Energy Requirements
- A Ceramic Tubed Reactor CAN Be Built Under Existing ASME Codes
- Several Ceramic Materials (ie SiC) Are Expected to Withstand Conditions
- Promising Market for Methanol and HiPHES

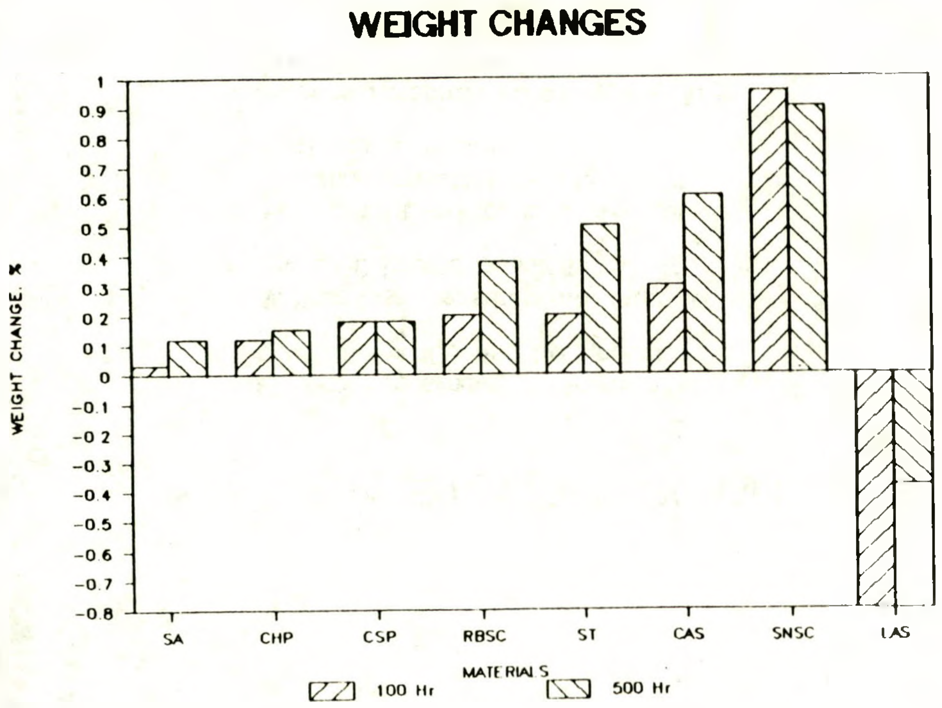
PHASE I TEST PROGRAM

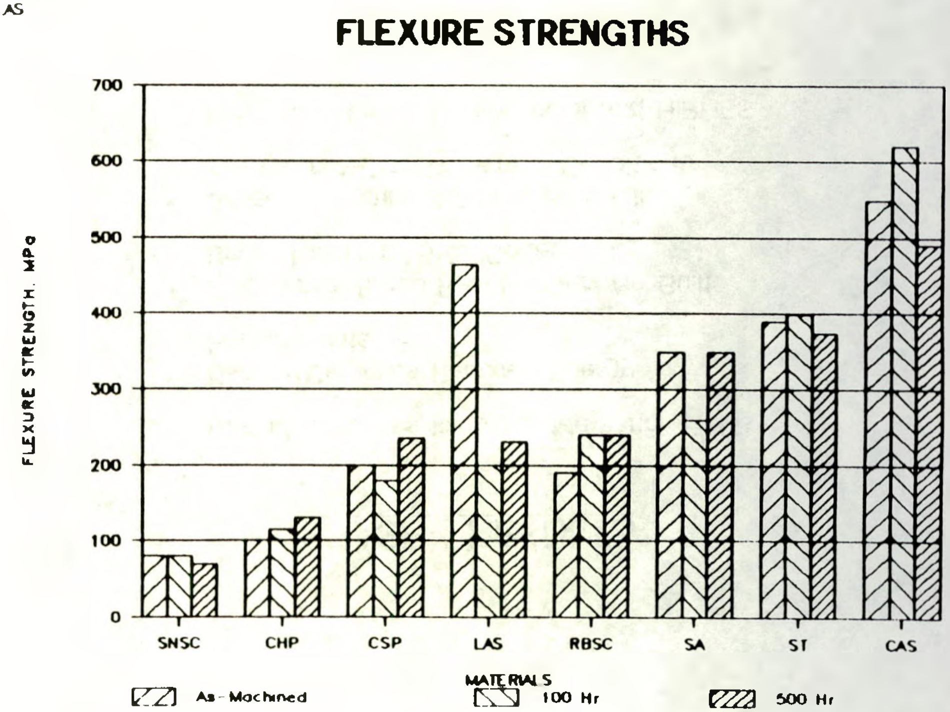
1. Exposure to simulated syngas at 2300 F for 100 and 500 hours.
2. Weight loss measurements after 100 and 500 hour exposures
3. Four point bend tests in as-received condition, and after 100 hour and 500 hour exposures.
4. Scanning electron microscopy before and after exposure.

MATERIALS TESTED

2300 F - 500 Hours

MATERIAL	SUPPLIER
Alpha SiC (SA)	Carborundum
SiC + TiB2 (ST)	Carborundum
High Purity RBSC (CHP)	Norton Co.
Standard Purity RBSC (CSP)	Norton Co.
RBSC (RBSC)	Coors Ceramics
Al2O3 + SiC (CAS)	Coors Ceramics
SNBSC (SNSC)	Ferro Corp.
Al2O3/Al/SiC (LAS)	Lanxide-DuPont


WEIGHT CHANGES
WEIGHT CHANGE, %
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-0.1
-0.2
-0.3
-0.4
-0.5
-0.6
-0.7
-0.8
SA
CHP
CSP
RBSC
ST
CAS
SNSC
LAS
MATERIALS
100 Hr
500 Hr


FLEXURE STRENGTHS
FLEXURE STRENGTH, MPa
700
600
500
400
300
200
100
0
SNSC
CHP
CSP
LAS
RBSC
SA
ST
CAS
MATERIALS
As-Machined
100 Hr
500 Hr

CONCLUSIONS

- Ceramics Can Produce Significant Savings
- Cost is Very Dependent on Cost of Ceramic Tubes
- Can Build Ceramic Reactor Under Existing ASME Vessel Codes
- Several Ceramic Materials Look Promising
- Market for Methanol in Next 10 Years is Very Promising

PHASE II WORK PLAN

- Prove Ceramic Materials and Joints Under Actual Conditions (T, P, Gas Comp)
- Creep and Fracture Toughness Tests
- Identify a Willing Host and Sign Host-Site Agreement
- Design Test Facility; Cost Estimate
- Phase III Plans and Cost Estimate

PHASE III WORK PLAN

- Engineering and Construction of HiPHES Test Reactor at Host Site
- Operation and Testing of HiPHES Test Reactor
- Commercialization Plan for HiPHES Reformer

DISCUSSION

HiPHES Development of a Convective Steam Reformer

Joseph J. Williams

Q: What size tubes were you considering for both the inside and outside?

A: We're considering a 3 1/2 inch O.D. outer tube, and a 1-inch O.D. inner tube. Tube length is in the range of 36 to 40 feet.

Q: In view of your very large activities in ethylene, I'm very surprised that your initial efforts in looking at ceramic applications is in the area of methanol and ammonia. Would you like to comment as to why you haven't been looking at ceramic tubes for ethylene manufacture?

A: We have looked at ethylene, as a matter of fact. At this point, we don't see an overwhelming advantage for the ceramics. It's a different operation, a low-pressure operation on the order of 20 to 30 pounds. We are still looking at it, however. We're also evaluating the use of ceramics in a ceramic-tubed pyrolysis device, but of all the applications that we evaluated, the synthesis gas production was the most viable.

Comment: I have a comment regarding the aluminum oozing or management of the residual metal. DuPont/Lanxide has now developed a technique that completely eliminates that problem, and we can provide samples that don't really have any weight change with that type of exposure.

Speaker: What percent aluminum?

Comment: It can be 8 percent.

A: That's good. As I said, after 500 hours the properties changed dramatically on the Lanxide material and gave us encouragement. That's why we certainly would want to include it in the Phase 2 testing.

Q: How would you support a 36-foot long tube with flow-induced vibration?

A: It will be supported from here (top tube sheet) and also at the bottom. These tubes will be very compactly spaced, and we'll have ceramic collars at various levels staggered within the bundle, so that all the tubes will actually be braced against each other. They will be designed much like we would design a regular heat exchanger.

Because the gaseous flow will be on the shell side, the combustion gases will be going parallel with the tubes which we feel will minimize the vibration versus having a cross-flow.

Q: You based your analysis on 300 pounds of pressure. Have you looked at increasing the synthesis gas pressure, especially in view of the fact that ammonia and methanol production requires a lot of compression energy? What impact would that have on the cost?

A: We have looked at that, and it would reduce the cost. The potential problem with going to a higher pressure is that the conversion drops off in the reformer reaction. However, we could increase the pressure somewhat and not lose too much, even though we would get a reduction in conversion within the reactor. Because we are already gaining quite a bit by going to the higher temperature, the overall effect would be a gain, and the compression requirements downstream would be lowered. Therefore, we'd have lower capital cost and lower compression charges. At this point in our plans for Phase II we are going to try to simulate conventional reforming operations. But higher pressures are certainly something we will be looking at in the future.

Q: You mentioned the heat transfer in the tubes, the process gas coming in and heating up to 1900 degrees (F), and then going out at a lower temperature. Could you go over that a little bit?

A: Process gases will be coming in at about 750°F. They will travel down the annular space and be heated by a counter current flow of 2300°F combustion gases, which are being cooled (of course) as they rise up through the shell side. By the time the gas reaches this point (pointing at lower end of tube) within the catalyst-filled tube, it's about 1900°F. The gas would then leave the catalyst bed and travel up though the central tube, where it would be cooled by transferring heat (as it passes up) to the annular space. It would be coming off at about 1050°F. The temperature profile of the process gas would be roughly 1000, 1900 to 1050°F.

Q: Is the catalyst a fairly solid matrix or a porous medium?

A: It's a conventional, nickel-type catalyst on an aluminum support, which is a form of a pellet or Raschig ring, and it would be filled within the tube. We've also been thinking about having the ceramic tube impregnated with a catalytic surface to reduce the pressure drop through the tube. But that doesn't seem to be in the immediate future.

Comment: I have just a couple of comments basically on some of what you talked about, and in particular on some of your proposed work. We have been doing some work on silicon carbide in particular and also discussed this with some other folks recently. I think we have identified several things that, with regard to mechanical testing to evaluate these materials, might be of interest and certainly need to be considered. One is behavior at high temperature, which a lot of standard test methods don't account for. We've had some experience with this type of behavior, producing some peculiar results that we couldn't explain. Just recently we think we know what is happening, and we're planning in our work this coming year to make further assessments. We've also got what we feel are some unique measurement capabilities that might be beneficial to your future test program. I'd certainly be interested in talking to you about it.

A: Very good. Thank you.

Q: In this testing program, you give the results of your corrosion testing in terms of weight changes. Is there any correlation between those numbers and some of the results from other corrosion testing you've done with sodium carbonate in terms of surface recession? Did you observe any surface recessions here, or increases in surfaces, or was it not measured?

A: There was no significant surface recession in this case. In this atmosphere, each material except the Lanxide material formed a glassy layer which was quite thin. I have looked at polished cross sections of these materials after the test and the glassy layer is of the order of 25 microns thick. So the only surface recession involved is what was required to produce that thin layer. And I'm afraid there isn't any correlation between these results and any of those in, say, sodium carbonate, or any other alkali-compound type of atmosphere.

HIGH PRESSURE HEAT EXCHANGER

William P. Parks
Babcock & Wilcox

DO NOT MICROFILM
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U. S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGER PROGRAM
PROJECT DESCRIPTION

TITLE: High Pressure Heat Exchanger

COOPERATIVE AGREEMENT NO: DE-AC07-IDI2798

CONTRACTOR: Babcock & Wilcox (B&W)
P. O. Box 11165
Lynchburg, VA 24506-1165

PRINCIPAL INVESTIGATOR: William P. Parks, Jr.

OBJECTIVE: Develop an advanced air heater based on ceramic composite materials capable of high pressure (>100 PSIA) and high temperature (>1500°F) operation. The air heater will feed an indirect-fired gas turbine. Refuse will be the primary combustion source.

TECHNICAL APPROACH: Phase 1 is a paper study designed to assess the potential of the proposed HiPHES System. Major topics were: concept definition, preliminary system design, identification of research and development needs, economic analysis, and planning for subsequent phases. Included in this were a review of the proposed market, review of the materials development status, and identification of a quality assurance program. This was summarized into a final Phase 1 report.

SYSTEM DESCRIPTION: A preliminary design of the high pressure heat exchanger system was established. The system would utilize refuse derived fuel (RDF) for combustion in a rotary kiln/afterburner. Spent flue gas would pass through the heat exchanger tubes and then into a conventional waste heat boiler package (boiler, superheater, condenser, air heater). High pressure (@135 PSI), preheated air from the ceramic air heater would be ducted to a modified gas turbine to generate electricity. Steam from the boiler side would be fed to a steam turbine and also generate electricity. The combined output would be a 8.9 MWe for the entire combined cycle.

The heat exchanger would utilize ceramic composite tubes, high pressure seals, and an insulated metallic tubesheet. The ceramic composite tubes would be based on zirconia and alumina.

CURRENT STATUS (10/89): Phase 1, including a summary report, has been completed. A Phase 2 effort is described in the report and is under review.

ACCOMPLISHMENTS:

The major accomplishments include:

- Design of a combined cycle utilizing a ceramic composite heat exchanger. This covered the entire system package including RDF processing plant, combustor-turbine-heat exchanger, boiler-steam turbine, and gas clean-up system.

- Design of the ceramic composite heat exchanger. This included system design, stress analysis, flow induced vibration, fluid flow, heat transfer, and material requirements.

- Identification of current composite materials for testing.

- Identification of research and development needs in order to move to a field test stage.

- Planning of future work.

FUTURE WORK: A Phase 2 workplan was identified. This included research into key issues such as tube development, seal development, and sub-scale prototype testing. A quality assurance plan was also formulated. Plans for a market survey were made.

CONCLUSIONS: A preliminary design for a high pressure heat exchanger was developed. The key technical development areas are in tube development, seal development and systems design. Economic production of the ceramic composites is also a key issue.

HIGH PRESSURE HEAT EXCHANGER SYSTEM DEVELOPMENT FOR A COMBINED RANKINE-BRAYTON CYCLE IN A RDF-FIRED PLANT

BABCOCK & WILCOX COMPANY

OVERALL PROJECT OBJECTIVES

- TO DEVELOP A HIGH PRESSURE HX UTILIZING CERAMIC COMPOSITE MATERIALS FOR USE WITH AN INDIRECT-FIRED TURBINE FOR POWER GENERATION

- THE HX SHOULD BE ABLE TO PRODUCE >100 PSIA, >1500F PREHEATED AIR

PHASE 1 OBJECTIVES:

- DEVELOP A PRELIMINARY DESIGN FOR THE HiPHES SYSTEM

- IDENTIFY CRITICAL RESEARCH NEEDS

- ESTIMATE ECONOMIC FEASIBILITY

- PLAN PHASE 2 ACTIVITIES

HiPHES
(DOE REQUIREMENTS)

- ADVANCED MATERIALS

- 0.01 QUADS/YR SAVINGS

- ECONOMICAL (COMMERCIALIZATION)

- SIGNIFICANT ADVANCE OVER CURRENT TECHNOLOGY

B&W HiPHES TEAM

BABCOCK & WILCOX

- RESEARCH AND DEVELOPMENT
- POWER SYSTEMS
- REFUSE MARKETING
- PGG ENGINEERING

DUPONT CERAMICS

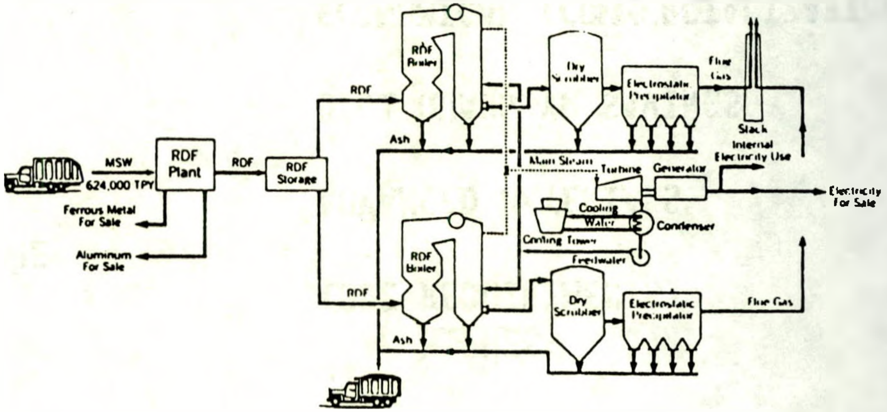
SUPPORT TECHNOLOGY

ALLISON TURBINE DIVISION

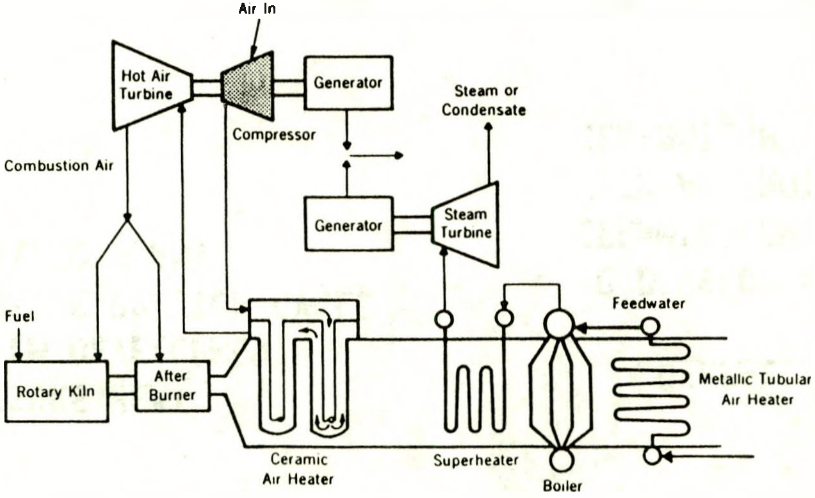
UNIVERSAL ENERGY SYSTEMS

ORNL

COMMERCIAL B&W REFUSE FACILITY



Combined Air Turbine/Steam Turbine Cycle



MAJOR COMPONENT REVIEW

CERAMIC HEAT EXCHANGER

COMBUSTOR (ROTARY KILN)

TURBINE

STEAM CYCLE

RDF PROCESSING SYSTEM

GAS CLEAN-UP SYSTEM

CERAMIC HEAT EXCHANGER

CERAMIC COMPOSITE TUBES

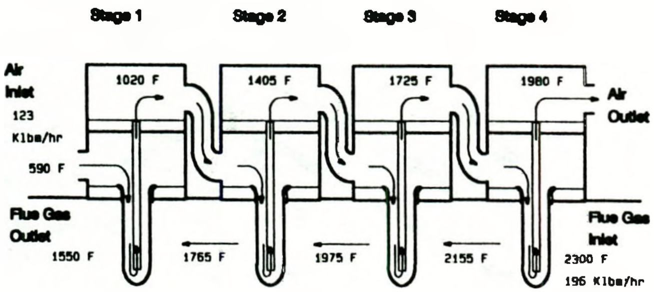
- CORROSION RESISTANCE
- RELIABILITY

HIGH PRESSURE SEALS

- COMPRESSIVE
- GLASS

INSULATED METALLIC TUBESHEET

HiPHES 4 Stage Heat Exchanger Performance

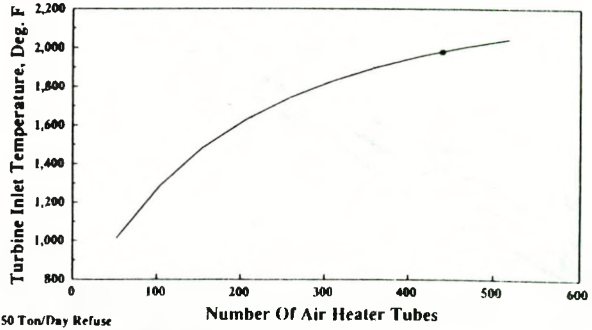


ΔP_{air}/Stage 5 in H_2O
ΔP_{fg}/Stage 0.1 in. H_2O

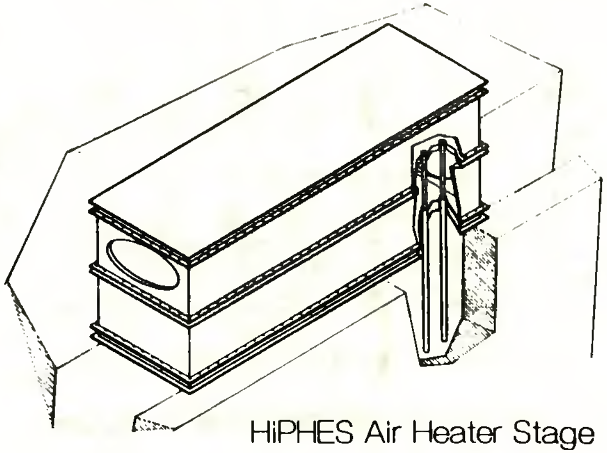
HiPHES

Cycle Performance

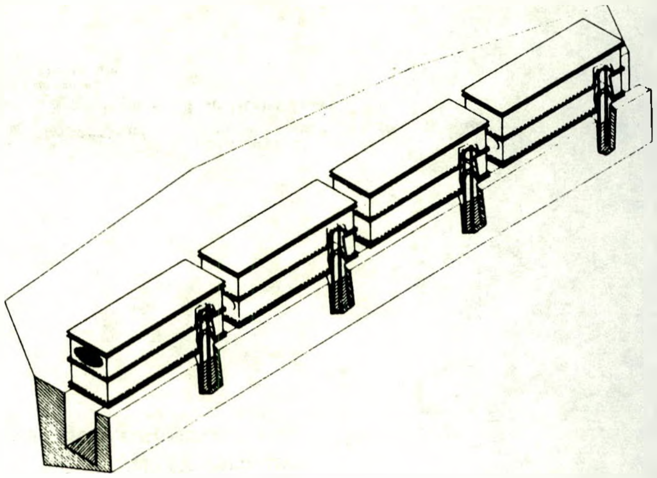
Turbine Inlet Temperature vs. Air Heater Size



250 Ton/Day Refuse
Four Pass Air Heater With Fouling
Outer Tube Length = 12 Ft, Dia. = 6 in.



HiPHES Air Heater Stage



HiPHES In-Line Arrangement

BAYONET-STYLE CERAMIC HEAT EXCHANGER

Geometrical Parameters

Number of Stages	4
Number Tubes/Stage	110

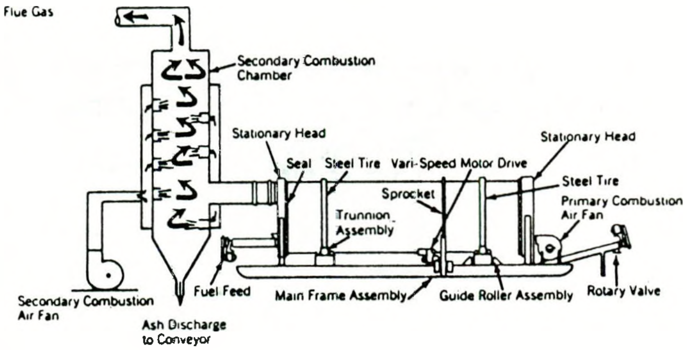
Inline tube arrangement

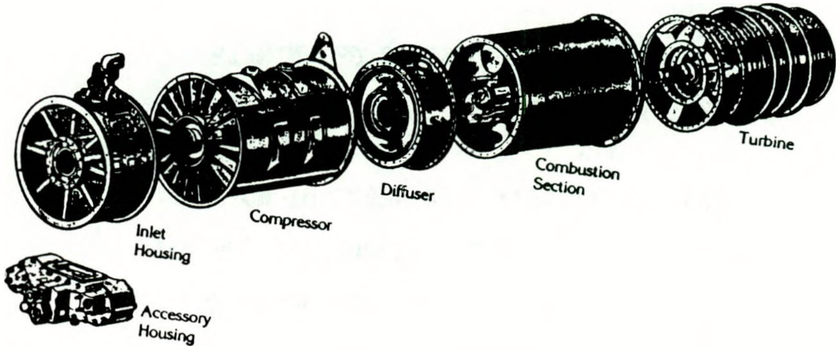
Outer tube	12′x 6″ x .25″
Inner tube	16′ x 4″ x .25″
Tube Spacing	24″

Thermal/Hydraulic Parameters

Air Flow Rate	123,000 lbm/hr
Air inlet temperature	980°F
Air outlet temperature	1980°F
Flue gas flow rate	414,600 lbm/hr
Flue gas inlet temperature	2300°F
Flue gas outlet temperature	1190°F
Heat transferred	3.5×10^6 Btu/hr
ΔP_{AIR} per stage	5 in. H_2O
ΔP_{AIR} per stage	0.1 in. H_2O

Rotary Kiln/Afterburner System





COMMERCIAL ALLISON 501-K TURBINE

SYSTEM OUTPUT

(1980F Turbine Inlet Temperature)

Total Output	8.9 MW_{ELEC}
Air Cycle	3.6 MW_{ELEC}
Steam Cycle (875 psia, 890F)	5.3 MW_{ELEC}

-REFUSE INCINERATION

-HAZARDOUS WASTE INCINERATION

-LOW GRADE COAL COMBUSTION

-WOOD, RICE HULLS, ETC.

-ADD-ON TO INDUSTRIAL FURNACE EXHAUST STREAMS

-CARBON CRACKING IN PETROLEUM REFINERIES

-CONTAMINATED SOILS CLEAN-UP

MUELLER REPORT
OCTOBER 1987

APPLICATION	TOTAL POTENTIAL SAVINGS
Power Generation	$1,472,000,000 Annually

REFUSE MARKET FORECAST

Year	Number of Plants	Capacity
1986	68	35,000 Tons/day
2000	278	241,000 Tons/day

EXPENDITURES 1986-2000: $19 BILLION

POTENTIAL BENEFITS OF HiPHES TO REFUSE

- 30% PROJECTED INCREASE IN CYCLE EFFICIENCY
- IMPROVED GAS COMBUSTION FOR REDUCED EMISSIONS INCLUDING NO_X, HCl, etc.
- ASH COMES OUT IN GLASSY SLAG FORM INSTEAD OF PARTICULATE
- 30-50% REDUCTION IN WASTE WATER NEEDS
- ABILITY TO COMBUST MANY TYPES OF WASTES
- MODULAR DESIGN FOR ABILITY TO SIZE IN 250 TON/DAY INCREMENTS
- UTILIZES CURRENT STRENGTHS IN B&W
    - o RDF TECHNOLOGY
    - o SYSTEMS DESIGN AND PACKAGING
    - o MARKET PRESENCE
    - o MATERIALS TECHNOLOGY

KEY DEVELOPMENT ISSUES

- ADVANCED CERAMIC COMPOSITE MATERIALS
- HIGH PRESSURE SEALS
- RELIABILITY
  - -COMPONENT
  - -SYSTEM
- COST

FUEL ANALYSIS

ANALYSIS	MASS	RDF	DEPOSITS
	-------WEIGHT	PERCENT	--------
CARBON	27.9	36.1	---
HYDROGEN	3.7	5.1	---
OXYGEN	20.7	31.6	---
NITROGEN	0.2	0.8	---
SULFUR	0.1	0.1	2-15
CHLORINE	0.1	0.1	0.05-25%
H_2O	31.3	20.2	---
ASH (CA, NA, K, SI, AL, PB, FE)	16.0	6.0	OXIDES

CORROSION

MATERIAL	STEAM	ALKALI
ALUMINA	.1 MM/YR	.1 MM/YR
ZIRCONIA	.1 MM/YR	.1 MM/YR
SILICON CARBIDE	6.0 MM/YR	3-7 MM/YR
SOURCE:	NASA LEWIS	ORNL

OPTIMAL COMPOSITE PROPERTIES

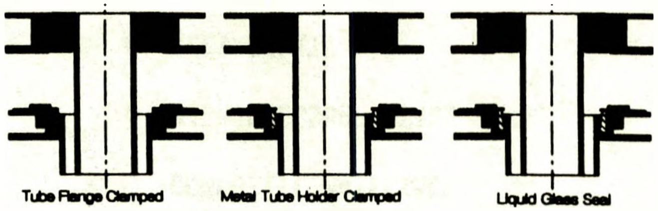
PROPERTY	DESIRED VALUE	HOW PRODUCED
POROSITY	10%	PROCESSING, DEPENDANT, MATRIX DEPENDANT
PERMEABILITY	0.1 SCFM AT 150 PSI	PROCESSING, COATINGS
STRENGTH 70°F	50-100 KSI	FIBER, PROCESSING INTERFACE CONTROL
2400°F	40-50 KSI	SAME
FRACTURE TOUGHNESS PARAMETER	20+	FIBER, MATRIX, PROCESS DEPENDANT
OXIDATION RESISTANCE	EXCELLENT	USE OXIDE FIBERS AND MATRICES
CORROSION RESISTANCE	MINIMAL REACTION	USE STABLE, COMPATIBLE MATRICES OR COATINGS

POTENTIAL CERAMIC MATERIAL SYSTEMS

ALUMINA-ZIRCONIA COMPOSITES

ALUMINA-SILICON CARBIDE COMPOSITES

SILICON CARBIDE-SILICON CARBIDE COMPOSITES



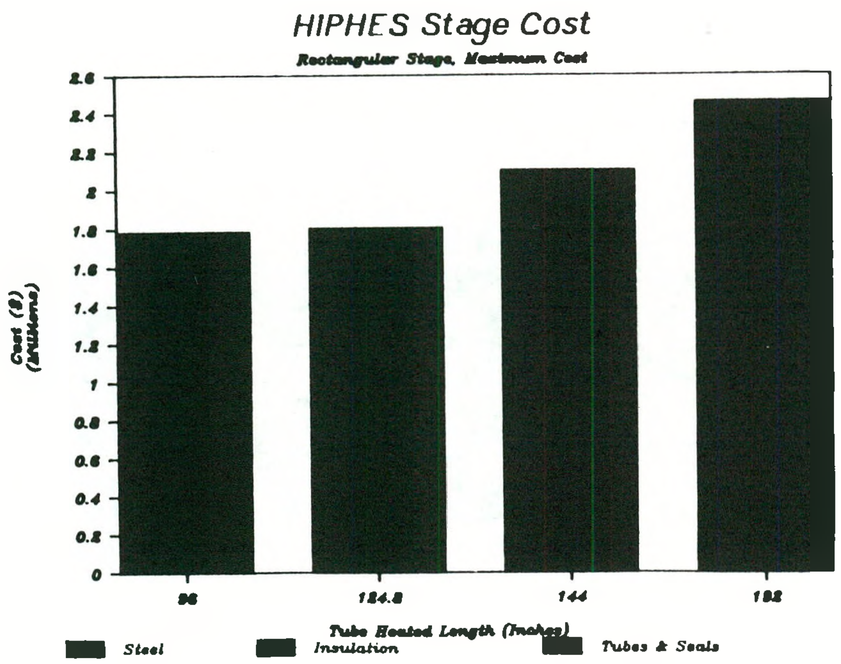
HiPHES Tube-to-Tubesheet Seal Concepts

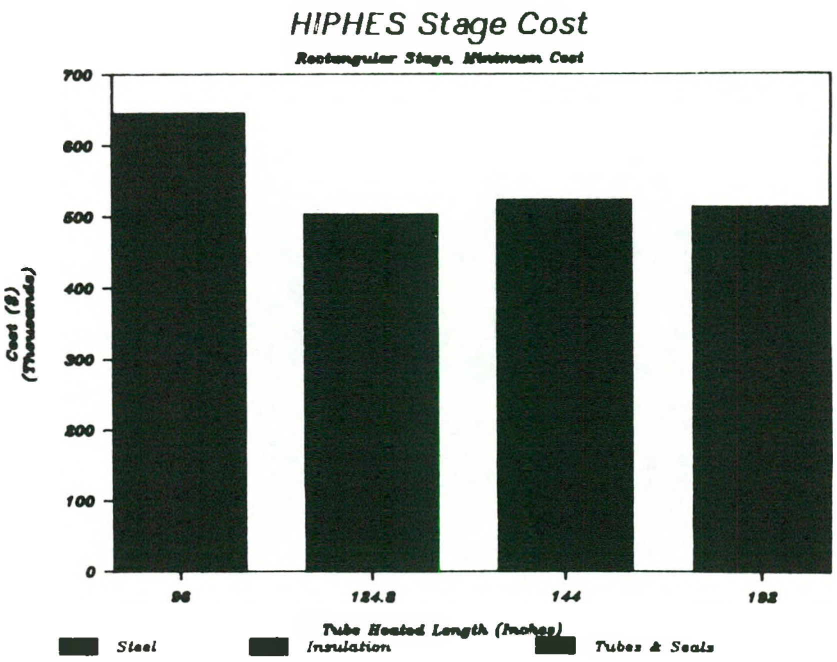
MATERIAL TRENDS FOR THE NEXT FIVE YEARS

- DEVELOPMENT OF THERMOCHEMICALLY STABLE FIBERS
- DEVELOPMENT OF MULTIPLE TOUGHENING TECHNIQUES
- DEVELOPMENT OF NEW PROCESSING TECHNIQUES
  - LANXIDE
  - SELF-PROPAGATING, HIGH-TEMPERATURE SYNTHESIS
  - MICROWAVE SINTERING
  - SOL-GEL
- DEVELOPMENT OF CHARACTERIZATION TECHNIQUES

ESTIMATED COST FOR 1000 TPD HiPHES FACILITY

COMPONENT	COST (MILLIONS)
4 KILNS @ 1 MILLION PER KILN	4
4 AIR TURBINES @ 1.5 MILLION PER TURBINE	6
4 250 TPD BOILER UNITS INCLUDING GAS CLEAN-UP	22
4 CERAMIC HX	8
4 STEAM TURBINES @ 1 MILLION EACH	4
1 1000 TPD RDF UNIT	25
MISC (BUILDINGS, DUCTINGS)	28
	110 MILLION DOLLARS





PHASE 2 PRELIMINARY PLANS

1. MATERIALS DEVELOPMENT AND CHARACTERIZATION
2. SEAL DESIGN AND TESTING
3. HEAT EXCHANGER DESIGN
4. SYSTEM DESIGN AND COMPONENT TESTING
5. PROTOTYPE TEST
6. SYSTEM COMPONENT VERIFICATION
    - KILN EMISSIONS
    - TURBINE PERFORMANCE
7. SITE ANALYSIS
8. PRESSURE VESSEL CODE DEVELOPMENT/ACCEPTANCE
9. QA PLAN
10. MARKET SURVEY

CONCLUSIONS

- A COMBINED CYCLE SYSTEM BASED ON A HIGH PRESSURE CERAMIC HEAT EXCHANGER HAS BEEN DESIGNED

- CRITICAL RESEARCH NEEDS WERE DETERMINED IN ORDER TO DEVELOP THE SYSTEM

- POTENTIAL MARKET APPLICATIONS WERE IDENTIFIED

CURRENT AND PLANNED HiPHES MATERIALS SUPPORT

J. I. Federer
ORNL

U. S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGER PROGRAM
TASK DESCRIPTION

TITLE: Current and Planned HiPHES Materials Support
WORK PROPOSAL NUMBER: CEED009
CONTRACTOR: Oak Ridge National Laboratory

PRINCIPAL INVESTIGATORS: R. A. Bradley, 615/574-6094 and J. I. Federer, 615/574-5131

OBJECTIVE: Provide support to contractors in testing and selection of materials for heat exchanger tubes and other critical components.

TECHNICAL APPROACH: (1) Provide existing materials technology in the form of papers, reports, etc., which are relevant to the contractors needs; (2) perform special laboratory tests, such as mechanical or corrosion testing; (3) assist the contractors in performing corrosion tests or other tests at industrial sites.

SYSTEM DESCRIPTION: A laboratory furnace is being used to investigate the effects of corrosion on flexure strength of eight candidate materials for the Stone & Webster Engineering Corporation project. Flexure bars of the materials are being exposed to an H_2-CO-CO_2-CH_4-H_2O atmosphere at 1260°C for 500 h with an intermediate examination after 100 h.

CURRENT STATUS (9/89): The 500 h exposure test has been completed. Flexure tests and specimen examinations are in progress.

ACCOMPLISHMENTS: Weight changes and flexure strengths were determined after the 100 h exposure. The results indicated that certain materials are viable candidates, but identification of best candidates depends upon results to be obtained.

FUTURE WORK: Negotiable.

CONCLUSIONS: None yet available.

BIBLIOGRAPHY: None.

CURRENT AND PLANNED HiPHES MATERIALS SUPPORT

J. I. FEDERER AND A. J. MOORHEAD
METALS AND CERAMICS DIVISION
OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

U. S. DEPARTMENT OF ENERGY
OFFICE OF INDUSTRIAL PROGRAMS
DIVISION OF WASTE ENERGY REDUCTION
WASTE ENERGY RECOVERY BRANCH

ADVANCED HEAT EXCHANGERS PROGRAM REVIEW
HERNDON, VIRGINIA
OCTOBER 11 AND 12, 1989

MATERIALS SUPPORT CAN BE PROVIDED IN VARIOUS FORMS

- REVIEW LITERATURE ON PROPERTIES, AVAILABILITY, FABRICABILITY, ETC.
- INTERPRET PUBLISHED DATA
- SELECT CANDIDATE MATERIALS ON THE BASIS OF CURRENT KNOWLEDGE
- DESIGN EXPERIMENTS TO OBTAIN SPECIFIC RESULTS
- CONDUCT TESTS, INTERPRET RESULTS, PREPARE PAPER OR REPORT
- PROVIDE INFORMATION CONCERNING FACILITIES AT ORNL, SPECIFICALLY THE HTML

CANDIDATE HEAT EXCHANGER MATERIALS WERE TESTED

PROJECT

STONE & WEBSTER ENGINEERING CORP.

PURPOSE

COMPARE CORROSION BEHAVIOR AND FLEXURE STRENGTH

METHOD

CORROSION TEST IN SIMULATED STEAM REFORMER ATMOSPHERE

MEASURED QUANTITIES

WEIGHT CHANGE
FLEXURE STRENGTH AT ≈25°C

OTHER EXAMINATIONS

SEM-EDX, OPTICAL MICROSCOPY, XRD

EIGHT CANDIDATE MATERIALS WERE TESTED

MATERIAL	I. D.	MANUFACTURER
SINTERED ALPHA SIC	SA	CARBORUNDUM
CRYSTAR, HIGH PURITY	CHP	NORTON
CRYSTAR, STANDARD PURITY	CSP	NORTON
REACTION BONDED SIC	RBSC	COORS
SI3N4-BONDED SIC	SNSC	FERRO
SIC-16 VOL.% TIB2	ST	CARBORUNDUM
Al2O3-SICw	CAS	COORS
Al2O3-SIC COMPOSITE	LAS	LANXIDE

CORROSION TEST CONDITIONS SIMULATED STEAM REFORMER CONDITIONS

ATMOSPHERE (vol.%)

H_2	53.3
CO	12.7
CO_2	3.7
CH_4	0.3
H_2O	30.0

FLOW RATE

2400 cm^3/min in 6.4 cm I.D. TUBE

TEMPERATURE

1260°C

PRESSURE

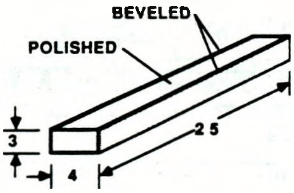
1 atm

DURATION

100 h, 500 h

WEIGHT CHANGES AND FLEXURE STRENGTHS WERE DETERMINED AFTER EACH EXPOSURE PERIOD

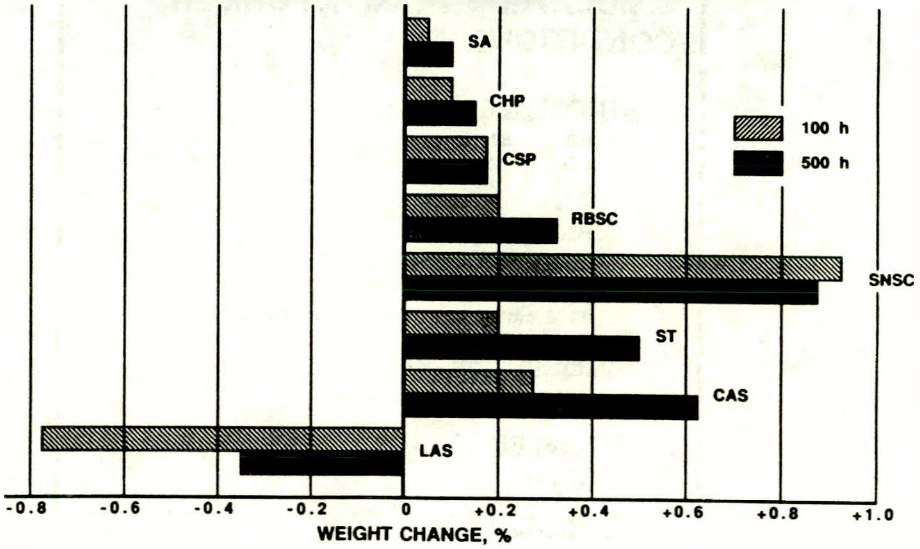
NUMBER OF SPECIMENS OF EACH MATERIAL, TOTAL NUMBER IN ( )		
CORRODED AT 1260°C	WEIGHED	FLEXURE TESTED AT ≈25°C*
	24 (192)	8 (64)
16 (128), 100 h	16 (128)	8 (64)
8 (64), 500 h	8 (64)	8 (64)



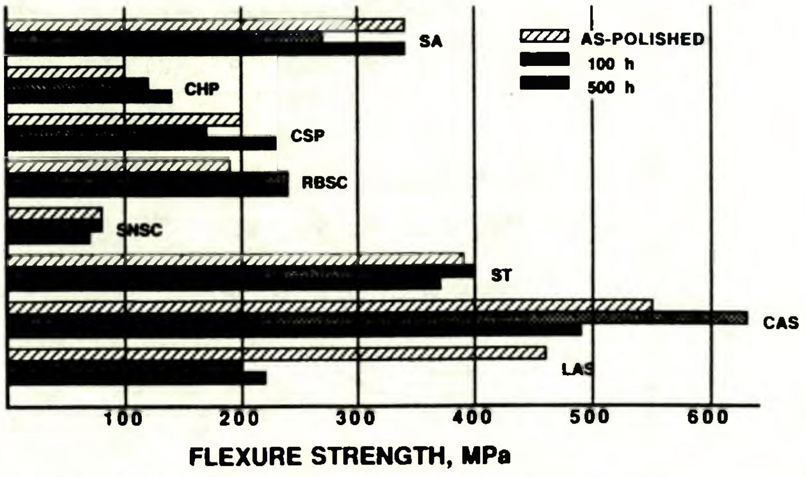
SPECIMEN

* 4-POINT LOADING WITH 19.05 mm OUTER SPAN AND 6.35 mm INNER SPAN

WEIGHT CHANGES AT 1260°C VARIED WITH COMPOSITION



CORROSION AT 1260°C CHANGED FLEXURE STRENGTHS

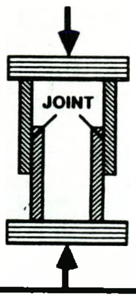


BASED ON INITIAL RESULTS, FOUR MATERIALS* WERE SELECTED FOR FURTHER EVALUATION

MATERIAL	STRENGTH		WEIGHT CHANGE		LARGE PORES	AVAILABLE AS TUBING	
	LOW	ACCEP	LOW	HIGH		YES	NO
SA*		X	X			X	
CSP*		X	X			X	
RBSC*		X				X	
LAS*		X		X		?	
CHP	X		X		X		X
SNSC	X			X	X	X	
ST		X		X			X
CAS		X		X			X

ADDITIONAL TESTS WILL BE USED TO IDENTIFY THE MOST SUITABLE MATERIAL

- CORROSION OF CANDIDATE MATERIALS
  - 1260°C FOR FLEXURE BARS AND CERAMIC/CERAMIC JOINTS
  - 565°C FOR CERAMIC/METAL JOINTS
  - 2000 h
  - 1atm
  - SIMULATED STEAM REFORMER ATMOSPHERE
- EXPOSED FLEXURE BARS
  - WEIGHT CHANGES
  - FLEXURE STRENGTH ($\bar{\sigma}_f$) AT 1260°C
  - TIME TO FAILURE AT 1260°C AND 25 TO 75% $\bar{\sigma}_f$
- EXPOSED JOINTS
  - STRENGTH OF CERAMIC/CERAMIC AT 1260°C
  - STRENGTH OF CERAMIC/METAL AT 565°C



DISCUSSION

Current and Planned HiPHES Material Support

J. I. Federer

Q: In this testing program, you give results of corrosion testing in terms of weight changes. Is there any correlation between those results and some of the results from corrosion testing with sodium carbonate in terms of surface recession. Did you observe surface recessions or increases in surfaces, or was corrosion measured in this way.

A: There were no significant surface recessions in these materials. In this atmosphere, each material except the Lanxide material formed a thin glassy layer. I have examined polished cross sections of these materials after the test, and the glassy layers were about 25 μm thick. So the only surface recession involved is the amount of material required to produce the thin layer. There isn't any correlation between these results and those in sodium carbonate or other alkali compound containing atmosphere.

ORNL OVERVIEW OF MATERIALS SUPPORT WORK

Michael A. Karnitz
ORNL

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
MATERIALS FOR ADVANCED HEAT EXCHANGER PROGRAM
TASK DESCRIPTION

TITLE: ORNL Overview of Materials Support Work
WORK PROPOSAL NO: CEED009
CONTRACTOR: Oak Ridge National Laboratory

PRINCIPAL INVESTIGATOR: R. A. Bradley, 615/574-6094 and M. A. Karnitz, 615/574-5150

DESCRIPTION OF WORK: This program provides material technology support to the Waste Energy Utilization Branch of the Office of Industrial Programs Conservation and Renewable Energy Technology and their contractors in two general categories: (1) heat exchanger project support and (2) technology development. The heat exchanger technology support involves assistance to DOE contractors in the development of advanced heat exchanger systems. The operating conditions of these systems require selection of metallic alloys and ceramics for critical components such as heat exchanger tubes, seals, and structural members. The purpose of the work is to identify potential material problems that could significantly affect system performance and reliability. The potential problems are resolved by application of existing materials technology or by performing the analysis and testing required for materials qualification. The technology development portion of the program focuses on materials and processes that enhance industrial acceptance of ceramic heat exchangers. The cost, performance, and reliability of ceramic materials and components are critical to the overall cost effectiveness of advanced heat exchanger concepts. The objective of this part of the program is to develop improved materials and processes, to overcome the factors that increase cost (e.g., ceramic powder production and component fabrication) or that limit performance and reliability (e.g., corrosion and toughness).

ORNL OVERVIEW OF MATERIALS SUPPORT WORK

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OFFICE OF INDUSTRIAL PROGRAMS
DIVISION OF WASTE ENERGY REDUCTION
WASTE ENERGY RECOVERY BRANCH

ADVANCED HEAT EXCHANGERS PROGRAM REVIEW
HERNDON, VIRGINIA
OCTOBER 11 AND 12, 1989

RESEARCH ON MATERIALS FOR INDUSTRIAL WASTE HEAT UTILIZATION

OBJECTIVE: **Develop Materials for Heat Exchange Systems that transfer Waste Heat from the Flue Gases of Industrial Furnaces to useful energy forms in other parts of the Industrial process.**

Two General Categories:

- **Heat Exchanger Project Support**
- **Technology Development**

HEAT EXCHANGER PROJECT SUPPORT

The Purpose of this Component of the Program, as Related to Heat Exchanger Systems, is to provide materials technology support to DOE industrial subcontractors.

- ORNL performing laboratory corrosion test for both:
  - HiPHES - High Pressure Heat Exchanger Systems
    - B&W and STI - Power generation from municipal or hazardous waste incinerator
    - SWEC - Developing a new convective steam reformer
  - AHET - Advanced Heat Exchanger Technology
    - B&W Developing recuperator for chemical or hazardous waste exhaust stream
- ORNL assisting in selection of ceramic materials

TECHNOLOGY DEVELOPMENT

The technology development portion of the program focuses on materials and process that enhance industrial acceptance of HiPHES and ceramic recuperators

- Gelcasting - Generic near net-shape forming technology
- Ceramic Coatings - Laboratory corrosion test on a variety of ceramic coatings
- Corrosion Probe - New Diagnostic Tool to provide industry capability to evaluate the corrosion potential for ceramic heat exchangers

TECHNOLOGY DEVELOPMENT (CONTINUED)

The technology development component of the program also includes several subcontractor tasks targeted at improving materials and processes

- Synthesis of high grade SiC powder
- HiPHES design methodology studies
- Membership - Penn State Center for advanced materials
- Study of biological hazards of fiber residue
- Develop manufacturing capability to fabricate whisker-toughened alumina tubes

METHODOLOGY STUDIES FOR CONTINUOUS-FIBER CERAMIC-MATRIX COMPOSITE

Michael A. Karnitz
ORNL

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
MATERIALS FOR ADVANCED HEAT EXCHANGER PROGRAM
TASK DESCRIPTION

TITLE: Methodology Studies for Continuous-Fiber Ceramic-Matrix Composite
WORK PROPOSAL NO: CEED009
CONTRACTOR: Oak Ridge National Laboratory and two university subcontractors (To be identified in FY 1990)

PRINCIPAL INVESTIGATORS: R. A. Bradley, 615/574-6094 and M. A. Karnitz, 615/574-5150

OBJECTIVE: The proposed effort is to develop and validate design methodologies for use of Continuous-Fiber Ceramic-Matrix Composite (CFCC) materials for advanced heat exchanger systems

TECHNICAL APPROACH: The general approach includes using two university subcontractors to work with ORNL to develop the design methodologies. The two projects will be carried out in parallel and in a similar phase manner. One subcontractor will focus on analysis methods, the other on design criteria. The initial emphasis will be on two or three continuous-fiber ceramic-matrix composite reference material systems. These composites are presently under consideration in the HiPHES program.

SYSTEM DESCRIPTION: Continuous-Fiber Ceramic-Matrix Composite Materials potentially offer advantages over today's materials for high-temperature heat recovery. They are generally more corrosion resistant in hostile environments than metal alloys. They are also tougher than monolithic ceramics. They can be designed to fail gradually in contrast to catastrophic failures with monolithic ceramics. However, there is a need to develop design methodologies for predicting stress in elements of continuous-fiber ceramic composites.

CURRENT STATUS: ORNL will be soliciting university subcontractors in FY 1990. Detailed statements of work are presently being prepared.

ACCOMPLISHMENTS: ORNL has completed a preliminary study to identify design needs for these composite materials. The report identifies several development and validation needs that must be satisfied in order to establish a viable design methodology for these new continuous-fiber ceramic composites materials for advanced heat exchanger systems.

CONCLUSIONS: The goal of the project is to develop and validate design methodologies for use of continuous-fiber ceramic composite materials to foster commercialization of high pressure heat exchanger systems (HiPHES). The project will be initiated in FY 1990 and will involve two universities and ORNL. The project emphasis will be on analysis methods and design criteria.

BIBLIOGRAPHY:

1. J. J. Blass, Design Methodology Needs for Fiber-Reinforced Ceramic Heat Exchangers (Draft), ORNL/TM-11012.

METHODOLOGY STUDIES FOR CONTINUOUS-FIBER CERAMIC-MATRIX COMPOSITE

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DIVISION OF WASTE ENERGY REDUCTION
WASTE ENERGY RECOVERY BRANCH

ADVANCED HEAT EXCHANGERS PROGRAM REVIEW
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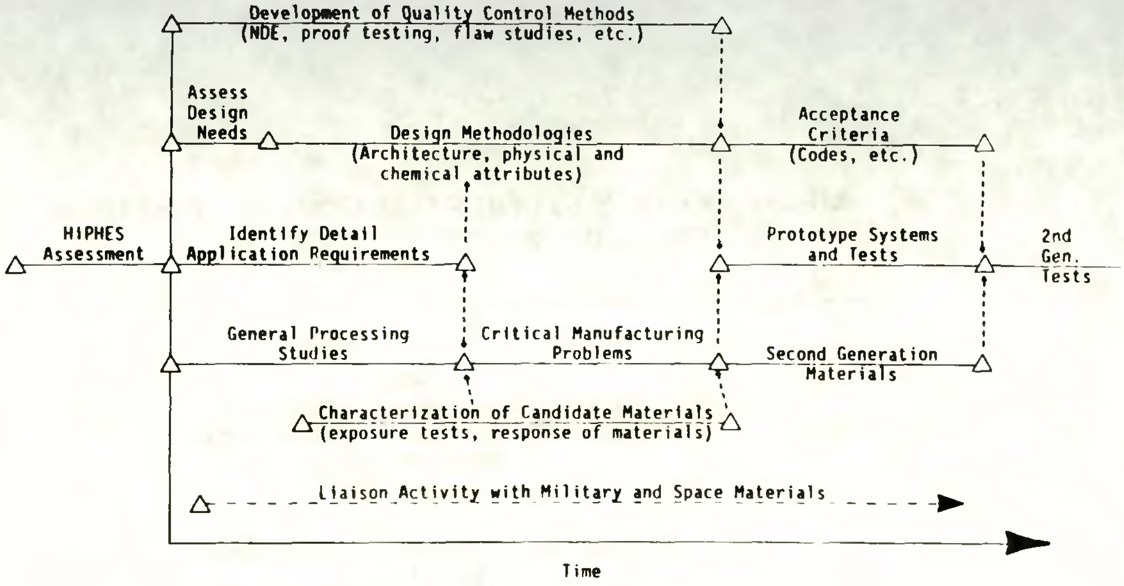
INTRODUCTION - PROJECT DESCRIPTION

We are attempting to develop and validate design methodologies for use of Continuous-Fiber Ceramic-matrix Composite (CFCC) materials for advanced heat exchangers systems

- HiPHES program based on rapidly developing advanced materials technology, including CFCC

- HiPHES assessment recommended time-phase program which provides the design methodologies



Recommended HiPHES Development Program

INTRODUCTION - HISTORY AND JUSTIFICATION

Continuous-fiber ceramic-matrix composites potentially offer advantages over today's materials for high-temperature waste heat recovery

- Stronger and tougher than monolithic ceramics
- More corrosion resistant in hostile environments than alloys
- Can fail "gracefully" in contrast to catastrophic failures with monolithic ceramics

---

There is a need to develop design methodologies for predicting stresses in elements of continuous fiber-ceramic matrix composites

- As a function of load
- As a function of "composite architecture"
  - Fiber volume, fiber orientation, and porosity of various fiber layups and cloths of various weaves

INTRODUCTION - GOALS

The objective of the proposed effort is to develop and validate design methodologies for the use of CFCC materials to foster the commercialization of high pressure heat exchanger systems

- Development of analysis methods
- Development of design criteria

PROJECT PARTICULARS

DOE - sponsored studies will be initiated soon and will involve two universities and ORNL

- One university will focus on analysis methods; the other, on design criteria
- ORNL will integrate and guide the university work as well as contribute in unique areas of expertise

OUTLINE OF ANALYSIS METHODS AND DESIGN CRITERIA

Analysis Methods

1.0 Micromechanical modeling
  - (a) Fiber-matrix interface modeling
  - (b) Fabric geometry modeling
  - (c) Laminate modeling
  - (d) Representation of properties

2.0 Macromechanical modeling

3.0 Fracture mechanics methods

OUTLINE OF ANALYSIS METHODS AND DESIGN CRITERIA

Design Criteria

1.0 Failure mechanisms

- (a) Matrix cracking
- (b) Time-dependent behavior
- (c) Fatigue
- (d) Ultimate strength and toughness
- (e) Test methods

2.0 Design limits and guidelines

- (a) Phenomenological design limits
- (b) Acceptance criteria
- (c) Probabilistic design methods
- (d) Risk/reliability assessments

THE METHODOLOGY STUDIES FOR CFCC WILL START IN EARLY CALENDAR YEAR 1990 AND REQUIRE 3 TO 5 YEARS TO COMPLETE

Detailed statements of work being prepared

- Phase I Survey of other pertinent methodologies
- Phase II Adaptation of key elements of the respective methodologies to HiPHES reference materials
- Phase III Complete the structural validation and testing of reference material and initiate work on alternate composite materials
- Phase IV Complete structural validation and testing of alternate composite materials

CONCLUSION

THE GOAL IS TO DEVELOP AND VALIDATE DESIGN METHODOLOGIES FOR THE USE OF CFCC MATERIAL TO FOSTER COMMERCIALIZATION OF HiPHES

- Project will be initiated in early FY 1990 and will involve two universities and ORNL
- Project emphase on analysis methods and design criteria
- Three to five years projects

DISCUSSION

Methodology Studies for Continuous Fiber Ceramic-Matrix Composite

Michael A. Karnitz

Q: I have a question relating to the internal flaws that were referred to earlier in another presentation. Are you making any plans to find better ways to detect internal flaws by non destructive testing so that people can evaluate pieces and determine what should be rejected due to internal flaws that could ultimately lead to failure? These flaws might be fairly small in size and difficult to detect.

A: Are you referring to composites when you ask that question? The presentations tomorrow, on strength limiting flaws from B&W and Idaho National Lab, are going to address the NDE and acceptance criteria development for monolithics. We haven't started a similar task on the composites. We want to wrap up the NDE project on the monolithics before we get into the composites. The composites are different because when you make a composite, there are multiple "flaws" all through it. One of the things we are going to have to be able to decide is which flaws should be considered strength limiting and which are just there and are inherent to the structure. We expect that fiber architecture can give us a little guidance on that.

GELCASTING OF MONOLITHIC CERAMICS AND CHOPPED FIBER REINFORCED COMPOSITES

Albert C. Young
ORNL

U. S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS MATERIALS FOR ADVANCED HEAT EXCHANGERS PROGRAM TASK DESCRIPTION

TITILE: Gelcasting of Monolithic Ceramics and Chopped Fiber Reinforced Composites
WORK PROPOSAL NO.: CEED009
CONTRACTOR: Oak Ridge National Laboratory

PRINCIPAL INVESTIGATOR: A. C. Young, (615) 576-5215

OBJECTIVE: Continue studies on the use of gelcasting technology in the production of monolithic ceramics and fiber reinforced ceramic composites.

TECHNICAL APPROACH: For all systems, the dispersion of ceramic powders in the gelcast solution is examined in terms of solids loading, viscosity, and castability. Gelation, drying, binder burn-out, and sintering processes of ceramic parts will be monitored. In addition, mechanical tests, SEM, Auger, and TEM will be used to study the ceramic composite properties.

SYSTEM DESCRIPTION: Monolithic: SiC. Composite: FP and PRD 166 reinforced alumina.

ACCOMPLISHMENTS: Gelcasting of oxide (alumina) has been completed. Initial studies on the fiber reinforced oxide composites were also completed and problems were identified.

CURRENT STATUS (9/89): Dispersion study of SiC powder in water and production of composites with high concentration of ceramic fiber are in progress.

CONCLUSIONS: Initial investigations of oxide-matrix composites reinforced with Du Pont FP and PRD 166 fibers indicate that gelcasting is a promising forming technique. The degree of uniformity in the distribution of fibers in the ceramic body is critical to the quality of the composite. Matrix densification increases as the sintering temperature increases; however, both FP and PRD 166 show thermal degradation in the form of grain growth and void formation. PRD 166 exhibits better stability at high temperature. Current results indicate that a thin carbon coating on the fibers is not effective in restricting reaction with the matrices. A thicker coating of a less reactive material is needed to preserve fiber integrity and prevent reaction between fiber and matrix.

FUTURE WORK: Current and future work involves searching for an adequate coating material and studying the effect of fiber concentration upon slurry viscosity and matrix densification. Further investigations of gelcasting with continuous fibers as well as other matrix materials such as SiC are planned.

BIBLIOGRAPHY: None

GELCASTING OF MONOLITHIC CERAMICS AND CHOPPED FIBER REINFORCED COMPOSITES

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DIVISION OF WASTE ENERGY REDUCTION
WASTE ENERGY RECOVERY BRANCH

ADVANCED HEAT EXCHANGERS PROGRAM REVIEW
HERNDON, VIRGINIA
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OUTLINE

- PROJECT DESCRIPTION: GELCAST NEAR-NET-SHAPE PROCESS
- RESEARCH - OVERVIEW
- RESULTS AND CONCLUSIONS

TRADITIONAL CERAMIC FORMING TECHNIQUES

PRESSING - Cold and hot pressing

CASTING - Slip casting

PLASTIC FORMING - Extrusion and injection molding

OTHERS - Tape forming, flame spray, green machining

LIMITATIONS

Not cost efficient

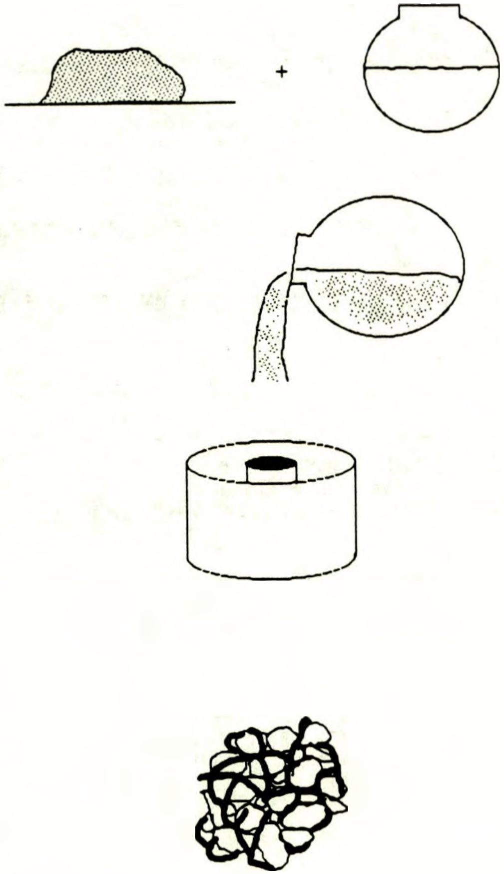
Complicated and difficult to execute

Simple shapes only

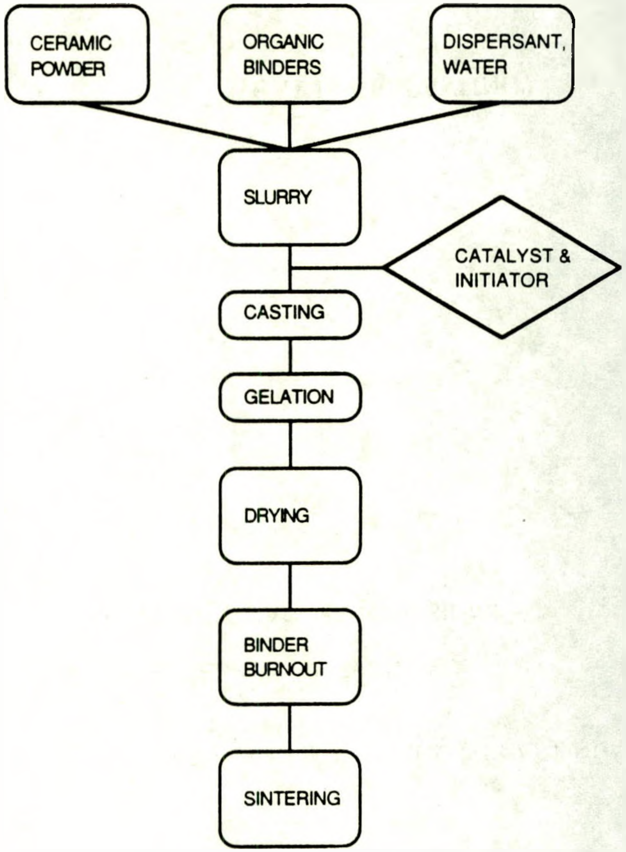
Poor reliability and reproducibility

WHAT IS GELCASTING?

A **new method** of making ceramic parts by using a ceramic powder suspension containing a small amount of **polymerizable** ingredients to obtain a **controllable casting and solidification process.**



PROCESS FLOW CHART



ADVANTAGES

- Applicable to all ceramic systems
- High solids loading, low viscosity slurry
- Complex shapes possible
- Strong, machinable green body
- Minimal final machining required

OBJECTIVES

- Fundamental understanding of the gelcasting processing
- Development of gelcast process for oxide and non-oxide ceramic systems
- Application of this technology to the production of ceramic fiber reinforced ceramic composites

MONOLITHICS - CURRENT STATUS

- Gelcasting of Al_2O_3 completed

    Slurry - solids loading, viscosity, dispersion
    mold materials
    Gelation process
    Drying
    Binder burn-out

- Currently working on gelcasting of SiC

    Dispersion of SiC powder in water

- Gelcasting of Si metal, ZrO_2, Sialon, etc.

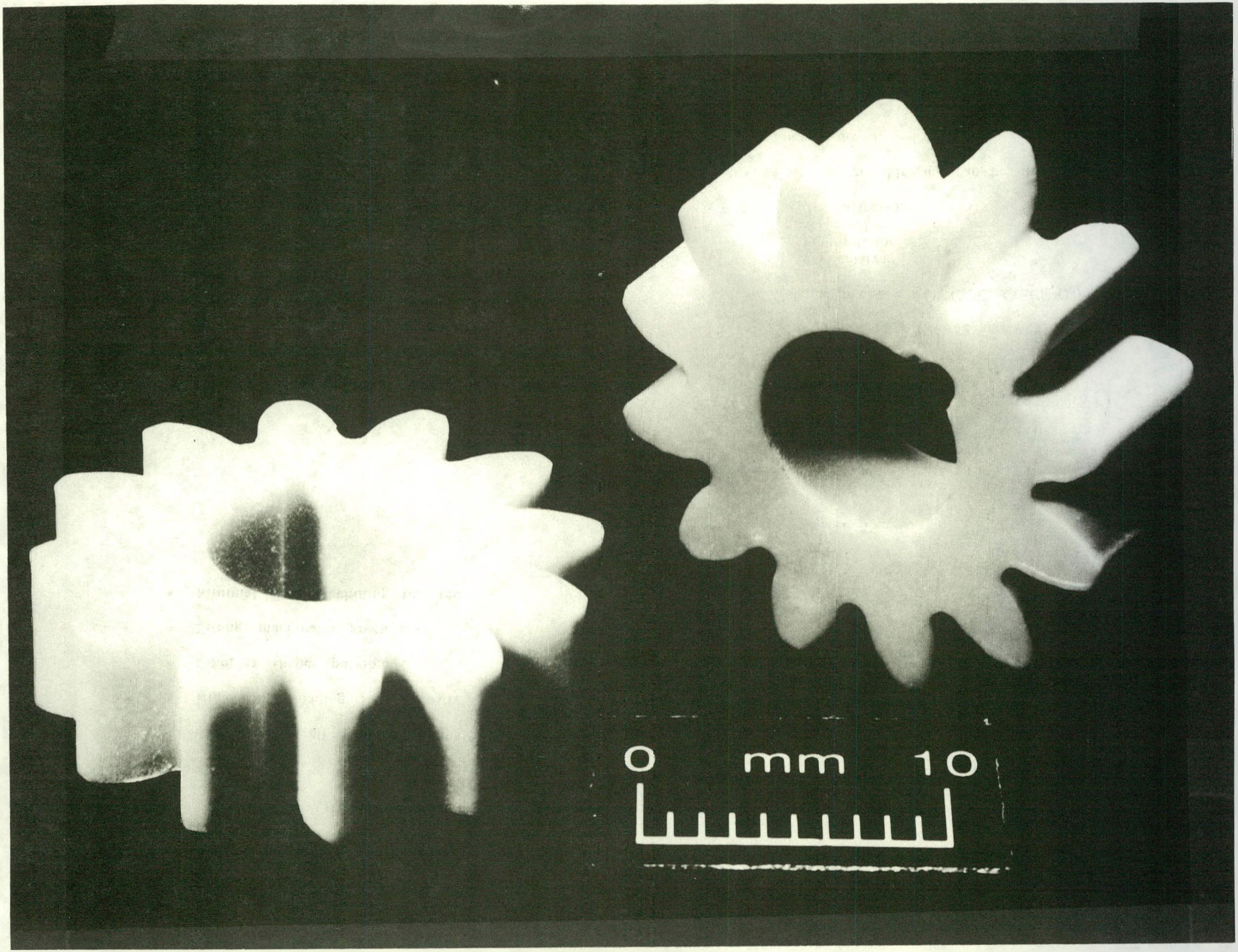
RESULTS

Al_2O_3

- 52-60 vol% slurry (RC152 and RCHP)
- Gelation process = f (T, C_c, C_i, C_s, C_d, O_2)

    c - catalyst
    i - initiator
    s - solid
    d - dispersant

- Knit line possible, but can be eliminated
- Sintered density > 99% T. D.
- Complex shapes are routine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COMPOSITES - CURRENT STATUS

- 6 vol% SiC whisker/Al_2O_3
- Initial studies on the gelcasting of fiber reinforced ceramic matrix composites completed
  - FP and PRD 166/Al_2O_3
- Continuing work on fiber reinforced composites
  - Fiber coating, coating materials
  - Fiber concentration
  - Fiber matrix interface
  - Mechanical properties

RESULTS

FP alumina fibers/Al_2O_3

PRD 166 alumina-zirconia fibers/Al_2O_3

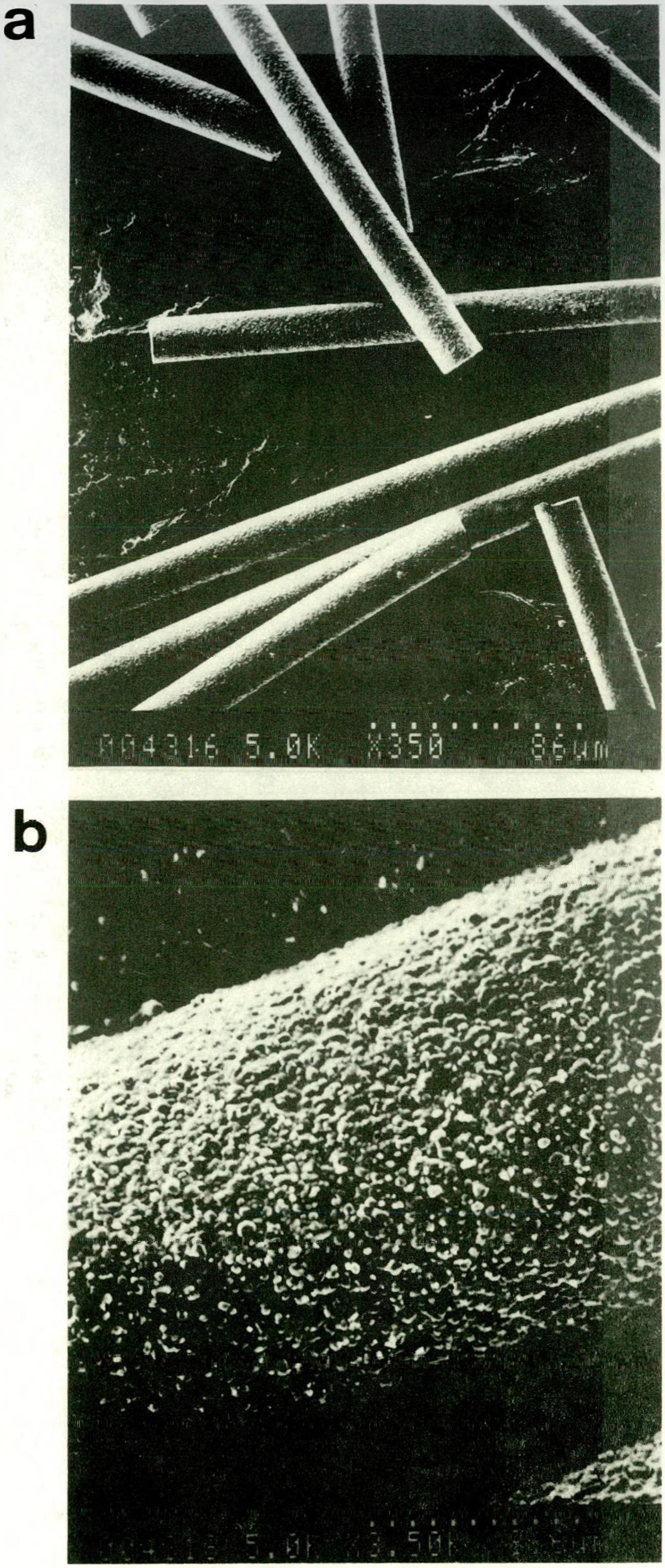
CONCLUSIONS

MONOLITHICS

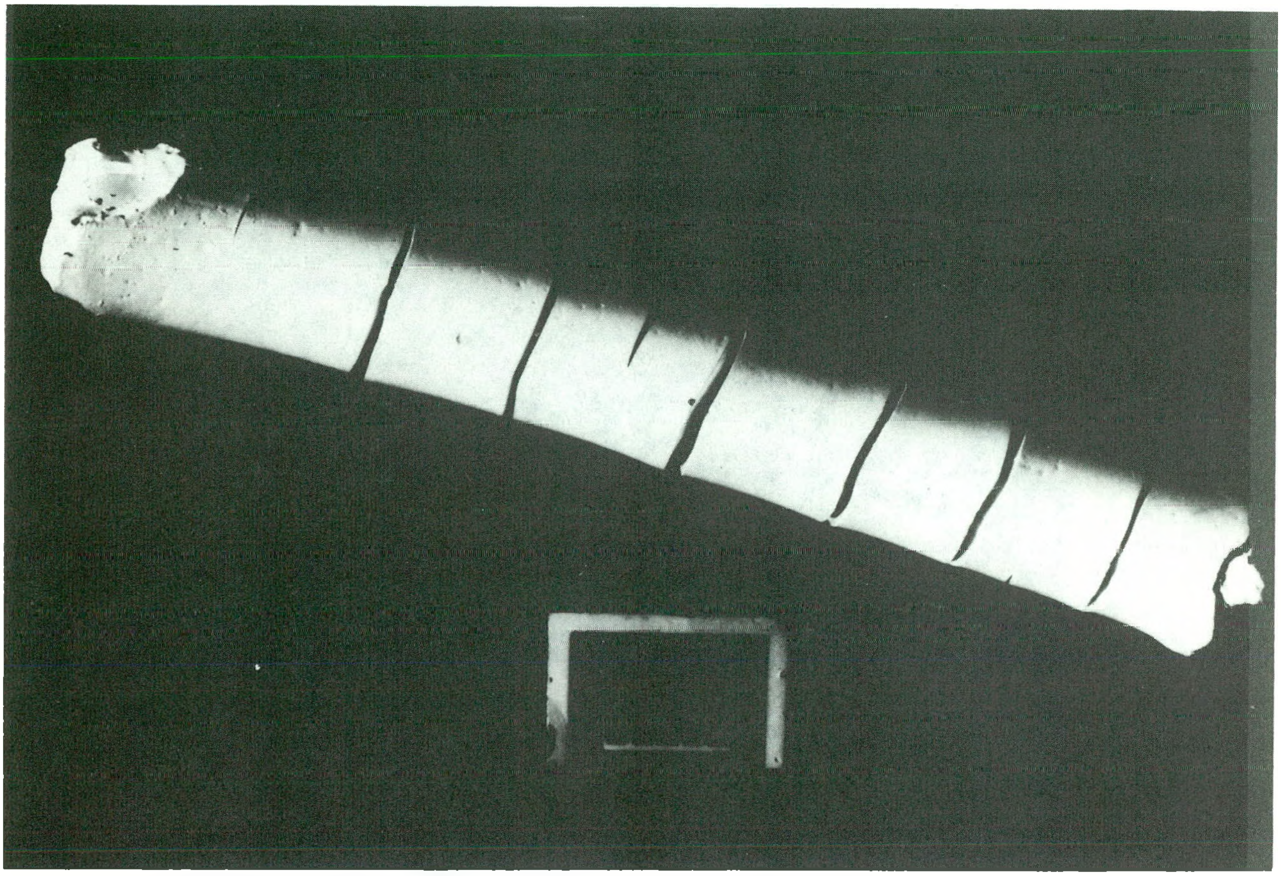
- Gelcasting is a promising near-net-complex-shape forming technique

COMPOSITES

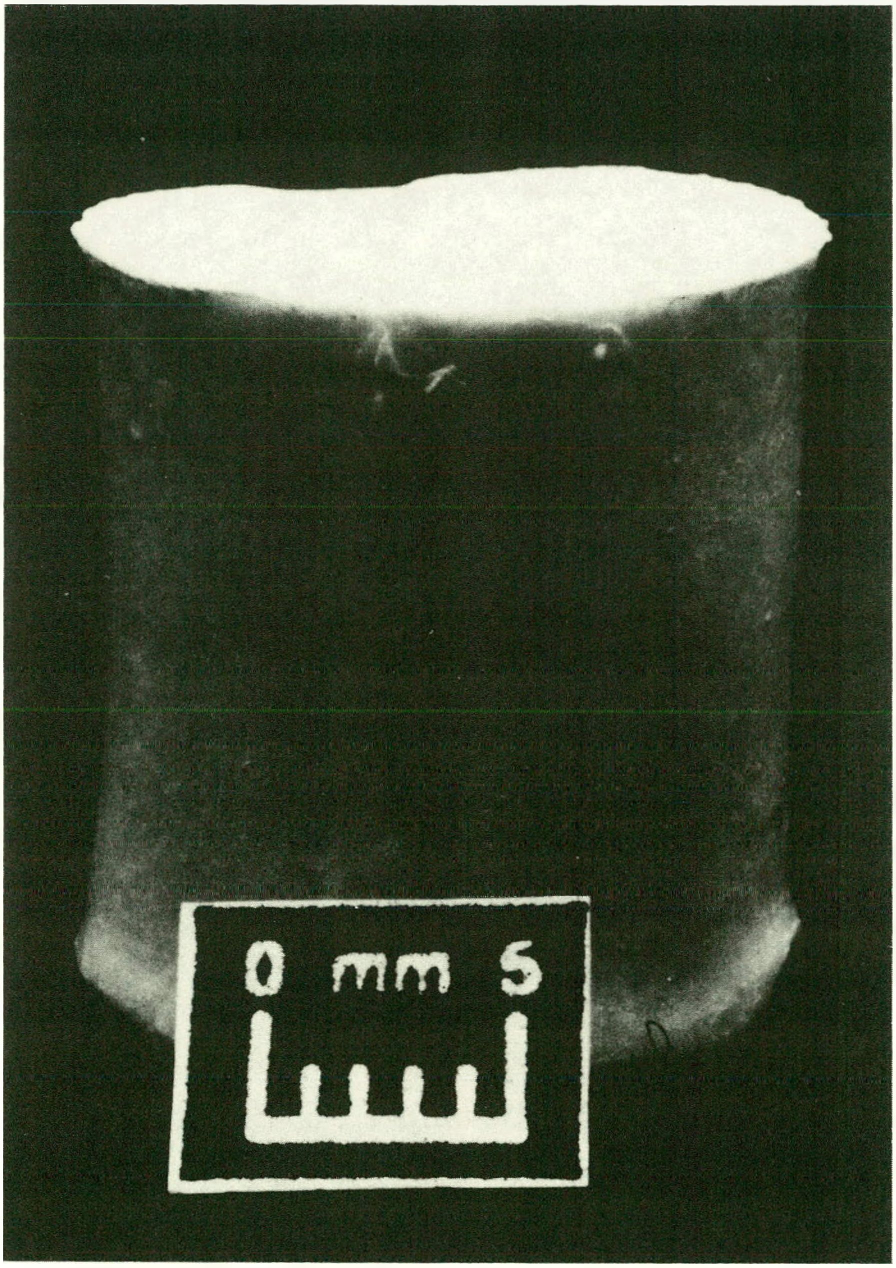
- Gelcasting can be used for production of fiber reinforced ceramic composites
- The uniformity of fiber distribution in ceramic body is critical to the quality of the composite
- Both FP and PRD 166 show thermal degradation in the form of grain growth and void formation at high temperatures
- A less reactive coating material is needed to preserve fiber integrity and prevent reaction between fiber and matrix



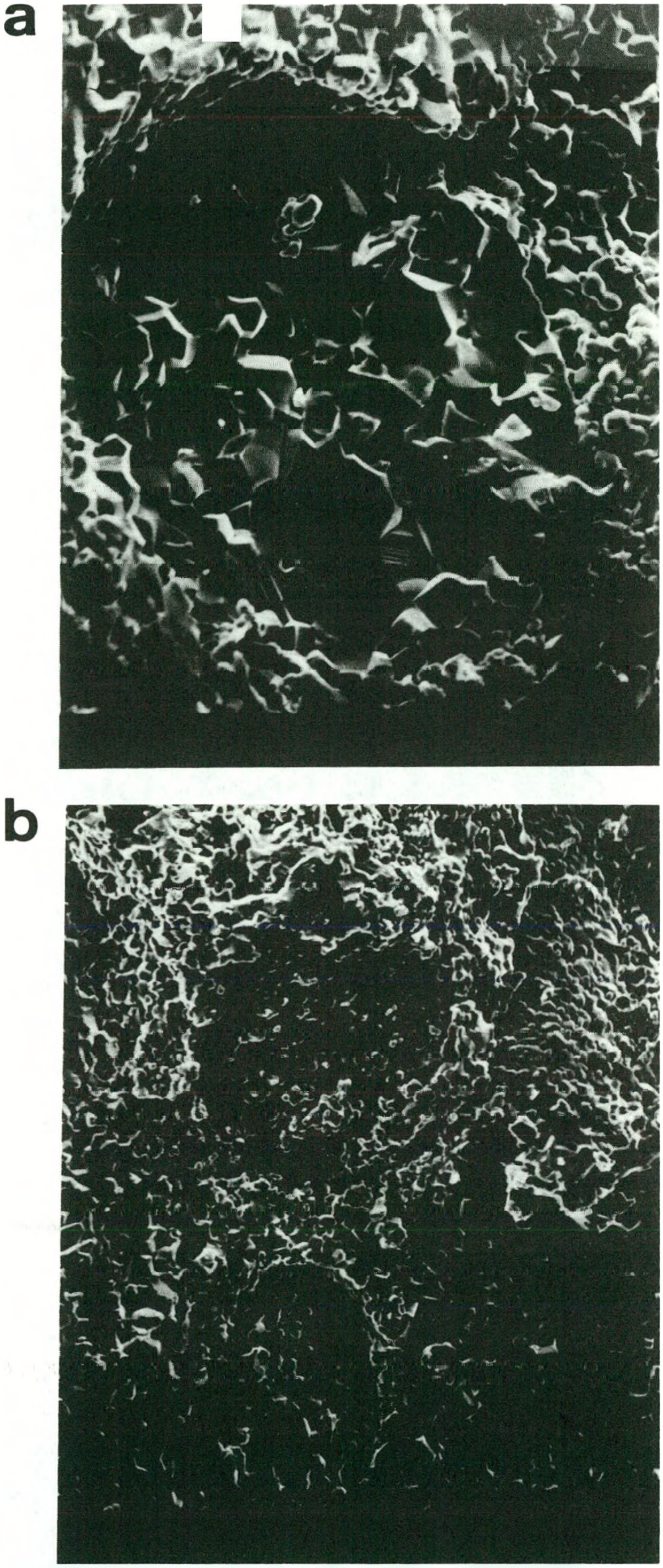
Scanning Electron Micrographs of FP Fibers. a) 350X; b) 3,500X.



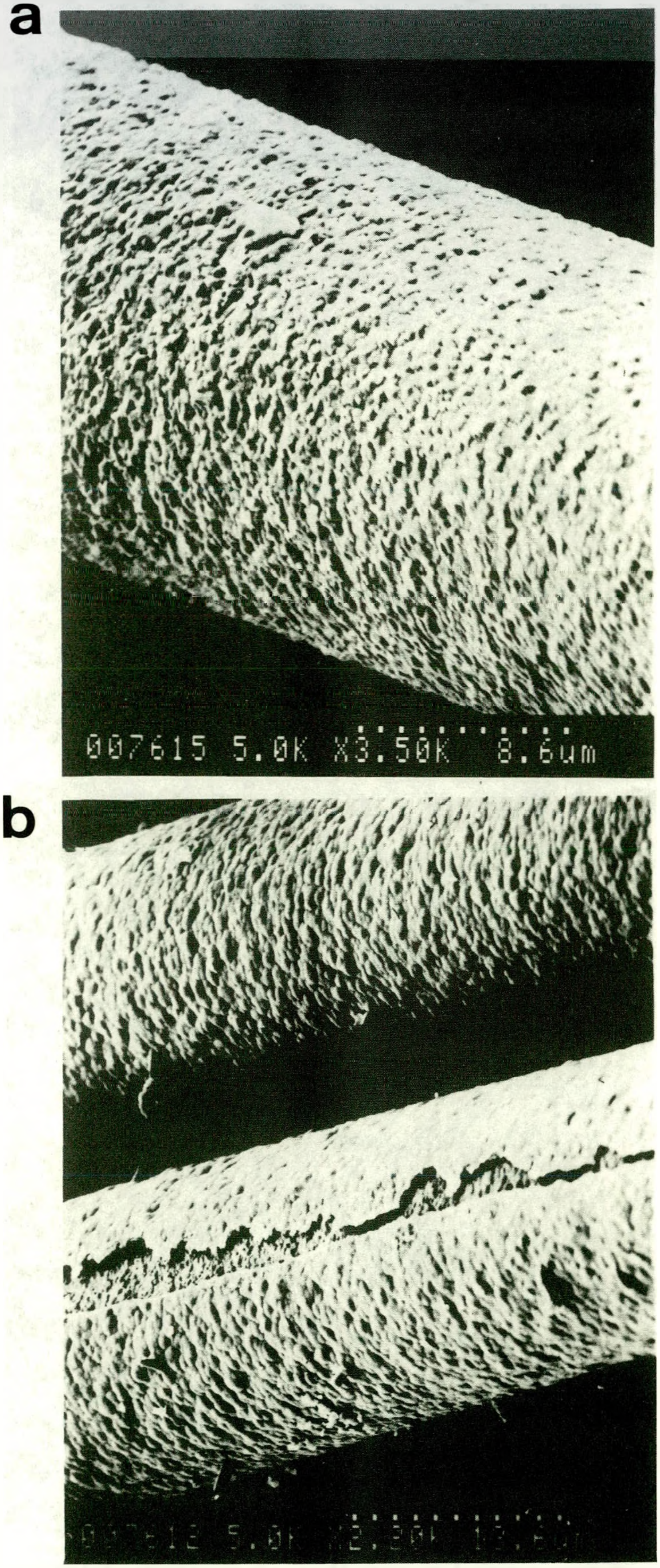
Continuous PRD 166 Fibers in TZ-3YS Zirconia, Fibers in Axial Direction. (Part 13AZAC, Sintered for 30h at 1300°C, in Air)



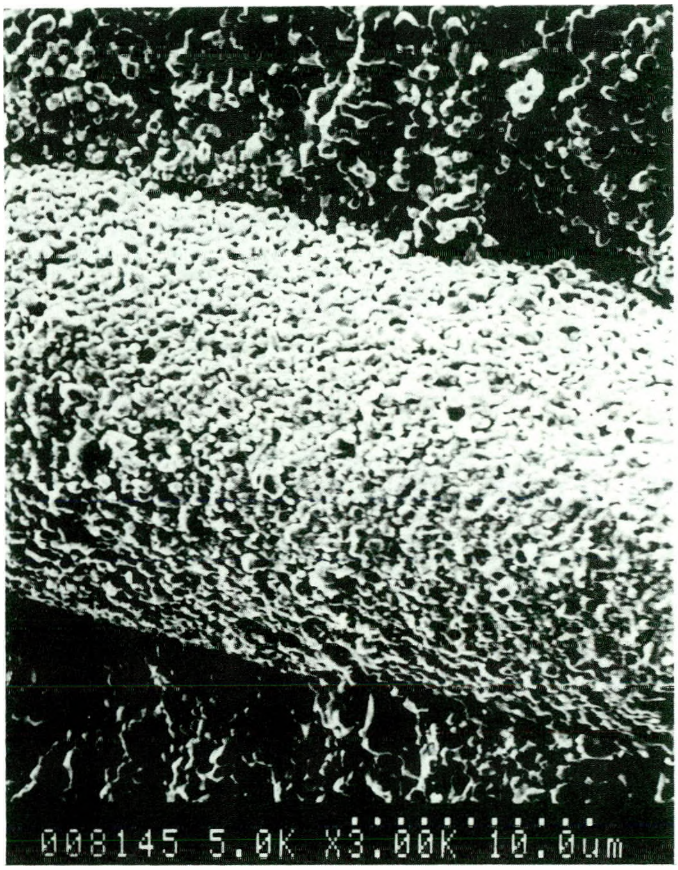
5 WT% FP Fibers in RCHP Alumina.
(Part 42AZAC, Sintered for 5h at 1400°C, in Air)



SEM of FP Fibers in RCHP Alumina.
a) 4,000X; b) 2,020X. (Part 32AZAC, Sintered for 0.5h at 1500°C, in Air)



SEM of Fibers with Carbon Coating.
a) FP Fiber, 3,500X; b) PRD 166 Fibers, 2,200X.



SEM of Carbon Coated PRD 166 Fiber in RCHP Alumina. 3,000X (Part 45AZAC, Sintered for 5h at 1400°C, in Air)

DISCUSSION

Gelcasting of Monolithic Ceramics and Chopped Fiber Reinforced Composites

Albert C. Young

Q: What is the quality of the water that you recommend for use in this gel casting process?

A: We usually use deionized water.

Q: How much shrinkage would you get from the cast state of the final sintered stage?

A: About 14 to 19 percent linear.

Q: You were showing an example where you have 5 percent of fiber in an aluminum matrix. What do you think is going to be the potential for increasing the volume fraction of the fiber in your composite with this technique? Many composites have 34, 40 percent fiber in them. Is this technique going to allow you to achieve that kind of fiber loading rate?

A: There should be no problem in principle to have 40 or 50 percent of fibers by volume in the slurries. The problem is the mixing - how do you get the fibers into the slurries? Either a better mixing method is needed or reduce the fiber aspect ratio. But, we think that because of low premix viscosities, and because the fibers are made of oxides, there will be no problem getting high fiber loading.

Q: What approximately is the time from mold filling to the time that you can remove a part from the mold that has sufficient handleability? You talked about the knit lines, which I assume is the timing problem. In other words, you are fighting a tradeoff here between this and gelation time. How does that translate into the size of parts you can make? What's the shelf life of the mixes that you deal with? And, in a somewhat unrelated question, what are the dimensional changes on gelation and on drying as opposed to firing?

A: The problem of how much time is needed from the casting to demolding depends on how much time it takes to gel the part. That can be determined by the temperature, and concentration of initiator and catalyst. For example, in thermal gelation you can heat a part using an oven and gelation would occur in two to three minutes after casting. And you can also make that ten minutes because the heating is determined by the operator. And you can control gelation time by controlling the concentration of initiator. If you only have initiator in the slurries, the slurry would not gel at room temperature before the heating takes place. So you control the timing of this casting to demolding. I would say 0 to 30 minutes is the range that you can control. We were able to completely eliminate the knit line problems using RCHP powders, which have much smaller particle size. When you cast the slurries, it takes some time for the solution to reach an equilibrium. With the smaller particle size we didn't have problems, although I'm not quite sure why. And also, using a thermal gelation, we were able to control the time before gelation; this eliminates knit line problem.

Q: How about the slurry shelf life?

A: You can have as long a shelf life as you want. All you need to do is add the initiator just before you make the part. We used premix that was made about 6 months ago and there was no problem.

Q: And the shrinkage on gelling?

A: About 1 percent of the difference before and after gelation. About 14 to 19 percent linear shrinkage from a wet green body to a fully densified part.

Q: This process seems to be very interesting and has potential. I'm familiar with thermal initiation and also other processes using UV initiation and doing the same kinds of things. Are you saying that when you start reducing the catalyst, then you need more initiator for a thermal initiation?

A: No. The cross linking process can be initiated two ways. One is to add a catalyst and the other way is to simply heat the part. In both cases, you have to have the initiator there. Actually these two process are the same in principle because catalysts just provide the additional activation energy for the reactions to take place.

Q: So what you're doing is taking the catalyst concentration down?

A: When you change the catalyst concentration, you are able to more or less control the gelation process. However, that's not as good as using thermal gelation which allows you to do whatever you need to do before heating the part.

Q: My question is related to distribution of temperature and threshold temperature for initiation. Specifically, the larger the part, the more difficult to have it uniform temperature profiled. And you may actually start generating gelation lines, net lines, although they are not formed because of the flow of slip but due to non-uniform gelation inside of your body. Have you seen anything like that?

A: We have made a one-inch part and we did not observe this problem As for larger parts, I don't know.

Q: Is the gel process exothermic?

A: Yes. I'd like to mention that you can obtain the information of this process through our technology transfer office in Oak Ridge National Laboratory.

SCALE UP OF A PROCESS FOR MANUFACTURE OF BETA SILCON CARBIDE POWDER

Peter T.B. Shaffer
Advanced Refractory Technologies

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
MATERIALS FOR ADVANCED HEAT EXCHANGER PROGRAM
TASK DESCRIPTION

TITLE: Scale-up of Process for Manufacture of Sinterable Beta Silicon Carbide Powder
WORK PROPOSAL NO: CEED009
CONTRACTOR: Advanced Refractory Technology Incorporated and Oak Ridge National Laboratory

PRINCIPAL INVESTIGATOR: Peter T. B. Shafer, 716/875-4091, R. A. Bradley, 615/574-6094, and M. A. Karnitz, 615/574-5150

OBJECTIVE: To develop a nonaqueous, dry purification process for silicon carbide powders synthesized via carbothermal reduction of silica.

TECHNICAL APPROACH: In this effort, Advanced Refractory Technology Incorporated (ART) will utilize its commercial production facility for the production of "crude" or unfinished silicon-carbide powder. In addition, ART will scale up the post furnace steps required to produce a truly finished sinterable silicon-carbide product.

SYSTEM DESCRIPTION: The scale up for the manufacturing of sinterable beta silicon carbide powder includes several process steps (1) blending (2) granulating/pelletizing (3) furnacing (4) dagglomertion (5) carbon removal (6) size reduction (7) silica removal. The latter four processing steps were carried out on a bench scale in a previous contract with Oak Ridge National Laboratory (ORNL), limiting the total production rate to less than 5kg per week. The proposed work is to scale up the level by more than an order of magnitude to 50kg per week.

CURRENT STATUS (9/89): Work by ART started in December 1988 and is scheduled for completion in January 1990. ART has completed the initial testing of all the processing steps.

ACCOMPLISHMENTS: ART has completed several short runs with the high temperature rotary kiln also the spray drier is fully operational. Several small tests were completed in August to check the operation of the unit.

CONCLUSIONS: ART is in the final phase of scaling up the processes for manufacturing silica beta silicon carbide powder. ART will deliver to Oak Ridge National Laboratory two 50kg lots of beta silicon carbide powder that meets the general acceptance of commercial specifications for sinterable powder. A final report will be prepared and delivered to ORNL, January 1990.

SCALE UP OF A PROCESS FOR THE MANUFACTURE OF BETA SILICON CARBIDE POWDER

DEPARTMENT OF ENERGY
OFFICE OF INDUSTRIAL PROGRAMS
CONTRACT P.O. 86X-SD119V

ADVANCED REFRACTORY TECHNOLOGIES, INC.

PRINCIPAL INVESTIGATOR: PETER T.B. SHAFFER, PhD
PROJECT ENGINEER: TOM MROZ

THE PROCESS

- REACTANTS: SILICA, CARBON
- BLEND, PELLETIZE
- FIRE: HIGH TEMPERATURE ROTARY FURNACE
  1750 - 1900 °C
  5-10 KG/HR PRODUCT
- CRUDE: BETA SiC
  FREE CARBON <10%
  SILICA < 2%
- DEAGGLOMERATE, SCREEN
- OXIDIZE: 600 - 650 °C, AIR
  (CARBON REMOVAL)
- ATTRITION MILL
- ACID WASH: HCl (REMOVE IRON FROM MILLING MEDIA)
  HF (REMOVE SILICA)
- PRODUCT

HISTORY

DEVELOPMENT OF A PROCESS FOR THE PRODUCTION OF INEXPENSIVE, REPRODUCIBLE, MICROCRYSTALLINE BETA SILICON CARBIDE POWDER(1).

PRODUCT:

S.A.:	9.2 m2/g
EQUIVALENT:	0.2 um
METAL IMPURITIES:	<63 ppm (2)

(1) DOE, CONTRACT 86X-00215-C
(2) ORNL, SPARK SOURCE MOSS SPEC

FINAL PRODUCT

(GOALS)

BETA SiC	>99%
FREE CARBON	<0.5
SILICA	<0.5
SURFACE AREA	10-20 m2/g
SIZE (EQUIVALENT)	0.1 - 0.2 um

DISCUSSION

Scale Up of a Process for Manufacture of Beta Silicon Carbide Powder

Peter T.B. Shaffer

Q: Are you adding any sintering aid to this powder or have you achieved these densities with the plain silicon carbide?

A: Yes, like all sintered SiC a sintering aid is required. We're using boron carbide graphite and resin additions. Interestingly enough we at ART have produced a boron carbide powder that has a very high boron/carbon ratio. It is a significantly better sintering aid than the standard arc furnace material. It has a ratio of about 4:1 (B/C). Whether it's just simply a function of higher boron loading, we don't know yet.

Q: Was there a cost goal along with the other goals?

A: We have estimated that the price of this material is probably going to be in the $10 to $15/pound range, when we get it into real production.

DEVELOPMENT OF A MANUFACTURERS CAPABILITY TO FABRICATE WHISKER-TOUGHENED CERAMIC TUBES

Michael A. Karnitz
ORNL

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
MATERIALS FOR ADVANCED HEAT EXCHANGER PROGRAM
TASK DESCRIPTION

TITLE: Development of a Manufacturers Capability to Fabricate Whisker-Toughened Alumina Tubes
WORK PROPOSAL NO: CEED009
CONTRACTOR: Oak Ridge National Laboratory and Subcontractor (to be indentified in FY 1990)

PRINCIPAL INVESTIGATORS: R. A. Bradley, 615/574-6094 and M. A. Karnitz, 615/574-5150

OBJECTIVE: To have a ceramic manufacturer develop manufacturing capability for fabricating large-diameter whisker-toughened alumina tubes.

TECHNICAL APPROACH: A subcontract will be placed with a ceramic manufacturer that will first identify and assess the potential market for the utilization of whisker-toughened alumina tubes for industrial applications such as heat exchanger and radiant tubes. After completion of the assessment, the manufacturer will develop a commercialization plan for selling the large-diameter tubes. The next step will be the development of a manufacturing plan for fabricating the large-diameter tubes. The final step is the development of the manufacturing capability for the production of the tubes. The ultimate goal is to have the manufacturer add the whisker-toughened alumina tubes to its product line.

SYSTEM DESCRIPTION: ORNL recently completed a study on the feasibility of extruding small-diameter (1 in diameter) whisker-toughened alumina tubes. These small-diameter extruded tubes had good strength and excellent thermal-shock properties. In addition, recent furnace exposure test have shown that the whisker-toughened alumina tubes are more corrosion-resistance in industrial environments than silicon-carbide. Based on these factors, the whisker-toughened alumina is an excellent candidate material for use as heat exchanger and radiant tubes.

CURRENT STATUES (9/89): A request for proposal will be issued in October to solicit a ceramic manufacturer to develop the capability to fabricate the whisker-toughened alumina tubes.

ACCOMPLISHMENTS: ORNL has completed an initial study on extruding small-diameter whisker-toughened alumina tubes. A statement of work has also been prepared for the scale-up of the manufacturer's capability.

CONCLUSIONS: It's been demonstrated that small-diameter whisker-toughened alumina tubes have good strength and excellent thermal-shock properties. The extruded whisker-toughened alumina composites represent a prime candidate material for use as heat exchangers and radiant tubes.

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1. T. N. Tiegs and P. F. Becher, "Sintered Al_2O_3-SiC Composites, "Bull. Am. Ceram. Soc. 66(2), 339-42 (1987).

2. M. J. Janney, E. S. Bomar, and M. C. Vance, Extrusion of Alumina-Silicon Carbide Whisker
Composites, ORNL TM 10373 (September 1989).

DEVELOPMENT OF MANUFACTURERS CAPABILITY TO FABRICATE WHISKER-TOUGHENED ALUMINA TUBES

M. A. KARNITZ
METALS AND CERAMICS DIVISION
OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

U.S. DEPARTMENT OF ENERGY
OFFICE OF INDUSTRIAL PROGRAMS
DIVISION OF WASTE ENERGY REDUCTION
WASTE ENERGY RECOVERY BRANCH

ADVANCED HEAT EXCHANGERS PROGRAM REVIEW
HERNDON, VIRGINIA
OCTOBER 11 AND 12, 1989

INTRODUCTION - PROJECT DESCRIPTION

We are attempting to develop a manufacturer's capability to fabricate large-diameter whisker-toughened alumina tubes

- **For utilization in industrial applications such as heat exchanger tubes**
- **Soliciting ceramic manufacturers (RFP process)**

INTRODUCTION - HISTORY AND JUSTIFICATION

For some heat exchanger systems, silicon carbide is the material of choice; however, in many applications, SiC is rapidly attacked by constituents of the waste heat stream

- **Alumina (Al_2O_3) is much more corrosion-resistant**
- **However, alumina has poor thermal-shock resistance**
- **Whisker-toughened alumina appears to be an excellent candidate material for heat exchanger tubes**

ORNL has demonstrated that SiC whisker-toughened alumina composites possess both high strength and significantly improved thermal-shock resistance compared to normal alumina

- **Small-diameters extruded product had good strength (approximately 300 MPa)**
- **Excellent thermal-shock properties ($\triangle$Tcrit > 900°C)**

INTRODUCTION - GOALS

The primary goal of this project is to develop the capability for fabricating large-diameter (up to 6 in. diameter) whisker-toughened tubes

- The ultimate goal is to have the manufacturer add the whisker-toughened tubes to its product line
- More than one market might be needed in order to sustain the production capabilities

PROJECT PARTICULARS - INTERESTING ASPECTS

The statement of work for the project has been completed and the RFP will be issued during the month of October

- One award is contemplated
- Receive responses in November
- Award in December
- Project length 9-12 months

PROJECT PARTICULARS

Outline of statement of work

- Task 1 Identify market for Al_2O_3-SiCw tubes
- Task 2 Development of a commercialization plan
- Task 3 Development of a manufacturing plan
- Task 4 Development of the manufacturing capabilities
- Task 5 Quality assurance requirements

CONCLUSIONS

The goal is to have a ceramic manufacturer develop the manufacturing capability for fabricating large-diameter whisker-toughened alumina tubes

- For heat exchanger applications (corrosion-resistant)
- Goal to have manufacturer add this to its product line
- Project completed in calendar year 1990

DISCUSSION

Development of a Manufacturers Capability to Fabricate Whisker-Toughened Ceramic Tubes

Michael A. Karnitz

Q: You mentioned the strength of this material as 300 mega Pascals. Is that flexure strength or tensile strength?

A: That was flexure strength.

Q: Do you have any thermal conductivity data on this material? And was there any corrosion testing done on this material, say in the sodium carbonate environment at ORNL?

A: There is an ORNL report, but the corrosion work in the report was not on this particular material.

BIOLOGICAL EFFECTS OF SILICON CARBIDE WHISKERS

Neil F. Johnson
Inhalation Toxicology Research Institute

U. S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGERS PROGRAM
PROJECT DESCRIPTION

TITLE: THE BIOLOGICAL EFFECTS OF SILICON CARBIDE WHISKERS

COOPERATIVE AGREEMENT NO: ED 01018 AL10

CONTRACTOR: INHALATION TOXICOLOGY RESEARCH INSTITUTE
PO BOX 5890, ALBUQUERQUE, NM 87185.

PRINCIPAL INVESTIGATOR: Dr. NEIL F. JOHNSON, 505-844-9719

OBJECTIVE: The purpose of this study is to determine the physical characteristics and the in vitro toxicity of silicon carbide whiskers. Silicon carbide (SiC) whiskers are fine crystal structures possessing a fibrous morphology with high tensile strength, which are manufactured for use in advanced composite materials employed in the automotive and aerospace industries. Because there are few data in the literature with which to assess the potential toxicity of these whiskers, a battery of in vitro assays are being used to help determine the necessity of additional in vivo assays. The in vitro assays will provide important information such as the durability of the whiskers in mock biological fluids, their cytotoxicity towards established cell lines, primary lung epithelial cells and macrophages, and their ability to transform cell lines and primary lung cells. The respirability of silicon carbide whiskers will be determined by sampling an atmosphere of the material. While the results of any one assay are insufficient to suggest a potential health hazard, the results of all the assays taken together provide a basis for making such a judgment.

APPROACH: Three samples of silicon carbide whiskers, are being compared to crocidolite (blue asbestos), erionite (biologically active mineral), glass microfiber (JM Code 100), and a continuous glass filament (PRD-166). The materials have been characterized in terms of their density, surface area, dimension (length and diameter), chemistry (energy dispersive X-ray microprobe analysis) and crystallinity (electron beam diffraction). The cytotoxicity of these materials are being determined by examining their ability to reduce the number of cells in culture that are able to survive and divide to form colonies. The ability of these materials to induce genetic damage is being determined by assessing their ability to form micronuclei and to transform cells. The materials are being implanted into the pleural cavity of rats to determine their ability to induce mesothelioma. The respirability of the silicon carbide whiskers will be determined by sampling an atmosphere of the material.

CURRENT STATUS: The physical characterizations of the materials are almost complete; the final size distributions for erionite, crocidolite and JM code 100 are outstanding. The cytotoxicity assays for the samples are complete. The micronuleus assay is completed but not scored (one months work); the transformation assay is planned for late November 1989 and will take two months to complete. The animal implantation studies are being designed and will be underway within the next three months and will take approximately a year to eighteen months to complete. The respirability determinations and *in vitro* dissolution studies will start in November 1989 and take six months to complete.

ACCOMPLISHMENTS: The results of the surface area and density determinations are shown in table 1. The mean lengths and diameters and number of fibrous particles per mg are shown in table 2. The morphology of the silicon carbide whiskers was varied, with small numbers of particles possessing a beaded appearance or a "V" shape.

TABLE 1: Table showing the density and surface area of the various samples used in this project. The density was measured by air pycnometry and the surface area by nitrogen adsorption and desorption.

SAMPLE	DENSITY H2O = 1.00	SURFACE AREA m2/g
SiC # 1	3.37 + 0.18	3.0 + 0.2
SiC # 2	3.24 + 0.18	3.6 + 0.2
SiC # 3	4.29 + 0.02	1.4 + 0.1
CROCIDOLITE	3.19 + 0.13	7.0 + 0.3
JM CODE 100	2.54 + 0.32	2.5 + 0.2
ERIONITE	2.28 + 0.09	356
PRD-166 POWDER	4.21 + 0.17	1.5 + 0.1
PRD-166 FIBER	4.43 + 0.15	0.3 + 0.01

TABLE 2: Table showing the mean length and diameter and the number of whiskers (aspect ratio > 3:1 = fibers) per mg of sample.

SAMPLE	MEAN LENGTH	MEAN DIAMETER	WHISKERS/ mg
SiC # 1	4.54	0.42	0.76 X 107
SiC # 2	6.55	0.32	1.05 X 107
SiC # 3	20.12	0.75	1.61 X 105
CROCIDOLITE	3.10	0.19	3.60 X 109
JM CODE 100	4.70	0.40	2.30 X 109
ERIONITE	4.30	0.35	3.85 X 107

The cytotoxicity assays showed on a mass basis that SiC # 2 was more toxic than crocidolite, if the tests were based on whisker/fiber number then all three of the SiC samples were more toxic than crocidolite. The continuous glass filament sample; PRD 166 was less toxic than crocidolite. Preliminary data from the micronucleus assay shows only marginal genotoxic activity for the silicon carbide whiskers compared to crocidolite.

CONCLUSIONS: While extreme caution needs to be exercised over the interpretation of the results from the <u>in vitro</u> assays, the level of biological activity seen suggests that further animal assays; particularly an inhalation experiment should be undertaken. The physical dimensions of the SiC samples are similar to that of asbestos and may be expected to have similar airborne characteristics and therefore SiC whiskers should be handled with the same precautions as with asbestos.

ASSESSMENT OF CERAMIC WHISKER HEALTH HAZARDS

Study Director: **Dr. Neil F. Johnson**

INHALATION TOXICOLOGY RESEARCH INSTITUTE

P.O. Box 5890, Albuquerque, New Mexico, U.S.A. 87185 ITRI

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- OVERVIEW FIBER RELATED DISEASE
- HEALTH ASSESSMENT OF SILICON CARBIDE WHISKERS
    - Current knowledge
    - Present study
- SUMMARY AND CONCLUSIONS

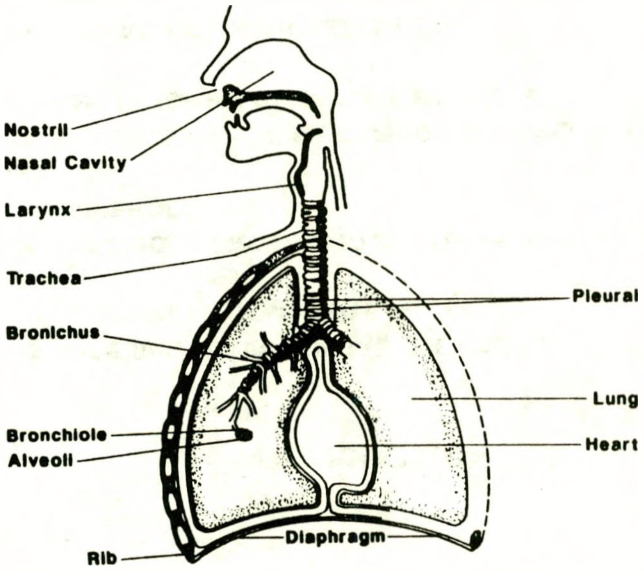
INTRODUCTION

- WIDE SPREAD POTENTIAL USE FOR SIC REINFORCED COMPOSITE MATERALS
- SIC SINGLE CRYSTAL STRUCTURES WITH FIBROUS MORPHOLOGY
- POSSIBLE EXPOSURE DURING SIC MANUFACTURE AND FINISHING COMPOSITE PRODUCT
- SIMILARITIES WITH ASBESTOS

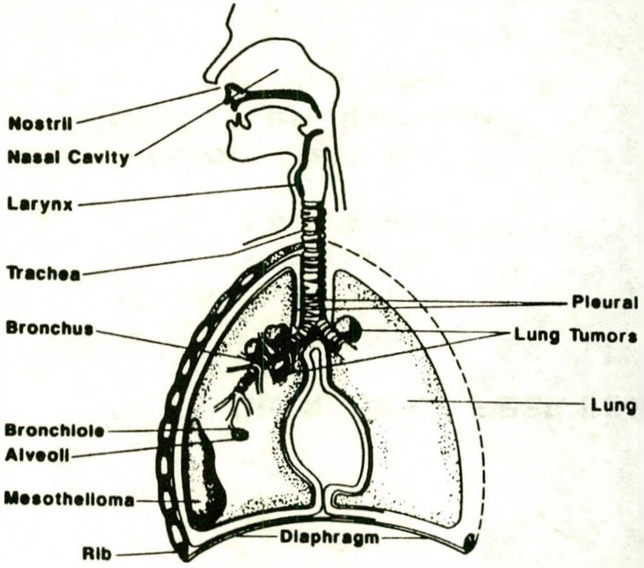
CONSEQUENCES OF FIBER INHALATION

- TUMOR INDUCTION
    - Lung tumors
    - Mesothelioma
- FIBROTIC REACTION
    - Intestitial fibrosis
    - Pleural fibrosis

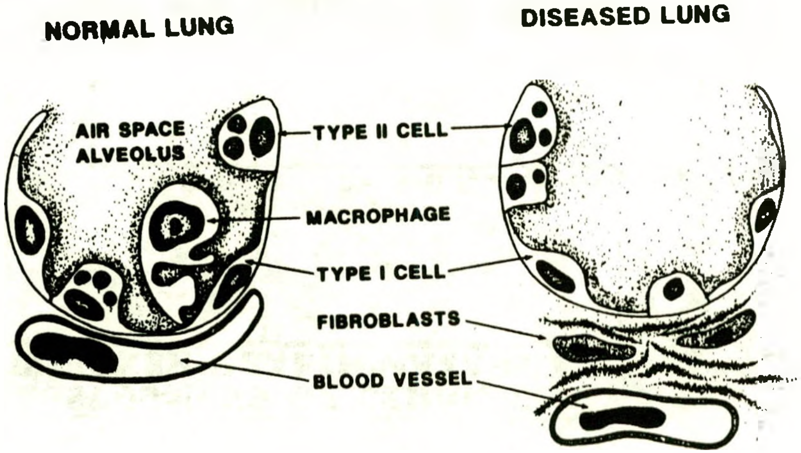
AMATOMY OF HUMAN RESPIRATORY TRACT



ANATOMY OF HUMAN LUNG CANCER AND MESOTHELIOMA



ANATOMY OF IMPAIRED LUNG FUNCTION


NORMAL LUNG

DISEASED LUNG

FIBER RELATED HUMAN DISEASE

- **LUNG CANCER**
  - **Dose dependent**
  - **Long latency**
  - **Fiber size relationship ?**
  - **Smoking history**
- **LUNG FIBROSIS**
  - **Dose dependent**
  - **Long latency**
- **MESOTHELIOMA**
  - **No dose dependency**
  - **No smoking relationship**
  - **Fiber size relationship**

TEST SAMPLES

- UICC CROCIDOLITE
- ERIONITE
- JM CODE 100 GLASS MICROFIBER
- PRD-166
- SAMPLE 1 SILICON CARBIDE WHISKERS
- SAMPLE 2 SILICON CARBIDE WHISKERS
- SAMPLE 3 SILICON CARBIDE WHISKERS

ASSESSMENT OF HUMAN HEALTH RISK


Epidemiology

HUMAN HEALTH RISK

Cellular Studies

Physico-Chemical Studies

Animal Studies

TIERED APPROACH TO ASSAYING BIOLOGICAL ACTIVITY OF SINGLE CRYSTAL STRUCTURES

TIER 1
Physico/Chemical Studies

TIER 2
in vitro Studies

TIER 3
Animal Studies

INSTILLATION / INOCULATION

INHALATION

- SIZE
- MORPHOLOGY
- CHEMISTRY
- DURABILITY

- CYTOTOXICITY
- FIBROGENICITY
- GENOTOXICITY

- MESOTHELIOMA
- FIBROSIS
- LUNG CANCER
- DURABILITY
- LUNG BURDEN

INCREASING COMPLEXITY COST →

INCREASING RELEVANCY TO HUMAN EXPOSURES →

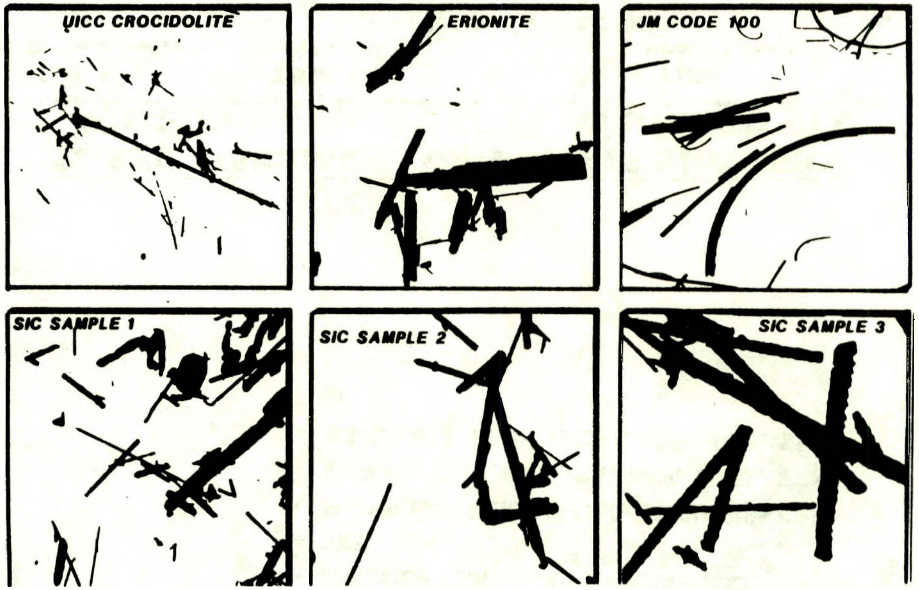
EXPERIMENTAL DESIGN

1. PHYSICO-CHEMICAL CHARACTERIZATION:
   - MORPHOLOGY
   - SIZE DISTRIBUTION
   - SURFACE AREA
   - DENSITY
   - CHEMISTRY
   - CRYSTALLINITY

FIBER MORPHOLOGY

TRANSMISSION ELECTRON MICROSCOPY

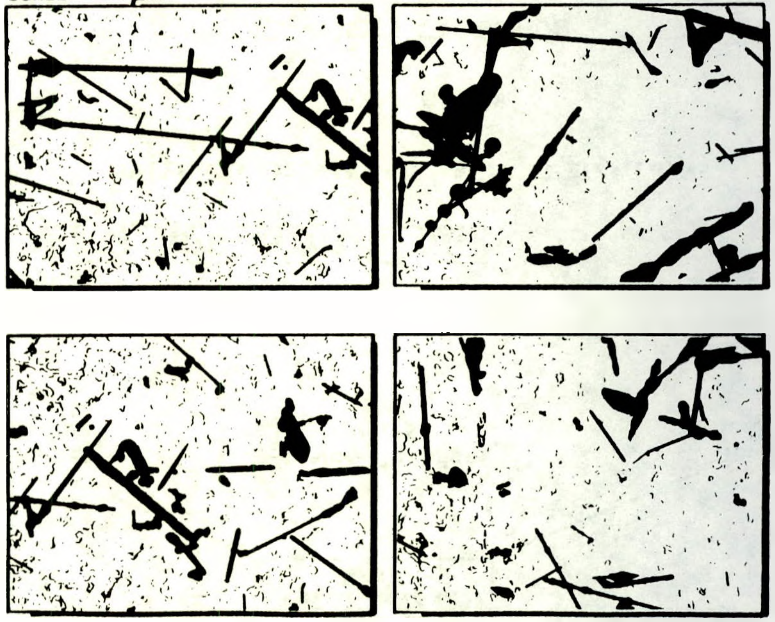
SCALE — 2µm



FIBER MORPHOLOGY

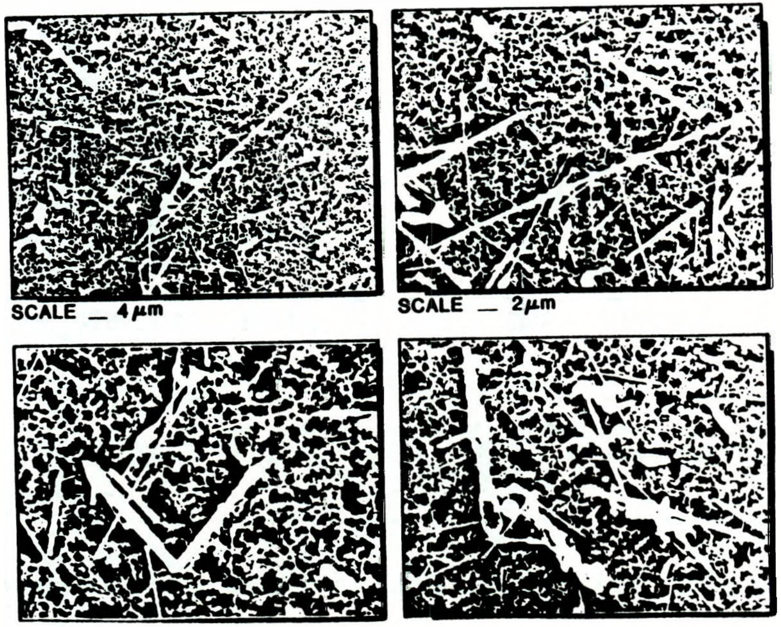
TRANSMISSION ELECTRON MICROSCOPY
SiC SAMPLE 3

SCALE — 4µm



FIBER MORPHOLOGY

SCANNING ELECTRON MICROSCOPY
SiC SAMPLE 3



LENGTH/DIAMETER

SiC SAMPLE 1 — **DIAMETER µm**

LENGTH µm	0-0.09	0.1-0.19	0.2-0.29	0.3-0.39	0.4-0.59	0.6-0.79	0.8-0.99	>1.0	TOTAL %
0-1.9	4.1	10.1	6.3	3.8	0.9				25.2
2.0-3.9		5.7	10.4	4.7	6.0	3.8	1.6		32.2
4.0-5.9		0.9	4.4	3.5	4.4	2.2	1.6	0.9	17.9
6.0-7.9			1.6	1.9	1.3	1.6	0.6	0.9	7.9
8.0-9.9			1.6	1.3	1.6	1.3	0.9	1.3	8.0
10.0-14.9			1.9	0.9	0.3	0.9	0.6	0.9	5.5
>15.0		0.3		0.6	0.3	0.3	0.9	0.6	3.0
TOTAL %	4.1	17.0	26.2	16.7	14.8	10.1	6.2	4.6	

n = 318

MEAN LENGTH 4.54 ± 0.23 µm (SEM) RANGE 0.53-25.87 µm
MEAN DIAMETER 0.42 ± 0.02 µm (SEM) RANGE 0.04-2.27 µm

LENGTH/DIAMETER

SiC SAMPLE 2 — **DIAMETER µm**

LENGTH µm	0-0.09	0.1-0.19	0.2-0.29	0.3-0.39	0.4-0.59	0.6-0.79	0.8-0.99	>1.0	TOTAL %
0-1.9	5.69	8.68	2.99	1.80	2.10				21.26
2.0-3.9	1.80	9.28	4.19	8.38	5.69	0.90			30.24
4.0-5.9	1.80	1.20	1.80	3.59	2.62	0.90	0.30		12.21
6.0-7.9		1.20	2.10	1.80	1.80	1.20	0.30	0.30	8.70
8.0-9.9		0.60	2.10	1.80	2.99	1.20			8.69
10.0-14.9		1.20	3.29	1.80	2.99	0.60		0.30	10.18
>15.0		1.20	0.60	2.40	3.30	1.20			8.70
TOTAL %	9.29	23.36	17.07	21.57	21.49	6.00	0.60	0.60	

n = 334

MEAN LENGTH 6.55 ± 0.40 µm (SEM) RANGE 0.8 - 55.47 µm
MEAN DIAMETER 0.32 ± 0.01 µm (SEM) RANGE 0.07 - 3.33 µm

LENGTH/DIAMETER

SiC SAMPLE 3 — **DIAMETER μm**

LENGTH μm	0-0.09	0.1-0.19	0.2-0.29	0.3-0.39	0.4-0.59	0.6-0.79	0.8-0.99	>1.0	TOTAL %
0-1.9									
2.0-3.9					0.3	2.5	0.8	0.3	
4.0-5.9		0.3	0.6		1.1	1.7	2.2	1.7	7.6
6.0-7.9		0.3	1.7		1.1	1.4	1.4	2.2	8.1
8.0-9.9		0.3	1.4	1.4	0.6	1.1	1.7	3.0	9.5
10.0-14.9		0.8	1.9	5.0	3.3	3.9	4.1	5.2	24.2
>15.0			2.5	8.8	12.1	7.5	3.0	13.0	46.9
TOTAL %		1.7	8.1	15.2	18.5	18.1	13.2	25.4	

n = 362

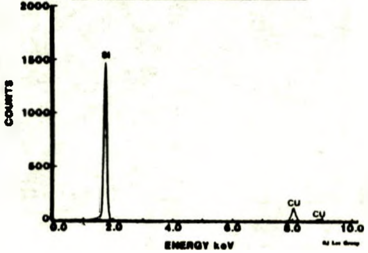
MEAN LENGTH 20.12 ± 1.01 μm (SEM) RANGE 2.35-122.95 μm
MEAN DIAMETER 0.75 ± 0.02 μm (SEM) RANGE 0.11-2.36 μm

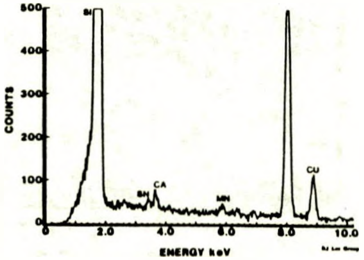
PHYSICAL DIMENSIONS

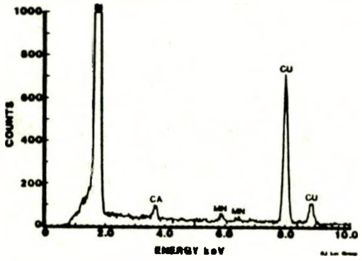
SAMPLE	MEAN LENGTH	MEAN DIAMETER	FIBERS/μg
SiC #1	4.54	0.42	0.76×10^{7}
SiC #2	6.55	0.32	1.05×10^{7}
SiC #3	20.12	0.75	1.61×10^{5}
CROCIDOLITE	3.10	0.19	3.60×10^{9}
JM CODE 100	4.70	0.40	2.30×10^{9}
ERIONITE	4.30	0.35	3.85×10^{7}

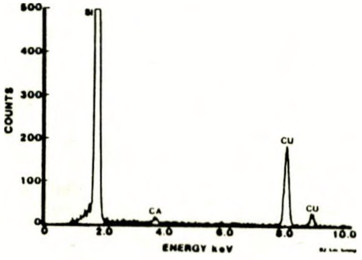
ENERGY DISPERSIVE X RAY ANALYSIS

SIC SAMPLE 2


REPRESENTATIVE OF MAJORITY OF WHISKERS

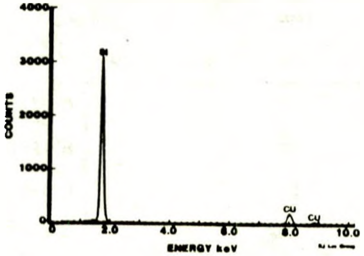

THICK, SMOOTH WHISKER

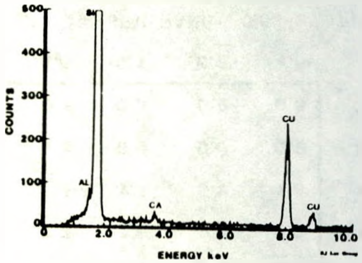

SERRATED WHISKER

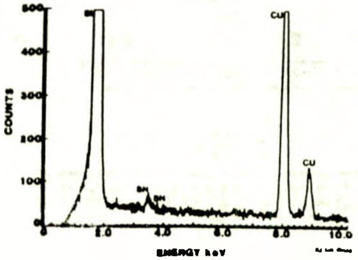

SERRATED WHISKER

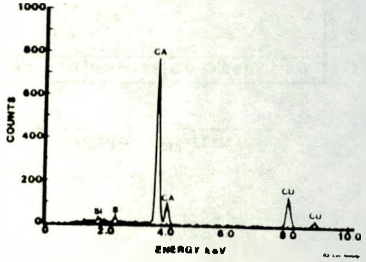
ENERGY DISPERSIVE X RAY ANALYSIS

SIC SAMPLE 3


REPRESENTATIVE OF MAJORITY OF WHISKERS & PARTICLES


FUSED NON-FIBROUS AGGLOMERATE


SILICON CARBIDE WHISKER


NON-FIBROUS PARTICLE

DENSITY AND SURFACE AREA

SAMPLE	DENSITY (H_2O + 1.00)	SURFACE AREA (m^2/g)
SIC #1	3.37 ± 0.18	3.0 ± 0.2
SIC #2	3.24 ± 0.18	3.6 ± 0.2
SIC #3	4.29 ± 0.02	1.4 ± 0.1
CROCIDOLITE	3.19 ± 0.13	7.0 ± 0.3
JM CODE 100	2.54 ± 0.32	2.5 ± 0.2
ERIONITE	2.28 ± 0.09	356
PRD-166 POWDER	4.21 ± 0.17	1.5 ± 0.1
PRD-166 FIBER	4.34 ± 0.15	0.32 ± 0.002

EXPERIMENTAL DESIGN

2. *In vitro* SOLUBILITY :

- **RELEASE OF SILICON**
- **MORPHOLOGY**
- **SIZE DISTRIBUTION**
- **SURFACE AREA**

EXPERIMENT DESIGN

3. *In vitro* TOXICITY

- **CYTOTOXICITY**
  - **Alveolar Macrophages**
  - **Fibroblasts**
  - **Tracheal Cells**
  - **Lung Epithelial Cell (LEC) Line**
  - **A549 Cell Line**
  - **V79-4 Cell Line**
- **FIBROGENICITY**
  - **Fibroblast Activation**
- **GENOTOXICITY**
  - **Micronuclei Formation (LEC)**
  - **Transformation :$C_3H10T_{1/2}$ Cell Line**
    :Tracheal cells

RELATIVE CELL SURVIVAL - 50 µg/mL

SAMPLE	CELL ASSAY LEC	A549	RTE	RAM
SIC #1	0.116	0.512	0.573	0.460
SIC #2	0.011	0.140	0.080	0.195
SIC #3	0.130	0.348	0.404	0.621
CROCIDOLITE	0.033	0.300	0.241	0.526
JM CODE 100	0.046	0.043	0.064	0.388
ERIONITE	0.187	0.548	0.398	0.734
PRD-166	0.548	0.760	0.961	0.767
UNEXPOSED CONTROL	1.000	1.000	1.000	1.000

RELATIVE CELL SURVIVAL - 50 µg/mL

SAMPLE	CELL ASSAY LEC	 A549	 RTE	 RAM	RANKING
SIC #1	4	5	6	3	4
SIC #2	1	2	2	1	1
SIC #3	5	4	5	5	5
CROCIDOLITE	2	3	3	4	3
JM CODE 100	3	1	1	2	2
ERIONITE	6	6	4	6	6
PRD-166	7	7	7	7	7

FIBER NUMBER - 50 µg/mL

SAMPLE	TOTAL NUMBER	FIBERS < 0.3 µM DIAMETER > 8.0 µM LENGTH PERCENTAGE	 FIBER NUMBER
SiC #1	380,000	3.8	14,440
SiC #2	525,000	9.0	47,250
SiC #3	80,500	5.9	4,750
CROCIDOLITE	180,000,000	7.4	13,332,000
JM CODE 100	115,000,000	6.7	7,7000,000
ERIONITE	190,000	14.6	27,740

CYTOTOXICITY RANKINGS - 50 µg/mL

SAMPLE	MASS CONCENTRATION	FIBER NUMBER	FIBER DIA < 0.3 µM LENGTH > 8.0 µM
SiC #1	4	3	2
SiC #2	1	2	4
SiC #3	5	1	1
CROCIDOLITE	3	5	6
JM CODE 100	2	6	5
ERIONITE	6	4	3
PRD-166	7	7	7

EXPERIMENTAL DESIGN

4. *In vivo* INTRAPLEURAL INOCULATION

- **MESOTHELIOMA INDUCTION**

SIC CONCERNS

- WHISKER DIMENSIONS
- HIGH BIOLOGICAL ACTIVITY WITH *in vitro* ASSAYS
- SIMILARITIES WITH ASBESTOS

FUTURE DIRECTIONS

- FUNDED
    - Respirability
    - Implantation
    - *In vitro* Solubility
    - Lung Retention and Clearance
- NON-FUNDED
    - Inhalation exposure

DISCUSSION

Biological Effects of Silicon Carbide Whiskers
Neil F. Johnson

Q: When you're doing all these studies, what protection do you and your co-workers at the Inhalation Research Institute use? The materials that you are using are potentially carcinogenic.

A: We are a very safety-conscious lab. We use respirators, wear double gloves, and wear white coats. The dry material is weighed in a fume hood with a full respirator. We take every available precaution about potential exposure. We are very concerned because the material is biologically active. Once it's in a liquid medium, then it really should be safe. We're less concerned about the wet material than dry. We're still concerned if we spill it, because it'll then dry out to potential powder exposure again. We take every precaution in handling material and storage -- they're stored in double bags in locked cabinets with carcinogen notices on them.

Q: You spoke at the beginning of your talk about the fact that you saw some pictures from one of the other presentations, here, where some of the fibers became available on the surface, probably as a result of corrosion. I thought up to now we considered that once the equipment was made, the fibers wouldn't be available to the general atmosphere.

A: That was my impression until I saw that scanning picture. That's why it wasn't included among the list of potential exposures on the introduction slide to my talk. It looked to me like it's a real possibility if there is sufficient corrosion. The other thing that potentially worried me was cristobalite, and that is one of the most active forms of silica. So a fibrous cristobolite has potent biological implications. It is not only fibergenic, but if it's a fiber, then it's potentially carcinogenic as well.

Q: That's the first indication I think we've heard that this material in use might be a problem. Up to now we have only been concerned that in the manufacture it was a problem.

A: I don't know how representative those corrosion pictures were. I don't want to cause problems, but it did make me suspicious that they were identifiable fibers on the surface of a corroded component. Previously, when I was thinking about potential exposure, I was thinking of catastrophic failure. However, corroding gracefully could be more dangerous than a catastrophic failure in terms of potential for releasing fibers.

A separate issue is ceramic or polycrystalline ceramic fibers if they are used as insulation material. At the interface between the heat source and the insulation material, those fibers can have a large amount of cristobolite on the surface. So we know that when polycrystalline ceramic fibers are used, they can have a cristobolite surface. This aspect is being investigated by a toxicology group in Geneva.

Q: Is one fiber as bad as a million?

A: Potentially yes.

Q: What mechanism could cause one fiber to be so dangerous?

A: I don't know. One of our real problems is that the only factor that seems to determine biological activity is shape. I'm not sure how shape might exert it's biological activity unless it actively impairs the ability of the cell to replicate. The cells divide into two; then their chromosomes divide and go into two daughter cells. If you inhibit or impair that ability, you can end up with an odd genetic mix in one part and a different mix in another. Whether it's just purely mechanical interference with the genetic process, no one knows. If you can come up with a plausible theory, we'd love to know because we've been looking at it now for 20 years, and we're still none the wiser. All we know is that it's fiber-size dependent.

Q: I'd like to know what other data exists, and particularly what other materials you might be including in your study. For example, what are other comments surrounding materials such as alumina? I'd also be very interested in whether you're going to look at any silicon nitride since, there are some interest in whiskers there. Typically how we get tougher silicon nitride is to get elongated grains. If any of those are released into the environment they might possibly act like whiskers. Do you expect to look at any of those materials?

A: Yes. We expect eventually that some inhalation toxicology groups will do this. We would very much like to look at the inhalation characteristics of silicon carbide, silicon nitride, and fibrous ceramics. In general, there is little known about their biological activity. Obviously they're going to be increasingly used in industry and as a starting material, and I'd be very concerned to have their toxicity evaluated.

Q: It would be interesting to find out what the effect of coating those fibers does to the toxicity of the silicon carbide. Have you got any thoughts on that?

A: It depends what the surface is.

Q: How about graphite-coated silicon carbide?

A: I don't think it would make much difference. I think if it's fiber-size dependent, altering a surface chemistry probably won't have much effect. I think in terms of cancer production, it won't make a lot of difference.

Q: The question on coatings is really relative to my question too. Have you looked at the pretreatment processes that are used to handle these whiskers? Most workers in the field do not use them as is, but they either clean them with HF or they do other things. Have any of those protocols in effect been given to you to look at the way they vary?

A: The four samples that I received were sent as-produced -- filled, sealed, and packaged. As far as I know they are the final product and not the pre-final products. Obviously it's important to look at the whole process and I think we need some further testing.

Q: A lot of study and work has been done over the past 20 years or so on materials like asbestos and fiberglass. This work was done by the mineral makers and the ceramic fiber people, and now you're setting out to do this. How does all that body of knowledge get transferred or communicated? Do you share information so that the subsequent studies can learn or not learn from what was done before?

A: All the major inhalation studies are in the open literature. All of our information will be in the open literature. Our work will be published very soon. Some companies I know are pursuing their own research programs, and whether they will publish their results in the open literature I don't know. Companies like DuPont have been very good at publishing their inhalation data. The majority of companies seem to be very responsible; once they've done a study, they publish it. We do share information; we actively collaborate with a lot of labs. But again, the end product is really getting the regulatory authorities to use it and define a permissible exposure level. To do that, you really have to perform an animal experiment to assure the regulatory authorities that it is a problem or isn't a problem.

Comment: At one of the early ASTM committee meetings on the MSDS (material safety data sheet) for inorganic fibers I believe, the people from GTE made an interesting statement. They had examined residue, from their grinders where they were grinding silicon nitride tools which were reinforced with silicon carbide whiskers. They examined many, many thousands of samples and they found fibers. Now the interesting thing was, all of the fibers they found were silicon nitride which were picked off the matrix. They found no silicon carbide whisker fibers that survived. The silicon carbide whisker fibers came off with about a one to one, one to two, L to D ratio.

A: That goes back to the problem of producing the matrix components.

Comment: What this says is that any time you're working with silicon nitride and doing any grinding, you better be careful.

A: I think there is also some evidence that grinding carborundum with silicon carbide particulates also releases fibrous materials. I think there are about three reports in the literature now showing samples recovered from lung tissue of exposed workers containing a small amount of fibrous material. So lifetime exposure to carborundum can also include minimal exposure to fibrous silicon carbide.

ENHANCED SHELL-AND-TUBE HEAT EXCHANGERS FOR INDUSTRY

Arthur E. Bergles
Rensselaer Polytechnic Institute

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGERS PROGRAM
PROJECT DESCRIPTION

TITLE: ENHANCED SHELL–AND–TUBE HEAT EXCHANGERS FOR INDUSTRY
COOPERATIVE AGREEMENT NO: DE–FC07–88ID12772 M002
CONTRACTOR: Rensselaer Polytechnic Institute
Troy, NY 12180–3590

CO–PRINCIPAL INVESTIGATORS: Arthur E. Bergles, 518–276–6203
Michael K. Jensen, 518–276–2843
Euan F.C. Somerscales, 518–276–6522

OBJECTIVE: The objective of this research is to develop enhanced heat transfer techniques for application in energy–intensive industrial processes. The enhancement techniques to be studied are structured surfaces for use in shellside boiling in tube bundles. Performance under normal and forced conditions is of interest.

TECHNICAL APPROACH: Boiling on the shellside of tube bundles will be investigated through the accomplishment of three main tasks: 1) Design, construct and test models of multitube bundles in crossflow with three surface structures (smooth and two enhanced surfaces) and two tube layouts. The specific enhanced tubes (Linde High Flux and Wolverine Turbo–B) will be chosen after pool boiling scoping tests have been performed.[1] 2) The fouling tendencies of these three surfaces will be investigated in a pool boiling apparatus. 3) The effect of multicomponent mixtures on boiling in enhanced tube bundles will be investigated after the pure liquid tests are performed.

APPROACH DESCRIPTION: This is primarily an experimental investigation. A wide range of surface geometries was tested in the pool boiling scoping tests to obtain the "best" surfaces for use in the bundle boiling tests. These surfaces will be tested over wide ranges of mass flux, heat flux, pressure and quality in the two bundle test sections. Heat transfer coefficient and pressure drop data are to be obtained. Tests will be run both with pure liquids and mixtures of two liquids. Fouling tendencies of the structured surfaces have been studied in water and refrigerant/oil mixtures. Pool boiling tests with a wide range of surface structure sizes for both enhanced surfaces were performed.

CURRENT STATUS (9/89): Of the four tasks in the formal Statement of Work, we are about on time on all of them. The major variances are the pool boiling experiments which are approximately 6–9 months ahead and the bundle boiling tests which are about 2–3 months behind.

ACCOMPLISHMENTS: All pool boiling experiments with pure liquid, binary mixtures, and fouling solutions have been run. The bundle test section has been constructed, experimental procedure developed and validated, and data are being obtained. Testing of the plain tube bundle with one layout is nearly complete.

FUTURE WORK: Bundle boiling data will continue to be obtained with the smooth tube in the second bundle layout and the High Flux and Turbo–B tubes in both bundle layouts. the loop will be modified for multicomponent operation, and experimental results will be incorporated into the computer simulation of a kettle reboiler. Details of the fouling of structured surfaces will be investigated by physical and mathematical models. Discussions with the industrial participants and others will be held to transfer to industry the knowledge developed in this program.

CONCLUSIONS: The project objectives are being achieved, valuable information is being obtained and a successful conclusion to the project is anticipated.

BIBLIOGRAPHY:

1. Jung, C. and Bergles, A. E., "Evaluation of Commercial Enhanced Tubes in Pool Boiling," Topical Report Submitted to D.O.E., Idaho Operations Office, DE–FC07–88ID 12772, March 1989.

ENHANCED SHELL-AND-TUBE HEAT EXCHANGERS FOR INDUSTRY

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Industrial Partners

A. Czikk
P. S. O'Neill
E. G. Ragi

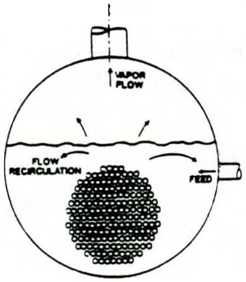
U.O.P.
Engineering Products
Tonawanda, NY

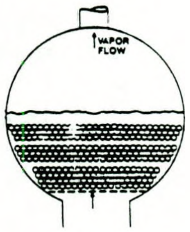
N. R. Clevinger
P. Thors

Wolverine Tube Inc.
Decatur, AL

PROJECT DESCRIPTION

- Experimental investigation to answer three main questions related to enhanced boiling heat transfer on the shellside of multitube bundles for the process and HVAC industries:
  - What are the effects of bundle geometry and enhancement technique on thermal-hydraulic performance?
  - How are thermal-hydraulics affected with multicomponent fluids?
  - Does the performance of enhanced surfaces degrade appreciably during boiling in the presence of typical contaminants?





HISTORY AND JUSTIFICATION

- Typical design for boiling on shellside used overall heat transfer coefficients

- What was occurring on individual tubes was unknown and ignored

- Realization that to design better and more efficient heat exchangers detailed knowledge is required

- Use of enhanced surfaces can result in smaller heat exchanger for same heat duty or larger heat duty for same size

- Gains resulting from using improved thermal hydraulic design and enhanced surfaces could be lost by fouling

- Details about the thermal-hydraulic behavior of multicomponent mixtures boiling on the shellside of tube bundles are unknown

- Project will examine these issues

OBJECTIVES

- Investigate the use of enhanced tubes in crossflow in multitube bundles

    - Perform single tube scoping tests in pool boiling to choose the surface geometry of two different surface enhancements for use in the bundle tests
    - Test the two enhanced surfaces and a plain surface in two tube bundle models
    - Simulate with a computer program the performance of a kettle reboiler using data obtained from the experiment
    - Perform the above tests with pure fluids and binary mixtures

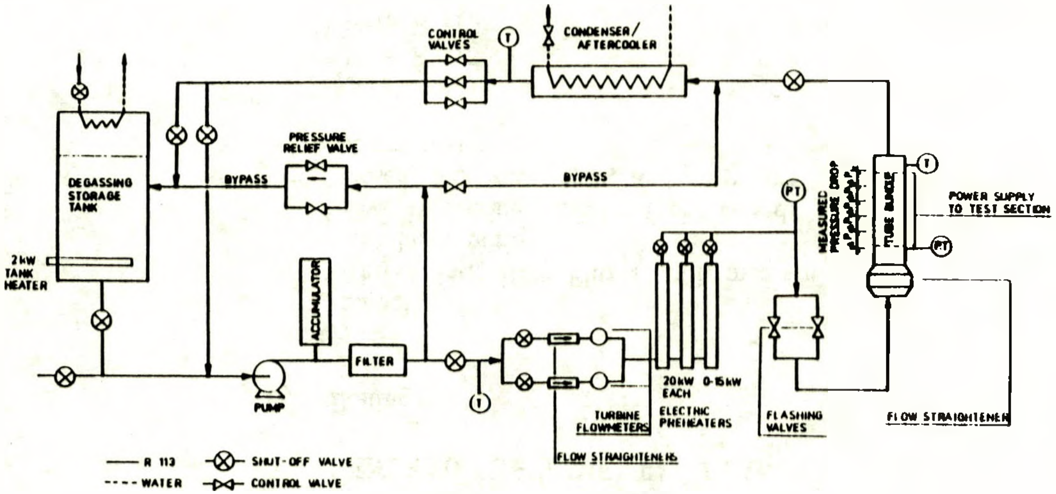
- Investigate the fouling tendencies of the enhanced surfaces

    - Perform pool boiling fouling tests with the enhanced and smooth surfaces
    - Develop a phenomenological model of the fouling process on enhanced surfaces

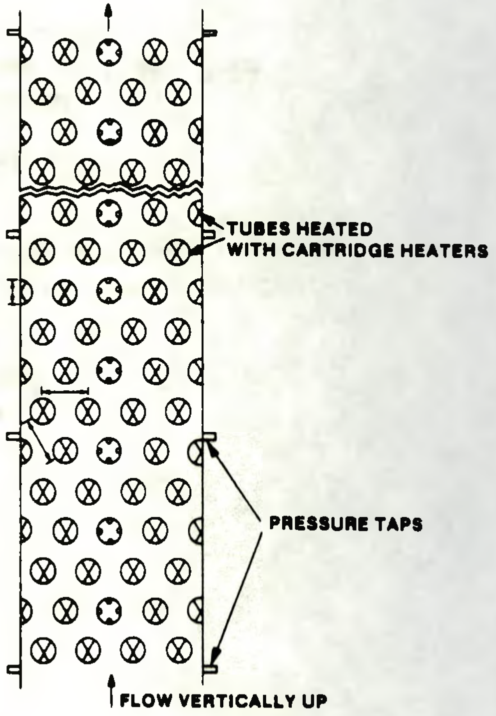
BUNDLE BOILING
GENERAL EXPERIMENTAL PLAN

- Pool Boiling
  - *Surfaces*
    - Smooth
    - Three Linde High Flux (small, medium and large pores)
    - Five Wolverine Turbo-B (very closed, closed, moderate, open, and very open structures)
  - *Liquids*
    - Pure R-113
    - Pure R-11
    - Mixtures of R-113 and R-11
  - *Operating Conditions*
    - One atmosphere
    - Saturated liquid
    - Range of imposed heat fluxes

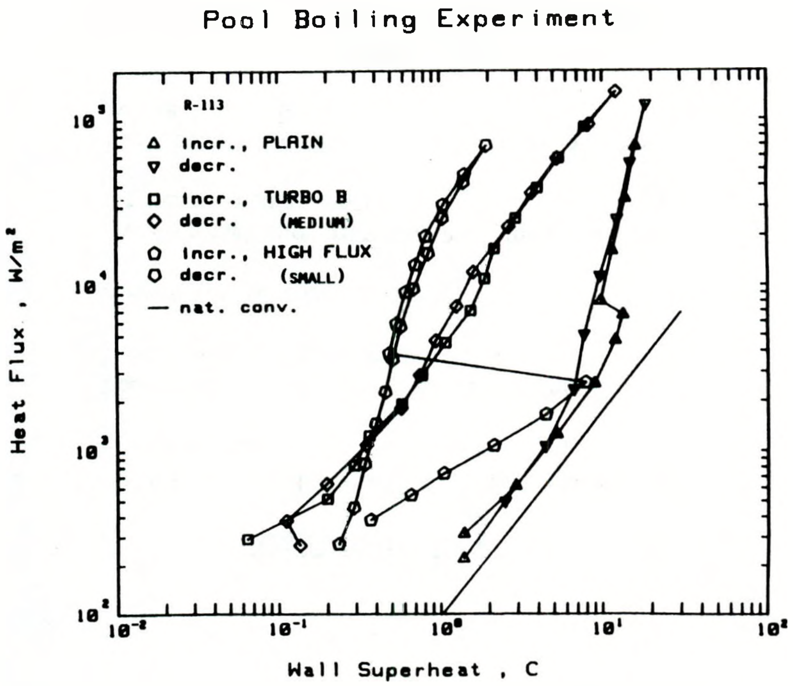
- Bundle Boiling
  - *Surfaces*
    - Smooth
    - "Best" High Flux
    - "Best" Turbo-B
  - *Bundle Layout*
    - Two staggered tube arrays with different tube-pitch-to-diameter ratios
  - *Liquids*
    - Same as pool boiling
  - *Operating Conditions*
    - Range of mass fluxes, heat fluxes, pressures and qualities

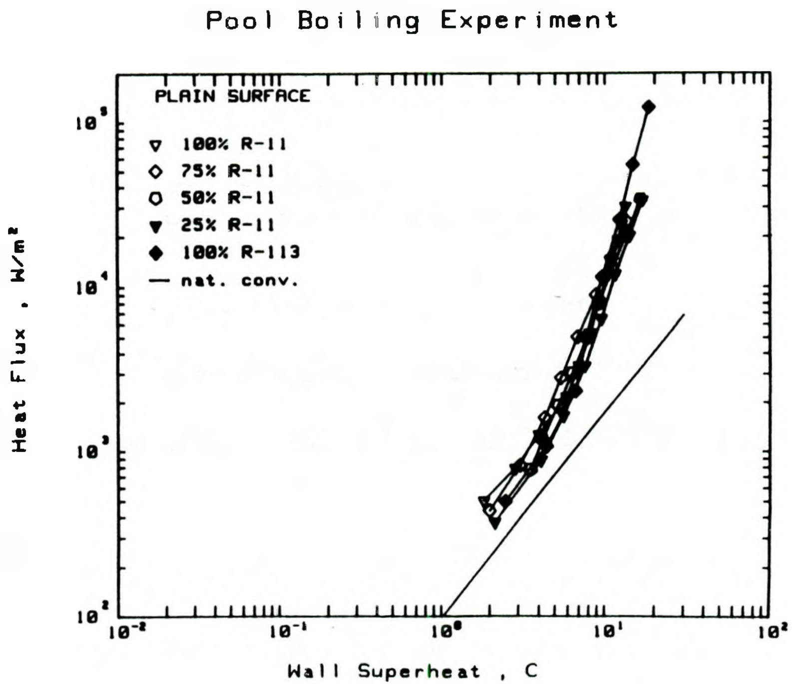


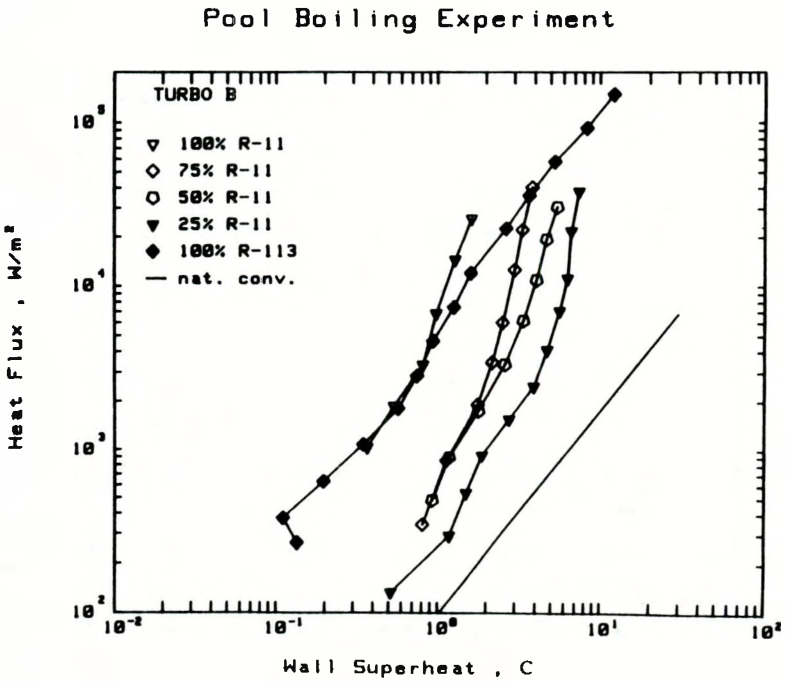
Flow Loop

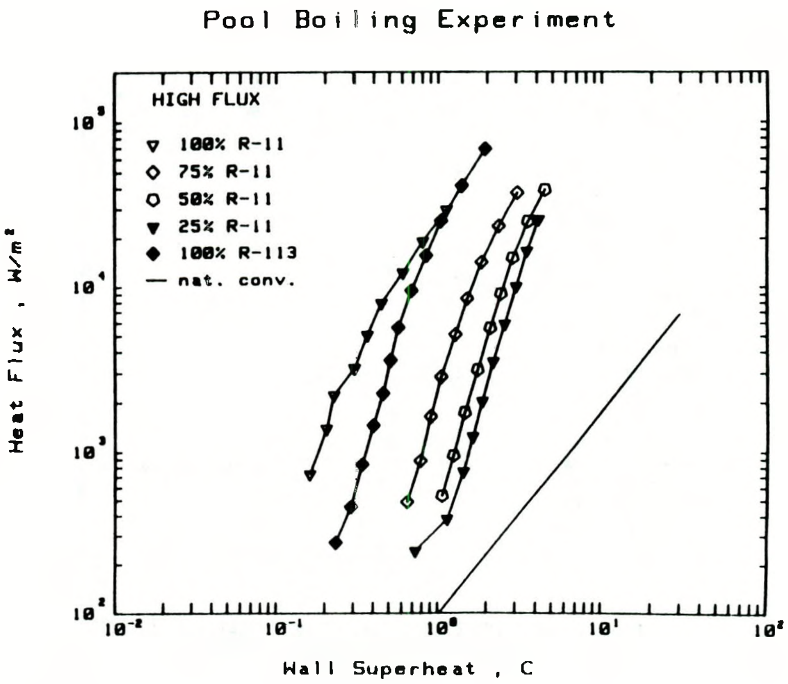


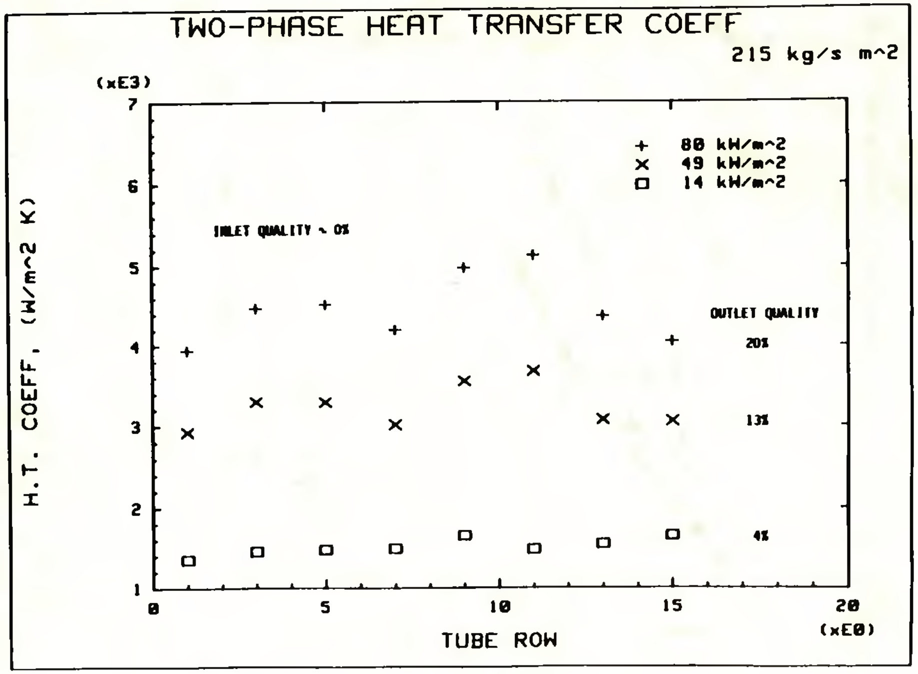
SCHEMATIC OF STAGGERED (EQUILATERAL) TEST SECTION

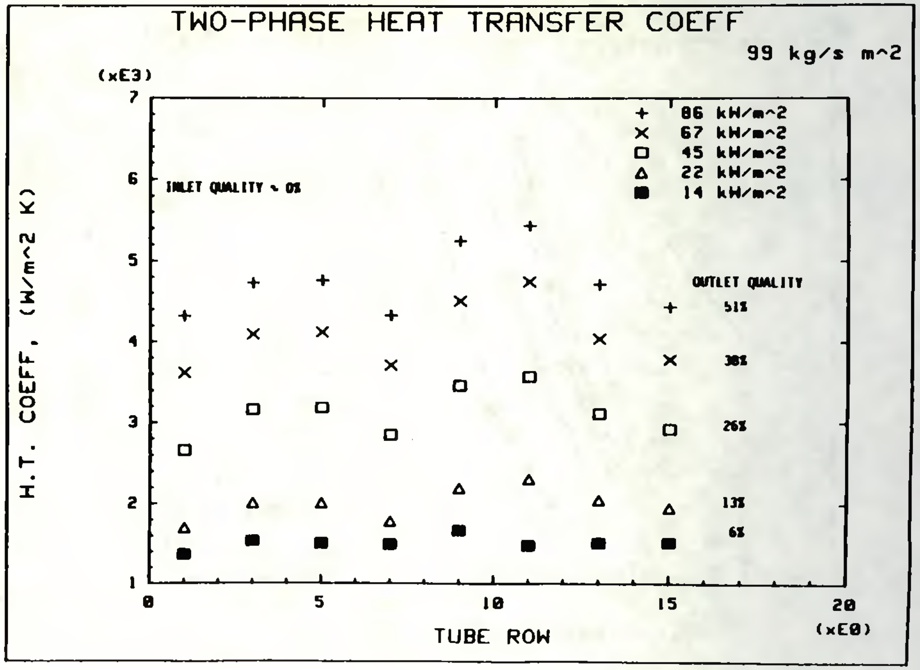

Pool Boiling Experiment
R-113
△ incr., PLAIN
▽ decr.
□ incr., TURBO B
◇ decr. (MEDIUM)
⬠ incr., HIGH FLUX
⬠ decr. (SMALL)
— nat. conv.
Heat Flux , W/m²
Wall Superheat , C
10²
10³
10⁴
10⁵
10⁻²
10⁻¹
10⁰
10¹
10²


Pool Boiling Experiment
PLAIN SURFACE
▽ 100% R-11
◇ 75% R-11
○ 50% R-11
▼ 25% R-11
◆ 100% R-113
— nat. conv.
Heat Flux , W/m²
Wall Superheat , C
10²
10³
10⁴
10⁵
10⁻²
10⁻¹
10⁰
10¹
10²


Pool Boiling Experiment
TURBO B
▽ 100% R-11
◇ 75% R-11
○ 50% R-11
▼ 25% R-11
◆ 100% R-113
— nat. conv.
Heat Flux , W/m²
Wall Superheat , C
10²
10³
10⁴
10⁵
10⁻²
10⁻¹
10⁰
10¹
10²


Pool Boiling Experiment
HIGH FLUX
▽ 100% R-11
◇ 75% R-11
○ 50% R-11
▼ 25% R-11
◆ 100% R-113
— nat. conv.
Heat Flux , W/m²
Wall Superheat , C
10²
10³
10⁴
10⁵
10⁻²
10⁻¹
10⁰
10¹
10²





RESULTS TO DATE

- All pool boiling tests with pure and binary mixtures done
- Pool boiling scoping experiments to identify "best" enhanced surfaces successful
- Bundle test sections constructed
- Experimental apparatus and procedures validated for bundle boiling tests
- Data have been obtained for the smooth tubes in one bundle layout
- Computer simulation program has been developed for the thermal-hydraulics in a kettle reboiler

FUTURE ACTIVITIES ON BUNDLE BOILING

- Continue obtaining experimental data
- Modify flow loop for multicomponent operation
- Begin incorporating experimental results into computer simulation

FOULING TENDENCIES OF ENHANCED HEAT TRANSFER SURFACES

E.F.C. Somerscales

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Department of Mechanical Engineering,
Aeronautical Engineering & Mechanics

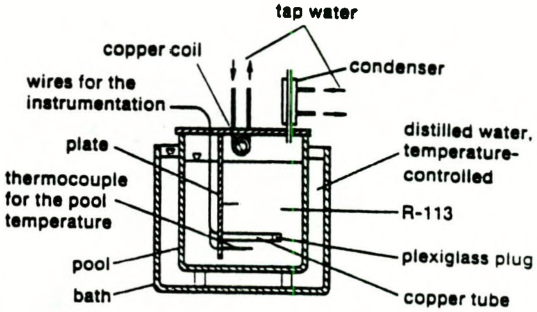
EXPERIMENTAL PLAN (Continued)

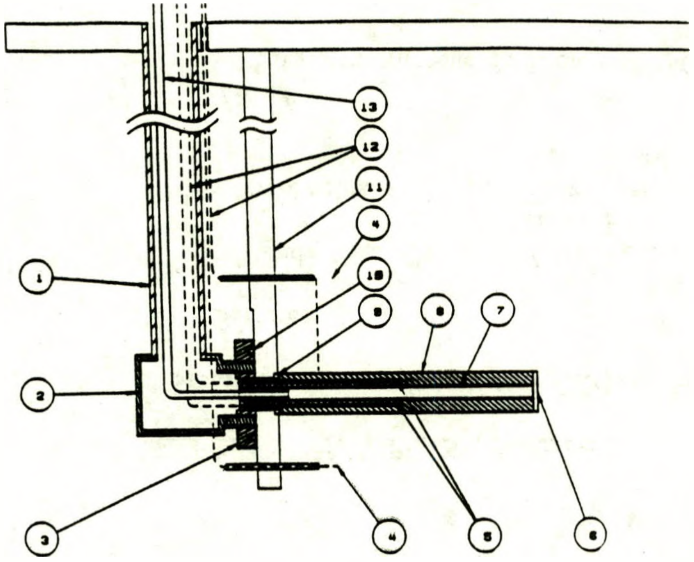
- Saturated Aqueous Solution of Calcium Sulfate
  - *Surfaces*
    - Plain
    - Linde High Flux: Small Pore
      - Large Pore
    - Wolverine Turbo B: Very Open
      - Very Closed
  - *Liquid*
    - Saturated Aqueous Solution of $CaSO_4$
  - *Operating Conditions*
    - Nucleate Boiling at 1 Atmosphere
    - Two Heat Fluxes
    - 10 kW/m^2
    - 50 kW/m^2

EXPERIMENTAL PLAN

- Refrigerant-oil Mixtures
  - *Surfaces*
    - Plain
    - Linde High Flux: Small Pore
      - Large Pore
    - Wolverine Turbo B: Very Open
      - Very Closed
  - *Liquids*
    - Pure R-113
    - Mixtures R-113-Oil
  - *Operating Conditions*
    - Nucleate Boiling at 1 Atmosphere
    - Saturated Liquid
    - Various Heat Fluxes

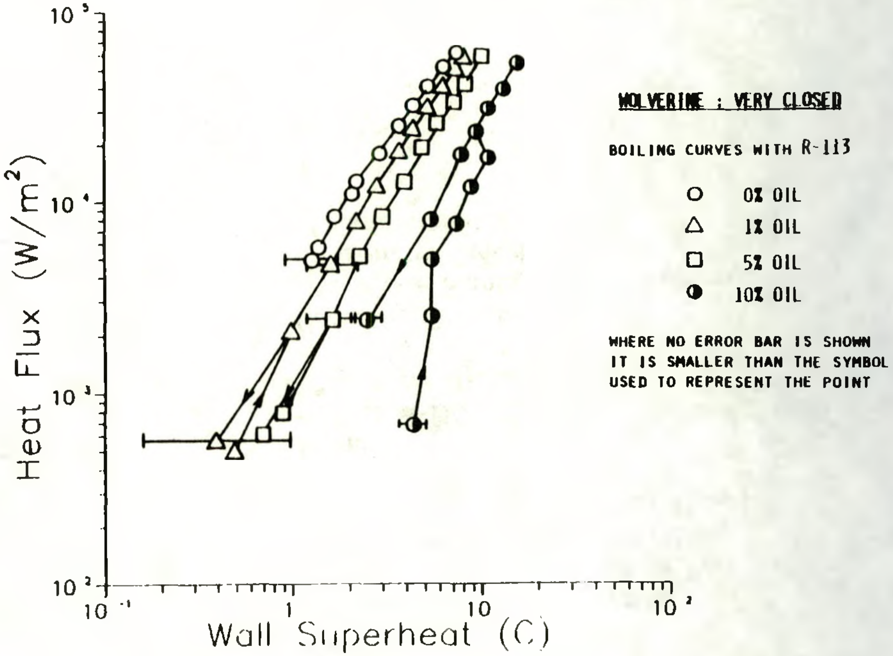
POOL BOILING TEST SECTION SCHEMATIC



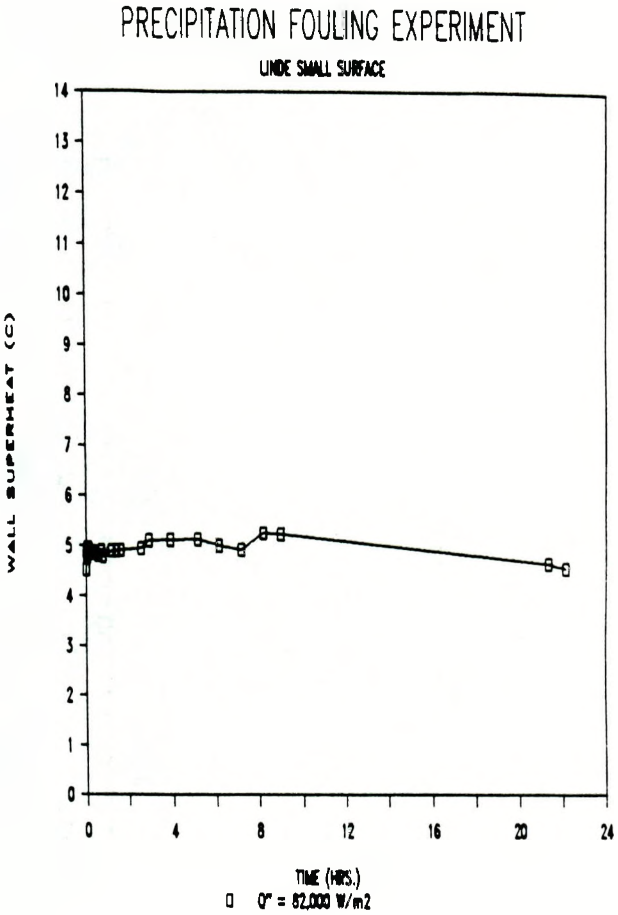


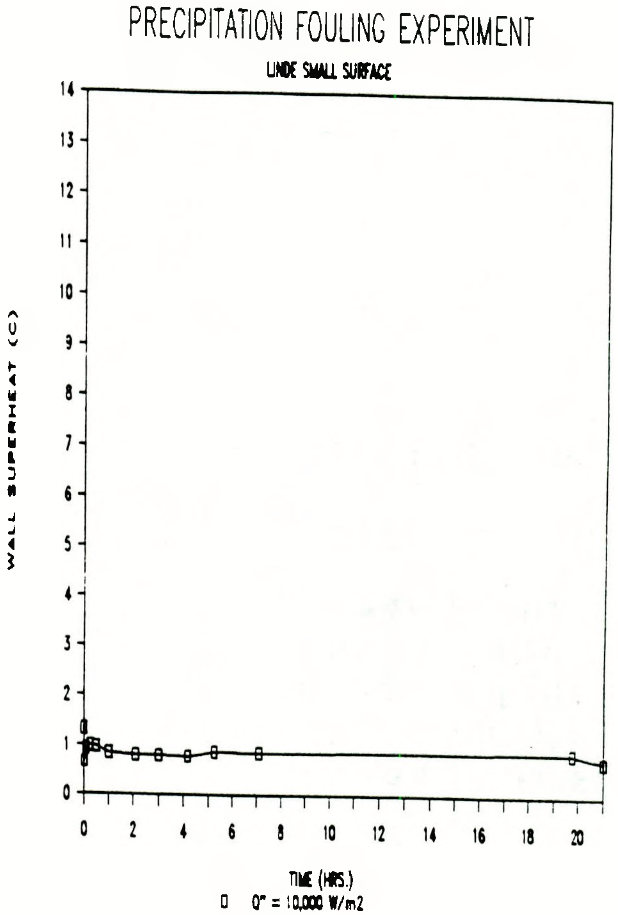
TEST SECTION

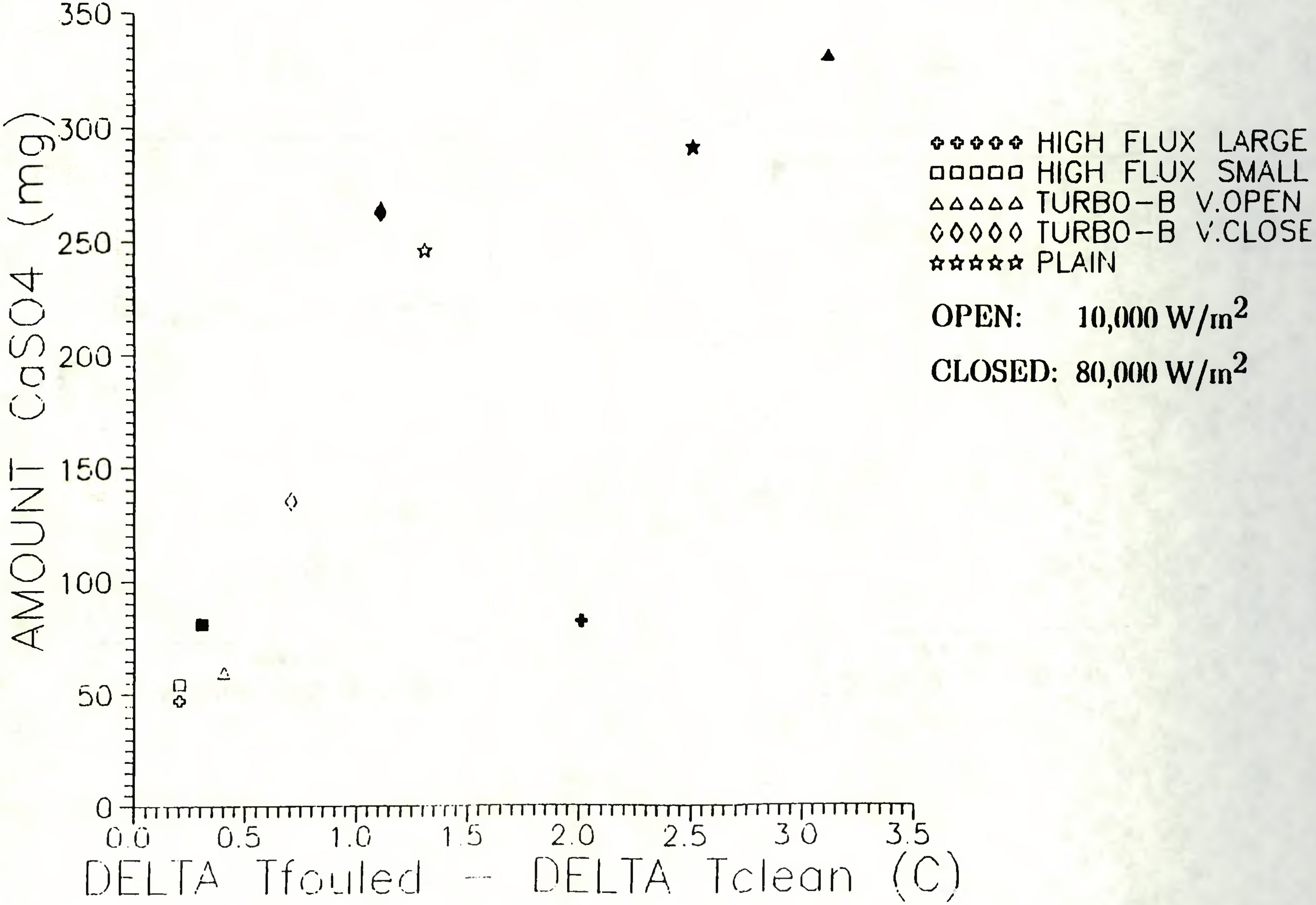
1. Protection tube for power and thermocouple leads
2. Elbow
3. Connector
4. Thermocouples recording fluid temperature
5. Thermocouples recording test section temperature
6. End seal
7. Cartridge heater
8. Test section
9. O-ring seal
10. O-ring seal
11. Test section support
12. Thermocouple leads
13. Power leads



WHERE NO ERROR BAR IS SHOWN IT IS SMALLER THAN THE SYMBOL USED TO REPRESENT THE POINT


PRECIPITATION FOULING EXPERIMENT
LINDE SMALL SURFACE
WALL SUPERHEAT (C)
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
TIME (HRS.)
0
4
8
12
16
20
24
□ Q" = 82,000 W/m2


PRECIPITATION FOULING EXPERIMENT
LINDE SMALL SURFACE
WALL SUPERHEAT (C)
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
TIME (HRS.)
0
2
4
6
8
10
12
14
16
18
20
□ Q" = 10,000 W/m2


AMOUNT CaSO4 (mg)
0
50
100
150
200
250
300
350
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
DELTA Tfouled – DELTA Tclean (C)
✣✣✣✣✣ HIGH FLUX LARGE
□□□□□ HIGH FLUX SMALL
△△△△△ TURBO–B V.OPEN
◇◇◇◇◇ TURBO–B V.CLOSE
☆☆☆☆☆ PLAIN
OPEN: 10,000 W/m^2
CLOSED: 80,000 W/m^2

RESULTS

- Refrigerant-oil Mixtures
  - Increasing amounts of oil degrade the heat transfer performance of enhanced surfaces
- Saturated $CaSO_4$ Solutions
  - Heat transfer performance of enhanced surfaces degraded by fouling
  - Fouling effects increase with increasing heat flux
  - Small pore surfaces (Linde) are less affected by fouling than surfaces with large openings

FUTURE ACTIVITIES

- Physically model fouling processes in cavities on an enhanced heat transfer surface
- Mathematically model fouling processes in cavities on an enhanced heat transfer surface

CONCLUSIONS

- Measured the effect on the performance under boiling conditions of advanced heat transfer surfaces of
  - R-113-Oil Mixtures
  - Aqueous $CaSO_4$ Mixtures
- Performance of surface is degraded. Depends on:
  - Liquid Composiiton
  - Heat Flux
  - Surface Geometry

SUMMARY

- Accomplishments
  - Evaluated three High Flux and five Turbo-B enhanced tubes in pool boiling and selected one of each for further bundle boiling tests
  - Designed, built and tested bundle test sections
  - Demonstrated effects of fouling on enhanced heat transfer surfaces under boiling conditions
- Future Goals
  - Obtain bundle boiling data for the enhanced surfaces in the two bundle layouts
  - Obtain bundle boiling data for multicomponent mixtures
  - Investigate details of fouling process by physical and mathematical models

DISCUSSION

Enhanced Shell-and-Tube Heat Exchangers for Industry
Arthur E. Bergles

Q: What was the diameter of the test tube in your activities?

A: They were half-inch in diameter, and three-fourths of an inch in diameter.

Q: You measure the temperature in the wall. Do you have some method developed for specifying or calculating the surface temperature as well for this kind of tube?

A: Yes, we just use the conduction equation for a cylinder to correct for the temperature drop between the thermocouple and the surface.

Comment: But the surface is irregular.

A: Yes, the surface is irregular, so we have to make an assumption of some average thickness of the wall.

Q: The fouling tests ran for 24 hour periods. Do you feel that's sufficient to describe long term fouling effects in these types of heat exchangers?

A: Looking at data from other work in this area, I think it probably is. Most of the changes seem to occur in the first 1 or 2 hours on those particular surfaces. The periods are not long term, but apparently the fouling effects start right at the beginning and just level off.

Q: I noticed on your deposits with reference to delta T, it appears that you are getting the same results for both the enhanced and the plain surfaces. It seems that your preliminary findings suggest that fouling of the enhanced surface and the plain surface are about the same. Is that a true statement?

A: It does vary depending upon the surface geometry. (Referring to results)

ENHANCED TUBES FOR STEAM CONDENSERS

Ralph L. Webb
Pennsylvania State University

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAM
ADVANCED HEAT EXCHANGERS PROGRAM

PROJECT DESCRIPTION

TITLE: ENHANCED TUBES FOR STEAM CONDENSERS
COOPERATIVE AGREEMENT NO: DE-FC07-88ID12708
CONTRACTOR: The Pennsylvania State University
University Park, PA 16802

PRINCIPAL INVESTIGATOR: Ralph L. Webb, 814-865-0283

OBJECTIVE: Develop tubes having enhanced surface geometries on the inside and outside surface, which will provide high performance in steam condensers, relative to the currently used plain tubes.

TECHNICAL APPROACH: The program goals are being pursued in three sub-programs. The first sub-program involves identification and development of steam side enhancement geometries. A computer program developed at Penn State is used to predict the steam condensation performance of integral fin tubes having different fin spacing, fin height and fin shape, for titanium and copper-nickel materials. The "optimum" fin geometry is then identified for manufacture and test. Wolverine Tube, Inc. (the industrial partner) will fabricate the test geometries. The steam condensation performance of these tubes will be tested in a steam condensation test cell having a vertical rank of five horizontal tubes.

The second sub-program involves identification of the preferred tube side enhancement. We are using semi-empirical correlations to predict the heat transfer coefficient of candidate surface enhancements as a function of the enhancement element height. Selection of the preferred enhancement geometry must account for its water side fouling and erosion characteristics.

Having selected candidate water and steam side enhancements, sub-program three will address the performance of candidate "doubly enhanced" tubes (steam and water side enhancements). A computer program will be used to predict the performance of the candidate doubly enhanced tubes in a simulated electric utility condenser. The goal is to attain the lowest possible condensing temperature for a tube-for-tube replacement. Wolverine will make tubes having the selected water side enhancement geometries. These tubes will be tested to measure the water side heat transfer coefficient, and to measure their fouling characteristics. Two types of fouling tests are being conducted. The first uses particulate fouling, and measures the ability of recirculating sponge balls to clean the tubes. The second will use biological fouling, and investigate the ability of sponge balls and chlorination to remove the foulant.

Finally, candidate tubes having the preferred steam and water side enhancements will be fabricated by Wolverine and tested in a manner that simulates the actual operation of a steam condenser tube. In this test, low pressure steam will condense on the outer tube surface, and the heat will be absorbed by cooling water at 6 ft/sec on the tube side.

CURRENT STATUS: Candidate steam and water side enhancements have been selected, and the needed tube geometries have been built. The steam condensation and water side heat transfer coefficient and fouling tests are in progress. The development of titanium tubes that will provide high performance has become a challenging goal. This is because titanium has very low thermal conductivity - less than 5% that of pure copper. The electric utility industry prefers titanium over copper alloys, because of its superior corrosion and erosion resistance.

ACCOMPLISHMENTS: We have identified tube geometries that are predicted to reduce the steam condensing temperature by 5 F, or more. Fouling tests have shown that the candidate water side enhancements appear to be cleanable using the sponge ball cleaning system.

FUTURE WORK: The experimental heat transfer program is in progress.

CONCLUSIONS: It appears that we will be able to attain practical tube geometries that will provide significant performance improvement for copper alloys. We are working on an innovative aluminum-titanium tube that appears promising for titanium tubes. We believe that the water side of the tubes will be cleanable.

file: projdesc

ENHANCED TUBE FOR STEAM CONDENSERS

R L. Webb, N-H. Kim, and H. Jaber
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INTRODUCTION

This program seeks to develop tubes having enhanced surface geometries on the inside and outside surface, which will provide high performance in steam condensers, relative to the currently used plain tubes. The program is applicable to industrial and electric utility steam condensers.

Steam condensers have typically used plain tubes. This is due to several reasons. Reliability is the major requirement for electric utility applications. A steam condenser tube is expected to last 20-30 years. Fouling, erosion and corrosion are contributors to tube failure. The electric utility has not favorably viewed enhanced tube geometries because of three reasons:

1. The controlling resistance is on the water side.

2. It was not thought possible to provide significant enhancement to the condensing side.

3. An enhanced water side tube geometry raises reliability concerns.

A commercially viable enhancement geometry must address the above three concerns. Approximately two-thirds of the total thermal resistance is on the water side of the plain tube. In order to obtain significant performance improvement, it is necessary to enhance the water side, in addition to the steam side. Until approximately 1984, it was believed that it was not possible to design a surface geometry that would significantly enhance the condensation performance. Further, there had been no work done to measure the fouling characteristics of enhanced tubes. It was generally assumed that they would show significant fouling, and not be cleanable. The electric utility industry has been moving away from copper alloy tubes, in favor of titanium tubes. This material choice is based on the belief that titanium tubes will have significantly erosion/corrosion problems.

Presently, there is no commercial tube available that provides significant enhancement on the water and steam sides. The only commercial enhanced tube that has been evaluated in electric utility steam condensers is the "Korodense" tube, as reported by Boyd et al (1983). This tube has helical ridges on the outer surface, which provide approximately 80% water side enhancement. However, it provides less than 30% steam side enhancement. It was installed in a TVA condenser and subjected to long term tests. It was observed that the dirty river water caused significant water side fouling, and loss of performance enhancement after about 30 days.

Significant research has been done on enhancement of steam condensation over the past four years. Wanniarachchi et al. (1986) report data that have been taken on integral fin tubes, and Webb (1988) describe an analytically based models that are applicable to the problem. We believe that it is possible to design a steam side enhancement geometry that will provide 50-300% steam side enhancement, depending on the tube material. However, the low thermal conductivity of titanium seriously degrades the performance of finned titanium tubes. Webb and Kim (1989) have been actively pursuing work on fouling and fouling control in enhanced tube geometries. Taprogge (1977) has developed a "recirculating sponge ball" cleaning system that has been installed in many electric utility plants having plain tubes. Other fouling research has shown that intermittent chlorination can effectively control biological fouling in plain tubes. This work has provided a knowledge base to identify potentially cleanable water side enhancements. Enhanced tube manufacturers have made significant advances in their ability to make cost effective doubly enhanced tubes. These advances indicate that it may be possible to develop a new doubly enhanced tube that can be used in steam condensers, and that it will be possible to control water side fouling.

The present program seeks to define a tube geometry that has special enhancement geometries on the tube (water) side and the steam (shell) side. Enhancement geometries will be developed for both cu/ni and titanium tube materials. Water side fouling and fouling control is a very important consideration affecting the choice of the tube side enhancement. Hence, we have consciously considered fouling potential in our selection of the tube side surface geometry.

It is envisioned that the enhanced tube would initially be used for retubing (tube-for-tube replacement) of existing steam condensers. This will yield a condenser tube that has a UA value 50-80% higher than the presently used plain tube. The increased UA value will allow reduction of the steam condensing temperature. This allows expansion of the steam to a lower turbine exhaust pressure, resulting in increased electricity generation. Analytical work completed to date (1) shows that the steam condensing temperature may be decreased 5-8 F for Cu-Ni or stainless steel tubes. Used in a 946 MW nuclear plant the simulation analysis (1) showed that the annual average generation rate would be increased as much as 2.6 MW, and the peak load generation was increased 10.8 MW. These performance improvements assumed no plant alterations, other than the tube-for-tube replacement. If it is possible to replace the tube sheets in the retubing, greater performance improvement is possible. Ideally, the turbine should be designed to take advantage of the lower condensing pressure. If this is done, for a new plant design, the full performance of the enhanced condenser tube may be attained.

PROJECT PARTICULARS

The project work is addressed in three sub-programs. They are:

1. Water Side Enhancement: This work involves selection of the water side enhancement geometry, and evaluation of its heat transfer and fouling characteristics.

2. Steam Side Enhancement: This addresses selection of the steam side enhancement geometry, and measurement of the steam condensation coefficient on a single tube, and on a bank of tubes.

3. After completion of the two sub-programs, the final candidate enhanced tubes will be manufactured having the desired outside and inside enhancements. The performance of these tubes will be predicted, and a test will be run that simulates actual condenser operating conditions.

Water Side Enhancement Sub-Program:

Four basic types of tube side enhancement geometries were analyzed. The calculations simulated the water-side performance in an actual electric utility condenser (fossil plant, 7/8" O.D. titanium tubes with 0.028" wall thickness) and compared the resulting steam condensing temperature with that of a plain tube. The analysis assumed that the steam side condensation coefficient is 80% greater than that of a plain tube. The analysis simulated a tube-for-tube replacement, and accounted for the increased water-side friction factor by calculating the flow rate at the new balance point on the pump curve. This procedure was applied to the fossil plant described by Webb and Haman (1984). Based on the computational results, the most promising roughness height and pitch were selected for each roughness configuration.

Table 1 shows the enhancement ratio (E_i) of the selected water side enhancements. Figures 1 through 6 shows illustrations of the different enhanced tubes that are discussed in this report. The "enhancement ratio" is defined as the ratio of the heat transfer coefficient in the enhanced tube (h_i) to that of a plain tube ($h_{i,p}$).

Because the water side thermal resistance in a plain tube is approximately twice that on the condensing side, significant increase of the overall heat transfer coefficient cannot be realized, unless the water side is enhanced.

Fouling tests are currently being conducted on the enhancement geometries. The present test series is measuring the fouling factor for particulate fouling. The tubes will also be cleaned with recirculating sponge rubber balls. The purpose of these tests is to compare the fouling rate and cleanability of the enhanced tubes to that of the plain tube. At the completion of the particulate fouling test series, we will perform biological fouling tests. These tests will investigate tube cleanability using sponge rubber balls, and intermittent chlorination.

Condensation Sub-Program

We have developed an analytically based computer model to predict steam condensation on horizontal integral fin tubes. This model is based on prior work of Webb et al. (1985), and Webb and Adamek (1989a, 1989b). Our computer model was used to predict the steam condensation coefficient on integral fin tubes as a function of fin height, fin spacing, fin shape, and material conductivity. Our model shows that the highest steam side performance will occur for approximately 19 fins/in. As the material thermal conductivity decreases, the optimum fin height will decrease. Our analysis shows that the preferred fin height is in the range of 0.020-to-0.040 in, with the lower fin height for titanium or stainless steel tubes. We have investigated two fin shapes - the "trapezoidal" shape commercially used, and a "profiled" shape, that has curved sides and a small fin tip radius. The profiled shape is described by Webb and Kedzierski (1989). Our predictions show that the profiled shape will give a higher condensation coefficient.

Table 2 shows the predicted enhancement ratio, E_o (ratio of condensation coefficients on the enhanced and plain tubes) for an integral fin geometry of 19 fins/in, 0.020 in high on a 1.0 in O.D. tube. This is the trapezoidal shape, whose fin base thickness is 0.012 in, and the tip thickness is 0.008 in. This table shows that the smallest enhancement ratio occurs with the low thermal conductivity titanium tube (k = 11 Btu/hr-ft-F). An admiralty tube (k = 70 Btu/hr-ft-F) gives a much higher enhancement ratio.

The predicted enhancement ratio for the titanium tube in Table 2 is 1.19. This is very small. In an attempt to improve the performance of the low thermal conductivity titanium tube, we have investigated the effect of increasing the fin base thickness. Table 3 shows the effect of fin base thickness on the steam side enhancement ratio for trapezoidal titanium fins having 0.008 in tip thickness. This table shows that the enhancement significantly increases as the fin base thickness is increased. This performance increases because the fin efficiency increases as the fin base thickness is increased.

Figure 2 shows as alternate condensation enhancement geometry, which may be important for titanium tubes. Used in steam a condenser, this would consist of 0.5 mm diameter aluminum particles sintered to the surface of a titanium tube. Because aluminum has 10 times the thermal conductivity of titanium, a high fin efficiency will exist. This tube is being made by Linde, who has a patent on the technology. The test tube will have copper particles on a cu/ni tube. If the performance of this tube is as high as expected, additional manufacturing development would be required by Linde to make a tube having aluminum particles on a titanium tube.

Table 4 shows the outside surface geometries we will test for steam condensation. We will measure the condensation coefficient using the test cell illustrated in Fig. 7. The cell has a vertical rank of five horizontal tubes. The tests will be run with steam condensation temperatures at 100-to-115 F. The finned tubes will have a trapezoidal fin shape, as compared to the higher performance profiled shapes, whose performance was given in Tables 2 and 3.

Proof of Concept Sub-Program

After the water side and steam condensation tests are completed, we will select the final enhancement geometries to be used for the cu-ni and titanium tubes. Then, we will make 8.0 ft long tubes having the chosen enhancements on the steam and water sides of this tube. This tube will be installed in a test cell and operated at steam and water flow conditions that approximate the conditions that exist in a steam condenser. This test will give the overall heat transfer coefficient (U-value) of the enhanced tube. At this time, it appears that the "proof of concept" tubes will include one or more of the doubly enhanced tube geometries listed in Table 5. We have recently determined that an electric utility steam condenser tube will have 0.018 in wall thickness, rather than the 0.028 in value that has been used in the analysis.

Application to Industrial Condensers

Our program focuses directly on electric utility steam condensers, because of the large application potential. However, it is also applicable to any area, where steam is condensed. The "industrial" applications include, all industrial condensers that use shell side condensation, co-generation plants that use water as the working fluid, absorption refrigeration condensers, and nuclear submarine condensers.

We are optimistic that we will successfully achieve a copper alloy tube geometry that will result in 80-100% higher UA-value than is provided by a plain tube. Problems exist for titanium tubes, because of its very low thermal conductivity. Use of a thicker fin base thickness (0.024 in) may provide better performance. The use of aluminum particles on a titanium base tube is believed to offer the best potential for titanium tubes.

Technical Advisory Group

It is our intent that the work be performed in a manner that is acceptable to the members of the electric utility industry and other industrial users. The test procedures, test results, and evaluation methodology should provide information that they need to reach application decisions. We wish to form an Advisory Group and inform the members of the work as it proceeds, so that their input and commentary can be considered in work planning, execution and evaluation of results.

The proposed interaction with the Advisory Group includes:

1. A Group meeting in October 1989. We will present the results of our theoretical predictions, surface selection, test planning, and demonstrate our test apparatus.
2. A Group meeting in August 1990, at the end of the present project. We will present test results and discuss the direction of future work.
3. The Advisory Group will be provided with quarterly progress reports.

FUTURE ACTIVITIES

Listed below is a brief description of the work to be performed in the second program year. 1990.

Manufacturing of the Candidate Tubes

Up to this point, theoretical analysis and the selection of the tubes are finished. Most of the candidate tubes will be manufactured at Wolverine Co. Some tubes will be obtained from other manufacturers. Listed below are the candidate tubes and the manufacturers.

Geom.	Inside Geometries	Manufacturer
1	3-dim. roughness	Wolverine
2	Wavy	Wieland
3	Corrugated, 45 deg helix angle	Yorkshire

Heat Transfer Measurements

Inside heat transfer coefficients of the 3-D roughness tubes are measured at Wolverine Co. For other geometries, the inside heat transfer coefficients will be estimated from the available correlations.

Fouling Measurements

Fouling tests are being conducted for Wolverine 3-D roughness tubes and a Wieland wavy tube using ferric oxide and aluminum oxide as foulant materials. The sponge ball cleaning tests will also be conducted. The O.D. of the tubes currently installed are 3/4". After the tests are over, the apparatus will be modified to be able to install 7/8" tubes. One Yorkshire corrugated tube and one 3-D tube will be tested. The O.D. of the tubes will be 7/8".

For those tubes which successfully passed particulate fouling tests, biological fouling tests will be conducted. This test is necessary because biofouling is the most severe problem in electric utility condensers. Prior to run biofouling, the copper piping of the apparatus will be replaced to PVC because the copper ions are toxic to microorganisms. Sponge ball cleaning system will also be tested.

Proof of Concept Test

This test will be conducted on an 8 ft section of tube having both water and steam side enhancements. Steam will condense at approximately 110 F on the outer surface, with water at 6 ft/sec on the inside.

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Table 1

Water Side Enhancement Ratio at Re = 40,000

Geometry	E_i
Plain	1.0
3-dim. roughness (Fig. 3)	1.65
Wavy (Fig. 4)	1.45
Corrugated, 45 deg helix (Fig. 5)	1.75
Corrugated 81 deg helix (Fig. 6)	1.90

Table 2

Effect Of Thermal Conductivity On The Enhancement Level for Trapezoidal Fin Shape
(19 fins/in, 0.020 in high on 1.0 in O.D. tube with 0.028 in wall;
0.012 in fin base thickness, and 0.008 in fin tip thickness)

k (Btu/hr-ft-F)	E_0
11	1.19
30	1.81
70	2.42

Table 3

Effect Of Fin Base Thickness On The Enhancement Level

(16 fins/in, 0.020 in high on 1.0 in O.D. tube with 0.028 in wall,
0.008 in fin tip thickness, t_b = fin base thickness, E_o = enhancement ratio)

t_b (in)	E_o
0.012	1.19
0.020	1.40
0.024	1.51

Table 4

Tube Geometries to be Tested in Condensation Test Cell

Geometry	Fin ht. (in)	Fins/in	Tube Dia. (in)	Material
Integral-fin	0.020	19	0.875	cu/ni
Integral-fin	0.040	11	0.75	cu
Integral-fin	0.040	11	0.875	cu/ni
Attached particles	0.040	none	0.75	cu particles on titanium tube

Table 5

Surface Geometries Selected for Experimental Evaluation

Number	Outside Geometry	Inside Geometry
1	13-19 fins/in, 1.0 mm fin height (Fig. 1)	3 - dimensional roughness (Fig. 3)
2	13-19 fins/in, 1.0 mm fin height	Wavy (Fig. 4)
3	13-19 fins/in, 1.0 mm fin height	Corrugated, 45 deg helix angle (Fig. 5)
4	3-dimensional distribution of 0.5 mm dia. attached metal particles (Fig. 2)	Corrugated, 45 deg helix angle

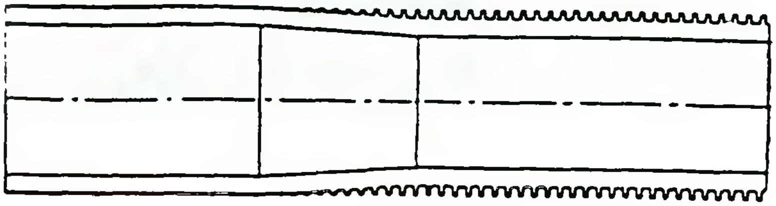


Fig. 1 Integral fin tube (19 fins/in, 0.020 in fin height).

Fig. 2a Aluminum particles sintered to base surface.

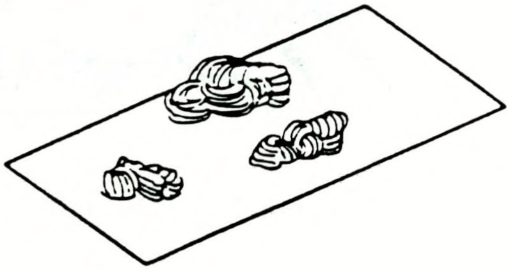


Fig. 2b Illustration of attached particles.

Fig. 3 3-dimensional roughened internal tube surface.

Fig. 4 Wavy internal tube surface.

Fig. 5 Corrugated tube (45 deg helix angle)

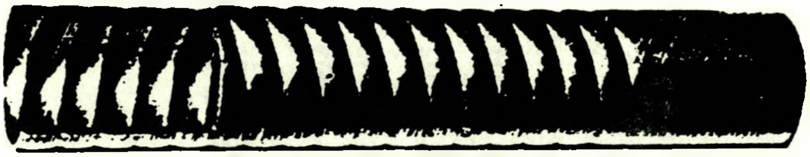


Fig. 6 Corrugated tube (81 deg helix angle)

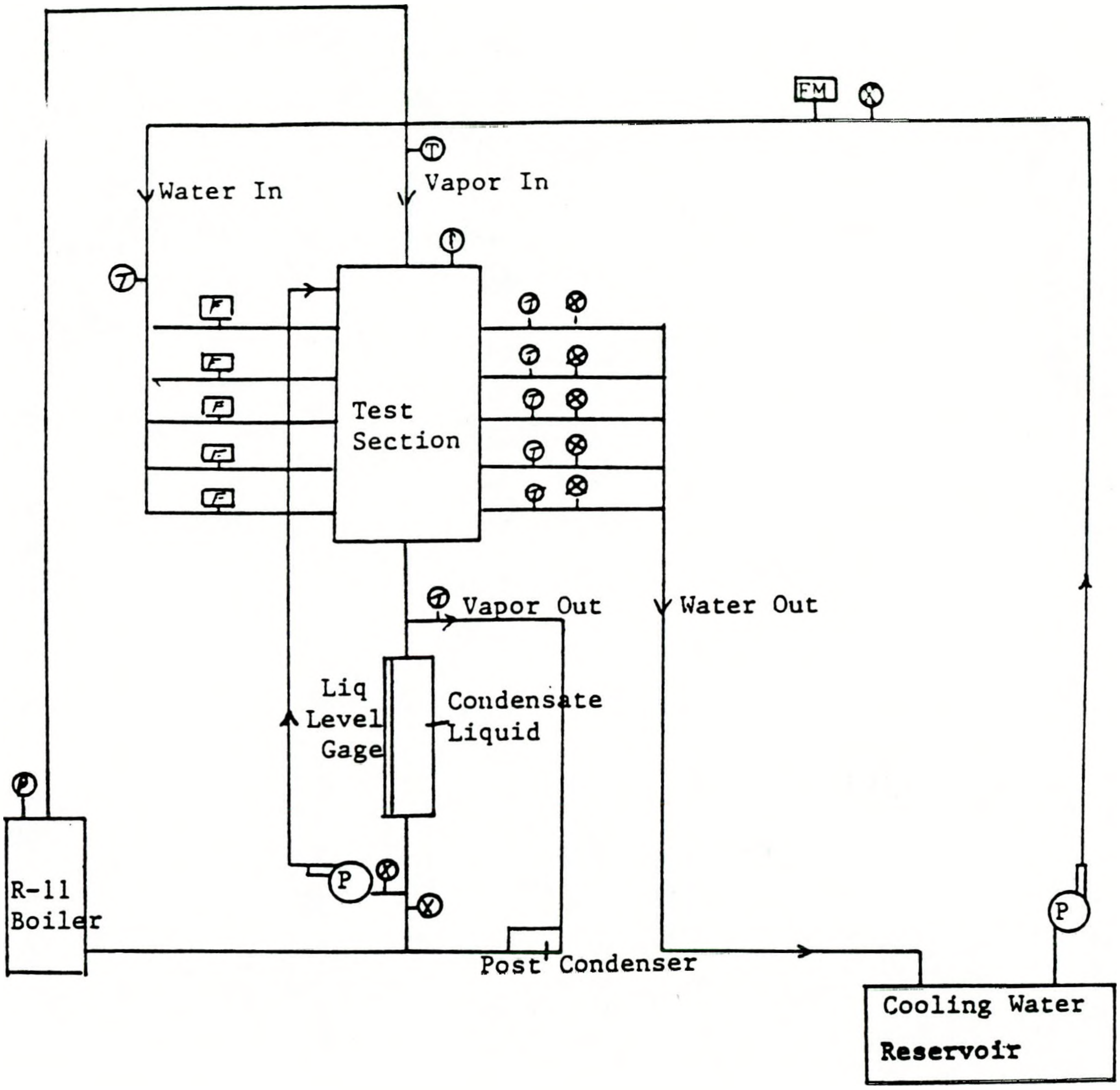


Fig. 7. Condensation Apparatus

DISCUSSION

Enhanced Tubes for Steam Condensers

Ralph L. Webb

Q: Please provide some input on the costs of these tubes. If we are going to be saving tube surface area, what do these enhanced tubes cost and what is the total cost benefit? Secondly, could you say something about whether there is a question of saving dollars, or could you address any savings in energy by using these tubes?

A: We have made the relevant predictions and published a paper that answers these questions. Also, in 1984 we published a paper at the ASME winter annual meeting in which we simulated installation of several different enhanced tubes in the same fossil plant and calculated performance. We did the same economic analysis that the electric utility industry does. First, let me explain how we use the tubes. Preferably I would like to use the tubes by building a new condensor, or by retubing a condensor with use of new tube sheets. However, we found that the most important current application is retubing a condensor by a tube-for-tube replacement. We did an economic payback and the numbers are given in the 1984 paper. The economic payback was considered very favorable by the electric utility industry. The tubes did cost more; a payback based on energy savings was calculated and was in the range of 2 1/2 to 3 1/2 years. One of the surprising things we found out was the impact on peakload capability. Apparently, this is a big bonus, because you don't have to buy gas turbines and run those to provide peakload capability. In the 600 megawatt plant analyzed, we increased the peakload capability of the plant around 3 megawatts, which is worth a lot of gas turbine dollars. Certainly we're paying more money for the tubes, but we're saving energy as a result of that, and getting higher peakload capability. The referenced paper is published in ASME symposium volume HTD Vol. 37, pp. 17-26. The paper is titled "Enhanced Tubes in Electric Utility Steam Condensers".

Q: It's pretty difficult to tube a unit with smooth tubes and in your write up you're talking about going to lighter wall tubes of titanium. Is there a notch sensitivity? In the application, the mechanical aspects of any enhanced surface tubing would appear to be very important, and have to be considered before the application.

A: I don't think the limiting factors in applying these tubes to electric utility steam condensers is necessarily how high you can increase the heat transfer coefficients or how well you can control the fouling. Erosion/corrosion stress concentration requirements as you describe them are really the bottom line issues. These can be answered only in long term field tests, which we're not involved in now. At the end of our next year, hopefully the industry would be interested in going on to that next step. I would say that the industry feels titanium tubes, as I understand, are what they want. As I understand it, the titanium tube is not highly sensitive to either corrosion or erosion. I'm hoping that we are not going to do anything to those tubes to effect durability. But that remains to be shown, and we're not going to do it in the next year. That's the reason we need the involvement of the electric utility industry.

We're only working on a part of the problem at this point. I believe that from start to finish, to actually do the work necessary to put these tubes into electric utility condensers, is probably a six to eight year job. What we're trying to accomplish in the next year is to show that the economic and performance potential is great enough to take the next step. At our workshop meeting at Penn State on October 31, we will present economic numbers on this.

Comment: In addition to the corrosion problem, one of the main reasons for utilities' going to titanium was the price advantage achieved with titanium. There would be a lot of resistance to go back to copper. Titanium is here to stay as a condensor material at sea-side plants. If we could overcome the notch sensitivity and fatigue problem that might be brought about by these enhanced surfaces, I think it would be an attractive concept. Let's compare copper-nickel to titanium. Titanium is much more expensive material. The incremental price increase required to enhance a titanium tube, I believe, is quite a bit less than a copper-nickel tube. You know the material cost is a dominant part of it, so it makes sense to enhance titanium tubes from a cost point of view.

Q: The original problem with the use of titanium tubes for steam condensers and waterside/oceanside plants was metal fatigue rather than any corrosion problem. And with these enhanced surfaces, it appears that this notch sensitivity would be your greatest problem. Do you have any comment on that? The original problem with use of titanium in condensers was failure due to fatigue, (thermal fatigue and low cycle fatigue). With the notched appearance of the tubes you might be bringing that problem back and that would be a major consideration to the non-sensitivity and fractured toughness of this material.

A: Let me try to answer that by saying first, I really don't know. I would say that we'd be willing to make our tubes out of any material you want. If you don't like titanium, tell us what you want. Our industrial partner, Wolverine, has more practical experience with this and can possibly provide a more specific answer to this question.

Comment: As explained, some of the tubes used in this program are not commercially available tubes; Penn State came up with a desirable geometry and asked us if we could make those tubes, and that's what we've done at this point, particularly with the copper. Some of those geometries we have not reproduced in titanium. They would be more difficult, of course, to reproduce in titanium. I really can't address the concern about the effect of geometry on long term fatigue of the titanium tubes. I'm only somewhat familiar with the history there and industrial refrigeration applications with the titanium tubes. But, as said before, we'll be glad to make the tubes in copper or copper-nickel or whatever material is preferred by the industry. There is a trend, particularly in the nuclear power industry to move toward staying with stainless steels and titanium condensor tubes. But we still do quite a good business in the non-ferrous condensor tubes as well.

ASSESSMENT OF STRENGTH LIMITING FLAWS IN HEAT EXCHANGER COMPONENTS

John R. Bower
Babcock & Wilcox

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGERS PROGRAM
PROJECT DESCRIPTION

TITLE: ASSESSMENT OF STRENGTH LIMITING FLAWS IN CERAMIC HEAT EXCHANGER COMPONENTS
COOPERATIVE AGREEMENT NO: DE-FC07-89ID12887, through DOE-ID
CONTRACTOR: Babcock & Wilcox
3315 Old Forest Road, PO Box 10935
Lynchburg, VA 24506

PRINCIPAL INVESTIGATOR: John R. Bower - 804-522-5742

OBJECTIVE: Develop nondestructive evaluation (NDE) and predictive modeling techniques to predict and measure the critical flaw population and subsequent flaw growth, if any, in ceramic heat exchangers under industrial operating conditions.

TECHNICAL APPROACH: Develop NDE techniques to examine materials: destructive testing with acoustic emission monitoring to determine flaw growth characteristics: and linear elastic fracture mechanics to model flaws in heat exchanger tubes.

SYSTEM DESCRIPTION: NDE:

Time of Flight Acoustic Microscopy (TOFAM) and microfocus x-ray computed tomography (CT) are being improved to provide rapid material inspections.

Destructive testing:

C-rings are being tested at high temperatures, over 2000^{o}F. Tube sections are pressure tested at room temperature. In both cases, acoustic emission is used to detect and locate crack growth. NDE is then used to attempt to determine the crack and its size.

Fracture Mechanics:

A model of the heat exchanger will be constructed, including mechanical and thermal stresses, with the information on physical properties and defects from the testing programs. Lifetime predictions will be made and tested.

CURRENT STATUS (8/89): Phase 2 is complete, Phase 3 is starting. NDE equipment improvements are underway.

ACCOMPLISHMENTS:

CT, TOFAM, and SLAM (Scanning Laser Acoustic Microscope) have been demonstrated capable of inspecting silicon carbide heat exchanger tubes.

Assessment of Strength Limiting Flaws in Heat Exchanger Components

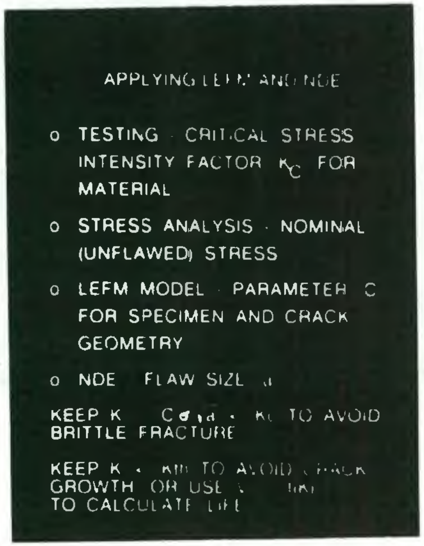
Phase III
July 1989 - November 1991

Babcock & Wilcox Co.

JOHN R. BOWER
Program Manager

Energy Efficient Lifetime

- **ACCEPTANCE CRITERIA**
- **ACCEPTANCE TESTING**
- **LIFETIME MODELING**



APPLYING LEFM AND NDE

- TESTING - CRITICAL STRESS INTENSITY FACTOR K_C FOR MATERIAL
- STRESS ANALYSIS - NOMINAL (UNFLAWED) STRESS
- LEFM MODEL - PARAMETER C FOR SPECIMEN AND CRACK GEOMETRY
- NDE - FLAW SIZE a

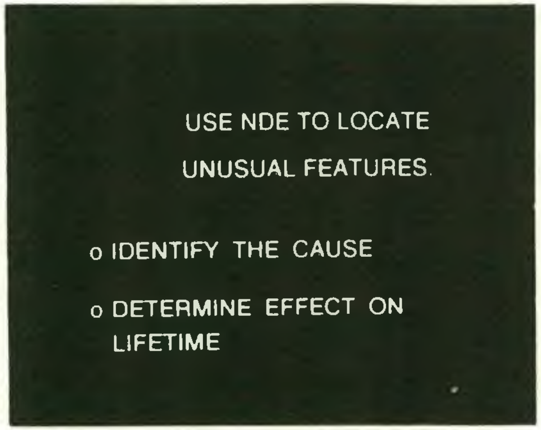
KEEP $K = C\sigma\sqrt{a} < K_C$ TO AVOID BRITTLE FRACTURE

KEEP $K < K_{TH}$ TO AVOID CRACK GROWTH OR USE $V = f(K)$ TO CALCULATE LIFE

SPECIMEN FAILURE

- IF $K > K_C$ THEN BRITTLE FRACTURE OCCURS
- IF $K < K_C$ BUT GREATER THAN A THRESHOLD VALUE K_{TH} THEN CRACK GROWTH WILL OCCUR AT SOME VELOCITY V UNTIL IT IS BIG ENOUGH TO CAUSE BRITTLE FRACTURE

FROM LABORATORY TESTING
$V = f(K)$

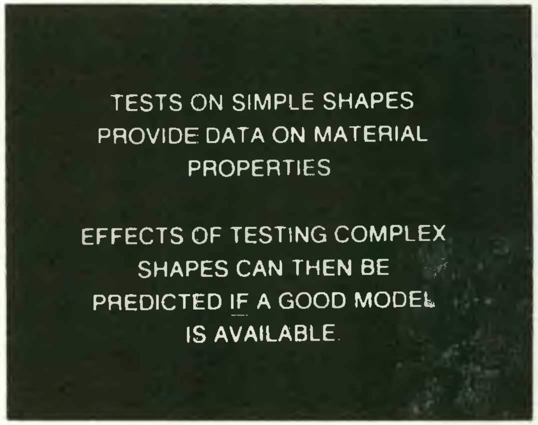


USE NDE TO LOCATE UNUSUAL FEATURES.

- o IDENTIFY THE CAUSE
- o DETERMINE EFFECT ON LIFETIME

Phase I

- o Microfocus X–ray, SLAM, ultrasonics are useful
- o Material properties limit testing
- o Various seeded defects used



TESTS ON SIMPLE SHAPES PROVIDE DATA ON MATERIAL PROPERTIES

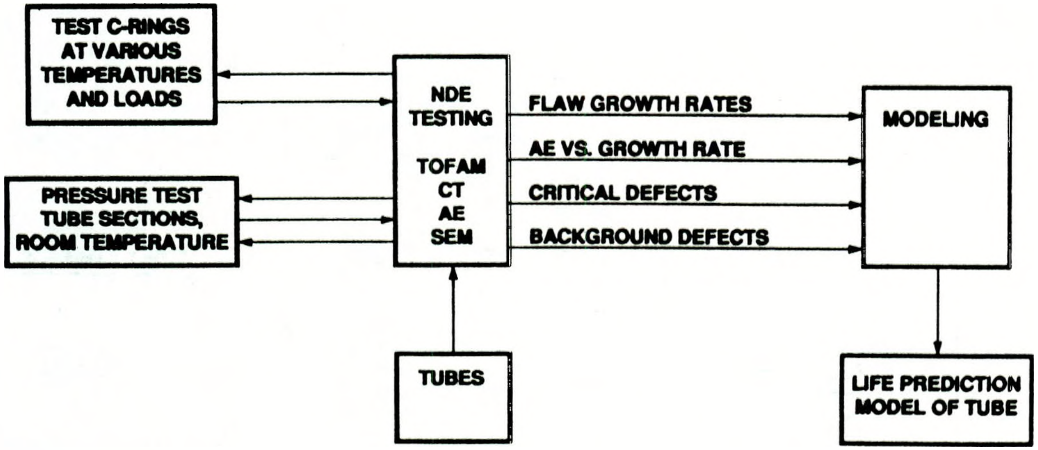
EFFECTS OF TESTING COMPLEX SHAPES CAN THEN BE PREDICTED <u>IF</u> A GOOD MODEL IS AVAILABLE.

Phase II

- o TOFAM and CT scanning added
- o Material properties measured
- o Tube materials used in testing

Assessment of Strength Limiting Flaws in Ceramic Heat Exchanger Components

Phase III Program



Task 1: **IMPROVE EQUIPMENT**

Task 2: **TEST FLAW BEHAVIOR**

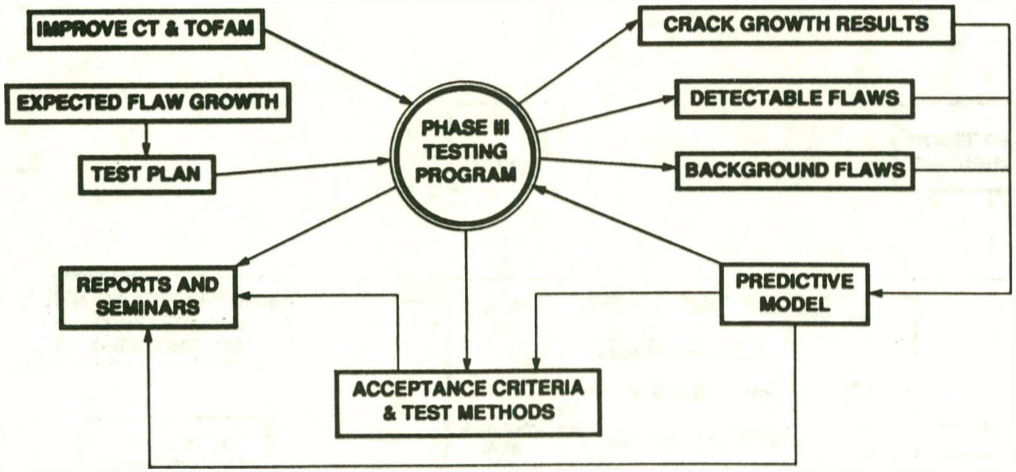
Task 3: **MODEL TUBES/FLAWS**

Task 4: **PREDICT AND VERIFY**

Task 5: **REPORTING**

Assessment of Strength Limiting Flaws in Ceramic Heat Exchanger Components

Phase III Activities



IMPROVE CT & TOFAM

EXPECTED FLAW GROWTH

TEST PLAN

PHASE III TESTING PROGRAM

CRACK GROWTH RESULTS

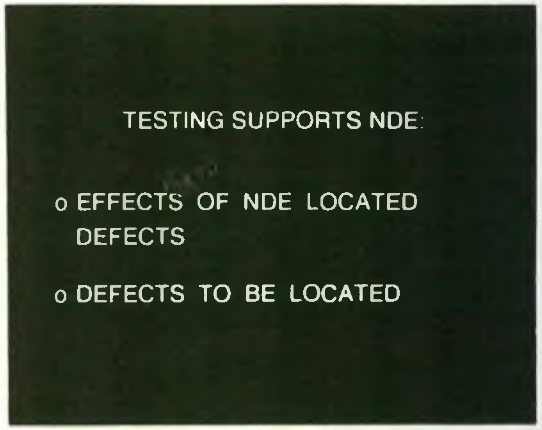
DETECTABLE FLAWS

BACKGROUND FLAWS

PREDICTIVE MODEL

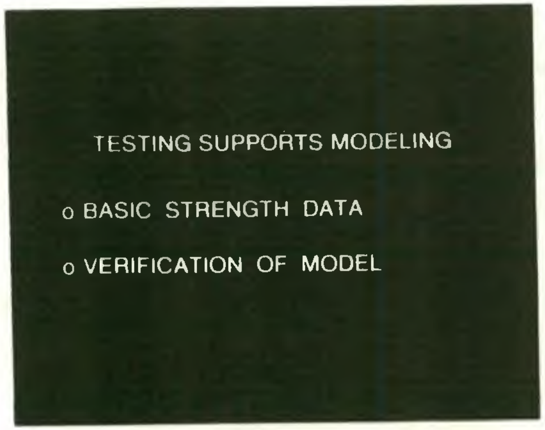
REPORTS AND SEMINARS

ACCEPTANCE CRITERIA & TEST METHODS



TESTING SUPPORTS NDE:

- o EFFECTS OF NDE LOCATED DEFECTS
- o DEFECTS TO BE LOCATED

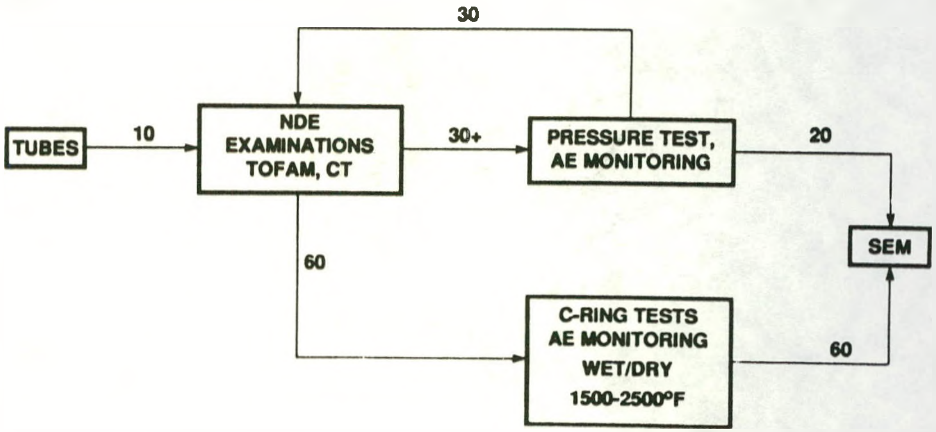


TESTING SUPPORTS MODELING

- o BASIC STRENGTH DATA
- o VERIFICATION OF MODEL

Assessment of Strength Limiting Flaws in Ceramic Heat Exchanger Components

Phase III Testing



TUBES

10

NDE EXAMINATIONS TOFAM, CT

30

30+

PRESSURE TEST, AE MONITORING

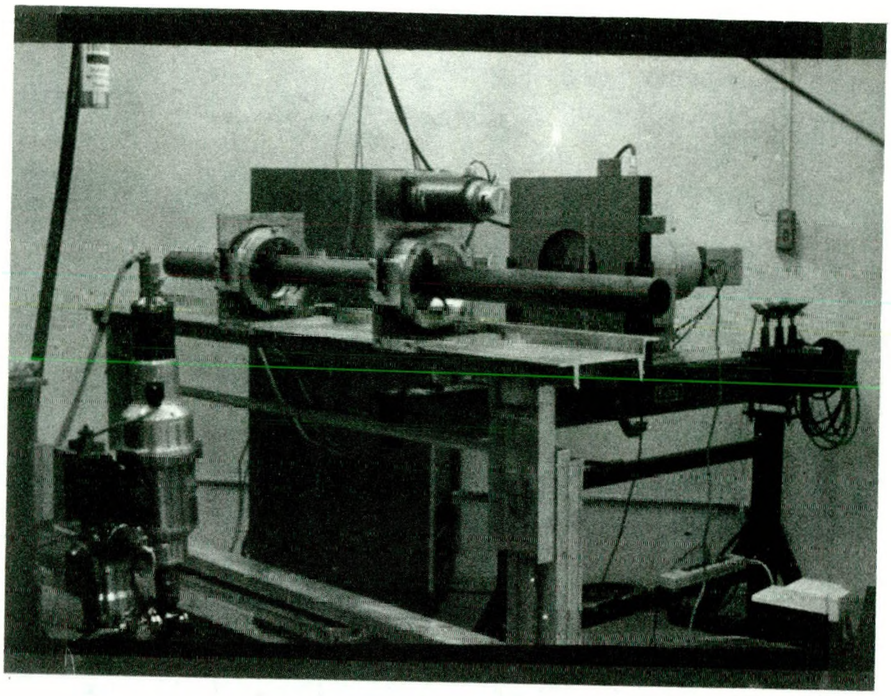
20

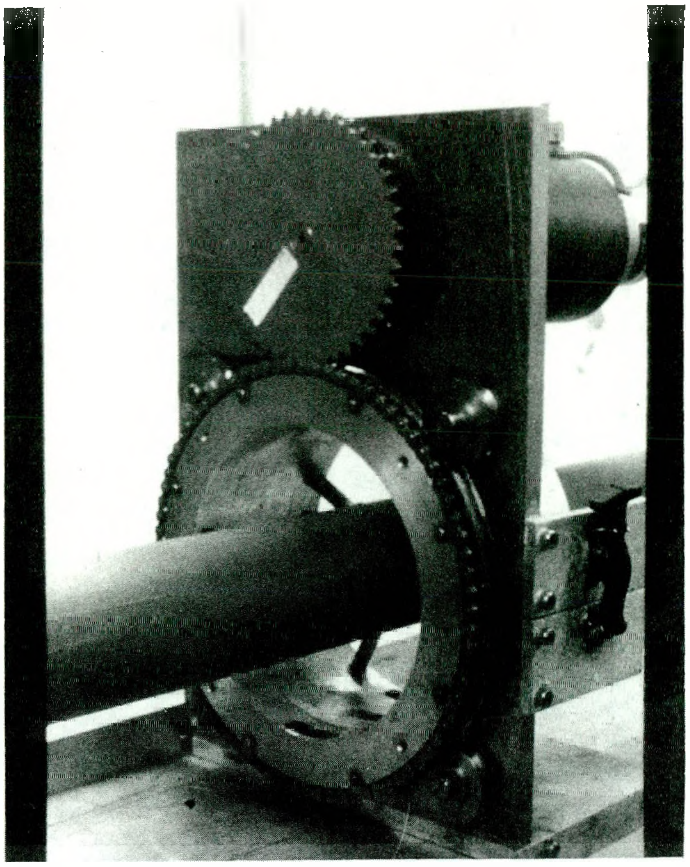
SEM

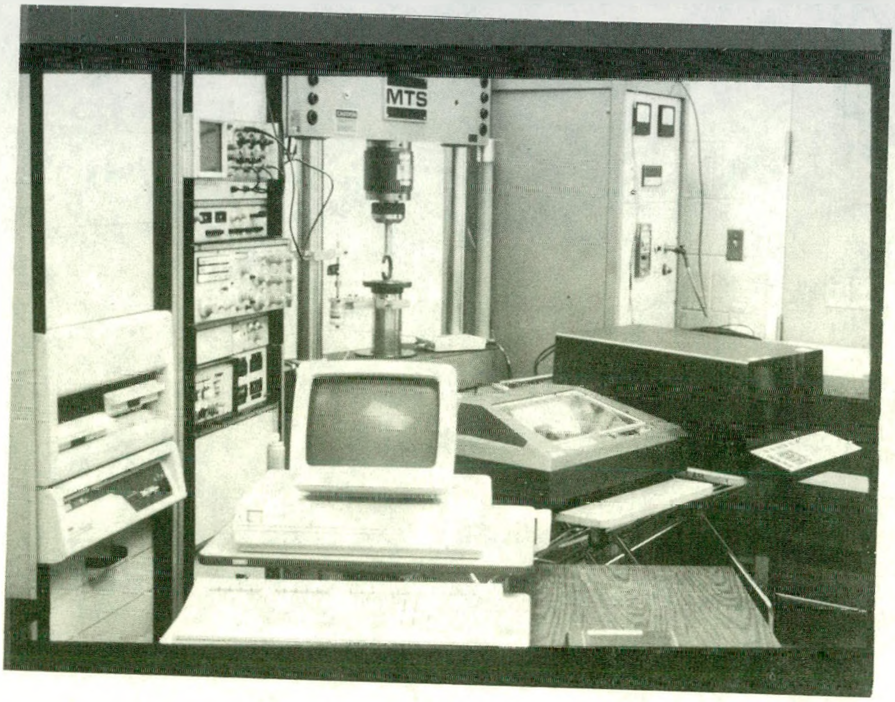
60

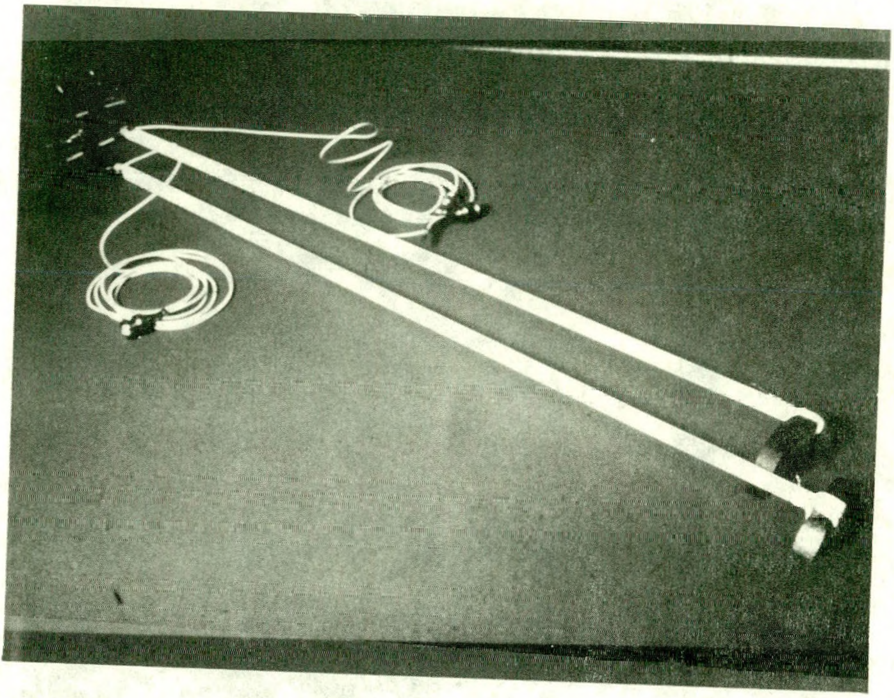
C-RING TESTS AE MONITORING WET/DRY 1500-2500°F

60

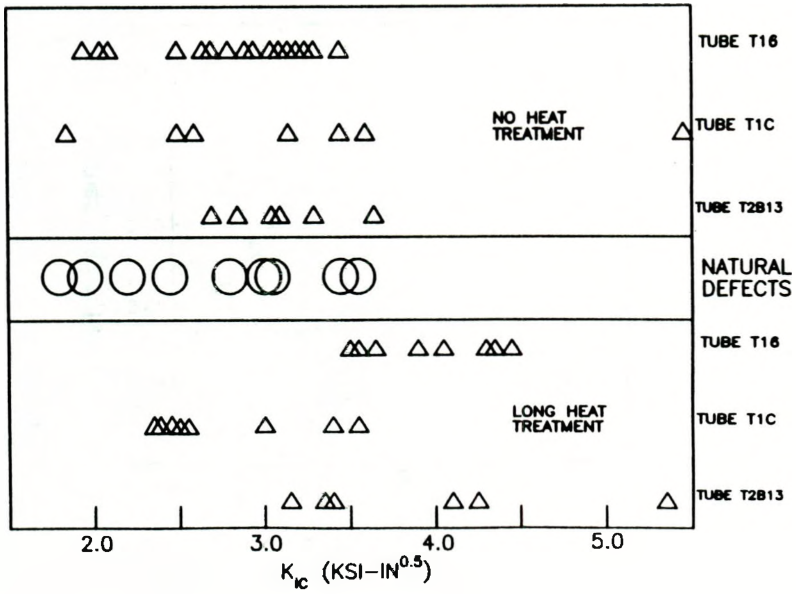




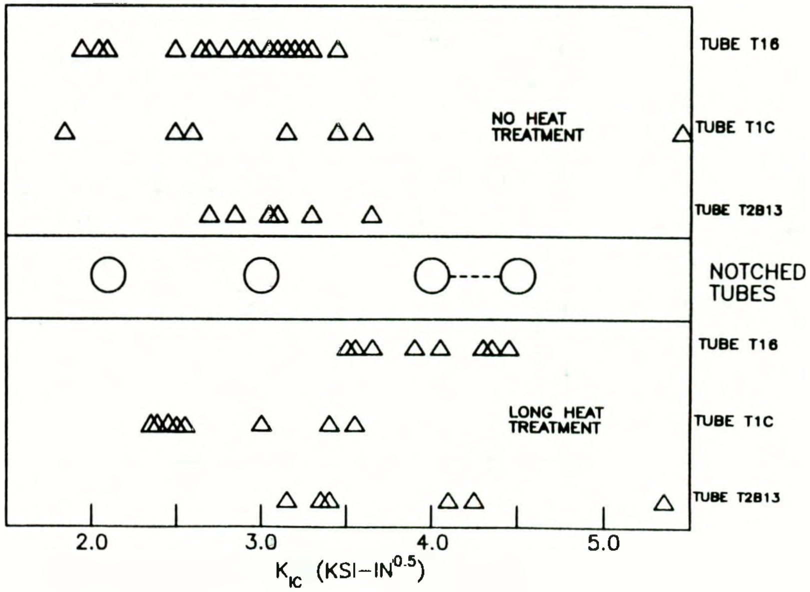

MTS



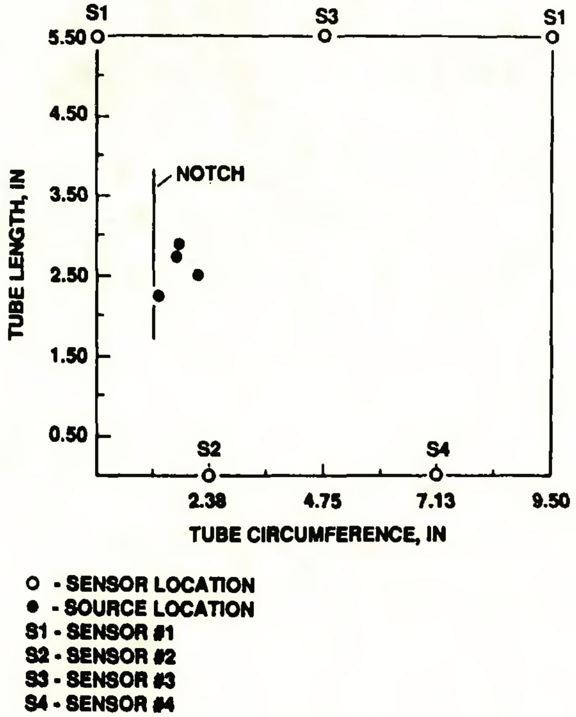
SUMMARY OF K_{IC} RESULTS



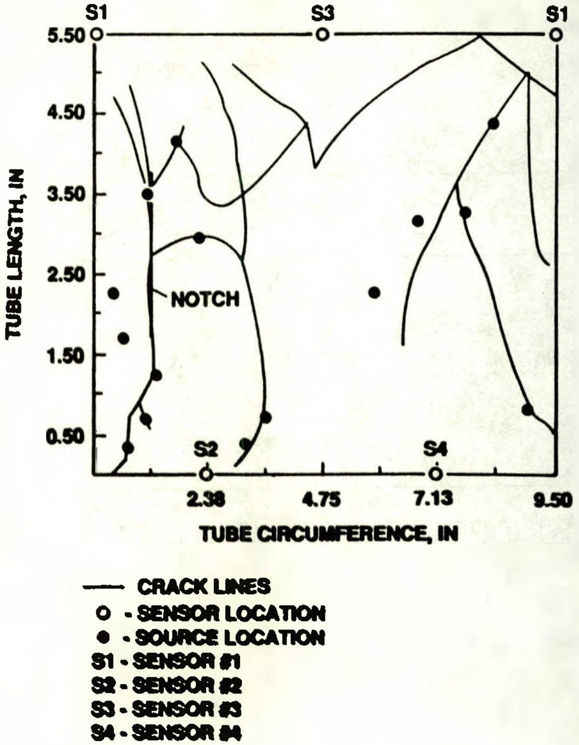
SUMMARY OF K_{IC} RESULTS



Source Location Plot, Notched Sample T1A17

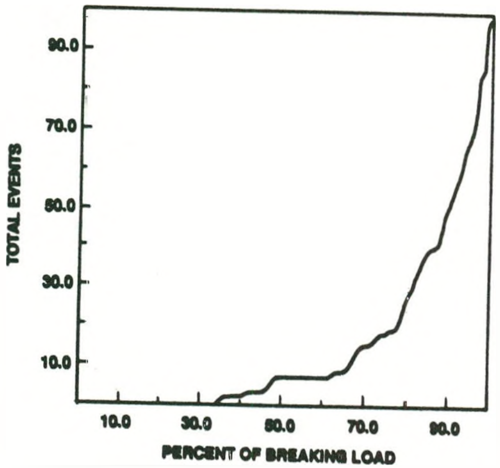


Source Location Plot, Notched Sample T15-2



Cumulative AE Events as a Function of Percentage of Failure Load for All 18 C-Ring Specimens

C-Ring History Plot



EASE OF TESTING

FLAT BARS

C-RINGS

TUBE SECTIONS

REALISM IN TESTING

MOVE TESTING TOWARD SERVICE CONDITIONS FOR MODEL VERIFICATION

GENERALIZE MODEL FOR VARYING SERVICE CONDITIONS AND VARYING MATERIAL CONDITIONS

IMPROVE NDE FOR DEFECT AND MATERIAL EVALUATION. LOCATE MORE CONDITIONS TO TEST

ESTABLISH ACCEPTANCE CRITERIA AND ACCEPTANCE TESTS

CRITERIA	TESTS
WALL THICKNESS	ULTRASONICS
OUTER SURFACE CRACKS	SLAM
INNER SURFACE CRACKS	TOFAM
INCLUSIONS, POROSITY	TOFAM, X-RAY

IN SILICONIZED SILICON CARBIDE, POROSITY IS EXTENSIVE.

PORES DO NOT CAUSE FAILURE.

DISCUSSION

Assessment of Strength Limiting Flaws in Heat Exchanger Components

John R. Bower

Q: Are you looking for other tube materials to test in this program or are you just going to stick with the Norton siliconized silicon carbide?

A: Our largest emphasis is on trying to get testing techniques that are usable. So I think for this program we'll stick with what we've got. We have seen some other materials, and realize there are variations. For the moment, to get a fracture mechanics model, I would assume that probably only some material properties would change, maybe not too significantly. K_{IC} values might be different. For the testing, surface condition is a primary limitation and I think we can get a good feeling for that with the material we're using. So rather than confuse the issues too much, we will just stay with one material. However, if you have samples you'd like to volunteer, we'll be happy to take a look at them.

Q: What flaw size can you detect with your Tofam and with CT?

A: The 250 micron deep EDM notch was detected with the Tofam. We have detection in a sense that the notch shows up above the background of normal material flaws. It can be set to be much more sensitive, but then the entire background fills in because the material has 50 micron grains and it has porosity of the same kind of size or larger. We pick up all the grain boundaries and porosities and can't separate anything, so the material property is limiting for this test. Tofam should be capable of getting much smaller defects. It has a focused beam running about 20 megahertz, and when run up to 50 or 100 megahertz, can detect very small spot sizes.

The CT scan resolution should be one or several pixels, and is limited by how many pixels you have in your image. In this case we have a three inch OD tube and about 500 pixels in each line of the image, so, that sets the resolution limit. The microfocus x-ray has a spot size of 2 to 5 microns, and if our rotation mechanism were stable enough we could presumably get down to that resolution on a small enough sample. The problem with CT-scan is that, for the reconstruction algorithms to work nicely you have to rotate the entire object in the beam, and then you're limited by the resolution of your image.

Q: I understand that you need a variety of equipment to detect various types of flaws, and that background really interferes with some of the critical flaws. Is it going to be practical for the large-scale user to use any of these techniques in the near future, for example, as a primary means of detecting critical flaws that would lead to rejection of tubes versus a proof testing, which is reasonably fast?

A: For the immediate future, proof testing is probably the best method that's available right now. Some kind of ultrasonic scanning like the Tofam system, when it is adapted to find flaws in any orientation on the inside or outside surface, could also go relatively quickly. In any case, it will be necessary to set some kind of a threshold that is above the natural material variations and reject anything with bigger indications than that. Then, find out what that threshold means in terms of the size crack you're ignoring, and determine statistically what your probability is for acceptance of that flaw. That is the direction we're going.

Q: So, for example, this Tofam technique could be used to reject any tubes that have any gross flaws, sort of as a preliminary screening prior to proof testing?

A: You could do it prior to proof testing, although I was thinking of it more as a substitute for proof testing. At this stage we're trying to develop the techniques, and have not looked into the economics of which way to do the testing.

Comment: The development of the Tofam and CT scan technologies is primarily as a research tool to provide us with a way of studying ceramic flaw populations so that we can develop acceptance criteria for ceramic heat exchanger components. Once we can define the acceptance criteria, we can then decide which type of NDE or proof test method best serves the purpose of QA in industry.

INEL SUPPORT FOR ASSESSMENT OF STRENGTH LIMITING FLAWS IN CERAMIC HEAT EXCHANGER COMPONENTS

Randy Lloyd
INEL

U. S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGERS PROGRAM
PROJECT DESCRIPTION

TITLE: **ASSESSMENT OF STRENGTH LIMITING FLAWS IN CERAMIC HEAT EXCHANGER COMPONENTS**
COOPERATIVE AGREEMENT NUMBER: DE-AC07-76ID01570 1HA110
CONTRACTOR: Idaho National Engineering Laboratory/
EG&G Idaho, Inc.
P. O. Box 1625
MS 2218
Idaho Falls, ID 83415

PRINCIPAL INVESTIGATORS: W. G. Reuter, 208-526-1708
D. C. Kunerth, 208-526-0102

OBJECTIVE: Verify applicability and develop capability to make minimum service lifetime predictions on structural ceramic heat exchanger components; establish material selection and acceptance criteria based on nondestructive examination techniques.

TECHNICAL APPROACH:

Fracture Mechanics - measure selected material's (Norton CS101K SiC) fracture toughness (K_{Ic}) using standard single edge notch bend [SE(B)] and arc chord support [A(B)] specimens and special (C-ring and chevron notch) specimens. Assess linear elastic fracture mechanics (LEFM) applicability to real structures by testing surface cracked specimens (rectangular specimens in bending and tube specimens by internal pressurization).

NDE - a Time-of-Flight Acoustic Microscopy (TOFAM) system is developed to examine both actual structural elements and test specimens, both before service or testing and after testing and delivered to B&W. The system can provide a defect "map" of the component or specimen. Appropriate technical advise regarding use of the system is also provided.

APPLICATION: Knowledge of the critical flaw size, provided by fracture mechanics, can be used to establish accept/reject criteria for NDE. NDE techniques can then be used to select components with the specified high reliability of performing their design function for the system's lifetime. Fracture mechanics concepts, including knowledge of significant environmental effects, combined with refined NDE techniques can be used to provide both more efficient and possibly lower cost heat exchanger components. The design/assessment model will also define the required material property data required for completion of a specific design.

CURRENT STATUS (08/89): Evaluation and testing of several fracture toughness test specimen geometries have been completed. SE(B) and C-ring specimens have been selected as the most appropriate for fracture toughness and environmental effect evaluation. The three-point-bend surface crack [SC(B)] specimen will be used for verification of applicability of LEFM-type data to the structural components; limited testing of tube segments may also be used for the same purpose. Use of acoustic emission monitoring is still being CURRENT STATUS

(08/89) (cont):
developed as an auxiliary measurement technique to assist in the establishment of threshold stress intensity factors.
The TOFAM system supplied to B&W is operational and in use to evaluate and characterize surface defects on the inside surface of tube segments.

ACCOMPLISHMENTS: It has been determined that LEFM may be used to predict structural integrity of the SiC components. K_{Ic} has been established for the SiC material at both 20°C and 1250°C. Effects of a 1250°C air environment have been examined in a limited effort. Unexpected elevation in fracture toughness was observed with extended exposure to high temperature and applied stress (together), as well as possible strain rate effects.

The TOFAM system supplied to B&W has been successfully used to detect surface-connected defects on the inside of tube segments. Enhancements to the system which will allow detection of a wider range of defect geometries have been identified.

FUTURE WORK: More extensive evaluation of environmental effects will be pursued. Both modifications to the AE system and development of a high temperature moire interferometry (HTMI) system will significantly improve the ability to assess environmental effects. An attempt to determine the failure mechanism associated with the high temperature oxidizing environment will be made; the HTMI system will be the focal point of this effort.

Improvements in the TOFAM system including data communications with other computer systems and outer surface flaw detection will be addressed.

CONCLUSIONS:

A design model for siliconized SiC (Norton CS101K) heat exchanger components, based on concepts of LEFM, has been developed and verified for a wide temperature range. To complete the service life assessment portion of the model, further studies into the subcritical crack growth mechanism of the material will be required; additional tests to accurately determine the threshold stress intensity factor (K_{th}) as a function of environment will also be needed.

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2. Reuter, W. G., Lloyd, W. R., Knibloe, J. R., and Storhok, E., Assessment of Strength Limiting Flaws in Ceramic Heat Exchanger Tubes, Phase II, EG & G Internal Report (currently being printed), January 1989.

SUPPORT FOR ASSESSMENT OF STRENGTH LIMITING FLAWS IN CERAMIC HEAT EXCHANGER COMPONENTS

PRESENTATION OUTLINE

I. A. Project Description

1. Provide experimental and analytical support to Babcock & Wilcox in their effort to develop characterization techniques and specification/acceptance criteria for ceramic heat exchanger materials.

2. Specifics include - fracture toughness (K_{Ic}) measurement, threshold stress intensity factor (K_{th}) determination, and assessment of environmental effects on defect behavior to specify a maximum allowable defect size. See Figure 1.

3. Provide improved nondestructive examination (NDE) capabilities for preservice flaw detection. See Figure 2.

I. B. History and Justification

It is critical to have the ability to predict the minimum service life of ceramic heat exchanger components and systems in order to have acceptance of these systems by industry. See Figure 3.

I. C. Goals

1. Reliably detect and characterize flaws (defects) in components using suitable NDE techniques.

2. Evaluate the applicability of using linear elastic fracture mechanics (LEFM) concepts to predict flaw growth and failure conditions in ceramic heat exchanger components subjected to high temperature, oxidizing environments. See Figure 4.

3. Develop a general model, based on LEFM, to assess a component's minimum service lifetime as a function of existing flaw size as well as providing procurement specifications and accept/reject criteria for new components. See Figure 5.

II. A. Results and Major Achievements

1. K_{Ic} can be measured for the SiC material (Norton CS101K, siliconized SiC with 15% free silicon metal) investigated and does not vary significantly from one component to the next fabricated from one batch of material, based on four separate tube sections tested at the Idaho National Engineering Laboratory (INEL).

2. Being very difficult to produce naturally sharp, straight front cracks in the SiC test specimens, electric discharge machined (EDM) notches were used. The effect on K measurements using EDM notched specimens versus sharp cracks has been quantified. See Figure 6.

3. Analytical solutions for stress intensity (K) as a function of applied load and crack length have been verified for the C-ring specimen (it is the most economical to fabricate from tube-shaped materials and is easy to test at high temperatures). See Figure 7.

4. Testing at 1250°C indicates that no environment-induced crack growth occurs for up to 900 hrs. at $K = 0.95\ K_{Ic}$. See Figure 8.

5. K_{Ic} (at both 21°C and 1250°C) is found to increase after exposure to high temperatures while subjected to various levels of applied K less than the untreated K_{Ic} value. See Figure 8.

6. Surface crack [SC(B)] specimens, simulating actual components containing defects, were tested to determine the maximum K value (K_{crit}) for flaw dimensions ranging from smaller than to much larger than calculated critical size. The results of these tests shows $K_{crit} > K_{Ic}$ for all flaw geometries evaluated, indicating LEFM can be applied with slightly conservative results. See Figures 9 and 10.

7. A time-of-flight acoustic microscopy (TOFAM) system has been successfully implemented to detect surface-connected cracks (these are the most critical type) on the inside surface of tube segments. See Figure 11.

II. B. Summary and Conclusions of Research

1. The ability to measure and to use K_{Ic} in an applicable equation are imperative to the application of LEFM concepts; both have been accomplished for the siliconized SiC material being studied.

2. The ability to reliably detect flaws (cracks), of a size determined by the use of LEFM, during preservice NDE inspection is required for the application of the proposed model. The TOFAM system can do this for flaws on the interior surface of tube segments (the most critical location and most difficult to inspect).

3. Preliminary studies indicate that no measurable crack growth occurs during 900 hrs. of exposure to an air environment at 1250°C with an applied K level at 95% of K_{Ic}. This indicates $K_{th} \approx K_{Ic}$, however, lack of knowledge of crack growth mechanisms in this environment does not allow significant extrapolation of the data past 900 hrs. for lifetime predictions.

4. A preliminary model for lifetime prediction has been developed. Certain areas within the model regarding environmental effects need more work before the model will be applicable for long term life prediction. See Figure 5.

II. C. Future Activities

1. Proceed with additional investigation of environmental effects on subcritical crack extension, with an aim to identify the specific mechanism by which it occurs in the environment(s) of interest. A high temperature moire interferometry (HTMI) system capable of sub-micron, full field surface displacement measurements at temperatures in excess of 1000°C will be developed to support this work.

2. Further work to provide more complete knowledge of the effects of sharp cracks versus EDM notches, particularly with respect to environmental effects, is planned.

3. Improvements to the TOFAM system's ability to communicate with other computer systems and in its user interface for easier operation is planned.

III. Review

A. An experimentally-based research program was implemented to evaluate the general applicability of using LEFM concepts to make service lifetime predictions of ceramic heat exchanger components.

   1. Fracture toughness, K_{Ic}, and environmental effects, K_{th}, were evaluated.

   2. The ability of LEFM to predict flaw behavior in the SiC material was investigated by comparing results of "standard" tests to results of tests on specimens which simulate actual structural components. See Figure 9.

   3. Capabilities of detecting defects of critical dimensions using NDE techniques were developed and verified.

B. In general, all goals of the project were achieved, culminating in the successful development of a LEFM-based model for prediction of structural integrity and service lifetime.

   1. The C-ring specimen was found to be economical to fabricate, easy to test, and able to produce reliable data.

   2. Limiting values of K_{Ic} (21°C and 1250°C) have been established for the SiC material used in the program.

   3. Provisional values of K_{th} (equal to K_{Ic}) have been measured, subject to verification through (a) additional testing of specimens with sharp cracks and (b) identification of the mechanism of subcritical crack growth for the SiC material in the environment(s) of interest.

   4. Applicability of using LEFM concepts for modeling the flaw growth behavior of the SiC material has been verified by comparing results of tests of "standard" specimens with results of tests on specimens simulating expected flaws in actual structural components.

   5. The above information has been incorporated into a general model of flaw behavior in the SiC material studied. See Figure 5.

   6. The TOFAM system has been successfully employed to detect critical size (from fracture mechanics analysis) defects in actual components.

SUMMARY OF INEL'S SUPPORT FOR ASSESSMENT OF STRENGTH LIMITING FLAWS IN CERAMIC HEAT EXCHANGER COMPONENTS

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ADVANCED HEAT EXCHANGERS PROGRAM REVIEW

11 - 12 October 1989
Ramada Renaissance
Herndon, VA

GENERIC TUBE SEGMENT SHOWING VARIOUS SPECIMEN TYPES, DEFECT ORIENTATIONS (IN SQUARE BRACKETS), AND METHOD OF LOAD APPLICATION

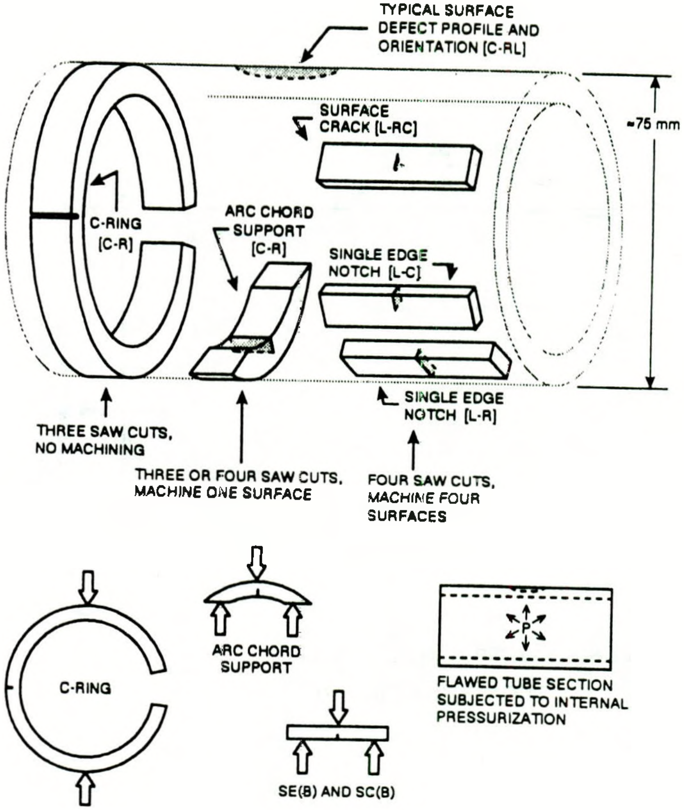


Figure 1

CROSS-SECTION OF COMPONENT SHOWING TOFAM TRANSDUCER SCANNING A POSITION WITHOUT A DEFECT (1) AND WITH A DEFECT (2)

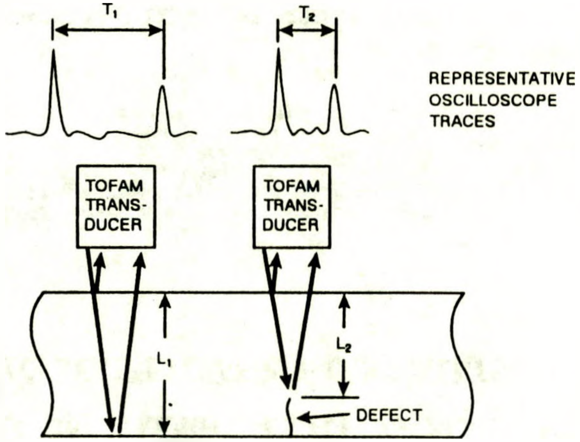


Figure 2

THE ULTIMATE GOAL

ACCURATE PREDICTION OF SERVICE LIFETIME

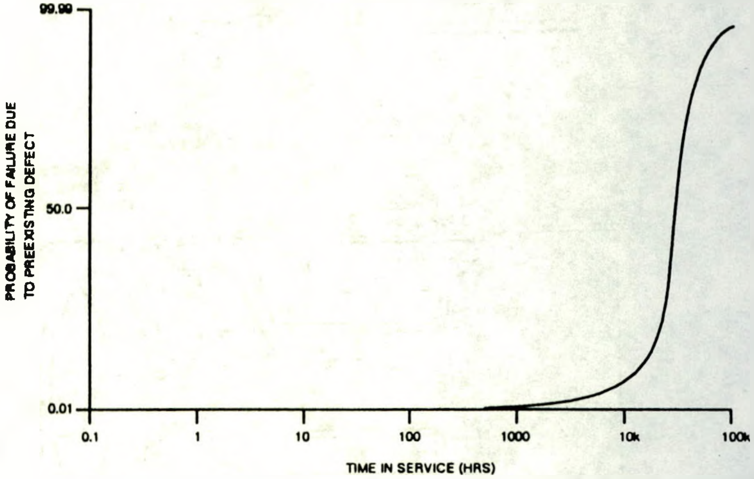


Figure 3

SCHEMATIC DIAGRAM ILLUSTRATING BASIC FRACTURE BEHAVIOR MODELING CONCEPT

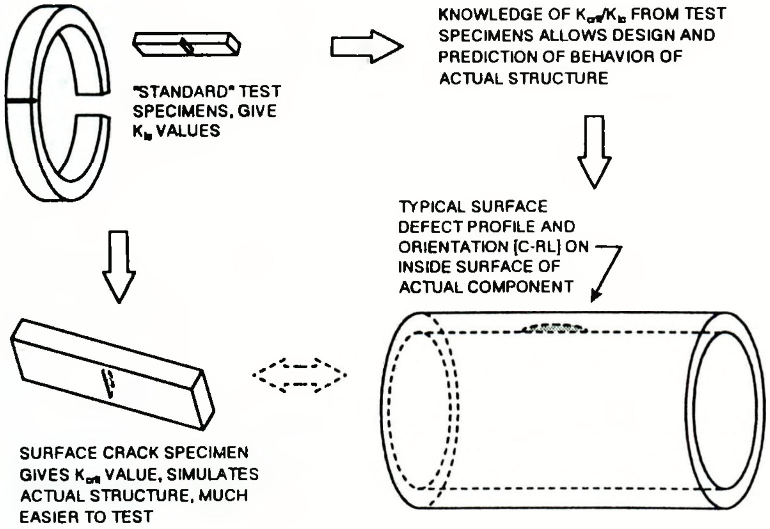


Figure 4

SCHEMATIC DIAGRAM OF DESIGN/ASSESSMENT MODEL BASED ON FRACTURE MECHANICS CONCEPTS

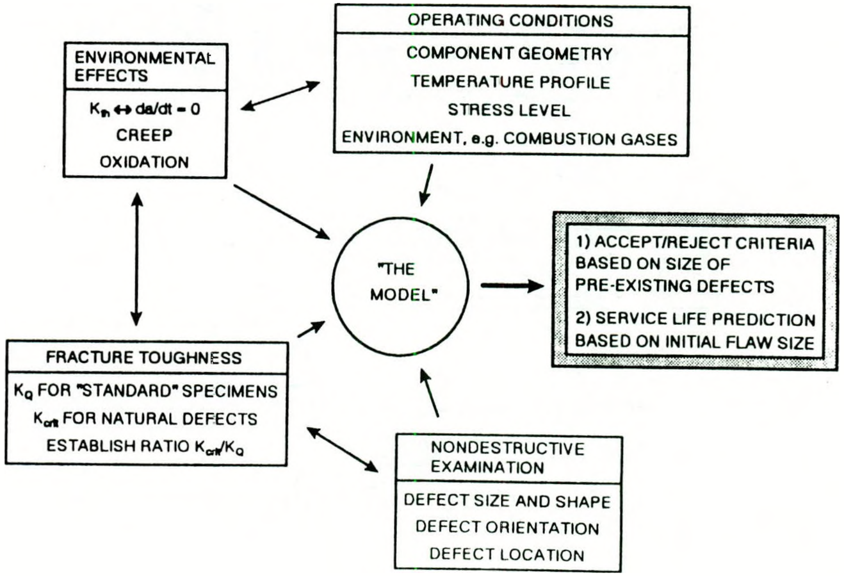


Figure 5

FRACTURE TOUGHNESS VS. NOTCH ROOT RADIUS

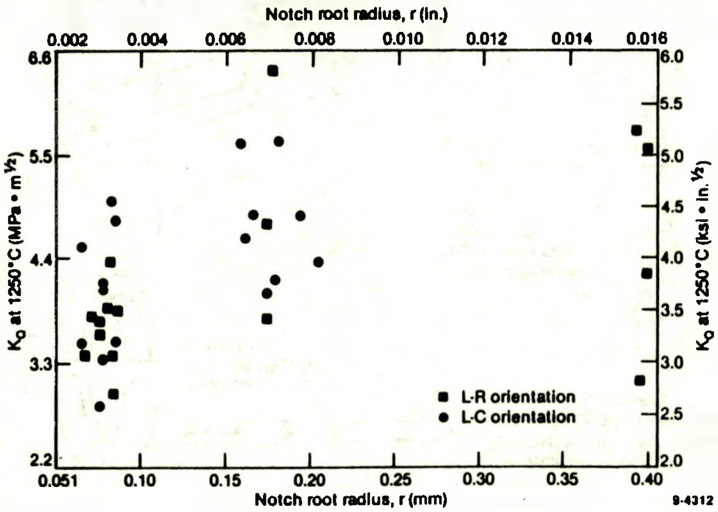
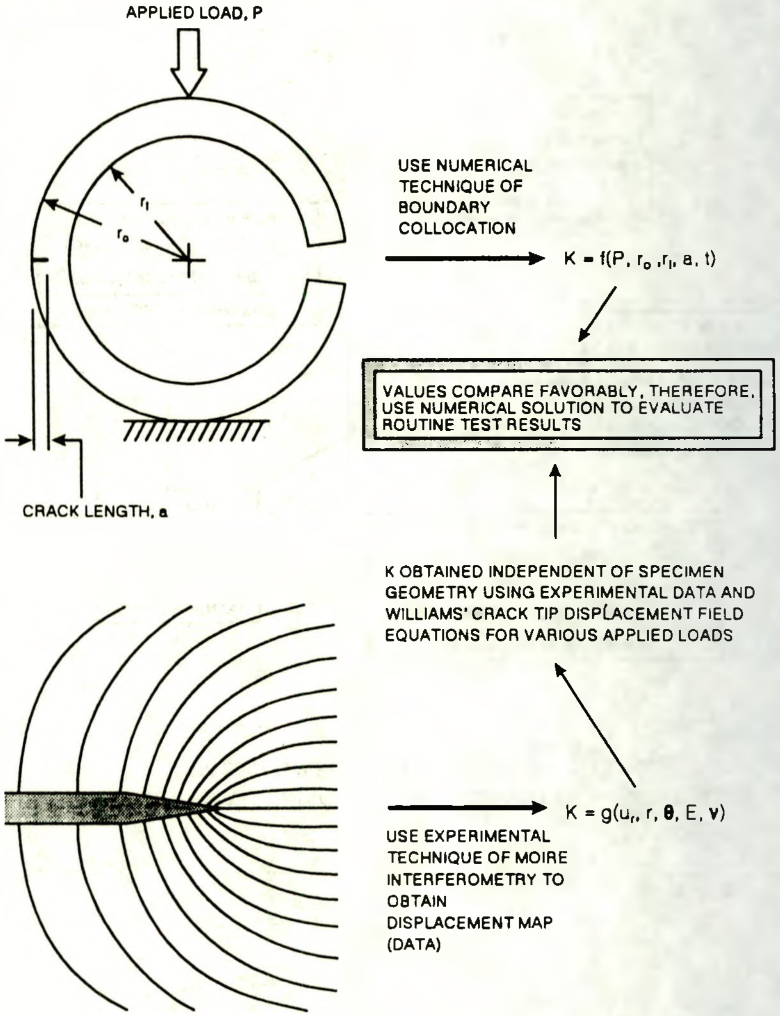


Figure 6

EXPERIMENTAL VERIFICATION OF NUMERICAL SOLUTION FOR C-RING STRESS INTENSITY FACTOR



USE NUMERICAL TECHNIQUE OF BOUNDARY COLLOCATION

$K = f(P, r_o, r_i, a, t)$

VALUES COMPARE FAVORABLY, THEREFORE, USE NUMERICAL SOLUTION TO EVALUATE ROUTINE TEST RESULTS

K OBTAINED INDEPENDENT OF SPECIMEN GEOMETRY USING EXPERIMENTAL DATA AND WILLIAMS' CRACK TIP DISPLACEMENT FIELD EQUATIONS FOR VARIOUS APPLIED LOADS

$K = g(u_r, r, \theta, E, \nu)$

USE EXPERIMENTAL TECHNIQUE OF MOIRE INTERFEROMETRY TO OBTAIN DISPLACEMENT MAP (DATA)

Figure 7

CRING_1.DRW
W. R. Lloyd 10 Sept 89

FRACTURE TOUGHNESS VS. TIME AT 1250°C

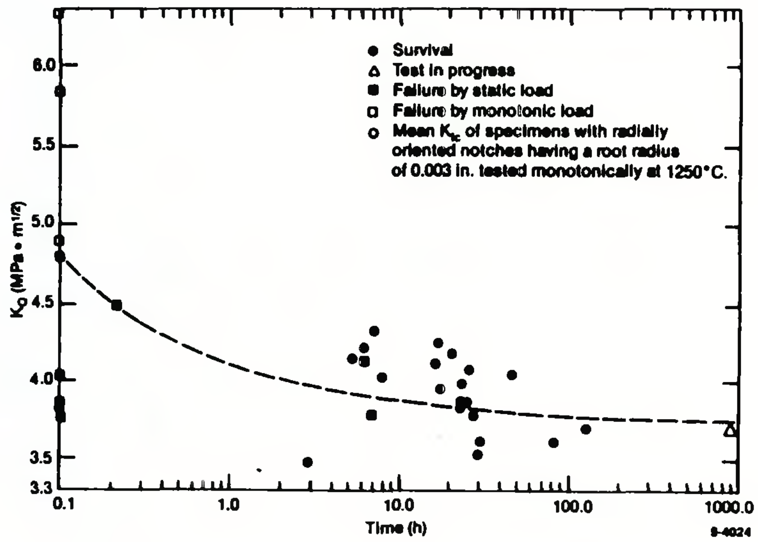
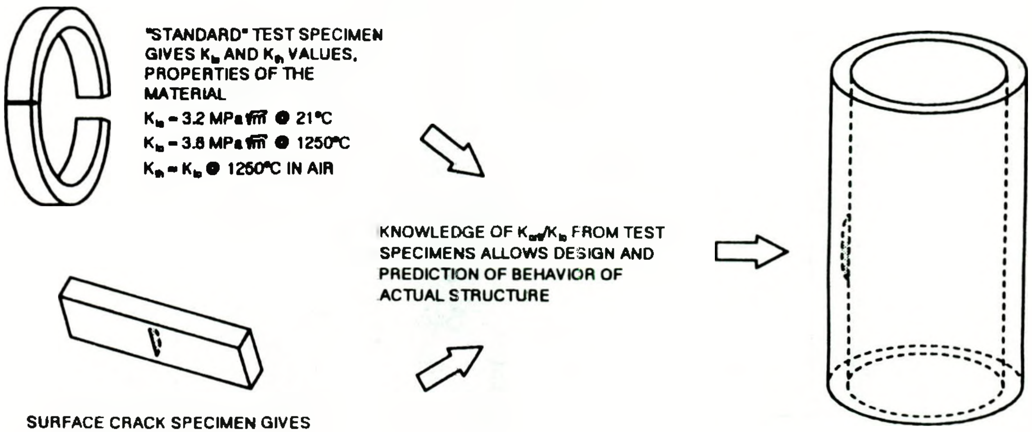


Figure 8

FRACTURE MECHANICS APPROACH TO PREDICTION OF STRUCTURAL INTEGRITY



"STANDARD" TEST SPECIMEN GIVES K_{Ic} AND K_{th} VALUES, PROPERTIES OF THE MATERIAL

K_{Ic} = 3.2 MPa$\sqrt{m}$ @ 21°C

K_{Ic} = 3.8 MPa$\sqrt{m}$ @ 1250°C

K_{th} = K_{Ic} @ 1250°C IN AIR

KNOWLEDGE OF K_{crit}/K_{Ic} FROM TEST SPECIMENS ALLOWS DESIGN AND PREDICTION OF BEHAVIOR OF ACTUAL STRUCTURE

SURFACE CRACK SPECIMEN GIVES K_{crit} VALUE, WHICH IS DEPENDENT ON GEOMETRY AND MODE OF LOAD APPLICATION - SIMULATES ACTUAL STRUCTURE, MUCH EASIER TO TEST

Figure 9

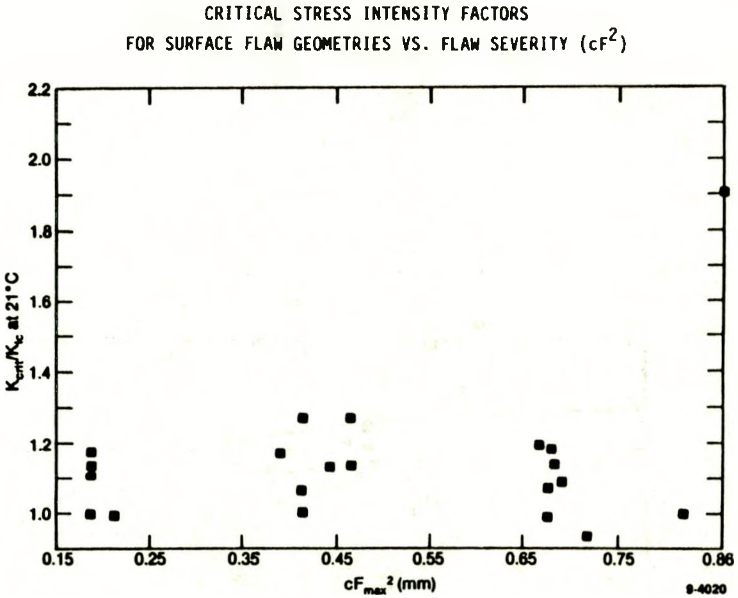
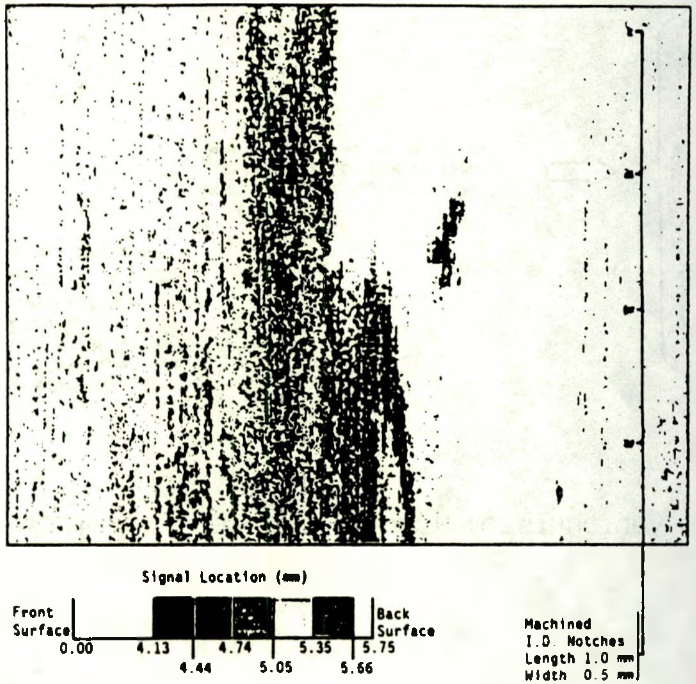


Figure 10

This is a poor black and white reproduction of a color image.



Time-of-Flight Scan
- Test Piece:
  - Slip cast silicon carbide tube (89.0 mm O.D. by 77.5 mm I.D.)
  - Known flaws - Variable depth I.D. notches (depth of notches from top to bottom 0.2, 0.4, 0.8, 1.0 mm)
- Color Coding:
  - Range - 52 ns corresponding to 0.31 mm of material thickness for each color
  - Exceptions - Yellow represents loss of back surface reflection resulting from scatter of acoustic wave
- Horizontal Axis:
  - Total circumference of tube - 279.6 mm
  - Step size - 0.79 degrees
- Vertical Axis:
  - Axis of tube - 50.0 mm
  - Step size - 0.159 mm

Figure 11

DISCUSSION

INEL Support for Assessment of Strength Limiting Flaws in Ceramic Heat Exchanger Components

Randy Lloyd

Q: Are linear elastic fracture mechanics (LEFM) concepts applicable for toughness assessment of fiber reinforced ceramic composites?"

A: The question, as it applies to organic matrix composites (e.g., graphite/epoxy systems) has been extensively investigated in recent years, with mixed results. The results indicate that the validity of applying LEFM to composites is strongly related to the local mechanism(s) of failure. As an example, local matrix cracking in a low fiber-volume fraction composite has been characterized using LEFM concepts. In other cases, the "toughness" data is strongly dependent on crack length and orientation. It is very likely that these trends will carry over to fiber reinforced ceramics. An important point to keep in mind is that LEFM concepts are based on homogeneous, isotropic material constitutive relations. The smaller the fiber size and the more isotropic the material response, the more likely it will be that LEFM concepts can be applied. Other factors influencing fracture, such as the fiber-matrix interface and associated fiber pull-out, have not been investigated enough to make any qualified statement about their effects. Work is being planned for the coming year at the INEL to begin studies of fracture in continuous fiber ceramic composites; preliminary results should be available early in 1991.

FOULING PROBE OVERVIEW

Gary R. Peterson
DOE-ID

Fouling Probe Overview

Gary R. Peterson, P.E
Project Manager
U.S. Department of Energy
Idaho Operations Office
Idaho Falls, Idaho

Fouling Costs U.S. Industry 2.9 Quads Per Year ($9 Billion at $3/MMBTU)

Research into HX Fouling Can Produce Significant National Energy Savings

---

Fouling Occurs By:

1. Scaling
2. Particulate Fouling
3. Chemical Reaction Fouling
4. Corrosion Fouling
5. Biofouling
6. Freezing Fouling

Fouling is Driven to the HX Surface By:

1. Particle Diffusion
2. Diffusiophoresis
3. Thermophoresis
4. Electrophoresis
5. Inertial Impaction
6. Ionic Diffusion
7. Sedimentation

DIAGNOSTIC TESTS IN A FLUE GAS STREAM - FOULING PROBE

Manohar Sohal
INEL

U.S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGERS PROGRAM
PROJECT DESCRIPTION

TITLE: DIAGNOSTIC TESTS IN A FLUE GAS STREAM - FOULING PROBE

COOPERATIVE AGREEMENT NO: DE-AC07-76ID01570,1HA116 through DOE-ID

CONTRACTOR: Idaho National Engineering Laboratory
EG&G Idaho, Inc.
P. O. Box 1625
Idaho Falls, ID 83415

PRINCIPAL INVESTIGATOR: Manohar S. Sohal (208) 526-9412

OBJECTIVE: Design, construct, and test a gas-side fouling probe for use in high temperature industrial exhaust gas streams. It should be capable of determining on-line gas-side fouling resistance.

TECHNICAL APPROACH: Designed a simple, sturdy, air cooled probe that can be easily installed and removed from an exhaust gas stream. Limited laboratory tests were conducted over selected ranges of gas temperatures, probe surface temperature, gas velocity and particulate concentration.

Two probes were constructed. The probe and the auxiliary instrumentation, such as surface temperature controller, automatic data recording system, were assembled ready to be shipped to a field test site. Fouling data were recorded. The data were analyzed to test the probe's suitability for measuring on-line gas-side fouling resistance in industrial streams.

SYSTEM DESCRIPTION or APPLICATION: The probe consists of a 1.0-in. OD circular cylinder with a heat flux meter embedded in the tube wall to measure the local wall heat flux. The probe surface is cooled with compressed air, and a temperature control system maintains a constant wall surface temperature during testing. The heat flux meter measures local fouling factors and calorimetric measurements simultaneously determine average fouling factors.

CURRENT STATUS (9/89): Field testing of the fouling probe at a coal fired power plant was completed. A final report on this phase of the project will be issued. One fouling probe was shipped to a coal fired plant in Massachusetts for further tests. Field testing of the fouling probe in a waste incineration plant is being planned.

ACCOMPLISHMENTS: A gas-side fouling measuring device was designed, constructed, and tested in laboratory and industrial settings. Laboratory tests were carried out using natural gas combustion products doped with slaked lime to serve as the foulant. Local and average fouling factors were obtained.

The results confirm that the probe is capable of determining both local and average on-line gas-side fouling factors. The fouling probe was tested in industrial exhaust gas streams over a broad range of parameters: probe wall temperature, 600 to 1300°F; gas temperature, 820 to 1840°F; heat flux, approximately 2800 to 8000 Btu/hr-ft^2; and gas velocity, 10 to 20 ft/sec.

FUTURE WORK: Field testing of the probe in a waste incineration plant exhaust gas streams is being planned. The objective of this project is to obtain the fouling data in support of the High Pressure Exchange System ((HiPHES) projects and to transfer the fouling probe technology to industry for commercialization. This objective will be met by performing further field tests.

CONCLUSIONS: An air-cooled gas-side fouling measuring device was designed, constructed, and successfully tested in laboratory and industrial settings. The results obtained confirm that the probe is capable of measuring on-line local and average gas-side fouling factors. It can also survive the conditions in the flue gas stream of a coal fired plant. However, additional data under more severe industrial conditions, such as a waste incineration plant, are needed to confirm validity of the device as a tool for high-temperature industrial applications.

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1. Marner, W. J. and MacDavid, K. S., Development of a Gas-Side Fouling Measuring Device, Final Report, Report No. DOE/ID/12138-7, Jet Propulsion Laboratory (Report No. JPL D-4065), May 1987.

2. Marner, W. J., "Gas-Side Fouling", Mechanical Engineering, Vol. 108. No. 3, 1986, pp. 70-77.

3. Marner, W. J., MacDavid, K. S., and Muzio, L. J., "Development of a Gas-Side Fouling Measuring Device", Presented at the 26th National Heat Transfer Conference, Philadelphia, PA, August 6-9, 1989.

Idaho
National
Engineering
Laboratory

Field Testing of a Gas-side Fouling Probe

Manohar S. Sohal

DOE Advanced Heat Exchanger
Program Review
October 11-12, 1989
Washington, D.C.

DOE FOULING WORKSHOP [1981]

- 41 PARTICIPANTS INCLUDING 30 INDUSTRIAL REPRESENTATIVES

- INDUSTRIES REPRESENTED INCLUDED (BUT NOT LIMITED TO):
  - Battelle Columbus Laboratories
  - AiResearch Manufacturing Company
  - Diamond Power Specialty Company
  - Occidental Research Corporation
  - PPG Industries, Inc.
  - Westinghouse R&D Center
  - Smith Engineering Company
  - Exxon Research & Engineering Company
  - Bethlehem Steel Corporation
  - Sunstrand Corporation
  - Standard Oil of California
  - Escoa Fintube Corporation
  - Riley Beaird, Inc.
  - Solar Turbines, Inc.
  - KTI Corporation
  - KVB, Inc.

- PARTICIPANTS MADE 7 HIGH-PRIORITY R&D RECOMMENDATIONS

BACKGROUND

- DOE GAS-SIDE FOULING WORKSHOP (JPL PUBLICATION 82-67)

- DOE/BATTELLE PNL GAS-SIDE FOULING AND CORROSION SURVEY (JPL PUBLICATION 83-74)

- SURVEY OF GAS-SIDE FOULING MEASURING DEVICES (JPL PUBLICATION 84-11)

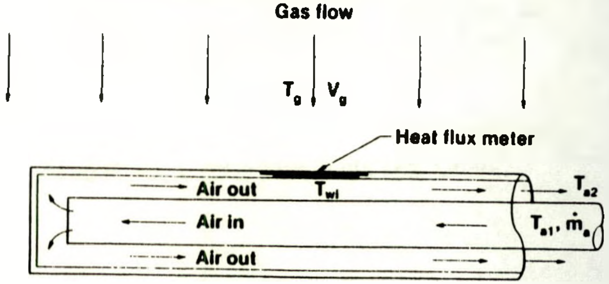
PURPOSE OF A GAS-SIDE FOULING PROBE

- USE PROBE TO INVESTIGATE FOULING CHARACTERISTICS OF EXHAUST GAS STREAMS TO PROVIDE DESIGN INPUT FOR <u>POTENTIAL</u> HEAT RECOVERY SYSTEMS

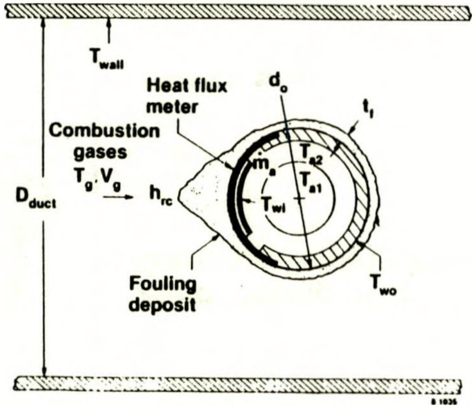
PAST ACTIVITIES UP TO PRESENT

- JPL DESIGNED AND FABRICATED A GAS-SIDE FOULING PROBE
- JPL TESTED THE PROBE IN A LABORATORY SETTING
- "PROOF OF CONCEPT" ACCOMPLISHED - FOULING RESISTANCE CAN BE MEASURED
- APPLICABILITY IN AN INDUSTRIAL ENVIRONMENT BEING TESTED
  - TESTED THE PROBE IN A COAL FIRED BOILER FLUE
- EXAMINED AND RECALIBRATED THE PROBES
- SHIPPED ONE PROBE FOR FURTHER TESTS IN A COAL FIRED BOILER

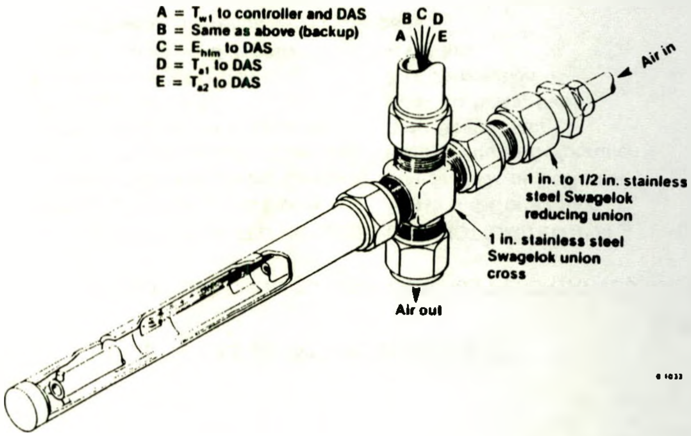
Schematic Drawing of Gas-Side Fouling Probe



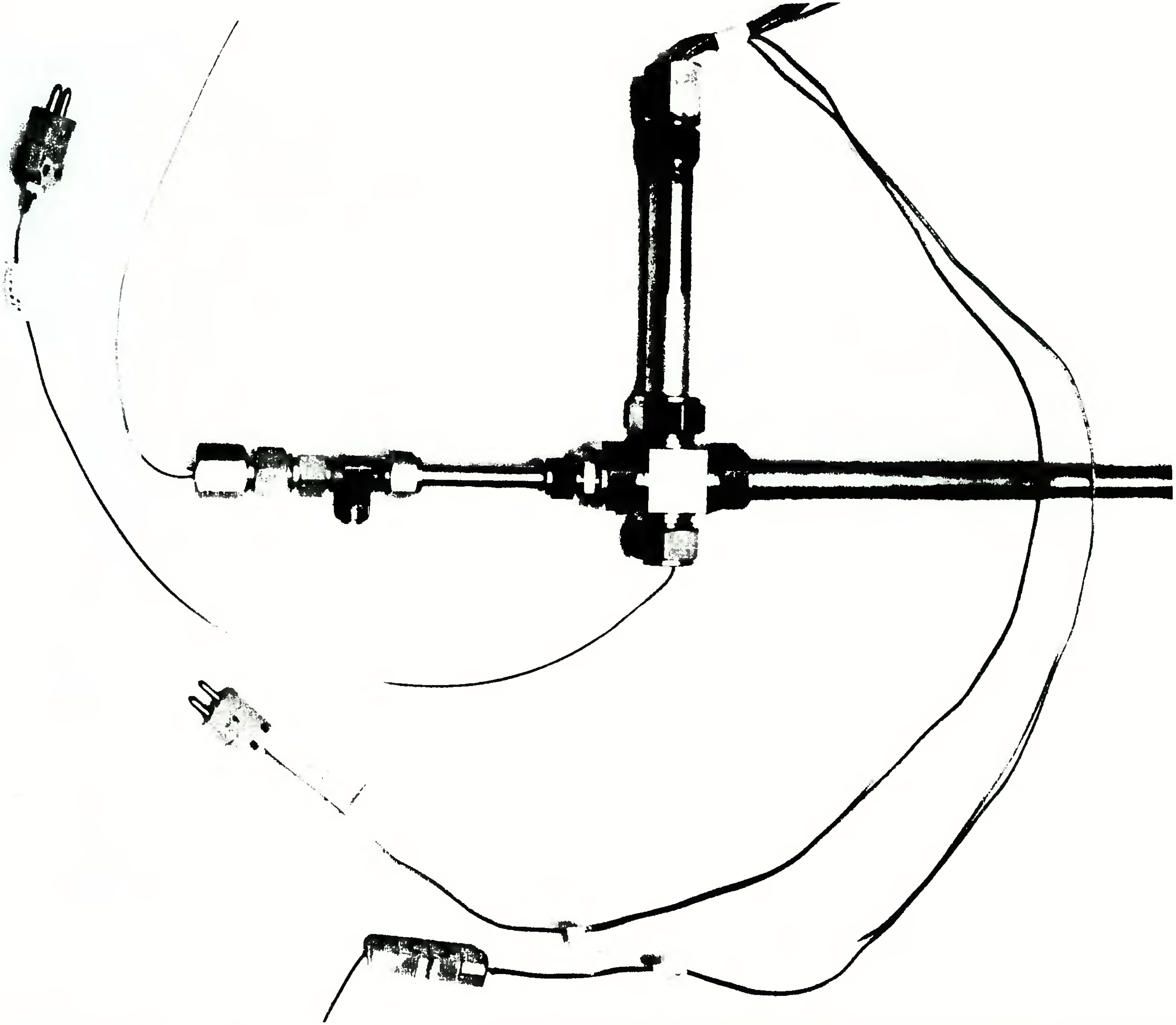
Deposit on a Fouling Probe



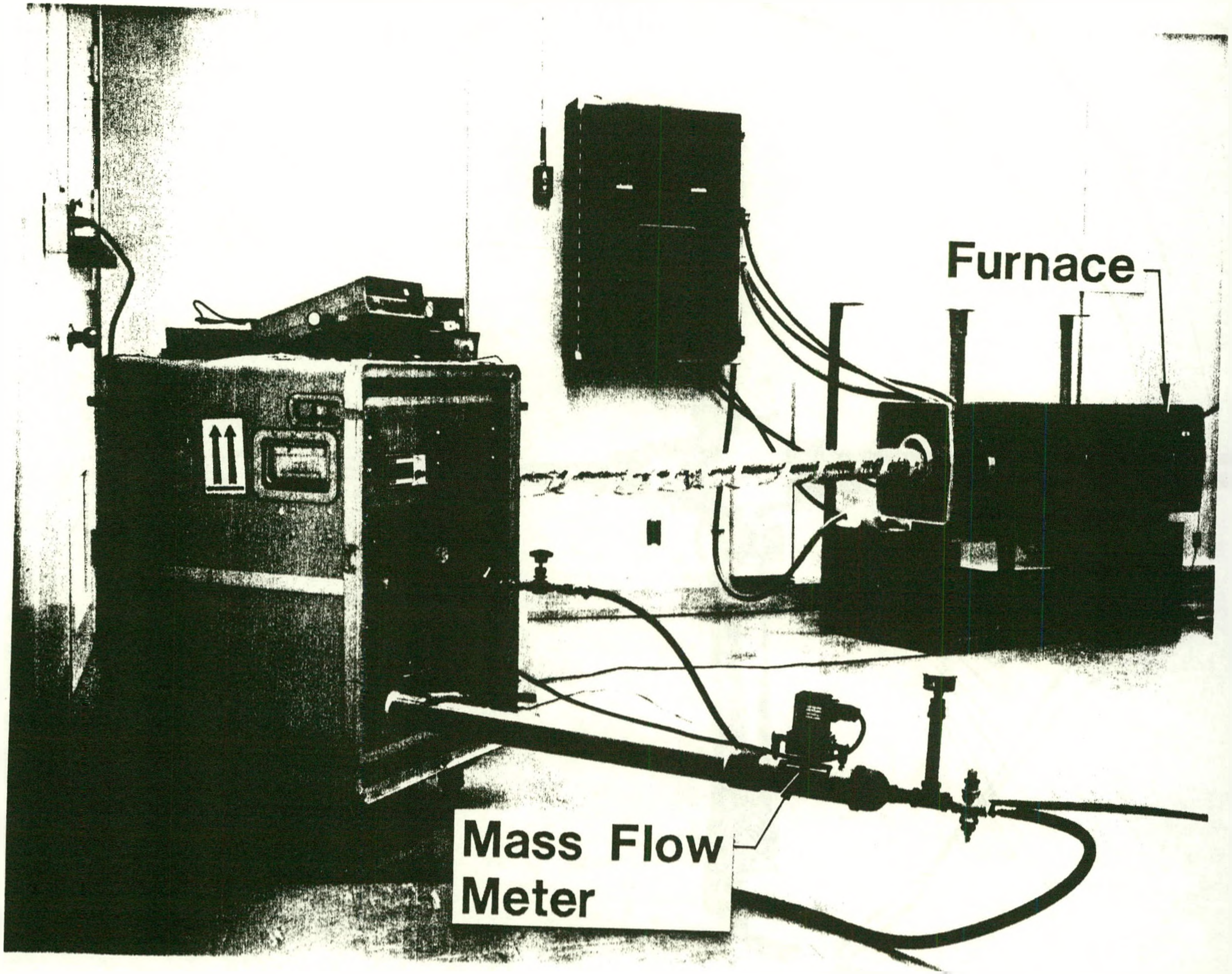
Fouling Probe Assembly



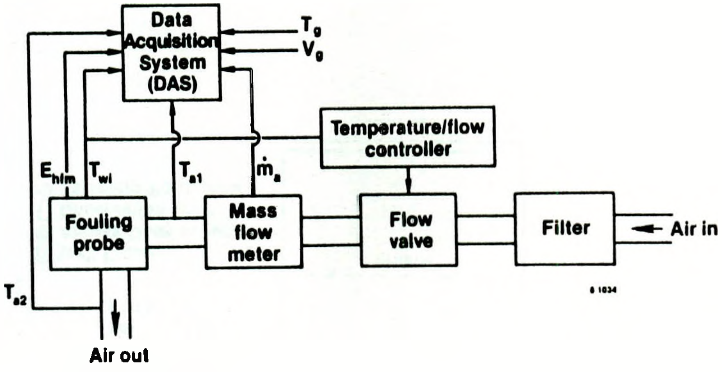
Fouling Probe



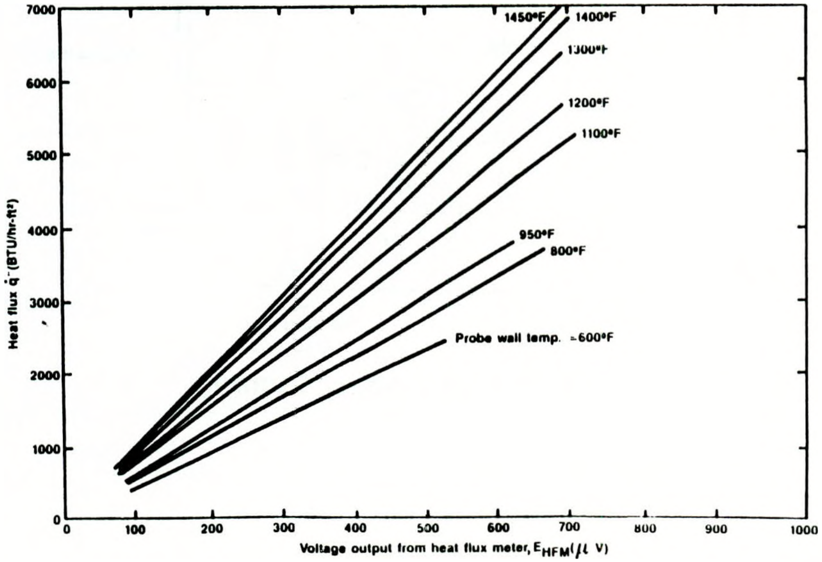
Calibration of Probe



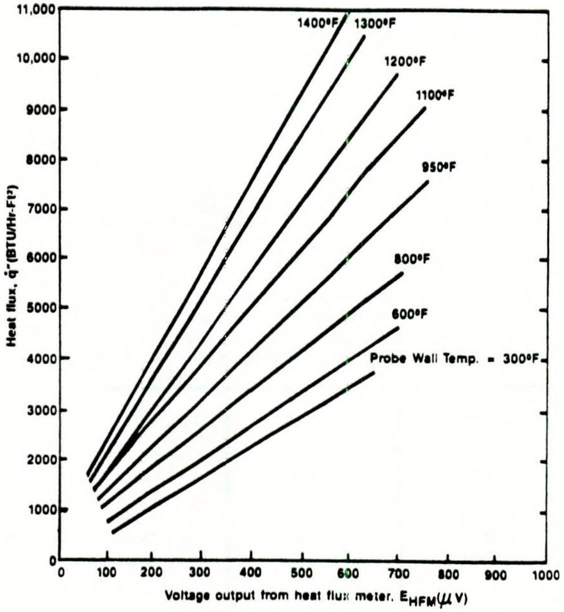
Fouling Probe Data and Control System



Calibration Curves for Fouling Probe No. 2



Calibration Curves for Probe No. 1

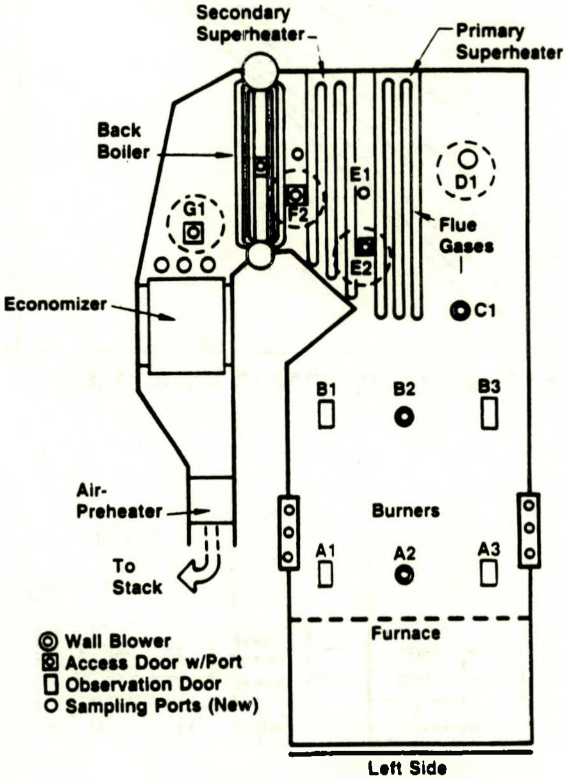


Calibration Constants for the Two Probes

$\dot{q}''(\text{Btu/hr-ft}^2) = E_{HFM}(\mu V) + C_2$

Probe No. 1		
Twall (°F)	**C_1**	**C_2**
300	5.55	-48
600	6.71	46
800	8.12	171
950	10.01	159
1100	12.04	283
1200	14.09	176
1300	16.67	313
1400	18.09	391
Probe No. 2		
Twall (°F)	**C_1**	**C_2**
300	5.08	-58
600	4.71	9
800	5.55	-9
950	6.09	16
1100	7.32	64
1200	8.07	36
1300	9.17	11
1400	9.79	-1
1450	10.23	-48

Fouling Probe Test Ports



Test Port Characterization

Insertion Depth	2'	4'	6'	8'
Test 13, Port E2				
Pre-Test				
Gas Temperature (°F)	1681	1516	1426	1398
Gas Velocity (ft/sec)	39.00	22.60	19.12	21.41
*Gas Flow Direction, degree	180	180	90	90
Excess Oxygen (%)	1.5	1.8	1.8	1.9
Carbon Monoxide (ppm)	95	96	109	101
Post-Test				
Gas Temperature (°F)	1651	1523	1456	1427
Test 15, Port F2				
Pre-Test				
Gas Temperature (°F)	1510	1241	1172	1165
Gas Velocity (ft/sec)	15.95	18.16	10.27	14.49
*Gas Flow Direction, degree	90	90	90	90
Excess Oxygen (%)	3.0	2.4	2.3	2.3
Carbon Monoxide (ppm)	120	140	155	158
Post-Test				
Gas Temperature (°F)	1585	1345	1221	1176

*Flow Direction: 0° Flow Down From Roof
90° Flow From Front Wall
180° Flow Up From Furnace
270° Flow From Rear Wall

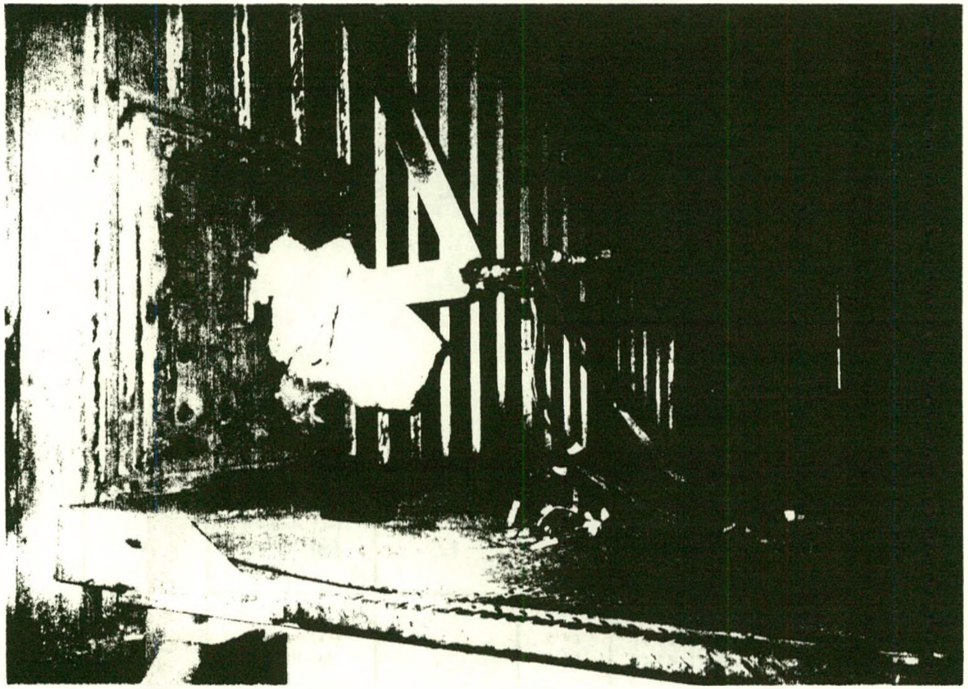
Flue Gas Composition

Test Number	Analysis Basis	Test Port	Composition of Combustion Gases				
			H_2O	CO_2	N_2	SO_2	O_2
13	Weight (%) Volume (%)	E2	5.04 8.33	22.72 15.38	69.91 74.34	0.46 0.22	1.87 1.74
15	Weight (%) Volume (%)	F2	4.93 8.15	22.24 15.04	70.06 74.44	0.45 0.21	2.32 2.16

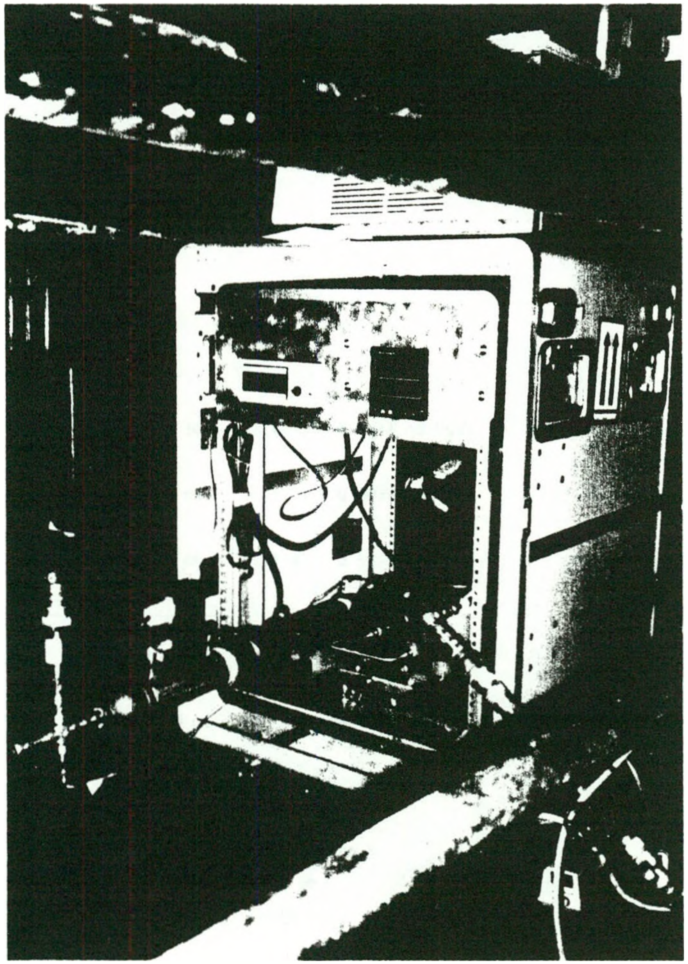
Field Tests at Richmond Power & Light Co. Whitewater Valley Station Unit #2 Richmond, Indiana

Test No.	Date (1989)	Port	Probe Temp. (°F)	Gas Temp. (°F)
4	Jan 11	E2	1100	1700
10A	Jan 31	E2	1100	1439
11A	Feb 1	D1	1300	1840
11C	Feb 1	G1	600	823
12A1	Feb 2	F2	800	1125
13	Feb 3	E2	950	1439
15	Mar 1	F2	800	1169

Field Testing of Fouling Probe



Instrumentation for the Probe



TYPICAL TEST DATA

FOULING PROBE FIELD TEST
RICHMOND POWER & LIGHT
WHITEWATER VALLEY STATION
UNIT 2: PORT F2

TEST 15 - 1 MARCH 1989
BOILER LOAD: 60 MW
PROBE TEMPERATURE: 800°F
GAS TEMPERATURE: 1169°F
DATA TIME: 5 MINUTES

TEST TIME HR	PROBE TEMP °F	AIR-IN TEMP °F	AIR-OUT TEMP °F	AIR-FLOW RATE LB/HR	HF-METER OUTPUT µV	METER-HF CALC B/FT^{2}-H	AVE-HF CALC B/FT^{2}-H	AVE-FF CALC	LOC-FF CALC
8.36	798.9	498.1	626.2	58.67	380.35	3258.00	3319.03	0.0028	0.0004
8.44	799.1	498.3	626.4	58.31	381.08	3263.90	3299.18	0.0035	0.0003
8.53	799.8	498.2	626.4	59.59	383.98	3287.50	3371.60	0.0011	-0.0001

FUTURE ACTIVITIES

- ISSUE A REPORT ON THE FIELD TESTING OF THE PROBE
- DESIGN AND FABRICATE A PROBE SUITABLE FOR TESTING IN A WASTE INCINERATOR PLANT
- TEST THE PROBE IN A WASTE INCINERATION PLANT

CONCLUSIONS

- A FOULING PROBE WAS DESIGNED AND FABRICATED
- THE PROBE CAN MEASURE THE FOULING RESISTANCE
- THE PROBE IS ROBUST ENOUGH TO WITHSTAND THE INDUSTRIAL ENVIRONMENT (A COAL FIRED PLANT FLUE)

DISCUSSION

Diagnostic Tests in a Flue Gas Stream - Fouling Probe

Manohar S. Sohal

Q: You showed a difference between the heat flux meter and the overall heat flux. An error was then found in the air flow meter, and a correction made, but the correction seemed to apply to the heat flux meter and not to the overall measurement.

A: That's correct. The problem was that the manufacturer had suggested the mass flow rate as measured by the mass flow meter to be only for one atmosphere pressure and 0°C (standard conditions). After looking carefully into it, I found that the mass flow meter gives you mass flow rate, not volume, so you don't have to make any corrections for pressure and temperature. In the data acquisition system, someone had made an adjustment to the meter reading to account for the deviation of the existing temperature and the pressure in the compressor from standard conditions because, in order to have correct flow. You had to have close to 100 psi in the air line. That correction wasn't needed.

Q: Are there any plans to take the data that you're obtaining and comparing it with predictive methods that are available? There are a number of methods for predicting impaction and thermal effects. At B&W, we've developed some of these models and we've gotten very good comparison of predictions with similar type of experiments. Particular application has been for the research program where they were injecting limestone to reduce NO_x in boilers and we've obtained some pretty good comparisons between the following rates and the predictions that we made.

A: I don't think there is any plan right now to get into predictive methods. In this particular project we are only looking at how can we correctly measure fouling at the different conditions.

Q: You were able to measure fouling performance. Just how did you predict fouling, or in what way would you use it to predict fouling?

A: You can install a probe into any gas stream and you can get what will be the fouling factor in that gas stream. In other words, you can take a probe and let it be in the gas stream for an amount of time, which will produce fouling. At the end of the test, you will get a fouling factor which you can use in heat exchanger design.

DISCUSSION

[illegible]
[illegible]

CORROSION PROBE TESTING AND DEVELOPMENT

J. I. Federer
ORNL

U. S. DEPARTMENT OF ENERGY, OFFICE OF INDUSTRIAL PROGRAMS
ADVANCED HEAT EXCHANGER PROGRAM
TASK DESCRIPTION

TITLE: Corrosion Probe Testing and Development
WORK PROPOSAL NUMBER: CEED009
CONTRACTOR: Oak Ridge National Laboratory

PRINCIPAL INVESTIGATORS: R. A. Bradley, 615/574-6094 and J. I. Federer, 615/574-5131

OBJECTIVE: Demonstrate that the temperature of the probe can be controlled relative to the flue gas temperature of the host facility.

TECHNICAL APPROACH: The probe will be inserted into the flue gas stream of a coal-fired furnace where the temperature of the probe would normally equilibrate with the flue gas temperature. The ability of the probe instrumentation to control the probe temperature at values different from the flue gas temperature will be evaluated by internally cooling and heating the probe.

SYSTEM DESCRIPTION: The probe consists of a SiC tube (representing the material being tested), a stainless steel end fixture for supporting the probe in a flue gas stream, internal air flow and/or gas combustion, and external instrumentation for controlling cooling air or gas combustion as required.

CURRENT STATUS (9/89): The prototype probe and instrumentation are completed. Facilities at the host site are installed. Testing is in progress.

ACCOMPLISHMENTS: Design and construction of the probe, design and construction of the instrumentation for internal heating and cooling of the probe, installation of services at the host site, preparation of a test plan for safety review.

FUTURE WORK: Construction and testing of an advanced probe incorporating several ceramic test specimens.

CONCLUSIONS: None yet available.

BIBLIOGRAPHY:

1. J. A. McEvers and J. I. Federer, "Design of a Controlled Temperature Flue Gas Corrosion Probe," proceedings of the 1988 National Heat Transfer Conference, Vol. 1, Houston, Texas, July 24-27, 1988, pp. 423-28, The American Society of Mechanical Engineers, New York, N.Y.

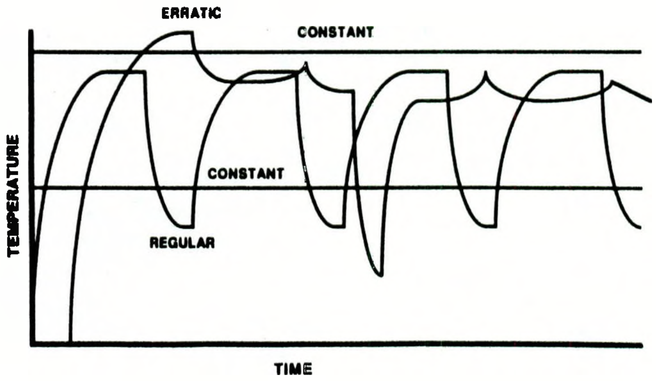
CORROSION PROBE TESTING AND DEVELOPMENT

J. I. FEDERER, METALS AND CERAMICS DIVISION
J. A. McEVERS, INSTRUMENTATION AND CONTROLS DIVISION
OAK RIDGE NATIONAL LABORATORY
OAK RIDGE, TENNESSEE

U. S. DEPARTMENT OF ENERGY
OFFICE OF INDUSTRIAL PROGRAMS
DIVISION OF WASTE ENERGY REDUCTION
WASTE ENERGY RECOVERY BRANCH

ADVANCED HEAT EXCHANGERS PROGRAM REVIEW
HERNDON, VIRGINIA
OCTOBER 11 AND 12, 1989

FLUE GAS AND PROBE TEMPERATURE



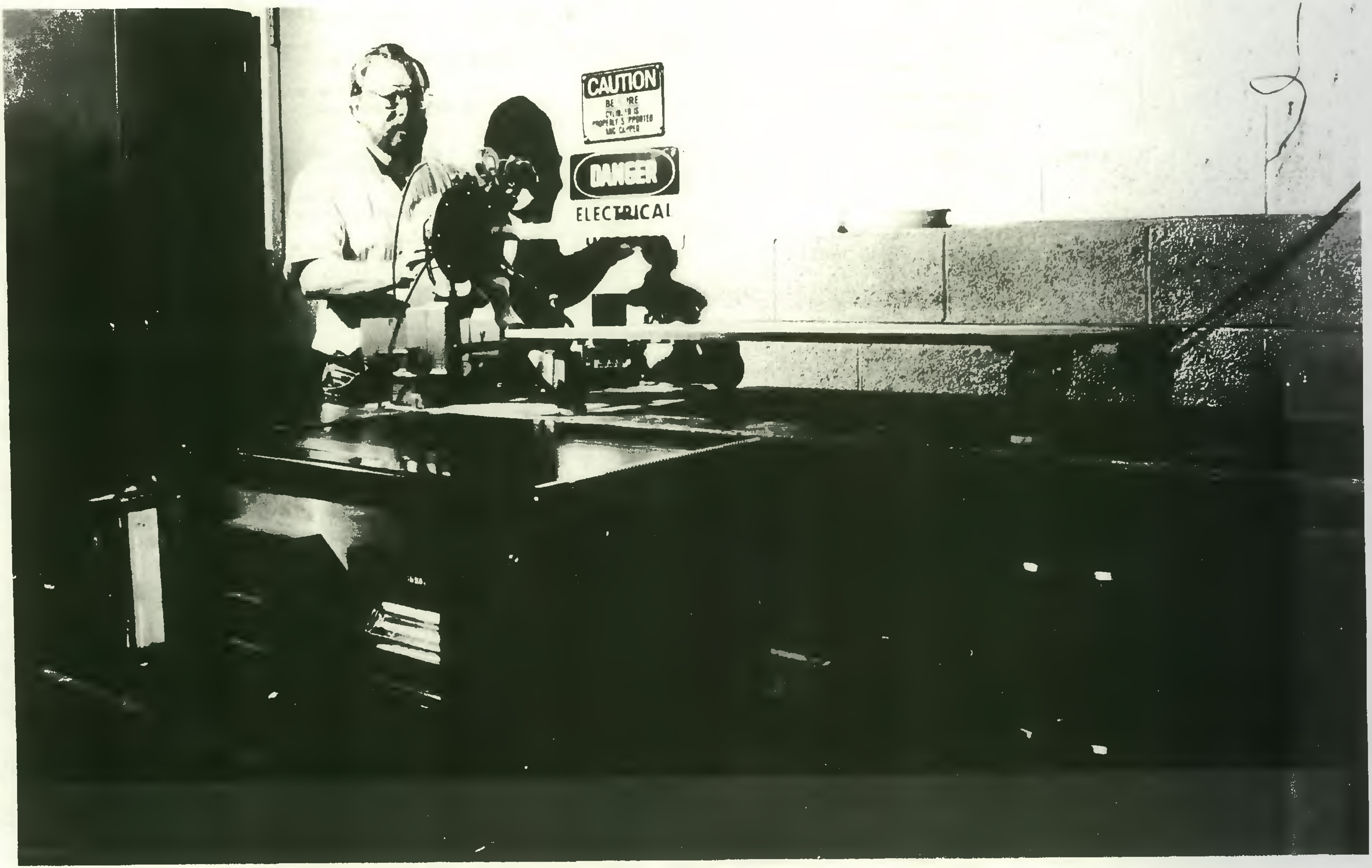
THE RESULTS OF CONVENTIONAL EXPOSURE TESTS ARE INCOMPLETE

- SPECIMEN TEMPERATURE IS NOT CONTROLLED OR ACCURATELY MEASURED
- FLUE GAS TEMPERATURE CYCLES FREQUENTLY IN SOME FURNACES; THEREFORE, SPECIMEN TEMPERATURE ALSO CYCLES
- CORROSION DATA REPRESENT COMPLICATED TEMPERATURE-TIME HISTORIES
- CORROSION RATE AT A PARTICULAR TEMPERATURE IS NOT DETERMINED
- EFFECT OF TEMPERATURE ON CORROSION RATE IS NOT DETERMINED
- EXTRAPOLATION OF CORROSION DATA TO OTHER FURNACES WITH DIFFERENT TEMPERATURE-TIME HISTORIES IS DIFFICULT
- STRESSES CAUSED BY THERMAL CYCLING MIGHT CAUSE ACCELERATED CORROSION OR OTHER DEGRADATION

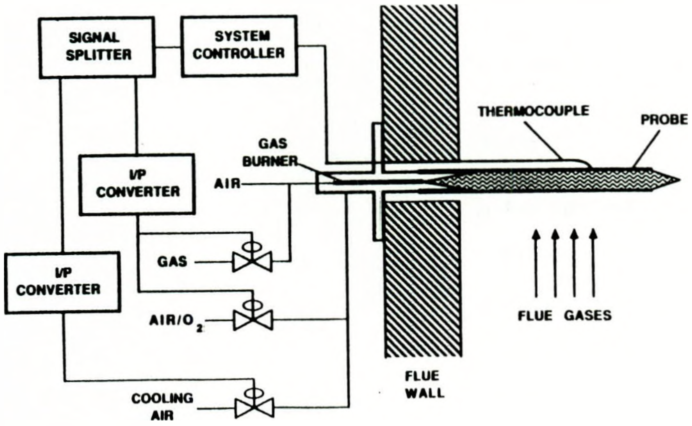
PRINCIPAL FEATURES OF A CONTROLLED TEMPERATURE CORROSION PROBE

- ONE OR MORE TUBULAR SPECIMENS
- THERMOCOUPLES
- INTERNAL HEATING
- INTERNAL COOLING
- AUTOMATIC TEMPERATURE CONTROL
- CONTINUOUS TEMPERATURE RECORD

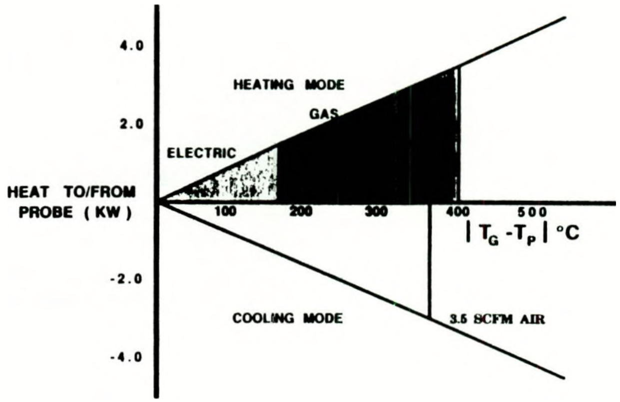
CORROSION PROBE BEING HEATED BY GAS COMBUSTION



SYSTEM CONFIGURATION WITH A GAS BURNER



CALCULATED PROBE OPERATING REGIME



TESTING IS BEING CONDUCTED IN AN ORNL STEAM PLANT FURNACE

COMBUSTION CONDITIONS

FUEL	NATURAL GAS	COAL
TEMPERATURE	≈1000°C	1100-1200°C
FOULING POTENTIAL	LOW	HIGH
CORROSION POTENTIAL	LOW	HIGH

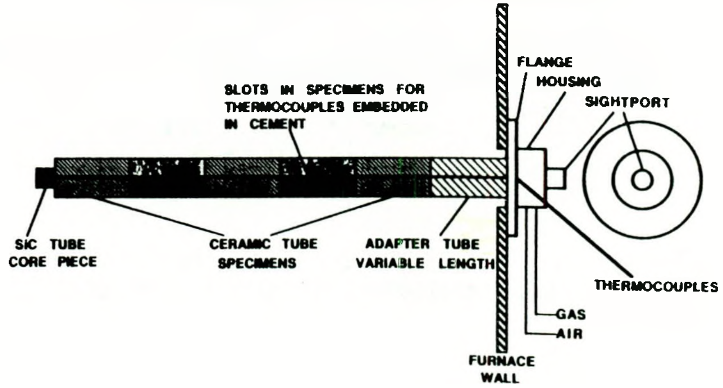
FACILITIES AT SITE

DEDICATED PORT
SPACE FOR INSTRUMENTATION
ELECTRICAL POWER
COMPRESSED AIR
PROPANE IN TANK

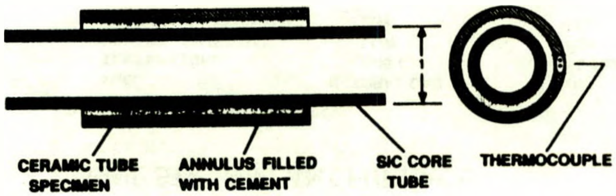
STATUS

PROBE INSTALLED
SAFETY REVIEW COMPLETED

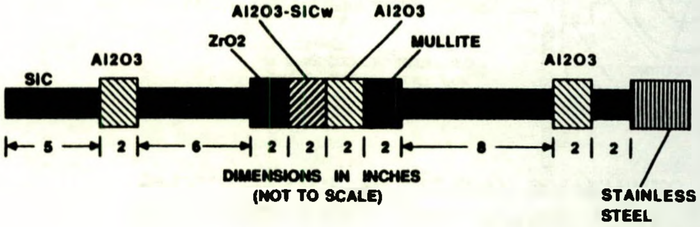
ADVANCED CORROSION PROBE WITH SPECIMENS OF DIFFERENT MATERIALS



CERAMIC SPECIMENS WILL BE SUPPORTED BY A SIC CORE TUBE



TENTATIVE MATERIALS ARRANGEMENT FOR ADVANCED CORROSION PROBE



SUMMARY

- A PROTOTYPE CORROSION PROBE AND TEMPERATURE CONTROL INSTRUMENTATION WERE DESIGNED AND FABRICATED
- THE HEATING CAPABILITY OF THE PROBE WAS INDICATED BY HEATING TO >1000°C IN THE LABORATORY
- THE PROBE WAS INSTALLED IN AN ORNL STEAM PLANT FURNACE, AND IS READY FOR TESTING
- COMPONENTS FOR AN ADVANCED PROBE CONTAINING SEVERAL TEST MATERIALS WERE DESIGNED, AND THE CONCEPT WAS TESTED IN THE LABORATORY

DISCUSSION

Corrosion Probe Testing and Development

J. I. Federer

Q: The concern was raised that if you heat or cool this probe, you may introduce another variable into your experiment, namely the heat transfer in the temperature gradient of the surface, which could effect transfer processes involving corrosion on the probe. If you attempt to heat the probe, will this produce a temperature gradient at the surface, and how will this affect corrosion?

A: Heat exchanger tubes typically operate at some temperature below the flue gas temperature, because they have cooling air flowing in them. So the probe represents a method to obtain corrosion data at specific temperatures of interest. Also, the probe could be heated to determine what will happen during a temperature excursion caused by loss of cooling air or overfiring. The corrosion occurs at the surface. I do not claim that we can measure the temperature of that surface with high accuracy, because the thermocouple is actually slightly below the surface. At least, we know the approximate temperature, and we know it over a period of time. In conventional exposure tests, the specimens often go through extreme changes in temperature, which cause a very complicated temperature-time history.

Q: On your advanced probe, is it your intention to have thermocouples on each of those specimen sections.

A: Probably not. Presently we foresee three thermocouples along the length, but we are not sure that it will be adequate. The present probe in the ORNL furnace appears to have a 50°C difference between the two thermocouples that are located twelve inches apart. Our experience with the probe now being tested will give us some information on how many thermocouples we will need in the advanced probe and how closely they and the specimens will need to be grouped in order to get the best temperature arrangement.

Q: How are you going to measure the corrosion rate? Does the geometry of the probe approximately simulate the geometry of the heat exchanger surface that you're trying to look at in the cross-flow mode?

A: In answer to the first question, the probe is inserted, operated for a period of time, withdrawn, and then subjected to a destructive type of examination to determine the corrosion rate. The diameters of specimens are measured before the exposure. Afterwards, optical microscopy methods are used to measure the diameters of the specimens again. Because of the corrosion product and/or fouling deposits that have formed on the outer surface, micrometers can no longer be used. Instead, we measure polished optical cross sections. The tubular probe would approximately simulate a heat exchanger tube exposed to flue gases in a cross-flow mode.

Advanced Heat Exchangers Program Review

October 11 & 12, 1989
Ramada Renaissance Hotel
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