INTERAGENCY COORDINATING COMMITTEE ON STRUCTURAL CERAMICS

ANNUAL MEETING

MAY 13, 1992

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ACKNOWLEDGEMENT

On behalf of the Committee, I wish to acknowledge Dr. Robert J. Gottschall of the Department of Energy for his considerable efforts at providing leadership, guidance and continuity to the activities of this organization.

Thomas W. Crooker
1991-1992 Chairperson
Interagency Coordinating Committee on
Structural Ceramics

INTERAGENCY COORDINATING COMMITTEE ON STRUCTURAL CERAMICS
ANNUAL MEETING
MAY 13, 1992

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INTRODUCTION

The Interagency Coordinating Committee on Structural Ceramics (ICCSC) held its annual meeting May 13, 1992. The primary function of the ICCSC is communication and coordination among managers of federally funded research and development programs in structural ceramics.

The focus of the Committee is on structural ceramic materials, including both monolithic ceramics and ceramic matrix composites. Twenty-three government program offices sponsored $120 million of unclassified research and development in fiscal year 1992 on these materials for applications in high performance transportation engine and turbine systems, cutting tools, heat exchanges, armor, bearings and aerospace engine and airframe components. These applications are critical to international commercial competitiveness since they represent large potential markets and also underpin the national defense posture.

This Committee regularly brings together federal program managers that have management responsibilities for such diverse research and development programs with a common denominator of interest and needs in structural ceramics. It provides a mechanism for maximizing the total effectiveness and resource sharing of government supported research and development in structural ceramics and assures that there is not redundancy or undesirable overlap in this coordinated effort.

Twenty-three federal program offices contributed to the proceeding of this meeting which was chaired Thomas W. Crooker of the National Aeronautics and Space Administration. Presentations were made which included both budgetary and programmatic information on the agencies with an emphasis on each program's mechanism of technology transfer. The following funding summary table has been extracted from the presentations and augmented with data supplied subsequently by committee members to determine fiscal year 1991, 1992 and estimated 1993 levels. The table is a summary which avoids duplicative data which would give an erroneously high estimate of funding in this field. It should be noted that the nature of research reported ranges from basic to applied and includes those portions of large component development projects which, in the judgement of the program managers, are clearly related to structural ceramics. Further, it should be understood that funding for fiscal year 1993 are estimates which may change.

A directory of the various program office representatives to this committee follows the following funding summary table.

The National Aeronautics and Space Administration appreciated the opportunity to host the ICCSC meeting and hence facilitate coordination of efforts in this critical field. The next meeting of the ICCSC is scheduled for May 12, 1993, Dr. David I. Lewis, III of the Naval Research Laboratory.

FEDERALLY SUPPORTED RESEARCH AND DEVELOPMENT IN STRUCTURAL CERAMICS

FUNDING SUMMARY TABLE ($M)

Organization	FY 1991	FY 1992	President's Request FY 1993
NSF	4.85	5.090	5.93
DOC-NIST	2.8	2.62	3.3
DOI-Bureau of Mines	2.99	3.19	2.47
DOE-BES/Materials Sciences	8.410	7.192	9.060
DOE-Transportation Tech. Ceramics Tech. for ADV. HT. ENG.	16.400	16.500	18.500
DOE-Transportation Tech. ADV. Turbine & Tech. Applications Program	11.725	12.148	11.5
DOE-ADV Industrial Concepts	1.80	2.10	2.15
DOE-Fossil Energy	1.29	1.192	0.808
DOE-Heavy Duty Transportation	0.35	0.5	0.8
DOE-Industrial Technologies Industrial Tech. Energy Efficiencies	5.775	7.875	7.9
NASA	18.4	25.4	34.9
DARPA	25	14	12
ARO-Durham	0.8	1.0	1.0
Army-MTL	6.57	2.65	3.76
Army Strategic Defense Cmd.	0.5	0.1	0.5
AFOSR	8.78	8.5	4.0
Wright Research & Development Laboratory	4.5	4.5	5.0
ONR	2.5	2.892	7.2
Naval Surface Warfare Ctr.	0.075	0.001	0
Navel Research Laboratory	0.264	0.920	1.200
Naval Air Warfare Center	0.9	0.85	1.5
Office of Naval Technology	0.05	0	0.3
Strategic Defense Initiative	0.53	0.53	0.53
Total	**125.3**	**119.8**	**134.3**

The above table represents an accurate summary of federal funding for research and development in structural ceramics. The numbers herein represent activity in structural ceramic research and development. They do not necessarily correspond to systems design or development programs budget line items, from which many structural ceramics efforts were estimated by pro-rating.

INTERAGENCY COORDINATING COMMITTEE
ON STRUCTURAL CERAMICS FY 1992
MAY 13, 1992
W. J. SCHAFER ASSOCIATES, INC.
1901 NORTH FORT MYER DRIVE, SUITE 800
ARLINGTON, VA 22209

AGENDA

Wednesday, May 13, 1992

9:00 am	Chairmens' Welcome	Mr. Thomas Crooker Mr. Stephen Moran
9:15 am	Keynote Address	Dr. Lyle Schwartz
9:45 am	National Institute of Standards & Technology	Dr. S.J. Dapkunas
10:00 am	Gas Research Institute	Dr. Michael Lukasiewicz
10:15 am	Department of Energy/Office of Basic Energy Sciences/Division of Materials Sciences	Dr. Alan Dragoo
10:30 am	Department of Energy/Office of Transportation Technology/Advanced Materials Division/AICD	Mr. Robert Schulz
10:45 am	Department of Energy/Office of Transportation Technology/Advanced Propulsion Division	Mr. Saunders Kramer
11:00 am	National Science Foundation	Dr. Frank Wang
11:15 am	Office of Naval Research	Dr. Steven Fishman
11:30 am	Office of Naval Technology	Dr. William Messick
11:45 am	Lunch	
1:00 pm	Naval Research Laboratory	Dr. David Lewis
1:15 pm	Naval Surface Warfare Center	Dr. Inna Talmy
1:30 pm	Naval Air Warfare Center/Aircraft Division	Mr. Randall Sands
1:45 pm	Naval Air Propulsion Center	Ms. Dawn Migliacci
2:00 pm	Army Strategic Defense Command	Mr. Doug Ennis
2:15 pm	Air Force Office of Scientific Research	Dr. Walter Jones
2:30 pm	Wright Laboratory/Materials Directorate	Dr. Allan Katz
2:45 pm	NASA Office of Aeronautics & Space Technology	Mr. Stephen Moran
3:00 pm	Defense Advanced Research Project Agency	Dr. William Coblenz
3:15 pm	Bureau of Mines	Dr. Sara Dillich Dr. Garret Hyde
3:30 pm	Army Research Office/Army Tank Automotive Command	Mr. Wilbur Simmons
3:45 pm	Army Materials Technology Laboratory	Dr. M.R. Fletcher
4:00 pm	Strategic Defense Initiative Office	Lt. Col. Michael Obal
4:15 pm	Department of Energy Environmental Restoration and Waste Management	Dr. Alan Dragoo, for Dr. Stanley Wolf

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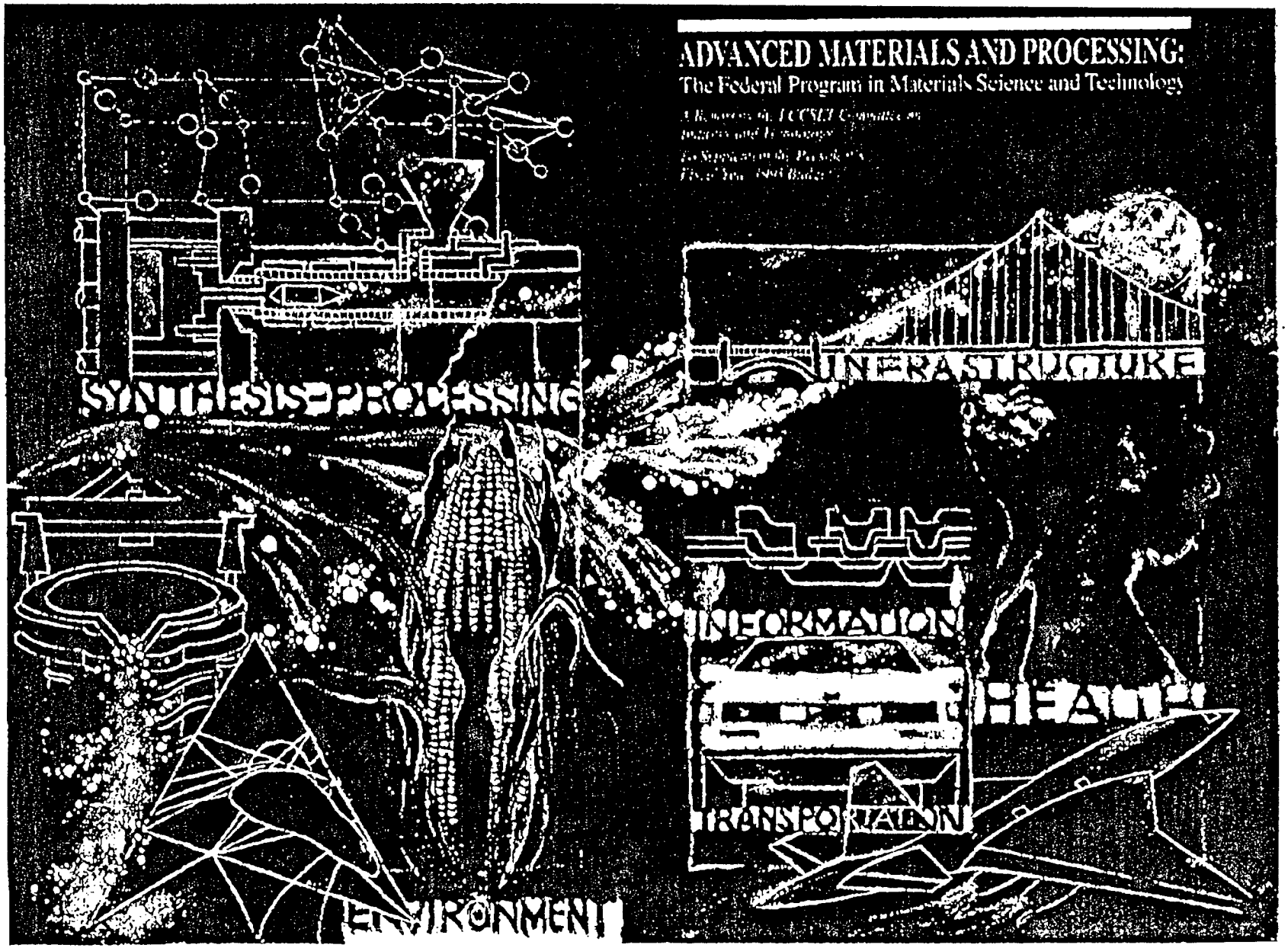
INTERAGENCY COORDINATING COMMITTEE ON STRUCTURAL CERAMICS

PRESENTATIONS

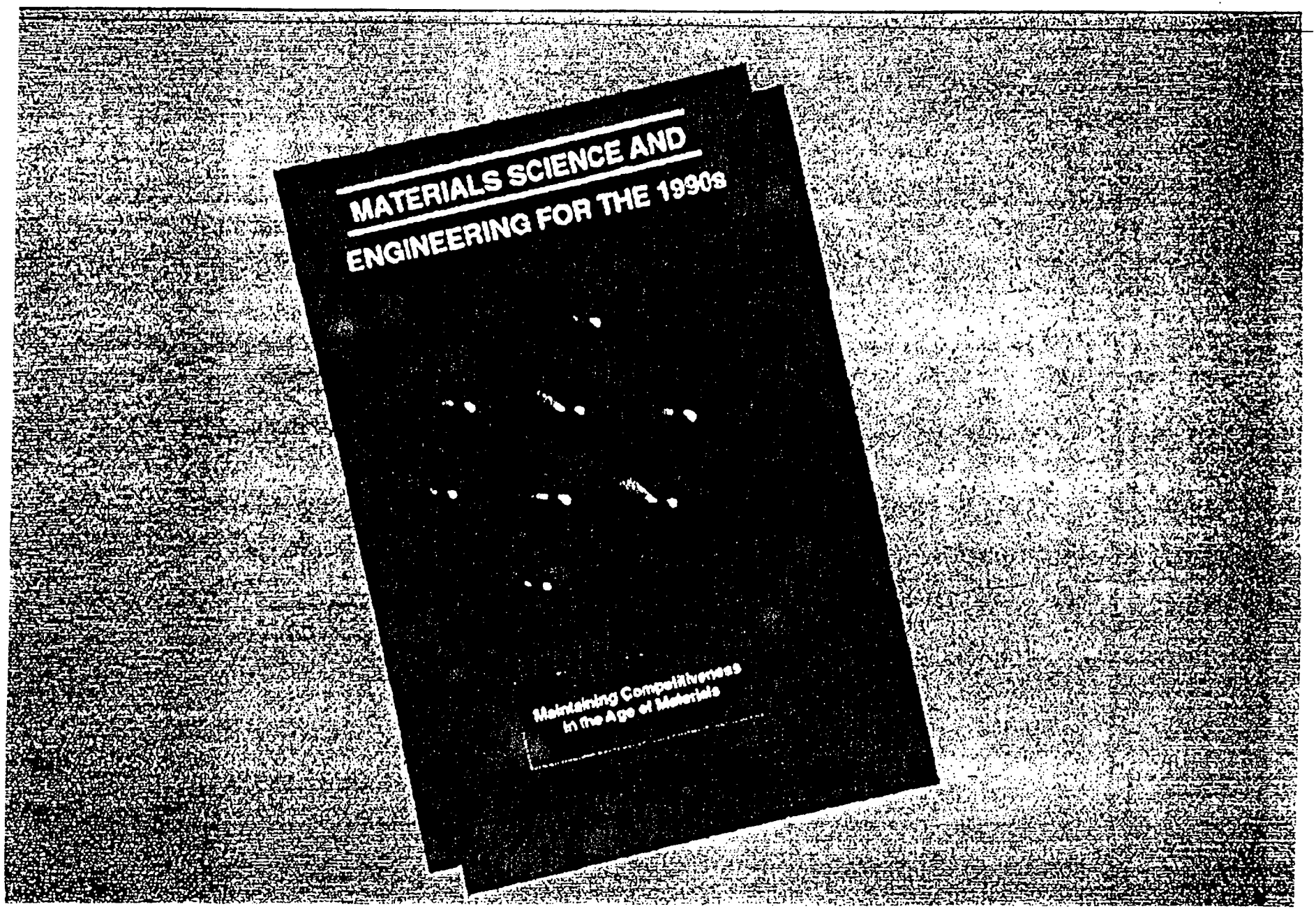


U.S. DEPARTMENT OF COMMERCE
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

DR. LYLE SCHWARTZ

PROLOGUE

- Materials are the Prototypical Enabling Technology
- Advanced Materials and Processing on Every Critical Technologies List
- NAS/NAE/NRC Study on Materials Science and Engineering for the 1990s Defined Issues
- Regional Meetings: A National Agenda for MSE
- Numerous Technical Assessments by Solid State Science Committee and National Materials Advisory Board Provide Detailed Background for Technical Planning
- Increased Public Awareness: Business Week; Public Television


MATERIALS SCIENCE AND
ENGINEERING FOR THE 1990s
Maintaining Competitiveness
in the Age of Materials

THE INFINITE VOYAGE

BANK MERGERS THE PAIN AND THE GAIN PAGE 24

AMERICA'S NEW WEAPON: LOW-COST CAPITAL PAGE 72

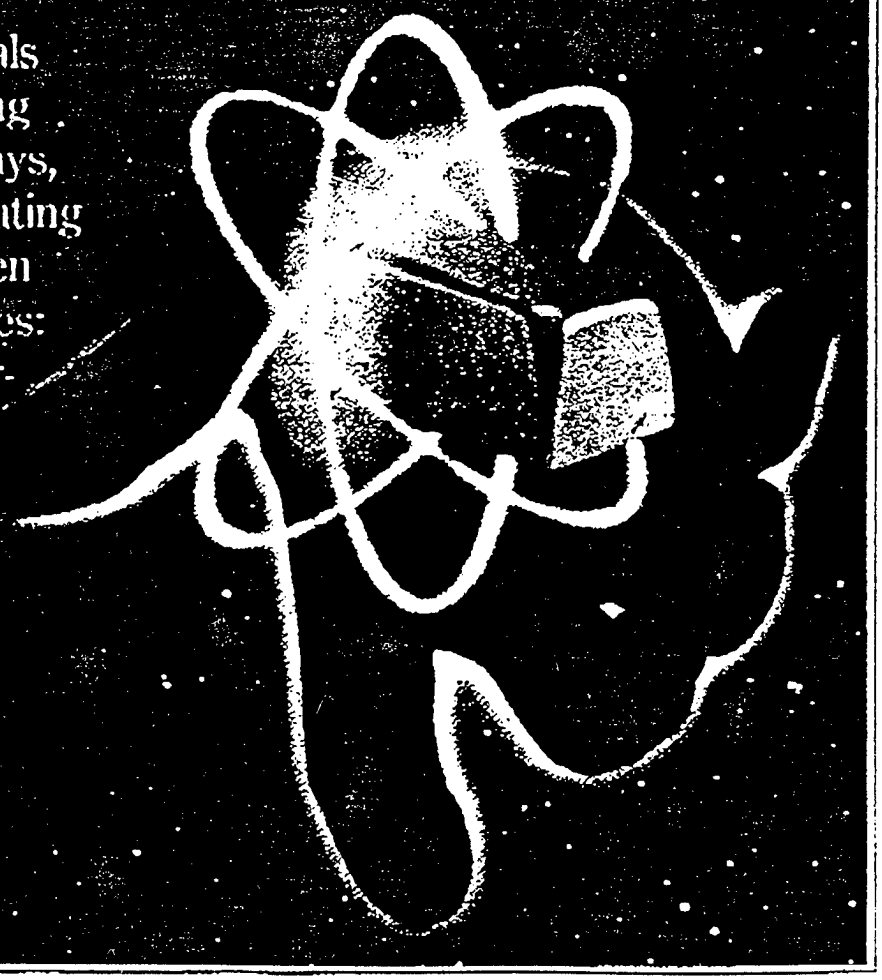
BusinessWeek

JULY 29, 1991 A McGRAW-HILL PUBLICATION $2.50

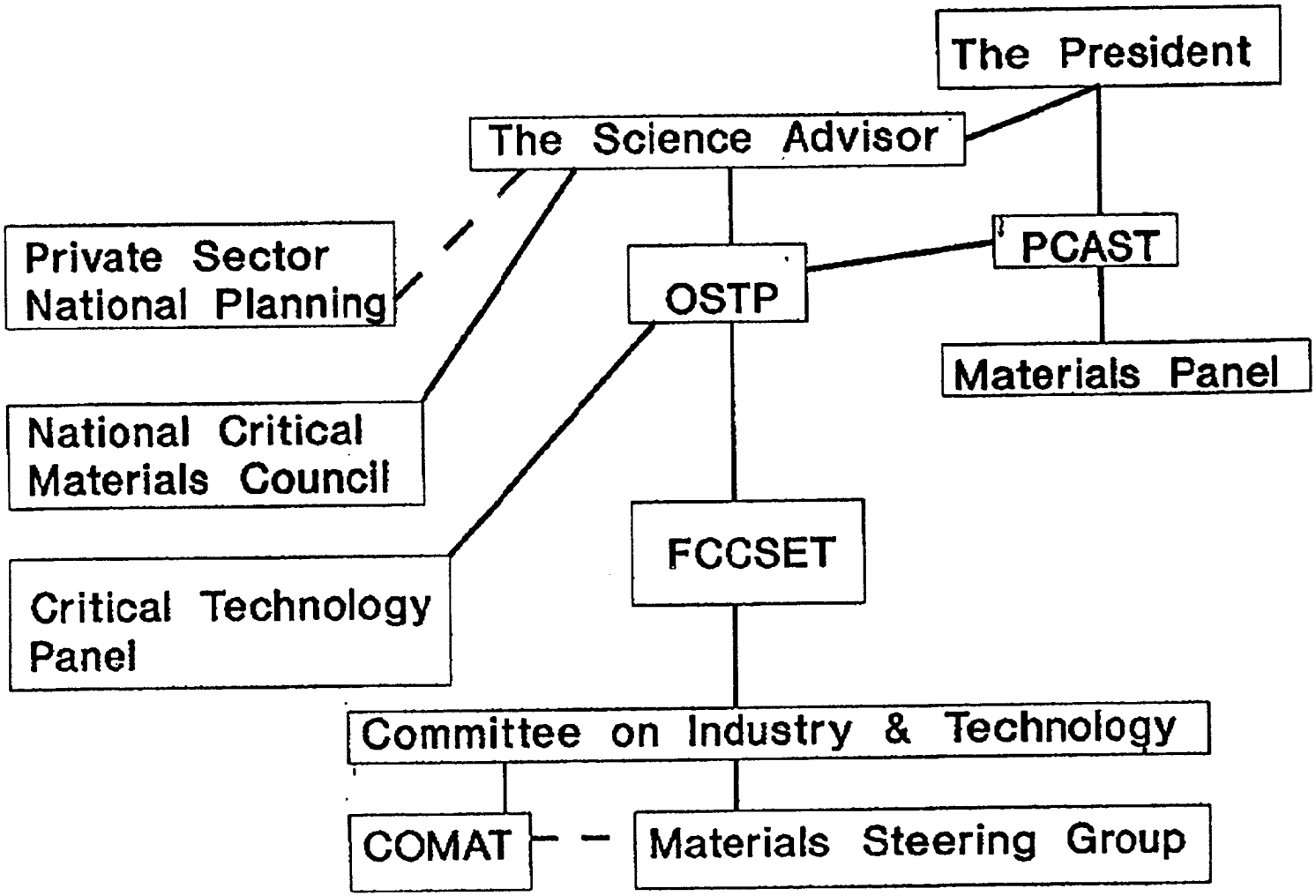
THE NEW ALCHEMY

Call it the Materials Age. By combining atoms in novel ways, scientists are creating materials that open up bold possibilities: Pocket-size supercomputers, superlight aircraft, bridges that warn when they're weak.

PAGE 40



MATERIALS-RELATED COMMITTEES



AMPP INITIATIVE STRATEGY

- Planning Framework and Process are in Place
- Priorities Reflect Private Sector Input
- Agency Support is Strong But Budget Constraints Limit Enhancement Opportunities
- Additional Planning With Private Sector is Critical for Applied Programs
- The AMPP Has Been Approved as a Presidential Initiative Based on a Multi-Year Phase-In Strategy to Achieve Breakthrough Opportunities

FRAMEWORK

- Goal

  Improve Manufacture and Performance of Materials to Enhance U.S. Quality of Life, National Security and Industrial Productivity and Economic Growth

- Objectives

  A. Establish and Maintain the U.S. Scientific and Technological Leadership Position in Advanced Materials and Processing

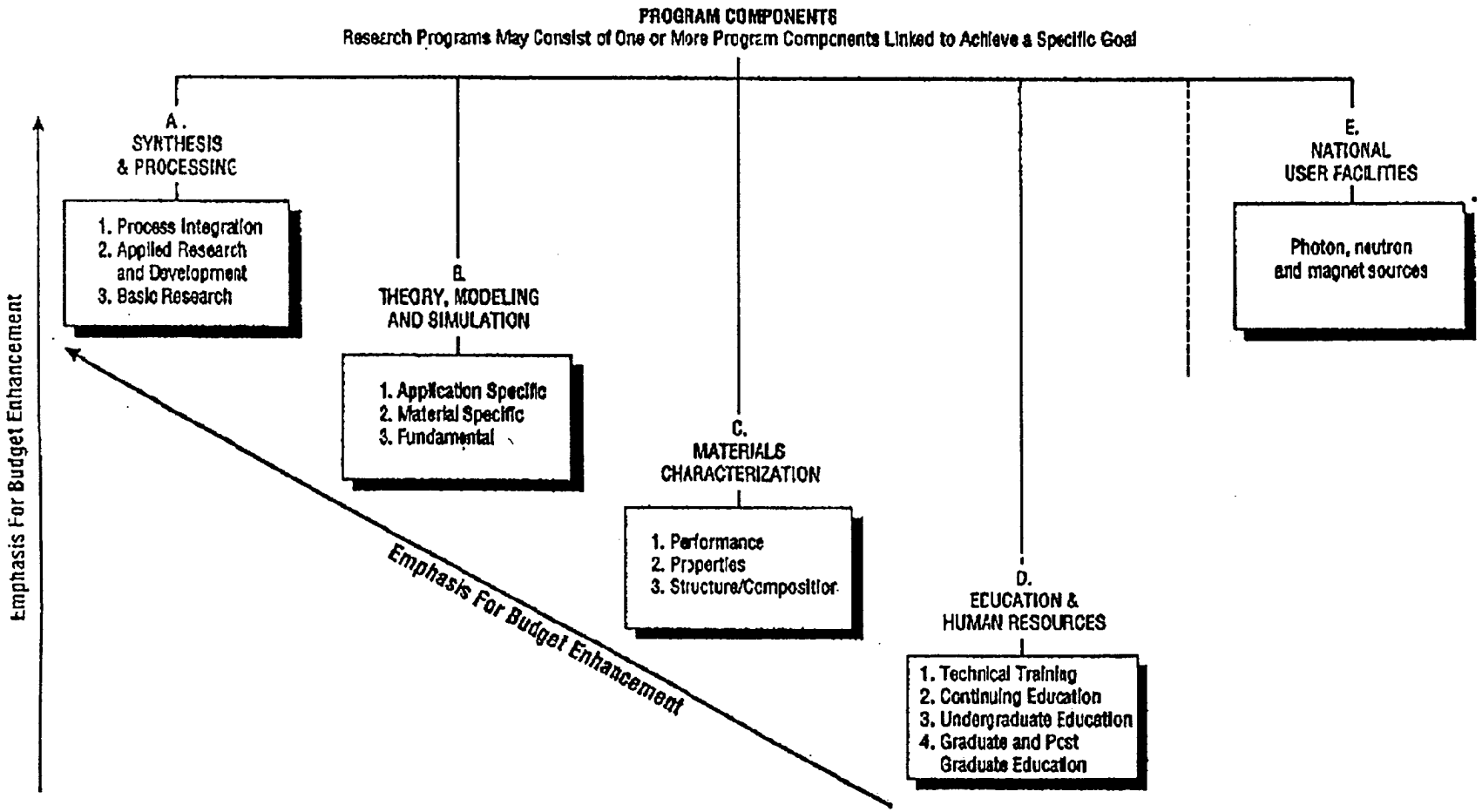
  B. Bridge the Gap Between Innovation and Application of Advanced Materials Technologies

  C. Support Agencies' Mission Objectives to Meet National Needs With Improvements in Advanced Materials and Processing

  D. Encourage University and Private Sector R&D Activities on Materials Technologies, Their Applications, and Their Implementation

- Breakthrough Opportunities

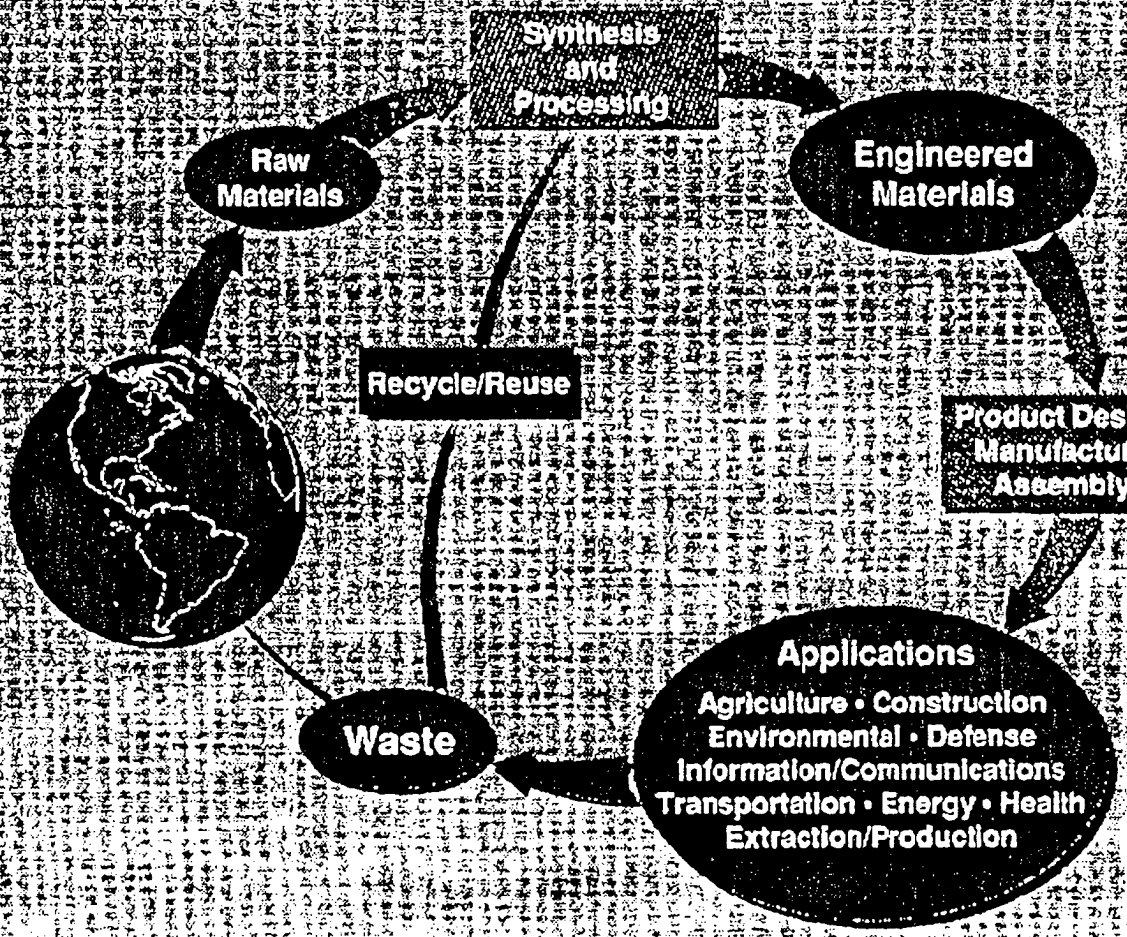
U.S. ADVANCED MATERIALS AND PROCESSING PROGRAM (AMPP)



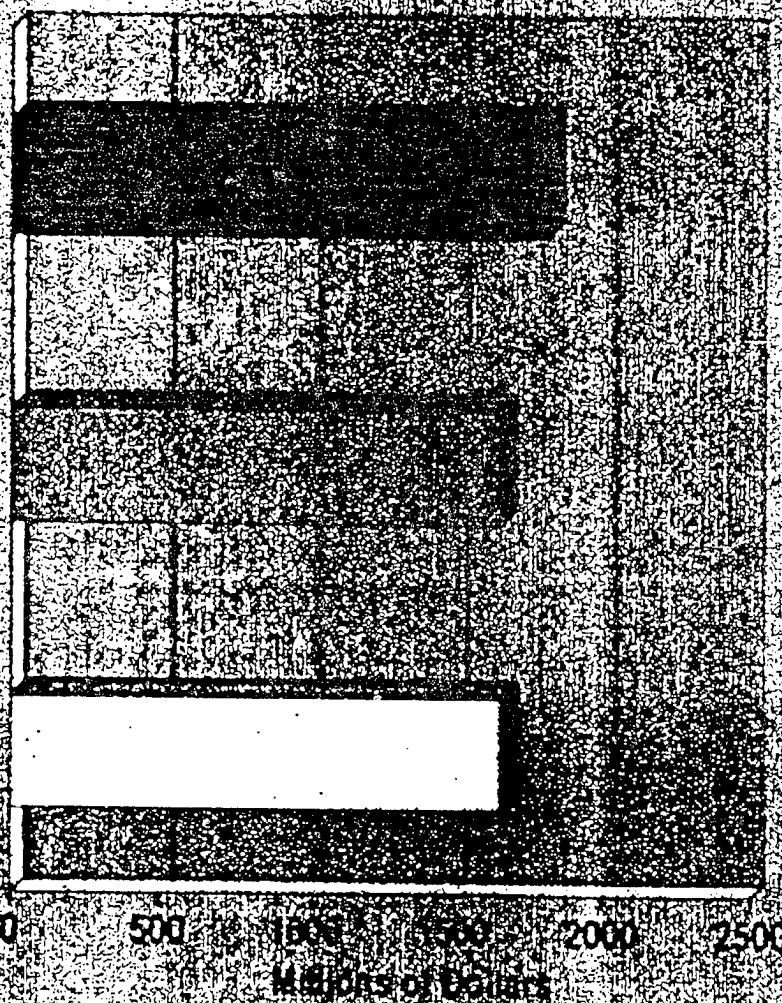
Breakthrough Opportunities

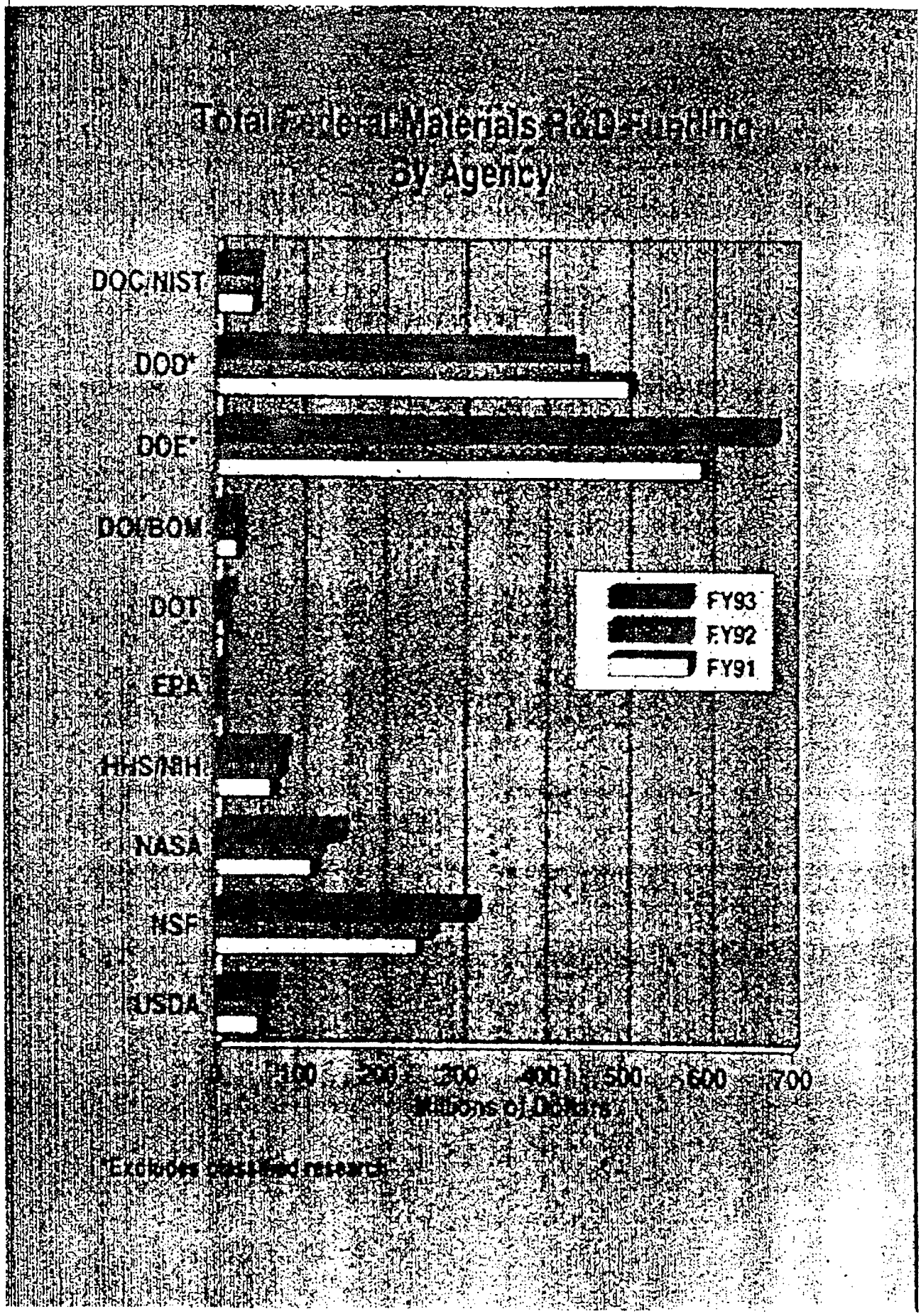
- Agriculture
- Defense
- Energy
- Environment
- Extraction/Production of Raw Materials
- Health
- Information/Communications
- Infrastructure/Construction
- Transportation
- Materials for the Future

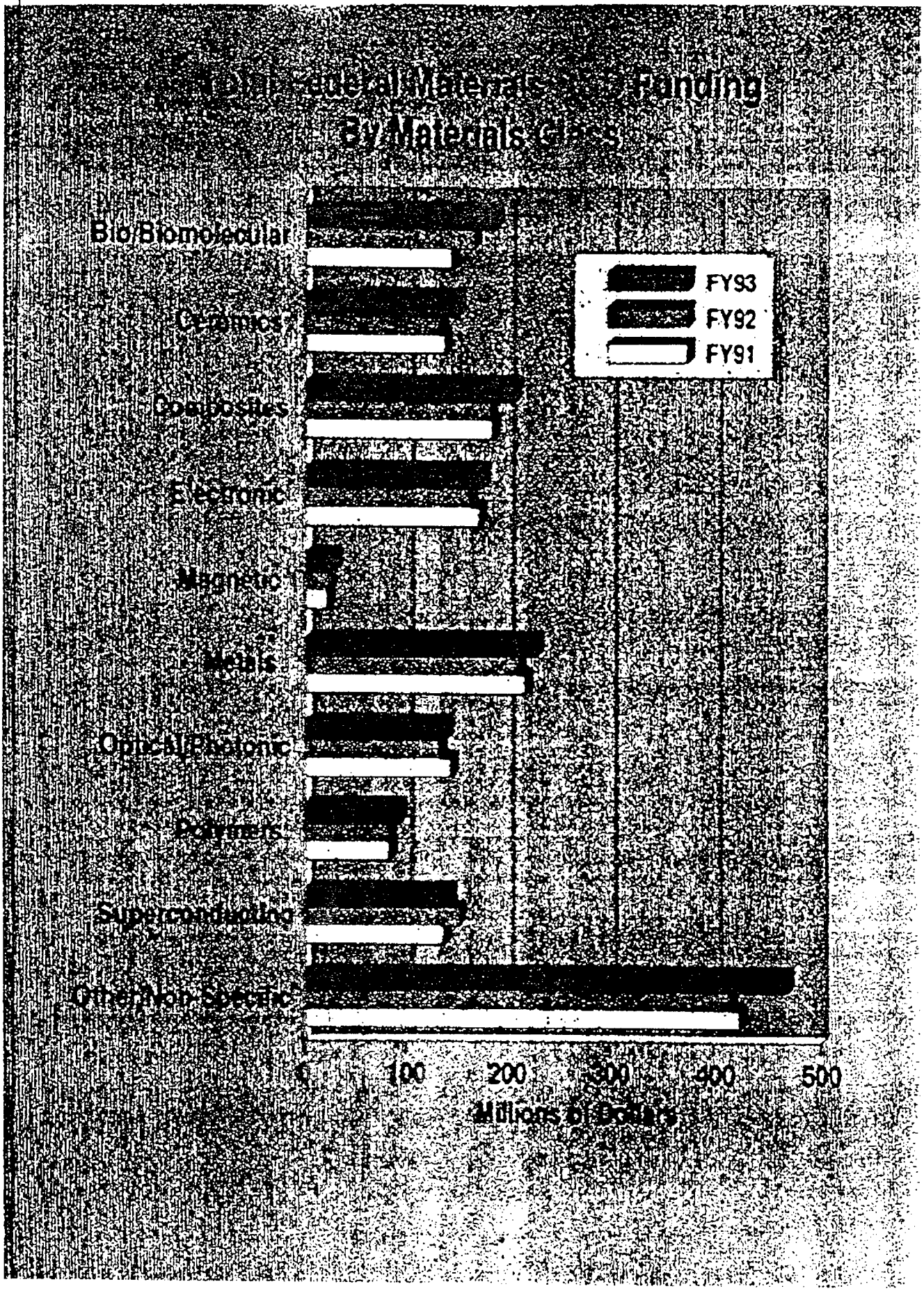
THE TOTAL MATERIALS CYCLE



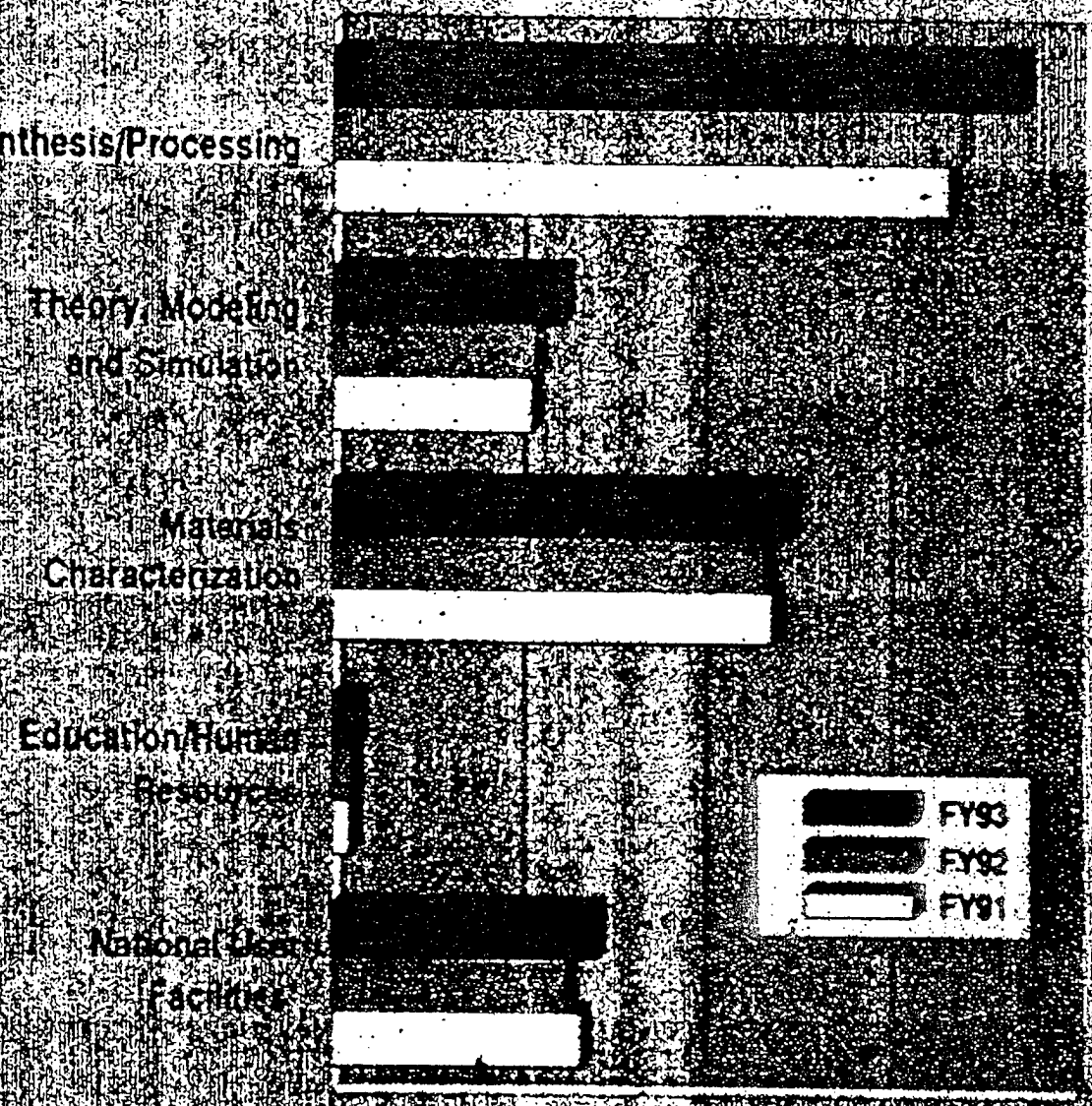
Total Federal Materials R&D Funding
All Agencies




Total Federal Materials R&D Funding
By Agency
DOC/NIST
DOD*
DOE*
DOI/BOM
DOT
EPA
HHS/NIH
NASA
NSF
USDA
FY93
FY92
FY91
0
100
200
300
400
500
600
700
Millions of Dollars
*Excludes classified research


Federal Materials Funding
By Materials Class
Bio/Biomolecular
Ceramics
Composites
Electronic
Magnetic
Metals
Optical/Photonic
Polymers
Superconducting
Other/Non-Specific
FY93
FY92
FY91
0
100
200
300
400
500
Millions of Dollars

Total Federal Materials R&D Funding By AMPP Research Components



Includes construction and operation costs

CROSSCUT - PROCESS

- The Crosscut Process Has Led to Significant Accomplishment in Advance of Any Budget Enhancement
- Increased Awareness of Materials Issues at High Levels in Agencies
- Increased Intraagency Planning and Coordination
- Increased Communication Amongst Agencies
- COMAT Role Strengthened
- Increased Awareness of Technical and Educational Areas Needing Attention

OPPORTUNITIES FOR FUTURE EMPHASIS IN AMPP

- Develop Programs Which Address All Breakthrough Areas
- Draw All Agencies Into Full Engagement
- Plan Applied Programs Demanding Private Sector Input
- Maintain and Strengthen Linkages Among Agencies
- Further Develop Linkages With Private Sector and Feedback Process

To obtain a copy of this document - send request to:
Committee on Industry and Technology/COMAT
c/o National Institute of Standards and Technology
Room B309, Materials Building
Gaithersburg, MD 20899
[Phone: (301) 975-5655)]

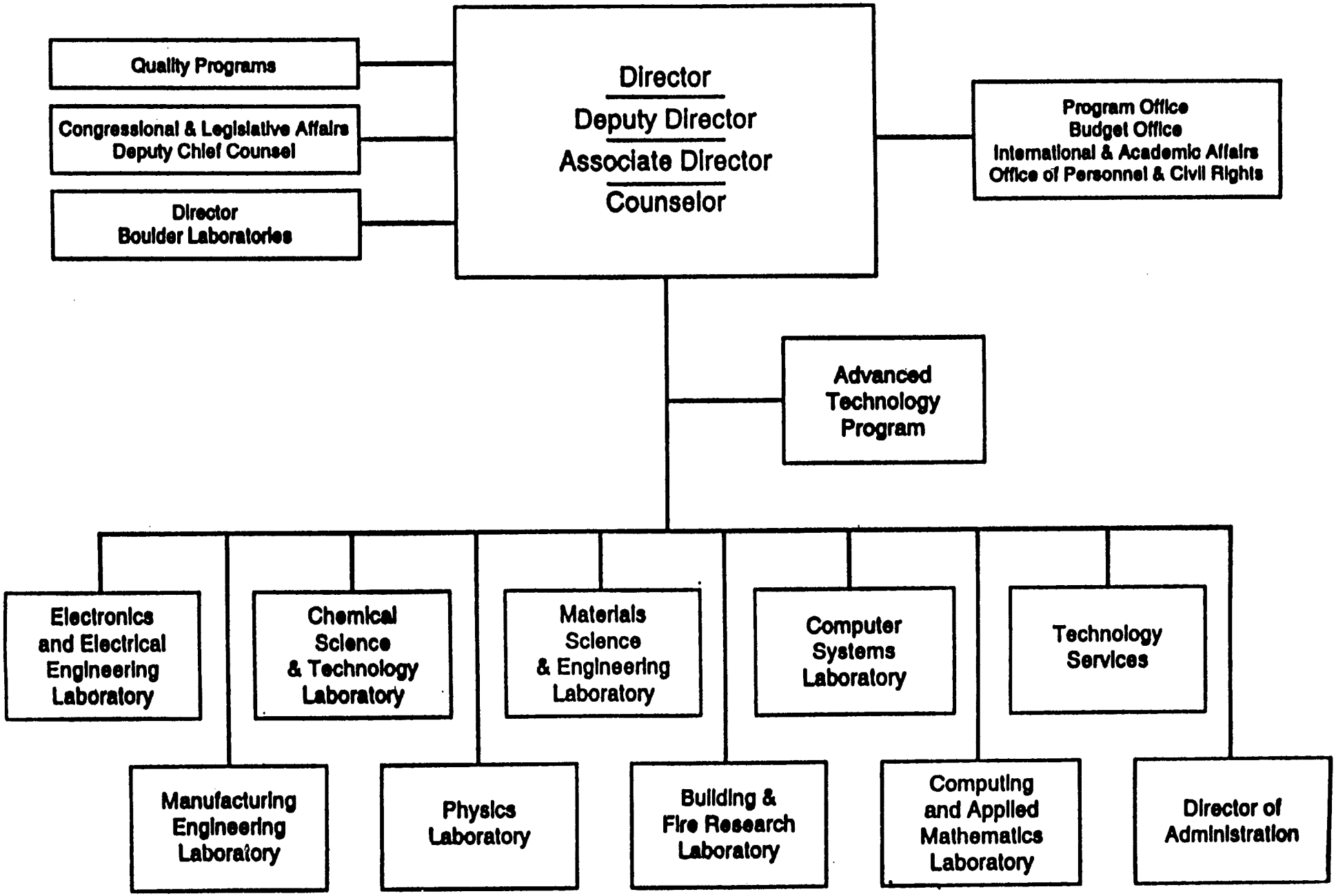
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
MATERIALS SCIENCE AND ENGINEERING LABORATORY

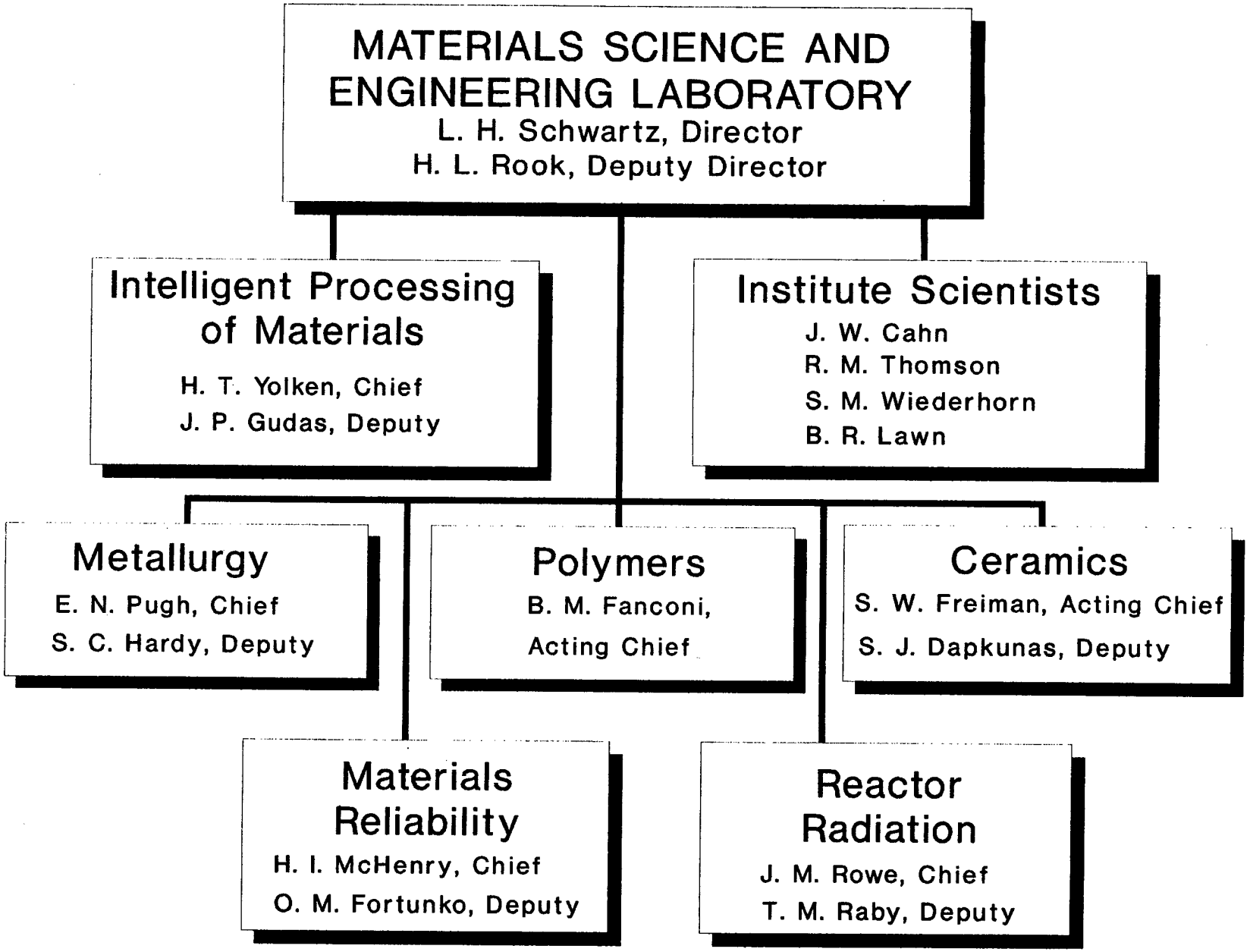
STRUCTURAL CERAMICS RESEARCH

S. J. DAPKUNAS
CERAMICS DIVISION

IACCSC
MAY 13, 1992

National Institute of Standards and Technology
ORGANIZATIONAL CHART

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H. L. Rook, Deputy Director
Intelligent Processing of Materials
H. T. Yolken, Chief
J. P. Gudas, Deputy
Institute Scientists
J. W. Cahn
R. M. Thomson
S. M. Wiederhorn
B. R. Lawn
Metallurgy
E. N. Pugh, Chief
S. C. Hardy, Deputy
Polymers
B. M. Fanconi, Acting Chief
Ceramics
S. W. Freiman, Acting Chief
S. J. Dapkunas, Deputy
Materials Reliability
H. I. McHenry, Chief
O. M. Fortunko, Deputy
Reactor Radiation
J. M. Rowe, Chief
T. M. Raby, Deputy

PROGRAM OBJECTIVES

- Provide models, measurement methods, standard reference materials and data to the advanced ceramics community.

- Assist industry in the development of technologies crucial for the cost effective manufacture of reliable ceramics.

STRATEGY

Through close interaction with industry and academia, identify technical barriers to implementation of advanced ceramics and underlying scientific issues. Utilize NIST capabilities and collaborative research with the ceramics community to address those issue

MAJOR STRUCTURAL CERAMICS ISSUES

Manufacturing Economics
- Powder Cost
- Machining Cost/Surface Condition

Reliability
- Improved Predictive Capability
- Cost Effective Test Methodology

Microstructural Modeling and Control

Availability of a Performance Database

Standards and Data

RESEARCH PROGRAM

Cost Effective Processing

- Powder Characterization and Intelligent Processing
- Ceramic Machining
- Low Temperature Si N Nanopowder Consolidation
- Ceramic Coatings (FGM)

Reliability and Property Measurement/Prediction

- Tensile Creep
- CMC Composite Modeling
- Microstructural Modeling
- Tribological Behavior

Standards and Data

- Powders-IEA, ASTM, Standard Reference Data
- Wear Maps, ACTIS
- Structural Ceramics Database

NIST STRUCTURAL CERAMICS FUNDING

	1991	1992	1993 (Est.)
DOC	2.8	2.62	3.3
Other Agency	2.1	2.38	
Industry	0.45	0.50	
	5.35	5.50	

Industrial Materials

Presentation to Interagency
Coordinating Committee on Structural Ceramics

Michael Lukasiewicz
Gas Research Institute

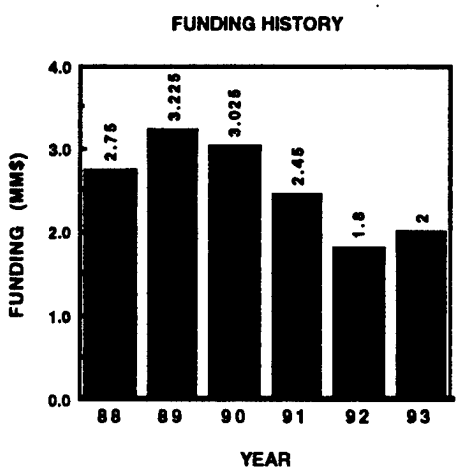
May 13, 1992
Arlington, VA

Industrial Materials: Objective

Develop cost-effective materials technology that enables industrial gas customers to improve productivity, product quality, and operating costs while meeting environmental regulations.

- Develop durable, reliable components and materials technologies for existing and emerging gas-fired process equipment.
- Reduce costs of advanced materials.
- Develop new materials to reduce pollution emissions and components to increase pollution control system durability.
- Improve scientific and engineering understanding of advanced materials.

Industrial Materials: Budgets



Industrial Materials: Strategy

Near-Term

Work with R&D Teams, including Manufacturers, to Develop and Field Test Advanced Materials and Components for Natural Gas-Fired Applications that are:

- Temperature and Corrosion Resistant
- Environmentally Acceptable
- Low Initial Cost
- Durable and Reliable

Long-Term

Establish Technology Base Projects to Provide New Concepts for Future Developments. Translate Basic Research into Practical Applications.

Industrial Materials: Research Emphasis

Strategic Element	No. of Projects	Budget (%)
Industrial Process Materials and Components	6	30
Material Design	1	10
Pollution Control Materials and Components	3	20
Technology Base	1	20
Power Systems Materials and Components	1	20
		100

- Nine Projects and Approximately 85 % of Budget is Dedicated to Structural Ceramics

Industrial Process Materials and Components

TITLE	CONTRACTOR	DESCRIPTION	STATUS
Materials for Gas-fired Boosting of Glass	Alfred University/ INEX and Lanxide	Identify materials solution for gas-fired boosting of glass.	Materials Screening test completed. Mo-MoSi2 deemed best.
Materials for Immersed Tube Aluminum Melting	Babcock & Wilcox/ BIRL	Identify materials solution for immersed htg. of alum.	Project initiated 4/92. Cofunded with project area 4.4.4..
Materials for High Pressure Methane Reformer	Stone & Webster/ CAM	Support DOE effort in high temperature, high pressure methane reform process.	Coupon corrosion tests initiated. Tube burst rig constructed.
Corrosion Resistant Coatings for Heat Exchanger Tubes	Solar Turbines	Develop oxide coating system for silicon carbide heat exchanger tubes.	Up to 3000 hrs tested on mullite coatings. Third 2000 hr test continuing.

Industrial Process Materials and Components

TITLE	CONTRACTOR	DESCRIPTION	STATUS
RBSC U-Tubes for Metals Furnaces	Coors	Develop RBSC U-Tubes for heat treat and reheat furnaces.	Field eval. with GM, Caterpillar, & Inland Steel.
SI-SIC U-tubes for Metals Furnaces	INEX	Develop INEX U-tubes for heat treat and reheat furnaces.	U-bends fabricated in cast and fired form. Shipped to burner company for testing.
New 1992 Starts	TBD	Furnace Components for High Temperature Furnaces.Application Assessment for Ceramic Radiant Tubes, and High Temperature Fibers for IR Burners	No new starts planned because of funding constraints.

Material Design

TITLE	CONTRACTOR	DESCRIPTION	STATUS
Carbon Filament Reinforced Composites Using Natural Gas	Penn State C.A.M./ Alcoa	Use Natural Gas to form Carbon Fibers in Pre-Shaped Molds. These preforms are then infiltrated with aluminum or ceramic to create composite components for industrial, power generation, and other end use areas.	Carbon fibers produced in unique catalytic reactor with natural gas feedstock.

Pollution Control Materials and Components

TITLE	CONTRACTOR	DESCRIPTION	STATUS
Direct Catalytic Reduction of NOx	Battelle	Investigate the feasibility of using low cost metallic fibers for direct conversion of NOx in industrial flue streams	Alloy materials Reduce NOx by 90%, however, reduction capabilities are zero in the presence of O2.
Low Cost Reticulated Ceramics for Industrial Burners	Hi-Tech Certamics	Development of Oxide Bonded Silicon Carbide for Applications Requiring $120/sqft max cost	Formulation of new material completed. Testing by Eclipse and refinement at Hi-Tech ongoing.
Reticulated Ceramics for Burners and also Catalyst Supports	Selee Corp.	Development of 3 Alternative Foam Ceramics for Burner and Catalyst Supports	Construction of combustion facility underway.

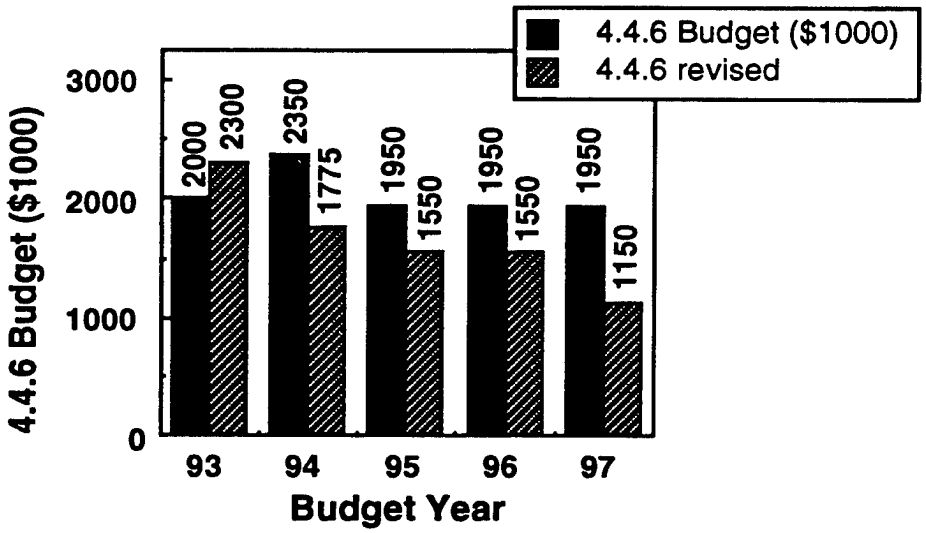
Technology Base

TITLE	CONTRACTOR	DESCRIPTION	STATUS
Center for Advanced Materials	Penn State University	Establish a Center for Advanced Materials development and characterization for accelerating application of advanced materials technology to natural gas users.	CAM is entering its 6th year as GRI support decreases. Industry and government support are increasing. Technology Transfer focus group meeting conducted. New manager of engineering services is facilitating advanced materials assistance to industry.

Power Systems Materials and Components

TITLE	CONTRACTOR	DESCRIPTION	STATUS
Ceramic Valve Seats for Reciprocating Engines	Southwest Research Institute	Apply Ceramic Valve Seats to Reduce Valve Train Wear in Power Generating Engines. This will yield lower operating costs and lower specific capital cost.	Norton/TRW selected as prime materials supplier. Coors/Eaton is backup supplier. Test facility design and equipment procurementt underway. NIST subcontract negotiations initiated.

Industrial Materials: Future Funding



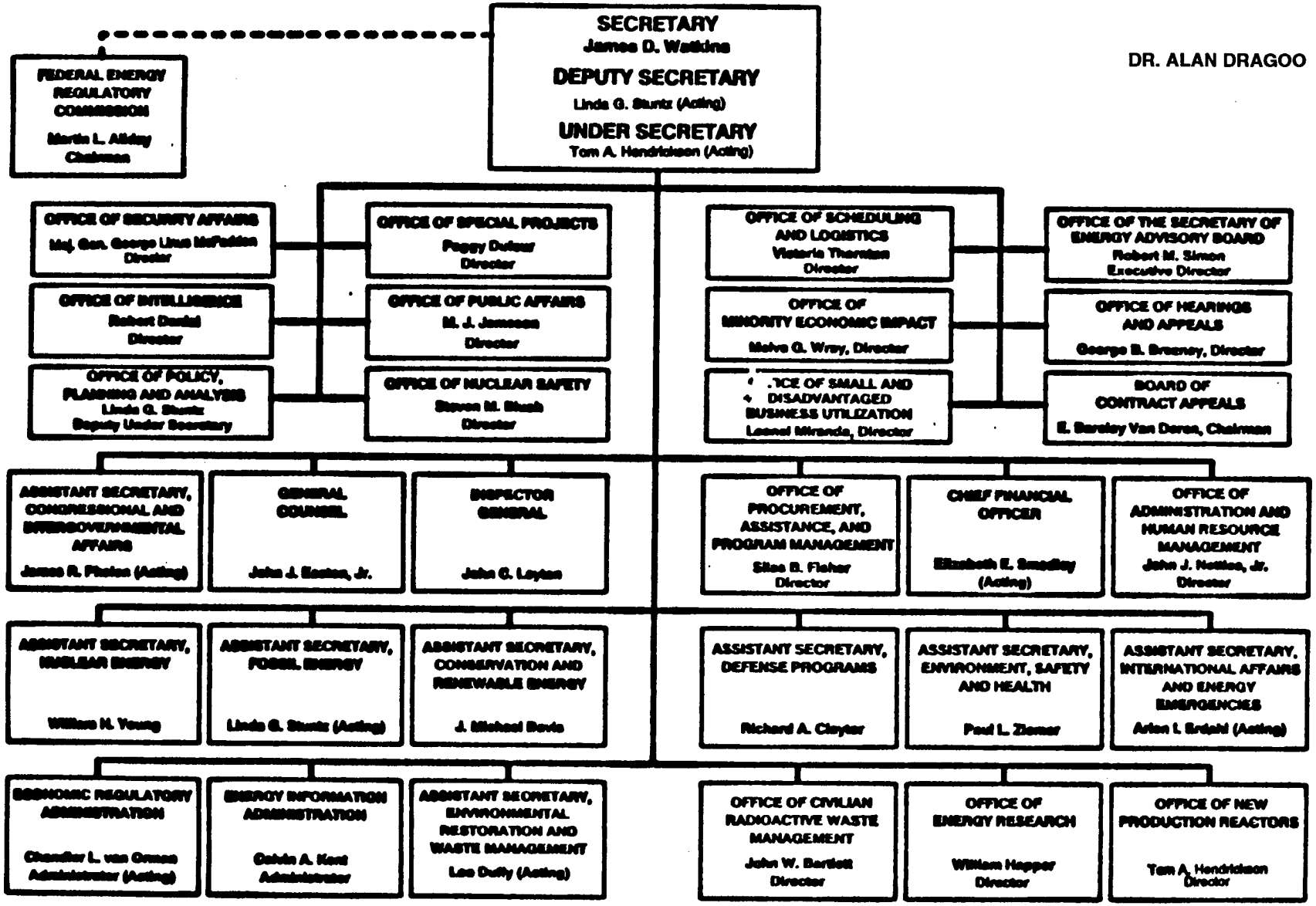
Industrial Materials: 1993 Funding Highlights

- 1993 Changes
  - Reduce First Year Funding of Ceramic Furnace Component R&D
- Outyear Revisions
  - Reduce Technology Base Projects to Minimum
  - Fund No Advanced Materials R&D Related to Prime Movers
  - Reduce Ultra High Temperature Radiant Tube R&D Efforts
- Implications to Structural Ceramics R&D
  - Percentage of Structural Ceramics Budget Declines to 60 % of Industrial Materials Program by 1996
  - Only Two Structural Ceramics Projects Scheduled in 1996 and 1997
  - GRI will seek to participate in cofunded projects to leverage limited funds in this area

THE DEPARTMENT OF ENERGY

U.S. DEPARTMENT OF ENERGY
OFFICE OF BASIC ENERGY SCIENCES

DR. ALAN DRAGOO



11/6/91

Prepared by Office of Energy Research
January 1994

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Principal Deputy Director

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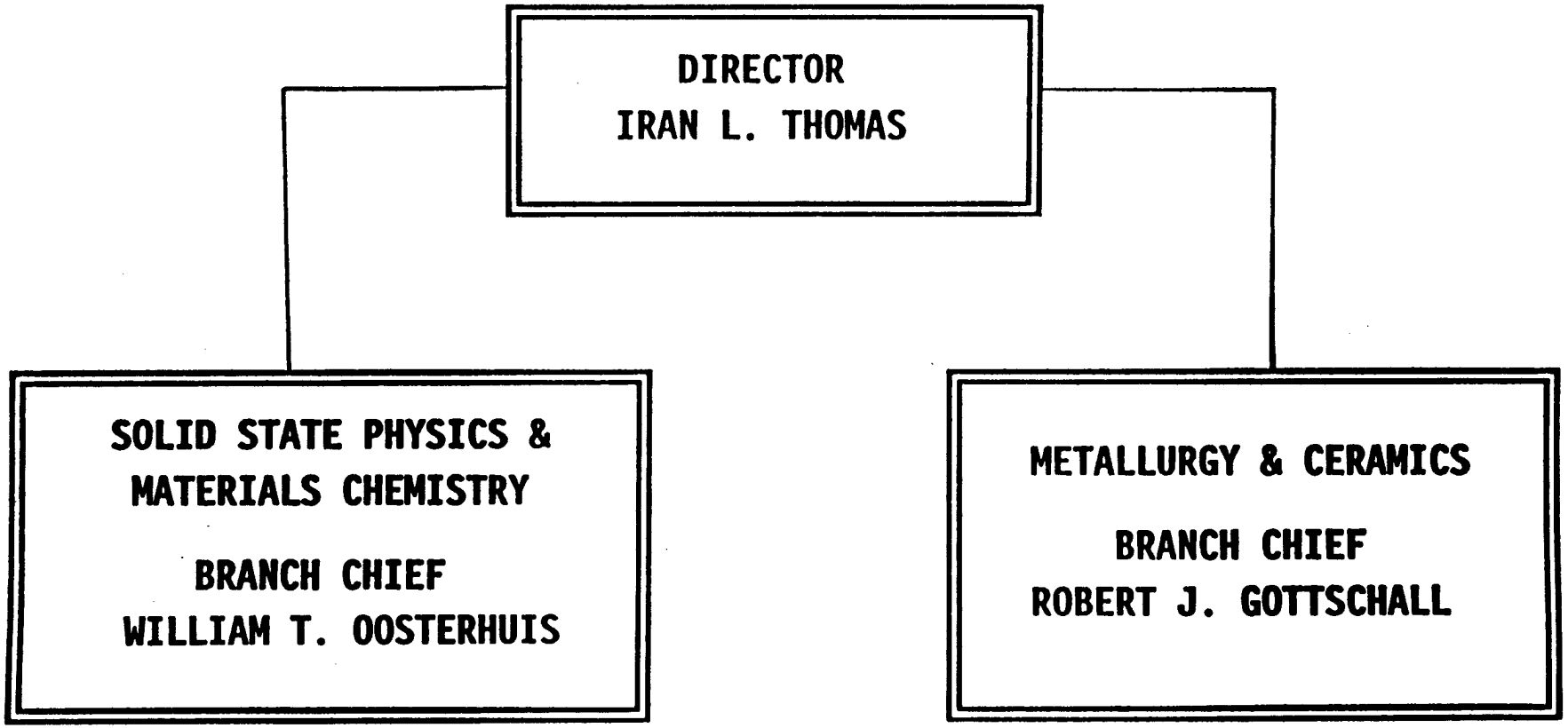
  Vacant, Director
- Precollege Programs Division
  Vacant, Director

DIVISION OF MATERIALS SCIENCES



MATERIALS SCIENCES

MODE OF OPERATION

STRATEGY:

- o USE APPROPRIATE EXPERTISE AT DOE LABORATORIES, UNIVERSITIES, AND INDUSTRY
- o MAINTAIN AN APPROPRIATE MIX OF RESEARCH AMONG ENERGY TECHNOLOGY SPECIFIC, MULTITECHNOLOGY, AND LONG TERM SCIENCE PROGRAMS
- o BALANCE THE FOREFRONT SMALLER RESEARCH PROGRAMS WITH MAJOR FACILITY RELATED ACTIVITIES

PROGRAM CHARACTERISTICS:

- o LABORATORIES - WORK IS GROUP ORIENTED, FACILITY ORIENTED, INTERACTIVE WITH TECHNOLOGIES
- o UNIVERSITIES - PROFESSOR/GRADUATE STUDENT ORIENTED, LONG TERM

FOCUS:

- o PANEL REPORTS
- o WORKSHOPS
- o STAFF ACTIVITIES
- o REVIEW PROCESSES

TECHNOLOGY/INFORMATION TRANSFER:

- o INFORMATION MEETINGS
- o RESEARCH ASSISTANCE TASK FORCE MEETINGS
- o PUBLICATIONS
- o CO-SITING OF APPLIED AND BASIC RESEARCH

MATERIALS SCIENCES OBJECTIVES

BASIC RESEARCH IN METALLURGY, CERAMICS, SOLID STATE PHYSICS, AND MATERIALS CHEMISTRY

- TO UNDERSTAND THE BEHAVIOR OF MATERIALS
- TO CREATE NEW OPPORTUNITIES IN MATERIALS
- TO UNDERPIN THE DEPARTMENT'S TECHNOLOGY PROGRAMS
- TO PLAN, CONSTRUCT, AND OPERATE UNIQUE FACILITIES NEEDED FOR MATERIALS RESEARCH

MATERIALS SCIENCES PROGRAM

METALLURGY AND CERAMICS ($59.5M):

- STRUCTURE OF MATERIALS
- MECHANICAL PROPERTIES
- PHYSICAL PROPERTIES
- RADIATION EFFECTS
- ENGINEERING MATERIALS

SOLID STATE PHYSICS ($50.6M)

- NEUTRON SCATTERING
- EXPERIMENTAL RESEARCH
- THEORETICAL RESEARCH
- PARTICLE-SOLID INTERACTIONS
- ENGINEERING PHYSICS

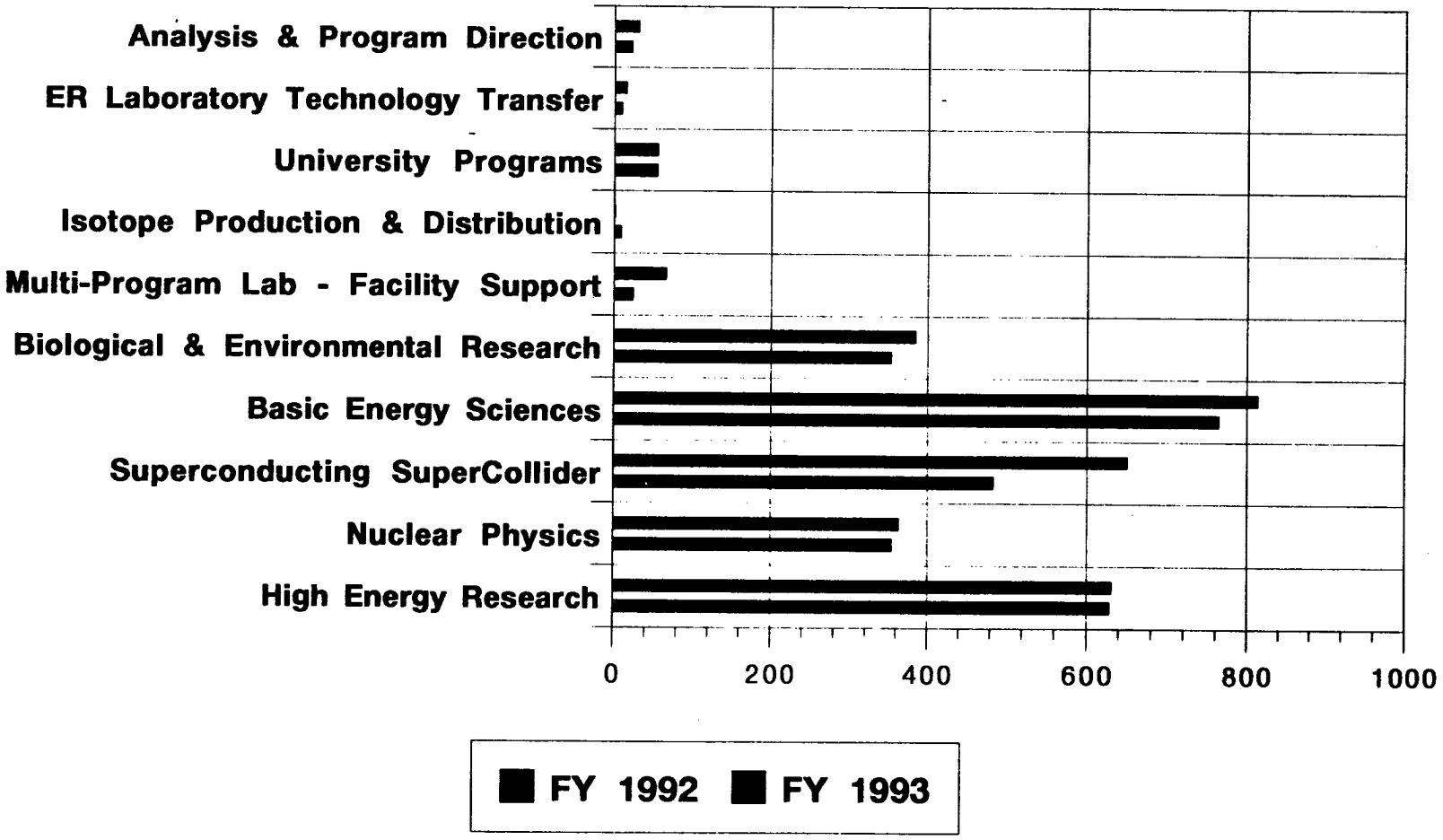
MATERIALS CHEMISTRY ($19.6M):

- SYNTHESIS AND CHEMICAL STRUCTURE
- POLYMER AND ENGINEERING CHEMISTRY
- HIGH TEMPERATURE AND SURFACE CHEMISTRY

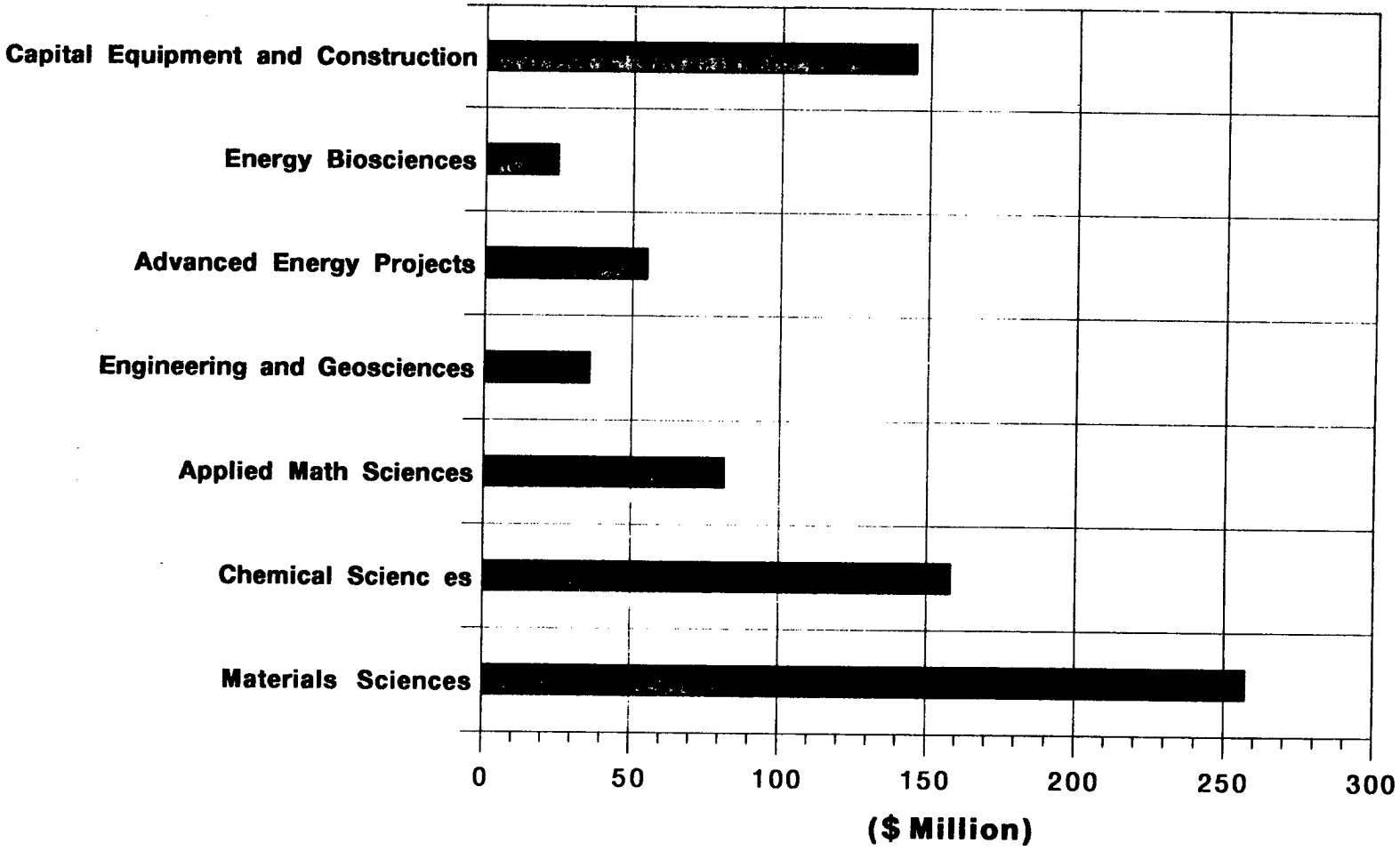
DOE COLLABORATIVE RESEARCH FACILITIES

- PRIMARY PURPOSE IS SCIENTIFIC RESEARCH.
- NO USER CHARGE FOR SCIENTIFIC INVESTIGATIONS PUBLISHED IN OPEN LITERATURE.
- COST RECOVERY FOR PROPRIETARY RESEARCH.
- SEVERAL BEAM LINES AT NATIONAL SYNCHROTRON LIGHT SOURCE AT BROOKHAVEN NATIONAL LABORATORY ARE DEDICATED FOR X-RAY PHOTO-LITHOGRAPHY PROPRIETARY RESEARCH.

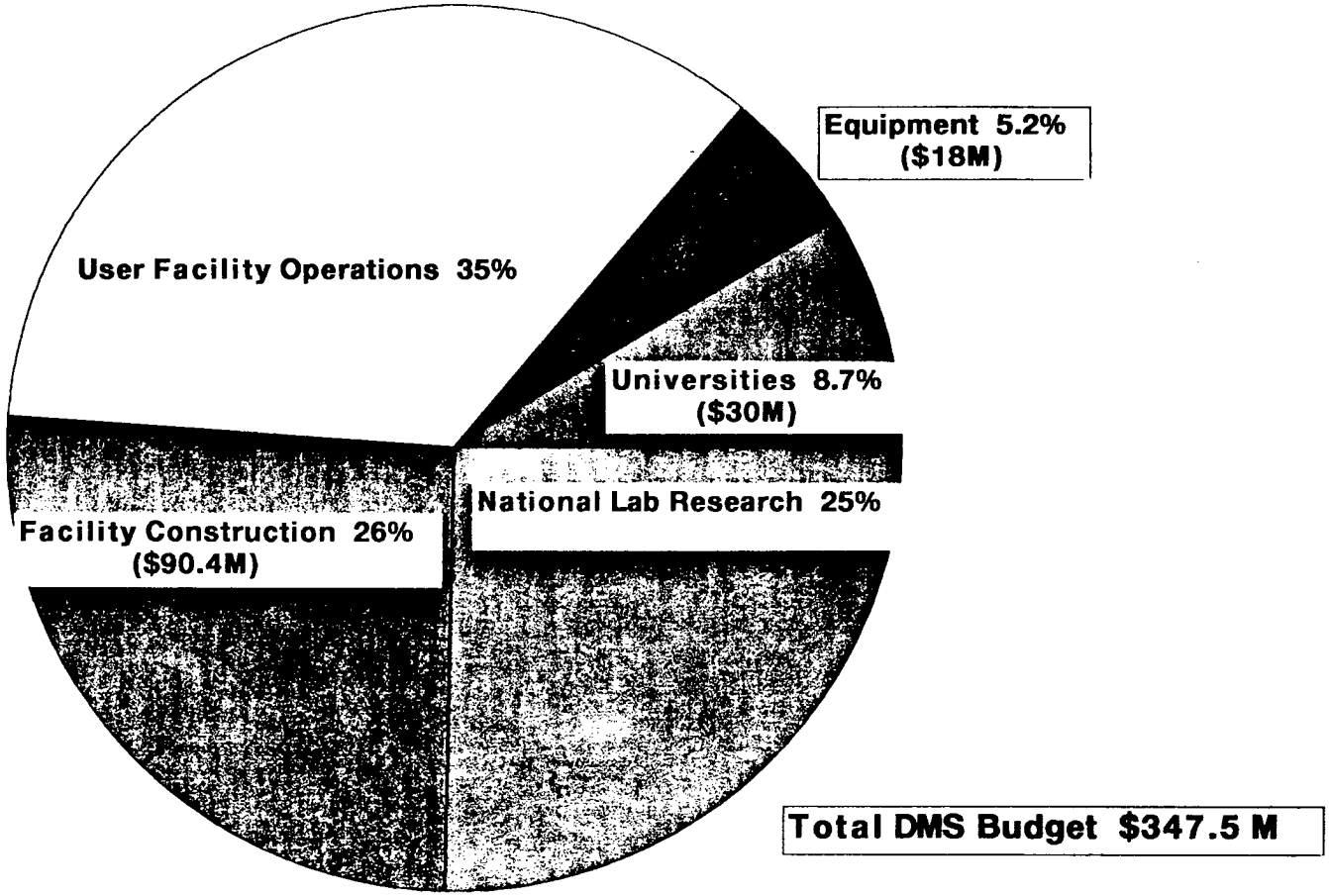
DOE Support for Fundamental Science Research ($M)



Basic Energy Sciences - FY1992



Division of Materials Sciences - FY 1992



ADVANCED MATERIALS AND PROCESSING PROGRAM

DIVISION OF MATERIALS SCIENCES

CENTER FOR EXCELLENCE FOR THE SYNTHESIS AND PROCESSING OF ADVANCED MATERIALS

- ESTABLISHED IN THE SUMMER OF 1991 THROUGH CONGRESSIONAL APPROPRIATION "TO ENHANCE SYNTHESIS AND PROCESSING IN THE DEPARTMENT OF ENERGY WITH THE OBJECTIVE OF DEVELOPING AND COMMERCIALIZING NEW ADVANCED MATERIALS."

- CENTER FOR EXCELLENCE IS COORDINATED BY SANDIA NATIONAL LABORATORIES AND INCLUDES: AMES LABORATORY, ARGONNE NATIONAL LABORATORY (ANL), BROOKHAVEN NATIONAL LABORATORY (BNL), UNIVERSITY OF ILLINOIS, LAWRENCE BERKELEY LABORATORY (LBL), LAWRENCE LIVERMORE NATIONAL LABORATORY (LLNL), LOS ALAMOS NATIONAL LABORATORY (LANL), NATIONAL RENEWABLE ENERGY LABORATORY (NREL), OAK RIDGE NATIONAL LABORATORY (ORNL), AND PACIFIC NORTHWEST LABORATORY (PNL).

- CENTER HAS 5 FOCUS AREAS:

  1. ATOMICALLY STRUCTURAL MATERIALS - BNL, LBL, LLNL, NREL, AND SNL (COORDINATED BY LBL)

  2. COMPLEX POLYMER SYSTEMS - AMES, ANL, BNL, LLNL, PNL, AND SNL (COORDINATED BY SNL)

  3. ADVANCED CERAMIC AND CERAMIC THIN FILMS - AMES, LANL, LBL, ORNL, AND SNL (COORDINATED BY ORNL)

  4. NANOPHASE MATERIALS - ANL AND UNIVERSITY OF ILLINOIS (COORDINATED BY UNIVERSITY OF ILLINOIS)

  5. EMERGING MATERIALS AND PROCESSING - AMES, ANL, BNL, LLNL, NREL, ORNL, AND SNL (COORDINATED BY AMES)

ADVANCED MATERIALS AND PROCESSING PROGRAM

DIVISION OF MATERIALS SCIENCES

CENTER FOR EXCELLENCE FOR THE SYNTHESIS AND PROCESSING OF ADVANCED MATERIALS

- **INDUSTRIAL PARTNERSHIPS AND COLLABORATIONS**
  - HEWLETT PACKARD
  - DOW CORNING
  - EVEREADY
  - IBM
  - EXXON
  - RAYTHEON
  - NORTON
  - Du PONT
  - SEMATECH
  - NANOPHASE TECHNOLOGIES
  - MICROELECTRONICS CENTER OF NORTH CAROLINA
  - GENERAL ELECTRIC
  - GENERAL MOTORS
- **2 CRADA'S SIGNED, SEVERAL MORE IN NEGOTIATION**

ADVANCED MATERIALS AND PROCESSING PROGRAM

MATERIALS SCIENCES SUBPROGRAM

FY 1993 REQUEST
(DOLLARS IN THOUSANDS)
B/A

OPERATING EXPENSES

	FY 1992	FY 1993	Δ
RESEARCH	$133,074	$148,000	+ 14,926
FACILITIES	$100,442	$121,227	+ 20,785
ANS	$ 23,600	$ 21,000	- 2,600
TOTAL	$257,116	$290,227	+ 33,111

METALLURGY AND CERAMICS THRUST AREAS

MICROCHARACTERIZATION

- MICROSTRUCTURE AND MICROCHEMICAL ANALYSIS
- DEFECT ANALYSIS INCLUDING POINT DEFECTS, DISLOCATIONS, GRAIN BOUNDARIES, RADIATION DAMAGE ANALYSIS, ETC.
- OPERATE FOUR NATIONAL USER CENTERS FOR MICROCHARACTERIZATION (ARGONNE NATIONAL LABORATORY, OAK RIDGE NATIONAL LABORATORY, LAWRENCE BERKELEY LABORATORY, UNIVERSITY OF ILLINOIS)

MECHANICAL BEHAVIOR

- RESPONSE AND BEHAVIOR UNDER APPLIED STRESS

PHYSICAL BEHAVIOR

- DIFFUSION AND TRANSPORT PHENOMENA
- SURFACE BEHAVIOR, CORROSION
- ELECTRONIC BEHAVIOR
- MAGNETIC BEHAVIOR
- SUPERCONDUCTING BEHAVIOR
- SEMICONDUCTING BEHAVIOR

RADIATION EFFECTS

- MECHANISMS OF IRRADIATION DAMAGE
- MODELING OF IRRADIATION INDUCED DEGRADATION

OTHER

- WELDING AND JOINING SCIENCE
- NON-DESTRUCTIVE FLAW DETECTION AND ANALYSIS
- SYNTHESIS AND PROCESSING

BES/DMS CERAMIC MATERIALS BUDGET

o	SUPERCONDUCTING CERAMICS	40%
o	STRUCTURAL CERAMICS	25%
o	ELECTRICAL CERAMICS	20%
o	OTHER	15%

DOE/BES PROGRAMS IN STRUCTURAL CERAMICS

PERFORMING ORGANIZATIONS -- NATIONAL LABORATORIES, UNIVERSITIES, AND INDUSTRY

OBJECTIVE -- INCREASE THE BASIC UNDERSTANDING OF MATERIALS PROPERTIES, BEHAVIOR, AND PHENOMENA IN STRUCTURAL CERAMICS. DEVELOPMENT OF NEW FACILITIES AND ANALYTICAL INSTRUMENTS FOR PROCESSING, CHARACTERIZATION, AND TESTING. UNDERSTAND SYNERGISTIC RELATIONSHIP BETWEEN PROPERTIES/ BEHAVIOR, STRUCTURE, AND PROCESSING PARAMETERS.

DMS/BES STRUCTURAL CERAMICS BUDGET

(IN THOUSANDS OF DOLLARS)

	FY 1990	FY 1991	FY 1992
NATIONAL LABS	$5,463	$6,494	$5,632
UNIVERSITY GRANTS	1,829	1,569	1,304
INDUSTRY GRANTS	247	247	206
SBIR PROJECTS	50	100	50
TOTAL	$7,589	$8,410	$7,192

FY 1992

DMS/BES STRUCTURAL CERAMICS

NATIONAL LABORATORIES	17 PROGRAMS
UNIVERSITIES	16 PROGRAMS
INDUSTRY	2 PROGRAMS
TOTAL	35 PROGRAMS

DMS/BES STRUCTURAL CERAMICS BUDGET

RESEARCH AREAS (%)

	FY 1991	FY 1992
SYNTHESIS/PROCESSING	43	42
MECH. PROPERTIES/FRACTURE	24	24
CHEM. REACTIONS/TRANSPORT	4	5
ATOMIC STRUCTURE/μ STRUCTURE	14	15
THEORY/MODELING	2	3
IRRADIATION EFFECTS	13	11

DIVISION OF MATERIALS SCIENCES

PUBLICATIONS, GRADUATE STUDENTS, AND POSTDOCTORAL

- ABOUT 4,000 PAPERS PUBLISHED IN FY 1990
- ABOUT 900 PRINCIPAL INVESTIGATORS SUPPORTED THROUGH 430 PROJECTS
- ABOUT 890 GRADUATE STUDENTS AND 300 POSTDOCTORALS SUPPORTED

BES/DMS STRUCTURAL CERAMICS PROGRAM

SUMMARY OF RESEARCH PROGRAMS FY 1991

SYNTHESIS AND PROCESSING

NATIONAL LABS

AMES LABORATORY
Iowa State University
Ames, IA 50011

(60%)[a]

20. SYNTHESIS AND CHARACTERIZATION OF NEW MATERIALS
J. D. Corbett, R. A. Jacobson, R. E. McCarley
(515) 294-3086 03-1 $550,000

Synthesis, structure and bonding in intermetallic systems–new Zintl phases, new ternary compounds stabilized by interstitials. The effect of common imparities on stability. Systematic variation of conduction, magnetic, and corrosion resistance properties. Synthesis, bonding, structure and properties of new ternary oxide phases containing heavy transition elements, especially metal-metal bonded structures stable at high temperatures. Low temperature routes to new metal oxide, sulfide and nitride compounds. Correlation of structure and bonding with d-electron count and physical properties. Development of diffraction techniques for single crystal and non-single crystal specimens, techniques for pulsed-neutron and synchrotron radiation facilities, and use of Patterson

IDAHO NATIONAL ENGINEERING LABORATORY
Idaho Falls, ID 83415

63. STRESS DISTRIBUTION IN GRADED MICROSTRUCTURES
B. H. Rabin
(208) 526-0058 01-5 $235,000

Develop fundamental understanding of the effects of microstructure, processing conditions, and specimen geometry on the residual stresses in graded materials intended to mitigate the effects of the mismatch in properties at dissimilar material interfaces. Fabrication of two-phase materials with controlled microstructural gradients and varying geometries by electron beam coevaporation and powder metallurgy techniques. Focus on materials systems in which significant property mismatch exists between components, e.g., Al_2O_3/Ni and Si/Ag. Measurement of residual stresses by high spatial resolution X-ray diffraction methods using synchrotron source. Comparison of experimental results with predictions from elastic-plastic finite element modeling of stress distributions.

Number in parantheses is the estimated percentage for structural ceramics research. If amount is not indicated, then the research is assumed to be concerned totally with structural ceramics.

UNIVERSITY OF ILLINOIS MRL
104 S. Goodwin Avenue
Urbana, IL 61801

88. PROCESSING OF MONODISPERSE CERAMIC POWDERS
C. Zukoski
(217) 333-7379 01-3 $277,386 (30%)

Low temperature processing of ceramics including precipitation of monodisperse oxide powders, rheology of monodisperse powders and mixtures, and studies of forces in colloidal suspensions, for the purpose of forming low flaw density, high performance ceramics.

LAWRENCE BERKELEY LABORATORY
1 Cyclotron Road
Berkeley, CA 94720

110. CAM CERAMIC SCIENCE PROGRAM
Lutgard C. De Jonghe
(510) 486-6138 01-1 $1,302,000

The CAM Ceramic Processing Science Program has three linked objectives: the development of predictive, quantitative theories of densification and microstructure development in heterogeneous powder compacts, the application of these theories to produce advanced structural ceramics with improved high temperature performance, and the evaluation of the mechanical properties of these ceramics. It develops model experiments that facilitate investigation of fundamental aspects of microstructural development and processing, and their application of model ceramic systems. It develops models and means for initial powder compact structural control including the production and use of coated powders; it examines the microstructural evolution and control during densification in relation to interface properties; it produces particulate ceramic composites based on SiC, and it tests mechanical properties of such ceramics in particular high temperature creep and fatigue.

LOS ALAMOS NATIONAL LABORATORY
P. O. Box 1663
Los Alamos, NM 87545

146. SYNTHESIS AND PROCESSING OF SINGLE CRYSTAL SAPPHIRE FILAMENTS
W. R. Blumenthal
(505) 667-0986 01-5 $235,000

The goal of this project is two-fold and uses a multiple disciplinary approach to study single crystal sapphire filaments for potential use as high temperature creep resistant composite reinforcements. One objective is to empirically relate growth parameters used to control the Edge-defined

Film-growth (EGF) and the Laser-heated Floating Zone processes to resulting microstructures and mechanical properties. A more challenging objective is to model the EFG process in order to not only optimize growth conditions for sapphire, but also for other candidate reinforcement oxides (e.g., YAG). Microstructural and mechanical property characterization meachanisms controlling filament strength.

OAK RIDGE NATIONAL LABORATORY
P. O. Box 2008
Oak Ridge, TN 37831-6117

176. SYNTHESIS AND PROPERTIES OF NOVEL MATERIALS
L. A. Boatner, M. M. Abraham, C. B. Finch, H. E. Harmon, J. O. Ramey, B. C. Sales
(615) 574-5492 02-2 $1,300,000

Preparation and characterization of advanced materials including the growth of single crystals and the development of new crystal growth techniques; development of new materials through the application of enriched isotopes; investigations of the physical, chemical, and thermal properties of novel materials using the techniques of thermal analysis, X-ray diffraction, Mossbauer spectroscopy, ion implantation and ion channeling, optical absorption, high performance liquid chromatography, EPR, and X-ray or neutron scattering; application of materials science techniques to the resolution of basic research problems; preparation and characterization of high-T_c superconducting oxides; synthesis and investigation of phosphate glasses; development and characterization of advanced ceramics; solid state epitaxial regrowth; growth of perovskite structure oxides, high temperature materials (MgO, CaO, Y_2O_3), refractory metal single crystals (Ir, Nb, Ta, V), fast-ion conductors, stainless steels, rapid solidification and microstructures.

180. THEORY OF CONDENSED MATTER
J. F. Cooke, J. H. Barrett, H. L. Davis, R. Fishman, K. Rensberg, M. S. Jonon, T. Kaplan, S. H. Liu, G. D. Mahan, G. D. Mostoller, O. S. Oen, S. Pettersson, M. Rasolt, M. T. Robinson, J. C. Wang, R. F. Wood
(615) 574-5787 02-3 $949,000

Theory of nonequilibrium solidification in semiconductors, lattice vibrations in metals and alloys, lattice dynamics and potential energy calculations of ionic crystals, computer simulation of radiation damage, sputtering, and ion implanation surface studies with backscattered ions, development of LEED theory and interpretation of LEED data, surface vibrations and relaxation, electronic structure of metal surfaces, magnetism in transition metals and local moment systems, neutron scattering at high energies, electronic properties of mixed-valent and heavy fermion systems, high temperature superconductivity, critical phenomena and phase transitions quantum Hall effect, diffusion and elastic vibrations of fractal systems, and self-organized critical systems. New directions include a study of the surface structure of disordered systems and the development of molecular dynamics theory and relevant computer programs for treating interfaces and, ultimately, crystal growth.

188. CHEMISTRY OF ADVANCED INORGANIC MATERIALS
E. J. Kelly, C. E. Bamberger, J. Brynestad, L. Maya, C. E. Vallet
(615) 574-5024 03-1 $1,083,000 (50%)

Application of ion implantation and ion beam mixing to the generation and systematic study of surface-modified materials of interest as catalysts, e.g., $M_xTi_{1-x}O_2/Ti$ (M = Ru, Ir, Rh, etc.) for electrocatalysis of Cl_2 and O_2 evolution; determination of the reaction mechanism, the specific catalytic activities, and the electronic properties of the catalysts via electrochemical, Rutherford backscattering, and in situ photoacoustic and photocurrent spectroscopic techniques. Development of new generalized methodologies for the synthesis of nonoxidic ceramic materials (BN, Si_3N_4, SiC, C-B-N ternaries, and borides, carbides, carbonitrides, and nitrides of the transition metals of groups 4, 5, and 6) in powder, fiber, film, or whisker forms; pyrolysis or photolysis of inorganic or organometallic precursors; synthesis of semiconducting C-N-B thin films via pyrolysis of borazine derivatives. Synthesis of TiN whiskers via reactions of titanates with NaCN or NH_3 at high temperatures; topochemically specific solid-state reactions. Synthesis and characterization of high-T_c superconducting oxides; composition/structure/property relationships and their utilization in optimization of synthesis/processing.

PACIFIC NORTHWEST LABORATORY
P. O. Box 999
Richland, WA 99352

193. MICROSTRUCTURAL MODIFICATION IN CERAMIC PROCESSING USING INORGANIC POLYMER DISPERSANTS
G. J. Exarhos, W. D. Samuels, I. A. Aksay (U. of Wash)
(509) 375-2440 01-1 $462,000

Fundamental studies of particle compaction phenomena in colloidal processing of ceramics using inorganic polymer dispersants. Localized particle-polymersolvent interactions probed by means of in situ molecular spectroscopic measurements. Integration of spectroscopic data with particle compaction measurements is used to evaluate packing efficiency and relate it to chemical functionality of derivatized inorganic polymer dispersants. Response of bound polymers in the greenbody to high temperatures during sintering also is being investigated. Improvement in mechanical properties of the fired ceramic is correlated with void density and distribution which evolve during processing and with the generation of interfacial phases formed by incorporation of the polymer dispersant with the ceramic matrix.

199. CERAMIC COMPOSITE SYNTHESIS UTILIZING BIOLOGICAL PROCESSES
P. C. Rieke, A. I. Caplan, A. I. Caplan, B. J. Tarasevich, A. H. Heuer (Case Western)
(509) 375-2833 03-1 $694,000

Studies of natural formation of hard tissue that use polymers as templates to control and orient ceramic crystal growth. Crystal growth on modified polymer surfaces and cell control of crystal growth. Surface, interface, and colloid chemistry of small atom cluster. Modeling of polymer surfaces and interactions with ions in solution.

UNIVERSITIES

CLARK ATLANTA UNIVERSITY
Atlanta, GA 30314-4381

256. THE SYNTHESIS, CHARACTERIZATION AND CHEMISTRY OF SI-C-N-O-M CERAMIC AND COMPOSITE POWDERS
Y. H. Mariam, Department of Chemistry
(404) 880-8593 01-3 $66,786

Preparation of Si-C-N-O-M/Si-C-N-M systems, where M=Ti or Zr, from silazane/organosilylamine polymer precursors. Molecular and chemical structures, microstructures, composition, morphology and microcrystallinity of powders investigated by SEM, TEM, EXAFS, EXELFS, etc. Detailed nitridation followed by physical- and chemical-state characterization. Computational modeling of certain reactions relevant to nitridation, decomposition and cross-linking performed using semiempirical molecular orbital methods to obtain reaction enthalpies, activation enthalpies and entropies, and potential energy surfaces. Modeling studies coupled with TGA/FTIR, decomposition kinetics, evolved gas analysis to investigate role of chemical reactivity and structure in formation chemistry of ceramic and composite powders.

INDUSTRY

SOUTHWEST RESEARCH INSTITUTE
San Antonio, TX 78284

403. CHARACTERIZATION OF PORE EVOLUTION IN CERAMICS DURING CREEP FAILURE AND DENSIFICATION

R. A. Page, Department of Materials and Mechanics
(512) 522-3252

K. S. Chan, Department of Materials and Mechanics
(512) 522-2053 01-2 $124,235

Characterization of pore evolution during sintering and cavitation during creep. Objectives of the sintering study are the statistical characterization of pore evolution during densification, identification of primary variables affecting pore removal, and development and evaluation of sintering models. Objectives of the creep study are to understand the effects of microstructural parameters and loading mode, including uniaxial tension, on the kinetics of various creep mechanisms, such as grain boundary sliding and cavity growth. Small angle neutron scattering (SANS) measurements (supplemented by TEM, SEM, precision density, and AES characterization), tensile-creep measurements, and grain boundary sliding measurements (using stereo-imaging technique). Cavity size, distribution, morphology, and nucleation and growth rates determined by SANS analysis. Materials investigated included alumina and silicon carbide.

SBIR

NANOPHASE TECHNOLOGIES CORPORATION
8205 South Cass Ave.
Darien, IL 60559

445. NET SHAPE FORMING OF NANOPHASE CERAMICS FOR MECHANICAL APPLICATIONS

J. C. Parker
(708) 963-0282 Phase I SBIR $50,000

Demonstrate net shape forming of various nanophase ceramics; characterization of resulting shapes with respect to hardness, fracture toughness, porosity and mechanical strength; development of database on mechanical and modeling properties of nanophase ceramics. Metal oxide powders prepared by gas phase condensation process; consolidated into simple shapes at different temperatures and pressing conditions; property characterization.

SYNERGETIC MATERIALS, INC.
P. O. Box 5574
Auburn, CA 95604

448. PLASTIC BEHAVIOR AND PROPERTIES OF TITANIUM CARBIDE AND TITANIUM CARBONITRIDE MONOLITHIC AND COMPOSITE MATERIALS

D. C. Halverson
(916) 823-0238 Phase I SBIR $50,000

Plastic behavior and property-microstructure relationships in titanium carbide (TiC) and titanium carbonitride (TiCN) materials. Monlithic TiC and TiCN ceramics synthesized using self-propagating high temperature synthesis (SHS); TiC-metal and TiCN-metal composites synthesized using SHS and liquid metal infiltration (LMI). Microhardness, macrohardness, fracture toughness; microstructure evaluation using metallographic, X-ray diffraction and electron microprobe analysis.

MECHANICAL PROPERTIES, FRACTURE AND FATIGUE

NATIONAL LABS

AMES LABORATORY
Iowa State University
Ames, IA 50011

5. MECHANICAL BEHAVIOR OF MATERIALS
W. A. Spitzig, B. Biner, J. Kameda
(515) 294-5082 01-2 $388,000 (30%)

Studies of the effects of environment and stress on the mechanical properties of metals, intermetallics, and ceramic composites. Effects of hydrogen on cracking in alloys under uniaxial and cyclic loading conditions. Interstitial effects on strength and ductility in both nonhydrogenated and hydrogenated V, Nb, and Ta. High-temperature-induced intergranular cracking in Ni base alloys. Effects of radiation-induced defects and solute segregation on intergranular embrittlement. Modeling of hydrogen embrittlement. Description of three dimensional arrays of defects and relationship of arrangement to ductility and mechanical properties. Correlation between defect structure and nondestructive measurement.

UNIVERSITY OF ILLINOIS MRL
104 S. Goodwin Avenue
Urbana, IL 61801

82. HIGH TEMPERATURE MECHANICAL BEHAVIOR OF CERAMICS
D. F. Socie
(217) 333-7630 01-2 $90,910

Behavior of engineering materials subjected top complex loading involving high temperatures, multiaxial state of stress, and time dependent state of stress. Macroscopic damage models are being developed on the basis of microscopic studies of defects accumulated in the materials. High temperature mechanical properties of ceramics under uniaxial, multiaxial, and fatigue conditions.

83. SUBCRITICAL CRACK GROWTH IN STRUCTURAL CERAMICS
J. F. Stubbins
(217) 333-6474 01-2 $80,884

Micromechanisms of failure at elevated temperatures under creep, fatigue and aggressive environmental conditions. Role of oxide films on crack initiation and propagation. Microstructural examination of regions in front of cracks and of the dislocation structures are related to micromechanics of failure. Crack propagation kinetics in ceramics at high temperatures and in aggressive atmospheres. Subcritical crack growth in ceramics.

OAK RIDGE NATIONAL LABORATORY
P. O. Box 2008
Oak Ridge, TN 37831-6117

168. TOUGHENING AND RELATED PROCESSING MECHANISMS IN CERAMICS
P. F. Becher, V Alexander, A. Bleier, C.-H. Hsueh
(615) 574-5157 01-5 $986,000

Experimental and theoretical approaches are being developed to provide new insights into mechanisms which improve the toughness, strength, and elevated temperature mechanical performance of ceramics with companion studies in ceramic processing leading to controlled densification, microstructures and compositions, in such toughened systems. The pertinent micro- and macro-scopic characteristics are directly related to phenomena that are controlled during powder synthesis, powder processing, and densification. Thus, this task incorporates interdisciplinary studies of the fundamental descriptions of powder synthesis and processing and their influence on densification mechanisms and microstructure evolution during densification. These are directly coupled with studies of the role of microstructure, composition, and defects in the mechanical behavior of ceramics and descriptions of toughening-strengthening and related mechanisms. A primary consideration of these studies is providing the fundamental insights for design and fabrication of ceramics and ceramic composites (e.g., transformation and second phase toughening behaviors).

UNIVERSITIES

BROWN UNIVERSITY
Providence, RI 02912

222. FATIGUE CRACK GROWTH UNDER FAR-FIELD CYCLIC COMPRESSION
S. Suresh, Division of Engineering
(401) 863-2626 01-2 $93,405

Experimental and theoretical investigation of stable crack growth under static and cyclic tensile loads in monolithic and ceramic matrix composites up to 1500 C. Effects of loading rate/cyclic frequency, hold time, cyclic means stress and test temperature on rates of subcritical crack growth; characterization of crack advance by fracture mechanics; characterization of crack-tip damage by transmission electron microscopy; effects of pre-existing amorphous films and amorphous films formed at test temperature. Finite element simulations of evolution of cyclic damage zones ahead of tensile fatigue cracks using constitutive formulations to represent experimentally determined damage mechanisms.

UNIVERSITY OF CALIFORNIA AT SANTA BARBARA
Santa Barbara, CA 93106

235. STRUCTURE AND CHEMISTRY OF METAL/CERAMIC INTERFACES
A. G. Evans, Materials Department
(805) 961-8275 01-1 $52,746 (24 months)

Different metals and ceramics joined under well-defined, instrumented, bonding conditions. Reaction layers for different metal/ceramic combinations identified and quantified by analytical electron microscopy. Defect structure determined by high resolution electron microscopy. Theoretical models of bonding and chemistry of interfaces.

CARNEGIE MELLON UNIVERSITY
3325 Science Hall
Pittsburgh, PA 15213

249. THEORETICAL MODELS FOR THE ULTIMATE STRENGTH AND FLAW RESISTANCE OF UNDIRECTIONALLY-REINFORCED CERAMIC-MATRIX COMPOSITES
P. S. Steif, Department of Mechanical Engineering
(412) 268-3507 01-2 $98,500

Theoretical study of microstructural determinants of strength and toughness in fiber-reinforced ceramics. Macroscopic properties include: the ultimate tensile strength parallel to the fibers and the resistance to matrix flaws which propagate normal to the fiber direction. Understanding of the extent to which these macroscopic properties depend on critical micro-level properties, including, the character of the fiber-matrix interface, as well as the fiber and matrix moduli, strength and strength variability. Theoretical approach to incorporate the influence of the interface via micro-mechanics models of the interface, that reflect either the presence of chemical bonding or the possibility of interfacial slippage.

UNIVERSITY OF DENVER
Denver, CO 80208

279. RESIDUAL STRESSES IN FIBER-REINFORCED CERAMIC COMPOSITES BY DIFFRACTION METHODS
P. K. Predecki, Department of Engineering
(303) 871-3570 01-2 $59,160

Residual stresses and strains in ceramic fiber/ceramic matrix composites by X-ray diffraction to obtain near surface stresses and by neutron diffraction to obtain the bulk microstresses in each crystalline phase. Diffraction measurements as function of temperature on well-characterized specimens in which either the thermal expansion of the matrix or the fiber surface treatment has been varied. Materials investigated include alumina fibers in silicate glasses and SiC whiskers in alumina. Noyan-Cohen analysis accounting for 3-dimensional nature of stresses and including, where possible, separation of macrostresses and microstress components in each phase. Results correlated with mechanical properties and thermal expansion via existing models for composite behavior. Objective is to provide a test for such models and to see if the techniques are useful for predicting strength, toughness, and thermal expansion of these materials.

MICHIGAN STATE UNIVERSITY
East Lansing, MI 48824

319. THEORETICAL STUDIES OF BREAKDOWN IN RANDOM MEDIA
P. M. Duxbury, Department of Phyics and Astronomy
(517) 353-9179 01-3 $64,000 (50%)

Scaling theories and numerical algorithms for predicting structure/extreme property relationships in random media. Use of concepts in statistical mechanics, disordered systems and nonequilibrium growth in conjunction with fracture mechanics, damage mechanics and dielectric breakdown to develop unified perspective of breakdown phenomena. Development of generic models and general methodology, and treatment of specific breakdown problems.

PENNSYLVANIA STATE UNIVERSITY
University Park, PA 16802

371. THE MECHANICAL BEHAVIOR OF SURFACE MODIFIED CERAMICS
D. J. Green, College of Earth and Mineral Sciences
(814) 863-2011 01-2 $80,000

Modification of surface layers of ceramics to introduce surface compression and increase hardness and fracture toughness of transformation toughened ZrO_2 and Al_2O_3. Surface infiltration when ceramic is pressed or partially sintered. Development of a second phase surface layer during final densification. Indentation cracking used to study crack nucleation and growth and determine fracture toughness. Stress and composition profiles determined by NSLS X-ray diffraction data.

STANFORD UNIVERSITY
Stanford, CA 94305-6060

407. A STUDY OF MECHANICAL PROCESSING DAMAGE IN BRITTLE MATERIALS
B. T. Khuri-Yakub, Department of Electrical Engineering
(415) 723-0718 01-5 $60,000

Study of mechanical damage and surface residual stresses associated with mechanical processing of brittle materials. Study of how defects in the green state of ceramic components evolve. Also, machine damage on curved surfaces. Development of a theory for calculating the behavior of surface wave propagation on curved surfaces. Materials include silicon nitride, porous silicon, and a variety of ceramic materials.

UNIVERSITY OF UTAH
Salt Lake City, UT 84112

417. ALUMINA REINFORCED TETRAGONAL ZIRCONIA (TZP) COMPOSITES
D. K. Shetty, Department of Materials Science and Engineering
(801) 581-6449 01-2 $90,712

Transformation toughening and reinforcement in composites; modeling of dependence of fiber-matrix interfacial properties on thermal expansion mismatch and processing temperature with glass-matrix composities; relationship between matrix cracking stress and interfacial properties. Effect of secondary additives on transformation toughening of Ce-TZP-alumina composites. Effects of fiber coatings on interfacial bonding and mechanical properties of alumina fiber-reinforced Y-TZP composites. Electrical-mechanical analog to evaluate stress-intensity factors for matrix cracks in fiber reinforced composites.

INDUSTRY

ROCKWELL INTERNATIONAL
Thousand Oaks, CA 91360

399. MECHANISMS OF MECHANICAL FATIGUE IN CERAMICS
B. N. Cox, Science Center
(805) 373-4128

D. B. Marshall, Science Center
(805) 373-4170 01-2 $123,161

Investigate the relationship between microstructure and fatigue behavior in fiber/whisker and metal reinforced ceramics. Distinguish crackbridging and crack-tip-shielding mechanisms by very percise measurements of crack opening displacements and displacements fields ahead of the crack-tip using a computer-based high accuracy strain mapping system (HASMAP). Study the rate of change of crack-bridging forces and the nonlinear constitutive behavior that causes crack-shielding. Systematic studies of the effects of variations in microstructure and changes in interface characteristics on fatigue.

ATOMIC STRUCTURE AND MICROSTRUCTURE

NATIONAL LABS

LOS ALAMOS NATIONAL LABORATORY
P. O. Box 1663
Los Alamos, NM 87545

147. INTERFACIAL AND RADIATION EFFECTS IN STRUCTURAL AND SUPERCONDUCTING CERAMICS
T. E. Mitchell, A. L. Graham, J. J. Petrovic, K. E. Sickafus
(505) 667-0938 01-5 $630,000 (60%)

Interface effects in structural ceramic composites. Synthesis of Si_3N_4, SiC and Al_2O_3 ceramics with VLS SiC whiskers. Interface modification. Characterization by high resolution and analytical electron microscopy. Interface adhesion and crack propagation in ceramic composites. Modeling of stress distribution and crack propagation by finite element codes. Irradiation-induced structures produced in high temperature superconductors by electronic excitation, ion bombardment and neutron radiation. Characterization by HREM, AEM, stored energy, electrical and magnetic property measurements. The role of irradiation in strength, fracture and interfacial properties of structural ceramics.

OAK RIDGE NATIONAL LABORATORY
P. O. Box 2008
Oak Ridge, TN 37831-6117

164. MICROSCOPY AND MICROANALYSIS
J. Bentley, E. A. Kenik, M. K. Miller
(615) 574-5067 01-1 $812,000 (20%)

Development and application of analytical electron microscopy (AEM) and atom-probe field-ion microscopy (APFIM) to determine the microstructure and microchemistry of materials. Equilibrium and radiation-induced segregation at grain-boundaries and interfaces by APFIM/AEM, correlation of GB structure and segregation. Radial distribution function determination by EXELFS and electron diffraction intensity profiles. APFIM/AEM studies of high-T_c superconductors. Lattice site location in alloys by electron channeling microanalysis. APFIM characterization of modulated structures, spinodals, early stages of phase transformations, and irradiated pressure vessel steels. GB phases and segregation in structural ceramics, ion-implanted ceramics, boron segregation and dislocations in Ni_3Al, short and long-range order in Ni_4Mo.

UNIVERSITIES

ARIZONA STATE UNIVERSITY
Tempe, AZ 85287

216. HIGH RESOLUTION ENERGY LOSS RESEARCH: SI COMPOUND CERAMICS AND COMPOSITES
R. W. Carpenter, Center for Solid State Science
(602) 965-4546

S. H. Lin, Department of Chemistry
(602) 965-3715 01-1 $108,600

High spatial resolution analytical electron microscopy investigation with a field emission source of the elemental composition and local electronic structure of small amorphous and crystalline regions in silicon carbide and silicon nitride and in interfacial reaction zones of metal/ceramic and ceramic/ceramic composites. Development of theoretical methods for EELS spectral analysis. Quantitative analysis of small-probe current distribution in real and reciprocal space for field emission gun analytical electron microscopes to permit quantitative analysis of compositional gradients.

UNIVERSITY OF CALIFORNIA AT SANTA BARBARA
Santa Barbara, CA 93106

234. FUNDAMENTAL STUDIES OF THE INTERRELATIONSHIP BETWEEN GRAIN-BOUNDARY PROPERTIES AND THE MACROSCOPIC PROPERTIES OF POLYCRYSTALLINE MATERIALS
D. R. Clarke, Materials Department
(805) 893-8232 01-1 $100,000 (50%)

Relationships between properties of individual grain-boundaries and macroscopic properties of polyphase, polycrystalline materials. Measurement of electrical properties and plastic deformation of grain-boundaries in bicrystals as a function of bicrystallography determined by electron channeling and high resolution transmission electron microscopy. Comparison of results from polycrystalline thin films and composed to simulations.

UNIVERSITY OF MINNESOTA
Minneapolis, MN 55455

264. CRYSTALLINE-AMORPHOUS OXIDE INTERFACES AND RELATION TO GRAIN-BOUNDARY FILMS
C. B. Carter, Department of Materials Science and Engineering *
(607) 255-4797 01-1 $137,751

Study of structure and chemistry of the interface between an amorphous material and crystalline form. Surface reaction on a bicrystal sample examined by cross-sectional transmission electron microscopy and reaction of a glass-forming vapor with a pre-thinned TEM sample. Materials studied are alumina, magnesia and silicon.

* Formerly at Cornell University.

CORNELL UNIVERSITY
Ithaca, NY 14853

269. CERAMIC FILMS AND INTERFACES: CHEMICAL AND MECHANICAL PROPERTIES
Rishi Raj, Department of Materials Science and Engineering
(607) 255-4040 01-2 $176,630

Structure and composition of interfaces between dissimilar ceramics and correlation between structure, chemistry and mechanical properties. Investigation will use alumina/zirconia interfaces in layered thin films under controlled oxygen partial pressures. Special emphasis on the formation of intergranular phases. Structure characterized by high resolution TEM, chemistry by scanning TEM, and mechanical properties by internal friction, fracture, and plasticity.

LEHIGH UNIVERSITY
Bethlehem, PA 18015

302. HIGH RESOLUTION MICROSTRUCTURAL AND MICROCHEMICAL ANALYSIS OF ZIRCONIA EUTECTIC INTERFACES
M. R. Notis, Department of Metallurgy and Materials Sciences
(215) 758-4225 01-1 $116,535

Eutectic interfaces studied in as-grown $MnO-ZrO_2$, $NiO-ZrO_2(Y^{20}3$ $CoO-ZRO_2(CaO)$, and $NiO-Y_2O_3$ systems. High resolution microstructural and microanalytical methods (HRTEM, CBED and PEELS), used to study interfaces in plan-view and conventional configurations. Local oxidation state across grain-boundaries in single phase MnO and $MnO-ZrO_2$ studied as function of oxygen partial pressure. Segregation effects due to ternary additions measured at interphase interfaces and at local defects and faults within interfaces.

UNIVERSITY OF MICHIGAN
Ann Arbor, MI 48109-2136

321. FUNDAMENTAL ALLOY DESIGN OF OXIDE CERAMICS AND THEIR COMPOSITES
I. -W. Chen, Department of Materials Science and Engineering
(313) 763-6661 01-2 $95,642

Three alloy design approaches to oxide ceramics for structural and energy applications. Aliovalent ions in solid solutions investigated for space charge segregation and effects on grain growth, dislocation creep and intergranular cavitation. Precipitation of spinel-based layers compounds studied for toughened and strengthened composites. Ceramic matrix composites with interpenetrating ductile phase prepared by infiltration of porous ceramic preforms with liquid under pressure. Structure property relationships established through variation of microchemical, microstructural, crystallographic, and other material parameters.

UNIVERSITY OF UTAH
Salt Lake City, UT 84112

416. FABRICATION, PHASE TRANSFORMATION STUDIES, AND CHARACTERIZATION OF SIC-ALN-AL_2OC
A. V. Virkar, Department of Materials Science and Engineering
(801) 581-5396 01-1 $80,683

Analysis of phase equilibria and phase transformations and the relationship between creep behavior and microstructure in the $SIC-AlN-Al_2OC$ system. Diffusional phase transformations leading to phase separation. Modulated microstructures developed by spinodal decomposition. Cellular precipitation. Dependence of creep behavior on composition and microstructure.

CHEMICAL REACTIONS AND MASS TRANSPORT

NATIONAL LABS

UNIVERSITY OF ILLINOIS MRL
104 S. Goodwin Avenue
Urbana, IL 61801

77. MICROSTRUCTURE EVOLUTION, INTERFACES AND PROPERTIES IN STRUCTURAL CERAMIC COMPOSITES
A. Zangvil
(217) 333-6829 01-1 $171,319

Microstructure and microchemistry of SiC with covalent additives, such as AlN, BN and BeO; solid solution formation in SiC based systems; effect of processing variables and additives on polytypism and microchemistry. Interfaces and toughening mechanisms in SiC- and mullite-matrix composites. Application of microanalytic methods to analysis of the structure and microchemistry of ceramic high-T_c superconductors.

UNIVERSITIES

OREGON STATE UNIVERSITY
Corvallis, OR 97331

367. HYPERFINE EXPERIMENTAL INVESTIGATION OF ZIRCONIA CERAMICS
J. A. Gardner, Department of Physics
(503) 737-3278 01-1 $110,497

Perturbed angular correlation (PAC) spectroscopy of nuclear gamma rays to investigate Zr-containing ceramics. PAC characterization of free energies, transformation mechanisms, equilibrium phase boundaries, diffusion and relaxation models, short range order, order-disorder reactions, and elevated-temperature/time dependent effects in various zirconia-based ceramics that contain either Hf-181 or In-111 as a probe. Investigation of Zr-91 in zirconia by nuclear quadrupole resonance (NQR) and of O-17 substituted zirconia by nuclear magnetic resonance (NMR). NQR/NMR experiments to complement and expand the studies of local structure and oxygen vacancy dynamics underway with PAC.

THEORY AND MODELING

NATIONAL LABS

ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
Argonne, IL 60439

36. MODELING AND THEORY OF INTERFACES
D. Wolf, S. Phillpot, S. Yip
(708) 252-5205 02-3 $265,000 (30%)

Computer simulation of the physical properties of solid interfaces, such as grain and interphase boundaries, thin films and superlattices, involving both atomistic simulation methods (lattice statics and dynamics, molecular dynamics, Monte Carlo). The atomistic simulations are used to determine, for example, the structure, free energy and elastic properties of solid interfaces as a function of temperature, the point-defect properties of interfaces, such as impurity segregation and diffusion, and the properties of voids in grain boundaries and in the bulk. Materials considered involve metals, semiconductors and ceramics as well as interfaces between them.

UNIVERSITIES

UNIVERSITY OF MISSOURI AT KANSAS CITY
Kansas City, MO 64110-2499

334. THEORETICAL STUDIES ON THE ELECTRONIC STRUCTURE AND PROPERTIES OF CERAMIC CRYSTALS AND GLASSES
W. -Y. Ching, Department of Physics
(816) 235-2503 01-1 $105,572 (30%)

Calculation by means of orthogonalized linear combination of atomic obitals (OLCAO) of electronic structure and linear optical properties for a larger number of oxide, nitride, phosphate, silicate, III-V semiconductors, metallic glass and high-T_c superconducting materials. Local density functional calculation of important bulk properties, phonon frequencies and structural phase transitions for selected materials. Formulation of calculational method for nonlinear optical properties. Calculation of magnetic properties of rare earth-iron-boron magnetic alloys and related intermetallic compounds.

OREGON STATE UNIVERSITY
Corvallis, OR 97331

362. THEORETICAL STUDIES OF ZIRCONIA AND RELATED MATERIALS
H. J. F. Jansen, Department of Physics
(503) 754-4631 01-3 $58,300

Total energy calculations of the electronic structure of zirconia and related materials used to obtain the electronic energy and the charge density as a function of atomic arrangement. Study of field-gradients, lattice relaxation, phonon spectrum, oxygen mobility and transport. Both Full Potential Linearized Augmented Plane Wave (FLAPW) and Monte Carlo techniques used.

IRRADIATION EFFECTS

NATIONAL LABS

LOS ALAMOS NATIONAL LABORATORY
P. O. Box 1663
Los Alamos, NM 87545

145. NEUTRON IRRADIATION INDUCED METASTABLE STRUCTURES
K. E. Sickafus, Jr. Clinard, F. W., M. Nastasi
(505) 665-3457 01-4 $269,000

Irradiation phenomena and damage microstructures resulting from neutron irradiation of ceramics and intermetallic compounds. Investigation of cascae damage events in model materials, complemented by physical property measurements and ion irradiation tests, where the latter can elucidate neutron damage effects. Computer simulation is used to assist in understanding the nature of damage events.

OAK RIDGE NATIONAL LABORATORY
P. O. Box 2008
Oak Ridge, TN 37831-6117

170. STRUCTURE AND PROPERTIES OF SURFACES AND INTERFACES
L. L. Horton, C. J. McHargue
(615) 574-5081 01-5 $695,000

Structure of ion-implanted Al_2O_3, SIC, and TiB_2 by backscattering-channeling and TEM, hardening, surface fracture toughening and wear of ion-implanted ceramics, structure and properties studied as a function of implantation parameters (temperature, fluence, energy, ion species) and annealing (temperature and environment). Mechanical behavior of thin films and interfaces, stress relaxation and dissipation. Adherence of oxide and metal films. Ion beam mixing and amorphization of multilayer metallic alloys and ceramics.

PACIFIC NORTHWEST LABORATORY
P. O. Box 999
Richland, WA 99352

197. IRRADIATION EFFECTS IN CERAMICS
W. J. Weber, N. J. Hess
(509) 375-2299 01-4 $50,000

Multidisciplinary research on the production, nature, and accumulation of irradiation-induced defects, microsturctures, and solid-state transformations in ceramics. Irradiations with neutrons, ions, and electrons to study point defect production and associate effects from both single displacement events and high-energy displacement cascades. Develop understanding of structural stability and irradiation-induced amorphization in ceramics. Computer simulations of defect production/ survivability, defect stability, and defect migration. The investigations utilize X-ray and neutron diffraction, electron microscopy, EXAFS, laser spectroscopies, ion beam techniques, and electrical property measurements to characterize the defects, microstructures, and transformations introduced by irradiation in simple and complex oxides, carbides, and nitrides. Work includes the development of techniques for in situ characterization during neutron and ion beam irradiations.

UNIVERSITIES

UNIVERSITY OF CALIFORNIA AT DAVIS
Davis, CA 95616

225. INVESTIGATION OF RADIATION DAMAGE AND DECOMPOSITION OF CERAMICS USING ELECTRON MICROSCOPY

D. G. Howitt, Department of Mechanical Engineering
(916) 752-0580 01-4 $95,000 (30%)

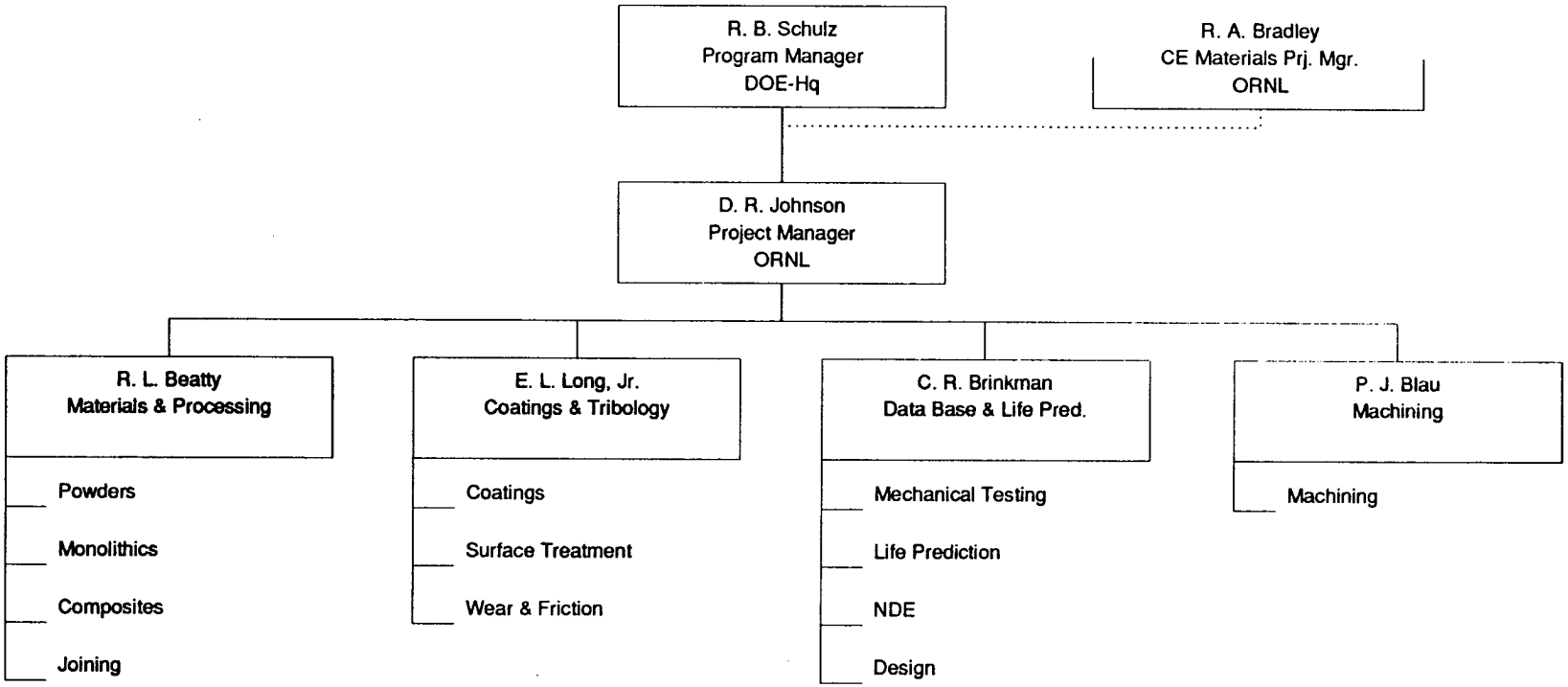
Investigation of electron and ion irradiation induced ionization, displacement damage, diffusion, and stimulated desorption by means of in situ analytical electron microscopy and mass spectroscopy. Study of ion mixing effects under ion irradiation. Materials include dielectrics and semiconductors. Study of free standing ceramics thin films.

Ceramic Technology Program
Office of Transportation Materials
U.S. Department of Energy

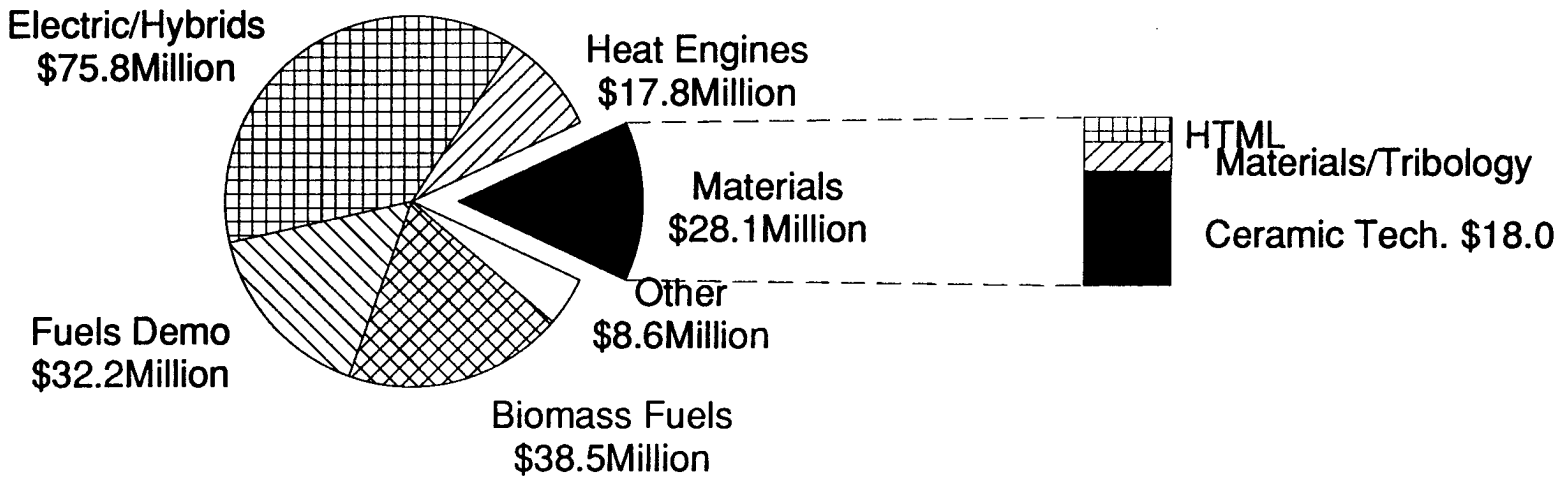
Robert B. Schulz, DOE
and
D. Ray Johnson, ORNL

Interagency Coordinating Committee
on Structural Ceramics Meeting
May 13, 1992, Arlington, VA

Ceramic Technology Project

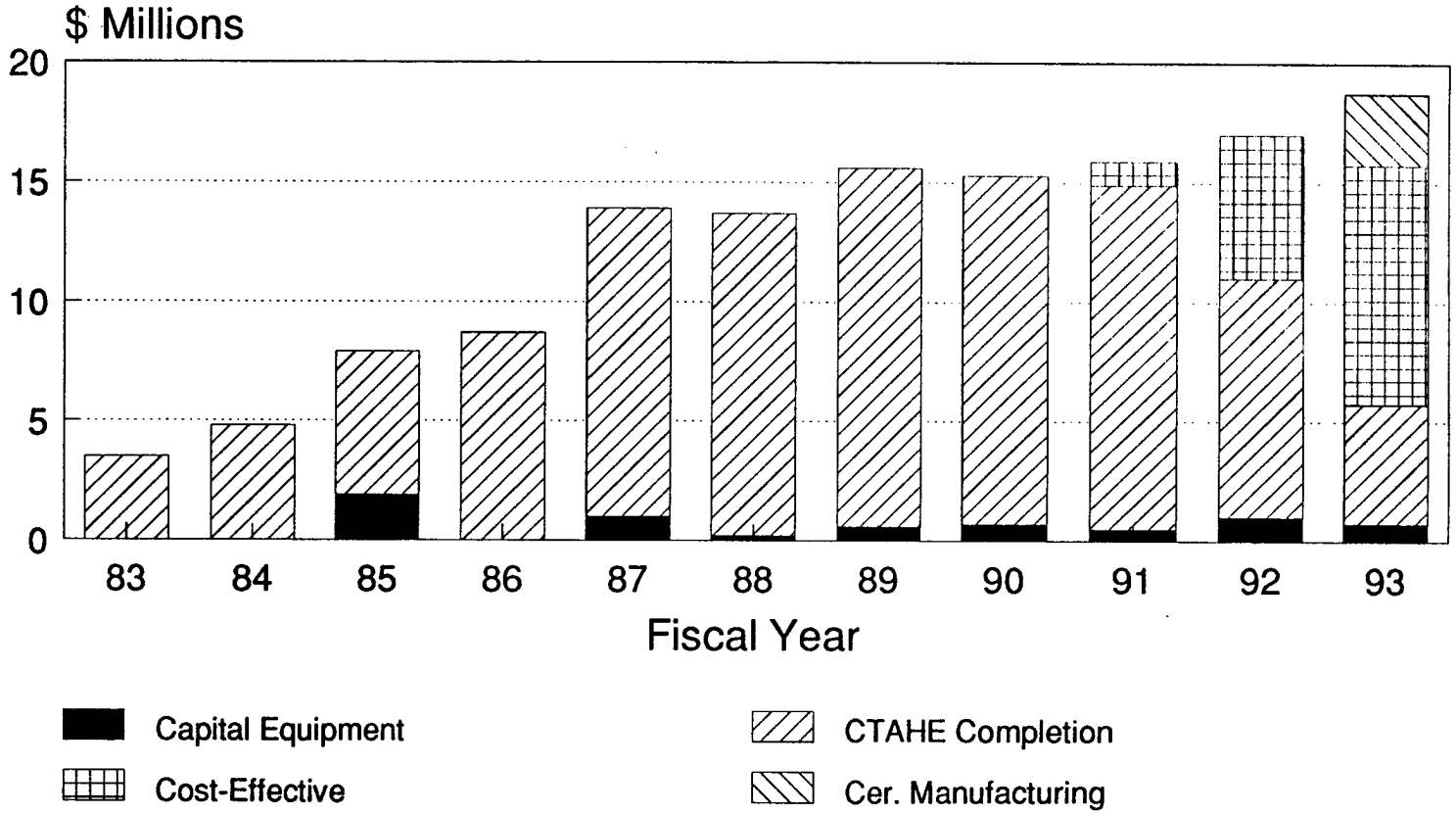


TRANSPORTATION TECHNOLOGIES
FY 1993 BUDGET REQUEST



A large share for Structural Ceramics

CERAMIC TECHNOLOGY PROGRAM
FY 1993 Changing Budget



AMPP Ceramic Manufacturing
Cost-Effective Ceramics for Heat Engines

CERAMIC TECHNOLOGY PROGRAM
FY 1993 Budget Request

CTAHE

In-Situ Toughened

Thermal Coatings

NDE

Testing/Data Base

Cost-Effective

Powder Synthesis

Innovative Processes

Near-Net Shape

Ceramic Machining

Low Expansion

Standards

Testing/Data Base

Adv. Manufacturing

Presidential Adv. Materials & Processing Program:

Automated Ceramic Manufacturing

Cost-Effective Machining of Ceramics
Joint DOE CE/DP Program

- CE Ceramic Technology Project
  -R&D contracts with Industry
  and National Labs
- DP Y-12
  -CRADAs with Industry
  -Ceramics Manufacturability Center
  in HTML and Y-12
- CE HTML
  -Materials characterization User Centers
  -Ceramics Manufacturability Center in HTML

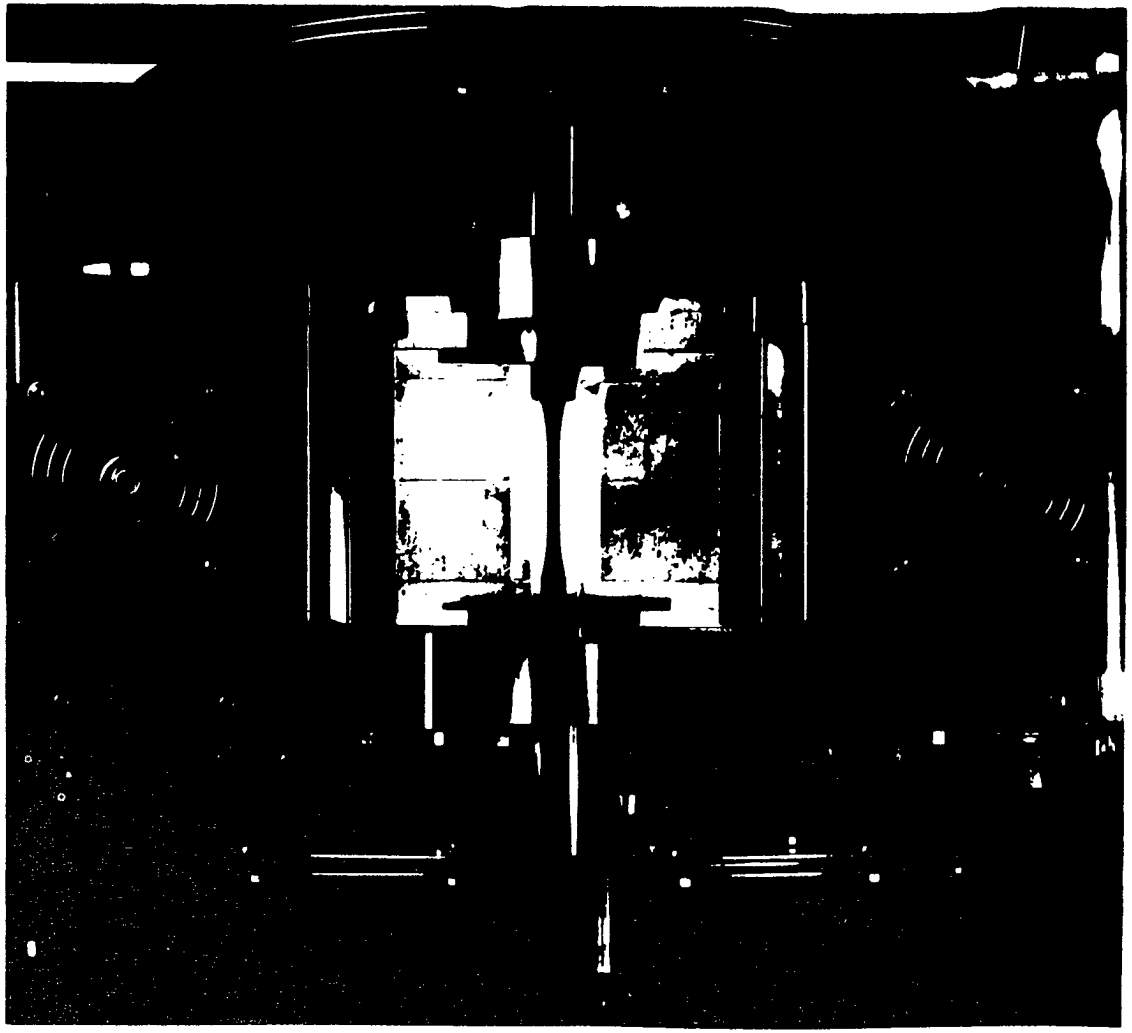
Cost-Effective Machining of Ceramics
Status Report

- President Bush witnessed signing of 1st CRADA with Coors Ceramics Co. at the HTML.
- Needs Analysis with Industry is underway to identify research priorities.
- Grinders and inspection equipment moving from Y-12 Plant to HTML Grinding Center.
- FY92-FY96 Resource Plan: DP/CE/Industry approx. $17 million each, $52 million total

Ceramic Manufacturing Project
FY 1993 AMPP Initiative

- "Bridge the Gap" Between Prototype and Production
- Emphasis on Statistical Process Control, Intelligent Processing, and Automation
- Develop Ceramic Supplier Infrastructure Capable of Meeting Automotive Quality/ Cost Requirements

High-Temperature Tensile Testing
Self-Aligning Super Grips



Norton Processing Program

- 3 Machining Procedures

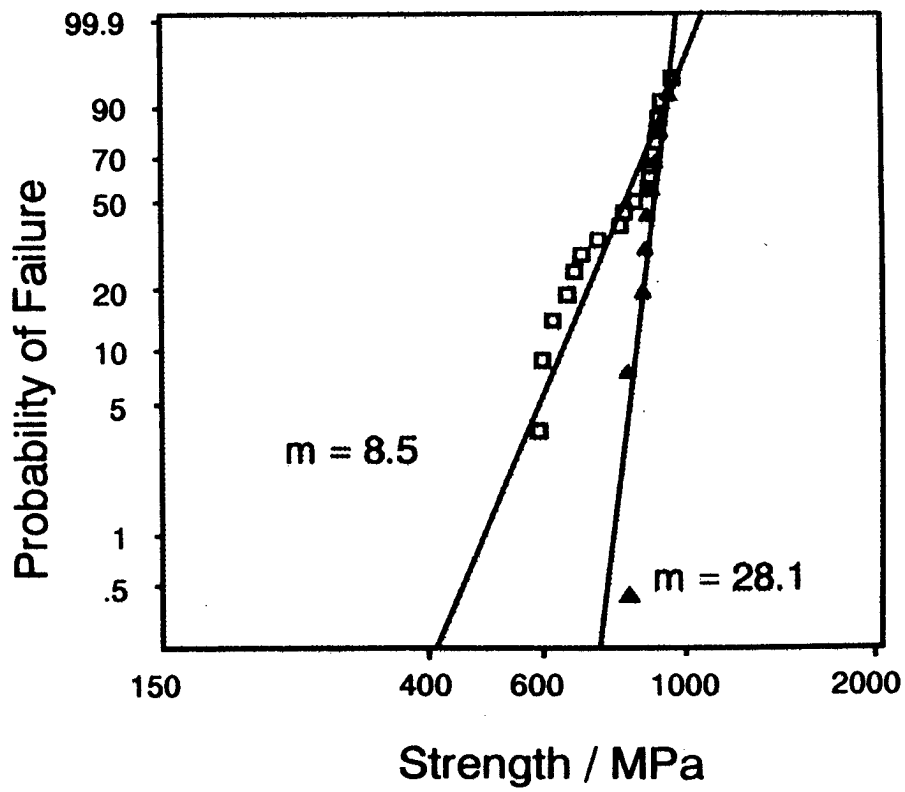
  Mean Strength 789 MPa (115 Ksi)

  Weibull m = 8.5

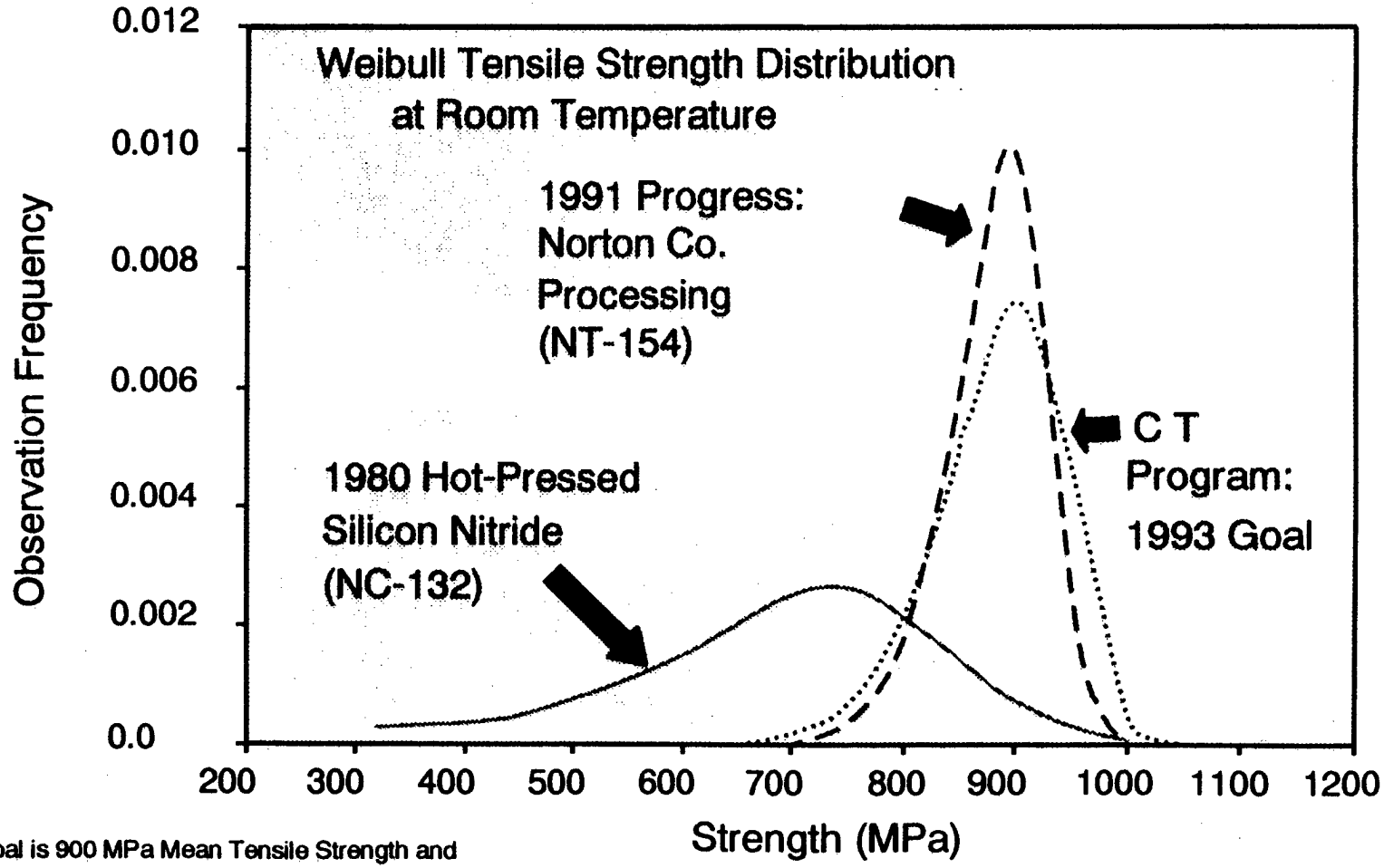
- Improved Machining

  Mean Strength 889 MPa (129 Ksi)

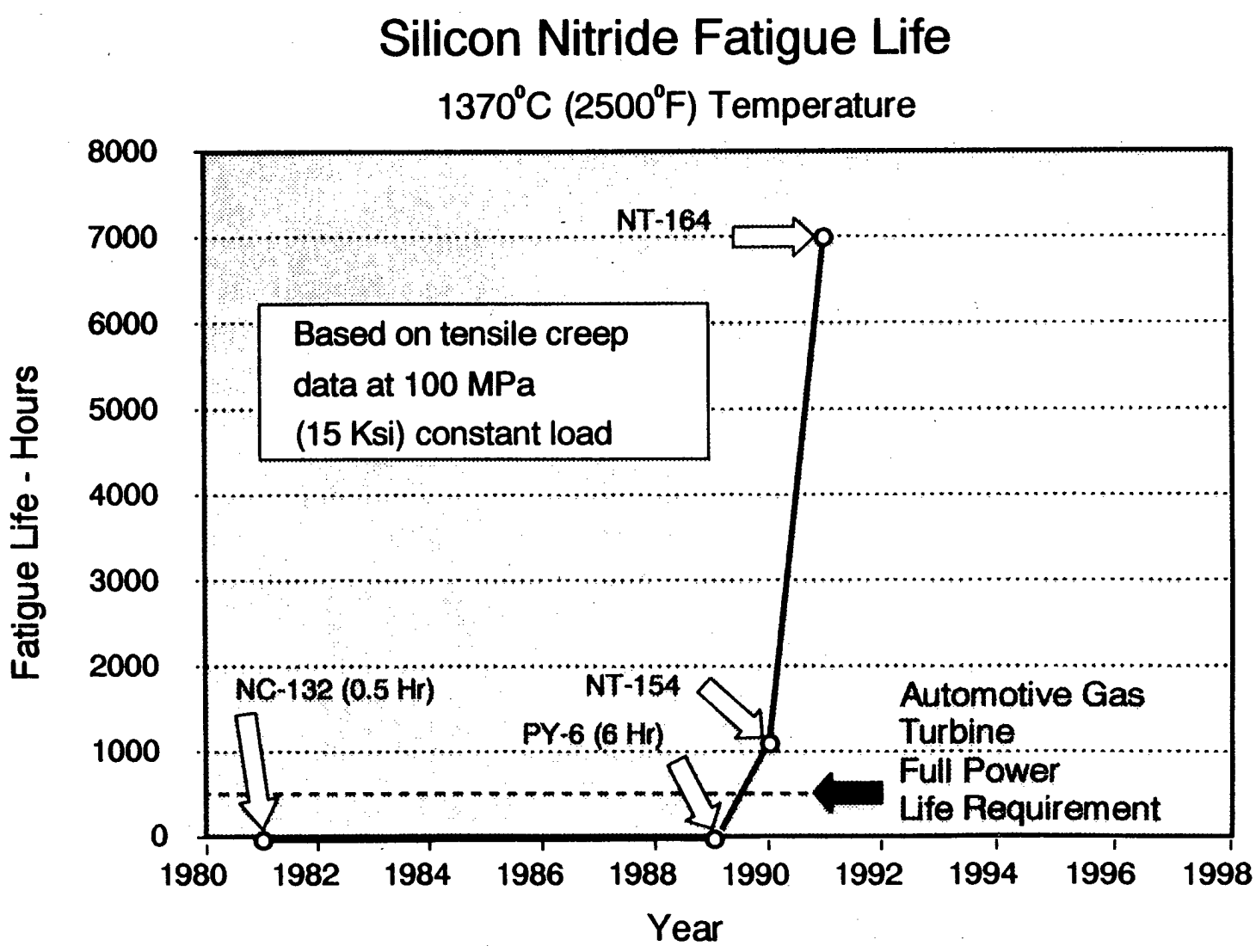
  Weibull m = 28



Improvements in Ceramic Processing and Flaw Tolerance



Goal is 900 MPa Mean Tensile Strength and
Weibull Modulus of m=20 in 200 Tensile Specimens


Silicon Nitride Fatigue Life
1370°C (2500°F) Temperature
Fatigue Life - Hours
8000
7000
6000
5000
4000
3000
2000
1000
0
1980
1982
1984
1986
1988
1990
1992
1994
1996
1998
Year
NT-164
Based on tensile creep data at 100 MPa (15 Ksi) constant load
NC-132 (0.5 Hr)
NT-154
PY-6 (6 Hr)
Automotive Gas Turbine Full Power Life Requirement

DOE - Transportation Materials
Ceramic Technology & Tribology

	FY 1991	FY 1992	FY 1993 Request
Ceramic Technology	$15,400	$15,800	$18,000
Tribology (part of)	1,000	700	500
TOTAL	$16,400	$16,500	$18,500

CURRENT PROJECTS AND FUNDING FOR THE
CERAMIC TECHNOLOGY PROJECT, U.S. DOE
OFFICE OF TRANSPORTATION TECHNOLOGIES,
R. B. SCHULZ, PROGRAM MANAGER

MATERIALS AND PROCESSING

High Temperature SX Silicon Carbide (WBS No. 1113)

PERFORMING ORGANIZATION: Carborundum Company

Carborundum Contact - Roger S. Storm, 716-278-2544

ORNL Contact - E. L. Long, Jr., 615-574-5172

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	287	0

Attrition Milling of Silicon Nitride Powder (WBS No. 1118)

PERFORMING ORGANIZATION: National Institute of Standards and Technology (NIST)

National Institute of Standards and Technology (NIST) Contact - S. Malghan, 301-975-6101

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	110	110

Microwave Sintering (WBS No. 1124)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - T. N. Tiegs, 615-574-5173

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	400	400

Cost Effective Silicon Nitride Powder RFP (WBS No. 1125)

PERFORMING ORGANIZATION: TBD

ORNL Contact: S. G. Winslow, 615-574-0965

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	500	1000

Novel Si_3N_4 Process (WBS No. 1126)

PERFORMING ORGANIZATION: Sullivan Mining Corporation

Sullivan Contact - T. M. Sullivan, 619-692-1180

ORNL Contact - E. L. Long, Jr., 615-574-5172

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	144	500

Cost Effective Sintering of Silicon Nitride Ceramics (WBS No. 1127)

PERFORMING ORGANIZATION: Southern Illinois University

Southern Illinois University Contact: D. E. Wittmer, 618-453-7006/7924

ORNL Contact: T. N. Tiegs, 615-574-5173

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	74	74

Cost Effective Manufacture of SRBSN Components (WBS No. 1128)

PERFORMING ORGANIZATION: Coors Ceramics Company

Coors Contact: Jack D. Sibold, 303-277-4441

ORNL Contact: R. L. Beatty, 615-574-4536

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	250	0

Advanced Processing (WBS No. 1141)

PERFORMING ORGANIZATION: Norton Company

Norton Contact - D. M. Tracey, 508-393-5811

ORNL Contact - R. L. Beatty, 615-574-4536

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	1575	200

Processing of Monolithics (WBS No. 1142)

PERFORMING ORGANIZATION: ORNL

ORNL Contact: S. D. Nunn, 615-574-9978

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	650	650

Advanced Composites (WBS No. 1225)

PERFORMING ORGANIZATION: University of Michigan

University of Michigan Contact: T. Y. Tien, 313-764-9449

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	260	281

In-situ Toughened Si_3N_4 (WBS No. 1226)

PERFORMING ORGANIZATION: Garrett Ceramic Components Division

Garrett Contact - H. C. Yeh, 213-618-7449

ORNL Contact - T. N. Tiegs, 615-574-5173

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	500	500

Dispersion Toughened Oxide Composites (WBS No. 1231)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - T. N. Tiegs, 615-574-5173

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	350	350

Low Expansion Ceramics (WBS No. 1242)

PERFORMING ORGANIZATION: Virginia Polytechnic Institute and State University

VPI & SU Contact - J. J. Brown, 703-961-6640

ORNL Contact - V. J. Tennery, 615-574-5123

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	162	0

Low Expansion Ceramics (WBS No. 1244)

PERFORMING ORGANIZATION: TBD

ORNL Contact: D. P. Stinton, 615-574-4556

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	500	500

Advanced Coating Technology (WBS No. 1311)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - D. P. Stinton, 615-574-4556

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	175	175

Coatings to Reduce Contact Stress Damage of Ceramics (WBS No. 1313)

PERFORMING ORGANIZATION: Boston University

Boston University Contact: V. K. Sarin, 617-353-2842

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	50	50

Wear Resistant Coatings (WBS No. 1331)

PERFORMING ORGANIZATION: Caterpillar

Caterpillar Contact - M. H. Haselkorn, 209-578-6624

ORNL Contact - D. P. Stinton, 615-574-4556

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	249	0

Wear Resistant Coatings (WBS No. 1332)

PERFORMING ORGANIZATION: Cummins Engine Company, Inc.

Cummins Contact - Malcolm Naylor, 812-377-5000

ORNL Contact - D. P. Stinton, 615-574-4556

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	0	400

Thick Thermal Barrier Coating Systems for Low Heat Rejection Diesel Engines (WBS No. 1342)

PERFORMING ORGANIZATION: Caterpillar Inc.

Caterpillar Contact - M. B. Beardsley, 309-578-8514

ORNL Contact: D. P. Stinton, 615-574-4556

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	139	175

Active Metal Brazing PSZ- Iron (WBS No. 1411)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - M. L. Santella, 615-574-4805

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	220	220

Ceramic-Ceramic Joints AGT (WBS No. 1421)

PERFORMING ORGANIZATION: Norton Company

Norton Contact - N. D. Corbin, 508-393-5660

ORNL Contact - M. L. Santella, 615-574-4805

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	234	0

Cost Effective Ceramic Machining (WBS No. 1500)

PERFORMING ORGANIZATION: TBD

ORNL Contact - P. J. Blau, 615-574-5377

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	700	1700

Chemically Assisted Machining of Ceramics

PERFORMING ORGANIZATION: National Institute of Science and Technology (NIST)

NIST Contact - Steven M. Hsu, 301-975-6119

ORNL Contact - P. J. Blau, 615-574-5377

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	150	150

Grinding Optimization for Advanced Ceramics

PERFORMING ORGANIZATION: National Institute of Science and Technology (NIST)

NIST Contact - Said Jahanmir, 301-975-6871

ORNL Contact - P. J. Blau, 615-574-5377

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	150	150

Cost Effective Ceramic Manufacturing (WBS No. 1502)

PERFORMING ORGANIZATION: TBD

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	200	3000

MATERIALS DESIGN METHODOLOGY

Coating Adherence on Ceramic Substrates (WBS No. 2212)

PERFORMING ORGANIZATION: University of Tennessee

University of Tennessee Contact - Debra L. Joslin, 615-574-4343

ORNL Contact - L. L. Horton, 615-574-5801

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	30	0

Advanced Statistics Calculations (WBS No. 2313)

PERFORMING ORGANIZATION: General Electric Research and Development Center

GE Contact - C. A. Johnson, 518-387-6421

ORNL Contact - M. K. Ferber, 615-576-0818

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	100	100

DATA BASE/LIFE PREDICTION

Microstructural Analysis (WBS No. 3111)

PERFORMING ORGANIZATION: National Institute of Standards and Technology (NIST)

NIST Contact - S. M. Wiederhorn, 301-975-2000

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	50	50

Mechanical Properties and Microstructural Characterization of Si_3N_4 Ceramics (WBS No. 3114)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - T. A. Nolan, 615-574-0811

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	200	200

Project Data Base (WBS No. 3117)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - B. L. Keyes, 615-574-5113

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	240	240

Fracture Behavior of Toughened Ceramics (WBS No. 3213)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - P. F. Becher, 615-574-5157

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	290	290

Cyclic Fatigue of Toughened Ceramics (WBS No. 3214)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - K. C. Liu, 615-574-5116

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	220	220

Tensile Stress Rupture Development (WBS No. 3215)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - K. C. Liu, 615-574-5116

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	300	300

Rotor Materials Data Base (WBS No. 3216)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - M. K. Ferber, 615-576-0818

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	200	200

Toughened Ceramics Life Prediction (WBS No. 3217)

PERFORMING ORGANIZATION: NASA-Lewis Research Center

NASA Contact - Stanley R. Levine, 216-433-3276

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	200	200

Life Prediction Methodology (WBS No. 3222)

PERFORMING ORGANIZATION: Allison Gas Turbine Division

Allison Contact - P. K. Khandelwal, 317-230-3805

ORNL Contact - C. R. Brinkman, 615-574-5106

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	0	0

Life Prediction Methodology (WBS No. 3223)

PERFORMING ORGANIZATION: Garrett Auxiliary Power Division

Garrett Contact - Carolyn McCormick, 602-220-3016

ORNL Contact - C. R. Brinkman, 615-574-5106

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	501	600

Environmental Effects in Toughened Ceramics (WBS No. 3314)

PERFORMING ORGANIZATION: University of Dayton

University of Dayton Contact - N. L. Hecht, 513-229-4341

ORNL Contact - V. J. Tennery, 615-574-5123

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	400	400

High Temperature Tensile Testing (WBS No. 3412)

PERFORMING ORGANIZATION: North Carolina A&T State University

North Carolina A&T State University Contact - J. Sankar, 919-334-7620

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	200	200

Standard Tensile Test Development (WBS No. 3413)

PERFORMING ORGANIZATION: National Institute of Standards and Technology (NIST)

NIST Contact - S. M. Wiederhorn, 301-975-2000

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	125	125

Non-Destructive Evaluation (WBS No. 3511)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - D. J. McGuire, 615-574-4835

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	360	360

Computed Tomography (WBS No. 3515)

PERFORMING ORGANIZATION: Argonne National Laboratory

Argonne Contact - W. A. Ellingson, 312-972-5068

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	90	60

Nuclear Magnetic Resonance Imaging (WBS No. 3516)

PERFORMING ORGANIZATION: Argonne National Laboratory

Argonne Contact - W. A. Ellingson, 312-972-5068

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	65	125

TECHNOLOGY TRANSFER

International Exchange Agreement (WBS No. 4115)

PERFORMING ORGANIZATION: ORNL

ORNL Contact - V. J. Tennery, 615-574-5123

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	400	400

Standard Reference Powders (WBS No. 4116)

PERFORMING ORGANIZATION: National Institute of Standards and Technology (NIST)

NIST Contact - S. Malghan, 301-975-6101

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	150	0

Development of K_{Ic} Standard (WBS No. 4121)

PERFORMING ORGANIZATION: National Institute of Standards and Technology (NIST)

NIST Contact - G. Quinn, 301-975-5765

ORNL Contact - D. Ray Johnson, 615-576-6832

FUNDING ($K) PROFILE:	FY 1992	FY 1993 (EST)
	100	100

DIVISION OF MATERIALS RESEARCH ORGANIZATION AND SCIENTIFIC STAFF

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Deputy Division Director
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Adriaan M. de Graaf

AMPP Program Director
Vacant

Administrative Manager
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FCCSET Programs Coordinator
Cross-Directorate Programs
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Bruce A. MacDonald

Ceramics
Franklin F.Y. Wang

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LaVerne D. Hess

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G. Bruce Taggart

Brian W. Dodson

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Lorretta J. Inglehart

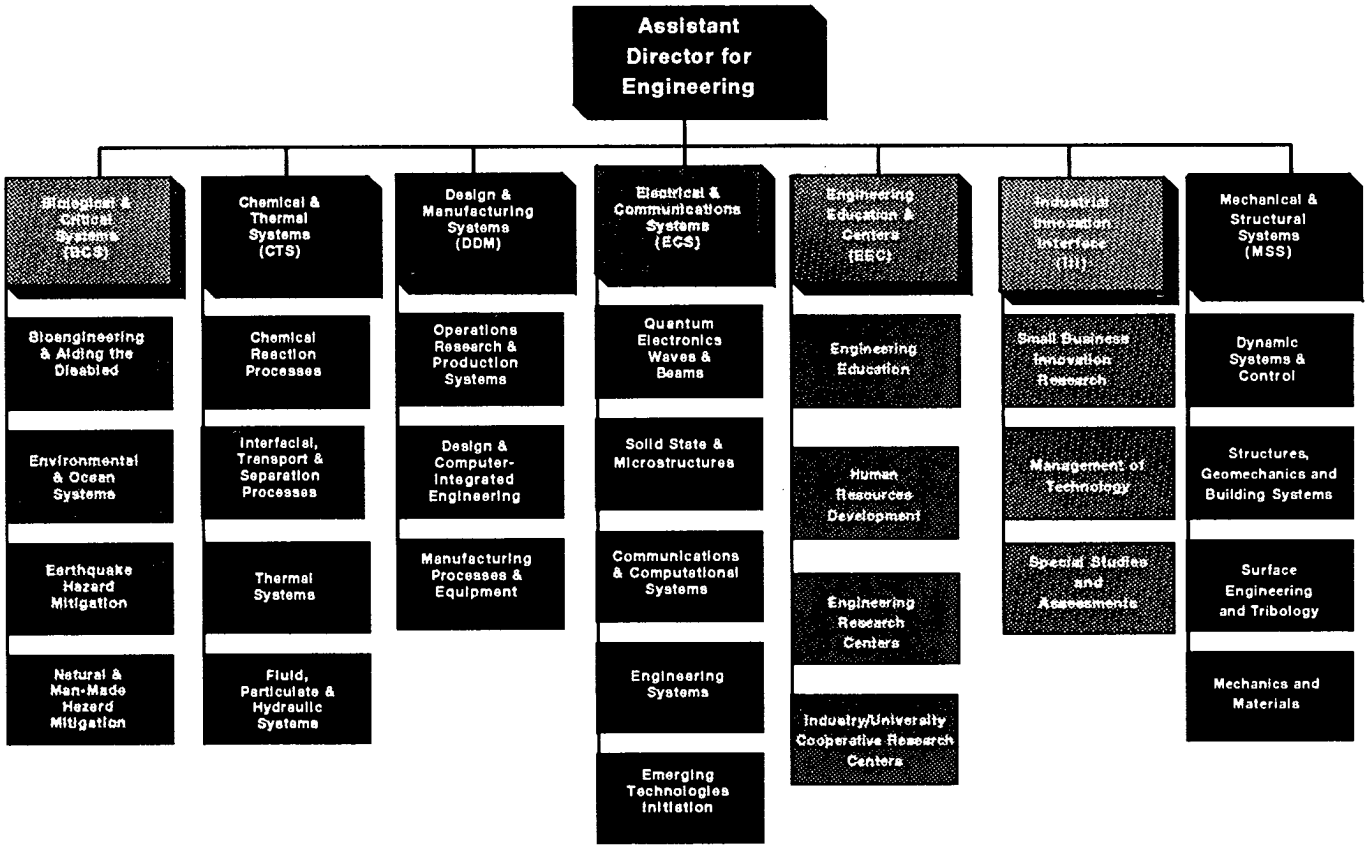
Materials Research Laboratories and Groups
(202) 357-9791

Materials Research Laboratories
W. Lance Haworth

Materials Research Groups
John C. Hurt

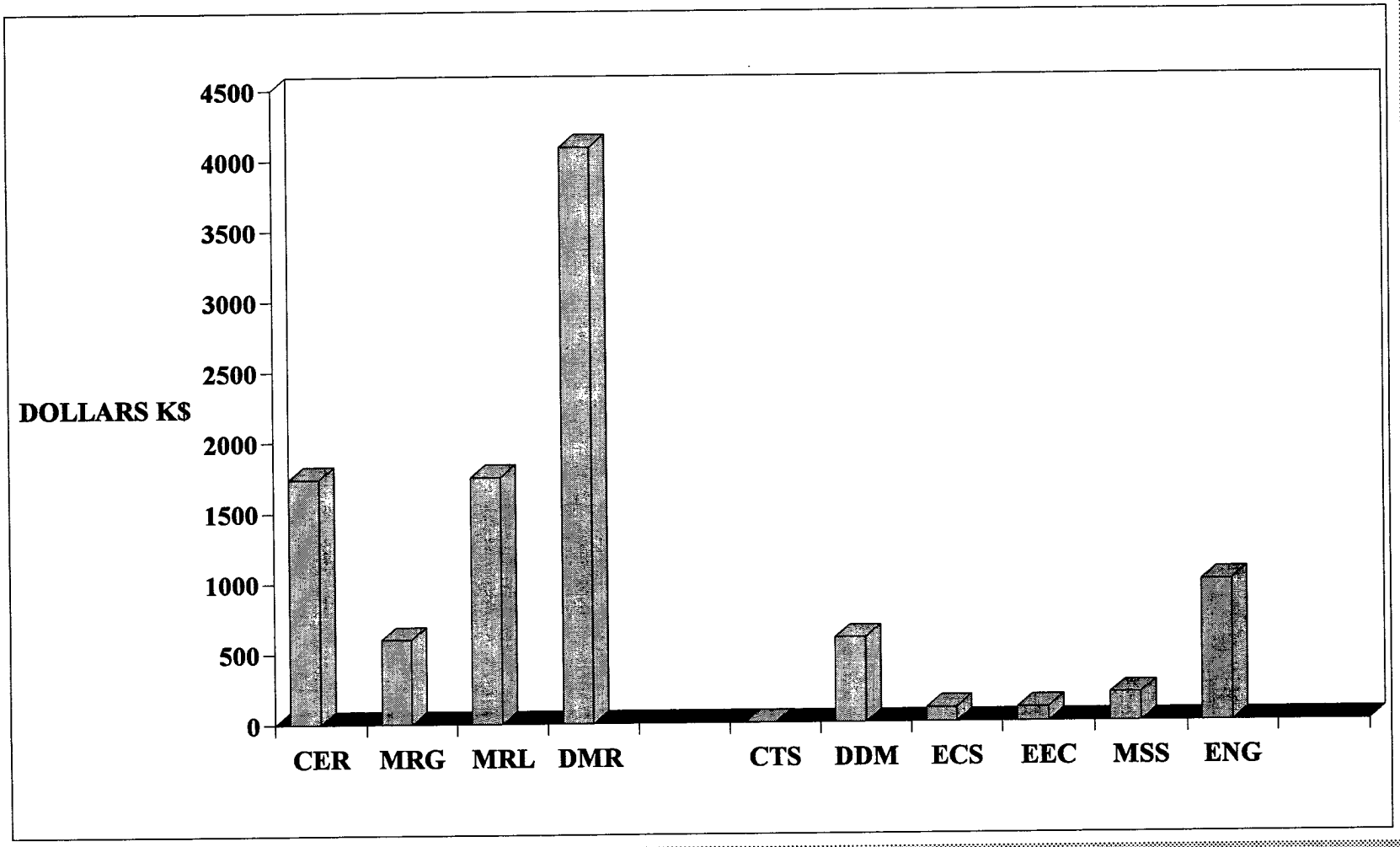
As of: May 1, 1992

Directorate for Engineering



NSF	STRUCTURAL CERAMICS K$	OTHERS K$
DMR Division of Materials Research		
CER Ceramics Program	1,740	1,520
MRG Materials Research Groups	600	1,070
MRL Materials Research Laboratories	1,750	200
SUBTOTAL:	4,090	2,790
ENG Directorate for Engineering		
CTS Chemical & Thermal Systems		100
DDM Design & Manufacturing Systems	600	600
ECS Electrical & Communications Systems	100	100
EEC Engineering Education & Centers	100	100
MSS Mechanical & Structural Systems	200	100
SUBTOTAL:	1,000	900
TOTAL:	$5,090K	$3,690K
MAY 12, 1992	58.0%	42.0%

STRUCTURAL CERAMICS, NSF FY92



MATERIALS SYNTHESIS AND PROCESSING:

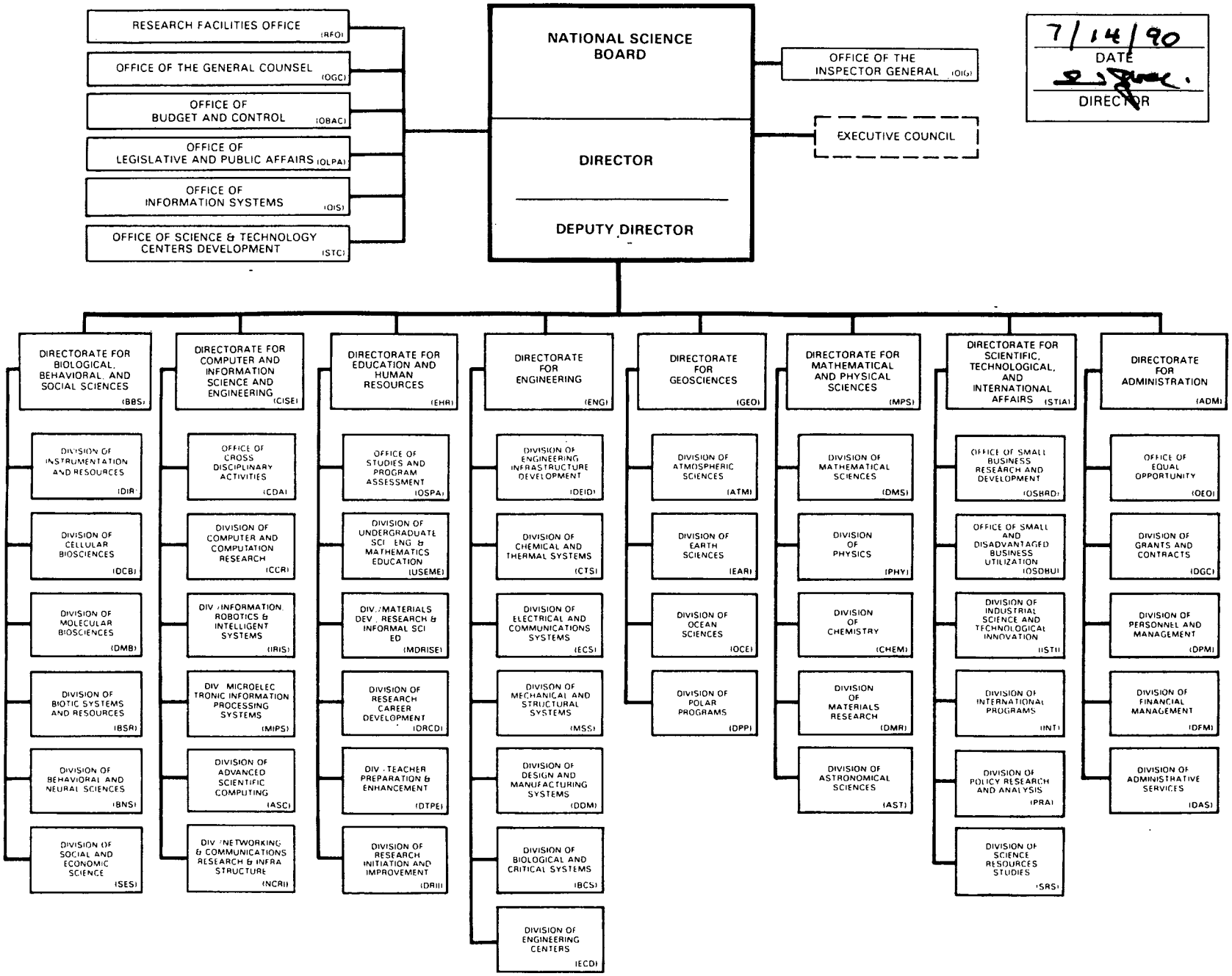
Research at the Interfaces of Materials Research, Engineering, Chemistry, and Biology

DIVISION OF MATERIALS RESEARCH
DIVISION OF CELLULAR BIOSCIENCES
DIVISION OF MOLECULAR BIOSCIENCES
DIVISION OF CHEMISTRY
DIVISION OF BIOLOGICAL AND CRITICAL SYSTEMS
DIVISION OF CHEMICAL AND THERMAL SYSTEMS
DIVISION OF DESIGN AND MANUFACTURING SYSTEMS
DIVISION OF ELECTRICAL AND COMMUNICATIONS SYSTEMS
DIVISION OF MECHANICAL AND STRUCTURAL SYSTEMS

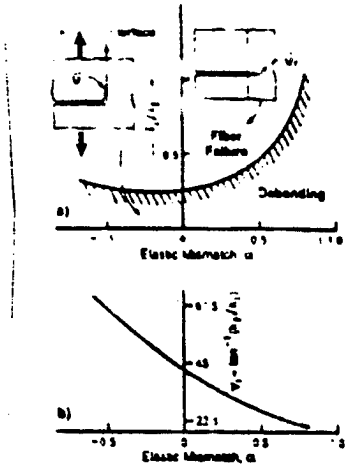
Deadline Dates:
Interdisciplinary, Collaborative Proposals - November 1, Annually
Single Disciplinary Proposals - Specific Program Date

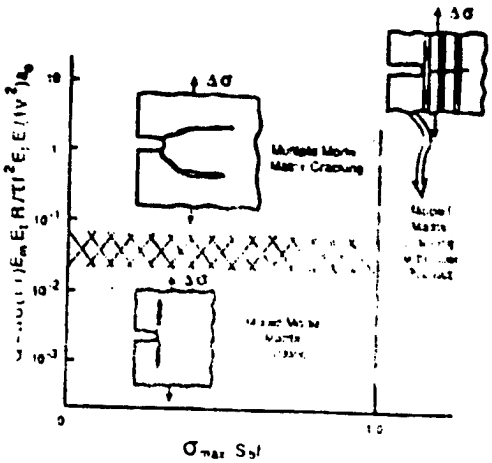
NATIONAL SCIENCE FOUNDATION

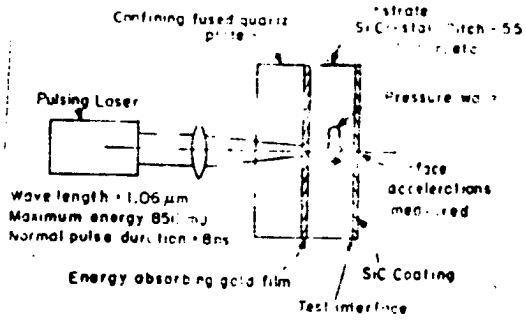
NATIONAL SCIENCE FOUNDATION

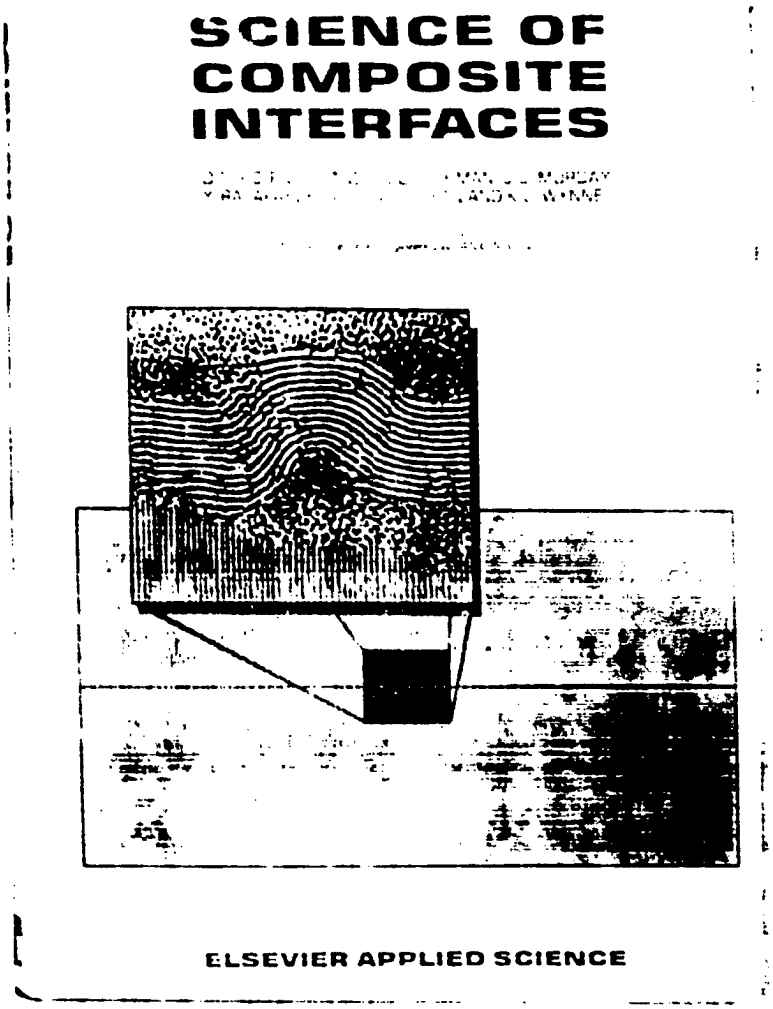


ONR INITIATIVE
COMPOSITE INTERFACES
1986 - 1991





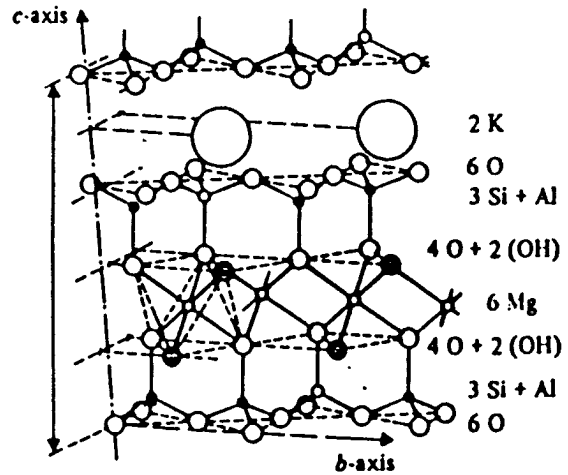




OFFICE OF NAVAL RESEARCH

DR. STEVEN FISHMAN

MOLECULAR DESIGN OF COMPOSITE INTERFACES



$KMg_3(Si_3AlO_{10})(OH)_2$

PHLOGOPITE

OBJECTIVES:

- TO UNDERSTAND RELATIONSHIP BETWEEN CHEMISTRY, MORPHOLOGY, AND MECHANICAL BEHAVIOR OF COMPOSITE INTERFACES
- TO INTERGRATE INTERFACIAL CHEMICAL UNDERSTANDING INTO PROCESSING SCIENCE OF STRUCTURAL COMPOSITES

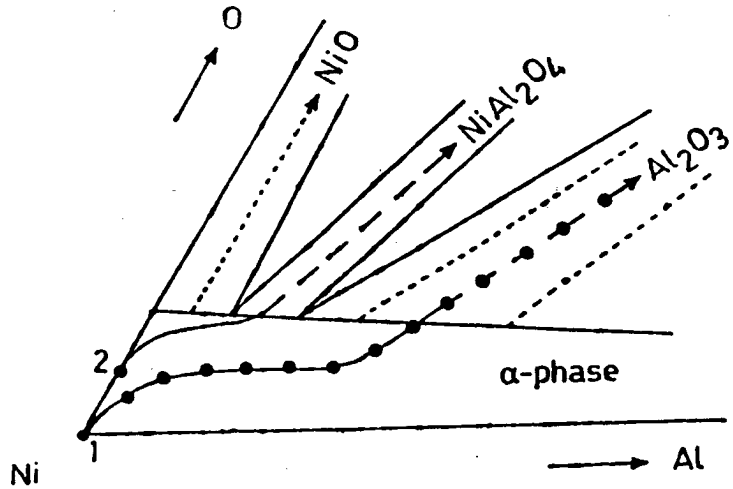
APPROACH:

- EXTEND AND APPLY NEW CHEMICAL CHARACTERIZATION TECHNIQUES TO INVESTIGATE INTERFACIAL CHEMISTRY IN REAL TIME
- NEW PRE-POLYMER ROUTES TO TAILOR CHEMISTRY OF ORGANIC AND INORGANIC COMPOSITE INTERFACES
- CORRELATE INTERFACIAL CHEMISTRY WITH MECHANICAL BEHAVIOR THROUGH COUPLED EXPERIMENT AND THEORY
- MODIFY COMPOSITE PROCESSING TO ENCOMPASS DESIRED CHEMISTRY/MORPHOLOGY

PAYOFF:

- ABILITY TO CONTROL AND OPTIMIZE COMPOSITE INTERFACIAL BEHAVIOR
- SYNTHESIS OF NEW FAMILIES OF COST EFFECTIVE, HIGH PERFORMANCE COMPOSITES

IN-SITU STRUCTURAL COMPOSTES



Example of Matrix Alloying to Modify the Reaction Path In Ni/Al_2O_3 Couple

OBJECTIVES:

- UNDERSTAND/EXPLOIT THERMODYNAMICS/KINETICS OF CONDENSED PHASE REACTIONS BETWEEN HETEROGENEOUS MATERIALS TO FORM "IN-SITU", AND AT THE SAME TIME, REINFORCEMENTS, MATRICES, AND INTERPHASES

APPROACH:

DEVELOP THEORY AND EXPERIMENTAL METHODS FOR PROCESSING SCIENCE AND MICROSTRUCTURAL CONTROL BY:

- KINETIC BIASING OF REACTION PATHWAYS
- SOLID DISPLACEMENT REACTIONS
- INTERNAL REDUCTION REACTIONS

PAYOFF:

- NEW FAMILIES OF HIGH PERFORMANCE STRUCTURAL MATERIALS WITH PROPERTIES AND MICROSTRUCTURES NOT NOW AVAILABLE
- COSTS AN ORDER OF MAGNITUDE LESS THAN CURRENT MATERIALS

ONR CERAMICS RESEARCH
CONTRACTOR LIST

CONTRACTORS	FY92	FY93
SOWRI, LANKFORD	140000	140000
NBS, STEVE FREIMAN	125000	125000
UCSB, TONY EVANS	332662	64700
UTRC, JOHN BRENNAN	125000	125000
NBS, BRIAN LAWN	105000	105000
USNA, DENNIS HASSON	35000	35000
BROWN U./S. NUTT	115000	120000
UCSB ISRAELACHVILI J	127310	0
PENN. STATE/PANTANO	122512	0
CORNELL/SASS	200000	200000
N.M. TECH/CHAWLA	89000	89000
CORNING GLASS WORKS	100000	0
UCSB/F. LANGE	160060	0
CLARKE/UCSB	146000	156000
U. OREGON/JOHNSON	74000	76000
ROCKWELL/D. MARSHALL	120000	119879
U. PA/WAYNE WORRELL	122000	100000
HALLORAN/U. MICH	25000	0
BROWN, S. SURESH	100000	108000
LEHIGH, H. CHAN	153630	140759
HENAGER/BATTELLE NW	150000	150000
U. TEX/H. MARCUS	100000	140000
U. MICH/R. LAINE	90000	90000
CAMBRIDGE U/M. ASHBY	35000	105000
TOTAL	2,892,174	2,189,338 *

*COMMITTED ONLY

OFFICE OF NAVAL TECHNOLOGY (ONT) CERAMICS PROGRAM

PRESENTED TO

INTERAGENCY COORDINATING COMMITTEE ON STRUCTURAL CERAMICS

13 MAY 1992

DR. WILLIAM T. MESSICK
MATERIALS TECHNOLOGY AREA MANAGER

F174/0050792/BM/832

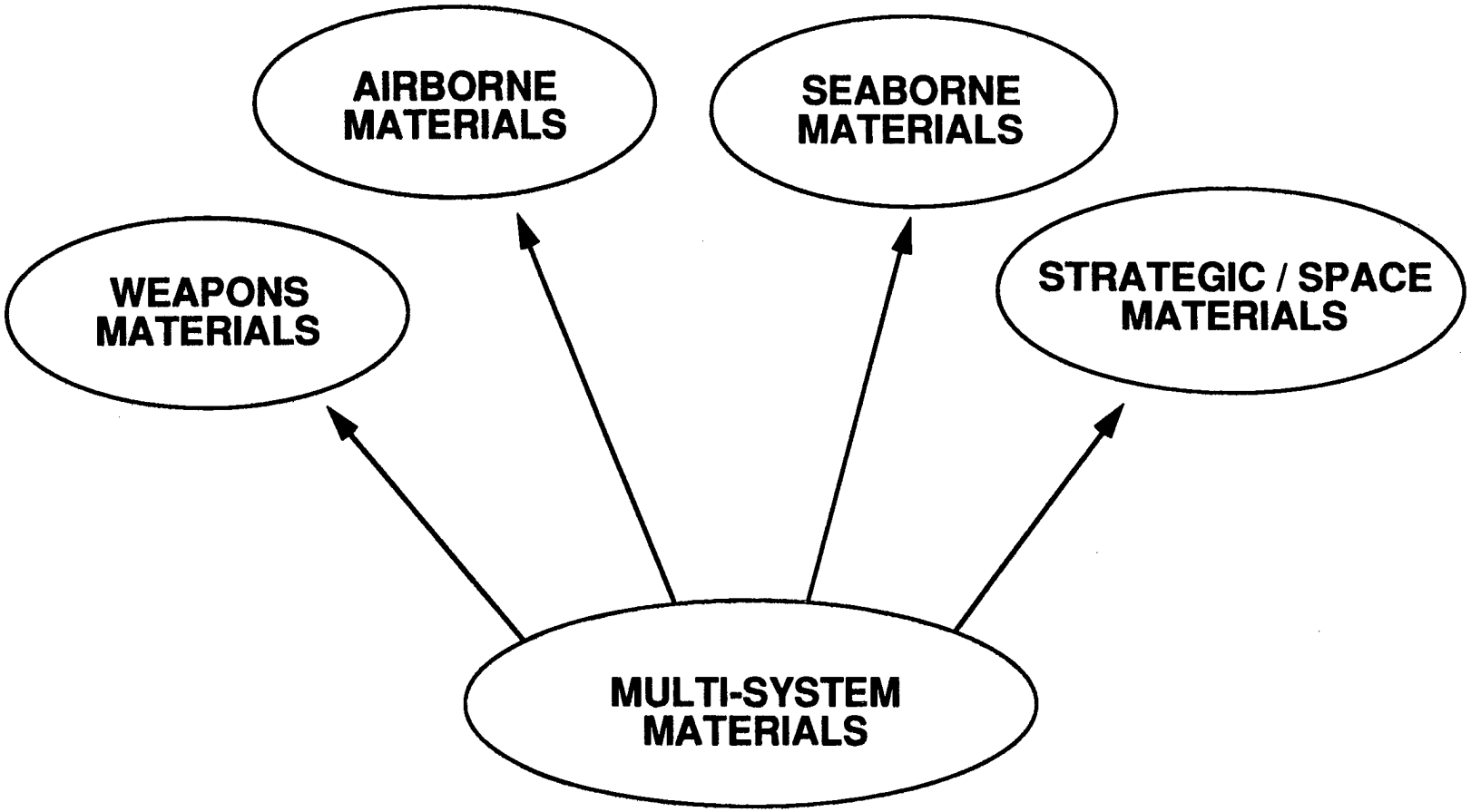
OUTLINE

- ONT MATERIALS PROGRAM OBJECTIVES
- PROGRAM ORGANIZATION
- CERAMIC APPLICATIONS
- ONT "IN-HOUSE" PROGRAM

OFFICE OF NAVAL TECHNOLOGY
MATERIALS PROGRAM OBJECTIVES

- EXPLORATORY DEVELOPMENT OF:
  - STRUCTURAL, PROPULSION, INERT WARHEAD, MACHINERY AND FUNCTIONAL MATERIALS AND PROCESSES
- FOR APPLICATION IN:
  - MISSILES, TORPEDOES, AIRCRAFT, SHIPS, SUBMARINES, SPACECRAFT AND SHORE FACILITIES
- IN ORDER TO:
  - INCREASE SYSTEM AFFORDABILITY (LOWER ACQUISITION COSTS, REDUCED MAINTAINABILITY AND LONGER LIFE)
  - INCREASED SURVIVABILITY
  - INCREASED PERFORMANCE

FIVE (5) MATERIALS TECHNOLOGY THRUST AREAS



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ONT PROGRAM PLAYERS

6.2 FUNDING: ONT

PERFORMERS:

WEAPONS MATERIALS

- NAVAL SURFACE WARFARE CENTER (NSWC) / DAHLGREN DIV (DD) / WHITE OAK
- NAVAL AIR WARFARE CENTER (NAWC) / WEAPONS DIV / CHINA LAKE

AIRBORNE MATERIALS

- NAWC / AIRCRAFT DIV (AD) / WARMINSTER
- NAWC / AD / TRENTON

SEABORNE MATERIALS

- NSWC / CARDEROCK DIV / ANNAPOLIS
- NAVAL CIVIL ENGINEERING LAB / PORT HUENEME
- NAVAL RESEARCH LAB (NRL)

STRATEGIC / SPACE MATERIALS

- NSWC / DD / WHITE OAK

MULTI-SYSTEM MATERIALS

- NRL
- ONT

F174/0050792/BM/832

POTENTIAL NAVAL APPLICATIONS FOR CERAMICS

APPLICATION AREAS	KEY DRIVERS
● ENGINE COMPONENTS (AIRCRAFT, MISSILES, SHIPS) — GAS TURBINES (ROTATING AND NONROTATING PARTS) — SOLID ROCKETS (NOZZLES)	● HIGHER OPERATING TEMPERATURES, WEIGHT REDUCTION, ENVIRONMENTAL RESISTANCE, DECREASED SIGNATURES
● AIRFRAME COMPONENTS (AIRCRAFT, MISSILES, REENTRY VEHICLES) — CONTROL SURFACES — ENGINE EXHAUST PROTECTION — PRIMARY STRUCTURE	● HIGHER OPERATING TEMPERATURES, ENVIRONMENTAL RESISTANCE, DECREASED SIGNATURES, WEIGHT REDUCTION
● ELECTROMAGNETIC TRANSPARENCIES — MISSILE RADOMES	● RAIN EROSION RESISTANCE, HIGHER OPERATING TEMPERATURES
● THERMAL PROTECTION COMPONENTS — VERTICAL LAUNCH SYSTEM LINERS	● ENVIRONMENTAL RESISTANCE, HIGHER OPERATING TEMPERATURES
● ELECTRONICS PACKAGING — CONSTRAINING CORE FOR PRINTED WIRING BOARDS	● THERMAL EXPANSION CONTROL, WEIGHT REDUCTION, STIFFNESS

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PRINTED WIRING BOARD (PWB) CONSTRAINING CORE

OBJECTIVE

DEVELOP CARBON FIBER-SILICON CARBIDE MATRIX COMPOSITES FOR CONSTRAINING GLASS-EPOXY PWB THERMAL EXPANSION

- REPLACEMENT FOR COPPER-INVAR-COPPER
- INCREASE LATERAL THERMAL CONDUCTIVITY

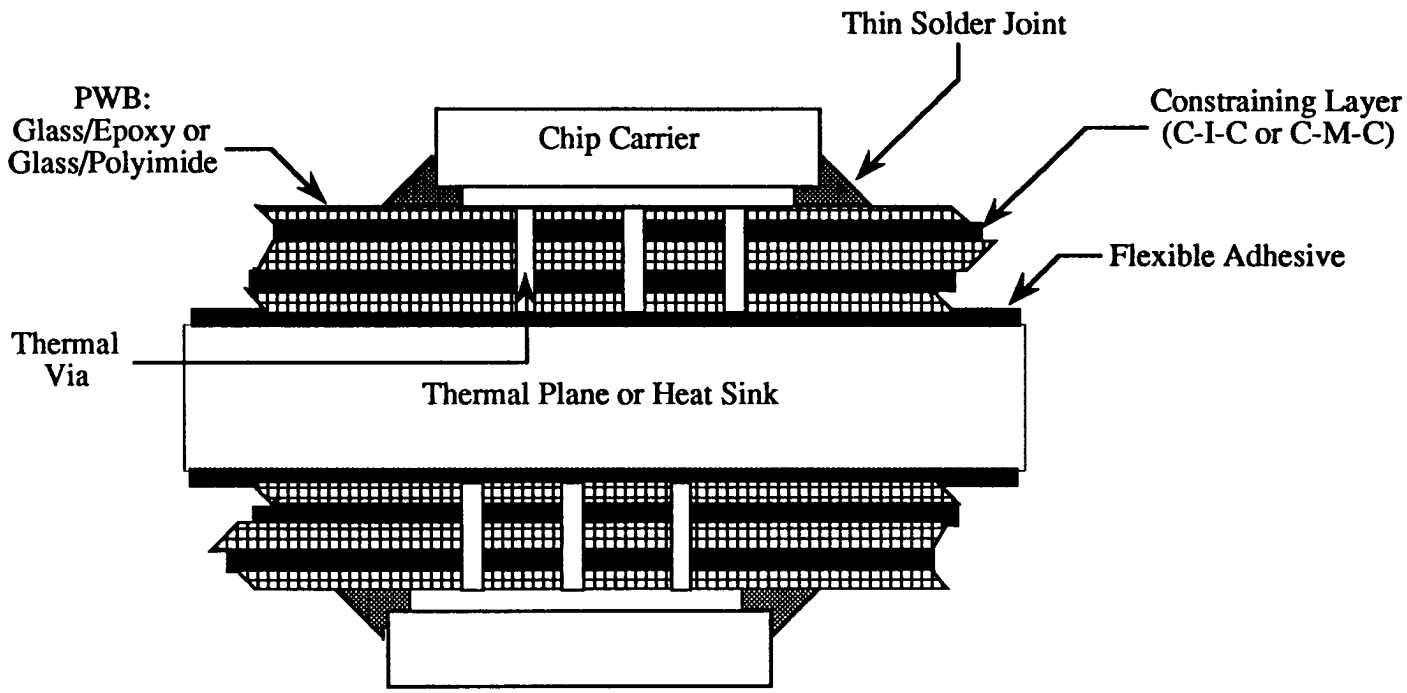
MAJOR THRUSTS

- STANDARD ELECTRONIC MODULE VERSION "E" (SEM-E) APPLICATIONS
- UTILIZE K1100 PITCH-BASED CARBON FIBERS
- INTRODUCE SiC MATRIX VIA CVD
- DEVELOP A ROBUST PROCESS

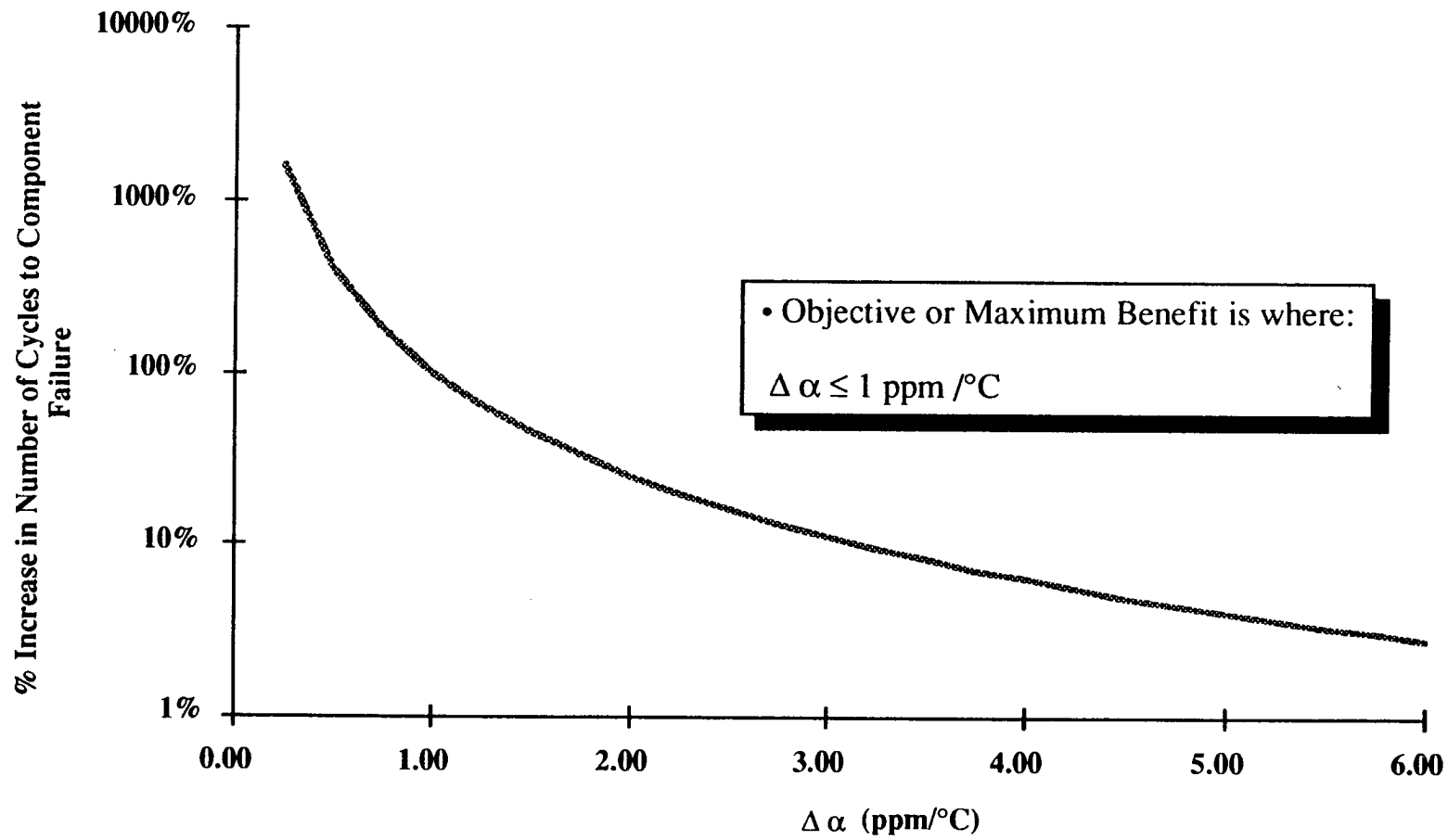
FUNDING	FY91	FY92	FY93
	$50K	$0K	$300K

F174/0050792/BM/832

SEM-E COMPONENT CONSTRAINING CORE



THERMAL EXPANSION MISMATCH



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PWB CONSTRAINING CORE REQUIREMENTS AND INCENTIVES

REQUIREMENTS

- Reduce CTE of glass-epoxy PWB
- Provide high lateral thermal conductivity

Properties of new graphite fibers suggest use of compositeconstraining cores

--> 135 Msi modulus
--> Negative thermal expansion
--> 1100 W/mK thermal conuctivity

INCENTIVES

Relative to Copper-Invar-Copper

--> Minimum volume
--> Minimum weight
--> Broader thermal expansion range for PWB
--> Higher thermal conductivity option

Structural Ceramics Programs

Composites and Ceramics Branch, Code 6370
Naval Research Laboratory
Washington, D. C. 20375-5000

David Lewis III
202-767-2131

Objectives:

- Develop basic science to support utilization of ceramics and ceramic composites in structural applications

- Develop materials and processing technologies suitable for production of structural ceramics and ceramic composites with costs consonant with particular applications

- Develop particular ceramic and ceramic composites systems suitable for specific Navy and DoD applications and work toward techology transfer and manufacturing technology development for these materials.

Strategies:

- Develop basic science necessary for successful development and utilization of ceramics and ceramic composites in structural applications:

  -Processing science for ceramics and ceramic composites
  -Testing techniques
  -Fracture, fracture toughness of ceramics and ceramic composites
  -Interface control in continuous fiber CMC's
  -'Correct' mathematical techniques for data analysis and parameter estimation
  -Non-linear modelling of thermal/physical/chemical processes at composite interfaces

Strategies (cont.):

- Develop affordable ceramics and ceramic composites:

  -Material systems with inherent 'low' cost
  -Material processing techniques with inherent 'low' cost
  -Net-shape processing techniques
  -Surface laminate approach to CMC's

Strategies (cont.):

- Develop specific systems for Navy and DoD applications:

  -Sandwich structure radome to meet requirements for high speed, rain erosion resistant radome
  -Sandwich structure utilizing ceramic cores and CMC skins for gas turbine components (combustor, augmentor, nozzle flaps)
  -Surface laminate toughened silicon nitride for rotors, compressors, APU rotors
  -Structural composites with embedded piezoelectrics for smart structures, damping

Strategies (cont.):

- Stress technology transfer and manufacturing technology development through ties to manufacturers and end users and related DoD programs:

  -GE, P&W, Garrett,..., for gas turbines
  -NSWC, 6.2 Materials Blocks for radomes, gas turbines

Major Thrusts:

- Smart structures using embedded piezoelectrics (active and passive)

  -Adaptive, self-repairing structures
  -Damped structures for space

- Ceramic Composite Surface Laminate Programs (DARPA, NRL 6.2 Emerging Materials Block)

  -Surface toughening of near-net shape ceramics
  -Sandwich structures for gas turbines, radomes, lightweight armor

Programmatic Changes:

- Greater emphasis on applied research
- Increased internal (NRL) 6.2 funding
- Increased emphasis on technology transfer, dual-use technologies,...

Funding:

	FY91	FY92	FY93
DARPA	$160k	$200k	$300k
NRL 6.2		$120	$150
NRL 6.2*			$250*
NRL 6.1	$264	$300	$250
SDIO		$ 50	$100
Totals:	$424k	$670k	$1050k* $ 800k

*Material processing facilities

CERAMIC MATERIALS
NAVAL SURFACE WARFARE CENTER
Dahlgren Division

Dr. Inna G. Talmy

Sponsors: NSWC IR, NSWC IED and ONT Weapons and Spacecraft Materials Block Programs

NSWCDD Ceramics Program

Task	FY91 ($K)	FY92 ($K)	FY93* ($K)	Performer
Phosphate Ceramics Research (IR)	117	45	45	NSWCDD
Phosphate Bonded Non-Oxide Ceramics (IED)	-	-	200	NSWCDD
Phosphate Bonded Non-Oxide Ceramic Composites (Engineering Development, Block)	150	275	300	NSWCDD
Thermally Stable Dielectric Oxide Ceramics (Celsian, IED)	189	10	-	NSWCDD
Celsian Radomes	350	344	515	Loral
Mullite Whisker Composites	25	-	-	NSWCDD
Mullite Felt Composites	50	10	-	Ceramatec, Northwestern
Nitroxyceram Radomes	180	50	-	Loral
VLS	-	142	200	NSWCDD
Nanophase Diamond Consolidation	-	20	150	NSWCDD
TOTALS	1061	896	1410	

**projected*

IA02.CAM 5/12/92

FY 92 CERAMICS SBIR's

Project	Funding ($K)	Performer
Injection Molding of Whisker Reinforced Composites	392	Lone Peak Engineering
Zirconium Diboride Matrix Continuous Fiber Composites	515	Advanced Ceramics Research
CVI of Carbide Matrix Carbon Fiber Composites	462	MSNW

Structural Ceramics Projects at NSWCDD

- **CELSIAN RADOME DEVELOPMENT**
  Advantages - improved temperature, erosion, and impact resistance
  Status - development complete, transitioned to Block Program for Scale-up by Loral Aeroneutronic (Newport Beach, CA)
  Awards - best NSWC IR FY88, best NSWC IED FY91
  Four US patents

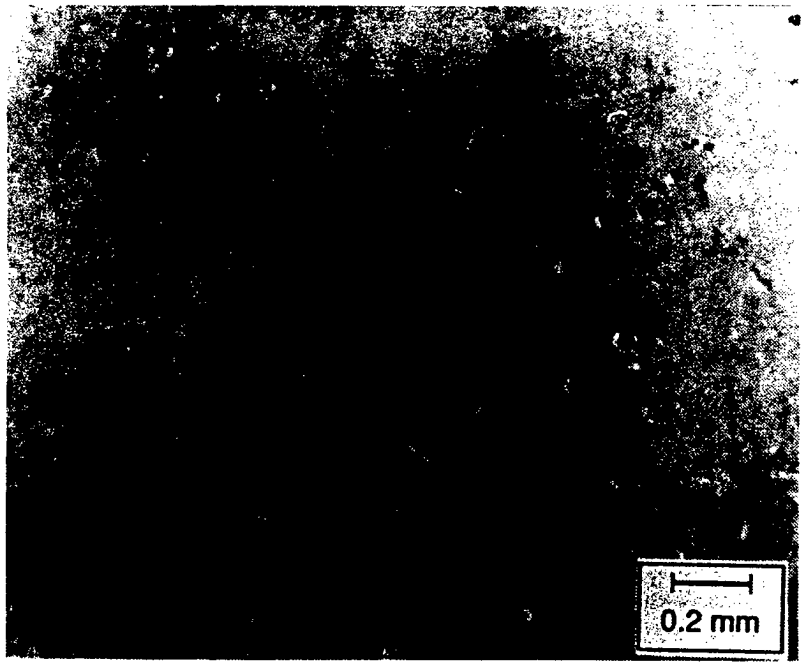
- **MULLITE WHISKERS AND FELT**
  Advantages - oxygen stable reinforcement for composites, in-situ net shape capabilities, high temperature chemical stability
  Status - development complete, technology transfer efforts ongoing (DOE coal gasification filters)
  Three US patents

- **PHOSPHATE BONDED CERAMICS**
  Advantages - low cost, low temperature processing, high temperature capabilities, excellent dielectric properties for radomes
  Status - ongoing, started in FY90

- **CVD AND CVI OF CERAMICS**
  Status - Equipment purchased in FY91 and set up in FY92

PHOTOMICROGRAPHS OF SAMPLE SURFACES AFTER SINGLE PARTICLE IMPACT TEST

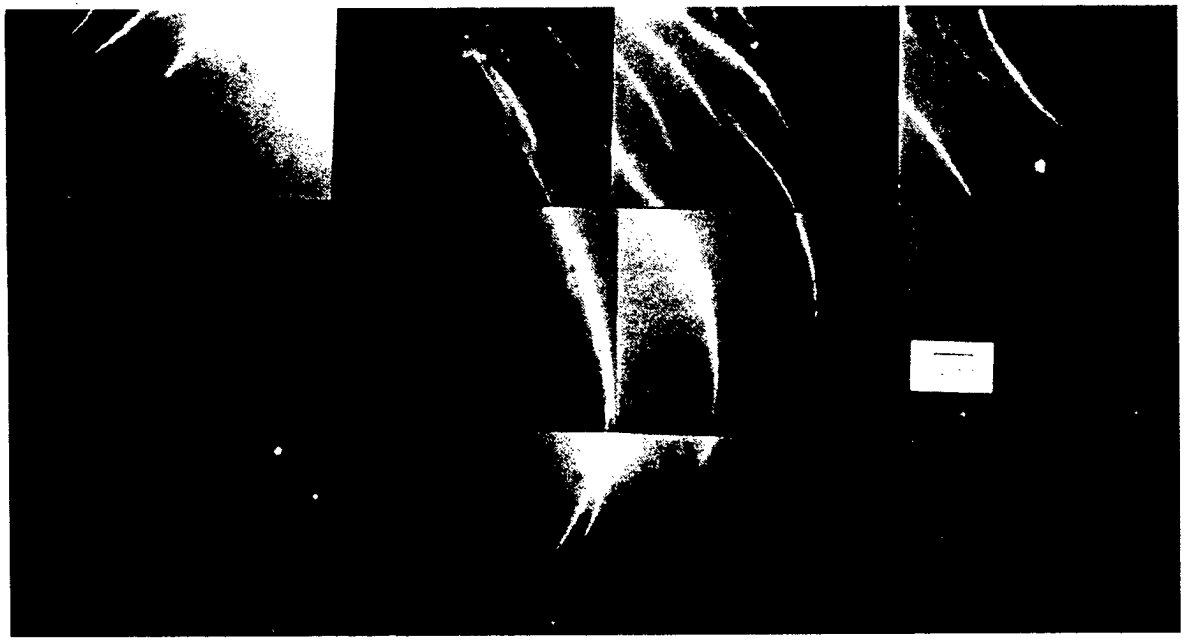
BAS
(1.71 Km/s)



Si_3N_4 W/BAS
(1.70 Km/s)

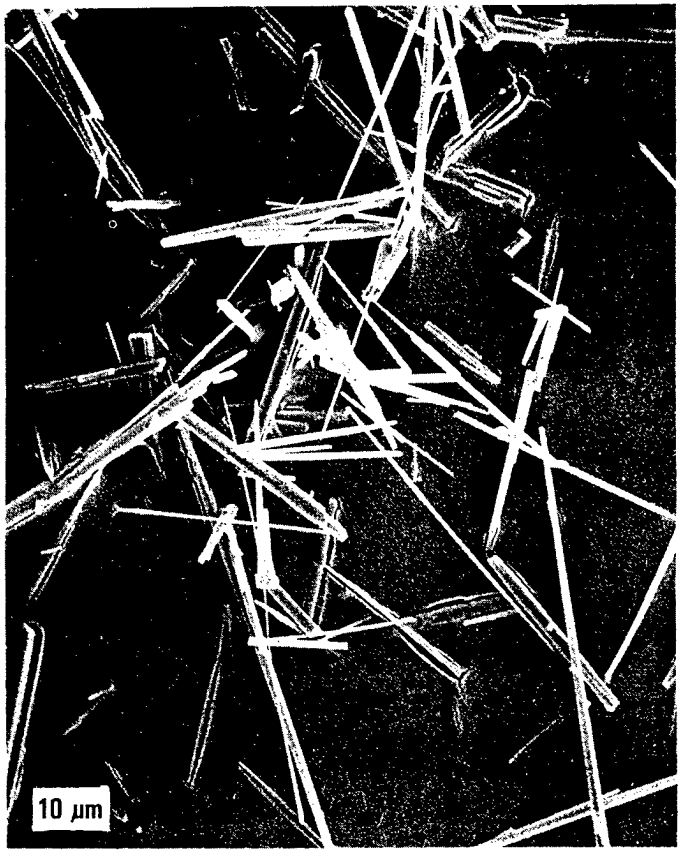
PHOTOMICROGRAPHS SHOWING SUBSURFACE CRACK PROPAGATION AFTER SINGLE PARTICLE IMPACT TEST

BAS
(1.51 Km/s)

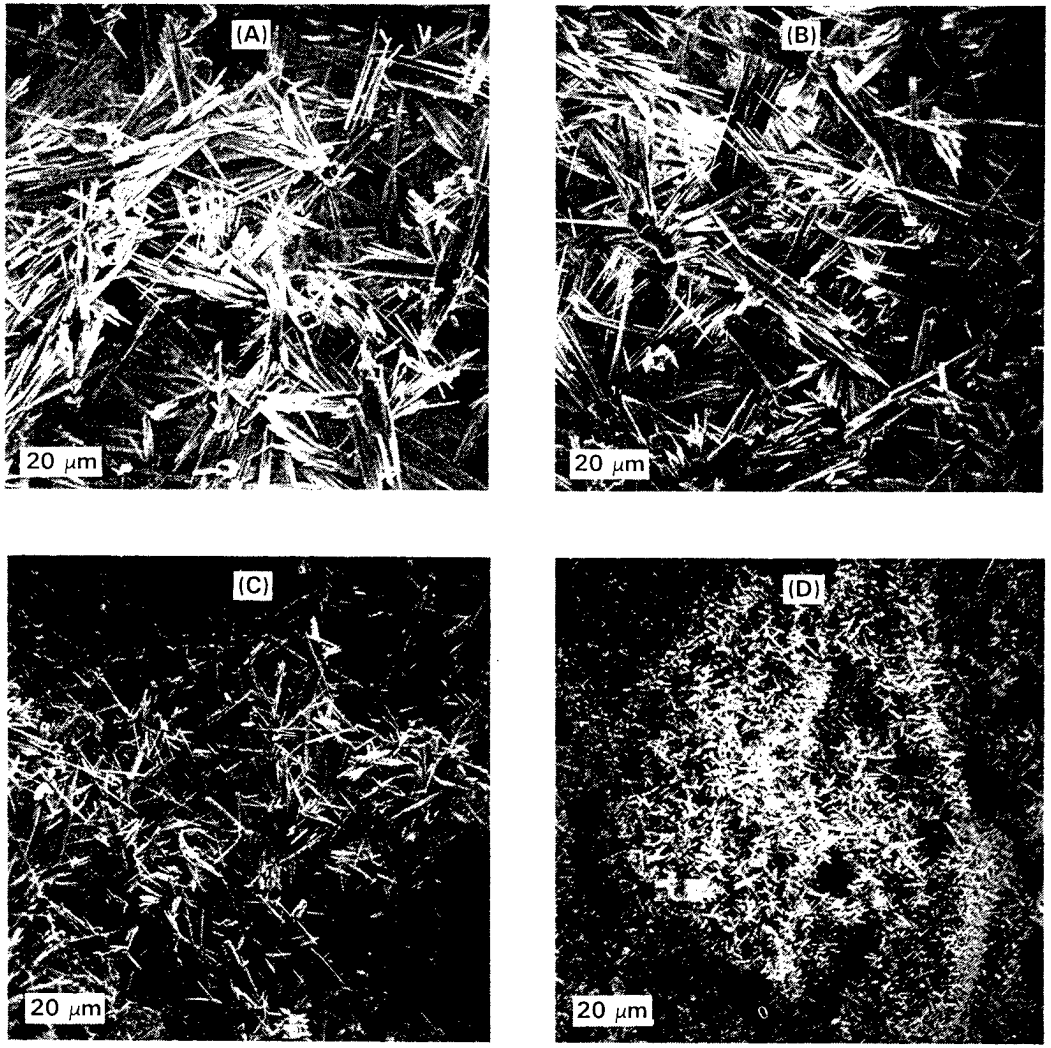


Si_3N_4 W/BAS
(1.49 Km/s)

SEM MICROGRAPHS OF MULLITE WHISKERS



MULLITE-WHISKER FELT WITH SUBSTITUTION OF AL_2O_3 FOR ALF_3



SCANNING ELECTRON MICROGRAPHS OF MULLITE-WHISKER FELT WITH SUBSTITUTION OF (A) 0%, (B) 25%, (C) 50%, AND (D) 75% Al_2O_3 FOR AlF_3

On-Going Efforts

- Upgrading equipment for felt preparation
- Provide felt samples to interested parties
- Transfer of technology
  - Strong interest from DOE for hot gas filtration applications (coal gasification, waste incineration)
    - Pore structure determined
    - Chemical compatibility is adequate
- Use of felt for composites
  - Sol-gel infiltration (Ceramatec)
  - Microwave-assisted CVI (Northwestern)
  - Surface laminates (NRL)

Metal Phosphates

Formation: Form by low temperature reactions of metal oxides, hydroxides, or salts with H_3PO_4 to develop sufficient strength for applications in monolithics and composites

Advantages:

- Low temperature processing
- High use temperature
- Low linear changes in processing
- Polymer matrix type processing

Applications:

- Advanced radomes
- Structural materials (leading edges, engine parts, ducts)
- High temperature adhesives
- Special applications

phosgen.dah

PHOSPHATE BONDED SILICON NITRIDE PROPERTIES
(FIRING TEMPERATURES: 700 – 900 °C)

COMPOSITION	DIELECTRIC CONSTANT	% CHANGE TO 1000°C	LOSS TANGENT	FLEXURAL STRENGTH (MPA)	THERMAL EXPANSION 30-1000°C (/°C)
30% $ALPO_4$	4.03	5.2	0.0055	51	3.56×10^{-6}
30% $ALPO_4$ (CIPED)	–	–	–	85	–
30% BPO_4	3.99	6.5	0.012	53	–
30% ZRP_2O_7	4.98	6.5	0.0031	95	–
30% $ZR_2P_2O_9$	5.32	6.5	0.0026	95	–
Radome Requirements	<9	<7	<0.1	>35	

Composite Components Evaluated

Binder

$AlPO_4$
BPO4
$AlPO_4$/ BPO_4
ZrP_2O_7
ZrP_2O_7/ BPO_4

Reinforcement

SiC Whiskers
SiC Platelets
Si_3N_4 Whiskers
SiO_2 Fibers (Astroquartz, HPQY)
Carbon Fibers
Tyranno
Nicalon
HPZ

Filler

Si_3N_4
Al_2O_3

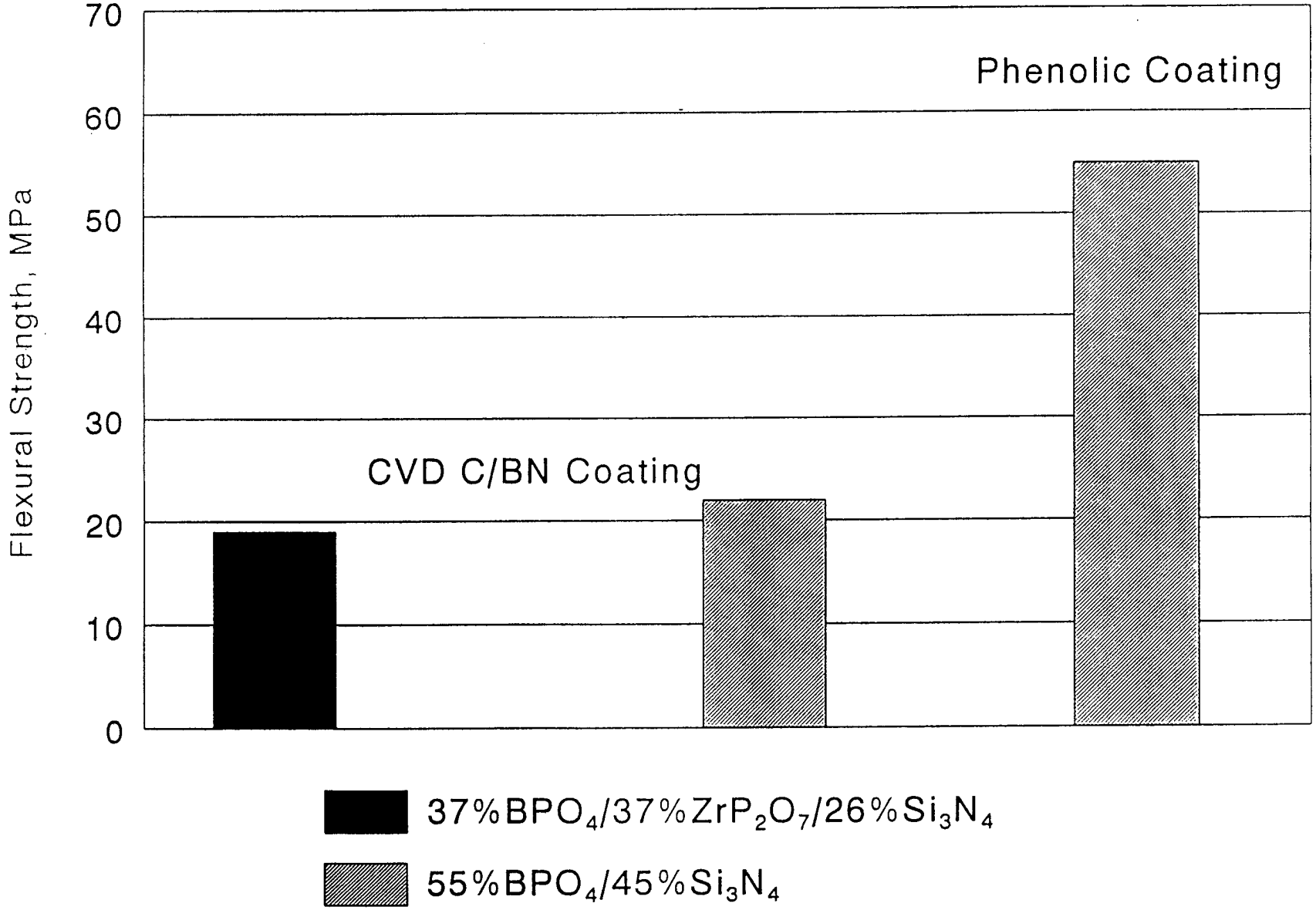
Interface Coating

CVD BN/C
CVD C
In House Phenolic Carbon

Reinfiltration

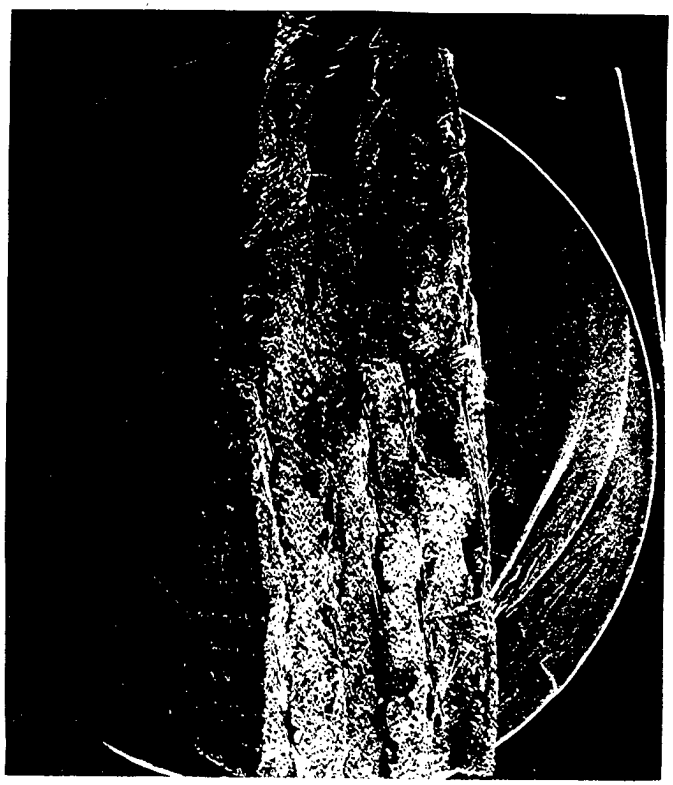
BPO_4
ZrP_2O_7
Monoaluminum Phosphate

Flexural Strength of HPQY Silica Fiber Phosphate Matrix Composites



HPQY Fiber Composites
BPO_4/45% Si_3N_4 Matrix

CVD 1 mm



PHENOLIC 1 mm

Naval Surface Warfare Center Ceramics Research

Naval Surface Warfare Center (R31)
Dahlgren Division, White Oak Detachment
10901 New Hampshire Ave.
Silver Spring, MD 20903-5000

Points of Contact:

Mr. David Sudduth	(301) 394-2262	Head, Nonmetallic Metallic Materials Branch
Mr. Richard Weller	(301) 394-1317	Group Leader, High Temperature Materials Group
Dr. Inna Talmy	(301) 394-2268	Senior Research Ceramist

STRUCTURAL CERAMICS

NAWC/AD - Warminster, Pa

Randy Sands Code 6063

Objective

* **To Improve Performance of Existing & Future Naval Aircraft Propulsion Systems by Development & Exploitation of Advanced Ceramics & Ceramic Matrix Composites**

Navy Aircraft Support

NAWC - Structural Ceramics Efforts

6.2 IHPTET Block

* SiC/LAS Compglas
* SiC/Si_3N_4 Composites

6.2 Aircraft Materials Block

* Environmental Effects On CMC's
  - Chemical Stability
  - Mechanical Stability

Manufacturing Technology

* Fabrication CMC's
  - Compglas
  - Other Glass-Ceramics

6.1 Basic Research ONR

* Interface Sciences
* High Temperature Mechanical Testing
* Novel Synthesis Pre-Ceramic Polymers

Funding Situation

	FY-92	FY-93	FY-Out Year
6.2 Aircraft Materials Block			
In-House	150 K	200 K	200 K
Contract	230 K	230 K	250 K
6.1 Basic Research			
In-House	200 K	200 K	250 K
Contract	100 K	50 K	?

Future Directions

* *Environmental Effects On CMC's*
  - *Naval Environment*
  - *Oxidation/Thermal Stability*

* *High Temperature Mechanical Testing*

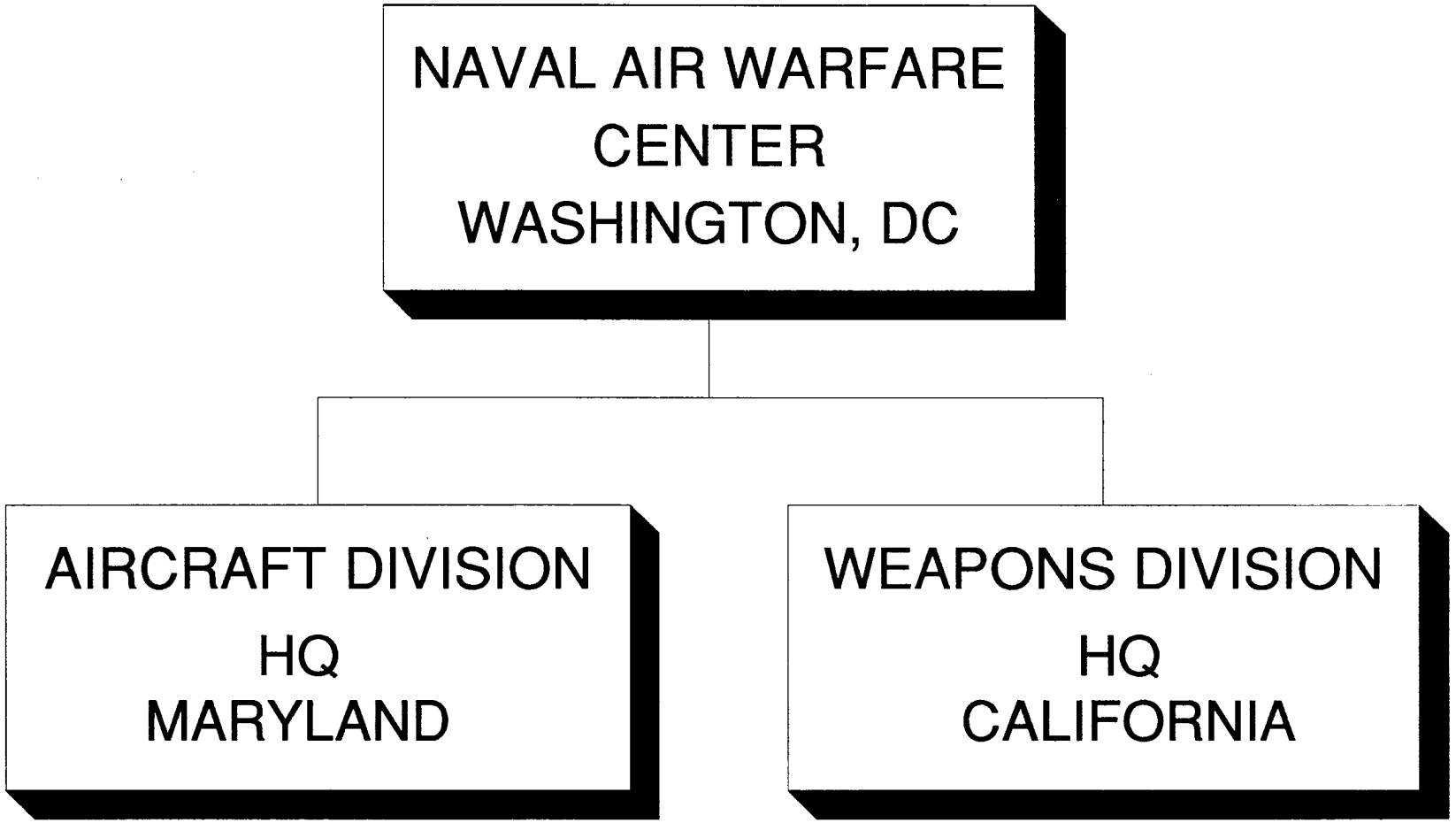
* *Novel Processing Routes*

* *High Temperature Ceramic Systems*
  - *Low Observables*

Naval Air Warfare Center
Aircraft Division
Trenton -

PROVIDE COMPLETE TECHNICAL AND ENGINEERING SUPPORT TO NAVAIR AND THE FLEET FOR:

- **AIR BREATHING PROPULSION SYSTEMS**
- **COMPONENTS**
- **ACCESSORIES**
- **FUELS AND LUBRICANTS**



NAVAL AIR PROPULSION CENTER, TRENTON
NAVAL AIR DEVELOPMENT CENTER, WARMINSTER
NAVAL AIR TEST CENTER, PATUXENT RIVER
NAVAL AIR ENGINEERING CENTER, LAKEHURST
NAVAL AVIONICS CENTER, INDIANAPOLIS

NAVAL WEAPONS CENTER, CHINA LAKE
PACIFIC MISSILE TEST CENTER, PT. MUGU
NAVAL ORDNANCE MISSILE TEST STATION
WHITE SANDS
NAVAL WEAPONS EVALUATION FACILITY
ALBUQUERQUE

OBJECTIVE

DEVELOP AND EVALUATE HIGH TEMPERATURE (2200-2700°F) OXIDATION AND CORROSION RESISTANT CERAMIC COMPOSITES WHICH POSSESS HIGH FRACTURE TOUGHNESS, HIGH STRENGTH AND THERMAL STABILITY. THESE CERAMIC COMPOSITE SYSTEMS HAVE THE POTENTIAL TO BE FABRICATED INTO IHPTET COMPONENTS THAT HAVE A SIGNIFICANT PAYOFF IN THRUST TO WEIGHT. PARTICULAR INTEREST IS IN DEVELOPING CERAMIC COMPOSITES FOR ROTATING COMPONENTS.

MAJOR THRUSTS

- o DEVELOP AND EVALUATE SILICON CARBIDE FIBER REINFORCED SILICON NITRIDE MATRIX COMPOSITES.
- o INVESTIGATE MODIFICATIONS TO THE FIBER/MATRIX INTERFACE IN ORDER TO IMPROVE THERMAL EXPANSION MISMATCH BETWEEN THE FIBER AND MATRIX.
- o INVESTIGATE NEWLY DEVELOPED FIBER COATINGS WHICH HAVE A HIGH PROBABILITY OF SUCCESSFULLY IMPROVING THE FIBER/MATRIX INTERFACE PROPERTIES.

STATUS AND FUNDING

STATUS: PRELIMINARY SiC/Si_3N_4 TAPES HAVE BEEN FABRICATED AND DENSIFIED. FULLY DENSIFIED COMPOSITES GAVE NO INDICATION OF FIBER/MATRIX REACTIONS.

FUNDING(FY/$K):	91	92	93
	150	200	150

Si_3N_4 COMPOSITE COATINGS

CURRENTLY FUNDING OAK RIDGE NATIONAL LABORATORY TO DEVELOP $Si_3N_4/MoSi_2$ COATINGS THAT WILL BE INCORPORATED INTO THE SiC/Si_3N_4 SYSTEM AS EITHER A FIBER COATING OR AS A COMPOSITE OVERCOAT. THESE COATINGS WILL BE EVALUATED TO DETERMINE POTENTIAL FOR IMPROVING MECHANICAL PROPERTIES AND OXIDATIVE STABILITY.

FUNDING(FY/$K):	92	93	94
	50	100	100

OBJECTIVE

IDENTIFY HIGHLY FABRICABLE, STRUCTURALLY EFFICIENT DESIGN APPROACHES AND FABRICATION METHODS TO IMPROVE PERFORMANCE AND REDUCE MANUFACTURING COSTS OF 2-D VECTOR NOZZLE STRUCTURES IN ADVANCED AIRCRAFT ENGINES. LOW COST GLASS-CERAMIC MATRIX COMPOSITE MATERIALS WILL REPLACE HEAVIER WELDED METALLIC STRUCTURES.

GLASS-CERAMIC COMPOSITE COMPONENT FABRICATION

MAJOR THRUSTS

- SUBELEMENT DESIGN AND EVALUATION. DETERMINE MOST EFFICIENT MANUFACTURING METHODS IN TERMS OF MACHINING, JOINING AND REPAIR.
- SCALE-UP AND COST STUDIES.
- FABRICATE SUBCOMPONENT, PREPARE SPECS, DETERMINE INSPECTION METHODS AND PROOF TEST.
- FULL SCALE NOZZLE DESIGN, FABRICATION AND TEST.

STATUS AND FUNDING

PROPOSALS UNDER NAVY BROAD AGENCY ANNOUNCEMENT (BAA) ARE EVALUATED. NAWCADTRN ANTICIPATES A 24-36 MONTH PROGRAM FUNDED AT OVER $ 1M IN FY93.

ADDITIONAL INFORMATION

NAVAL AIR WARFARE CENTER
TRENTON, NEW JERSEY

NAVAL AIR WARFARE CENTER
AIRCRAFT DIVISION, TRENTON
ATTN: D.M. MIGLIACCI, CODE PE34
P.O. BOX 7176
TRENTON, NJ 08628-0176

609-538-6927

M-920508-19U (C) (2129)

SILICON NITRIDE INJECTORS (U)

PRESENTED TO:
FY 1992 INTERAGENCY COORDINATING COMMITTEE ON STRUCTURAL CERAMICS ANNUAL MEETING

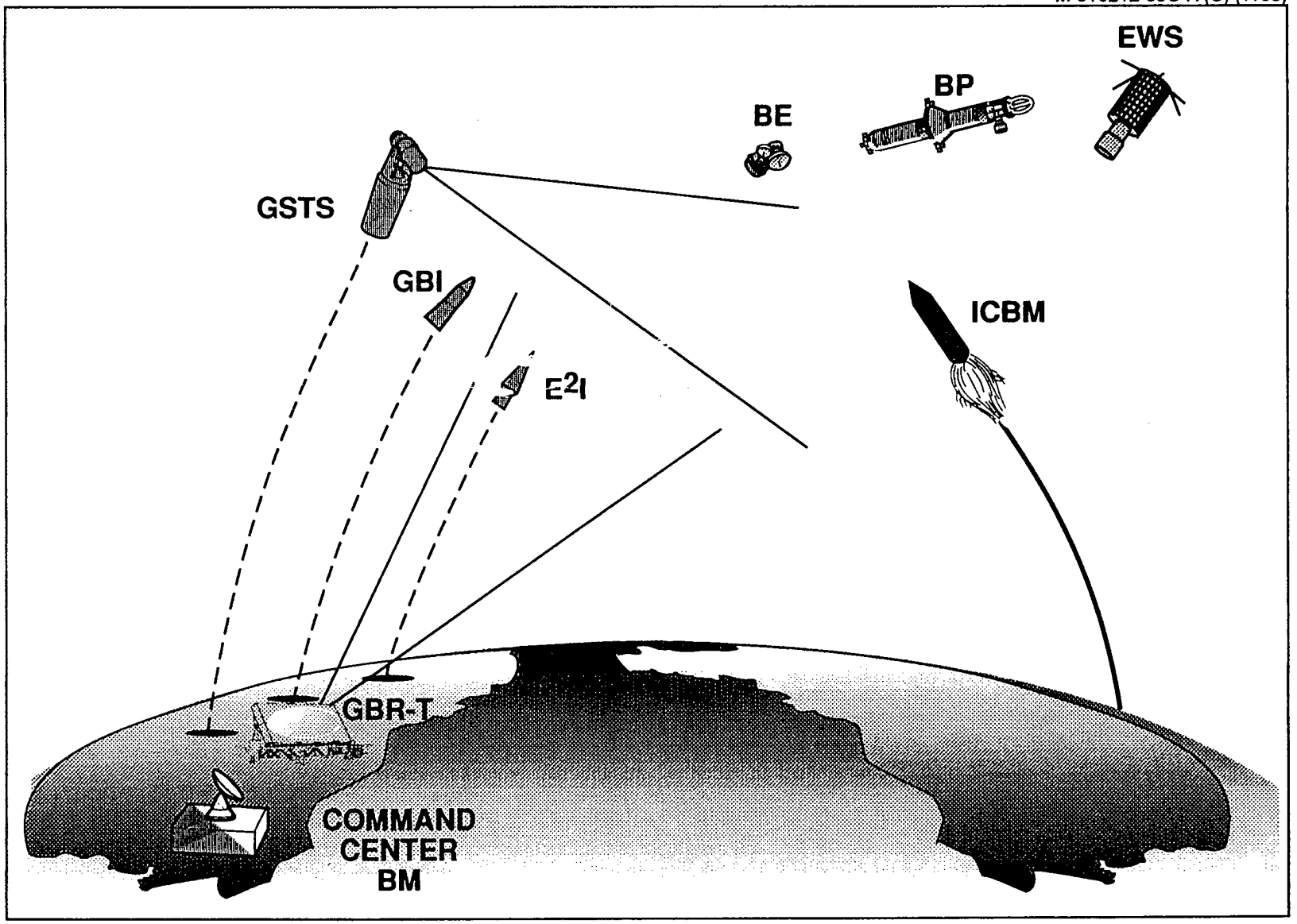
13 MAY 1992

MR. DOUGLAS ENNIS
UNITED STATES ARMY STRATEGIC DEFENSE COMMAND
HUNTSVILLE, ALABAMA

UNCLASSIFED

GPALS - STRATEGIC DEFENSE (U)

M-910212-39U-A (U) (1135)



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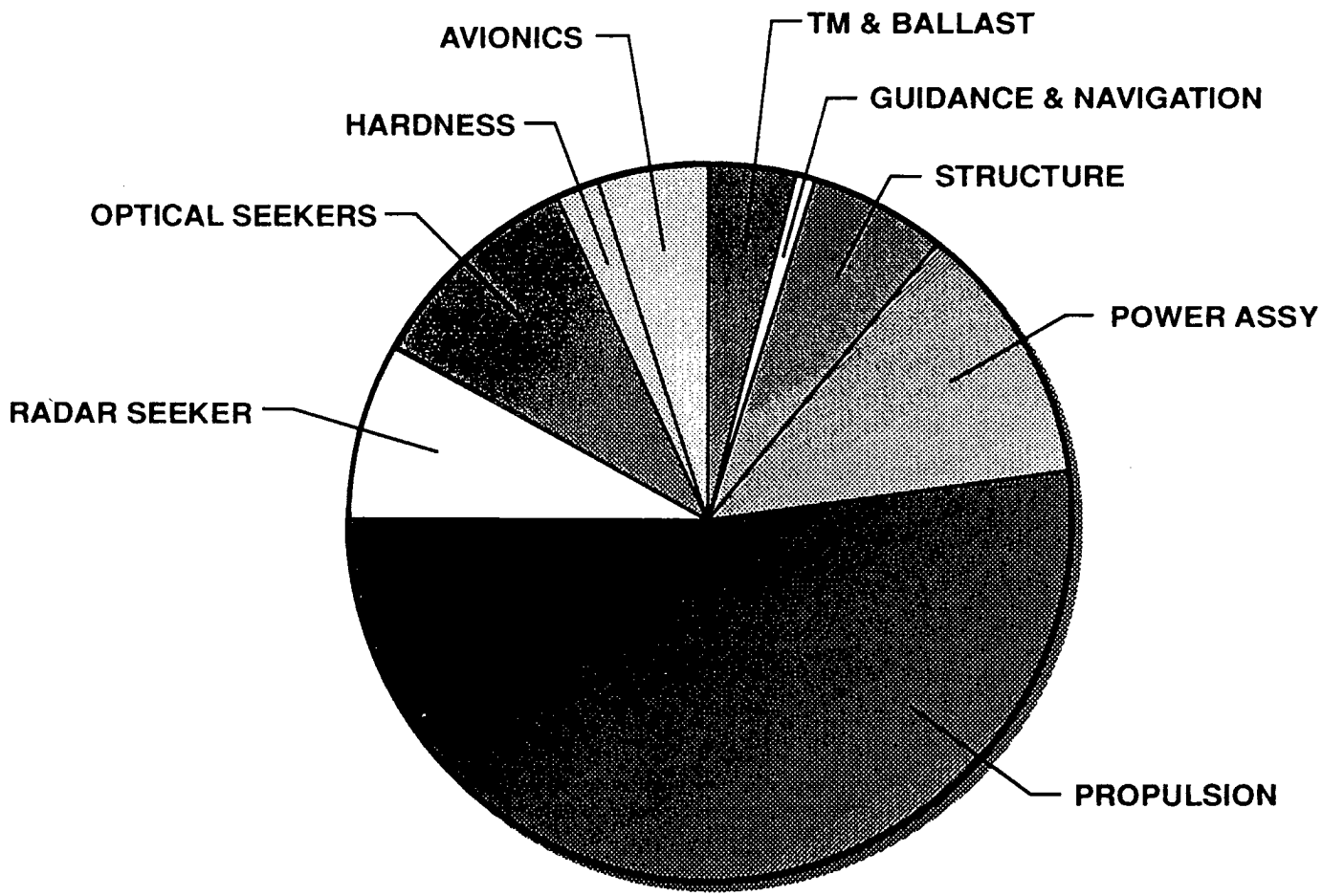
MATERIALS AND STRUCTURES
– SYSTEM MASS PROPERTIES FOR AN EXOATMOSPHERIC INTERCEPTOR – (U)

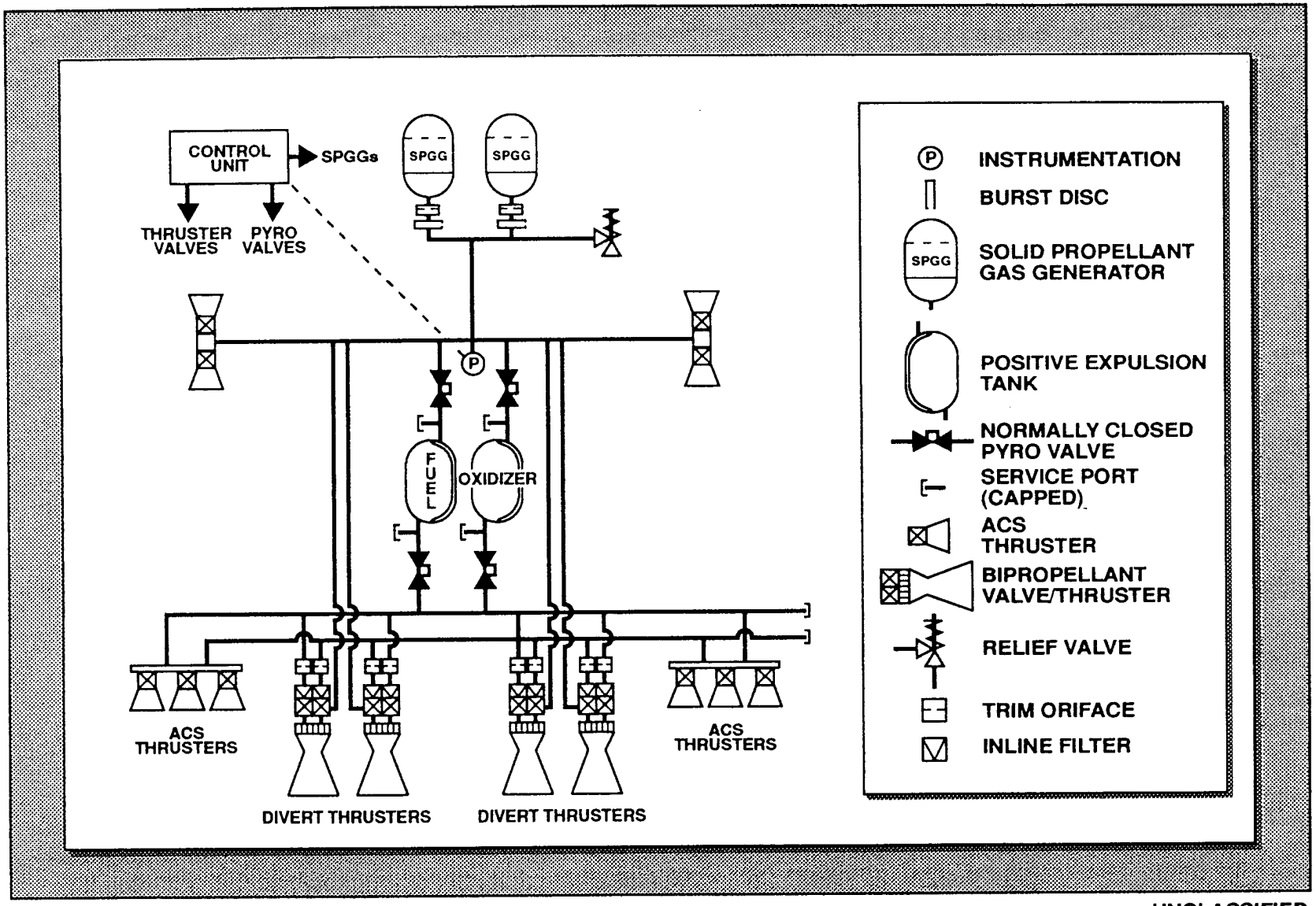
M-920508-03U (C) (2129)



UNCLASSIFIED

TACTICAL GBI
– TACTICAL GBI PROPULSION SYSTEM SCHEMATIC – (U)

M-920508-07U (C) (2132)



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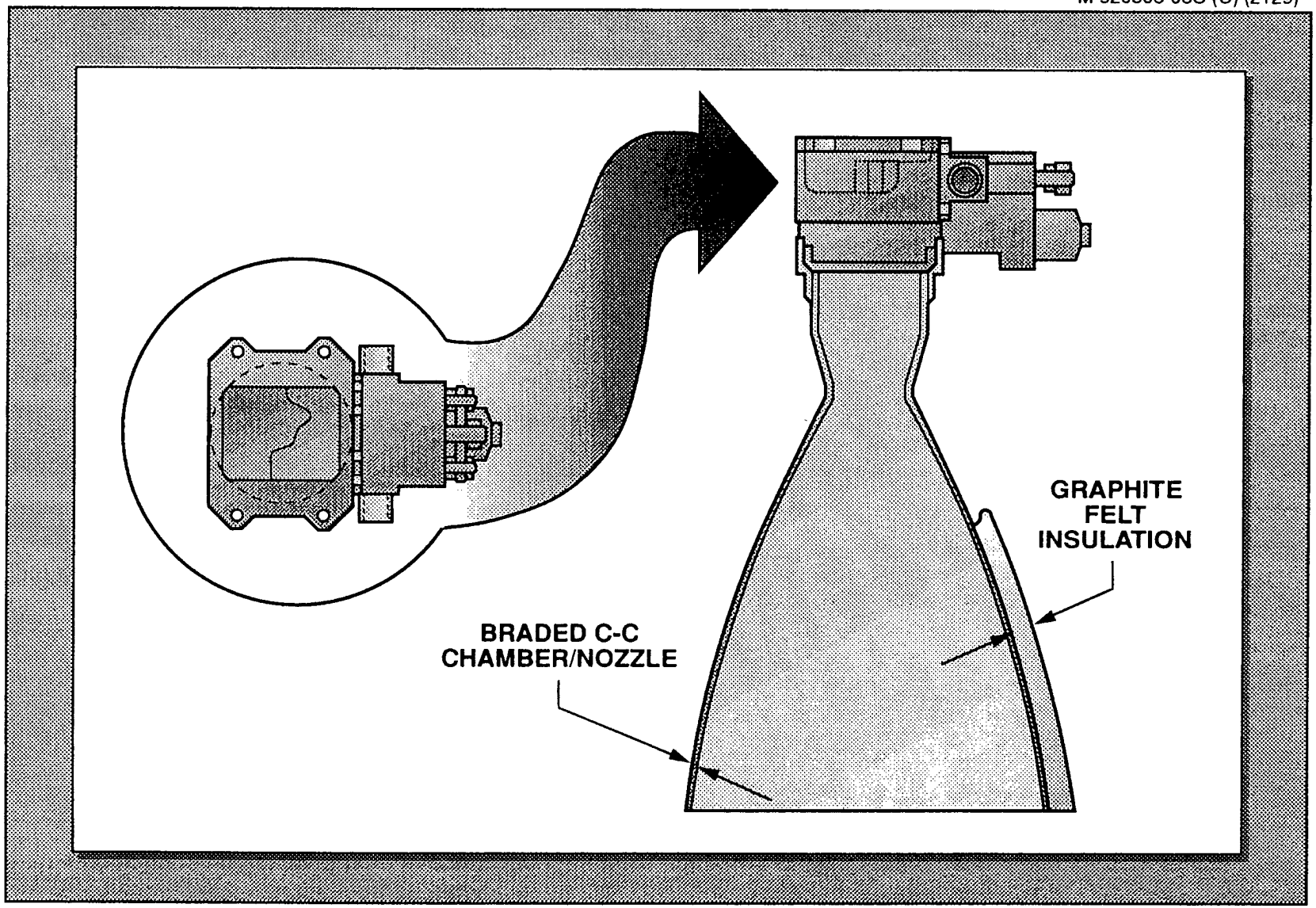
TACTICAL GBI
– CHAMBER/THRUSTER ASSEMBLY – (U)

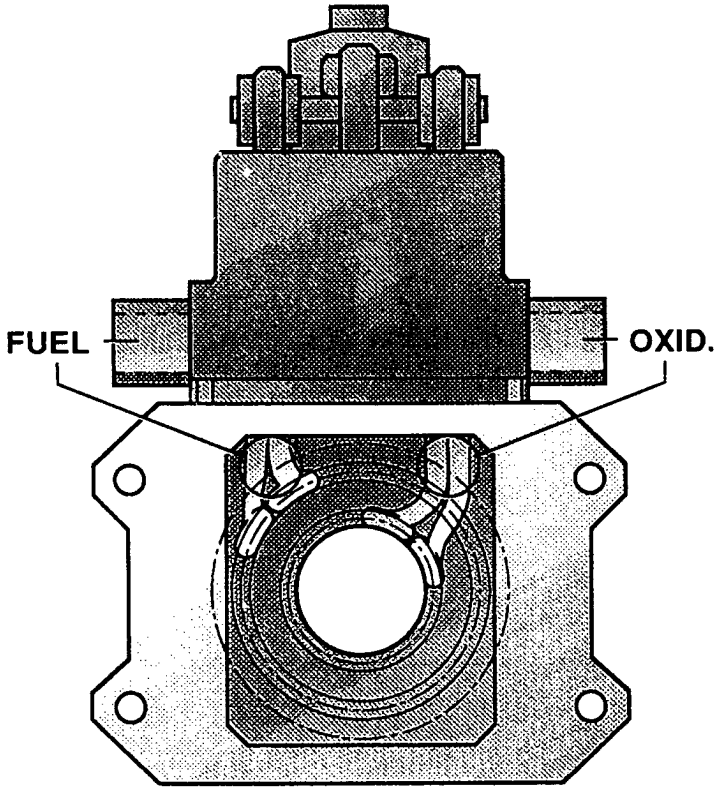
M-920508-08U (C) (2129)

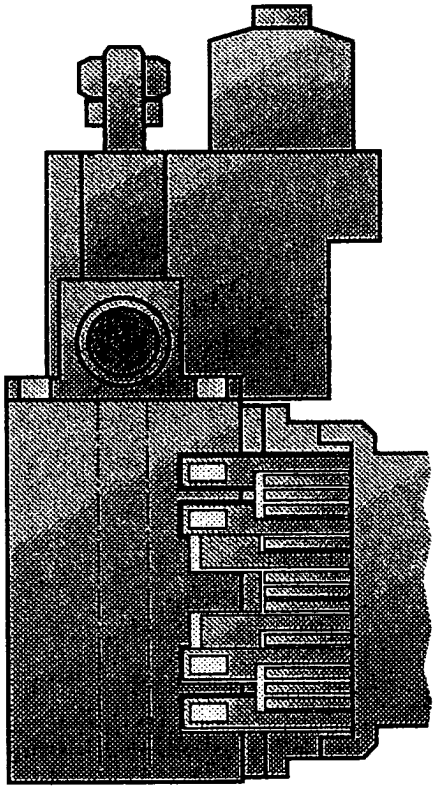


UNCLASSIFIED

TACTICAL GBI
– INJECTOR DESIGN - MANIFOLDING – (U)

M-920508-05U (C) (2129)





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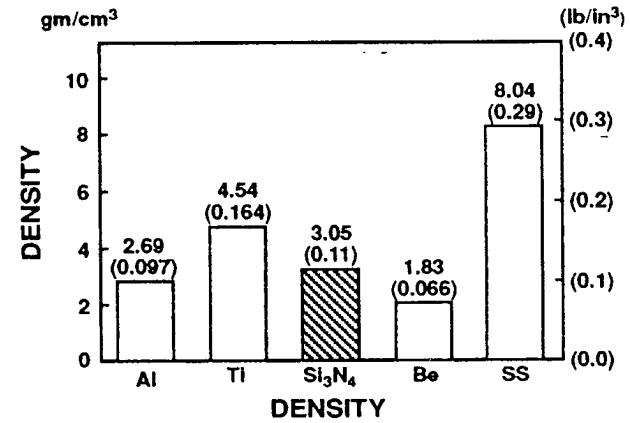
MATERIALS AND STRUCTURES
– MATERIAL REQUIREMENTS FOR PROPULSION APPLICATIONS – (U)

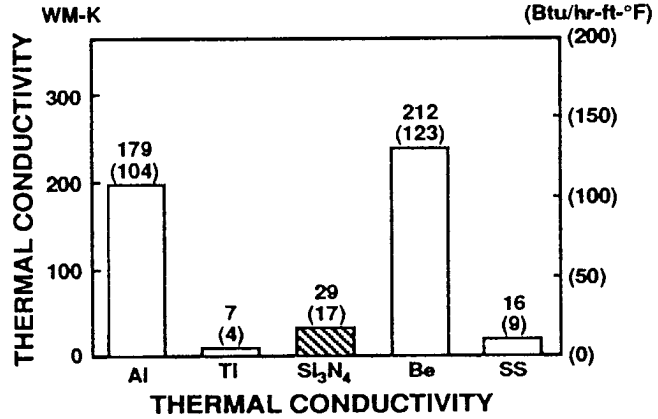
M-920508-01U (C) (2129)

- LIGHT WEIGHT
- HIGH OPERATING TEMPERATURE CAPABILITIES
- SELF QUENCHING IN OXYGEN RICH ENVIRONMENT
- GOOD THERMAL SHOCK RESISTANCE

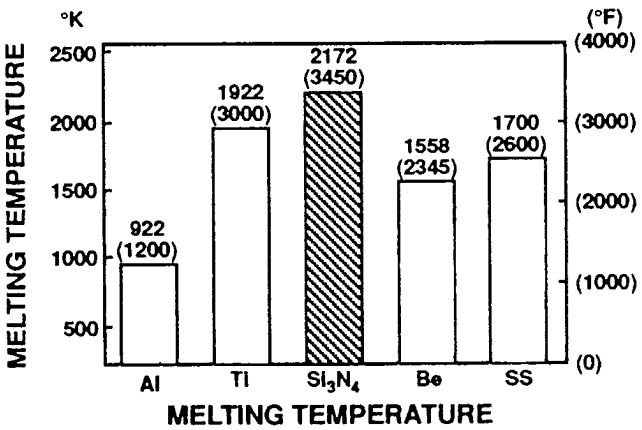
MATERIALS AND STRUCTURES
– PROPERTIES COMPARISON: Al, Ti, Si_3N_4, Be, SS – (U)

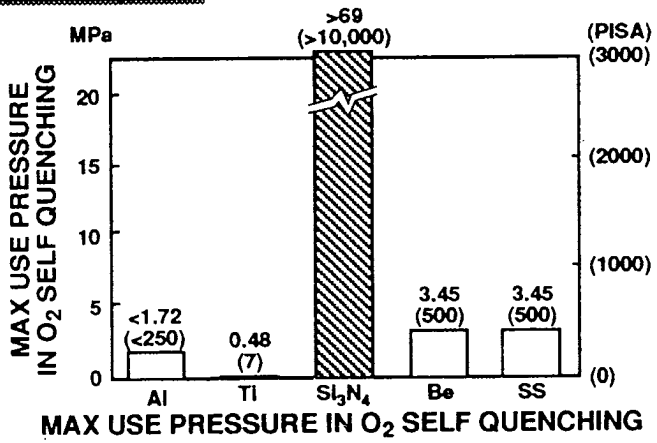
M-920508-04U (C) (2132)





Ti (6AL-4V) SS (CRES 347)
Al (6061) Si_3N_4 (TAPE CAST)
Be





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MATERIALS AND STRUCTURES

– SILICON NITRIDE: STRENGTH AT ELEVATED TEMPERATURES – (U)

M-920508-20U (C) (2129)

MATERIAL	Si_3N_4 (HOT PRESSED) 4 PT BEND	ALUMINUM (6061-T6)	Ti-6A-4V	BERYLLIUM	CRES 347
RT	840*	459	812	552	260
1000 F	840	---	440	360	170
2000 F	830	---	---	---	20
2500 F	550	---	---	---	---

* *UTS/DENSITY, [ksi/lbm per in.^{3}]*

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MATERIALS AND STRUCTURES
– PROPERTIES OF PSG* SILICON NITRIDE – (U)

M-920508-22U (C) (2129)

PROPERTY	VALUE
BULK DENSITY (g/cc)	3.27
HARDNESS (45N)	88
FRACTURE TOUGHNESS ($MPa \cdot m^{0.5}$)	5.7
CHARACTERISTIC STRENGTH (RT, 4 pt) MPa (ksi)	751 (109)
WEIBULL MODULUS	18
ELASTIC MODULES GPa (Mpsi)	303 (44)
POISSON's RATIO	0.24
THERMAL EXPANSION (10^{-6}/K) RT-1273K	3.4
THERMAL CONDUCTIVITY (W/m K)	29.5
THERMAL SHOCK FACTOR (ΔTc)	666K

* *PRESSURELESS SINTERED GLASS*

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MATERIALS AND STRUCTURES
– SILICON NITRIDE HYPER THIN INJECTORS PROGRAM – (U)

M-920508-23U (C) (2129)

OBJECTIVE:	DEMONSTRATE THE FABRICABILITY AND INTEGRITY OF TAPE CAST LAMINATED SILICON NITRIDE INJECTORS
APPROACH:	DESIGN, FABRICATE AND TEST PROTOTYPE INJECTORS
PRIME CONTRACTOR:	AEROJET, SACREMENTO, CALIFORNIA
PRINCIPAL SUBCONTRACTORS:	CERCOM INC., VISTA, CALIFORNIA COORS CERAMICS CO., GOLDEN, COLORADO

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MATERIALS AND STRUCTURES
– INJECTOR DESIGN FOR TECHNOLOGY DEMONSTRATION – (U)

M-920508-24U (C) (2129)

THRUST	2900N	660 lbf
Pc	10.3MPa	1500 psia
FUEL	MMH	
OXIDIZER	NTO	
$\dot{\omega}$ToT	1.2 kg/s	2.65 lb/s
$\dot{\omega}$ FUEL	0.45 kg/s	1.00 lb/sec
$\dot{\omega}$ OX	0.75 kg/s	1.65 lb/sec
ΔP OX	7.5 MPa	1093 psia
ΔP FUEL	3.3 MPa	481 psia
ΔP/Pc FUEL	0.32	0.32
ΔP/Pc OX	0.72	0.72
P OX INLET	17.9 MPa	2593 psia
P FUEL INLET	13.7 MPa	1981 psia
Kw FUEL (SG = .88)		0.0486
Kw OX (ST = 1.44)		0.042
PROOF AND LEAK CAPABILITY	24 MPa	3500 psi

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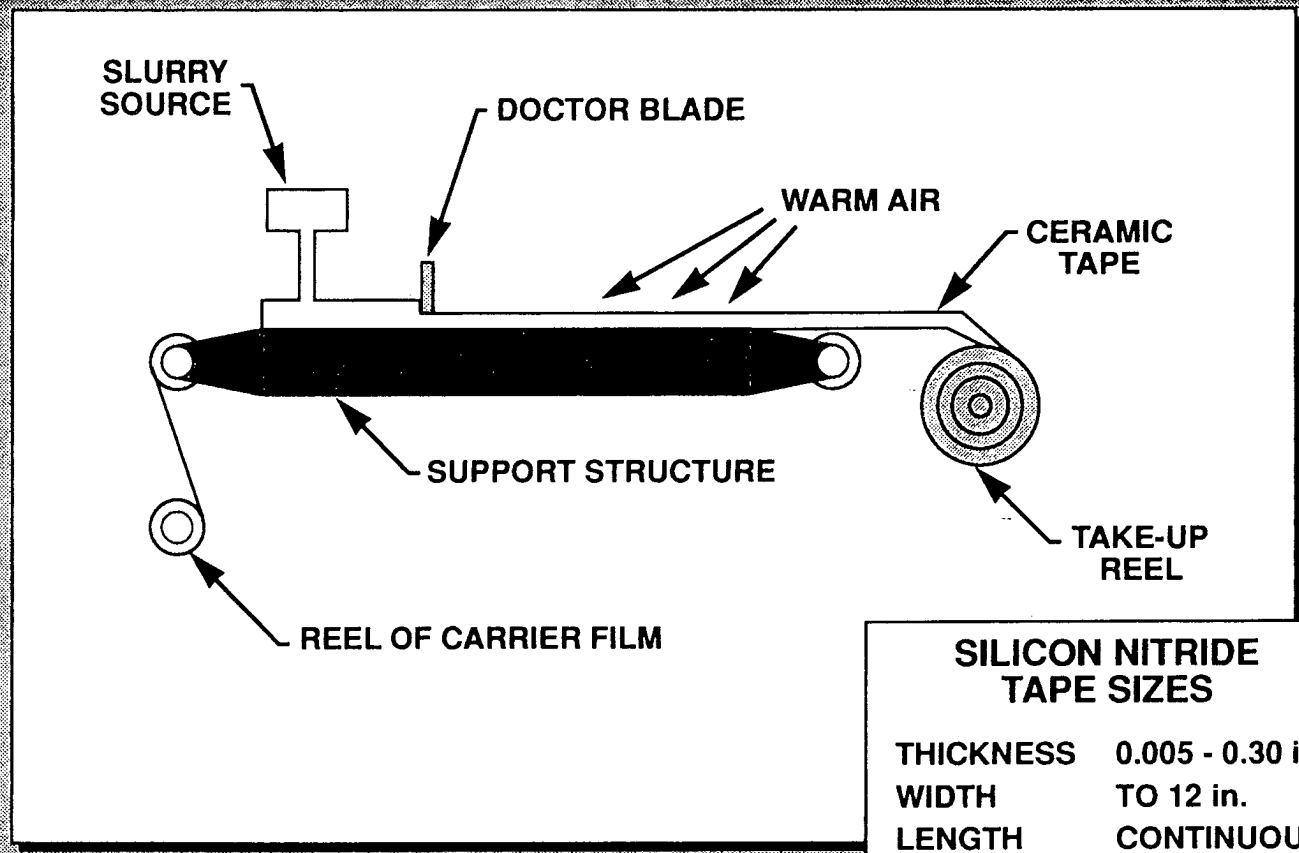
MATERIALS AND STRUCTURES
– DOCTOR BLADE PROCESS FOR PRODUCING CONTINUOUS ROLL SILICON NITRIDE TAPE – (U)

M-920508-25U (C) (2129)



SILICON NITRIDE TAPE SIZES

THICKNESS	0.005 - 0.30 in.
WIDTH	TO 12 in.
LENGTH	CONTINUOUS

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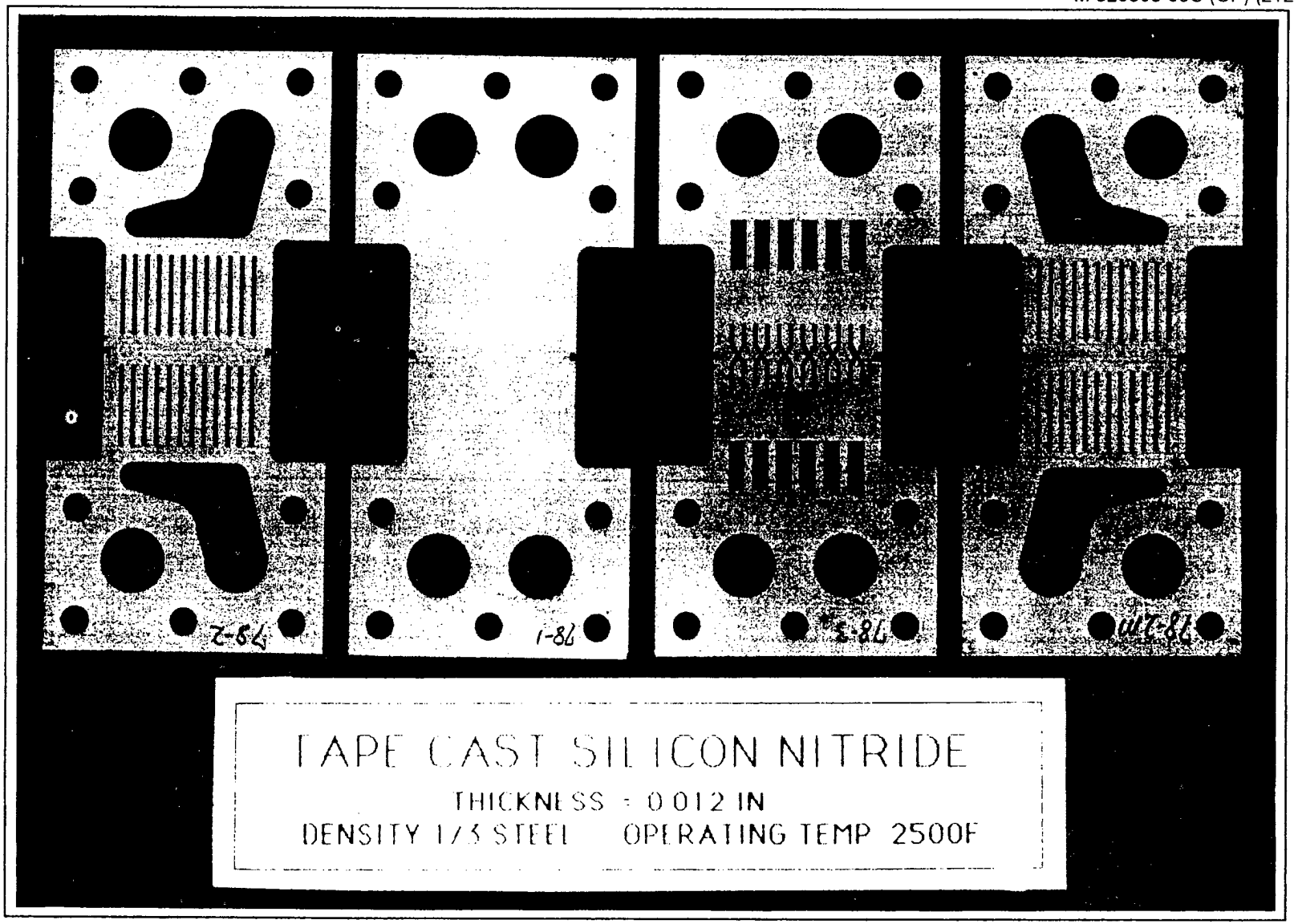
MATERIALS AND STRUCTURES
– TAPE CAST SILICON NITRIDE – (U)

M-920508-09U (CP) (2129)



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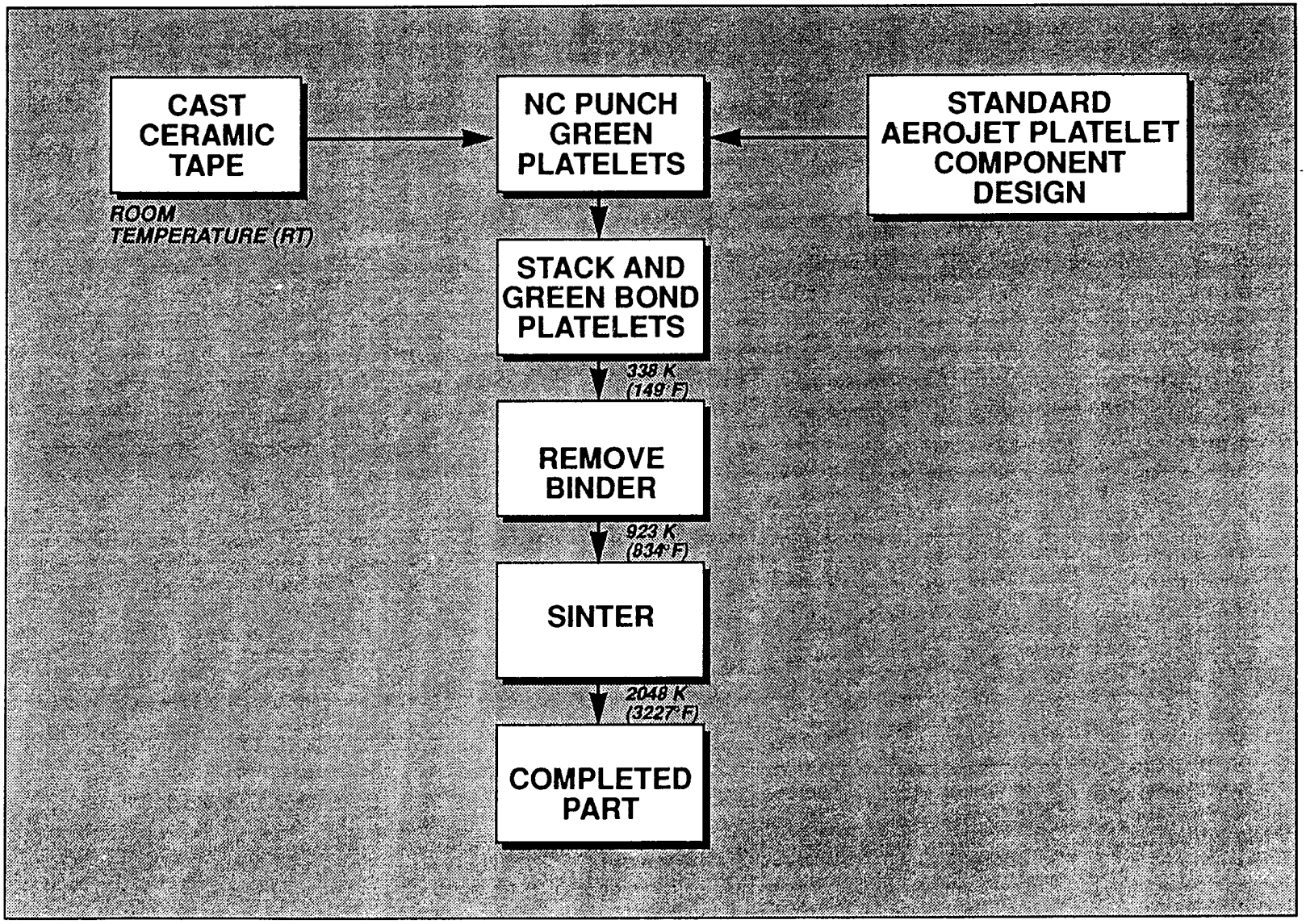
MATERIALS AND STRUCTURES
– SILICON NITRIDE PLATELET FABRICATION PROCESS – (U)

M-920508-26U (C) (2129)



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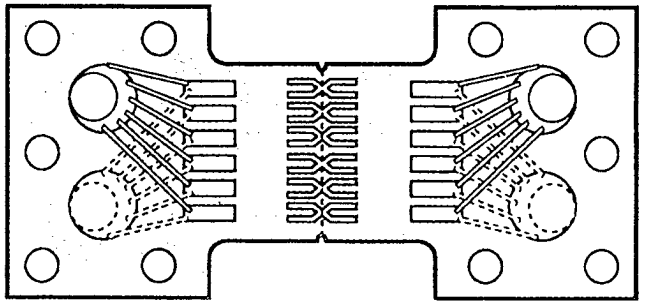
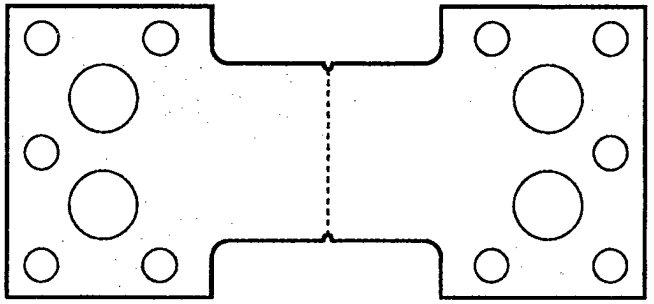
MATERIALS AND STRUCTURES
– SILICON NITRIDE INJECTOR DESIGN – (U)

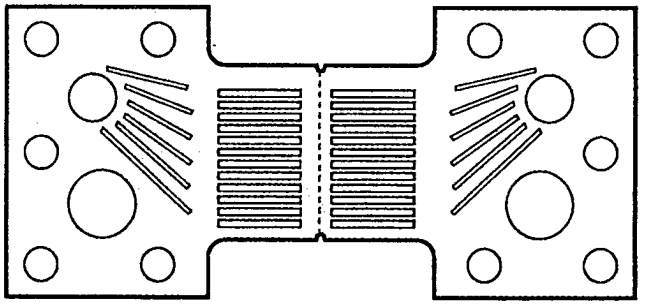
M-910404-04U (C) (1094)



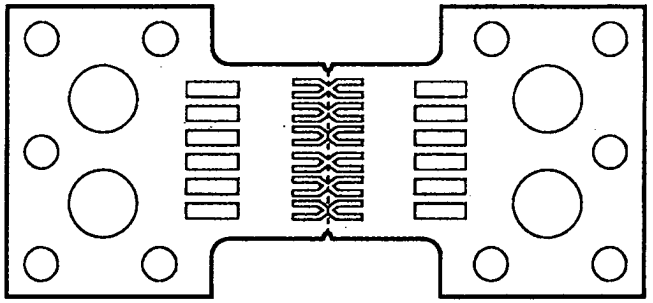
STACKED PLATELET THROUGH VIEW



SEPARATION PLATELET



MANIFOLD PLATELET



METERING PLATELET

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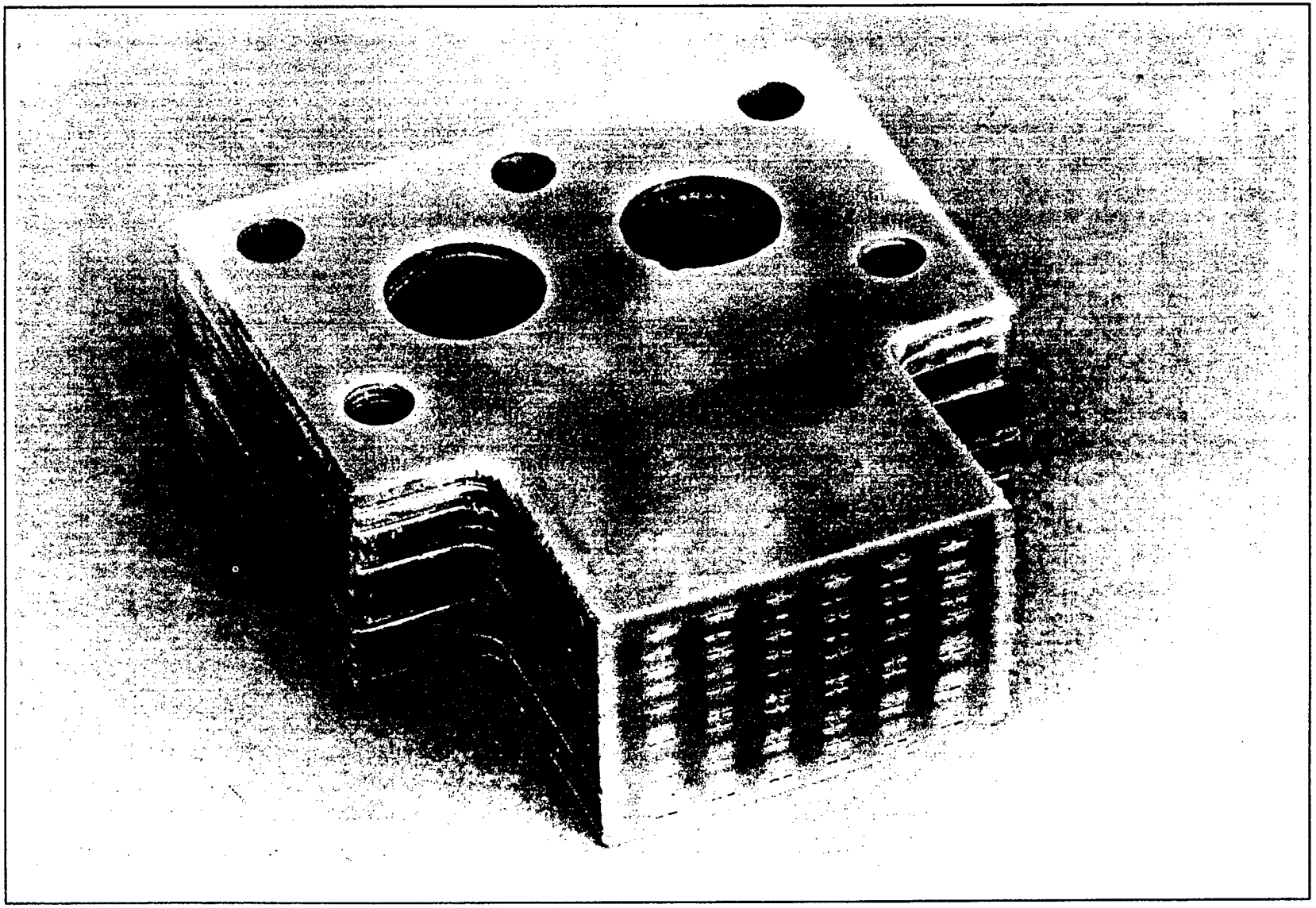
MATERIALS AND STRUCTURES
– DELAMINATED SILICON NITRIDE INJECTOR – (U)

M-920508-10U (CP) (2129)



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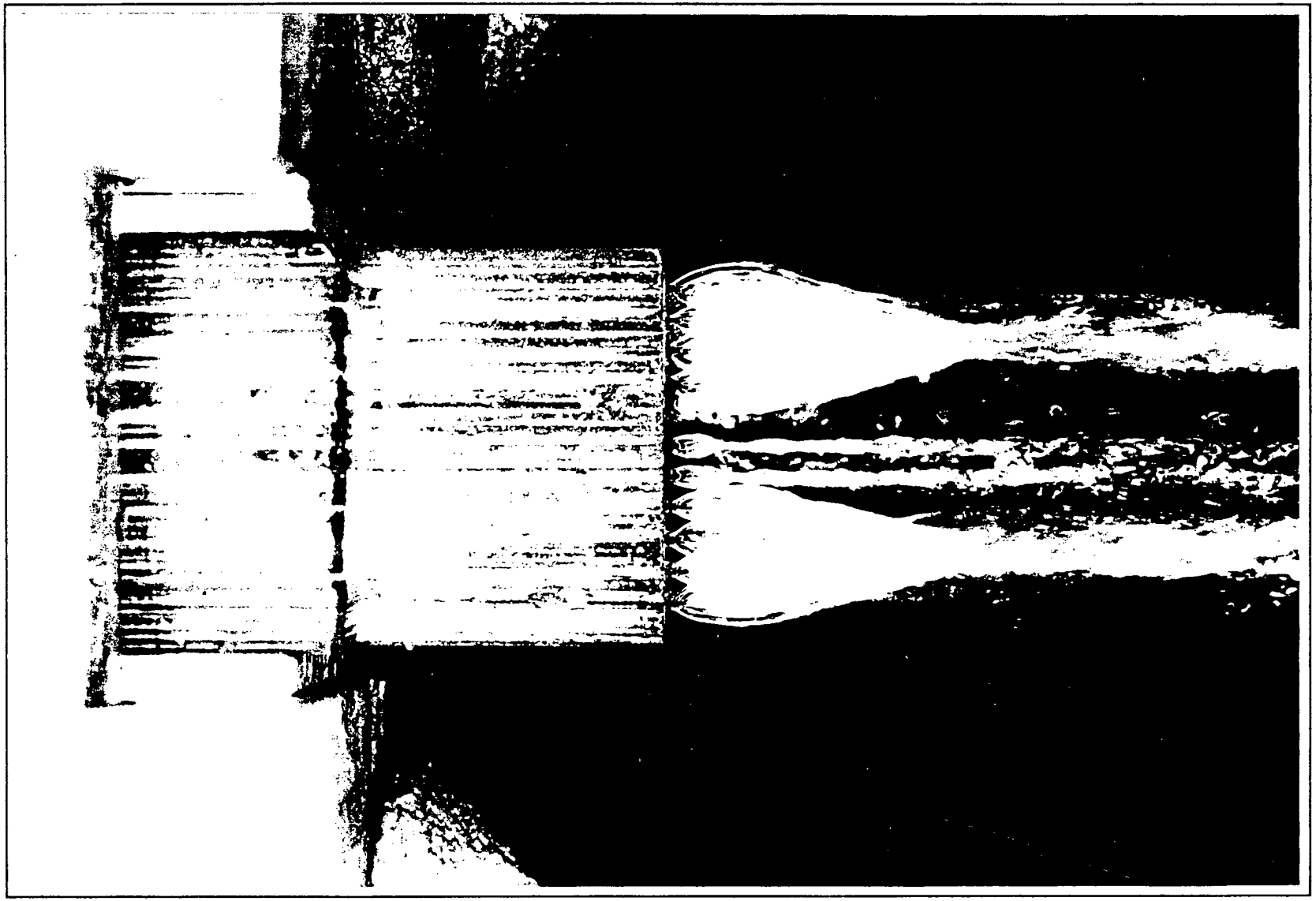
MATERIALS AND STRUCTURES
– COLD FLOW TEST OF SILICON NITRIDE INJECTOR – (U)

M-920508-12U (CP) (2129)



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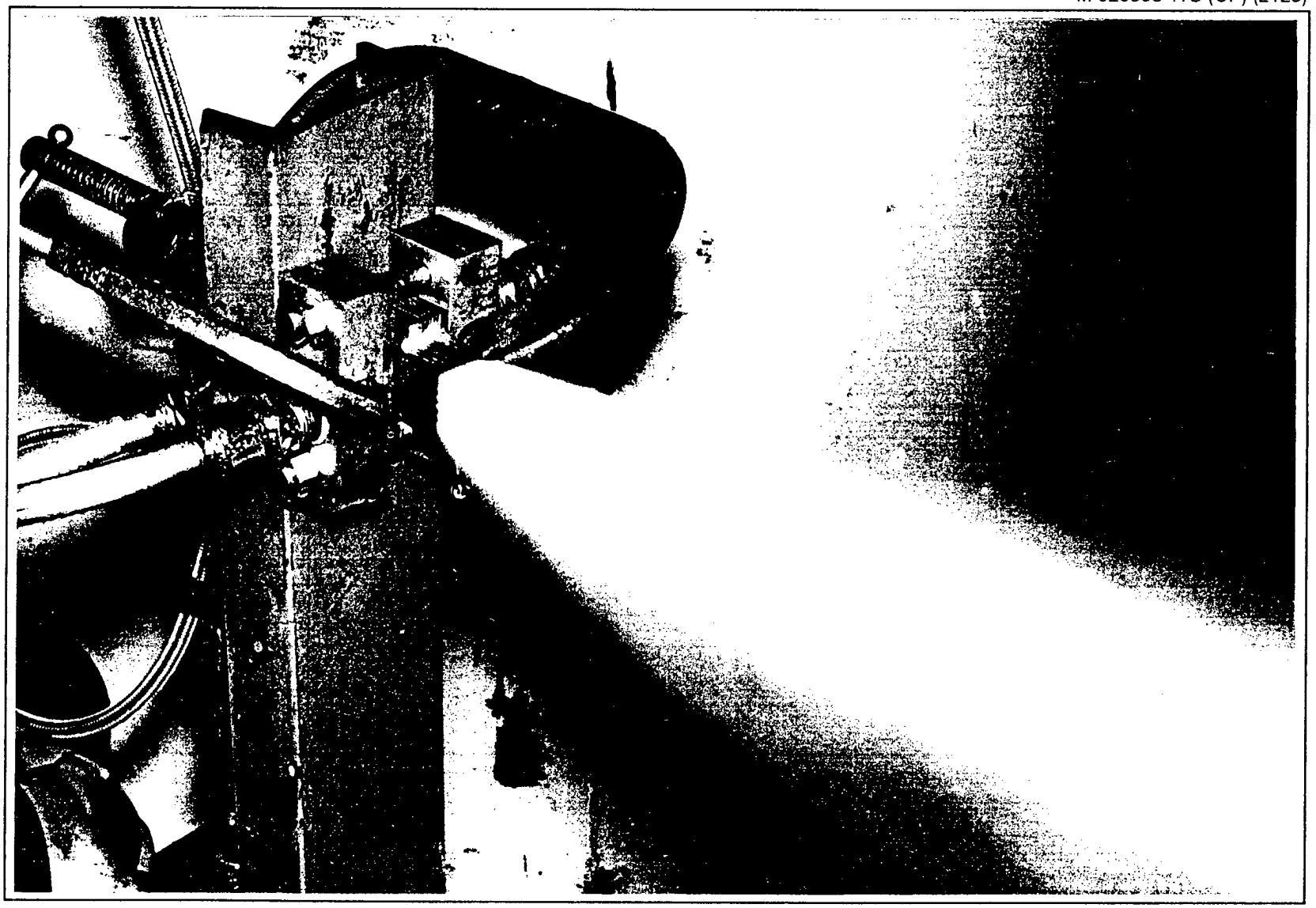
MATERIALS AND STRUCTURES
– COLD FLOW TEST OF SILICON NITRIDE INJECTOR – (U)

M-920508-11U (CP) (2129)



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MATERIALS AND STRUCTURES
– INJECTOR A-1 WATER FLOW TEST RESULTS – (U)

M-920508-27U (C) (2129)

MASS FLOWRATE

	DELTA P (psi)	COLLECTOR TIME (s)	(lbm)	(lbm/s)	Kw	Kw DESIGN GOAL
DUAL CIRCUITS	1000	16.74	23.40	1.40	0.044	
	500	15.93	16.24	1.02	0.045	
	300	15.75	12.69	0.81	0.046	
	300	15.75	12.69	0.81	0.046	
					0.0456	0.091
OX CIRCUIT	1000	15.92	10.89	0.68	0.022	
	1000	15.65	10.89	0.70	0.022	
	500	15.8	7.89	0.50	0.022	
	500	25.96	12.93	0.50	0.022	
	300	26.06	10.11	0.39	0.022	
					0.022	0.042
FUEL CIRCUIT	1000	16.41	12.30	0.75	0.024	
	1000	15.65	8.49	0.54	0.024	
	500	16.05	8.75	0.54	0.024	
	500	26.03	11.02	0.42	0.024	
					0.0242	0.049

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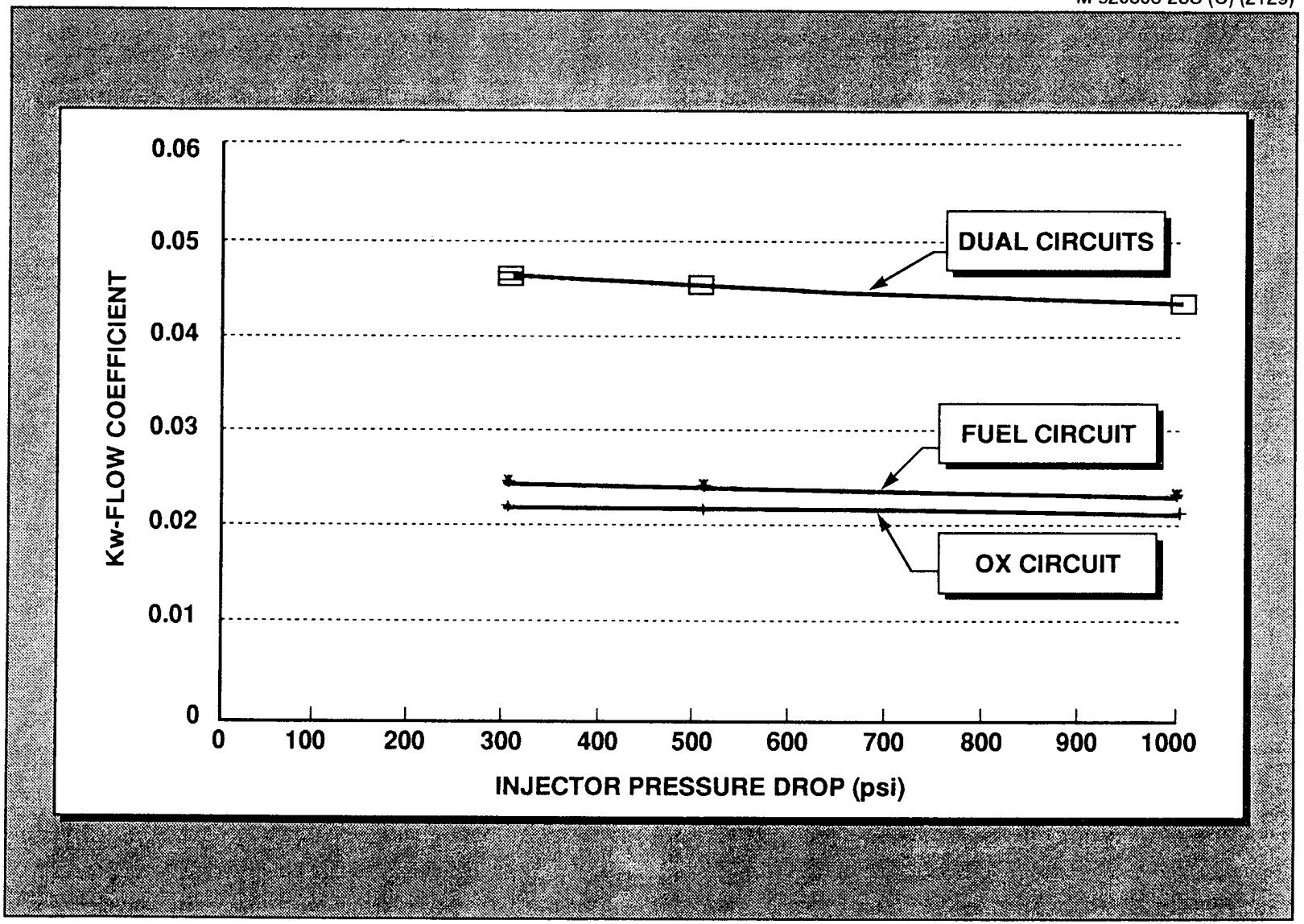
MATERIALS AND STRUCTURES
– INJECTOR A-1 WATER FLOW TEST RESULTS – (U)

M-920508-28U (C) (2129)



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BRIEFER:

DR. WALTER F. JONES

Program Manager
Directorate of Aerospace Sciences

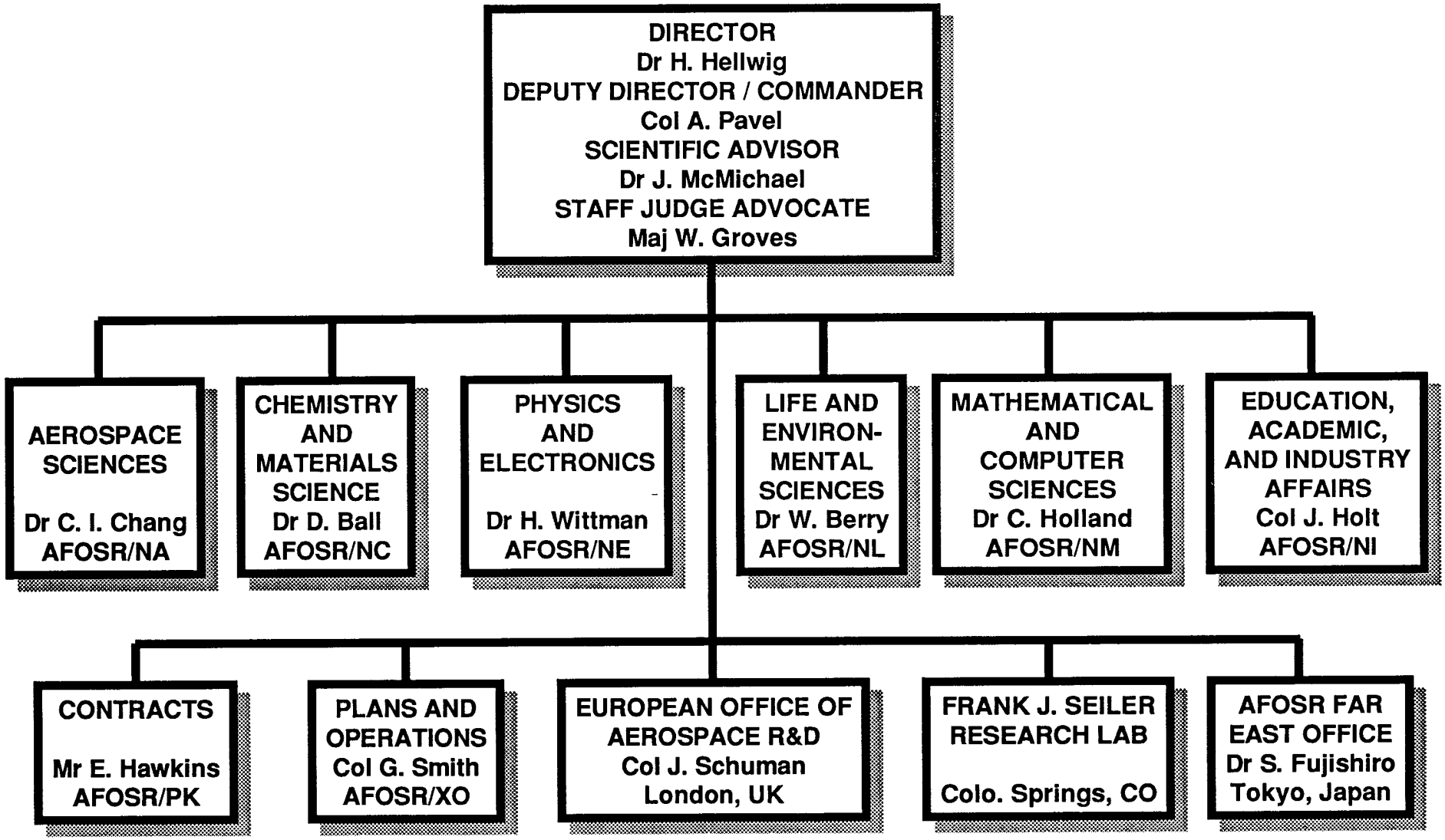
Air Force Office of Scientific Research
Bolling Air Force Base
Washington, DC 20332-6448
Phone: (202) 767-0470
DSN 297-0470

AIR FORCE BASIC RESEARCH PROGRAM IN STRUCTURAL CERAMICS

INTERAGENCY COORDINATING COMMITTEE ON STRUCTURAL CERAMICS
ARLINGTON, VIRGINIA
13 May 1992

Air Force Office of Scientific Research

Bolling Air Force Base, DC 20332-6448



May 1992

Air Force Basic Research

Structural Ceramics Program

Aerospace Sciences | *Chemistry and Materials Science*

Structural Ceramics Basic Research

Project 2302/BS
MECHANICS OF MATERIALS
Dr. Walter F. Jones
(202) 767-0470

- **WL/MLBM** ***MECHANICS OF BRITTLE-MATRIX COMPOSITES*** **Dr. N. J. Pagano**
- **WL/MLLN** ***LIFE PREDICTION*** **OF ENGINE MATERIALS** **Dr. T. Nicholas**

Project 2306/BS
CERAMIC AND NONMETALLIC MATERIALS
Lt. Col. Larry W. Burggraf
(202) 767-4960

- **WL/MLLM** ***HIGH-TEMPERATURE CERAMIC COMPOSITES*** **Dr. R. J. Kerans**
- **PL/RK** ***CARBON MATERIALS RESEARCH*** **Dr. W. Hoffman**

Air Force Basic Research

Structural Ceramics Program

PROGRAM OBJECTIVE

Establish the fundamental understanding required for the design, processing, and thermomechanical performance prediction of current and evolving structural ceramic material systems

CERAMICS TOUGHENING: Control microstructure to create tough ceramic composite materials.

MICROMECHANICAL MODELING: Relate ceramic microstructure to mechanical properties and guide the development of new material systems.

HIGH-TEMPERATURE CERAMICS: Understand structural stability of ceramics and ceramic interfaces at elevated temperatures.

INTERFACES AND COATINGS: Tailor interface structures and coatings for thermomechanical stability.

COMPOSITE PROCESSING: Develop processing/microstructure relations.

Air Force Basic Research

Structural Ceramics Program

FY 1992 INITIATIVE - *STRUCTURAL CERAMICS*

SCIENTIFIC ISSUES AND RESEARCH NEEDS

- IDENTIFY AND MODEL THE ROLE OF TEMPERATURE ON MECHANISMS INFLUENCING INTERFACIAL BONDING
  - Diffusion and Oxidation
  - Chemical Reactions
  - CTE Mismatch
  - Coefficient of Friction
- NEED TO IDENTIFY INTERPHASES THAT ARE
  - Stable at High Temperatures
  - Bond Poorly to Either Reinforcement or Matrix
- IDENTIFY DESIRABLE THERMOMECHANICAL AND CHEMICAL PROPERTIES FOR CANDIDATE COATINGS
- DETERMINE APPROPRIATE "FLAVOR" OF MECHANICS FOR TREATING MATERIAL BEHAVIOR (Continuum, LEFM, Damage Mechanics, etc)

Air Force Basic Research

Structural Ceramics Program

CERAMICS TOUGHENING

Carnegie-Mellon Univ (Prof Michael Readey) - ***Microstructure and Reliability of Ceramics***

Univ of Illinois (Prof Waltraud Kriven) - ***Displacive Transformations in Ceramics***

Lehigh Univ (Prof Helen Chan) - ***Microstructural Design for Tough Ceramics***

Lehigh Univ (Prof Helen Chan) - ***Multiphase Ceramics for Mechanical and Structural Reliability at Low and Elevated Temperature***

National Inst of Standards and Technology (Dr Brian Lawn) - ***Strength, Fatigue, and Microstructure of Ceramics***

Rockwell Int'l Science Center (Dr David Marshall) - ***Transformation Toughening of Ceramics***

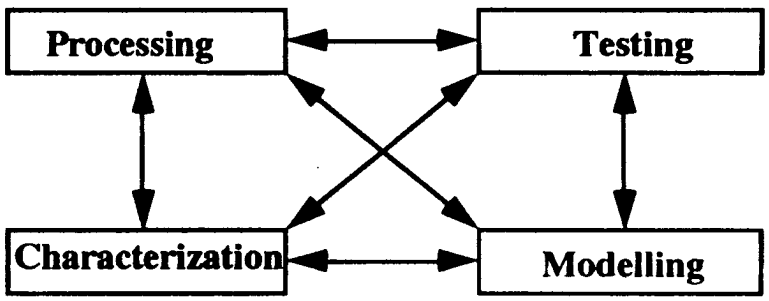
MICROSTRUCTURE & MECHANICAL PROPERTIES OF CERAMICS

Helen Chan and Brian Lawn
Lehigh University

OBJECTIVES

- **Understand role of microstructure in mechanical properties**
- **Design & fabricate ceramics with optimum mechanical properties**
- **Develop tests for characterization of strength and toughness**

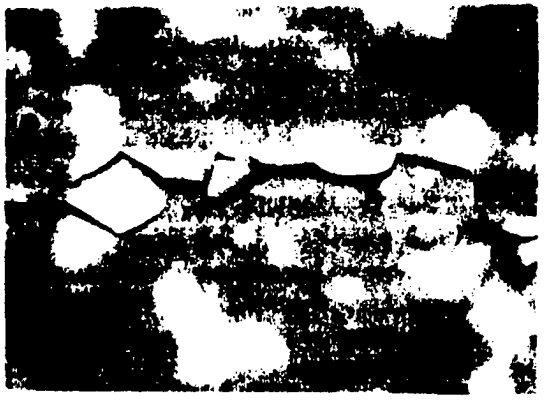
APPROACH



ACCOMPLISHMENTS

- **Developed novel particulate-reinforced composite: alumina/aluminum titanate**
- **Designed innovative tests for evaluation of short-crack toughness curves**
- **Investigated microstructure-strength relations in two-phase ceramics**

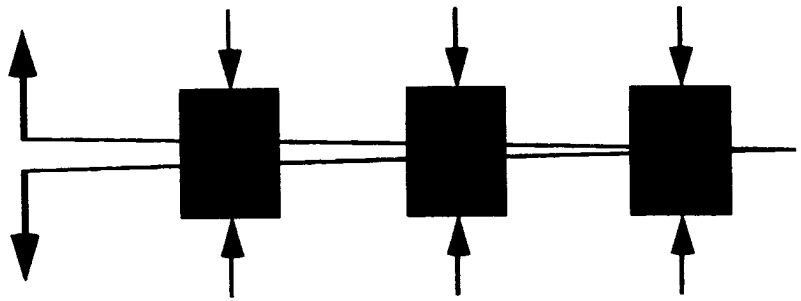
GRAIN BRIDGING



MICROSTRUCTURAL TAILORING

- **MODELLING**

Toughening by grain-interlock crack bridging

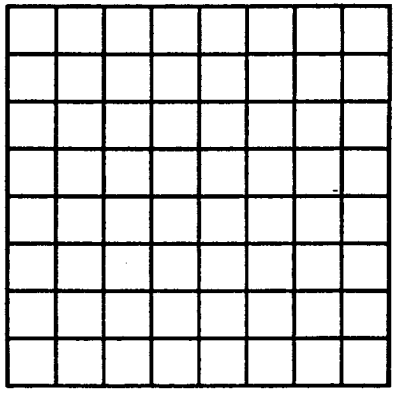


Optimize toughness by tailoring microstructure and introducing internal residual stressses

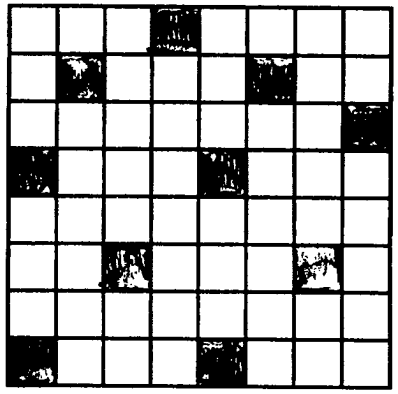
- **PROCESSING**

  - **Microstructure tailoring by heat-treatment and powder processing (Incorporate large grains and second-phase particles)**
  - **Residual stress tailoring by interface chemistry (Add Al_2TiO_5 to Al_2O_3)**

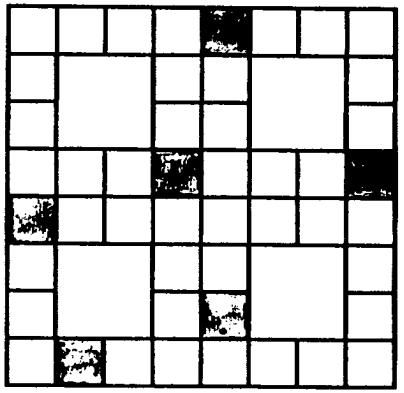
MICROSTRUCTURAL DESIGN



Single-phase unimodal

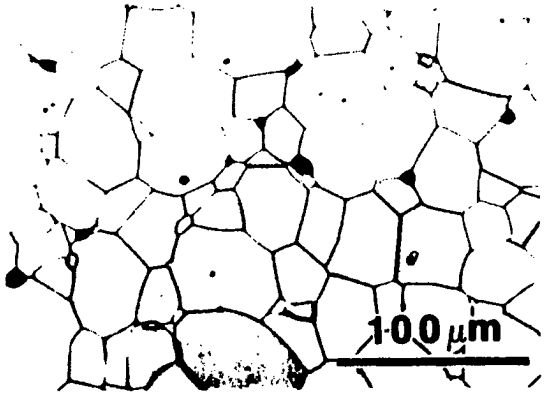


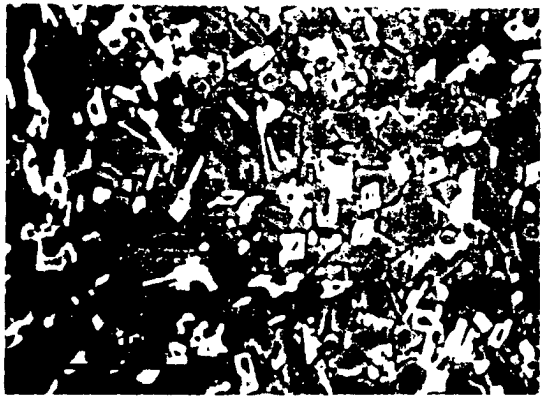
Duplex-unimodal

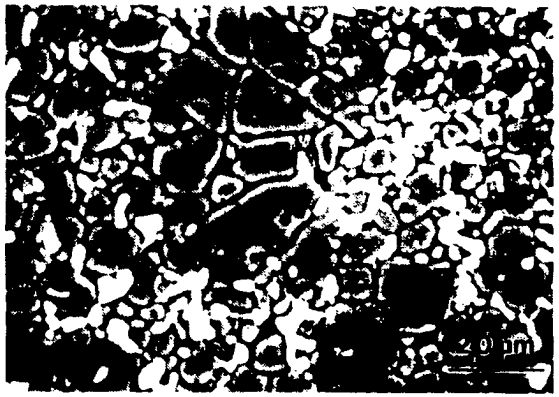


Duplex-bimodal

Alumina **Aluminum titanate**

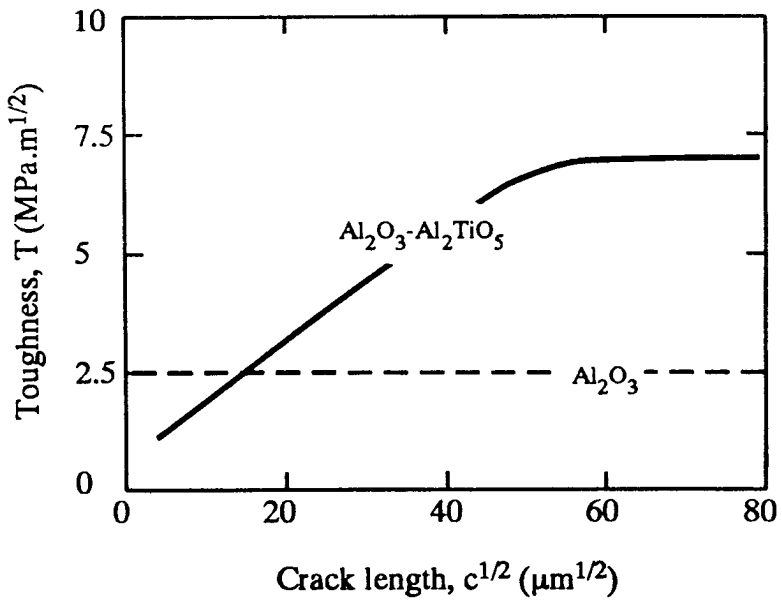






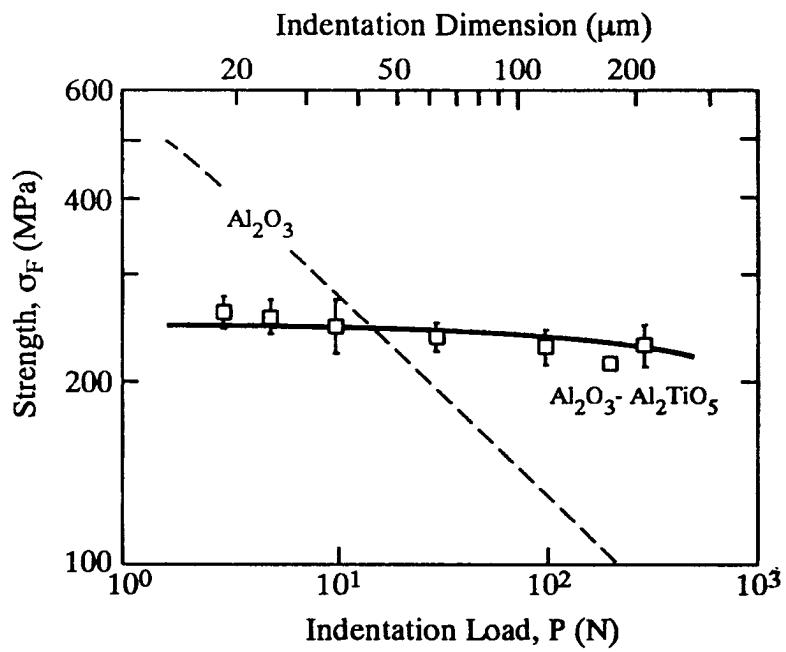
MECHANICAL PROPERTIES

- **Short-crack T-curves from *in situ* microscopy**



Enhanced toughness

- **Indentation-strength in bending**



Flaw tolerance

Air Force Basic Research

Structural Ceramics Program

MICROMECHANICAL MODELING

Cambridge Univ, UK (Prof Peter Beaumont) - *Development and Characterization of Tough Ceramic-Matrix Composites*

Carnegie-Mellon Univ (Prof Paul Steif) - *Flaw Sensitivity in CMC*

Massachusetts Inst of Technology (Prof Ali Argon) - *Mechanical Properties of Porous, High-Temperature Structural Materials*

Massachusetts Inst of Technology (Prof Michael Cleary) - *3-D Analysis and Verification of Fracture Growth Mechanisms in Fiber-Reinforced CMC*

City College of New York (Prof Feridun Delale) - *Micromechanical Prediction of Tensile Damage for CMC at High Temperature*

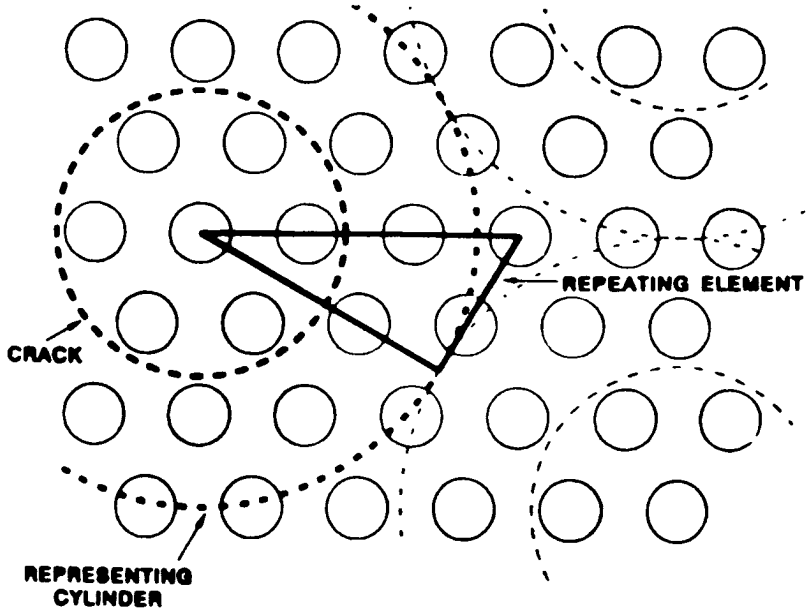
Northwestern Univ (Prof Isaac Daniel) - *Characterization of Deformation and Damage in Brittle-Matrix Composite Materials*

Air Force Basic Research

Aerospace Sciences

Damage and Failure Mechanisms in Certain Ceramic-Matrix Composites Are Influenced by Imperfections in the Matrix

Group Pullout in C/SiC CMC



Models for Crack Stress Analysis

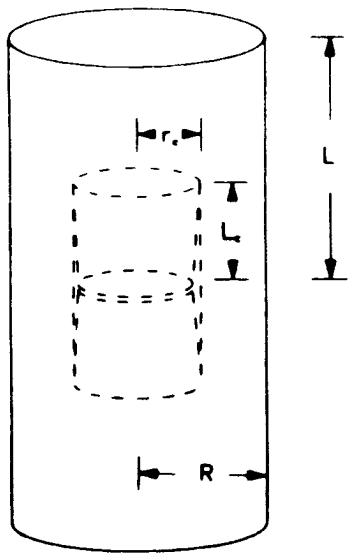
Air Force Basic Research

Aerospace Sciences

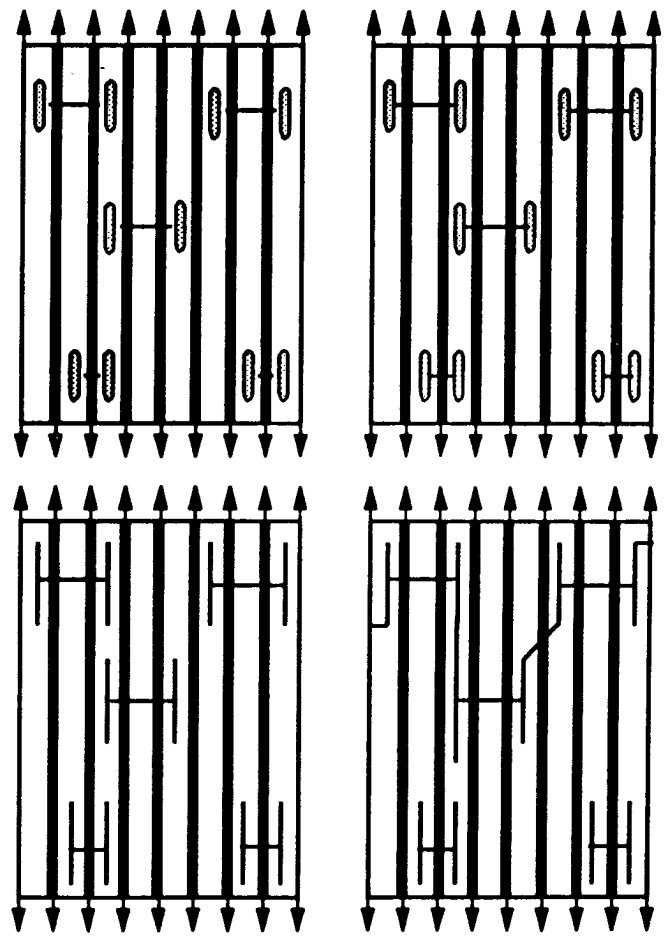
Damage and Failure Mechanisms in Ceramic-Matrix Composites Which Exhibit Dispersed Local Failures Have Been Modeled

CMC Failure Scenario

1. Matrix cracks grow and are arrested by flaws
2. Fibers fracture; Longitudinal cracks grow in stable manner
3. "H cracks" merge to form catastrophic flaws



Representative Volume Element



C. F. Yen/Materials Sciences Corp

Air Force Basic Research

Structural Ceramics Program

HIGH-TEMPERATURE CERAMICS

Univ of California, Santa Barbara (Prof Fred Lange) - *High-Temperature Stability of Binary Microstructures Derived from LIquid Precursors*

Univ of Michigan (Prof I-Wei Chen) - *High-Temperature Fatigue of Structural Ceramics*

Univ of Michigan (Prof John Holmes) - *Mechanics of Elevated-Temperature Fatigue Damage in Fiber-Reinforced Ceramics*

Nat'l Inst of Standards and Technology (Dr John Hastie) - *Thermodynamic and Kinetic Stability of Refractory Materials at High Temperature*

Southwest Research Inst (Dr Richard Page) - *High-Temperature Failure Mechanisms in Ceramics*

Air Force Basic Research

Structural Ceramics Program

INTERFACES AND COATINGS

Brown Univ (Prof Brian Sheldon) - *Nucleation of Crystalline Films from Vapor Phase Reactants*

Oak Ridge Nat'l Lab (Dr Ted Besmann) - *Nucleation and Growth in Chemical Vapor Deposition*

Oak Ridge Nat'l Lab (Dr Rodney McKee) - *Molecular Beam Epitaxy Grown Oxides*

United Tech Research Center (Dr John Brennan) - *Processing and Properties of Coated HPZ Fiber/Glass Ceramic-Matrix Composites*

United Tech Research Center (Dr John Brennan) - *Interfacial Studies of Coated Fiber-Reinforced Glass Ceramic-Matrix Composites*

Air Force Basic Research

Structural Ceramics Program

COMPOSITE PROCESSING

Univ of Missouri, Rolla (Prof Mohamed Rahaman) - ***Pressureless Densification of Ceramic-Matrix Composites***

Univ of New Mexico (Prof Martin Weiser) - ***Pressureless Sintering of Ceramic Composites***

Southwest Research Inst (Dr Stuart Schwab) - ***Microstructure Design Through Preceramic Polymer Chemistry***

United Tech Research Center (Dr Jim Strife) - ***Novel Precursor Approaches for Ceramic-Matrix Composites Derived by Polymer Pyrolysis***

Air Force Basic Research

Structural Ceramics Program

DARPA-Funded Ceramic Programs

Ceramatec, Inc (Dr Raymond Cutler) - *New Mechanisms for Toughening of Ceramics*

General Atomics (Drs Streckert and Gulden) - *Ceramic Fiber Coating Development and Demonstration*

Pratt and Whitney Aircraft (Dr Bob Emiliani) - *Fiber Coatings by Sputtering for High-Temperature Composites*

Air Force Basic Research

Structural Ceramics Program

UNIVERSITY RESEARCH INITIATIVES - FY 1990

Drexel Univ (Prof Albert Wang et al) - *Comprehensive Study of Matrix Fracture Mechanisms in Fiber-Reinforced Ceramic-Matrix Composites*

Univ of Illinois (Prof Waltraud Kriven et al) - *Transformation Toughening of Ceramic Composites*

Northwestern Univ (Prof Leon Keer et al) - *High-Temperature Heterogeneous Materials*

Air Force Basic Research

Aerospace Sciences

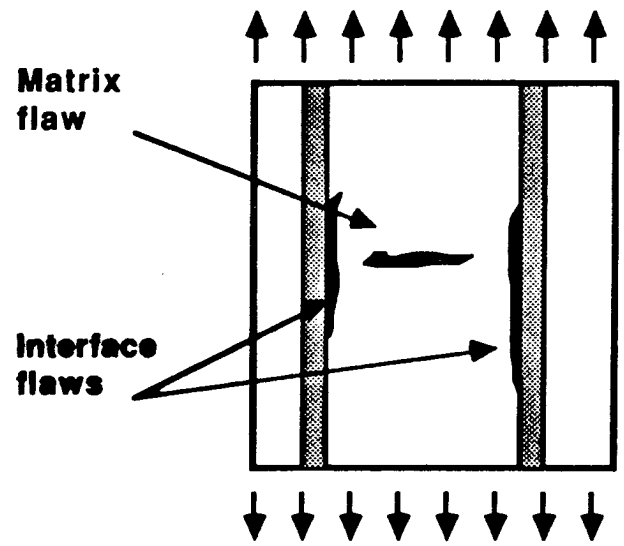
Understanding of Initiation and Propagation of Matrix Fracture in Ceramic-Matrix Composites Requires Combined Analytical and Experimental Efforts

Physical Basis of Model

- Multiphased Griffith Solids: A Composite Micro-Cell Which Distinguishes Fiber, Matrix, Interface, and Inherent Flaws
- Coupled Analysis of the Micro-Cell for Flaw-Crack Initiation and Evolution under Thermomechanical Loading

Experimental Variables

- Nicalon, SCS-6, and Carbon Fibers
- Borosilicate, Aluminosilicate, Zircon, and SiC Matrices
- Room and Elevated Temperatures

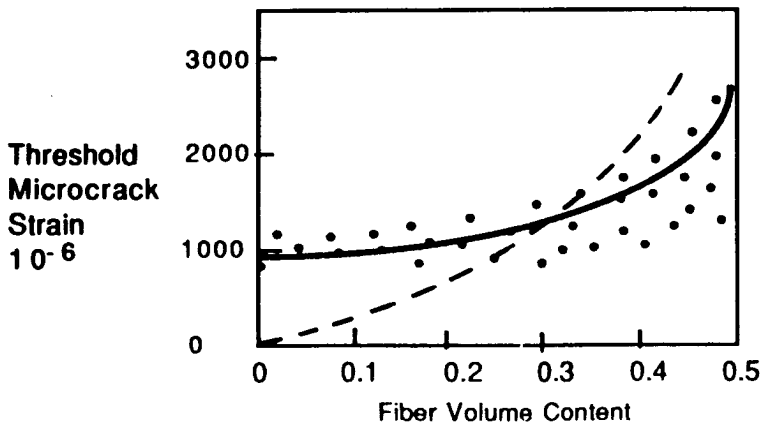


Idealized local flaws and interaction configuration

Air Force Basic Research

Aerospace Sciences

Local Flaw Model Predicts the Behavior of Microcracking in Fiber-Reinforced Ceramic-Matrix Composite Materials



Experiments ●

Existing Model - - - -

Crack Interaction Model ———

Desirable Properties

- Tougher Matrix
- Stiffer Fiber; $E_f/E_m > 2$
- Thermal Compatibility
- Smaller Fibers
- High Fiber Volume Fraction
- Small Fiber Spacing
- Uniform Fiber Distribution

Research: Validate Model Using CMC's Capable of Sustaining Temperature > 1600°C

A. S. D. Wang/Drexel

Air Force Basic Research

Structural Ceramics Program

UNIVERSITY RESEARCH INITIATIVE - FY 1993

HIGH-TEMPERATURE AEROSPACE STRUCTURAL MATERIALS

- Extend the use temperature of advanced material systems, including polymer-matrix composites, metal-matrix composites, CERAMIC-MATRIX COMPOSITES, and carbon-carbon
- Develop new families of aerospace structural material systems
- Study relationship of atomic phenomena to bulk mechanical properties; microstructure/property relationships
- Develop understanding of thermomechanical behavior through appropriate experiments and modeling

Air Force Basic Research

Structural Ceramics Program

FY 1991-1992 FUNDING SUMMARY

	FY 1991 (K$)		FY 1992 (K$)	
	AFOSR	DARPA	AFOSR	DARPA
CERAMICS TOUGHENING	1319	187	1510	-
MICROMECHANICAL MODELING	1406	-	973	-
HIGH-TEMPERATURE CERAMICS	657	-	481	-
INTERFACES AND COATINGS	539	1274	765	787
COMPOSITE PROCESSING	360	-	287	-
TOTAL FUNDING	4281	1461	4016	787

WRIGHT LABORATORY MATERIALS DIRECTORATE

CERAMIC COMPOSITE PROGRAMS

13 MAY 1992

Allan P. Katz

WL/MLLM

CERAMIC COMPOSITES OBJECTIVE

- CERAMIC COMPOSITE DEVELOPMENT FOR GAS TURBINE ENGINE APPLICATIONS
  - CMC'S ARE CRITICAL TO MEETING IHPTET PHASE II AND PHASE III GOALS
  - CMC'S ARE DESIRED FOR NEAR-TERM SYSTEMS USE (e.g., F100/110 UPGRADES, F119)
- ALTERNATE APPLICATION OUTLETS TO BE CONSIDERED FOR FUTURE ACTIVITIES

CERAMIC COMPOSITES STRATEGY

- MAINTAIN A BALANCED PROGRAM WHICH ADDRESSES THE CRITICAL PACING ISSUES FOR CMC DEVELOPMENT AND APPLICATION
  - FIBERS
  - INTERFACES
  - PROCESSING
  - LIFE PREDICTION AND MECHANICS
  - MATERIAL TO COMPONENT TRANSITION
- PROMOTE THE APPLICATION OF CERAMIC COMPOSITES IN AF SYSTEMS
  - ML 6.2/6.3 PROGRAMS
  - LEVERAGED RESOURCES WITH OTHER GOVERNMENT ORGANIZATIONS AND WITH INDUSTRY
  - TECHNICAL LEADERSHIP OF GOVERNMENT-WIDE CMC EFFORTS

Ceramic Composites Key Activities

Wright Laboratory | Materials Directorate

- RESEARCH IN-HOUSE ON CRITICAL GENERIC ISSUES
- PROCESSING SCIENCE
  - CVI PROCESS MODELING
  - CVI MANUFACTURING SCIENCE (PLANNED FY93 7.8 NEW START)
  - CMC PROCESS DEVELOPMENT (PLANNED FY94 6.2 NEW START)
- TECHNOLOGY FOR MATERIALS/COMPONENT INTEGRATION
  - CARBON AND SiC FIBER REINFORCED SILICON CARBIDE NOZZLE FLAPS FOR F-100 ENGINE UPGRADE (TO 2500 ^{0}F)
  - CARBON FIBER REINFORCED SILICON CARBIDE FOR LIMITED LIFE ROTORS (TO 2500^0 F)
  - DURABILITY ENHANCEMENT FOR GLASS-CERAMIC MATRIX SYSTEMS (TO 2200^0 F)
  - JOINT ML/PO PROGRAMS UNDER IHPTET PRDA III (TWO PLANNED FY92 6.2 NEW START)

Ceramic Composites Key Activities (Cont.)

Wright Laboratory | Materials Directorate

- CMC EVALUATION FOR LIMITED LIFE COMBUSTORS (TO 2500^0 F, DARPA FUNDED)
- HPZ FIBER REINFORCED CAS, MAS, BMAS, AND SiCN FOR ENGINE EXHAUST COMPONENTS
- ADVANCED EXHAUST SYSTEM COMPONENT DEVELOPMENT (PLANNED FY93 6.3 NEW START)

• FIBER DEVELOPMENT FOR >2500^0 F THROUGH FEASIBILITY STUDIES AND GOVERNMENT/ENGINE INDUSTRY CONSORTIUM

- ASSESSMENTS OF SINGLE AND POLYCRYSTAL YAG, OXIDE EUTECTICS INCLUDING AL_2O_3- YAG, SINGLE CRYSTAL Mg AL_2O_4, AND CVD SiC
- GOVERNMENT/ENGINE INDUSTRY CONSORTIUM TO DEVELOP MULTIFILAMENT AND MONOFILAMENT FIBERS FOR CMC AND MMC (PLANNED FY92 6.2 START)

CERAMIC MATRIX COMPOSITES IN-HOUSE RESEARCH PROGRAM

OBJECTIVE

- UNDERSTAND THE DESIGN, PROCESSING, AND BEHAVIOR OF HIGH TEMPERATURE COMPOSITES

APPROACH

- BASIC MECHANICS OF BRITTLE MATRIX COMPOSITES
- CONTROLLED STUDIES OF THE ROLE OF THE FIBER/MATRIX INTERFACE
- INVESTIGATE MECHANISMS THAT LIMIT USE TEMPERATURE OF COMPOSITES
- CONTROLLED PROCESSING FOR DESIGNED MICROSTRUCTURES

CURRENT ACTIVITIES

- MECHANICS OF BRITTLE MATRIX COMPOSITES
  - MECHANICS
  - FAILURE MORPHOLOGY
- CONTROL OF THE FIBER/MATRIX INTERFACE
  - COATINGS
    - SOL-GEL CHEMISTRY
    - CHEMICAL VAPOR DEPOSITION
- HIGH TEMPERATURE COMPOSITES
  - ALUMINA-YAG COMPOSITES
  - ENVIRONMENTAL DEGRADATION OF INTERFACES
- PROCESSING
  - COATING OF FIBER PREFORMS
  - PRESSURE INFILTRATION
  - DENSIFICATION WITH CONSTRAINTS
    - CONTROLLED POROSITY
- FIBER RESEARCH
  - CREEP MECHANISM
  - CREEP RESISTANT MATERIALS
  - OXIDE SINGLE CRYSTALS (WITH NASA LEWIS)

WRIGHT LABORATORY MATERIALS DIRECTORATE

CURRENT STRUCTURAL CERAMIC COMPOSITE PROGRAMS

5 MAY 1992

In-House Research/On-Site Contract Research - Generic research on critical issues for understanding and developing ceramic composites. Emphasis on fiber-matrix interface behavior and control. System specific studies of high temperature oxides, e.g., Yttrium-Aluminum-Garnet (YAG) and YAG-Alumina. Cooperative efforts underway with NASA-Lewis.

Processing Science - Analytical process modeling of isothermal and forced flow CVI. Models developed in conjunction with Georgia Tech are being experimentally validated. Effort to conclude in FY92 with scaleup demonstration of forced flow approach and infiltration of prototypical fiber preform.

Rotor Development - Carbon fiber reinforced SiC by CVI. Subcontract to Williams International. Builds on prior rotor work of Williams with SEP for limited life engines. To examine effects of key variables on material behavior. Rotors to be made and tested.

Nozzle Development - Fiber reinforced SiC by CVI. Subcontract to Pratt & Whitney. To examine effects of key variables, including fiber type, on material behavior. Commitment from F100 Engine SPO to test prototype divergent flap in an engine.

Thermally Durable CMC - Examines two approaches to avoid oxidative embrittlement of glass-ceramic matrix composites. (1) Borosilicate glass and SiC-whisker doped Magnesium Aluminosilicate matrix/NICALON. Glass inhibits crack propagation and whiskers toughen matrix. Preliminary results promising. (2) Sheet silicate interface treatments for Calcium Aluminosilicate (CAS)/NICALON. Weak, oxidatively stable interfaces. Work on sheet silicates to start late FY92 so as to build on work ongoing in a DARPA/ONR program. Subcontract to Pratt & Whitney to help focus material testing toward application needs. Emphasizes material optimization in iterative fashion.

Combustor Evaluation - DARPA funded program. Follows similar effort just being completed. To look at state-of-the-art CMC in combustor configuration for limited life engines. Data generation and full up engine tests.

Adv CMC For C3D - Augmentation of Wright Lab Flight Dynamics Directorate's Ceramic Composite Component Demonstration (C3D) program to develop CMC nozzle sidewall. Subcontracts to Dow Corning and Corning to evaluate HPZ-fiber-reinforced SiCN (polymer derived) and CAS, respectively.

LHPG YAG Fiber - Feasibility of the laser heated pedestal growth (LHPG) method to make single crystal YAG and zircon fibers.

Oxide Fiber Feasibility - Assessment of potential for oxide eutectic, YAG, and magnesia alumina spinel single crystal fibers. Emphasis on generating creep data at very high temperature. Builds on prior work to identify candidate constituent materials for ultrahigh temperature CMC.

WRIGHT LABORATORY MATERIALS DIRECTORATE

CURRENT STRUCTURAL CMC PROGRAMS (CONT.)

Creep Resistant YAG Fiber - Feasibility of polymer derived fiber. To examine producing aligned grains for creep resistance by aligning dopants during fiber making process.

Polycrystal YAG Fiber - Feasibility of sol-gel derived fiber. Will incorporate dopants for grain growth control and stability.

EFG YAG Fiber - Feasibility of single crystal fiber by edge defined film fed growth (EFG).

SiC CVD Fiber - Feasibility of SiC fibers by CVD onto multifilament carbon fiber tow.

YAG-Alumina Fiber - Feasibility of EFG eutectic fiber. Builds on successful SBIR Phase I program.

HPZ Fiber CMC - Evaluation of HPZ-fiber-reinforced glass ceramics. To incorporate appropriate interface treatments and composite processing.

WRIGHT LABORATORY MATERIALS DIRECTORATE

CURRENT STRUCTURAL CERAMIC COMPOSITE PROGRAMS

13 MAY 1992

TITLE	ORGANIZATION	P.I.	FUNDS (PE*/$TOTAL)		START	DURATION
In-House Research	WL/MLLM	R. Kerans	6.1A	$430K/YR	NOV 83	Ongoing
On-Site Contract Research	UES	T. Mah	6.1A	$1280K	APR 88	48 Mo
"	"	"	6.2	$600K	"	"
"	Univ. Dayton	N. Ashbaugh	6.2	$600K	APR 91	48 Mo
Processing Science	Oak Ridge NL	T. Besmann	6.2	$1750K	OCT 88	48 Mo
Rotor Development	DuPont	R. Klacka	6.2	$252K	AUG 90	24 Mo
"	"	"	6.2P	$973K	"	"
Nozzle Development	DuPont	J. Halada	6.2	$173K	SEP 90	30 Mo
"	"	"	6.2P	$795K	"	"
Thermally Durable CMC	Corning	D. Larsen	6.2	$1360K	AUG 90	48 Mo
"	"	"	6.2P	$175K	"	"
Combustor Evaluation	Williams Intl.	W. Fohey	6.2D	$1623K	SEP 91	36 Mo
Adv CMC For C3D	Pratt & Whitney	C. Alloca	6.3	$200K	SEP 91	12 Mo
"	"	"	6.3P	$300K	"	"
LHPG YAG Fiber	Lasergenics	R. Schlecht	6.5	$49K	MAY 91	6 Mo
Oxide Fiber Feasibility	Pacific NW Lab	E. Courtright	6.2	$385K	JUL 91	15 Mo
Creep Res YAG Fiber	Univ. Michigan	J. Halloran	6.2	$417K	SEP 91	27 Mo
Polycrystal YAG Fiber	General Atomics	K. Mazdiyasni	6.2	$407K	AUG 91	27 Mo

*See PE nomenclature explanation on following page.

WRIGHT LABORATORY MATERIALS DIRECTORATE

CURRENT STRUCTURAL CMC PROGRAMS (CONT.)

TITLE	ORGANIZATION	P.I.	FUNDS (PE/$TOTAL)		START	DURATION
EFG YAG Fiber	Saphikon	H. Bates	6.2	$426K	AUG 91	27 Mo
SiC CVD Fiber	Georgia Tech	J. Lackey	6.2	$225K	SEP 91	15 Mo
YAG-Alumina Fiber	UES	T. Mah	6.2	$485K	AUG 91	24 Mo
HPZ Fiber CMC	UTRC	J. Brennan	6.2	$296K	AUG 91	18 Mo

PE Nomenclature:

6.2	Materials Directorate 6.2
6.3	" " 6.3
6.5	" " 6.5 (SBIR)
6.1A	AFOSR 6.1
6.2P	Propulsion Directorate 6.2
6.2D	DARPA 6.2

CERAMIC COMPOSITES SUMMARY

- MAINTAIN STRONG GENERIC TECHNOLOGY BASE THROUGH IN-HOUSE PROGRAM
- ENHANCE PROCESSING ACTIVITY WITH COORDINATED IN-HOUSE/CONTRACTUAL EFFORTS
- TRANSITION STATE-OF-THE-ART CMC'S TO APPLICATION VIA ML 6.2/6.3 PROGRAMS AND JOINT ACTIVITIES WITH OTHER GOVERNMENT ORGANIZATIONS
- WORK ADVANCED CMC'S (~2800°F CAPABLE) FOR IHPTET VIA FIBER PROGRAMS AND COMPOSITE EVALUATIONS/DEVELOPMENT
- INVESTIGATE NON-PROPULSION OUTLETS FOR FUTURE OPPORTUNITIES

Office of
Aeronautics and
Space
Technology

NASA

STRUCTURAL CERAMICS RESEARCH

Presentation to

INTERAGENCY COORDINATING COMMITTEE ON STRUCTURAL CERAMICS

Stephen G. Moran
Program Manager for Materials
High-Speed Research Division
May 13. 1992

NASA STRUCTURAL CERAMICS FUNDING ($M)

OAST

Total NASA Funding	**12.2**	**18.8**	**28.1**
- Advanced High Temperature Engine Materials Technology	3.2	3.2	3.2
- Enabling Propulsion Materials	2.0	8.5	18.0
- Thermal Protection Systems	3.6	3.3	3.1
- Other Base R&T	3.4	3.8	3.8
Civil Service Professionals	**62**	**66**	**68**
Other Agency Funding	**0.7**	**0.7**	**0.7**
- DOE	0.5	0.5	0.5
- Army Research Office	0.2	0.2	0.2

Compiled By:
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- Dr. Hugh R. Gray, Manager, HITEMP Program (216) 433-3230
- Mr. Joseph R. Stephens, Manager, EPM Program (216) 433-3195

NASA Ames Research Center
- Dr. Daniel J. Rasky, Chief, Thermal Protection Materials Branch (415) 604-1098

NASA STRUCTURAL CERAMICS RESEARCH

OAST

FOCUS: FIBER REINFORCED CERAMIC MATRIX COMPOSITES
THERMAL PROTECTION SYSTEMS

APPLICATIONS: ADVANCED AEROSPACE PROPULSION, POWER, AND THERMAL PROTECTION SYSTEMS

- **Aircraft Gas Turbines**
- **NASP**
- **Space Power Systems**
- **Aerobrakes**
- **Rocket Engines**

MAJOR PROGRAM COMPONENTS

- **Advanced High Temperature Engine Materials Technology (HITEMP)**
- **Enabling Propulsion Materials (EPM)**

ADDITIONAL EFFORTS

- **NASP:** Hydrogen Compatibility, Seals, TPS
- **Civil Space Technology Initiative (CSTI):**
  CFCC for Advanced Rocket Engines
- **R&T Base: Space, Aeronautics**
- **DOE:** ATTAP - Testing, Design, Life Prediction
  CTAHE - Toughened Ceramics Life Prediction
- **ARO:** CFCC Validation w/Williams, Textron Lycoming

NASA'S

ADVANCED HIGH TEMPERATURE ENGINE MATERIALS TECHNOLOGY PROGRAM

HITEMP

HITEMP

....is a lab-scale materials and structures project

....is focused on key technical issues in order to accelerate the utilization of composite materials for generic engine components

....actively seeks cooperative projects with engine companies to resolve key technical issues and evaluate LeRC materials in rig/engine tests

OUR APPROACH

- FOCUSED MATERIALS AND STRUCTURES RESEARCH ....
  - Builds upon base R&T and generic hypersonics
  - Feeds laboratory-scale composites and analysis methods into component development programs (EPM, IHPTET, NASP)
  - Closely coordinated with development programs
- INTERDISCIPLINARY TECHNOLOGY TEAMS ....
  - Materials
  - Structures
  - Sensors
  - Aerothermal Loads

  → Composite Materials Technology Readiness
- UTILIZES SKILLS OF RESEARCHERS FROM ....
  - LeRC
  - Universities
  - U.S. Industry

  → Working Cooperatively

TURBINE MATERIALS
Ceramic Matrix Composites

FY 90 | 91 | 92 | 93 | 94 | 95 | 96

Issues

- Fiber/interface development
- Composite development
- Analysis
- Test methods

SiC (SCS-6) /BAS	x/XAS
Al_2O_3 (Sx) / Al_2O_3 SiC (SCS-6) /RBSN SiC (SCS-6) /RFSC	Oxide/Oxide SiC (x) / RBSN

OBJECTIVES

CMC's > 1095°C (2000°F)

CMC's > 1370°C (2500°F)

- Turbine Components Blades, Vanes, Disks

Focus Areas

Fiber/Interface Development
- Materials design/selection
- High strength
- Thermal/mechanical stability
- Small diameter
- Stable interface coatings
- Engineered interface

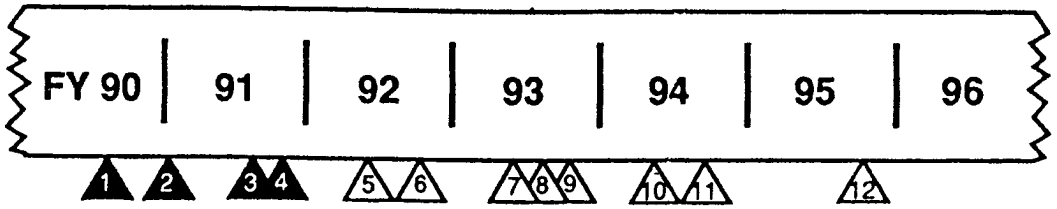
Composite Development
- Materials design/selection
- Characterization
- Failure mechanisms
- Thermal/mechanical stability
- 3D Fabricability
- Environmental durability

Analysis
- Life prediction
- Structural applications
- Architecture
- Residual stresses
- Fiber/matrix creep mismatch

Test Methods
- NDE
- Fiber/Interface testing
- Smart composites
- Instrumentation
- Standard test methods
- Benchmark tests

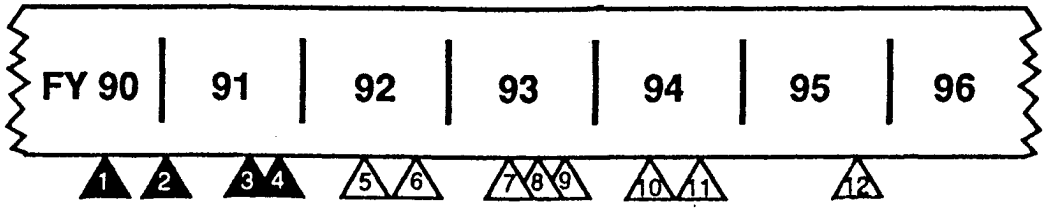
MICS-Narrative

Ceramic Matrix Composites
Fiber/Interface Development



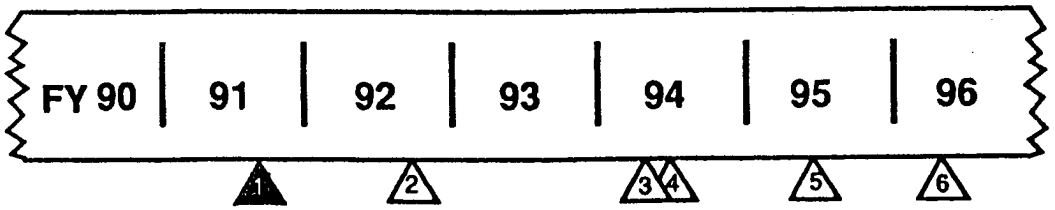
1. C-rich SiC coating on SCS-6 fiber characterized.
2. Strong, small diameter sapphire (Al_2O_3) fibers by laser processing demonstrated.
3. Creep model for current polycrystalline SiC fibers developed.
4. Strong, small diameter eutectic (Al_2O_3 -$Y_3Al_5O_{12}$) fibers demonstrated.
5. Develop models for fracture, strength and creep of sapphire fibers up to 1650 °C (3000°F).
6. Demonstrate CVD SiC fibers with improved creep resistance.
7. Demonstrate strong, small diameter(<20um) polycrystalline SiC fibers stable to > 1370 °C (2500°F).
8. Identify oxide fibers that are stronger and more creep resistant than sapphire fibers.
9. Demonstrate >1370 °C (2500°F) interface coatings for oxide fibers.
10. Determine feasibility of oxidatively stable, carbon-containing interface coatings for SiC fibers.
11. Determine feasibility of >1370 °C (2500°F) non-carbon interface coatings for SiC fibers.
12. Demonstrate continuous single crystal SiC fibers.

Ceramic Matrix Composites
Fiber/Interface Development



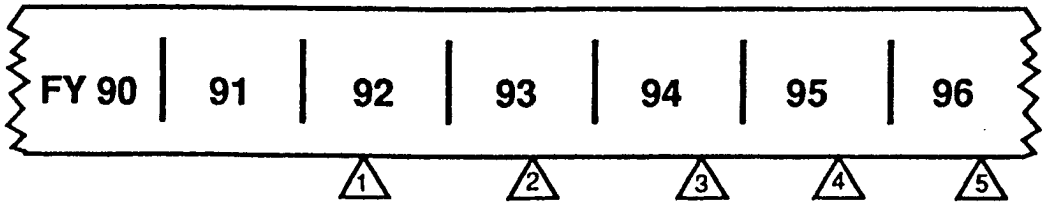
1. C-rich SiC coating on SCS-6 fiber characterized.
2. Strong, small diameter sapphire (Al_2O_3) fibers by laser processing demonstrated.
3. Creep model for current polycrystalline SiC fibers developed.
4. Strong, small diameter eutectic (Al_2O_3 -$Y_3Al_5O_{12}$) fibers demonstrated.
5. Develop models for fracture, strength and creep of sapphire fibers up to 1650 °C (3000°F).
6. Demonstrate CVD SiC fibers with improved creep resistance.
7. Demonstrate strong, small diameter(<20um) polycrystalline SiC fibers stable to > 1370 °C (2500°F).
8. Identify oxide fibers that are stronger and more creep resistant than sapphire fibers.
9. Demonstrate >1370 °C (2500°F) interface coatings for oxide fibers.
10. Determine feasibility of oxidatively stable, carbon-containing interface coatings for SiC fibers.
11. Determine feasibility of >1370 °C (2500°F) non-carbon interface coatings for SiC fibers.
12. Demonstrate continuous single crystal SiC fibers.

Ceramic Matrix Composites
Composite Development >1095° C (2000° F) CMC



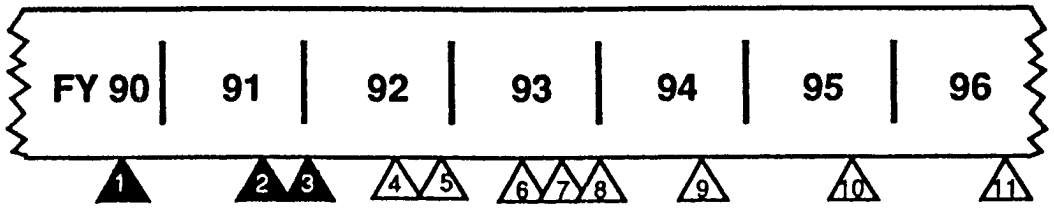
1. Processing for strong, tough SCS-6/BAS (Barium AluminoSilicate) developed.
2. Optimize processing for strong, tough SCS-6/SAS (Strontium AluminoSilicate).
3. Determine factors limiting use temperature/life of SiC/BAS and SiC/SAS.
4. Optimize processing for 3D X/XAS.
5. Determine feasibility of improved X/XAS.
6. Determine structural properties for optimum X/XAS.

Ceramic Matrix Composites
Composite Development: >1370° C (2500° F) Oxide CMC



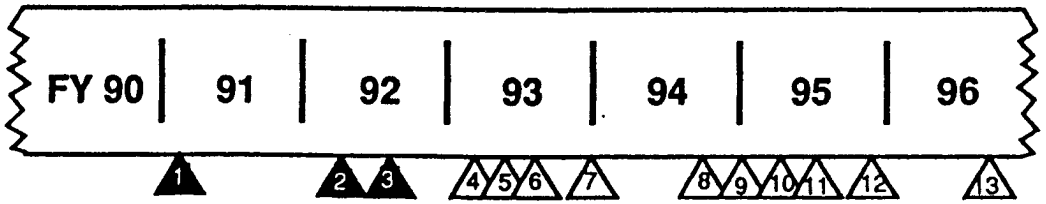
1. Demonstrate strong, tough Al_2O_3(Sx:single crystal) /Al_2O_3.
2. Determine factors limiting use temperature/life of Al_2O_3/Al_2O_3 .
3. Determine feasibility of improved oxide/oxide.
4. Optimize processing for 3D oxide/oxide.
5. Determine structural properties for optimum oxide/oxide.

Ceramic Matrix Composites
Composite Development: >1370° C (2500 °F) Non-Oxide CMC



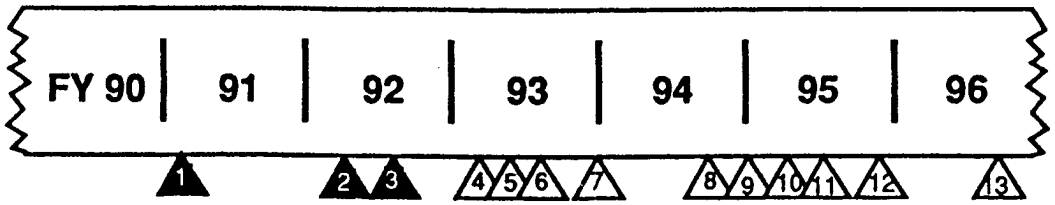
1. Room temperature SCS-6/RBSN (Reaction Bonded Silicon Nitride) laminate properties determined.
2. Feasibility of high density CMC's (HIPed RBSN and reaction-formed SiC) demonstrated and transitioned to EPM program.
3. Microstructural approaches for improved SiC/RBSN identified.
4. Determine tensile strength, creep and fatigue properties of SCS-6/RBSN at elevated temperatures.
5. Identify oxidation-resistant RBSN coatings to >1370°C (2500°F).
6. Identify environmental factors limiting use temperature/life of SCS-6/RBSN.
7. Identify microstructural factors limiting use temperature/life of SCS-6/RBSN.
8. Determine processing cycle for 2D RBSN composites with small diameter fibers.
9. Optimize processing for SiC/RBSN with >1370°C (2500°F) capability.
10. Optimize processing for 3D SiC/RBSN.
11. Determine structural properties for optimum SiC/RBSN.

Ceramic Matrix Composites Analysis



1. Acoustic emission technique for determining failure in SiC/RBSN demonstrated.
2. Controlling failure mechanisms for SiC/RBSN identified.
3. Analysis on radiation/conduction interaction to establish heat transfer capability conducted.
4. Develop code for composite ceramic analysis and reliability evaluation of structures (C/CARES).
5. Characterize failure mechanisms of model 2-D/3-D woven and braided CMC's to 1650°C (3000°F).
6. Establish crack growth threshold and crack growth rates in model CMC's. (e.g. Corning CAS)
7. Simulate thermomechanical behavior of SiC/RBSN.
8. Establish microscale failure mechanisms of SiC/SiC.
9. Develop and verify quasi-static fracture and damage models for 2-D/3-D CMC's.
10. Incorporate inelastic analysis into BEM for transient heat transfer and thermoelastic analysis.
11 Augment C/CARES to account for load redistribution.
12. Demonstrate fracture toughness test and study R-curve behavior for SiC/CAS and SiC/SiC CMC's.
13. Develop time dependent reliability models.

Ceramic Matrix Composites Analysis



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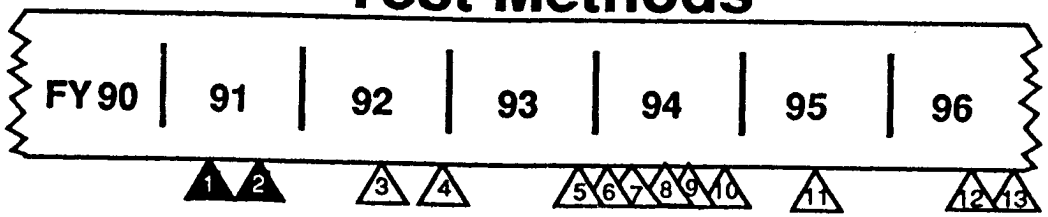
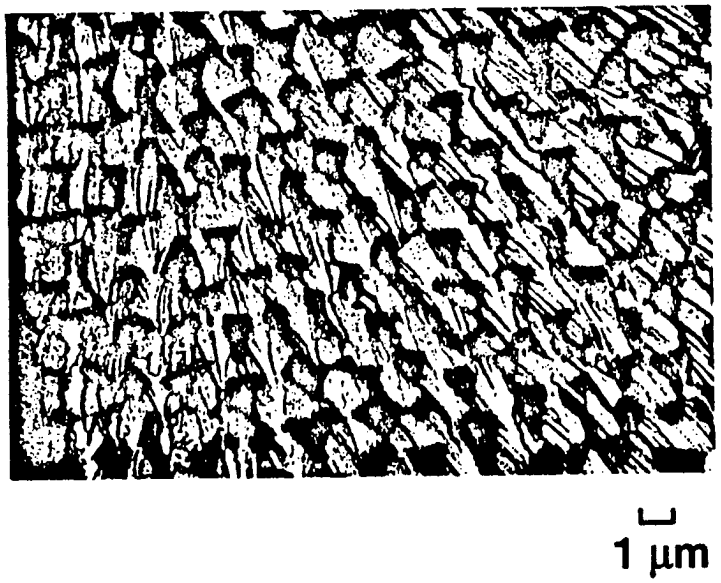
Ceramic Matrix Composites
Test Methods

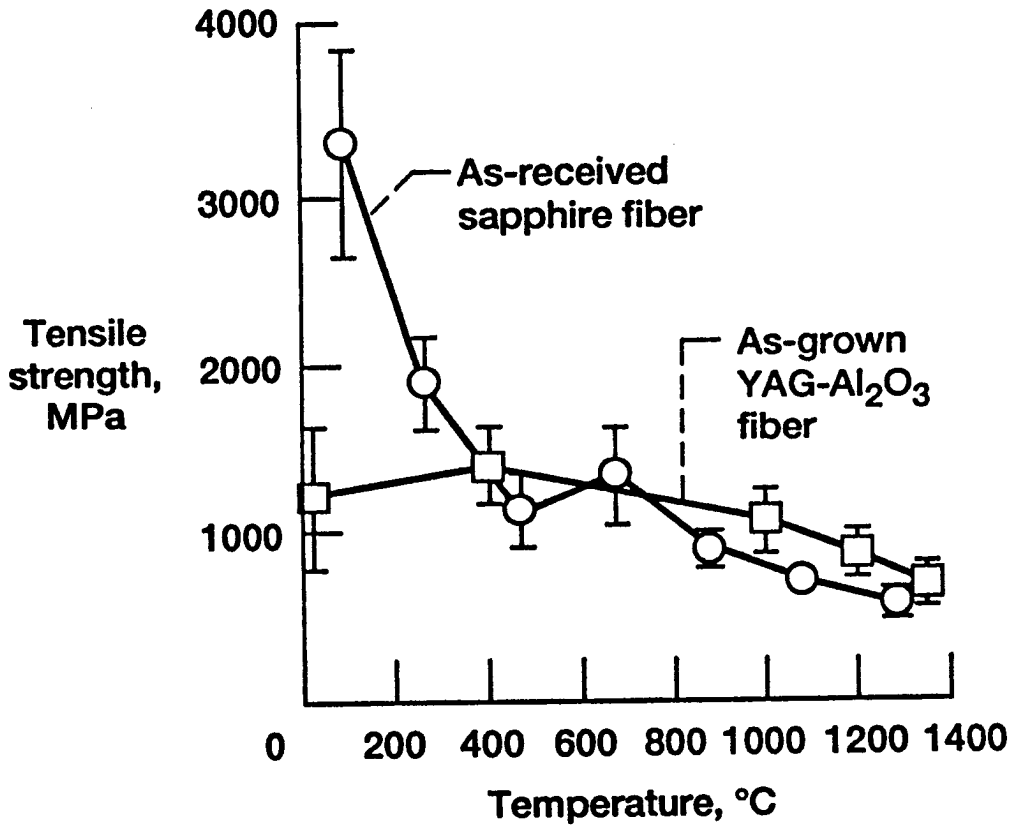


1. Thin film thermocouples to 1370°C (2500°F) demonstrated.
2. Thermomechanical test system to 1650°C (3000°F) completed.
3. Demonstrate heat flux sensing elements to 1370°C (2500°F).
4. Develop preliminary consensus "Standards" for conducting mechanical tests to 1650°C (3000°F).
5. Develop system for creep/recovery testing to 1650°C (3000°F).
6. Establish ultrasonic methods for addressing fiber-matrix bond quality/strength/degradation.
7. Complete round robin testing program.
8. Demonstrate field operable pyrometry scheme with emittance and radiation corrections.
9. Verify fracture, damage accumulation models with acoustic emission methods on 2D/3D woven samples.
10. Develop fiber push out capability to 1370°C (2500°F).
11. Demonstrate thin film heat flux gage to 1650°C (3000°F).
12. Apply constitutive NDE to selected generic structural shapes in conjunction with C/CARES and CARES/LIFE to predict load response, fracture modes, and life expectancy.
13. Demonstrate full field heat flux measurements using pyrometry with emittance and radiation corrections incorporated.

Laser Heated Float Zone Processing for Strong Directionally Solidified YAG-Al_2O_3 Fibers Demonstrated



Mirror region of fiber fracture surface
YAG - light, Al_2O_3 - dark



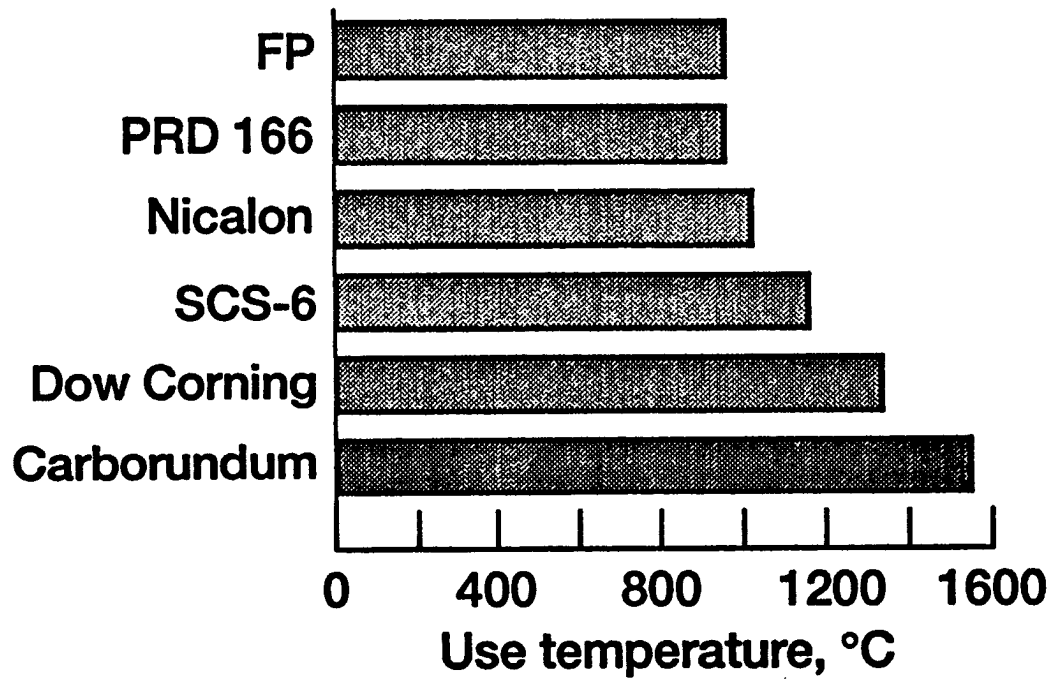
- Better intermediate and high temperature strength

CD-91-57403

Creep Model Developed and Creep Parameters Experimentally Established for Current Polycrystalline SiC Fibers

- Primary stage model: $E_c = A_o \sigma^n t^p \exp[-B/T]$

Example fiber ranking by use temperature for 20 ksi/100 hr/ 0.1% E

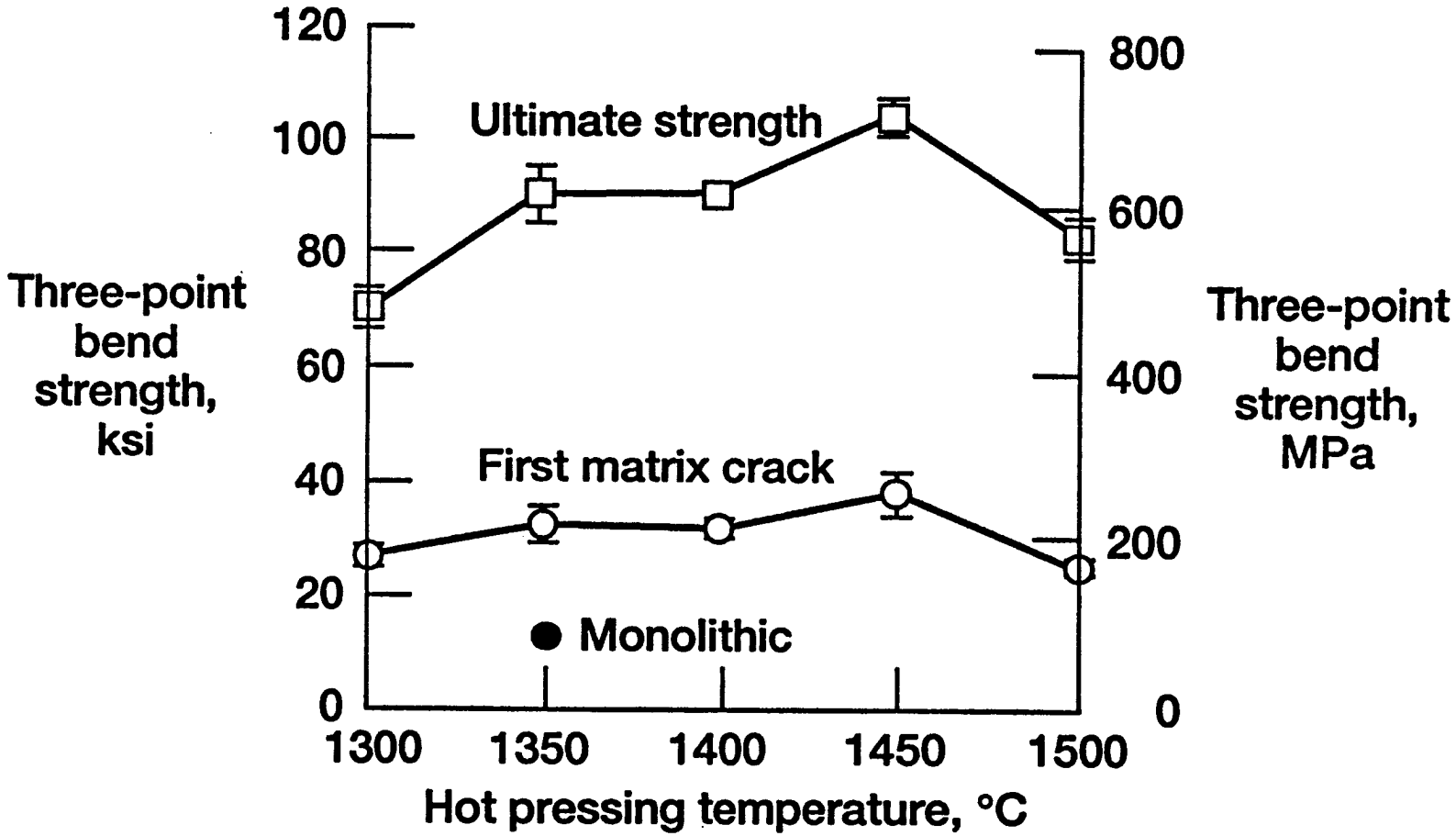


- Use temperature can be predicted for any $\sigma/E/t$
- Creep model aids structural analysis of advanced composites

CD-91-57406

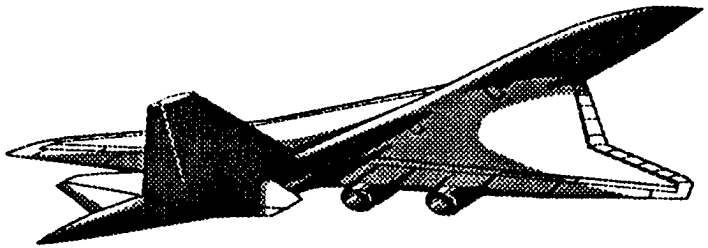
Hot Pressing Conditions Optimized to Achieve Strong, Tough SCS-6/BAS Composites

($V_f \simeq 23\%$)



- Maximum toughness achieved by hot pressing at 1450 °C
- Three invention disclosures filed '90-'91

CD-91-57405



Enabling Propulsion Materials (EPM)

for the

High Speed Civil Transport

Enabling Propulsion Materials

Objective

By 1999 develop and demonstrate in cooperation with U.S. industry the technical feasibility of high-temperature, lightweight composites for critical engine components of the High Speed Civil Transport

FY '92 ACCOMPLISHMENTS

- Contract signed with P&W/GEAE Team December 16, 1991
- Fourteen sub-contractors on-board
- Baseline engine cycle & HSCT mission identified for materials-design benefits analysis
- METCAN selected as the fiber property prediction code
- CSTEM selected as computer code for composite analysis

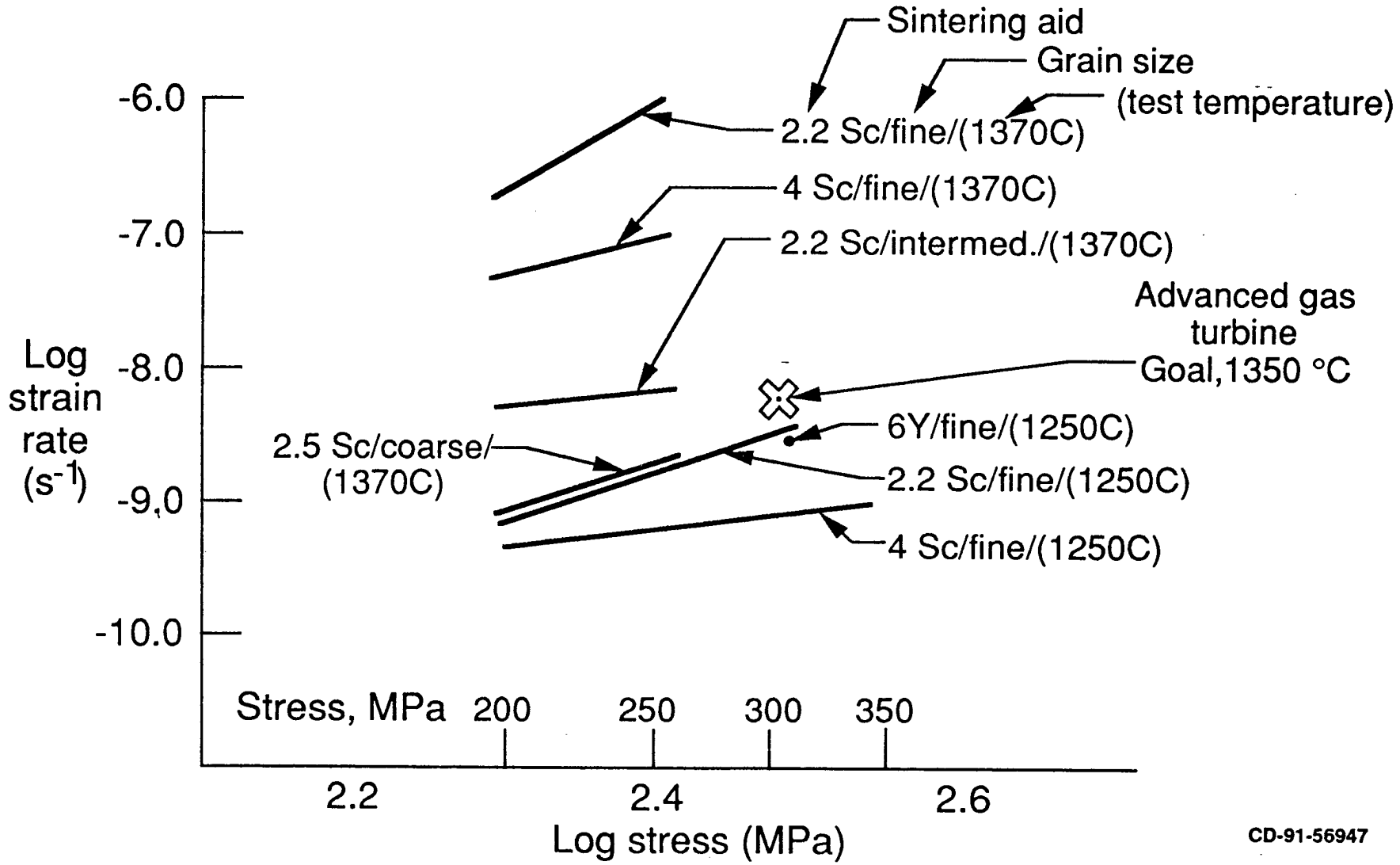
FY '93 MAJOR MILESTONES

- Identify alternate materials and design for combustor
- Select processing technique, for CMC composites & deliver coupons to NASA for independent evaluation
- Primary fiber selection for nozzle
- Identify low-cost fabrication options for nozzle sub-components
- Review and up-date other critical engine components

• RESULTS FOR SOME ADDITIONAL EFFORTS

- ○ NASP: HYDROGEN COMPATIBILITY, SEALS
- **• CSTI (CIVIL SPACE TECHNOLOGY INITIATIVE)**
  - **• FRC FOR ADVANCED ROCKET ENGINES**
- **• RESEARCH AND TECHNOLOGY BASE: SPACE, AERONAUTICS**
- **• DOE TRANSPORTATION**
  - **• ATTAP: MATERIALS ASSESSMENT, DESIGN AND LIFE PREDICTION**
  - ○ CTAHE: TOUGHENED CERAMICS LIFE PREDICTION

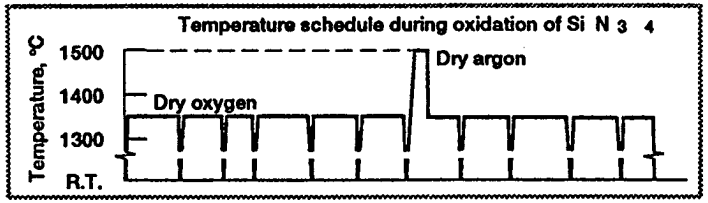
Improved Flexural Creep of Sintered Si_3N_4 Matrices

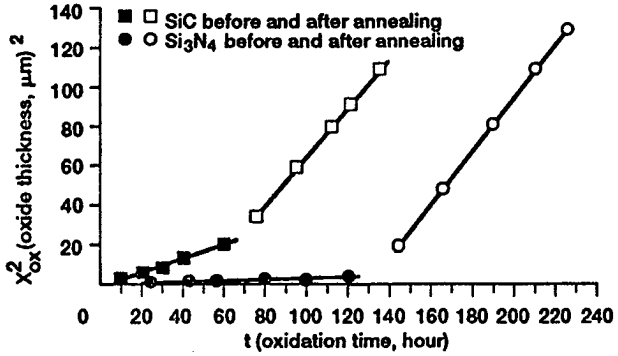


CD-91-56947

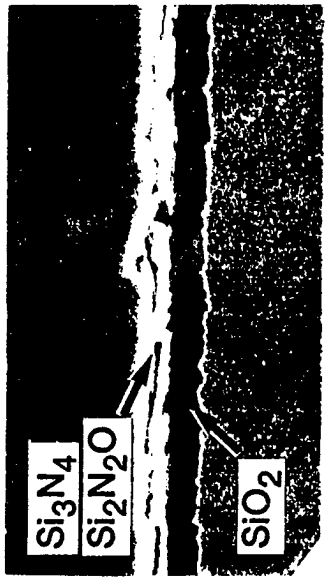
Oxidation Rates of Si_3N_4 Ceramic at 1350°C are Sharply Increased by Brief Exposure to 1500°C.

Effect of Thermal Excursion (1.5 hr, 1500 °C) on the Oxidation Rates of SiC and Si_3N_4 at 1350 °C





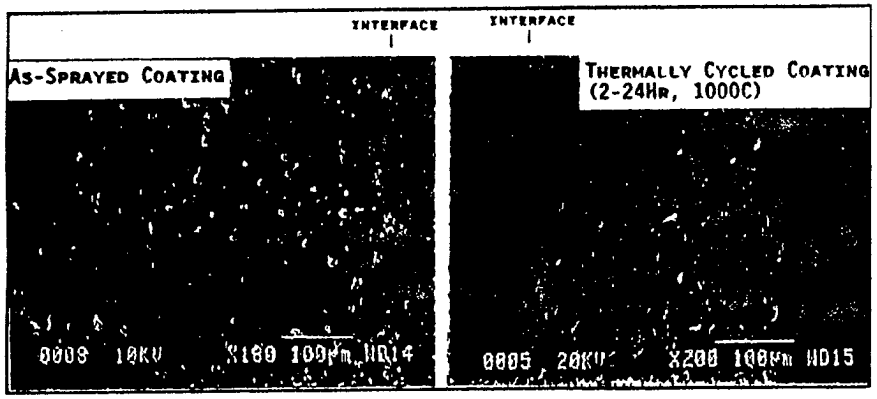
SiC and Si_3N_4 oxidation at 1350°C accelerate over 50-fold after a 90-minute excursion to 1500°C.

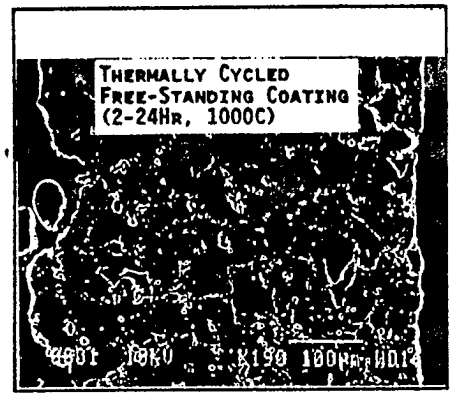


Si_3N_4 oxidation at 1350°C yields an SiO_2 top layer and a Si_2N_2O inner oxide layer. Excursion to 1500°C eliminates the Si_2N_2O and induces microcracks across the SiO_2.

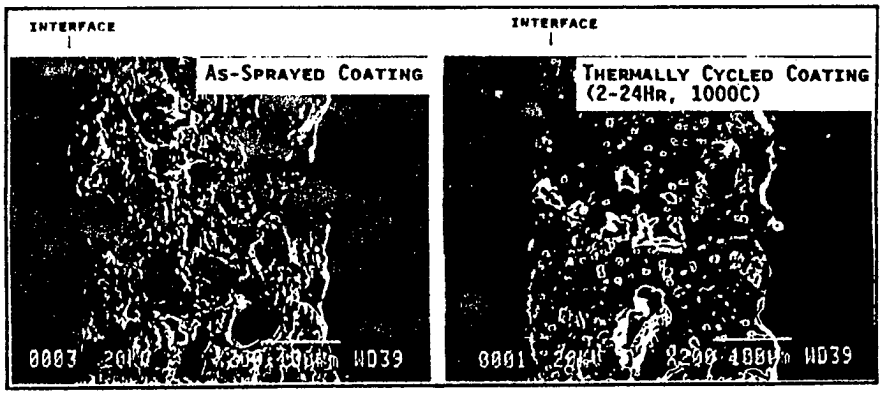
CD-92-60022

Phase Stability is Identified as the Key Factor for Thermal Shock Resistance of Plasma-Sprayed Mullite Coating on SiC





Mullite coating cracks on thermal cycling. Close CTE match between mullite and SiC, and the cracking of free-standing sprayed mullite indicate that predominant factors causing the cracking are within the mullite.



Chemical etching of mullite coating shows the crystallization of amorphous mullite during thermal cycling (amorphous mullite is dissolved in HF). Shrinkage of the coating during crystallization is the key factor for the cracking.

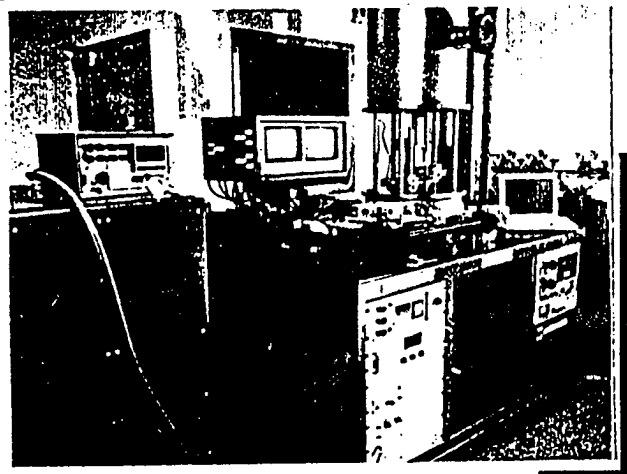
NASA
C-91-03887

AEROSPACE TECHNOLOGY DIRECTORATE

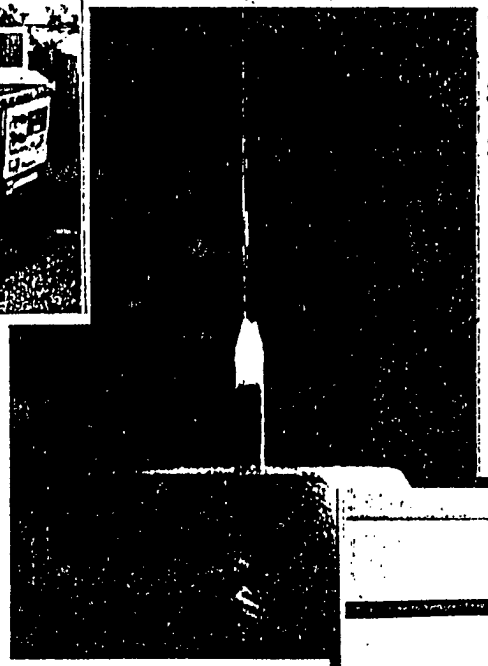
MATERIALS DIVISION

NASA
Lewis Research Center

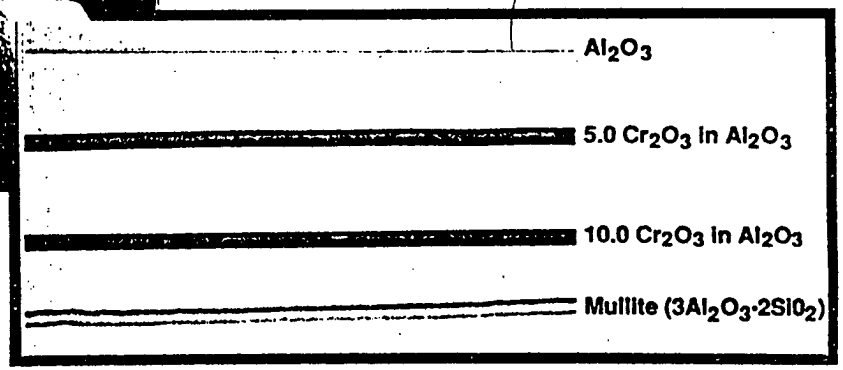
Laser Heated Floating Apparatus Used to Conduct Research on Single Crystal Structural Fibers



Floating zone system



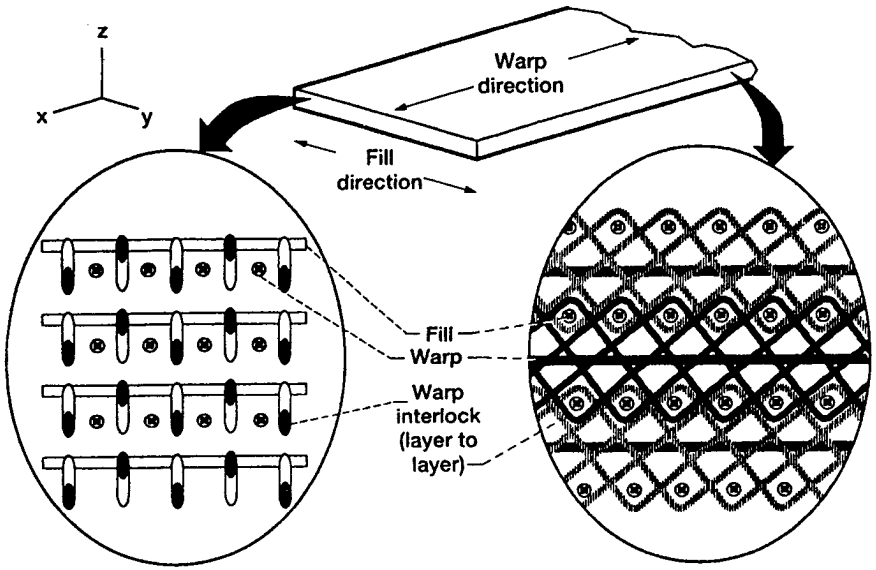
Processing of high strength (6 GPA) Al_2O_3 single crystal fibers

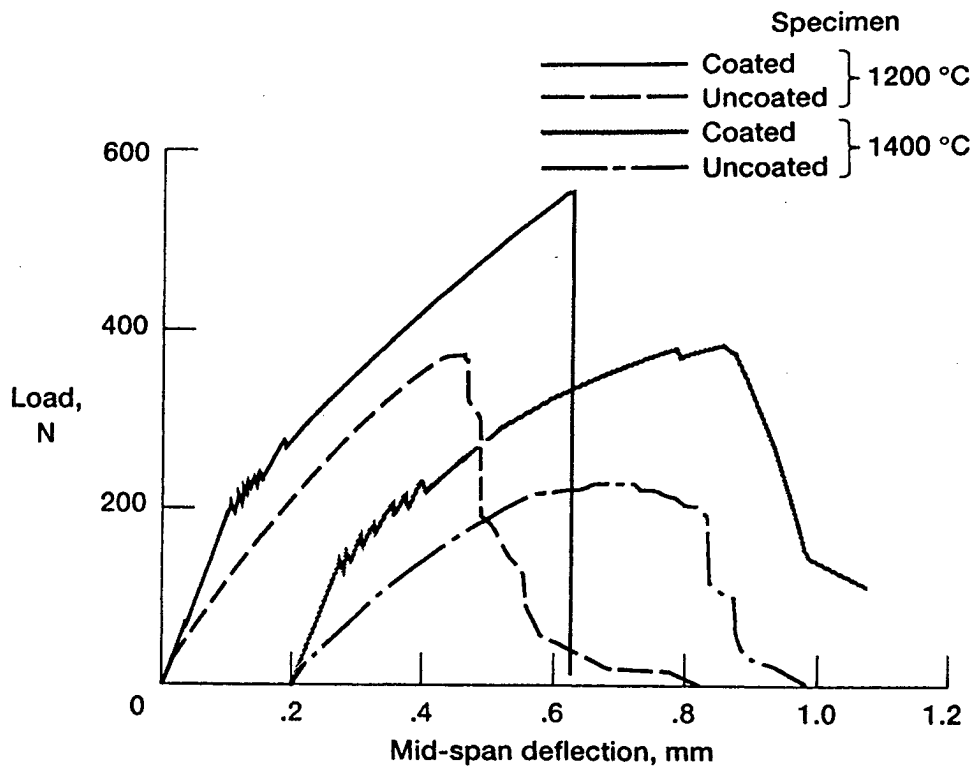


Initial doped fibers grown

CD-91-54732

High Temperature Behavior of Coated and Uncoated 3D Woven SiC Fiber/SiC Ceramic Matrix Composites





- 3D woven fiber architecture prevents delamination, increases impact tolerance and expedites CVI processing.
- Specimens coated with CVD/SiC protect composites from severe oxidation.
- Results show increased composite strength and stiffness.

NASA
C-90-14375

AEROSPACE TECHNOLOGY DIRECTORATE

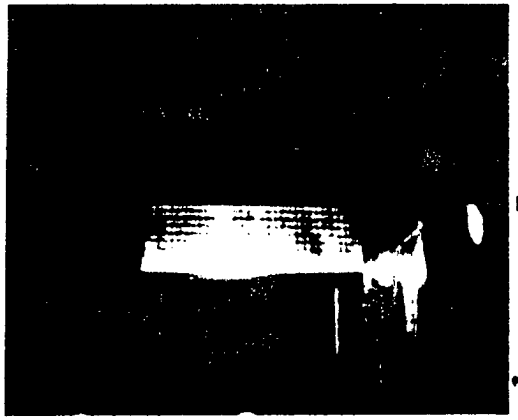
CERAMICS BRANCH

NASA
Lewis Research Center

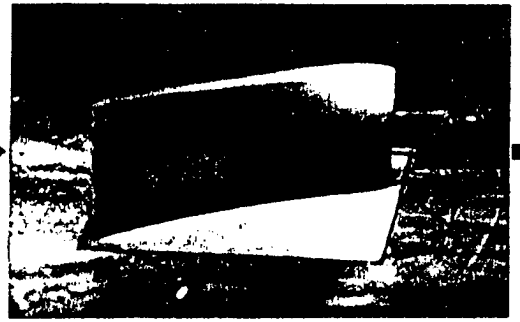
FRCMC TESTING IN ROCKET ENGINE ENVIRONMENTS

1989, Nozzle/combustion chamber

1988-Present, Coupon Tests

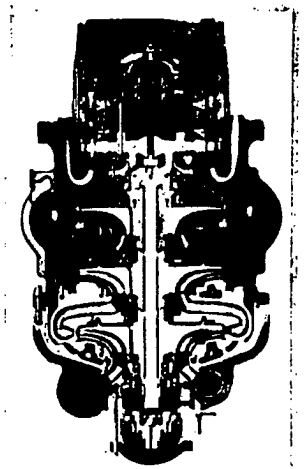


1990, SiC/Si_3N_4 turbine blade



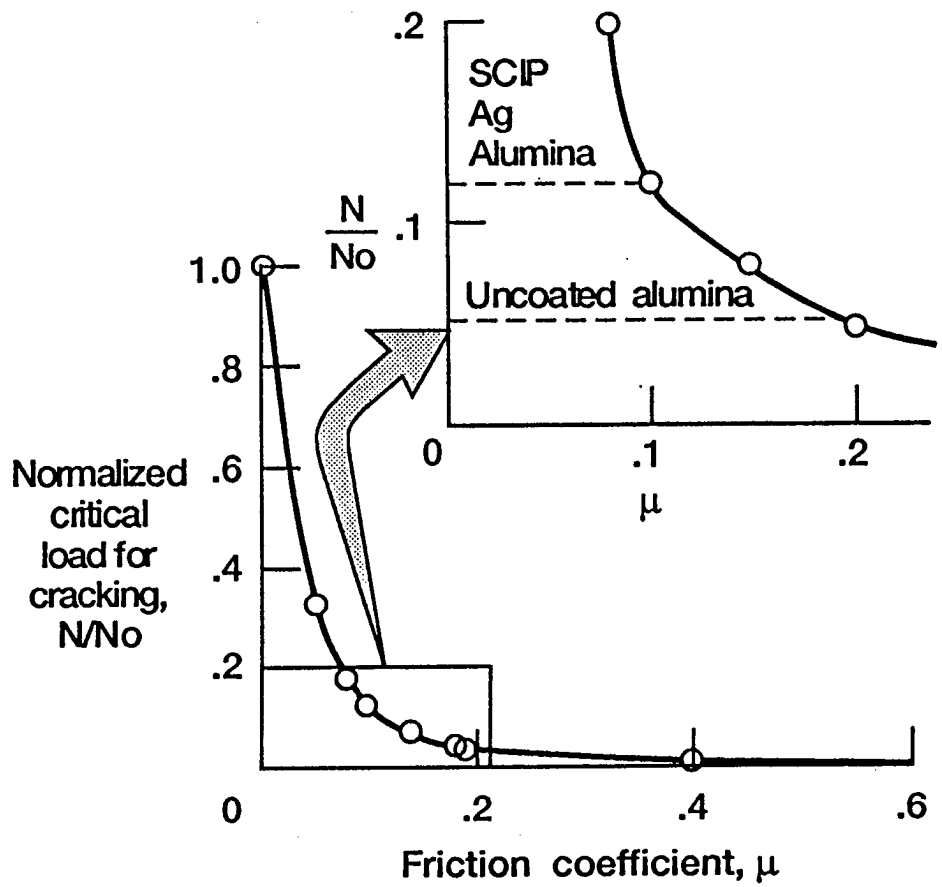
1990, C/SiC turbine blade

1995, Full-scale FRCMC turbopump test



CD-90-51252

Effect of Ion-Plated Silver on Mitigating Surface Tensile Stress and Microfracture During Sliding on Alumina



SCIP Silver reduces friction–allowing increased bearing load

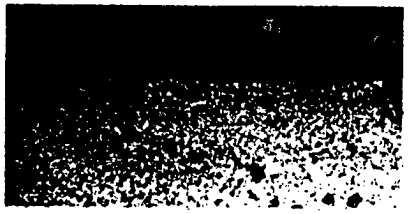
CD-91-58008

Microfracture and Grain Pull-Out in Alumina

Uncoated alumina

SCIP Silvercoat coated alumina

1.5 N



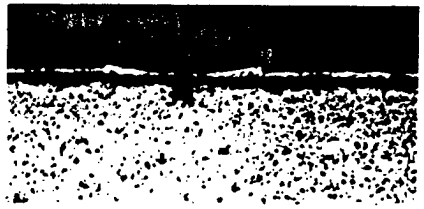
No damage

50 µm

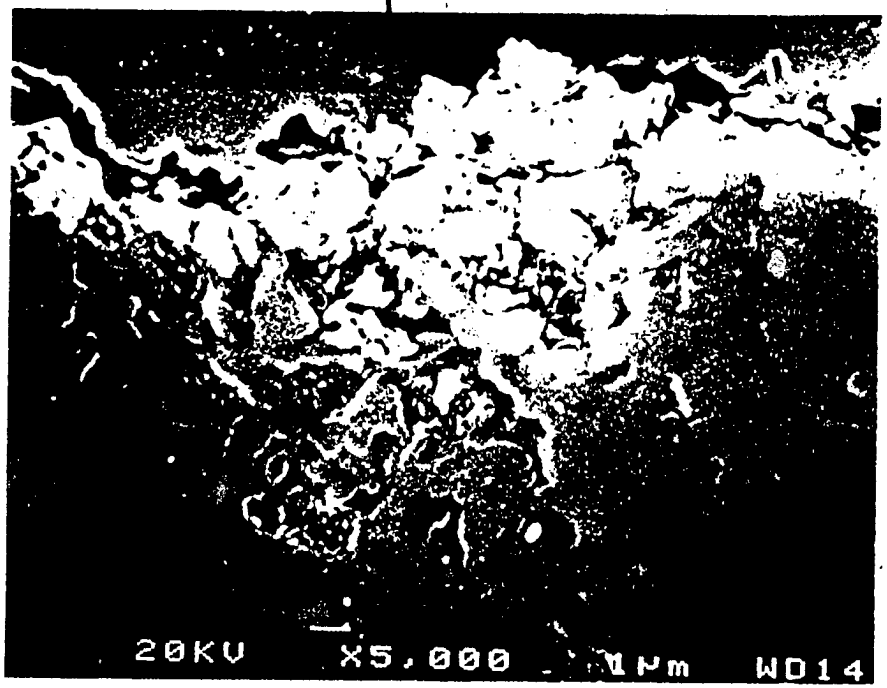
10 N

5.5 N

Early microfracture damage



14.7 N



Features of screen cage ion plated "SCIP" silver

- Lubricates (reduces friction)
- Increases critical load for microfracture
- Coats "around corners" (high throwing power)

CD-91-57484

THERMAL PROTECTION MATERIALS

Ames ⇄ Vehicle Developer ➡ Ground/Flight Experiments ➡ Flight TPS

TPS Technology Development, Arc-Jet Testing & Computational Analyses

Past: 1970-1982
TPS for 2400°F Applications

- Key Shuttle TPS Contributions:
  - LI2200*, FRCI-12*
  - RCG Coating*
  - AFRSI**
  - Gap Fillers*
- X-24B/C First Supersonic Flight Test of Tile TPS
- In Flight Proof of Catalytic Effects on Shuttle
- Arc-Jet Testing for Shuttle, Galileo, Apollo, and DoD Vehicles

* Ames Patent
** Tech Brief

1982 - Present
TPS for 3000°F Applications

- NASP:
  - CMC & Coatings TMP'S
  - GWP #93 & #95
  - Arc-Jet Testing
- Lead Center for SEI Aerobrake TPS
- 3 AFE Experiments
- 3 OEX Experiments
- Pegasus Flight Exp's
- New Materials and TPS:
  - AETB*, TUFI*
  - TABI**, CFBI*
  - CMC Development
  - TOP HAT*
- Numerous Applications of Ames Technology: ATF, A-12, F-117A, B-2

Future to 2005
TPS for 4000°F + Applications

- New Very-High Temp. Ceramics
- Hot Structure CMC TPS
- Durable All-Weather TPS
- Advanced Ablators:
  - Structural Ceramic
  - Volume Reflecting
- TPS for Future Programs:
  - X-30, SSTO'S, HYFLEX
  - Lunar/Mars Missions
  - MESUR, Neptune Probe
- Catalytic Effects for Mars
- Nuclear Rocket Reentry
- Advanced TPS Modeling and Optimization

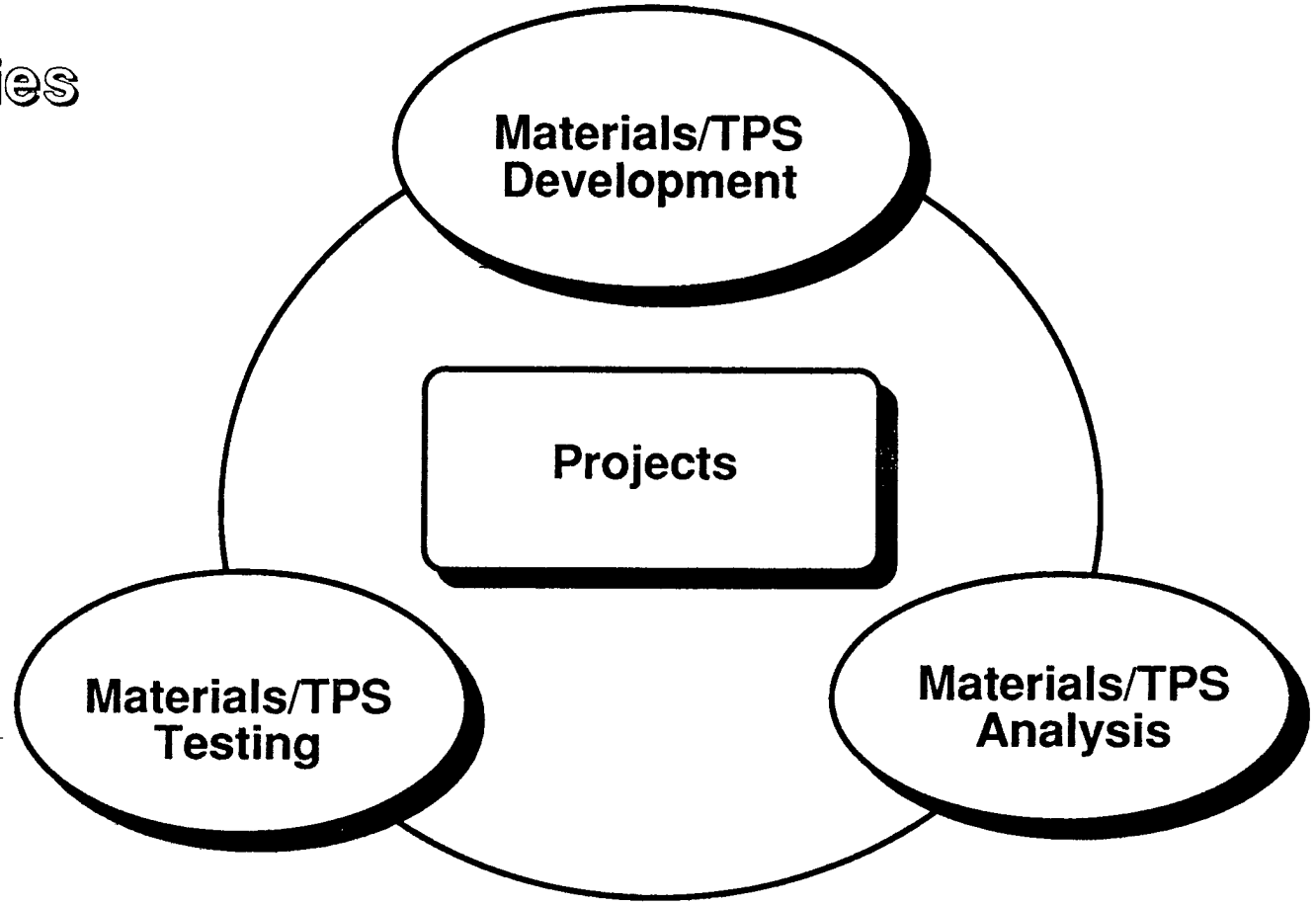
NASA-Ames Research Center Program

- Ceramic Reusable and Ablative Thermal Protection Systems (TPS)
- Ceramic Matrix Composites
- Organometallic Precursors

Major Goals

- New very-high temperature ceramic matrix composites/TPS for 4000°F+ reusability
- High temp. & specific strength ceramic matrix composites for structural TPS applications
- Light weight, rigid, ceramic insulations with improved temperature capability
- Flexible, light weight ceramic insulations with improved durability and insulation capability
- New very-light weight structural ceramic ablators with substantial weight savings compared to state-of-the-art materials

Activities



- A Synergistic, Multidisciplinary Approach
- Continual Research/Technology Development Supports Projects

Projects

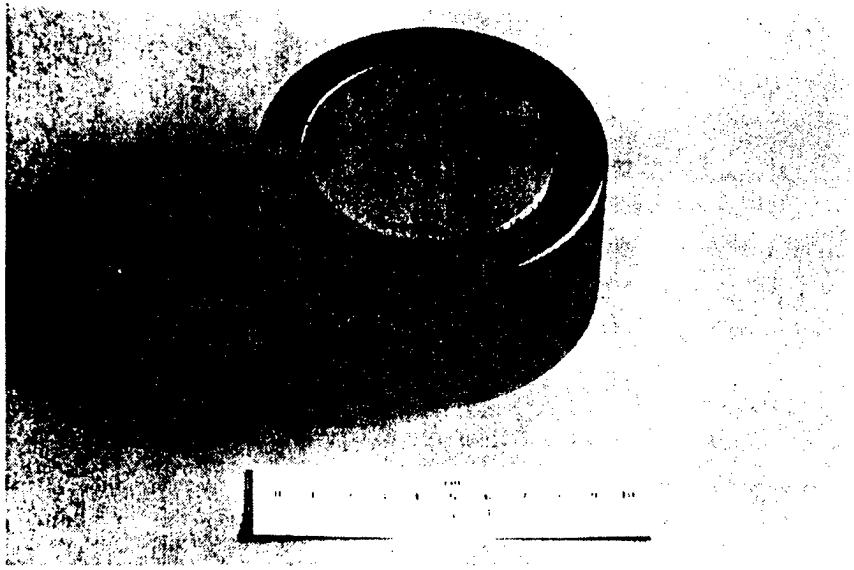
- National Aerospace Plane (GWP #93 & 95)
- SSTO's (Deltaclipper)
- Aerobrakes (Lunar/Mars)
- Planetary Probes (MESUR, ESA/Rosetta)
- Hypersonic Aircraft (Pegasus, Waveriders)

Advanced Material Families

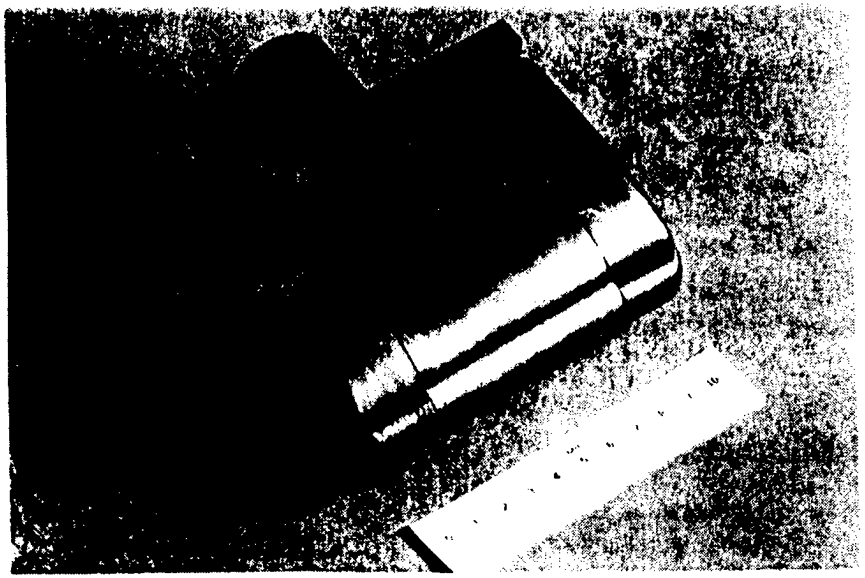
- <u>Ceramic Matrix Composites</u>
  - Very-High Temperature (Diborides)
  - High Temp. & Specific Strength (C/SiC/TiB_2)
  - Polymer Precursors (Si/C/B fibers, tape casting)
- <u>Light Weight Ceramic Insulations</u>
  - Rigid Tiles (TUFI, AETB, SMI)
  - Aerogel Studies (Silica, Zirconia)
  - Flexible Blankets (TABI, CFBI)
- <u>Structural Ceramic Ablators</u>
  - Fibrous Silica & Carbon Substrates
  - Polymer Fillers

Diboride Components

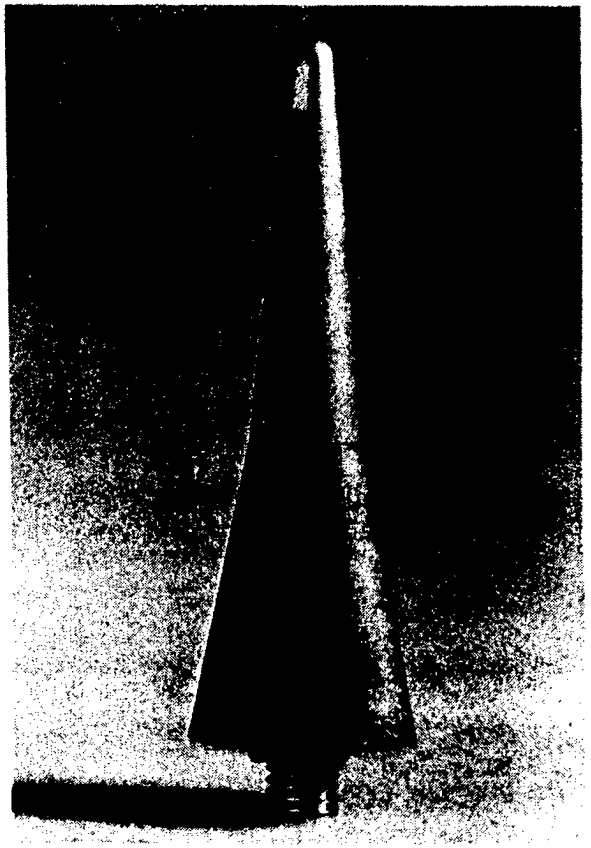
ZrB_2 + 20v/o SiC Skirt
HfB_2 + 20v/o SiC NoseTip



Disk Sample (2.8" diam. x 0.25")



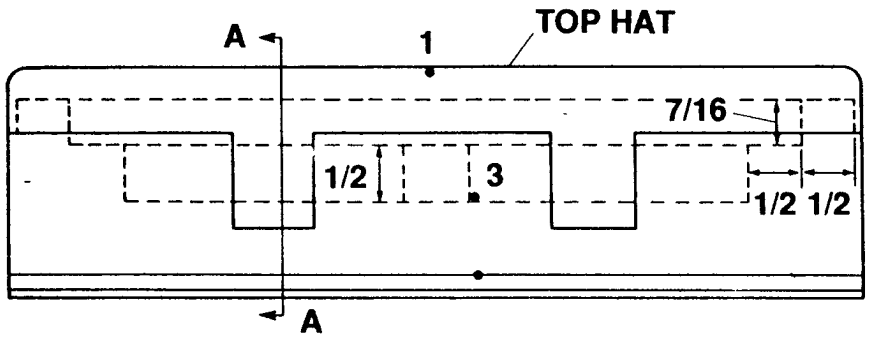
Leading Edge (2.75" x 0.75" diam.)

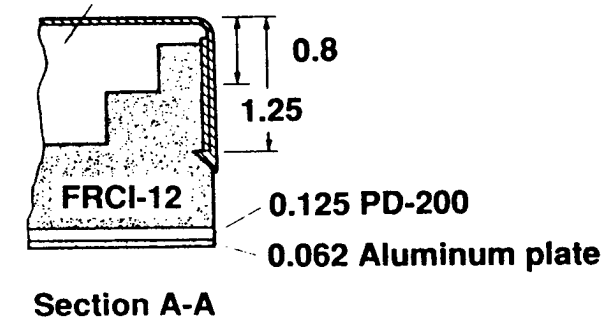


Hypersonic Vehicle Nosetip
(0.141" nose radius)

CERAMIC MATRIX COMPOSITES PROGRAM

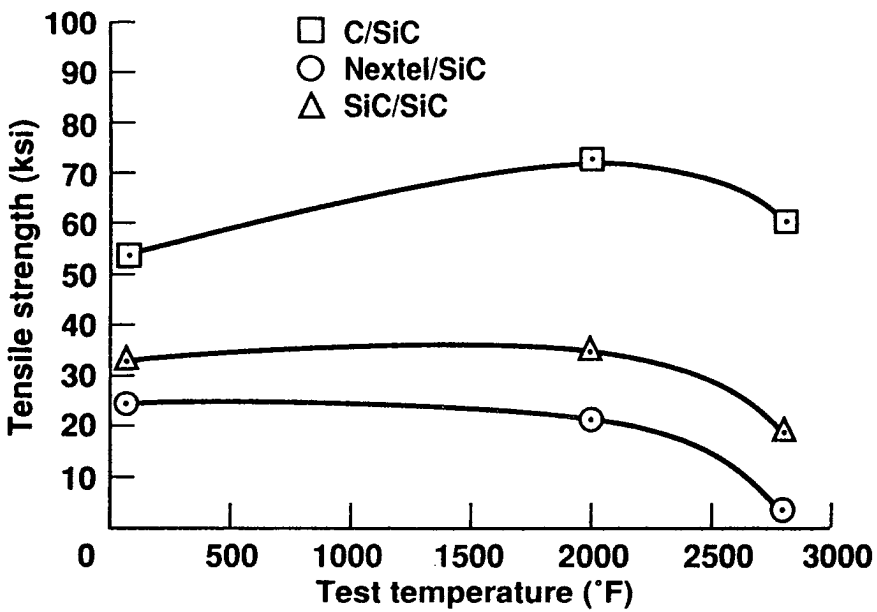
"TOP HAT" Assembly
Edge View



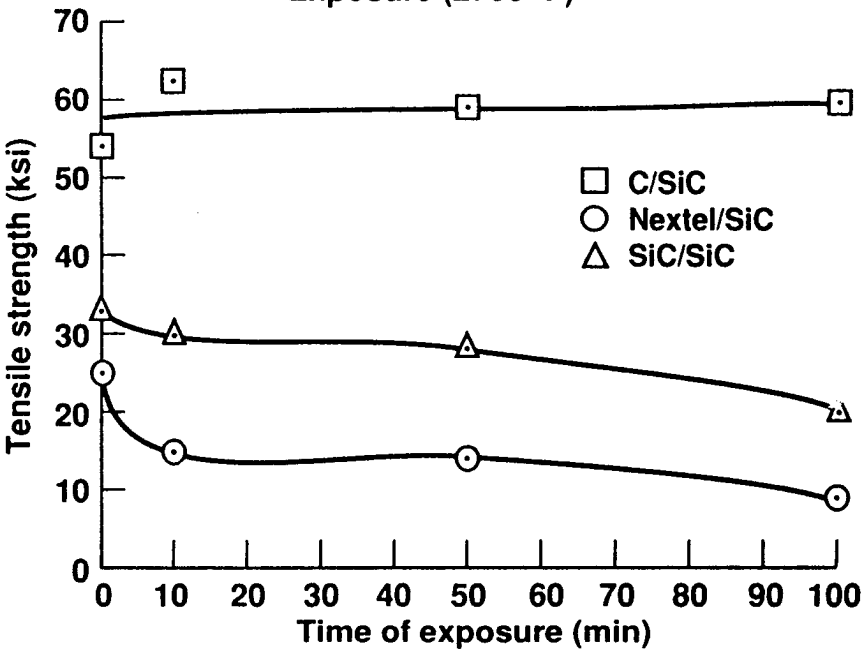


Section A-A

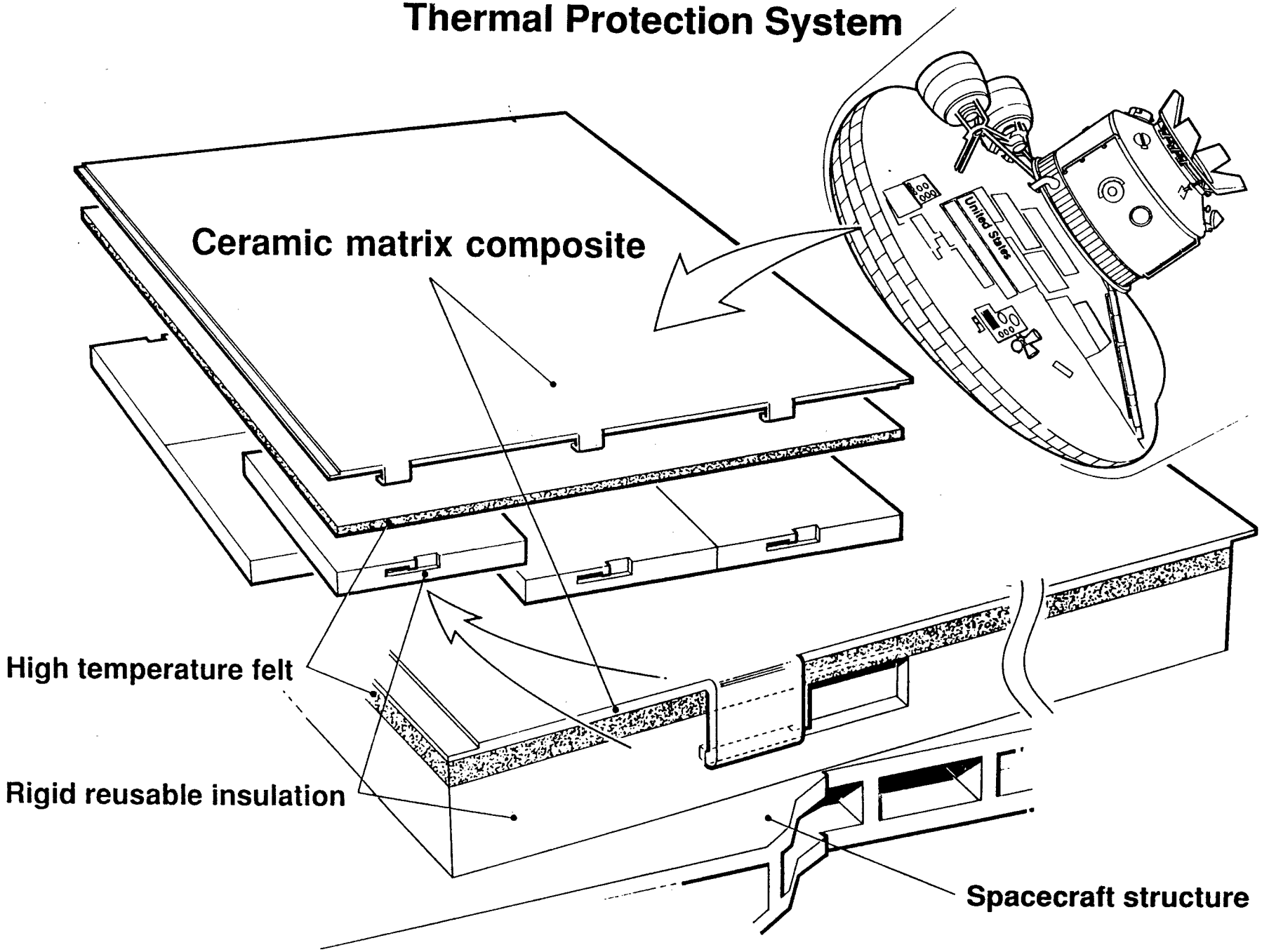
Average Tensile Strength vs. Temperature
Pre-aeroconvective Exposure (U)



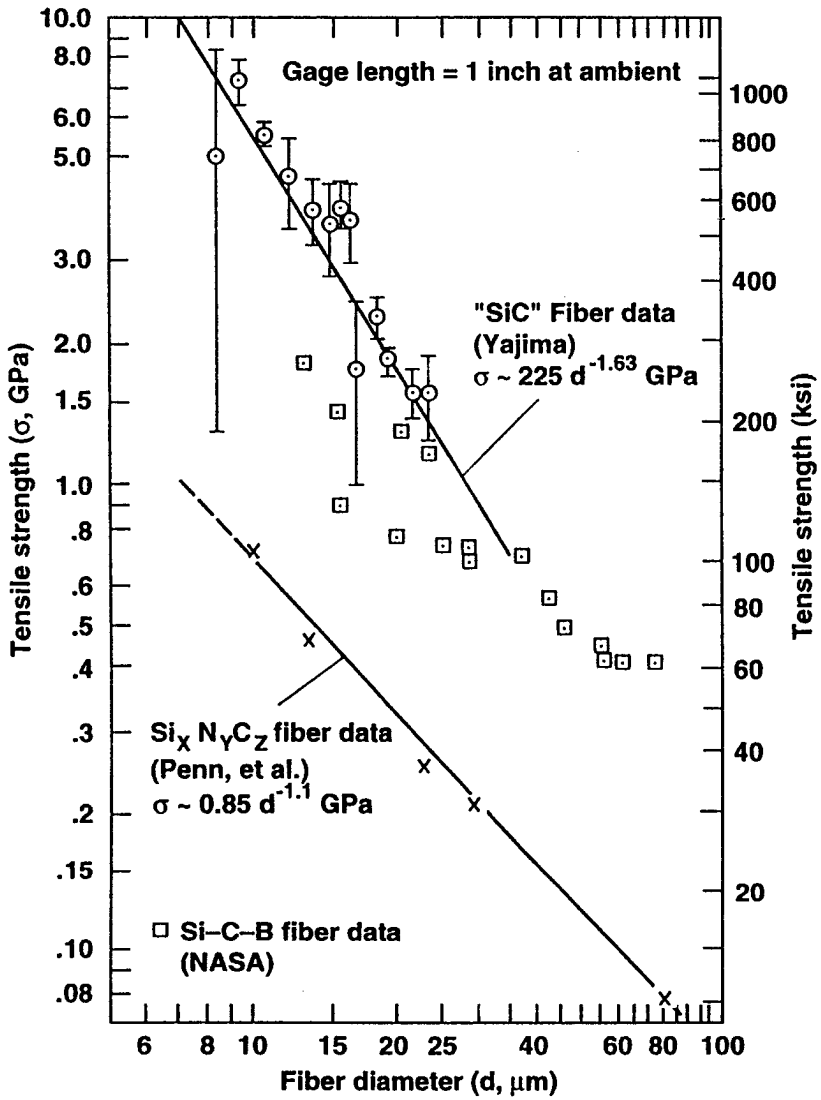
Average Tensile Strength (70 ˚F, Ar) of Carbon/SiC, Nextel/SiC, and SiC/SiC vs. Time of Aeroconvective Exposure (2700 ˚F)



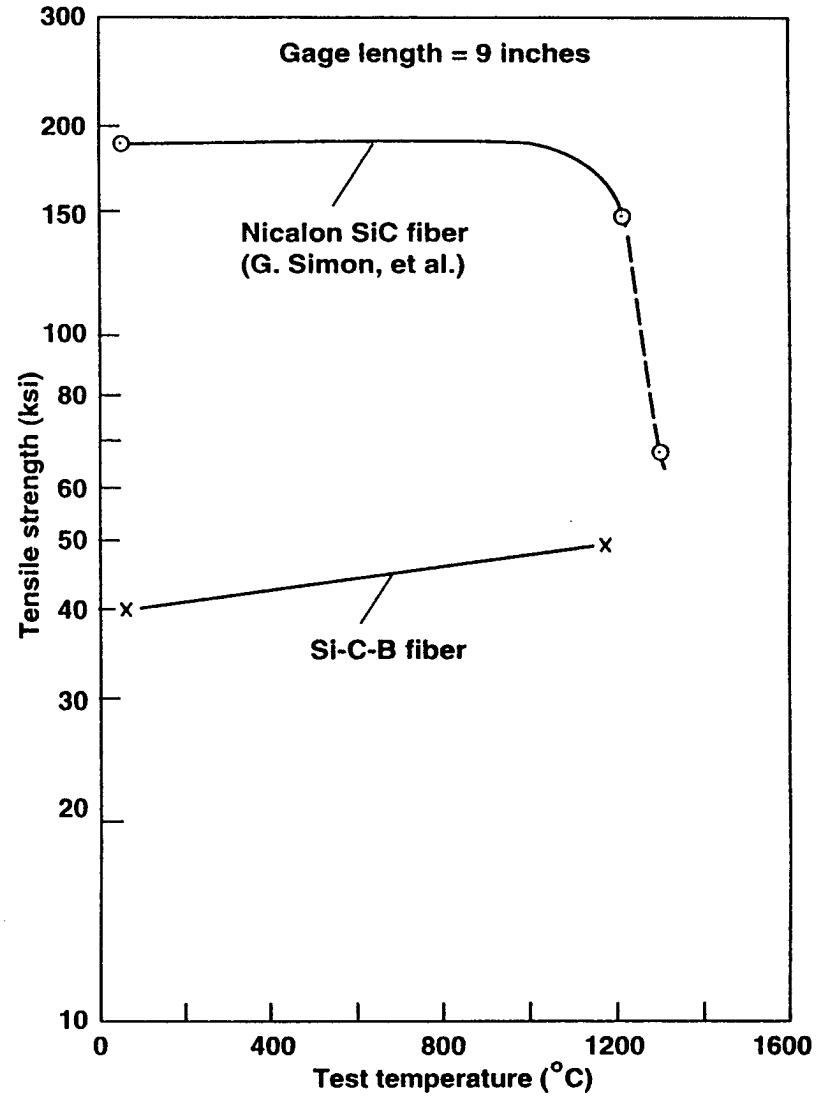
TOP HAT
Thermal Protection System



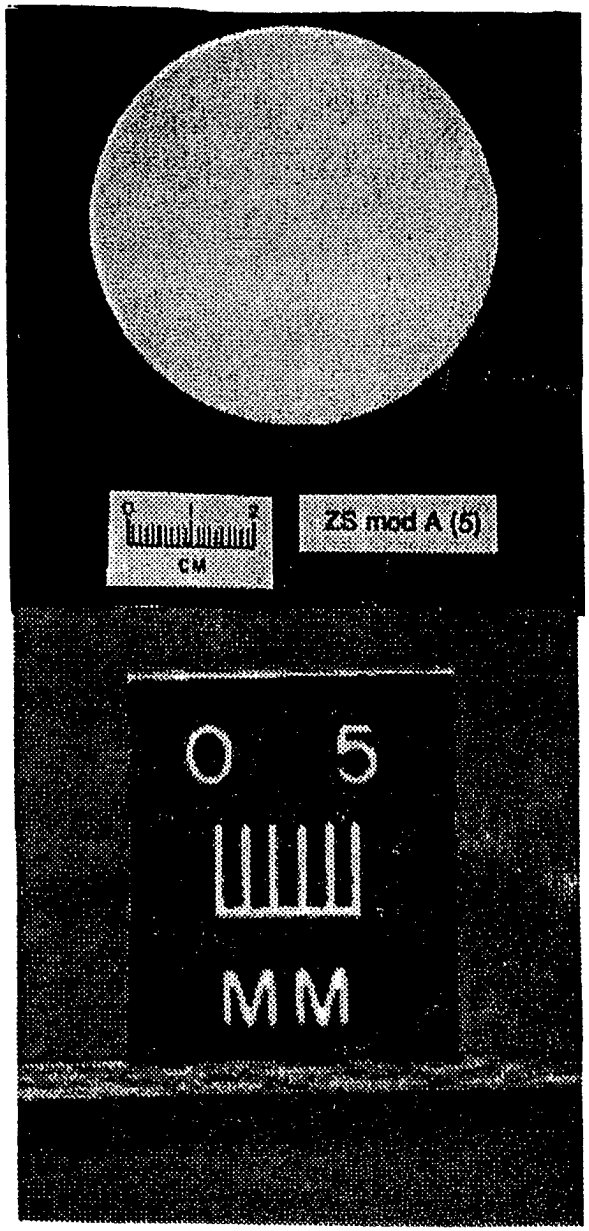
TENSILE STRENGTH OF SiC, Si-C-N and Si-C-B FIBERS AS A FUNCTION OF FIBER DIAMETER



TEMPERATURE EFFECTS ON FIBER TENSILE STRENGTH IN AIR

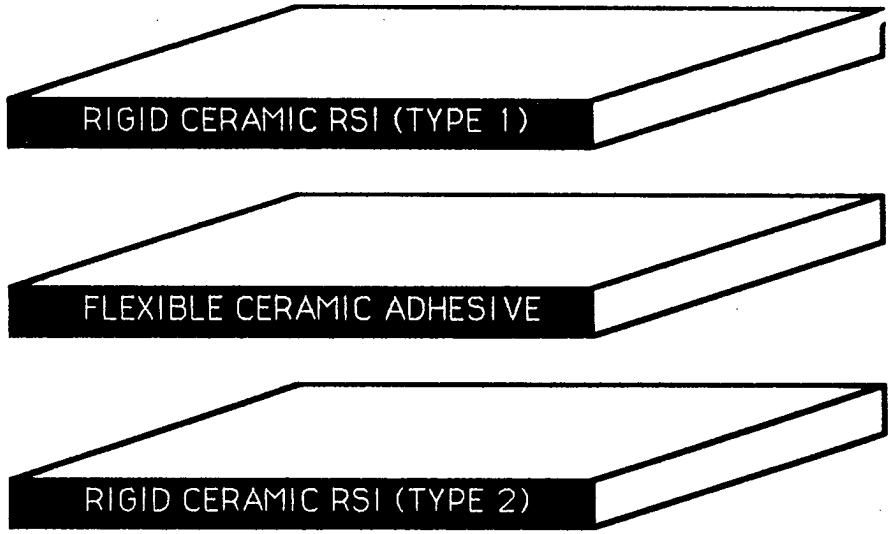


Ref. K.J. Wynne and R.W. Rice, Ann. Rev. Mater. Sci., 14, 297 - 334 (1984).



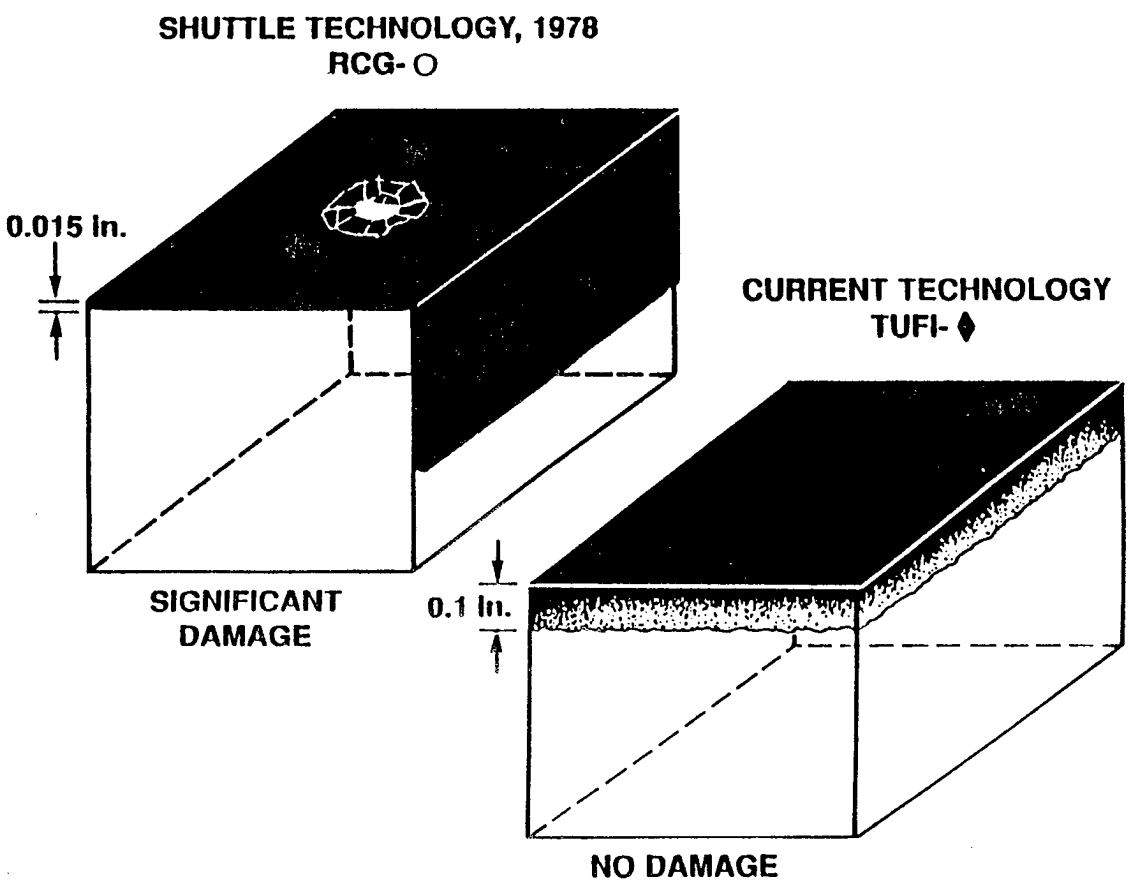
Cross-Section of ZS mod A(12)
3-Ply Laminate (Green State)

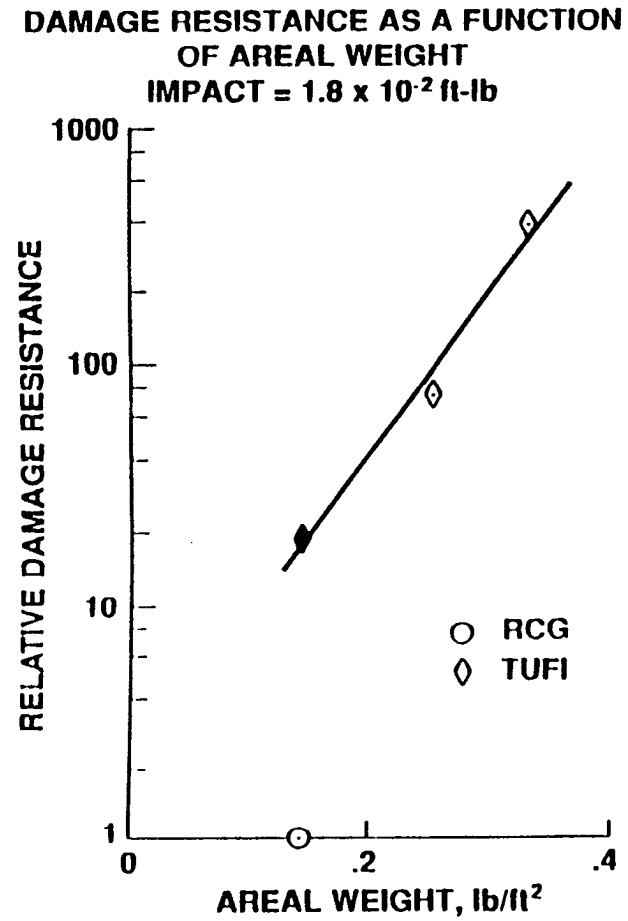
As illustrated below, two or more different RSI materials can be joined at room temperature and subsequently bonded together at the melting point of the tape cast ceramic. The thermal properties of the ceramic tape can be tailored to accommodate the thermal properties of the RSI by simply adjusting the ceramic composition of the tape.



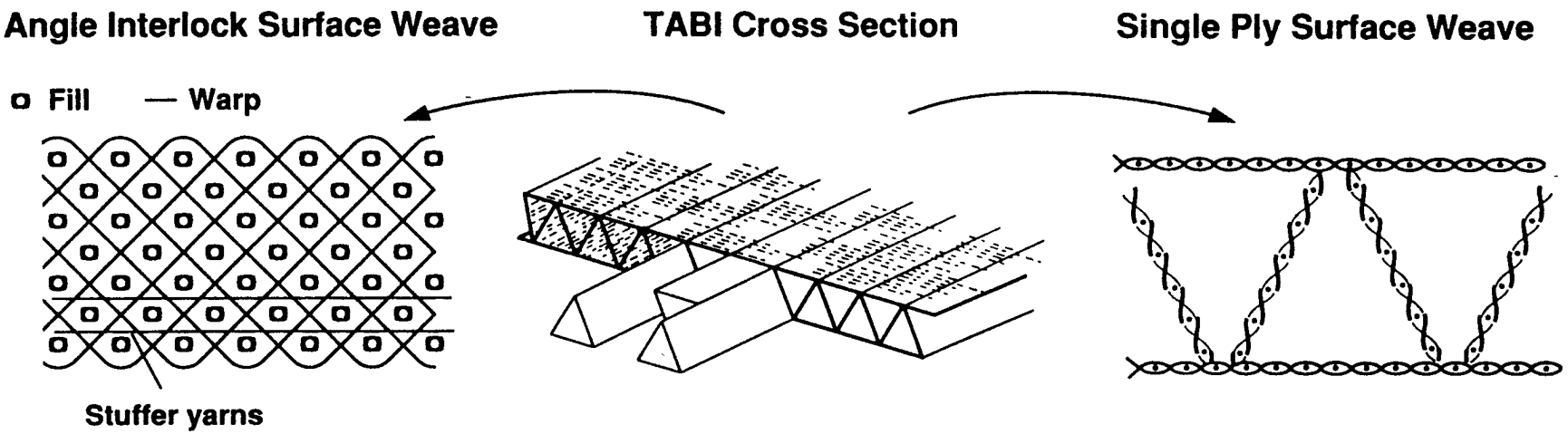
TAPE CASTING OF CERAMIC MATRIX COMPOSITES

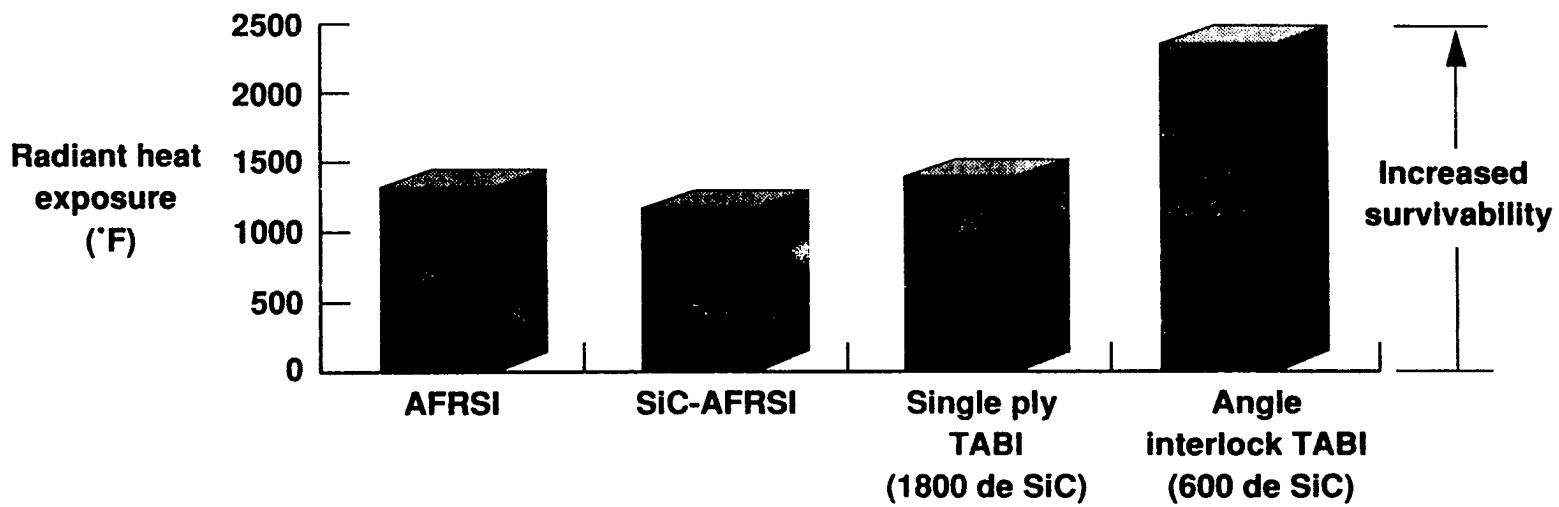
IMPACT RESISTANCE OF RSI COATING SYSTEMS





SURFACE TOUGHENING OF TABI TO AEROACOUSTIC ENVIRONMENTS



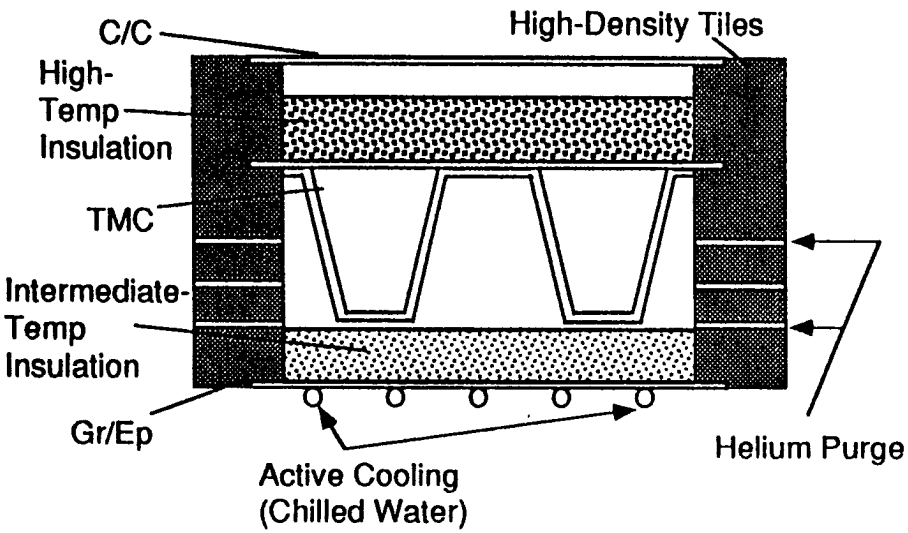


Aeroacoustic survival of flexible TPS after 600 sec at 170 dB (after exposure to radiant heat cycle)

NASP Insulation System Development (GWP#95)

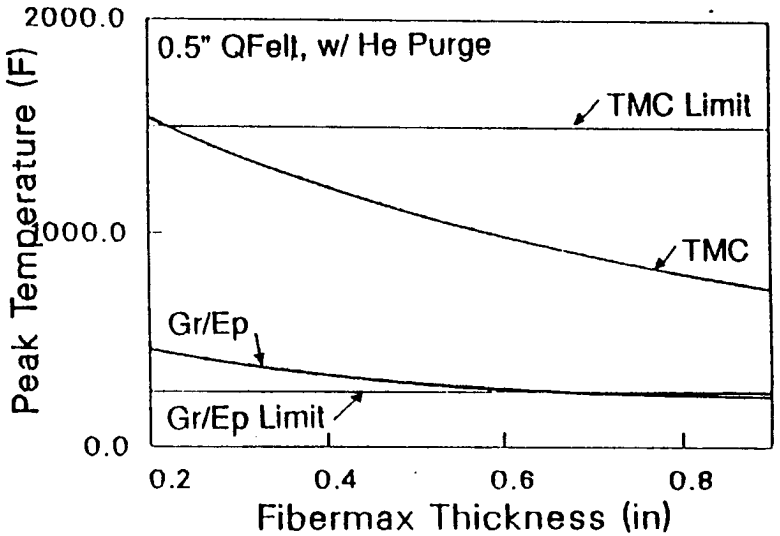
Acreage Insulation Arc-Jet Test Article

Simulates baseline structure without NPO structural attachments to assess insulation performances for acreage protection.



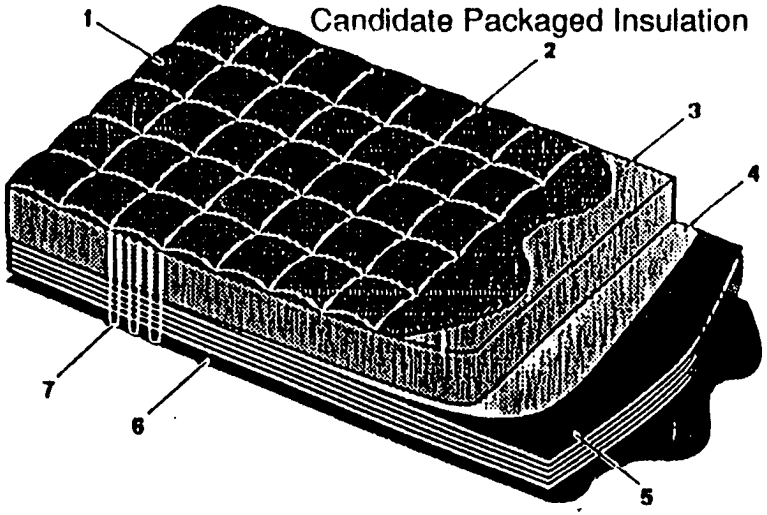
Thermal Performance of Baseline Batting

High-Temp: Fibermax
Intermediate-Temp: QFelt



Composite Flexible Blanket Insulation (CFBI)

Candidate Packaged Insulation

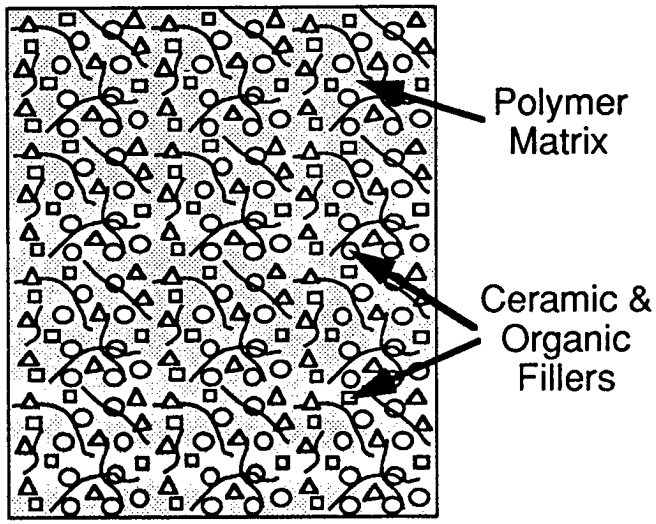


1 OML Fabric
2 OML Thread
3 Batting
4 Scrim Cloth
5 MLI Foil
6 IML Fabric
7 IML Thread

Development of New Structural Ceramic Ablators

30 to 50% Weight Reduction Compared to Traditional Ablators

Traditional Ablators*

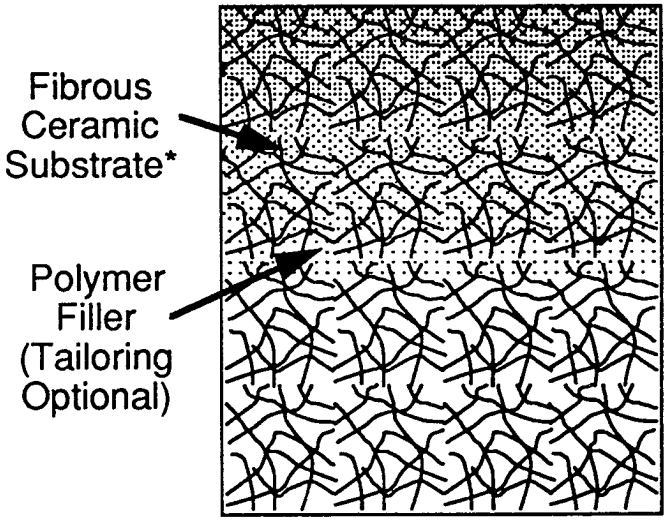


(*e.g. Avcoat 5026, SLA-561)

Pros:
- Mature technology
- Commercially available
- Flight tested

Cons:
- Little strength at high temperature
- Mechanical mass loss at high heat fluxes
- Tailoring of fillers very difficult

Structural Ceramic Ablators

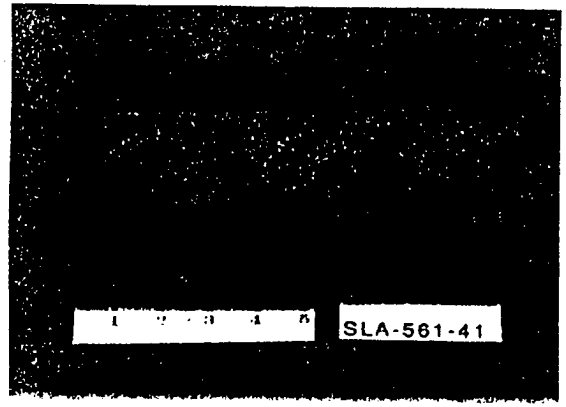


(*e.g. silica, carbon, alumina fibers)

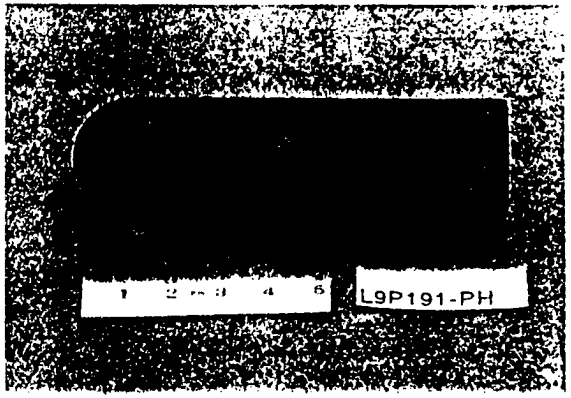
Pros:
- Good structural integrity at high temperature
- Avoids pyrolosis gas buildup and mechanical loss
- Filler can be tailored to optimize ablative and insulative performance

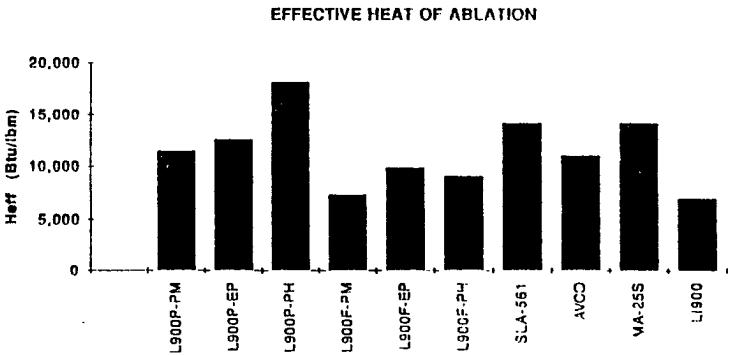
Cons:
- Material system under development
- Verified flight performance required

STRUCTURAL CERAMIC ABLATOR DEVELOPMENT



TEST CONDITION:
Q_{cw} = 830 Btu/ft^2-sec
P_{t2} = 0.081 Atm





MICROSTRUCTURE OF SLA-561

MICROSTRUCTURE OF LI900-PHENOLIC

NASA STRUCTURAL CERAMICS RESEARCH

OAST

SUMMARY

Diverse Array of Applications

- Aircraft Gas Turbines
- NASP
- Space Power Systems
- Thermal Protection Systems
- Rocket Engines

Enabling Propulsion Materials is Premier Program (>60% of Total FY 1993 Funding)

50% Funding Increase in FY 1993 to $28M w/66 Professionals

DARPA
STRUCTURAL CERAMICS PROGRAM

Presented to

INTERAGENCY COORDINATING COMMITTEE ON STRUCTURAL CERAMICS

By

WILLIAM S. COBLENZ
PROGRAM MANAGER - STRUCTURAL CERAMICS
DEFENSE SCIENCES OFFICE

13 MAY 1992

CERAMIC TECHNOLOGY INSERTION PROGRAM

- GOAL: DEMONSTRATE THE UPGRADE POTENTIAL OF ADVANCED STATE-OF-THE-ART STRUCTURAL CERAMICS IN FIELDED MILITARY SYSTEMS
- SELECTION OF CONTRACTORS
  - MILITARY BENEFITS: MISSION CAPABILITY, ACQUISITION AND MAINTENANCE COSTS....
  - SOUNDNESS OF THE TECHNICAL PLAN
  - SERVICE PROGRAM SUPPORT
  - VARIETY OF APPLICATIONS DEMONSTRATED
- MEASURE OF SUCCESS: SERVICE PROGRAM OFFICE FOLLOW ON FUNDING LEADING TO QUALIFICATION OF CERAMIC COMPONENTS

CERAMIC BEARING TECHNOLOGY PROGRAM

- GOAL: ENHANCE THE INDUSTRIAL TECHNOLOGY BASE CAPABILITIES FOR CERAMIC - HYBRID AND ALL CERAMIC BEARINGS TO BE USEI IN ADVANCED DoD SYSTEMS
- SELECTION OF CONTRACTORS (BAA, MARCH 1991)
  - BENEFITS FROM PROPOSED RESEARCH
  - TECHNICAL PLAN
- TECHNOLOGY CHOICES
  - DESIGN DATA BASE
  - NDE METHODS
  - BALL FINISHING
  - RACES
  - NEW MATERIALS DEVELOPMENT

PRE-COMPETITIVE CONSORTIA OR PARTNERSHIPS

BACKGROUND: DARPA Authorized (10 U.S.C. 2371) to enter into "cooperative agreements or other transactions". DARPA Agreements Authority is potentially applicable where contracts or grants are not appropriate instruments. Congress has directed DARPA to support pre-competitive generic technologies with dual-use applications through consortia. Congressional funding provided in FY 91 +92

CRITERIA FOR CONSIDERATION:

1. The technology, when developed, will fulfill an important military need and be identifiable with the DoD Critical Technologies Plan

2. The technology must fall within a DARPA major thrust area.

3. The consortium concept (business strategy) is reasonably mature, with expressed interest from the private sector. Private sector cost sharing should be greater than 50%

4. The consortia should be self-sustaining after the initial government investment.

WHAT IS PRE-COMPETITIVE TECHNOLOGY?

We know that competition is key to rapid technological advancement. How then can competitors work productively together? (SEMATECH, MOSIS, Japan Fine Ceramics Center, ATP)

CERAMIC FIBER PRECOMPETITIVE CONSORTIA

OBJECTIVES

- Develop ceramic fibers for reinforcing ceramic Matrix (and possibly metal matrix) composites
- Ceramic matrix composite use temperature 2500 to 3000 ^{o}F
- Ready for commercial scale-up in 3 to 5 years

APPROACH

- Seven major U.S. gas turbine engine companies form a not-for-profit consortium
- Research funding will be from consortium partners DARPA and Air Force
- Consortium will fund developers of ceramic fibers

CHARACTERISTICS OF CFC

- Not-for-profit
- Board of directors from engine companies (ex officio representatives from government)
- CFC awards contracts to U.S. Industries and Universities from competitively selected proposals
- Participation of government program managers in contract selection/project management

MEMBERS

- Allied-Signal Aerospace Company-Garrett Engine
- Teledyne CAE
- Williams International
- Allison Gas Turbine - General Motors Corporation
- Textron Lycoming
- General Electric Aircraft Engines
- Pratt & Whitney

HIGH TEMPERATURE STRUCTURAL MATERIALS PROGRAM

- FIBER INTERFACE COATING TECHNOLOGY
- CERAMIC FIBER TECHNOLOGY MONITORING
- CERAMIC FIBER PROCESSING
- COMPOSITE FABRICATION

FIBER INTERFACE COATING TECHNOLOGY

- Corning Glass: Sheet Silicate Coated Fibers - Glass Ceramic Matrix Composites Oxidatively Stable Debond Layers (K. Chyung) {co-funded with ONR}
- General Atomics: Ceramic Fiber Coating Development (H. Streckert) Continuous Coating of Nicalon® fiber tows with gas phase deposition of BN Liquid phase coating; Yttria, Zirconia, BN, Hafnia
- Pratt & Whitney: Fiber Coating by Sputtering for High Temperature Composites Identification of coatings which promote debonding in intermetalic and ceramic matrix composites; Yttria coated Nb fiber TiAl matrix (M.L. Emiliani)
- U. Florida: Innovative Processing of Composites for High Temperature Applications (R. Abbaschian) 'In-Situ' coating process (alumina coated Nb in Nb-3Al matrix; Atomic Layer Deposition Of TiC on alumina
- U. California - Santa Barbara (A. Evans) Fugitive phases for oxide-oxide systems; Decohesion at oxide-metal interfaces

TECHNICAL EVALUATION OF CERAMIC FIBERS

- Institute for Defense Analysis (W. Hong)

  Collection and critical evaluation of test data for ceramic fibers
  Manufacturing Process, Strength vs. Temp., Creep Rates, etc.

- Georgia Tech Research Institute (T. Starr)

  Fiber Characterization: HPZ, Tyrano, BN, SiNC from France, 3M alumina

  Physical Properties [density, filament count, diameter}
  Composition and Phase Analysis
  Strength (RT - 1400 ^{o}C), Modulus, thermal expansion
  Dielectric properties

- Rensselaer Polytechnic Institute (S. Sternstein)

  Creep and thermal expansion measurements (RT - 1500 ^{o}C)
  Self-Resistance heating of SCS-6 SiC fibers

CERAMIC FIBER PROCESSING

- Case Western Reserve: (A. Heuer)
  Investigation of Single Crystal Sapphire (MIT Laser Float Zone)
  Tensile Strength Dip at ~400°C, strength reduced ~50%
  Twinning without slip vs. Environmental effects?

- 3M: Part of Bill Barker's Metal Matrix Composite Model Factory
  Sol-Gel derived alpha alumina fiber

- NIST: (C. Handwerker and L. Cook)
  Seeded secondary grain growth to produce alumina fibers with crystal length to fiber diameter ratios >20, Single crystal properties at polycrystalline prices!
  Single Crystal alumina fibers via directional solidification of a Cryolite-alumina eutectic followed by matrix leach

- GE / Saphikon: Part of Bill Barker's IPM Program
  Improved Quality and Reduced Costs through IPM, Sapphire pulled from the melt via edge-defined film growth

- University of Florida: Sintered Mullite fibers via transient viscous phase sintering and low oxygen polymer derived SiC fibers. (M. Sacks)

COMPOSITE FABRICATION

- Lanxide: SiC reinforced alumina combustor plates and metal matrix electronic packaging (T. D. Claar) also Ceramic Armor for Desert Storm
- Naval Research Laboratory: Fiber Composite Surface Laminates (D. Lewis III)
- Northwestern University: Thermal Gradient CVI with microwave heating Alumina-alumina with debond layers, and SiC fiber with either SiC or aluminosilicate matrix (D. Lynn Johnson)
- University of Florida: Transient viscous sintering of mullite matrix composites Infiltration Processing (M. Sacks)
- University of Michigan: Compaction of coated 'green' fibers followed by pressureless sintering (J. Halloran)
- Williams International: Evaluation of vendor supplied combustor liners and plates for WR 24-7 Engine with collection of material engineering property data base (W. Fohey)
  Vendors: Lanxide, Amercon, Kaiser Aerotech

CERAMIC FIBER PROCESSING CONTINUED

- University of Illinois: Part of Rich Loda's Program (J. Economy)
  Nitriding of melt spun boria fibers to produce low cost BN fibers

MECHANISM-BASED DESIGN OF COMPOSITE STRUCTURES

Research & Development Expenditures For High Temperature Structural Composites By DOD, NASA, and DoE of ~$70M/year

ENABLING TECHNOLOGY

Tactical Fighter
Commercial / Transport (Subsonic)
High Speed Civil Transport
Hypersonics (NASP)

Thrust-To-Weight
Thrust Specific Fuel Consumption
Emissions and Noise
Weight and Thermal Management

DESIGN RELATED REQUIREMENTS: The high degree of anisotropy in properties, and their relationship to fiber architecture for ceramic and metal matrix composites, combined with the lack of design experience or established design methodology have hindered the efficient utilization of these newly developed materials.
A new generation of designers capable of utilizing advanced design methodologies will be needed to integrate manufacturability and performance of these advanced high-temperature composite materials.

NEW URI TOPIC
MECHANISM - BASED DESIGN OF COMPOSITE STRUCTURES

- OBJECTIVE: Develop A Mechanism-Based Design Approach For High Temperature Composite Structures Leading To A Computer Aided Design Capability

- APPROACH: Utilize Formalisms Derived From Micromechanics Descriptions Of The Physical Mechanisms Of Creep, Fracture, Fatigue, Etc., As The Basis For The Numerical Procedures

  Experimental Technique Development For Required Material Property Measurements

  Processing Capability Consistent With The Design Strategy

  Development of Sensors Which Provide Information About The Material State, Both During Processing and In Use

- NEW PARADIGM: From Material Replacement to Designing For Functionality And Manufacturability (Concurrent Engineering)

Interagency Coordinating Committee on Structural Ceramics

May 1992

U.S. BUREAU OF MINES
Division of Minerals and Materials Science

Materials Research

CONSERVATION
Sara A. Dillich
FTS 501-9282

SUBSTITUTE MATERIALS
Garrett R. Hyde
FTS 501-9281

STRUCTURAL CERAMICS RESEARCH
($1000)

TUSCALOOSA RESEARCH CENTER, Tuscaloosa, Alabama
Research Supervisor: vacancy

o <u>Processing of Advanced Ceramics</u> $1000

Effects of processing parameters on properties. Focus is on a single processing operation

- Microwave processing
  -- MIP Processing of Advanced Ceramic Powders
  -- Developed a technique for high T measurement of dielectric properties of advanced ceramics

- Organometallic Precursors for Advanced Ceramics
  -- Synthesis of SiC/AlN Ceramics by Chemical Routes

- Laser Processing (Melting) of SiC - wt University of Alabama

o <u>Structure and Properties of Advanced Ceramics</u> $600

Relationships between composition, microstructure/properties and performance in severe service environments. Focus is on relationships between treatment and performance of finished material.

- Alumina oxycarbide substitute for flake graphite (and chromite) in refractory Brick

- Titanium-aluminum oxynitride-spinel composites - high MOR
  -- composition and heat treatment effects on strength and electrical properties

- Corrosion of Ceramic Materials $250

  Mechanisms of corrosion in advanced ceramic materials in severe corrosive environments up to 1000 C

ALBANY RESEARCH CENTER, Albany, Oregon
Research Supervisor: Richard Walters

- Fundamentals of Wear and Machining $345

  Erosion of monolithic ceramics -- investigation of the mechanisms of material removal and how they are affected by changes in the scale of the damage relative to the scale of the microstructure

- Micromechanisms of Compressive Fracture in Heterogeneous Systems $275

  Analytical study of the role of interfaces between reinforcing particulates and brittle matrices under multiaxial compressive loading

IDAHO NATIONAL ENGINEERING LABORATORY, Idaho Falls, Idaho

- Fracture Mechanics of Interfaces in Heterogeneous Microstructures $242
  J.S. Epstein, Principal Investigator

  Real time (Moire interferometry) study of mechanisms of interface fracture in heterogeneous microstructures

- <u>Oxynitride Based Ceramic Composites</u> $417
  M.H. O'Brien, Principal Investigator

  Synthysis and characterization of composites based upon oxynitride matrix compositions
  -- Oxynitride sintering of Si_3N_4

  -- Reaction sintered composites
     TZP reinforced SiAlON
     SiC-SiCAlON composite fiber, fiber drawing

- <u>Noncontacting NDE for Materials Characterization</u>
  K.L. Telschow, Principal Investigator

  Ceramics Component: $60
  -- Ceramic Densification During Microwave Sintering

POSSIBLE NEW DIRECTIONS FY 1993

TUSCALOOSA RESEARCH CENTER, Tuscaloosa, Alabama

- Design and Synthesis of functionally layered nanocomposites by intercalation of layered structures e.g., clay, graphite

- Development of a Gas-Fluidized Bed Turbomill in which grinding media are fluidized with a gas (air) instead of liquid

ARO - MATERIALS SCIENCE DIVISION

ICCSC FY 1992

ARMY RESEARCH OFFICE

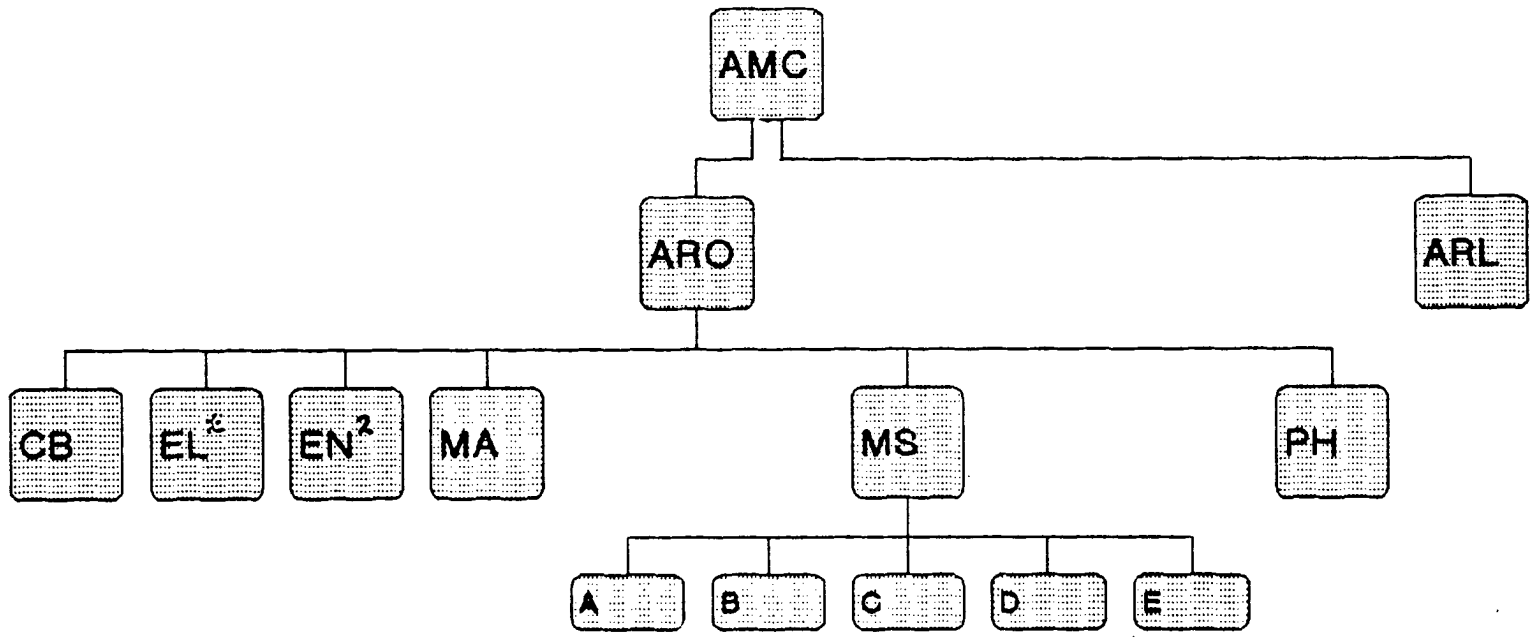
Presentation

13 May 1992

Wilbur C Simmons, PhD
(919) 549-4329
dsn/fax; 832-4310

92.05.08 WCS

ARMY RESEARCH OFFICE



92.05.09

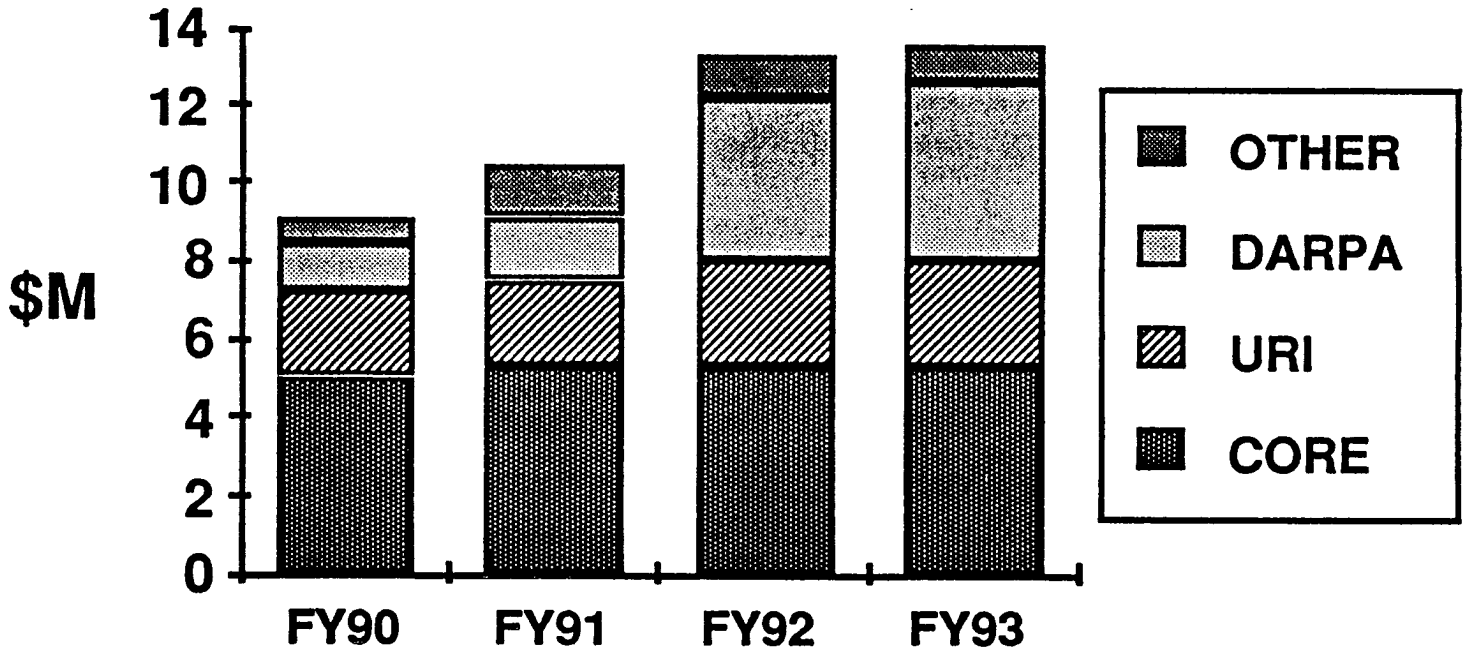
MATERIALS SCIENCE DIVISION PROGRAM RESPONSIBILITIES

Division Director Dr. Andrew Crowson

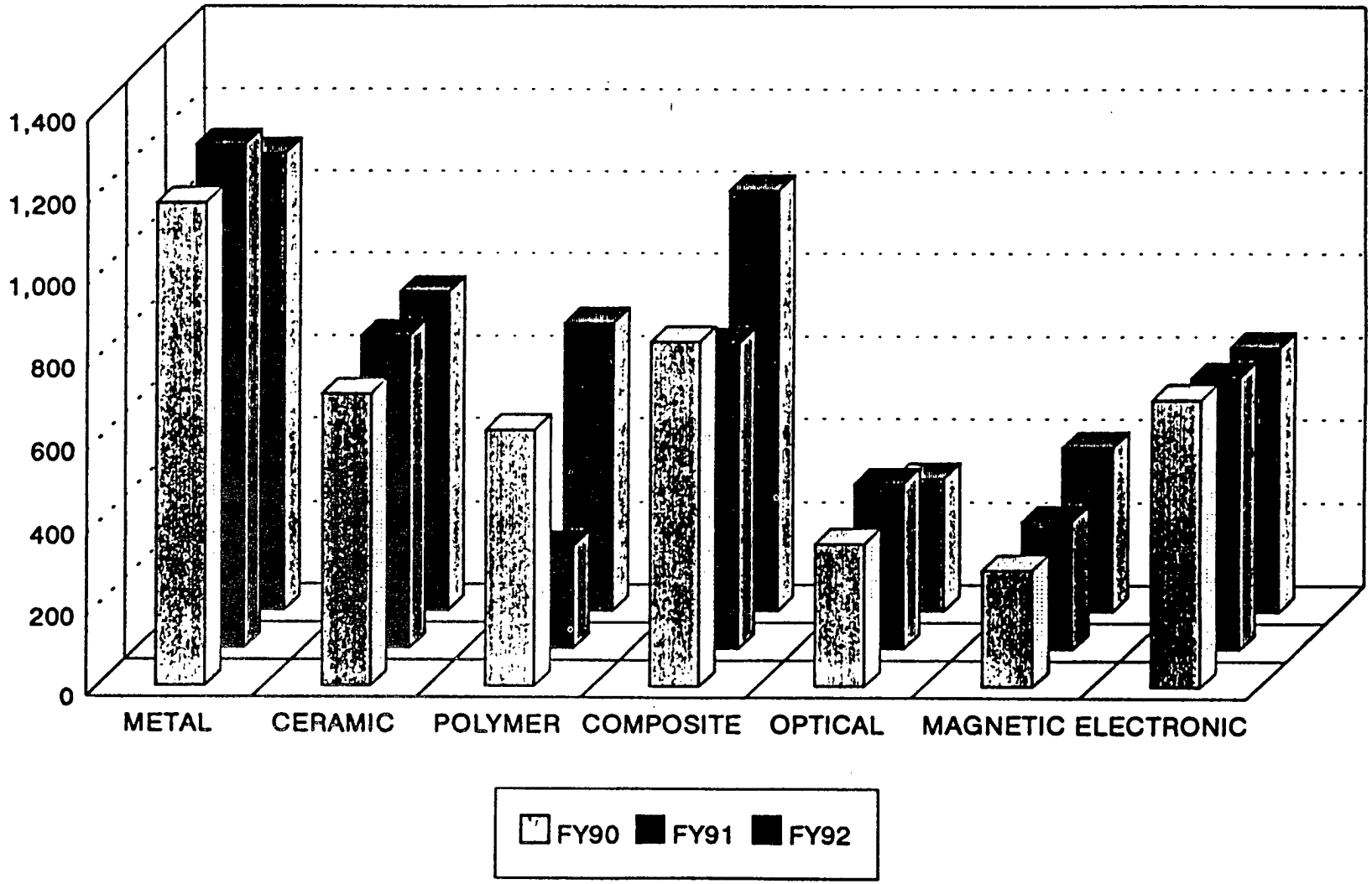
Associate Director Dr. John Prater

A. Degradation, Reactivity & Protection	Dr. Robert Reeber
B. Mechanical Behavior	Dr. Wilbur Simmons
C. Synthesis & Processing	Dr. Edward Chen
D. Physical Behavior	Dr. John Prater
E. Dynamic Behavior	Dr. Kailasam Iyer
Far East Office	Dr. Iqbal Ahmad

MATERIALS SCIENCE DIVISION BUDGET ($M)



ARO MS DIVISION BUDGET
BY MATERIAL CLASS



FY 1992
31 March 1992

URI PROGRAMS

- CENTER FOR MANUFACTURING SCIENCE (U of Delaware)
  Establishment of a science base for advanced, automated manufacturing of thick section polymer matrix composites

- CENTERS FOR ULTRADYNAMIC PERFORMANCE MATERIALS (UCSD/BROWN)
  Enhanced understanding of the response of materials to high rates of loading, impingement of energy at high densities and rates and to shock waves

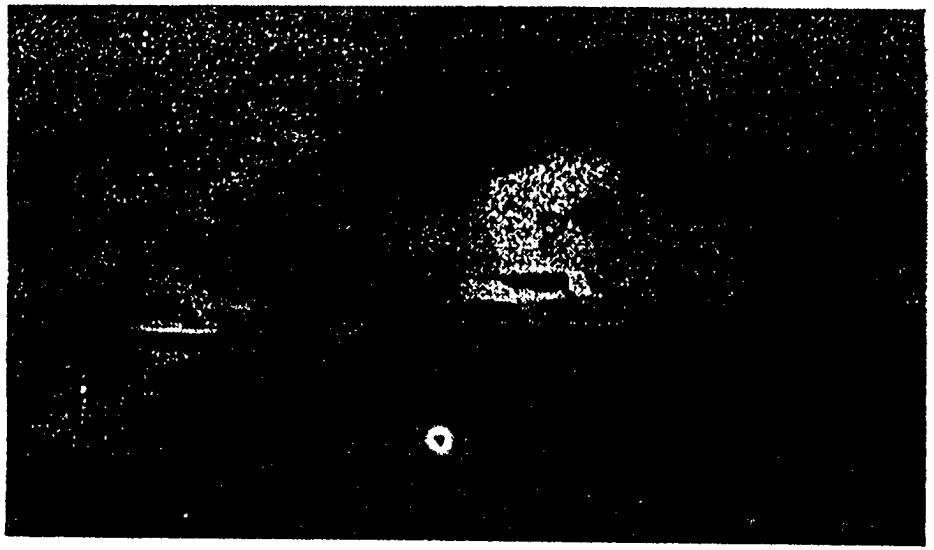
- BIOMIMETIC PROCESSING OF MATERIALS (CASE WESTERN/U OF WASHINGTON/PRINCETON)
  Fundamental understanding of relationships between biological processes and material performance in order to synthesize new advanced composites

- SMART MATERIALS AND STRUCTURES
  Multi-disciplinary efforts on promoting fundamental research on materials and structures which have embedded sensor, control and response mechanisms capable of sensing and responding to a stimulus in an appropriate fashion

DOD/ARMY URI PROGRAM

DYNAMIC BEHAVIOR OF BRITTLE MATERIALS

(BROWN UNIVERSITY)



OBJECTIVE:

- PROVIDE THE SCIENCE BASE FOR THE QUANTITATIVE UNDERSTANDING OF THE BEHAVIOR OF "BRITTLE" MATERIALS UNDER CONDITIONS OF IMPACT AND PENETRATION.
  - –TRACTABLE EXPERIMENTS
  - –POST-TEST CHARACTERIZATION
  - –PHYSICS-BASED MODELS

RESEARCH:

- CONTROLLED HIGH STRAIN RATE INSTRUMENTED RECOVERY EXPERIMENTS FOR SHOCK AND STRENGTH PROPERTIES OF CERAMICS.
- MODEL EXPERIMENTS TO UNDERSTAND THE BEHAVIOR OF MICROSCOPICALLY HETEROGENEOUS "BRITTLE/DUCTILE" COMPOSITES.
- MICROMECHANICS-BASED ANALYTICAL MODELS FOR INELASTIC DEFORMATION, INSTABILITY, AND FLOW OF PULVERIZED "BRITTLE" MATERIALS UNDER PRESSURE

RETURN ON INVESTMENT:

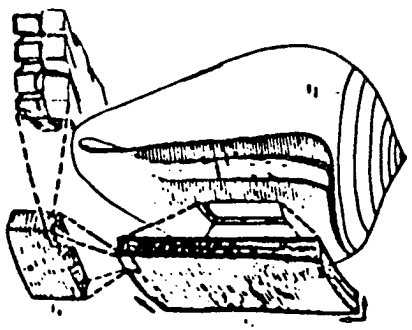
- IDENTIFICATION OF FAILURE MECHANISMS
- IMPROVED VULNERABILITY ANALYSES
- GUIDELINES FOR OPTIMIZING MATERIAL DESIGN
- PHYSICS-BASED MODELS IN CODES (IMPROVED ARMOR DESIGN)
- LAB/CENTER PARTICIPATION
- TRAINING OF FUTURE DOD/INDUSTRY SCIENTISTS/ENGINEERS

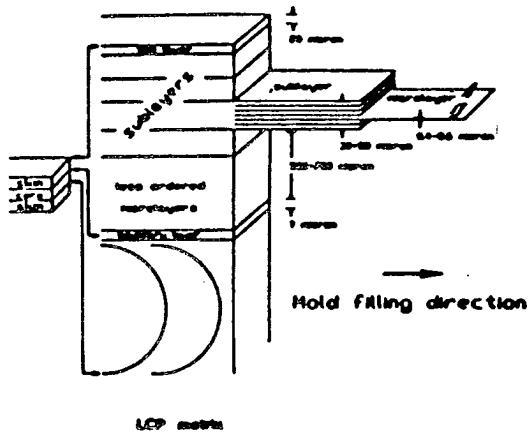
31 March 1992

ARO - MATERIALS SCIENCE DIVISION

URI: BIOMIMETIC PROCESSING OF MATERIALS

CWRU & UW & PRINCETON





OBJECTIVE:

ESTABLISH SCIENCE BASE FOR THE SYNTHESIS OF NEW ENGINEERED MATERIAL SYSTEMS AT THE MOLECULAR & NANO-CRYSTALLINE LEVEL FOR NANO-ELECTRONIC DEVICES, STRUCTURAL MATERIALS, AND FUTURE SMART MATERIALS.

RESEARCH

- LEARN LESSONS FROM NATURE
  (STRENGTH & TOUGHNESS)
- MIMIC BIOLOGICAL STRUCTURES
  (KNOWN MATERIALS SCIENCE & TECH)
- BIODUPLICATE MATERIALS & STRUCTURES
  (CONTROLLED NUCLEATION & GROWTH ON PROTEINS & LBF)
- MOLECULAR/NANO-LEVEL ASSEMBLY

RETURN ON INVESTMENT

- NEW PATHWAYS FOR MAKING HIGH PERFORMANCE MATERIALS
- NEW ELECTRO-OPTIC DEVICES
- BETTER ARMOR PROTECTION
- MULTIFUNCTIONAL "SMART" MATERIALS
- ARMY LABORATORY INVOLVEMENT
- GRADUATE TRAINING OF S&E'S

SURFACE MODIFICATIONS/COATINGS PROGRAM FY 1992

ION BEAM MIXING/IMPLANTATION

- ION BEAM MIXING of CERAMICS
  (Mayer - Cornell Univ.)
- PLASMA ENHANCED ION BEAM MIXING TiN in STEEL
  (Conrad - Univ. Wisconsin, Madison)
- CORROSION INHIBITION PLASMA ENHANCED ION BEAM MIXING OF N IN STEEL (Roth - Univ. Tenn., Knoxville)
- HIGH CURRENT METAL ION IMPLANTATION
  (Brown - Lawrence Berkeley Lab)
- ION IMPLANTED REFRACTORY COATINGS
  (Bunker - Implant Science) +

March 31, 1992

NOVEL CHARACTERIZATION of SURFACES/INTERFACES

SPECTROSCOPY/SPECTROMETRY

- Photoelastic Modulated Laser Enhanced IR Spectroscopy (White - Univ. Mo.)
- Positron Studies of Polymer Free Volume (McGervey - Case Western Reserve)
- Laser Brillouin Spectroscopy (Chelluri - IAP Research Inc)
- Chemical Luminescence (Bray - Texas Research Institute)
- Scanning Tunneling Spectroscopy (Tsong - Arizona State)

March 31, 1992

PROTECTIVE COATINGS
FY 1992 ACTIVE PROJECTS

- Metal Conversion Coatings
  (Sugama - Brookhaven)
- Plasma Sputtered Refractory Oxides
  (Aita - Univ. Wisconsin, Milwaukee)
- CVD Refractory Metal Laminates
  (Bilello - Univ. Michigan)
- Electrically Conductive Polymer Films
  (Barkey - Univ. New Hampshire)

ARO-MS-B: MECHANICAL BEHAVIOR
STRENGTH AND TOUGHNESS

- CERAMICS & CMCs
  - * Laser Induced Controlled Flaw testing in Ceramics — Chou/Lehigh
  - * High-Toughness Glass-Ceramics Through Transformation Toughening — Tomozawa/RPI

- POLYMERS & PMCs
  - * Fracture Behavior of Ionomers and Ionomer Blends. — Hara/Rutgers

- ALL COMPOSITES
  - * Dependence of Strength & Toughness on Interfacial Strength — Gupta/Dartmouth
  - * Mechanical Properties of Materials by Thermalwave Imaging — Favro & Thomas/ Wayne State U

ARO-MS-B: MECHANICAL BEHAVIOR
SUPERPLASTIC PHENOMENON

- Superplasticity in Fine-Grain Ceramic Composites — Nieh/Lockheed
- Nanograin Superplastic Processing of Ceramics — Raj/Cornell
- Superplastic Ceramics — Sherby/Stanford

(Conrad/NCSU)

POWDER SYNTHESIS & CONSOLIDATION

• Integrated Synthesis/Processing of SiC and AlN	Kingon/NCSU
• SHS Processing of Functionally Gradient Materials	Stangle/Alfred
• Heavily Deformed In–Situ Composites	Courtney/Virginia
• Two–Phase Nanocrystalline Materials	Baker/Dartmouth
• AlN: A New Low Cost Ceramic Armor	Logan/Georgia Tech
• Processing/Microstructural Effect on Tungsten Heavy Alloy Performance	German/Penn State

NONEQUILIBRIUM MATERIALS

• Solidification Processing of TaW/W Refractory Metal Base Alloys	Lavernia/UC Irvine
• Synthesis/Properties of RS Alloys with Quasiperiodic Structures	Poon/Univ Virginia
• Processing of High Temperature Intermetallics (DARPA)	Perepezko/Wisconsin
• Solidification Structure Synthesis in Undercooled Liquids	Perepezko/Wisconsin

HIGH RATE LOADING PROGRAM
MS DIV., USARO

TITLE/PROPOSAL #	INSTITUTION/PI	
1. DYN. BEH. OF BRITTLE MATLS	BROWN UNIVERSITY/FREUND	DoD/NEW START
2. DYN. BEH. OF DUCTILE MATLS	UCSD/NEMAT-NASSER	DoD/NEW START
3. DYN. MATL RESPONSE	BROWN UNIVERSITY/CLIFTON	ARO /JOINTLY WITH EN
4. SHEARING DEF. IN W ALLOYS	JOHNS HOPKINS/RAMESH	ARO
5. INELASTIC DEF. IN CERAMICS	WASH. ST./GUPTA	ARO/NEW START
6. MODELLING OF GRANULAR FLOW	UCSD/NEMAT-NASSER	DARPA/NEW STA.
7. DYN. BEH. BRITTLE MATERIALS	CALTECH/AHRENS	DARPA+BH57/NEW JOINTLY WITH EN DIV

INNOVATIVE PROCESSING

• Intelligent Synthetic Polymers	Calvert/Univ Arizona
• Shock Induced Reactions in Ceramics	Johnson/Cal Tech
• High Performance Carbon Fibers Using Supercritical Fluid Extraction (DARPA)	Thies/Clemson Univ

U.S. ARMY RESEARCH OFFICE
MATERIALS SCIENCE DIVISION

FUTURE THRUSTS

- Manufacturing and Processing Science
- Composite materials
- Smart Materials
- Hierarchical Structures
- Dynamic Behavior of Materials
- Surface Modification
- Resilient Structures

92.05.11

WORK UNIT TITLE: Materials - Ceramic Matrix Composites

PE/PRJ/TSK/WP: 61102/AH45/B/PRD1002

CONTRACTOR: In-House

POC/PHONE: R. Bhatt (216) 433-5513

OBJECTIVE/PROBLEM STATEMENT:

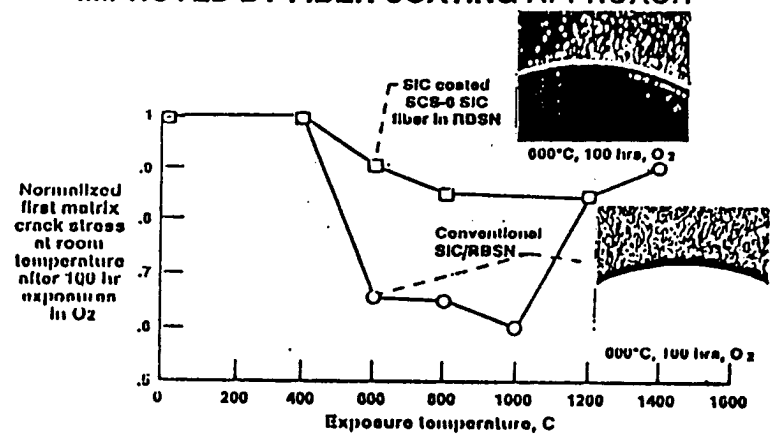
- Determine and optimize the performance of SiC/Si_3N_4 composites for strength, toughness, thermal-oxidative stability, and fabrication capabilities

DELIVERABLES:

- Alter composite microstructure by altering fiber, interface, and matrix
- Correlate microstructure with performance and theory

70 K Away

OXIDATIVE STABILITY OF SIC/RBSN COMPOSITES IMPROVED BY FIBER COATING APPROACH



PROGRAM SCHEDULE

TASKS	92	93	94	95
HIPing studies				
Fabrication and characterization				
Impact behavior				
Creep behavior				
Oxidation protection studies				

ACCOMPLISHMENTS:

FY 91:
- Determined strength stability of SCS-6 fibers in argon environment
- Studied densification behavior of SCS-6/RBSN composites in presence of sintering additives
- Explored NDE techniques for analyzing as-fabricated flaws and for assessing damage in SiC/RBSN composites
- Investigated oxidative stability of coated SiC/RBSN composites

PLANS:

FY 92:
- Develop methods for fabricating 2-D and 3-D SiC/RBSN composites
- Study reaction kinetics between silicon nitride matrix mixed with sintering additives and commercially available small diameter SiC fibers
- Measure elevated temperature strengths of SiC/RBSN and HIPed SiC/RBSN composites

WORK UNIT TITLE: Structures - Ceramic Fracture Mechanics Research

PE/PRJ/TSK/WP: 61102/AH45/B/PRD1002

CONTRACTOR: In-house

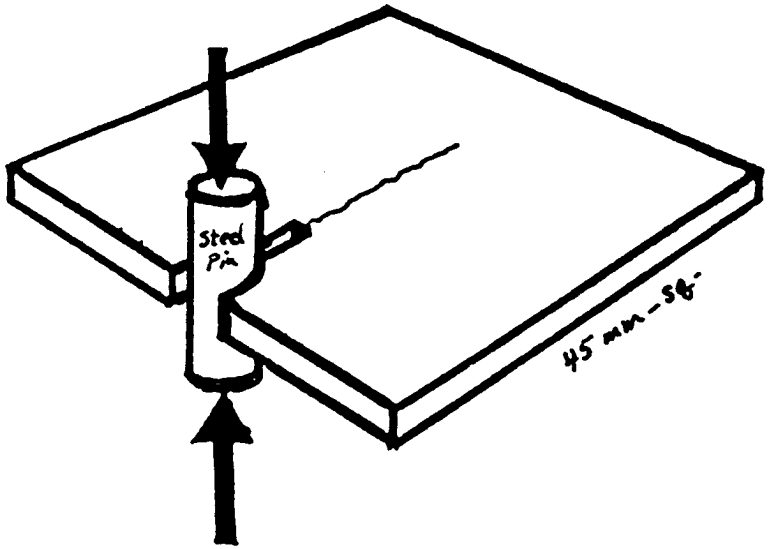
POC/PHONE: D. Brewer (216) 433-3304

OBJECTIVE/PROBLEM STATEMENT:

- Collect ceramic fracture behavior data suitable for structural analysis code validation
- Develop standard testing procedure for structural ceramics

DELIVERABLES:

- Analytical tools: Improved structural ceramic life prediction model through increased understanding of failure behavior and damage accumulation



PROGRAM SCHEDULE

TASKS	90	91	92	93	94	95
Specimen geometry and loading techniques						
Fatigue crack growth studies						
Develop controlled environment testing capability						
Test at high temperature						
Life prediction model and code validation						
Model tuning, incorporation into design criteria						

ACCOMPLISHMENTS:

FY 90:
- Specimen design finalized
- Loading method finalized

FY 91:
- Fatigue crack growth studies conducted in NC-132
- Specimen/Load train interaction investigated
- Environmental testing equipment fab underway

PLANS:

FY 92: - Demonstrate environmental capability

FY 93: - Demonstrate high temperature capability

FY 94: - Life prediction model and code development

FY 95: - Model tuning and incorporation into structural design criteria

HEAT RELEASE PHENOMENA IN LOW HEAT REJECTION ENGINES (TACOM)

STATUS: (2QTR FY92)

-
-
- FULLY COOLED ENGINE BASELINE RUNS COMPLETED
-
- LHR ZIRCONIA COATED PISTON TESTS COMPLETED
- SEAL COATED ZIRCONIA PISTON TESTS INITIATED

DR. WALTER BRYZIK
ADVANCED PROPULSION SYSTEMS
USATACOM

CERAMICS

PROPOSAL NUMBER	P. I. INSTITUTION	TITLE	FY92 FUNDS: ARO/ OTHER/ TOTAL	SUBFIELD
26427MS	Carolyn R. Aita Wisc, Univ-Milwaukee	Process Parameter/Growth Environment/ Film Property Relationships/for Reactive Sputter Deposited Metal---	$0 $0 $0	A
26806MS	James W. Mayer Cornell Univ	Ion Beam Modification of Ceramics: Mechanical Properties and Structure	$85,997 $0 $85,997	A
27572MS-SM	Louis Cartz Marquette Univ	Actuators of Mica Layer Structures	$0 $79,405 $79,405	A
29037MS	I. S. T. Tsong Arizona State Univ	Scanning Tunneling Microscopy & Spectroscopy of Bonding Mechanisms Between Advanced Ceramics & Thin Metal--	$85,060 $0 $85,060	A
29577MS-YIP	Lorraine Falter Francis Minnesota, U-Minneap	Processing & Characterization of Porous Oxide Coatings	$0 $0 $0	A
29807MS-AAS	James W. Mayer Cornell Univ	Ion Beam & Laser Modification of Zirconia	$0 $0 $0	A
30335MS-SB2	Stephen N. Bunker Implant Sciences Corp.	Strongly Adherent Ceramic Surface Layers by Ion Implantation	$0 $0 $0	A
30460MS-SBI	Todd E. Schlesinger Hittman Materials & Medical Comp.	Solid Lubricants for Ceramics	$0 $0 $0	A
30461MS-SBI	Stephen N. Bunker Implant Sciences Corp.	Single Crystal Diamond Films	$0 $0 $0	A
30462MS-SBI	John L. Lawless Space Power, Inc.	cBN Insulator for TFE Trilayer (SBIR-SPI-156)	$0 $0 $0	A

CERAMICS

PROPOSAL NUMBER	P. I. INSTITUTION	TITLE	FY92 FUNDS: ARO/ OTHER/ TOTAL	SUBFIELD
24583MS	Dinesh K. Shetty Utah, Univ	Stress State Effects on Strength & Fracture of Partially-Stabilized Zirconia	$0 $0 $0	B
26673MS-A	Angus I. Kingon North Carolina State Univ	Integrated Synthesis & Post-Processing of SiC and AlN	$100,000 $0 $100,000	B
26747MS-A	Rishi Raj Cornell Univ	Nanograin Superplastic Processing of Ceramics	$0 $0 $0	B
28549MS	Y. T. Chou Lehigh Univ	Laser-Induced Controlled Flaw Testing in Ceramics	$112,553 $0 $112,553	B
28761MS	Oleg D. Sherby Stanford Univ	Superplastic Ceramics	$120,000 $0 $120,000	B
28780MS	Robert L. Thomas Wayne State Univ	Thermal Wave Imaging & Characterization of Solids	$0 $0 $0	B
29934MS-AAS	Angus I. Kingon North Carolina State Univ	Plasma Synthesis of Nanocrystalline Materials	$0 $0 $0	B
30349MS-URI	Ilhan A. Aksay Case-Western Reserve Univ	Layered Nanocomposites by Biomimetic Processing	$0 $0 $0	B
25526MS	Robert S. Averback Illinois, Univ	Nonphase Ceramics	$0 $0 $0	C
26021MS-A	Kathryn V. Logan Georgia Inst of Tech	ADVANCED ARMOR MATERIALS DEVELOPMENT TiB2/A1203	$49,999 $0 $49,999	C

CERAMICS

PROPOSAL NUMBER	P. I. INSTITUTION	TITLE	FY92 FUNDS: ARO/ OTHER/ TOTAL	SUBFIELD
26392MS-A	Marc A. Meyers Calif, Univ-San Diego	Production & Evaluation of Dense Ceramic Compounds by Combustion Synthesis & Dynamic Compaction	$0 $0 $0	C
26485MS-A	Louis Zernow Zernow Technical Services	To Study and Evaluate Special Reactive Armor Concepts	$0 $0 $0	C
27373MS	James E. Mark Cincinnati, Univ	Polymer Gels as Precursors to High-Performance Materials	$96,700 $0 $96,700	C
28368MS	Marc Andre Meyers Calif, Univ-San Diego	Production & Evaluation of Dense Ceramic Compounds by Combustion Synthesis & Dynamic Compaction	$0 $0 $0	C
29098MS	Gregory C. Stangle N.Y.St. Col. of Cer. at Alfred Univ	Functionally Gradient Refractory Materials Fabrication by the Self-Propagating High-Temperature Synthesis Proc--	$51,596 $0 $51,596	C
29857MS-AAS	James E. Mark Cincinnati, Univ	Hybrid Organic-Inorganic Aerogels	$0 $0 $0	C
28884MS	Thomas J. Ahrens Calif Inst of Tech	Dynamic Consolidation of Crystalline & CVD Diamond	$20,000 $60,000 $80,000	E
30124MS	Yogendra M. Gupta Washington State Univ	Strength & Inelastic Deformation of Shocked Ceramics	$75,000 $0 $75,000	E
30347MS-URI	L. Ben Freund Brown Univ	Dynamic Behavior of Brittle Materials	$0 $0 $0	E
30351MS	Siavouche Nemat-Nasser Calif, Univ-San Diego	Dynamic Behavior of Brittle Materials	$0 $0 $0	E

CERAMICS

PROPOSAL NUMBER	P. I. INSTITUTION	TITLE	FY92 FUNDS: ARO/ OTHER/ TOTAL	SUBFIELD
30352MS	Thomas J. Ahrens Calif Inst of Tech	Dynamic Behavior of Brittle Materials	$0 $50,000 $50,000	E

TOTAL ARO FUNDS: $796,905
TOTAL OTHER FUNDS: $189,405
TOTAL ALL FUNDS: $986,310

COMPOSITES

PROPOSAL NUMBER	P. I. INSTITUTION	TITLE	FY92 FUNDS: ARO/ OTHER/ TOTAL	SUBFIELD
28509MS	John C. Bilello Michigan, Univ	Growth and Characterization of Refractor y Metal Multilayers: Potential Ultra-Hig h Strength-High Temp Surface Coatings	$105,000 $0 $105,000	A
25390MS	John J. Lewandowski Case-Western Reserve Univ	Micromechanisms of Deformation & Fracture in Aluminum Based MMC'S - Interface Effects	$34,750 $0 $34,750	B
28198MS-SAH	Arturo Bronson Texas, Univ-El Paso	Electromagnetic Processing of Refractory Metal Boride/Oxide Composites	$0 $99,646 $99,646	B
28480MS	Vijay Gupta Dartmouth College	Measurement of Interface Strength, Intrinsic Toughness & Their Dependence on Interfacial Segregants	$109,798 $0 $109,798	B
28577MS	Minoru Tomozawa Rensselaer Polytech Inst	High-Toughness Glass-Ceramics	$82,052 $0 $82,052	B
28826MS	Terence G. Langdon Southern Calif, Univ	Effect of Processing Parameters on the High Temperature Creep of SiC Whisker-Reinforced Alumina	$110,000 $0 $110,000	B
25202MS	John S. Haggerty MIT	Processing & Properties of Silicon Carbide Reinforced Reaction Bonded Silicon Nitride Composites	$0 $0 $0	C
26439MS	E. J. Lavernia Calif, Univ-Irvine	Mechanical Behavior & Processing of Aluminum Metal Matrix Composites	$0 $0 $0	C
26571MS	C. S. P. Sung Connecticut, Univ-Storrs	In-Process Cure Monitoring of Composites via Fiber-Optic Fluorescence	$50,000 $0 $50,000	C
26591MS-A	Mark C. Thies Clemson Univ	Supercritical Fluid Extraction: A New Process for Producing High-Performance, Low-Cost Carbon Fibers	$0 $0 $0	C

COMPOSITES

PROPOSAL NUMBER	P. I. INSTITUTION	TITLE	FY92 FUNDS: ARO/ OTHER/ TOTAL	SUBFIELD
26751MS-A	T. G. Nieh Lockheed Miss & Space Co.	Superplasticity in Fine-Grained Ceramic Composites	$98,061 $0 $98,061	C
26882MS-A	Alan K. Miller Stanford Univ	Enabling Technologies for Economical Manufacturing of Composites	$0 $0 $0	C
27198MS-SM	Nisar Shaikh Nebraska, Univ	Smart Structural Composites: Piezoelectric Film Deposition on Intercalated Carbon Fibers	$0 $0 $0	C
28560MS	Donald G. Baird VPI & State Univ	Processing Studies on Composites Based on Blends of Thermotropic Liquid Crystalline Polymers with Thermoplastics	$110,000 $0 $110,000	C
30095MS	Mark C. Thies Clemson Univ	Supercritical Fluid Extraction: Preparing a Superior Mesophase Precursor for Carbon Fibers	$0 $133,000 $133,000	C
30365MS-URI	Tsu-Wei Chou Delaware, Univ	ARO/URI Program in Manufacturing Science of Polymeric Composites	$0 $0 $0	C
24102MS	Stephan Bless Dayton, Univ	Penetration Mechanics of Fiber Laminate Composites	$0 $0 $0	E
26246MS	Robert R. Reeber North Carolina, Univ, Chapel Hill	Crystal Chemistry of Ceramic/Mineral Systems	$20,628 $0 $20,628	F
28620MS	Ronald D. Taylor Nat Academy of Sci	Scientific Assessment of Self-Assembling and Biomolecular Materials	$1,500 $2,500 $4,000	F
29012MS	A.S. Abhiraman UNKNOWN INSTITUTION	1991 Gordon Research conference on the Frontiers of Fiber Science	$0 $0 $0	F

COMPOSITES

PROPOSAL NUMBER	P. I. INSTITUTION	TITLE	FY92 FUNDS: ARO/ OTHER/ TOTAL	SUBFIELD

TOTAL ARO FUNDS: $721,789
TOTAL OTHER FUNDS: $235,146
TOTAL ALL FUNDS: $956,935

BUSINESS AREAS AT MTL

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

* HIGH-TEMPERATURE MATERIALS
* ARMOR AND ARMAMENT MATERIALS
* MATERIALS FOR LIGHTWEIGHT STRUCTURES
* ENVIRONMENTAL DURABILITY
* MULTIFUNCTIONAL MATERIALS
  - CHEMICAL PROTECTION
  - DIRECTED-ENERGY WEAPONS
  - LOW-OBSERVABLES
  - ELECTROMAGNETIC MATERIALS
  - SMART MATERIALS

MATERIALS SCIENCE AND TECHNOLOGY

PROCESSING RESEARCH / MANUF. TECHNOLOGY

* Major involvement of structural ceramics

OBJECTIVES

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

BUSINESS AREA	OBJECTIVES
HIGH-TEMPERATURE MATERIALS	DURABLE THERMAL-BARRIER COATINGS FOR DIESELS HIGH-TOUGHNESS, LOW-CREEP SILICON NITRIDE MONOLITHS EXPENDABLE TURBINE ENGINE WITH MONOLITHIC TURBINE ROTOR IMPROVED FIBERS FOR HIGH USE-TEMPERATURE (<1000 C) CMC's HIGH-MODULUS FIBERS FOR PMC's and MMC's DEVELOPMENT OF HIGH-TEMPERATURE TENSION TESTING METHODOLOGY STANDARDS DEVELOPMENT

OBJECTIVES

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

BUSINESS AREA

LIGHTWEIGHT STRUCTURES (& SDIO)

OBJECTIVES

HIGH-MODULUS OXYNITRIDE-GLASS FIBERS FOR STRUCTURAL APPLICATIONS

NEW ROUTES TO HIGH-MODULUS GLASS AND CERAMIC FIBERS (SOL-GEL, POLYMERIC PRECURSORS)

NEW ROUTES TO GLASS CHARGES FOR CONVENTIONAL MELT DRAWING

3-D BRAIDS AND "SMART" FIBER INCORPORATION IN CMC's

OBJECTIVES

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

BUSINESS AREA	OBJECTIVES
ARMOR/ARMAMENT MATERIALS	FRACTOGRAPHY HANDBOOK FAILURE MODES OF MONOLITHS AND COMPOSITES NEW STANDARDS (MIL STD AND ASTM)
ENVIRONMENTAL DURABILITY	CERAMIC BEARINGS - METHODOLOGY AND MATERIALS EVALUATION APPLICATION METHODS AND EVALUATION OF DIAMONDLIKE WEAR-RESISTANT COATINGS

HIGH-TEMPERATURE MATERIALS

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

	FY91 $K	FY92 $K	FY93 $K
BASIC RESEARCH	426	292	752
DEVELOPMENTAL RESEARCH	551	383	250
ADVANCED RESEARCH	100	100	290
TOTAL	1,077	775	1292

LIGHTWEIGHT STRUCTURES

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

	FY91 $K	FY92 $K	FY93 $K
BASIC RESEARCH	220	327	338
DEVELOPMENTAL RESEARCH	100	27	520
TOTAL	320	354	858

ARMOR

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

	FY91 $K	FY92 $K	FY93 $K
BASIC RESEARCH	375	288	285
DEVELOPMENTAL RESEARCH	304	254	235
TOTAL	679	542	520

OTHER BUSINESS AREAS

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

	FY91 $K	FY92 $K	FY93 $K
BASIC RESEARCH	15	125	235
DEVELOPMENTAL RESEARCH	15	50	50
ADVANCED RESEARCH	4467	800	800
TOTAL	4497	975	1085

HIGH TEMPERATURE MATERIALS FY91

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

	$K
HIGH-TEMPERATURE REACTIONS, COMPOSITES AND GLASS-CERAMICS	78
MONOLITHIC ENGINE MATERIALS : IN-HOUSE	125
CONTRACT	100
HIGH-TEMPERATURE TENSION TEST DEVELOPMENT	59
HIGH-TEMPERATURE CORROSION	50
FOREIGN MATERIALS - CMC TESTING	5
TESTING AND TEST DEVELOPMENT	45
CERAMIC BEARINGS	50
HIGH-TEMPERATURE FIBERS (BAA)	169
MONOLITHIC CERAMIC MICROSTRUCTURES (BAA)	203

LIGHTWEIGHT STRUCTURES - FY91

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

	$K
HIGH-MODULUS GLASS FIBERS: IN-HOUSE	193
CONTRACT (BAA)	84
CERAMIC BONDING (new)	50
SELF-PROP. HIGH-TEMP. SYNTHESIS (SHS) (D650)	21
AROFE	6

ARMOR - FY91

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

	$K
PROCESSING (HIP TECHNIQUES)	88
MECHANICAL TESTING	164
HIGH-STRAIN-RATE, STRUCTURAL CERAMICS	200
BALLISTIC TESTING - FRACTURE ANALYSIS	90

ENVIRONMENTAL DURABILITY - FY91

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

	$K
BEARINGS AND GEARS	50
DIAMOND-LIKE COATINGS ON STEEL	100
DIAMOND-LIKE COATINGS ON POLYMERS	25

THRUST CHANGES 1990 - 1995

US ARMY
LABORATORY COMMAND

MATERIALS TECHNOLOGY LABORATORY

FORMER	ADDITIONS
TESTING MONOLITHS	TESTING COMPOSITES
TESTING BARE SAMPLES	TESTING WITH CORROSIVE COATING
TESTING/EVALUATING COMPOSITES	FABRICATING COMPOSITES, SUPPORTING BRAIDING STUDIES
POWDER, MELT, SOL-GEL PROCESSING	NEW STARTING MATERIALS: PRECERAMIC POLYMERS, NANOPHASE MAT'LS, BIOMIMETICS
BEND AND COMPRESSION TESTING	BASIC RESEARCH ON HIGH-T TENSION; COMPOSITES
METALS TRIBOLOGY	CERAMIC BEARINGS, COATINGS; SOLID LUBES
EVALUATING TBC's WITH TACOM	IN-HOUSE & CONTRACT TBC WORK
HIGH-MODULUS GLASS FIBER PRODUCTION IN-HOUSE	PRODUCTION OF GLASS & SC FIBER UNDER CONTRACT;VSSP EVALUATION

SDIO Structural Ceramics Programs

Lt.Col. Michael Obal

Interagency Coordinating Committee on
Structural Ceramics

Annual Meeting

13 May 1992

SDIO Programs

IS&T

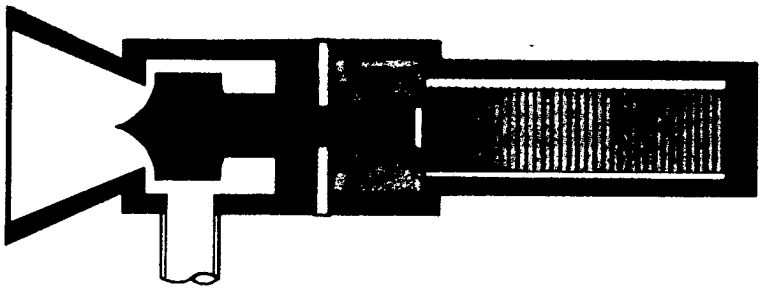
Graphite/Glass Composites

M&S

Actuator Ceramics for Adaptive Structures

KEY TECHNOLOGIES
– WARM GAS FAST ACTING CONTROL THRUSTER – (U)

M-910426-39U (C) (1117)



OBJECTIVES

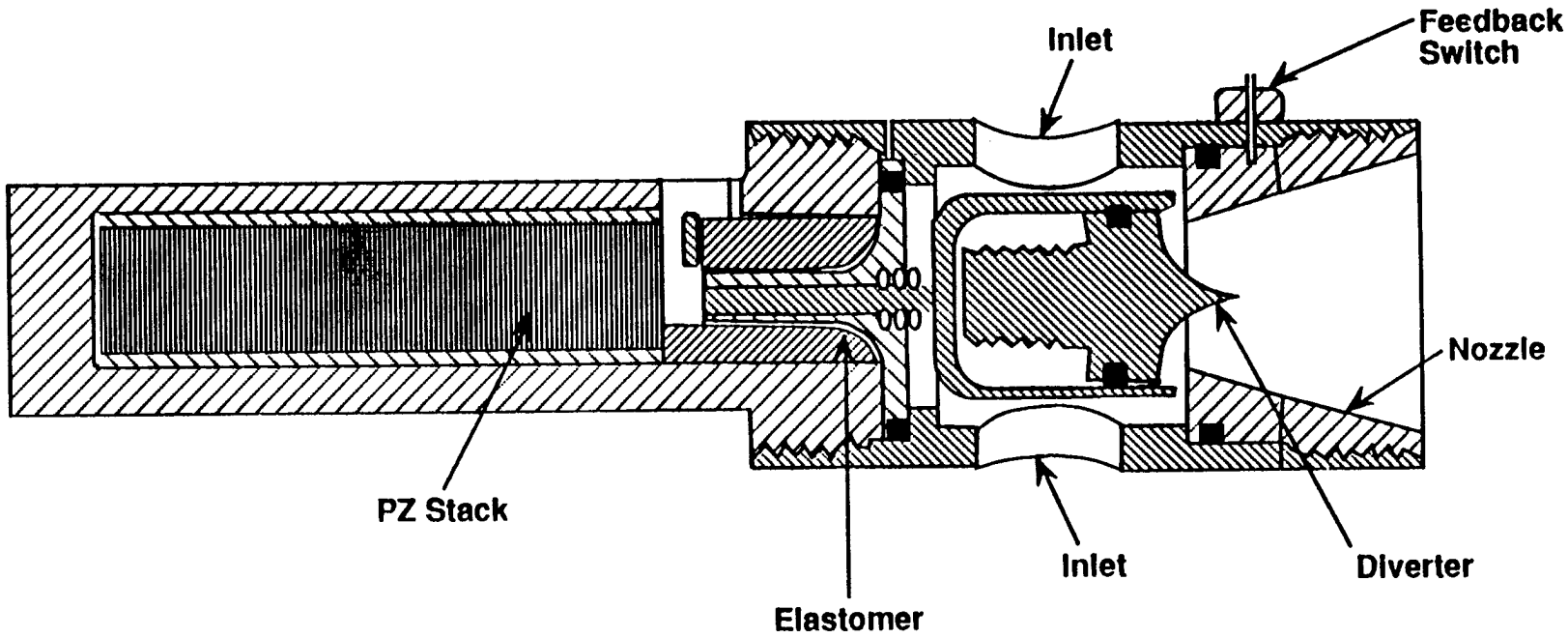
- DEVELOP AND DEMONSTRATE A FAST ACTING, PROPORTIONAL, ATTITUDE CONTROL THRUSTER UTILIZING WARM GAS ON-BOARD EXO/ENDOATMOSPHERIC INTERCEPTORS
- DEVELOP/DEMONSTRATE FAST RESPONSE/ LOW VOLTAGE REQUIREMENT

TECHNICAL CHALLENGES

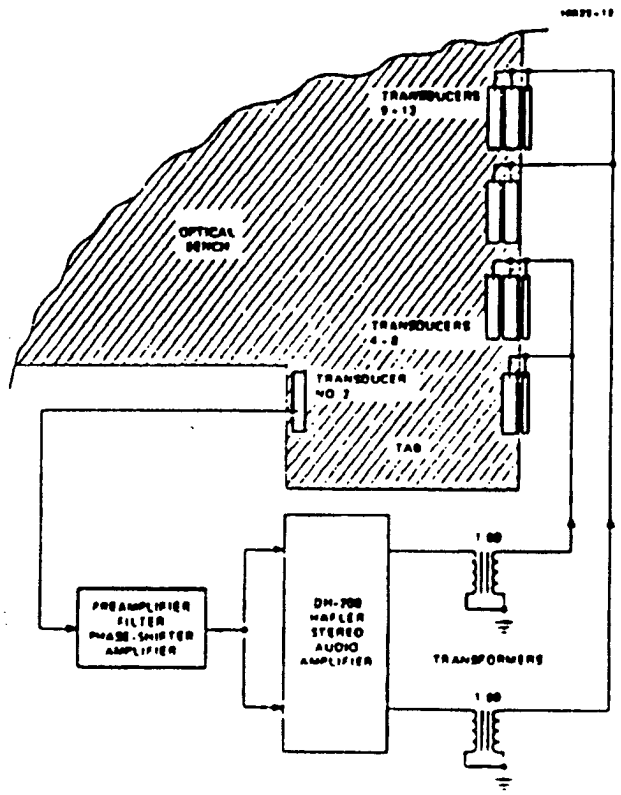
- MAXIMUM SUSTAINED THRUST/HEATING LOAD
- OPERATION OF PIEZO/ELECTROSTRICTIVE MATERIALS IN HIGH THERMAL ENVIRONMENT
- POWER AVAILABILITY

BENEFITS

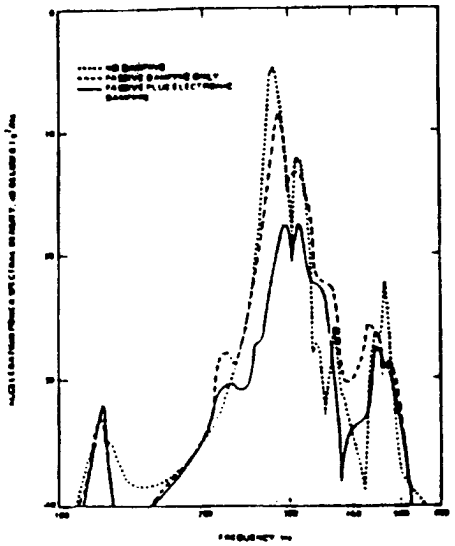
- LIGHWEIGHT/PROPORTIONAL CONTROL THRUSTER
- 1 MILLISECOND RESPONSE TIME
- HIGH THRUST-TO-WEIGHT RATIO
- HIGH PRECISION ATTITUDE CONTROL ADJUSTMENTS
- REDUCED DYNAMIC IMPULSE TO STRUCTURE


Inlet
Feedback Switch
Nozzle
PZ Stack
Elastomer
Inlet
Diverter

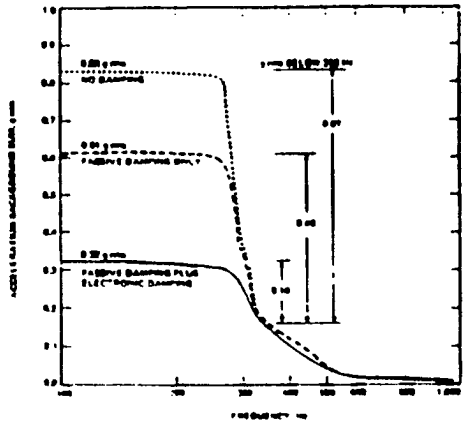
Laser Weapon Optical Bench
Active/Passive Vibration Suppression



Electronic Damping
Feedback Loop

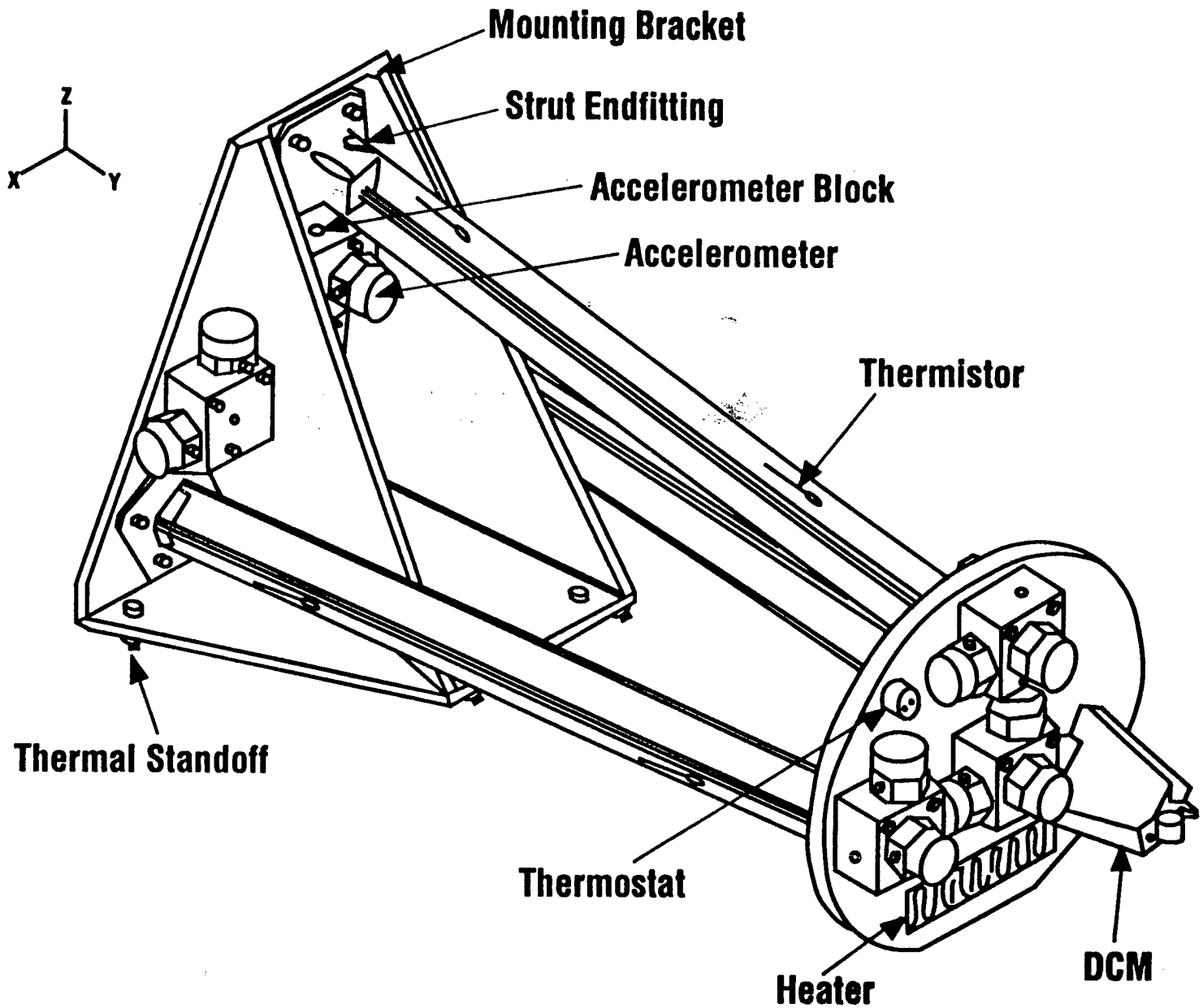


Acceleration Power
Spectral Density



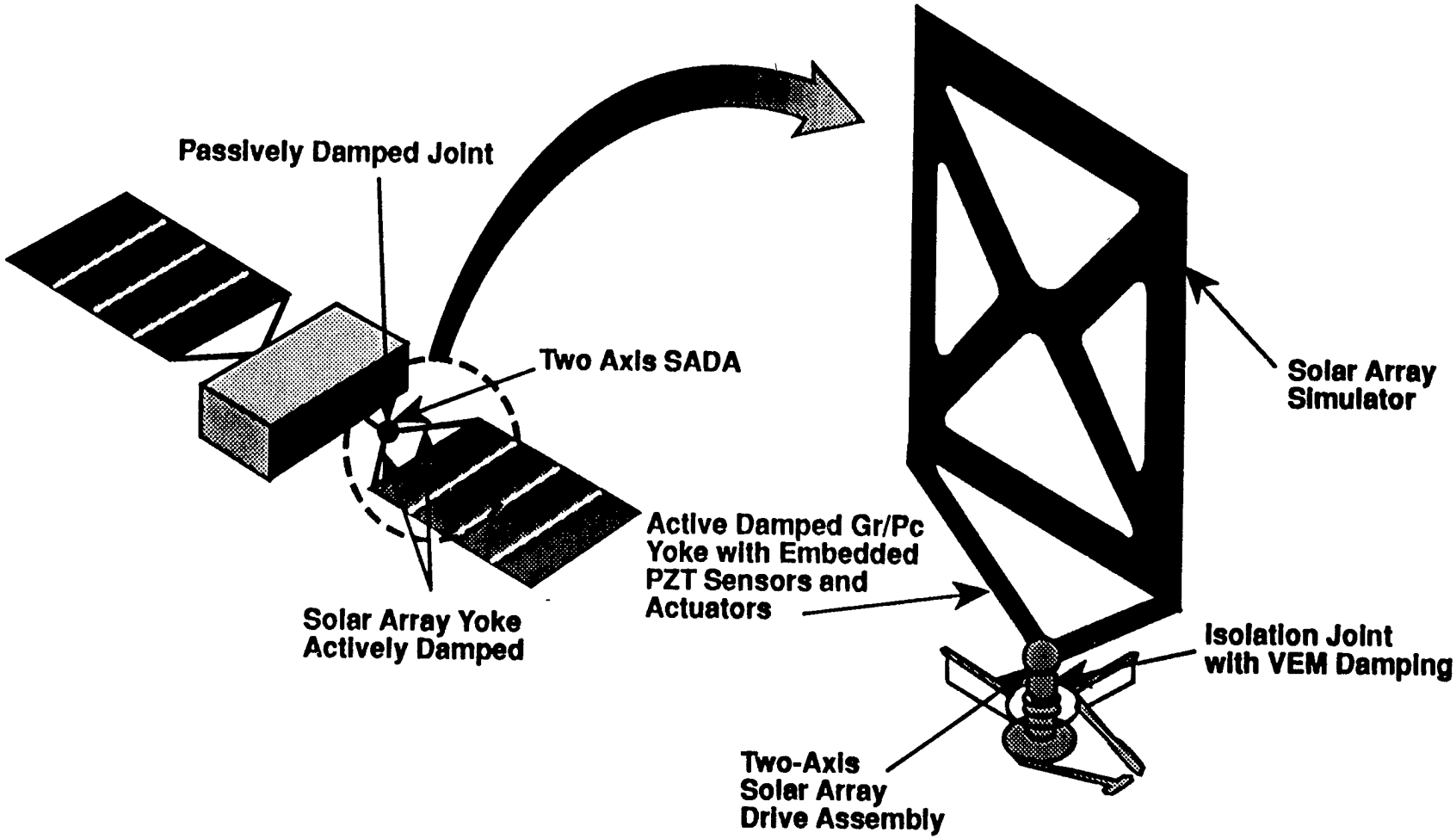
Back Sum of
Foward Acceleration

ACTEX Tripod



3-15-92-15M

AMASS Validation Structure



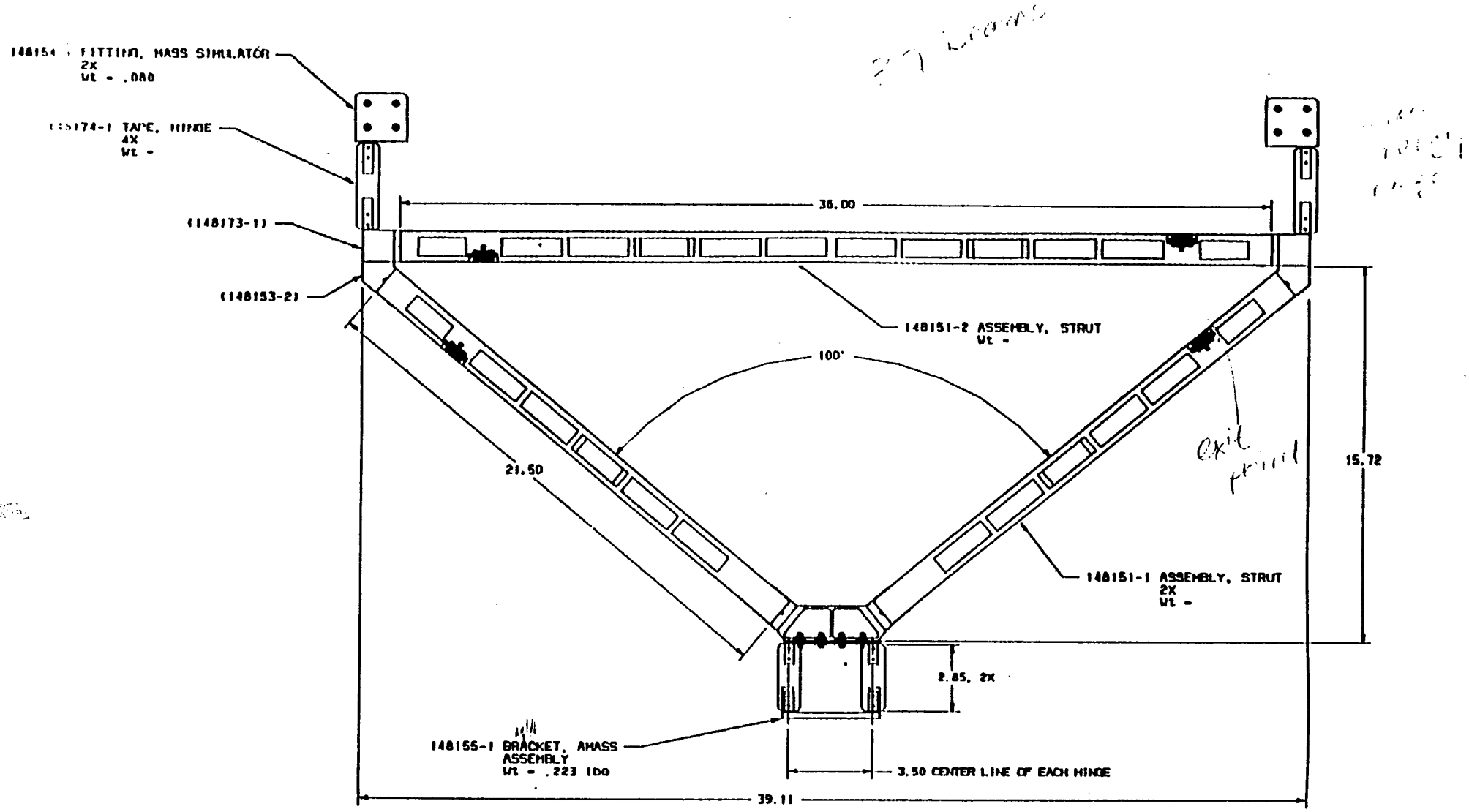
SDI Spacecraft **Test Structure**

3-13-92-10m

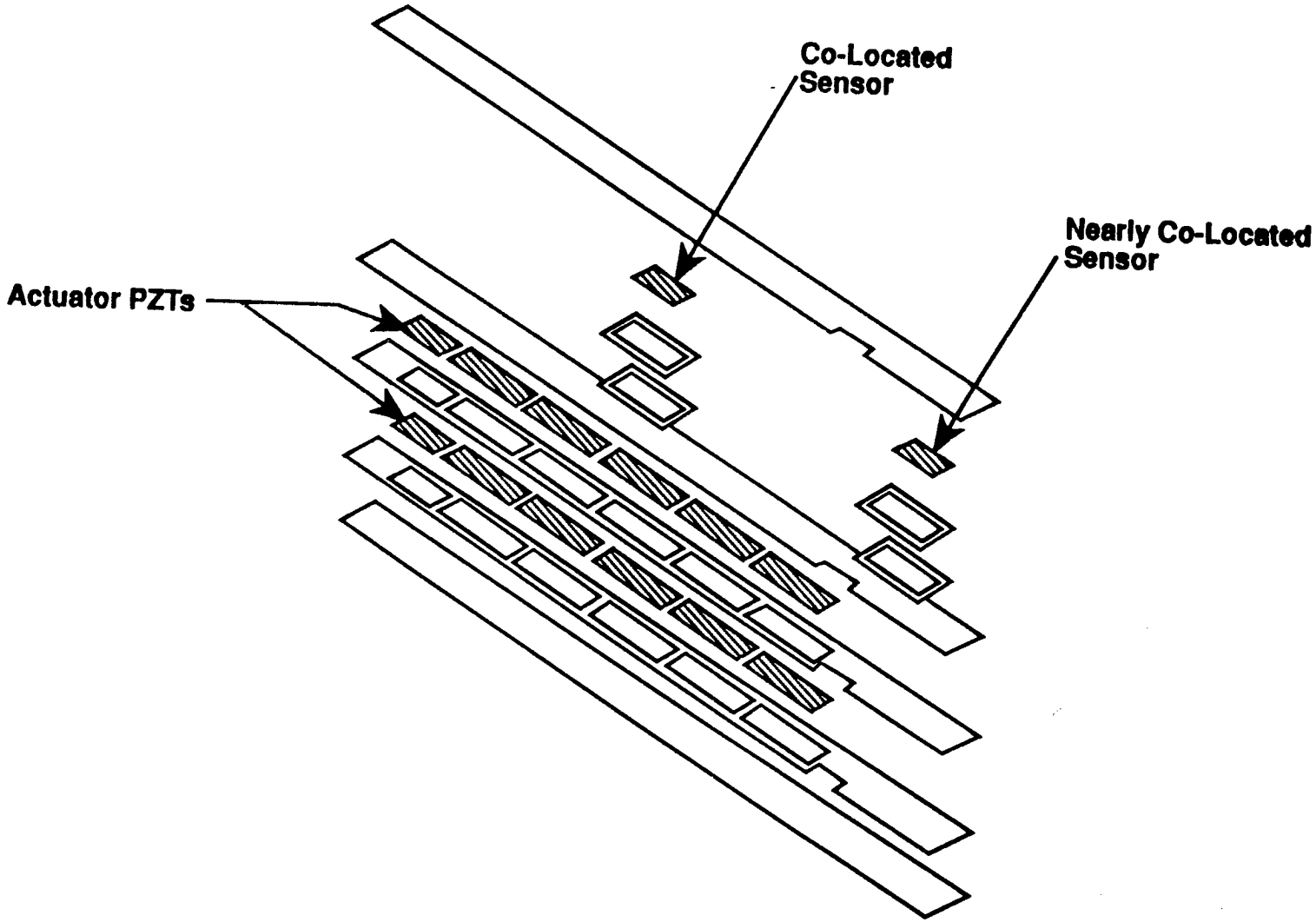
AMASS

YOKE STRUCTURE

TRW

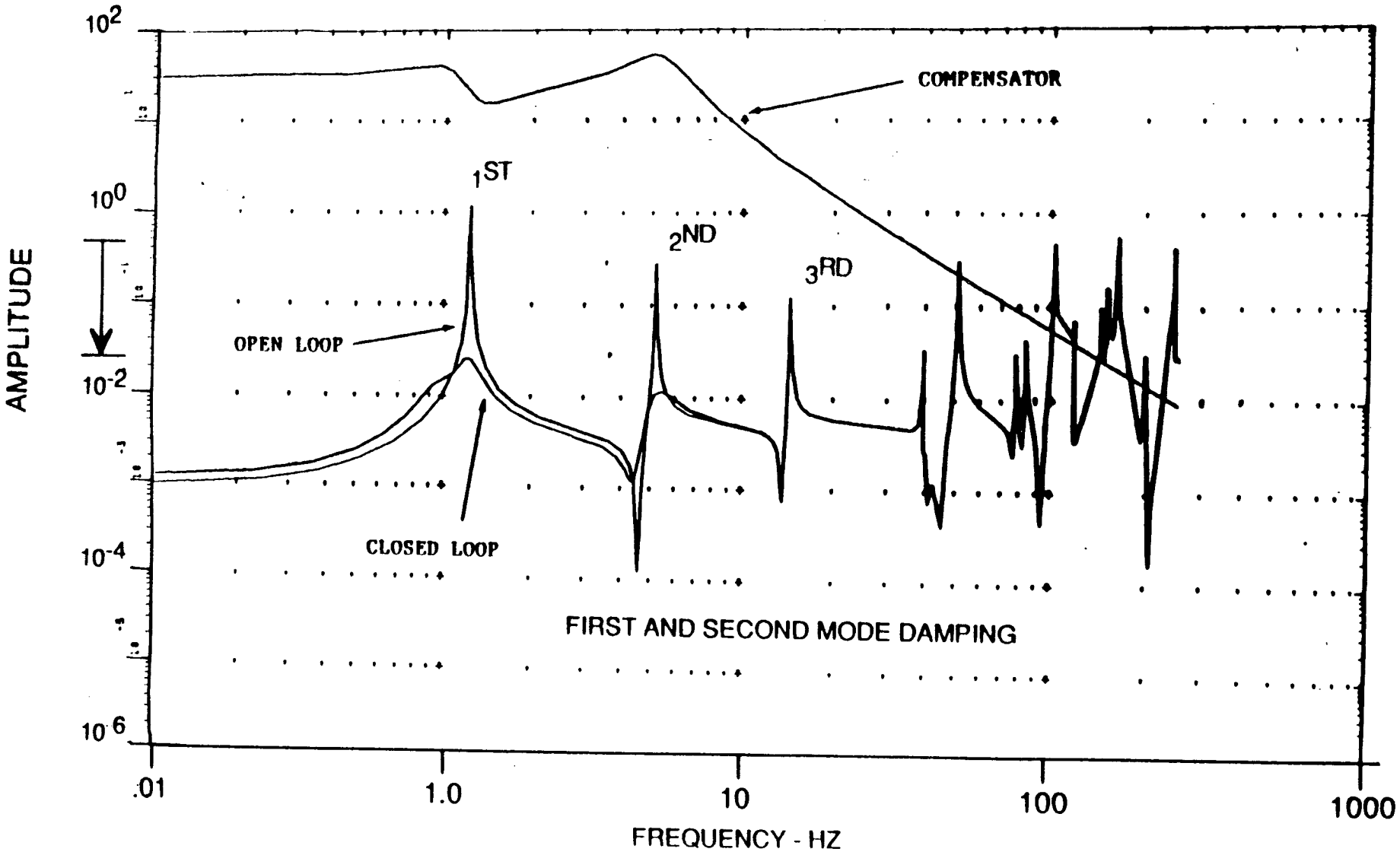


AMASS Struts

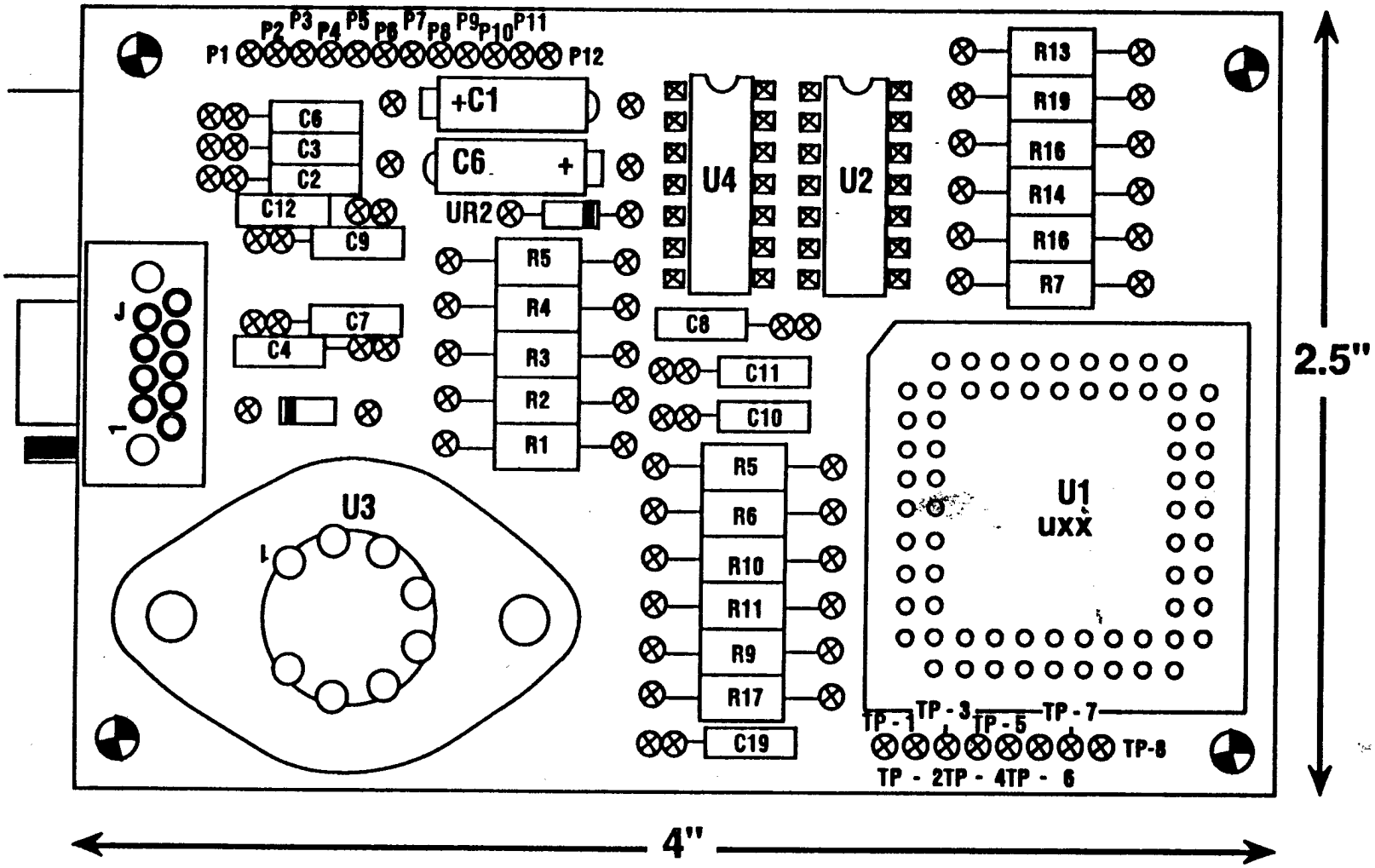


3-13-92-12m

AMASS PRELIMINARY TEST RESULTS



AMASS Electronics Board Layout



3-13-92-13M

Piezoceramic Material and Device Issues

d_{31} vs. d_{33} performance
Displacement/volt, power requirements
Fatigue life at high strains
Tensile strain-to-failure
Aging behavior, hysteresis
Temperature/environmental effects
Ceramic material/actuator fabrication
Surface finish
Dimensions, shape
Material handling
Electrode materials, application to actuator
Post-composite fabrication actuator performance
Actuator-structure interface

SDIO - ACTUATOR MATERIALS PROGRAM

INTERIM REPORT ON NRL WORK

A) DEVELOPMENT OF
LOW FIRING PZT FOR LOW VOLTAGE (THIN LAYER) ACTUATORS
AND COST REDUCTION OF INTERNAL ELECTRODE MATERIAL.
SINTERING TEMPERATURES HAVE BEEN REDUCED BY >150°C
AND SIGNIFICANTLY ENHANCED DENSIFICATION EFFECTS OF
WET CHEMICAL FLUX ADDITION WERE DEMONSTRATED.

B) ADDITION OF ZrO_2 WHISKERS TO PZT RAISED AREA OF FRACTURE SURFACE
(AND THEREFORE RELATIVE TOUGHNESS)
BUT INCREASED DENSITY OF DISCONTINUITIES (FLAWS)
IMPACTED ON FRACTURE STRENGTH.

C) PNZST AND PLZST ANTIFERROELECTRIC-FERROELECTRIC PHASE CHANGE CERAMICS
WERE PREPARED AND TESTED.
WITH CURIE TEMPERATURES NEAR 175°C
STEADY STATE DIMENSIONAL CHANGES OF >1700 μ STRAINS WERE OBSERVED.

SDIO - ACTUATOR MATERIALS PROGRAM

PROPOSED RESEARCH THRUSTS FOR 1992-1993

A) MATERIALS

1) RESEARCH INTO PHASE CHANGE (HIGH STRAIN) TEMPERATURE STABLE MATERIALS AND CHARACTERIZATION OF KNOWN COMPOSITIONS
2) EVALUATION OF KNOWN HIGH STRAIN PIEZOELECTRICS (I.E. PZN)
3) COMPOSITIONS AND METHODS FOR FLUXED (LOW FIRING) PZT
4) EXPLORATION OF TANTALATE ANALOGS FOR CRYOGENIC ACTUATORS
5) PREPARATION OF PZT FROM CHEMICAL PRECURSORS

B) ACTUATORS

1) STRENGTH CHARACTERIZATION OF ACTUATORS AND ACTUATOR MATERIALS
2) ELECTRODES, CERAMIC MATERIALS AND MANUFACTURING TECHNOLOGY FOR LOW VOLTAGE (THIN LAYER) ACTUATORS
3) DEVELOPMENT OF PERFORMANCE TEST CRITERIA AND SPECIFICATIONS FOR STRUCTURE INTEGRATED ACTUATORS
4) DEVELOPMENT OF WIDE TEMPERATURE RANGE AND OF MANUFACTURING TECHNOLOGY FOR FLEXTENSIONAL ACTUATORS (MOONIES)

ESTIMATED COST OF FULL PROGRAM $\$10^6$

MAY 13, 1992

ADVANCED TURBINE TECHNOLOGY APPLICATIONS PROGRAM (ATTAP)

Performing Organization

U.S. DEPARTMENT OF ENERGY
OFFICE OF TRANSPORTATION TECHNOLOGIES
OFFICE OF PROPULSION SYSTEMS
ADVANCED PROPULSION DIVISION

SAUNDERS B. KRAMER

CCE31603

EXECUTIVE SUMMARY
ATTAP PROGRAM

THE ATTAP (ADVANCED TURBINE TECHNOLOGY APPLICATIONS PROGRAM) WAS INITIATED FOR THE PURPOSE OF DEVELOPING STRUCTURAL CERAMICS FOR THE HOT SECTION OF THE ADVANCED AUTOMOTIVE INDUSTRY AS A VIABLE STARTING POINT FOR THE INDUSTRY TO ENGAGE IN MANUFACTURING ENGINEERING VIS-A-VIS THE USE OF THE GAS TURBINE AS THE NEW PROPULSION SYSTEM FOR AUTOMOBILES.

THE PROGRAM COMMENCED IN MID-1987 AND HAS A FY '92 BUDGET OF $12.15 MILLION. THE TWO MAJOR CONTRACTORS, OPERATING IN A COOPERATIVE MODE (I.E., NOT COMPETITIVE FOR THE PURPOSE OF DEVELOPING THE AFORESAID CERAMICS) ARE THE ALLISON GAS TURBINE DIVISION OF GM AND THE GARRETT AUXILIARY POWER DIVISION OF THE ALLIED-SIGNAL AEROSPACE CORP. THESE CONTRACTORS EACH HAVE A FIVE YEAR CONTRACT, ENDING IN 1992, WHICH IS INCREMENTALLY FUNDED ON AN ANNUAL BASIS. ALL CERAMIC COMPONENT RESEARCH AND DEVELOPMENT IS SUBCONTRACTED TO DOMESTIC INDUSTRY BY THE TWO MAJOR CONTRACTORS. THE CHOICE OF CERAMIC SUBCONTRACTORS WAS MADE AFTER A DETAILED SURVEY OF FORTY POTENTIAL MEMBERS BY THE TWO TEAMS. NINE SUCH MEMBERS WERE CHOSEN AND SOME OF THESE PROVIDE R & D TO BOTH ALLISON AND GARRETT. BOTH ALLISON AND GARRETT ARE NOW TESTING CERAMIC COMPONENTS IN THE 2500 F (1371C) TEMPERATURE REGIME.

ATTAP Objectives

- Establish the Reliability of Ceramic Component Designs and Materials for Use in Automotive Gas Turbine Applications
- Develop the Analytical Tools Required to Support Industry in the Application of Ceramics to Long Life Gas Turbine Engines
- Expand the Ceramic Component Experimental Data-Base in the Gas Turbine Operating Environment

ATTAP

ADVANCED TURBINE TECHNOLOGY APPLICATIONS PROGRAM

SPECIFIC INTENTS OF ATTAP

- Ceramic component processing methods will be developed in the U.S.A.
- Ceramic component design methodology will have been developed
- Reliable ceramic components will have been reproducibly fabricated, tested evaluated in rigs and in test-bed engines
- Technology developed in the program will have been transferred to the US Auto and Gas Turbine Engine Industries

bce11796

MAJOR ACCOMPLISHMENTS DURING FY 1991 & 1st HALF FY 1992

- SILICON NITRIDE (KYOCERA) AXIAL ROTOR TESTED FOR 1001 HOURS:

  AT TEMPERATURES UP TO 2500 F AND UP TO FULL SPEED -- 63000 RPM

- ALL CERAMIC GASIFIER SECTION OF TURBINE ENGINE TESTED FOR 100 HOURS:

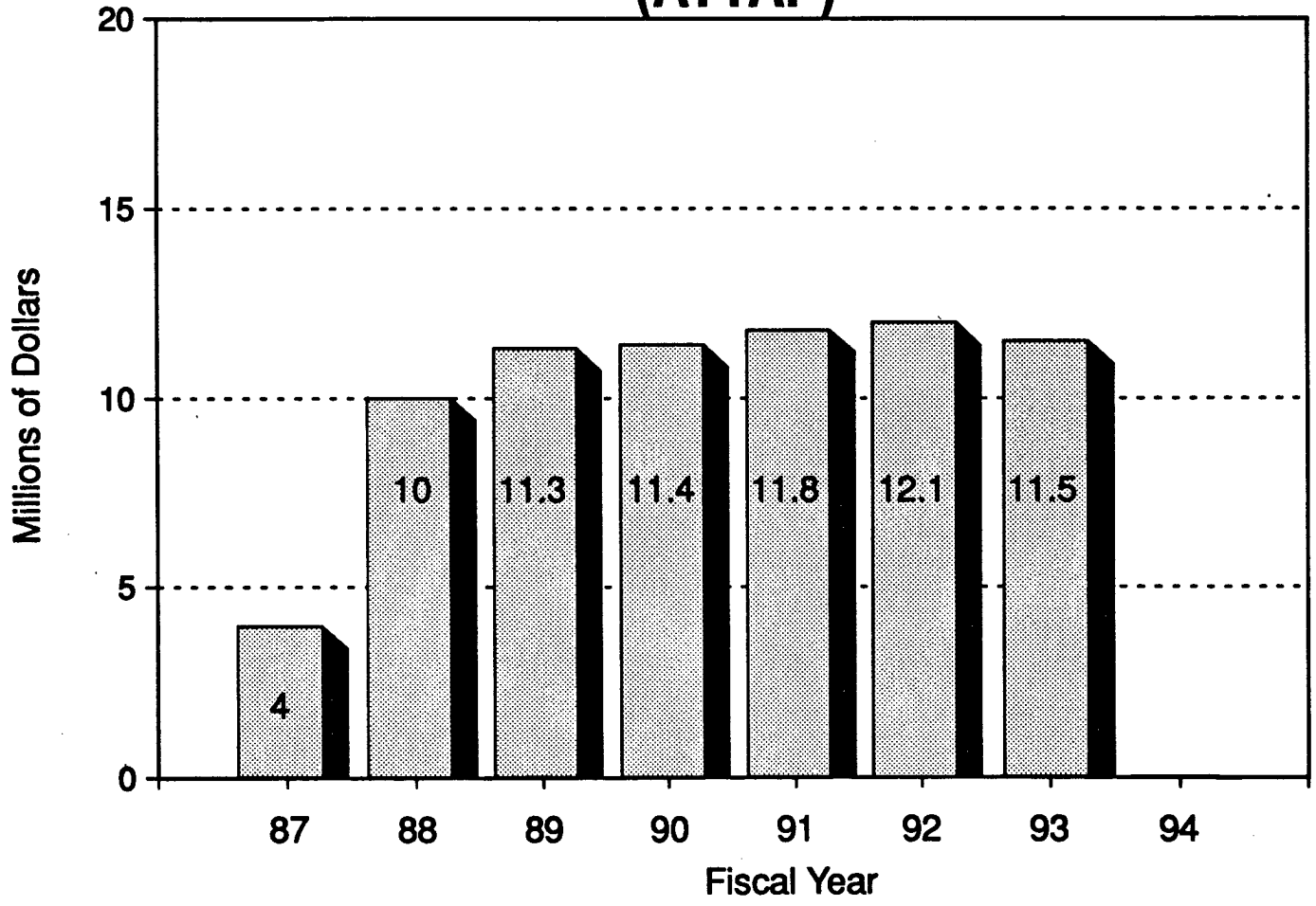
  THEN TESTED FOR AN ADDITIONAL 96 HOURS

- SILICON NITRIDE (NORTON/TRW) AXIAL ROTOR TESTED FOR 262 HOURS AT TEMPERATURES UP TO 2500 F AND UP TO FULL SPEED

- COMPLETED INITIAL RIG TEST OF MIXED FLOW ROTOR DESIGN

ACE52041/first

ADVANCED TURBINE TECHNOLOGY APPLICATIONS PROGRAM (ATTAP)



ACE52041/first

INTERAGENCY COORDINATING COMMITTEE FOR STRUCTURAL CERAMICS

Annual Meeting, May 13, 1992

DR. BRIAN VOLONTINE

ADVANCED INDUSTRIAL CONCEPTS MATERIALS PROGRAM

Office of Industrial Technologies, Conservation and Renewable Energy

U.S. Department of Energy

The mission of the AIC Materials Program is to conduct applied research and development to help bridge the gap between basic research and industrial application of energy saving materials and materials processing methods. This is accomplished through development of materials that save energy by enabling improved system efficiencies and increased service lives and of processing methods using less expensive raw materials and fewer processing steps. The Program serves as a resource for end-use programs withing Conservation and Renewable energy and works directly with industry to identify needs, conduct cooperative research and development, and transfer technology.

STRUCTURAL CERAMICS PROJECTS - FY 1992

Chemical Vapor Infiltration of TiB_2 - Oak Ridge National Laboratory - Ted Besmann, P.I. Funding: $200,000

The purpose of this task is to develop continuous filament TiB_2 matrix composites by use of a forced-flow chemical vapor infiltration method. The work is coordinated with extensive modeling efforts at Oak Ridge and Georgia Institute of Technology and the work on computerized X-ray tomography at Lawrence Livermore National Laboratory.

Microwave Assisted Chemical Vapor Infiltration of SiC - Los Alamos National Laboratory - Robert Currier, P.I. Funding: $200,000

Work is being conducted to deposit silicon carbide by chemical vapor infiltration into woven SiC fiber reinforcements. Application of microwave power creates an inverse thermal gradient so that deposition proceeds from the center outward, minimizing porosity and eliminating the need for periodic machining to reopen channels for gas infiltration.

Microwave Sintering of Oxides - Oak Ridge National Laboratory - Mark Janney, P.I. Funding: $500,000

The work has shown that microwave sintering of oxide ceramics is a low energy alternative to resistance heating and results in increased sintering rates and beneficial microstructures. Additives to couple with microwave energy without significant property changes have been found and extensive modeling of microwave energy distribution has been done. A variable frequency microwave furnace has been developed and is being commercialized. The work is

coordinated closely with microwave sintering of silicon nitride supported by the Office of Transportation Technologies.

Microwave Spray Drying of Oxides - Los Alamos National Laboratory - Gerald Vogt, P.I. Funding: $400,000

The project is to determine the feasibility of using microwave energy to spray dry oxide ceramic powders by continous flow through a cylindrical microwave cavity. Results have shown that uniform, spherical particles, with a minimun of agglomeration can be obtained.

Chemical Vapor Composites - Sandia National Laboratories, Livermore - Mark Allendorf, P.I. Funding: $320,000

The formation of SiC composites by chemical vapor deposition with particulate or short fiber reinforcements entrained in the gaseous reaction stream was developed by Thermoelectron Technologies. This task includes modeling of the kinetics and mechanisms of the process, coupled with experimental verification at the Combustion Research Facility of SNLL.

Synthesis and Processing of Composites by Reactive Metal Infiltration - Sandia National Laboratories, Livermore - Ronald Loehman, P.I. Funding: $350,000

Infiltration of a porous preform of ceramic powder by a reactive metal, such as molten aluminum, can result in a wide variety of reaction products depending on the chemical identities and ratios of the reactants. This work is being done to develop a variety of net shape or near net shape composites, with either ceramic or metal matrices, using inexpensive raw materials and relatively low temperature processes. The work is coordinated with a project at Oak Ridge to develop oxide and carbide composites reinforced with intermetallic alloys.

Computerized X-Ray Tomography of Composites - Lawrence Berkeley Laboratory - John Kinney, P.I. Funding: $180,000

Three dimensional images of porosity in chemical vapor infiltrated composites can be obtained by this method. A specially constructed CVI reactor makes it possible to interrupt the CVI process at any stage for XTM, thereby providing a means of determining the progress of deposition, the geometry of remaining pore spaces, the extent to which pores have been closed to further deposition. The resolution if sufficient to map both density and morphology of the matrix with respect to position within the reinforcing fibers. The work is closely coordinated with other composites work in the AIM Materials Program.

ADVANCED INDUSTRIAL CONCEPTS MATERIALS PROGRAM

Structural Ceramics Funding
($000)

Project	FY 1991	FY 1992	FY 1993
Chemical Vapor Infiltration of TiB_2	200	200	200
Microwave Assisted CVI of Sic	200	200	200
Microwave Sintering of Oxides	500	450	450
Microwave Spray Drying	400	400	400
Chemical Vapor Composites	300	320	350
Composites by Metal Infiltration	0	350	350
X-Ray Tomography	200	180	200

U.S. DEPARTMENT OF ENERGY

OFFICE OF INDUSTRIAL TECHNOLOGIES CE-20

Alan J. Streb, Deputy Assistant Secretary, 586-9232

Denise Swink, Assoc. Deputy Assis. Sec., 586-0559
Beatrice L. Cunningham, Secretary, 586-9232
Kenneth M. Friedman, 586-9236, Special Assistant
Noel K. Cole, 586-9238, Budget Analyst

Office of Waste Reduction CE-22

Don K. Walter, Director, 586-6750
Alfreda F. Jenkins, Secretary, 586-2090
Virginia B. Gordon, 586-2387, Budget/Administration
Gerry Dorian, 586-2369, Environ. Liaison

Industrial Energy Efficiency Division CE-221

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Violet Bruce, Secretary, 586-2098
Scott L. Richlen, 586-2078, Heat Exchangers, Ceramics
William P. Parks, 586-2093, Cogeneration
Ramesh C. Jain, 586-2381, Combustion Efficiency Improv.
Gideon Varga, 586-0082, Combustion
Paul E. Scheihing, 586-7234, Heat Pumps, Electric Motors

Waste Material Management Division CE-222

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Marion V. Gunter, Secretary, 586-6750
(Vacant), 586-2369, Waste Utiliz./Conver.
Alan C. Schroeder, 586-1641, Liquid Waste Utilization
Stuart L. Natof, 586-2370, Solid Waste Utilization
H. Bruce Cranford, 586-9496, Waste Minimization
Simon Friedrich, 586-6759, Municipal Solid Waste
Cliff Carwile, 586-5584, Solar-Thermal
Frank W. Wilkins, 586-1684, Solar Detoxification
(Vacant), 586-xxx, Municipal Solid Waste

Implementation and Deployment Division CE-223

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Marilyn T. Burgess, Secretary, 586-2366
Charles J. Glaser, 586-1298, Energy Analy. & Diag. Centers
Marsha L. Quinn, 586-2097, Technology Transfer
(Vacant), 586-xxxx, Analyst

Office of Industrial Processes CE-23

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Sheila A. Traynham, Secretary, 586-9487
Barbara G. Euell, 586-2079, Information Services

Improved Energy Productivity Division CE-231

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W. Eugene Eckhart, 586-8668, Steel, Foundries
Matthew J. McMonigle, 586-2082, Aluminum, Glass, Mining
Gobind N. Jagtiani, 586-1826, Steel, Foundries
William A. Obenchain, 586-3090, Metals
Stanley F. Sobczynski, 586-1878, Paper, Separations, Sensors/Controls
William M. Sonnett, 586-2389, Separations, Membranes, Drying, Food
Mary K. Corrigan, 586-1708, Agriculture, Food, Drying, Sensors/Controls
Linda Beth Schilling, 586-8091, Petroleum, Chemicals

Advanced Industrial Concepts Division CE-232

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Shanere Waller, Secretary, 586-5377
(Vacant), 586-7543, Combustion
David Boron, 586-0080, Biocatalysis, Catalysis, Chemicals
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23-Feb-92

THE ADVANCED INDUSTRIAL CONCEPTS MATERIALS PROGRAM
FY 1992

Mission and Goals

The mission of the Advanced Industrial Concepts Division (AIC), within the Office of Industrial Technologies, Conservation and Renewable Energy, is to support generic, long-term, high-risk applied research and development in those processes and technologies that underpin industrial unit operations. The AIC output provides a technology base to improve energy use efficiency and advance industrial capability to use alternative energy resources. Materials-related research in AIC is conducted in the AIC Materials Program. The Materials Program develops generic materials technologies in order to advance them to a stage at which private industry or other government programs can carry them on to technology and engineering demonstration. The Program emphasizes materials as an enabling technology for industrial energy conservation. For purposes of definition and planning, the following general guidelines are considered appropriate.

- Generic. Research is conducted on those materials and processes with a broad range of applications in industrial operations. If materials and processes are applicable to other sectors, such as transportation, buildings, and utilities, the applicability is to be identified and coordination is established with the appropriate Program Managers.

- Applied Research and Development. Emphasis is on those materials and processing technologies that have the potential for real industrial application within a reasonable period of time. Technologies that have advanced beyond basic research but are not ready for practical applications are appropriate for AIC materials research. The Program attempts to take a lead role in "bridging the gap" in materials research and development for commercialization.

- Technology Base. For any materials or processing technology that has not found industrial use, there is knowledge needed to enable practical application. In the spectrum between discovery and manufacturing, that missing knowledge may be at any point between basic research and the factory floor; it is a proper role for the AIC Materials Program to identify the technology (knowledge) needs and to conduct appropriate research to satisfy those needs.

- Towards Technology and Engineering Demonstration. The goal of AIC materials research is to "bridge the gap" between basic research and proof-of-concept or prototype production. The difficult task is to determine the point at which technology is picked up from basic research and the point at which it should be delivered to others for implementation. These are different for

each material or process.

• Private Industry or Other Government Programs. A logical continuation of the previous statements is that, at some stage, the research conducted will be ready to turn over to others for industrial use. At present, AIM researchers, assisted by technology transfer offices, are doing an excellent job of interesting industry in the materials and processes resulting from their work. These efforts are properly considered within the missions of other DOE programs or even those of other government agencies and concerted effort is exerted to pass the technology. The Program can, as a result, identify new technology concepts and have the flexibility to act upon them.

FY 1991 Accomplishments

Following the successful improvement and development of new materials and processes for industrial energy efficiency and transfer of technology in 1990, the Engineered Industrial Materials and Materials Manufacturing Technologies programs were even more successful in 1991. During those two years, researchers in the programs produced over 130 technical papers, held 8 workshops and technical exchange meetings, and worked closely with 59 manufacturing companies. The work was recognized by 4 R&D 100 awards and nine peer awards. Direct indicators of technology transfer were the 9 Cooperative Research and Development Agreements and 6 Patents, with 11 Patent applications pending and 6 more Patent disclosures filed. Two licenses have been granted to industry and two new companies have been formed to use the new technology.

Research on intermetallic alloys at ORNL and LANL resulted in solutions to several barriers to use of these materials at very high temperature in hostile environments. The inherent low temperature brittleness and high temperature creep were eliminated by alloy additions and microstructural control through processing; the first successful method for welding nickel aluminides was developed; and the shape memory effect in nickel aluminide was characterized. Industrial testing of these materials showed that they have great energy saving potential and will be more reliable and durable than currently used materials.

At NREL, cost effective processes for recovery of monomers from six different mixed plastic waste streams were developed. The monomers can be used for making new polymeric materials at lower cost. The first successful process to produce thermoset plastics that can be chemically recycled was developed by NREL researchers, and new polymer composites were made from wood products.

Aerogels, developed at LBL, demonstrated record high insulating values, more that four times those of any other material, and have attracted interest from automobile, appliance, aerospace,

and construction materials manufacturers. Research on aerogels and other sol-gel processes will lead to an entirely new class of functional materials to serve as filters, membranes, catalyst carriers, and electolyte carriers.

Work at ORNL, LLNL, LANL, and SNLL has advanced understanding of ceramic composites formation for very high temperature structural uses by modeling chemical vapor deposition processes, devising new methods of infiltrating reinforcing preforms, using microwaves to densify advanced ceramic materials with a fraction of the usual energy use, and developing an X-ray tomography camera that detects pores and flaws of microscopic size in the interior of the component. Other important ceramic materials being developed include oxide and carbide ceramics bonded with intermetallic alloys to produce high strength, ultratough, wear resistant and corrosion resistant composites.

Other significant materials developments include chemically specific surfaces, with potential for wide application as membranes, sensors, and ion absorbers, being developed at SNLA; commodity plastics strengthened by polymerization in a magnetic field at LANL; ultratough polymers with surfaces nearly as scratch resistant as diamond being produced at ORNL; and oxide films with controlled crystal structure and orientation, being made at PNL by room temperature processes that mimic biological processes. All these projects, though underway for only a short time, have been remarkably productive and promising.

Properties and performance capabilities of materials now used place severe limits on energy efficiency, systems durability, and environmental compatibility of industrial processes. The goals of the AIC Materials Program are to identify materials that will enable industrial systems to operate with greater thermal and mechanical efficiencies, greater reliability, and increased service lives and to conduct research and development on energy efficient processes to synthesize materials to be used to assemble practical components and devices.

FY 1992 Research Activities

Engineered Industrial Materials--In FY 1992, research, development, and technology transfer efforts is devoted to alloys, primarily nickel aluminides, iron aluminides, titanium aluminides, and molybdenum disilicides. Properties of these materials have been improved to the extent that successful industrial trials have been conducted, several CRADA's have been signed, and potential new applications have been identified. Given the number of alloys and the multitude of industrial applications, a program plan for continuation of work on intermetallics and for identification of other important alloys systems is needed and will be developed. Work on rapidly solidified alloys (INEL) is continuing; during FY 1991, three CRADA's were signed with industrial partners and three more will

be completed early in FY 1992. FY 1992 goals include 1) demonstration of modeling validity and manufacturability of $MoSi_2$ reinforced with continuous fibers (LANL), 2) completion of melting and solidification characterization of castable nickel aluminide alloys, and 3) completion of mechanical property and shape memory characterization of nickel aluminide.

Research and development of polymers (lightweight and biobased materials) will continue (NREL), with increased emphasis on transfer of the highly successful work on recycling technology and consolidation and enhancement of work on production of biobased composites. Investigation of the effects of magnetic fields applied during polymerization and processing of plastics on properties is continuing at LANL and INEL. Major efforts include 1) assessment of unsaturated polyester streams for highest value to be recovered, 2) demonstration of magnetic field induced effects in commercially available composite prepregs, and 3) establishment of new collaborative efforts with other organizations. Recent discoveries at LANL have shown that conducting polymers have useful membrane characteristics and can be tailored to many specific applications; additional emphasis is being given to this work.

Work on advanced ceramic materials and processing includes a coordinated effort in modeling and synthesis of materials by chemical vapor infiltration and deposition (CVI and CVD), microwave sintering of ceramics and microwave drying of ceramic powders, and thermally insulating materials. Goals for FY 1992 include 1) assessment of microwave drying of ceramic powders and initiation of collaborative effort with an industrial partner to explore microwave processing of commercial ceramic fibers (LANL), 2) completion of work on synthesis of hollow ceramic spheres (aerospheres) and continuation of technology transfer efforts (Georgia Tech), 3) completion of baseline data development for processing/structure/property relationships for fiber reinforced titanium diboride and 4) initiation of cooperative work by LBL and the National Institute of Standards and Technology on synthesis of amorphous foams by supercritical drying (aerogels) and by sol-gel methods; this work extends the potential uses for such materials beyond thermal insulation into applications as membranes and filters, carriers for nanocrystalline functional materials, and ion exchange media.

Materials Manufacturing Technologies--Surface modification research is continuing at LANL with emphasis on conducting polymer coatings for PC board applications, at ORNL on ion implantation to increase surface hardness of polymers, at SNLA on chemically specific coatings, and at PNL on biomimetic synthesis. This research area is targeted for enhancement after improvement of coordination of current efforts and identification of new research areas and collaborative groups. Highlights for FY 1992 include 1) initiating cooperative efforts with the printed circuit board industry to develop technology for placing conducting polymer coatings on insulators, 2) development of

highly sensitive and chemically specific coatings and membranes, and 3) initiation of an industry-university-laboratory group effort to develop new wear resistant and corrosion resistant coatings.

New materials and processing are the objects of intensive planning in FY 1992. The objectives are to determine what unique processes can be developed using inexpensive raw materials and new processing methods that can reduce the number of processing steps and energy use during production. New synthesis methods for nanocrystalline and amorphous materials, membrane materials, and new ceramic and metal matrix composites will receive attention. The first new process initiated in FY 1992 is "Synthesis and Processing of Composites by Reactive Metal Infiltration" at SNLA. The method will produce either metal matrix or ceramic matrix composites to net shape or near net shape at relatively low temperatures (above the melting point of the reactive metal). The number of possible systems and reaction sequences is large. Major efforts for FY 1992 are 1) to determine the chemistry, thermodynamics, and kinetics of the most promising systems, 2) determine the effects of experimental variables on physical properties of selected materials, and 3) fabricate near-net-shape demonstration parts. A major effort in engineered porous materials is being planned, to include portions of existing projects, enhanced by new efforts. The work will be coordinated with other programs within the Office of Industrial Technologies.

THE U.S. DEPARTMENT OF ENERGY'S CONTINUOUS FIBER CERAMIC COMPOSITE PROGRAM

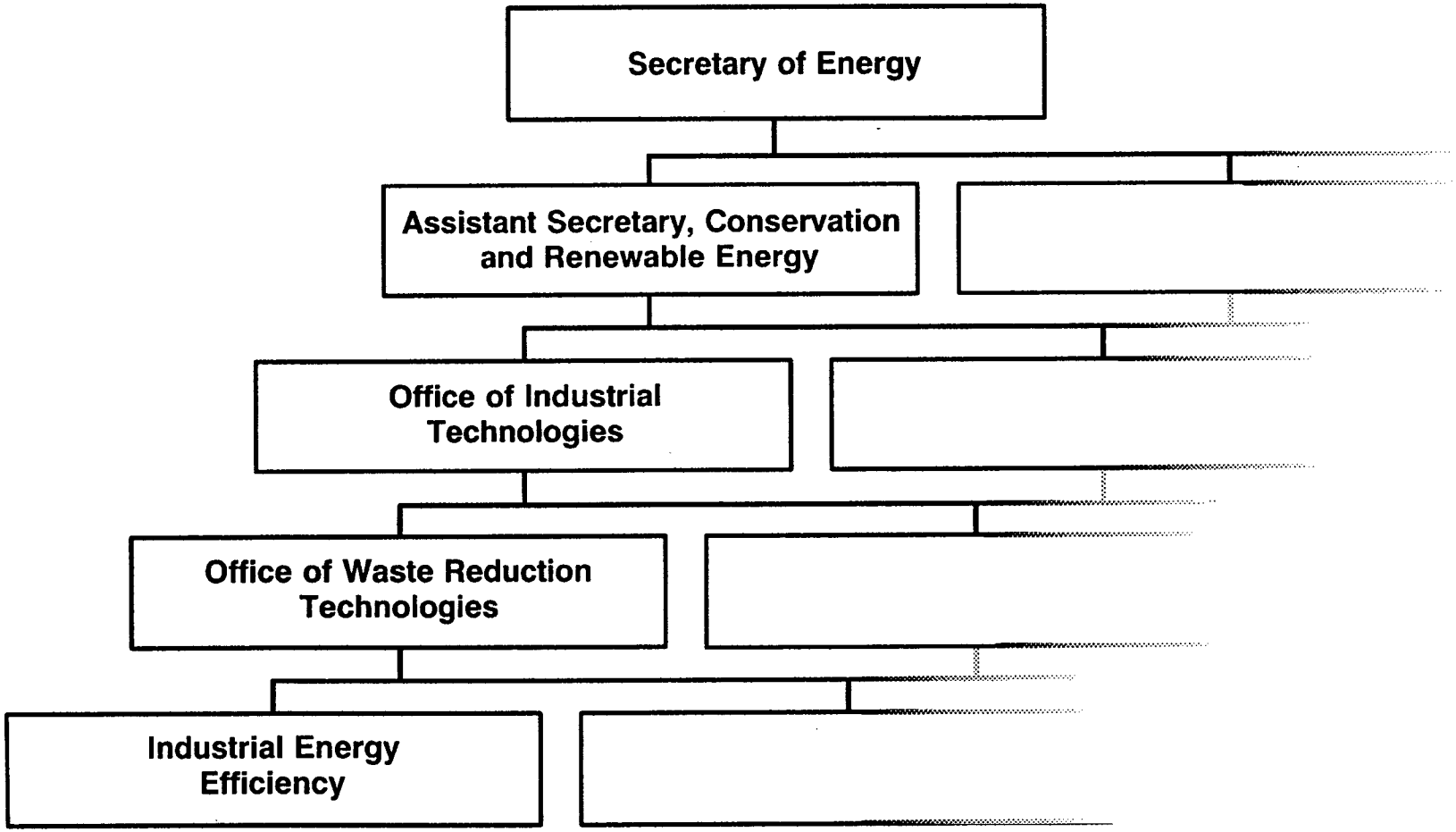
CE50790.01

CFCC PROGRAM GOAL

... TO DEVELOP, IN U.S. INDUSTRY, THE PRIMARY PROCESSING METHODS FOR THE RELIABLE AND COST-EFFECTIVE FABRICATION OF CONTINUOUS FIBER CERAMIC COMPOSITE (CFCC) COMPONENTS FOR INDUSTRIAL APPLICATIONS

CE50611.05

DOE ORGANIZATION



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MOTIVATION

- Save Energy, Increase Efficiency, and Improve Productivity by Enabling a New Generation of Processes and Products Based on CFCC Components
- Establish U.S. Leadership in Manufacture and Use of CFCC Industrial Components

CE42662.03

BASIS

- Higher Temperatures ➡ Higher Efficiencies
- Ability To Design Beyond Capabilities of Available Materials
- Progress in Monolithics
- Desire for "Graceful Failure"

CE42662.04

POTENTIAL INDUSTRIAL APPLICATIONS FOR CFCC COMPONENTS

- Stationary Heat Engines (Primarily Gas Turbine Components)
- Heat Recovery Systems (Air Preheaters, Recuperators, Remelters)
- Advanced Burners/Combustors:
  - Radiant Tube Burners
  - "Catathermal" Combustors
  - Low Temperature Porous Radiant Combustors
- Process Equipment (Reformers, Reactors, Hi-Pressure HXs)
- Waste Incineration (Handling Equip., Furnace Internals, Cleanup)
- Separation/Filtration Systems (Gas Turbine Inlet; Diesel Exhaust)
- Refractory Products (Furnace Linings, Kiln Furniture, Crucibles)
- Structural Components ("Infrastructure" Repair; Niche Markets)

CE42662.06

INDUSTRIAL BENEFITS

- 1.1 Quad Savings in Limited Applications
- 600,000 Ton/Year NOx Reduction
- Multi-Billion Dollar Markets

CE42662.05

PROCESSING ISSUES

- Multiple Approaches
- Immature Technology
- Correspondence to Applications Not Established

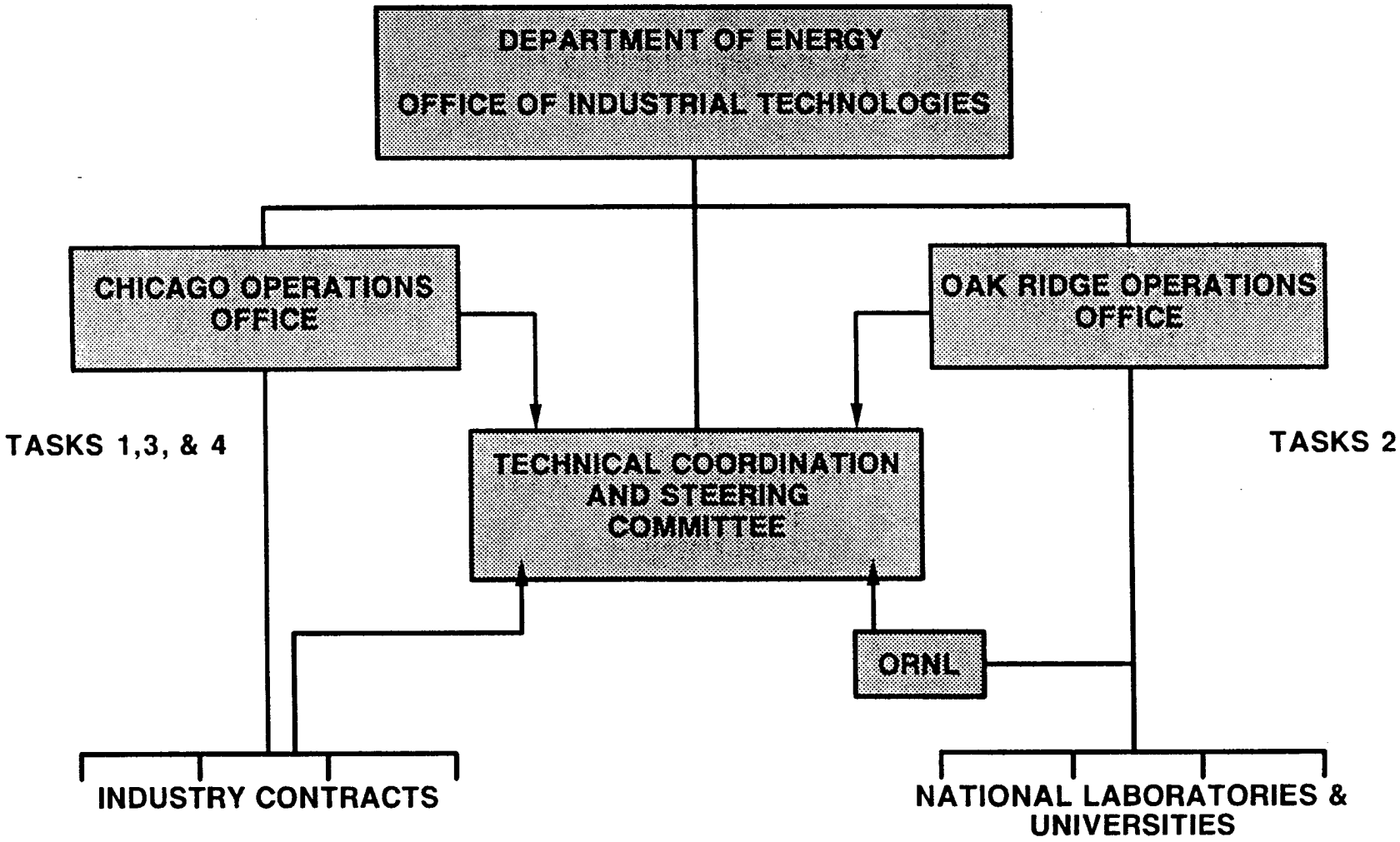
CE42662.08

CFCC PROGRAM OBJECTIVES

- DEMONSTRATE PILOT SCALE PROCESS LINE
- DEVELOP NECESSARY SUPPORTING TECHNOLOGIES
- PROVIDE REPRESENTATIVE COMPONENTS FOR TEST
- NON-REDUNDANT

CE50611.07

STRUCTURE OF THE CFCC PROGRAM



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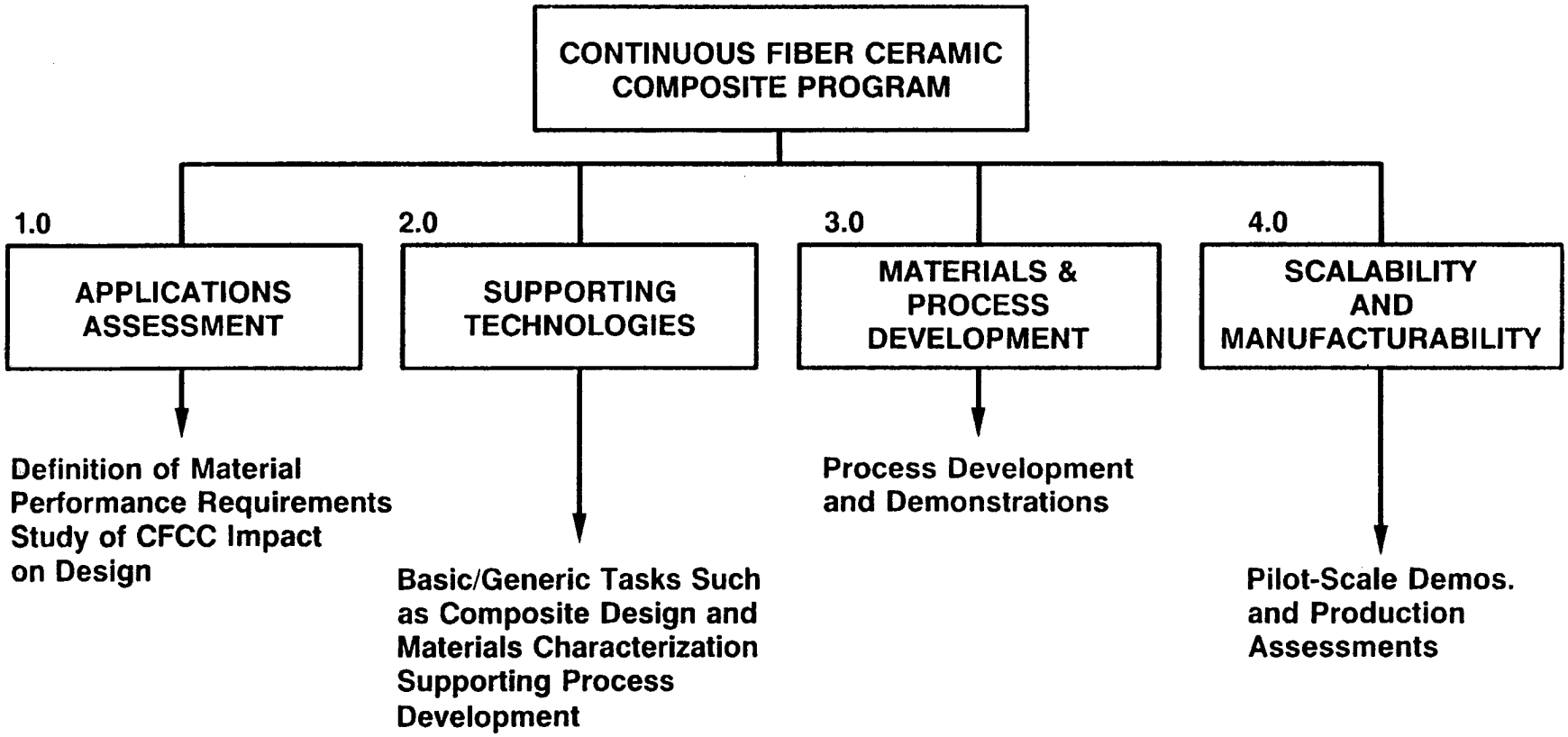
CFCC WORK BREAKDOWN STRUCTURE



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PROGRAM STRATEGIES

- Support Development of a Range of Processes
- Process Development Guided by Application Performance Requirements
- Define Clear Paths to Commercialization
- Encourage "Vertical" Teaming
- Track Foreign Developments
- Protect Intellectual Properties

CE42662.11

PROCESSING METHOD

APPLICANT	CVI/CVD	DIMOX	SLIP INFILTRATION	SOL-GEL	POLYMER PRECURSOR	GAS-SOLID REACTION BONDING	MELT INFILTRATION SILICONIZATION
ALZETA CORP.							
AMERCOM	■						
BABCOCK & WILCOX				■			
DOW CHEMICAL CO.			■				
DOW CORNING					■		
DUPONT ADVANCED COMPOSITES AND FOSTER-WHEELER	■						
DUPONT LANXIDE		■					
GARRETT CERAMIC COMPONENTS							
GENERAL ELECTRIC RESEARCH & DEVELOPMENT							■
TEXTRON						■	

CE50790.06

CFCC PHASES

- PHASE I - EXPLORATORY DEVELOPMENT
- PHASE II - PROCESS ENGINEERING AND COMPONENT DEVELOPMENT
- PHASE III - PILOT SCALE PROCESS DEVELOPMENT

CE50611.16

PHASE I - EXPLORATORY DEVELOPMENT

- 6 - 24 MONTHS DURATION
- TASK ACTIVITIES:

  1.0 APPLICATIONS ASSESSMENT

    1.1 ANALYSIS

    1.2 DEFINITION

    1.3 DESIGN STUDIES

  3.0 MATERIALS AND PROCESS DEVELOPMENT

    3.1 PROCESS FEASIBILITY

CE50611.17

PROJECTED ACHIEVEMENTS - PHASE I

- IDENTIFY APPLICATIONS
- ASSESS FEASIBILITY OF COMPOSITE SYSTEM
- MATCH-UP COMPOSITE SYSTEM WITH APPLICATIONS
- IDENTIFY APPLICATION IMPACTS

CE50790.09

PHASE II - PROCESS ENGINEERING AND COMPONENT DEVELOPMENT

- 2 - 3 YEARS DURATION
- TASK ACTIVITIES:

  3.0 MATERIALS AND PROCESS DEVELOPMENT

  - 3.2 PROCESS ENGINEERING
  - 3.3 COMPONENT FABRICATION AND TESTING
  - 3.4 COMPONENT EVALUATION
  - 3.5 JOINING

CE50611.18

PROJECTED ACHIEVEMENTS - PHASE II

- ESTABLISH PROOF-OF-CONCEPT
- COMPLETE LAB DEVELOPMENT
- IDENTIFY CONTROL/PROCESSING ISSUES
- ACTIVE COMMERCIAL PURSUIT

PHASE III - PILOT SCALE PROCESS DEVELOPMENT

- 3 - 5 YEARS DURATION
- TASK ACTIVITIES:

  4.0 SCALABILITY AND MANUFACTURABILITY

  - 4.1 ANALYSIS
  - 4.2 PRODUCTION ENGINEERING
  - 4.3 PROCESS CONTROLS
  - 3.5 JOINING

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PROJECTED ACHIEVEMENTS - PHASE III

- DEVELOP NECESSARY SUPPORTING TECHNOLOGIES
- PROVIDE REPRESENTATIVE COMPONENTS FOR TESTING
- DEMONSTRATE PILOT SCALE PROCESS LINE

ACHIEVEMENTS TO DATE

- ESTABLISHED DoD / DOE / NASA COORDINATION
- INITIATED ASME BOILER CODE DISCUSSIONS
- DRAFT PROTOCOL FOR HEALTH ASSESSMENT
- INITIATED GENERIC & SUPPORT R&D
- SELECTED INDUSTRY PLAYERS

CE50611.30

PROPOSED MEETINGS

- Working Group Meeting
  (Tasks 1, 2, & 3) . . . . . Chicago, Fall '92

- Technical Reviews . . . . . Cocoa Beach, Jan '93

- Program Review . . . . . Washington, D.C., Winter '93

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OFFICE OF INDUSTRIAL TECHNOLOGIES

Continuous Fiber Ceramic Composite Program

Office of Waste Reduction, Industrial Energy Efficiency Division

INTRODUCTION

Advanced materials have been cited by the National Research Council as critical to the continued economic growth and prosperity of U.S. industry. After examining the role of materials in eight major U.S. industries (which collectively employed 7 million people and had sales of $1.4 trillion in 1987), they found a universal need for materials that were lighter, stronger, more corrosion resistant, and capable of performing at elevated temperatures.[1] Continuous fiber ceramic composites (CFCCs), which have high tensile strength in addition to high-temperature capability and corrosion resistance, offer the potential to satisfy material demands across a broad spectrum of industrial applications.

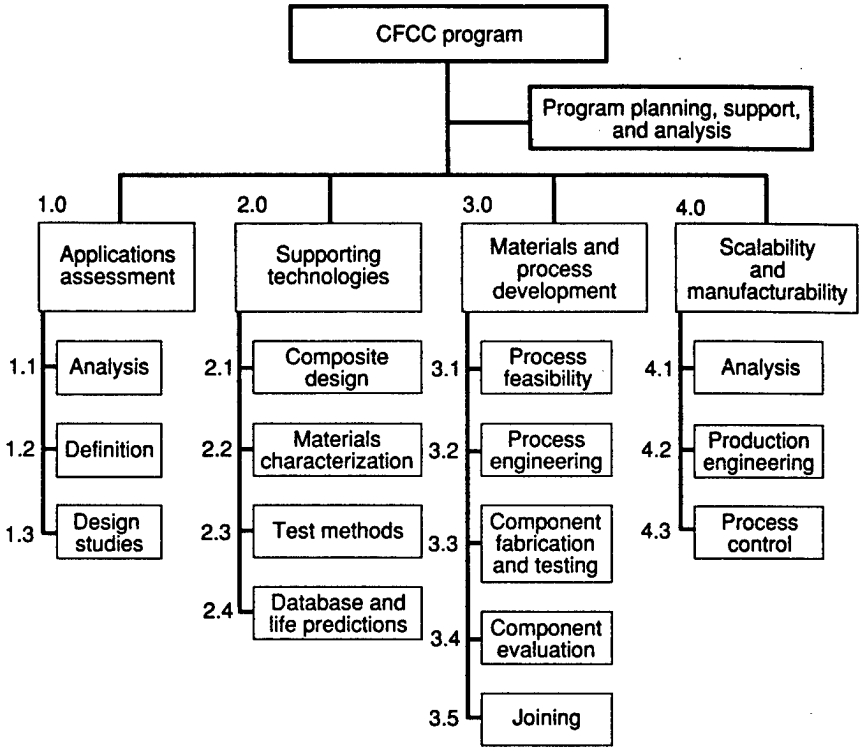

Figure 1 Structure of research tasks in the CFCC Program.

Successful deployment of CFCC components in industry can provide substantial benefits in the form of improved energy efficiency and reduced environmental impacts. CFCC components can be used in systems designed for the efficient high-temperature incineration of municipal and industrial waste streams, which are generated at alarming rates every year.[2] Application of CFCCs in the demanding environments of incineration systems could facilitate the reduction of the waste disposal burden, decrease the toxicity of airborne pollution, and displace fuel consumption through useful energy recovery from waste burning. The potential for energy saved through power generation and heat recovery from waste streams has been estimated at more than 0.5 quadrillion Btus per year. CFCCs are also being considered for use as components in gas turbines. By allowing turbines to operate at higher inlet temperatures, the use of CFCCs could increase thermodynamic energy-conversion efficiency by as much as 4 percent, which is equivalent to a 13-percent reduction in fuel consumption. Over a 15-year time span, this improvement in efficiency could provide an estimated fuel cost savings of $12.7 billion.[3]

Knowledge of how to design, fabricate, and test CFCC components is at a preliminary stage, and a substantial effort on the part of both industry and government is needed to develop the necessary characterization, processing, and evaluation techniques. A comprehensive strategy and plan to conduct a major research effort on the potential of CFCCs for application in industry was completed by the Office of Industrial Technologies (OIT) in 1990. The plan reflects the major challenges that must be overcome before the significant potential of these materials can be realized. With input from more than 120 companies, universities, and government laboratories, four principal tasks were designated as the basis for the CFCC Program. The tasks include application assessments, supporting technologies, materials and process development, and scalability and manufacturability. The structure

of each research task is shown in Figure 1 (Ref. 4). The ultimate goal of the Program is to develop, within U.S. industry, the capability to produce reliable and cost-effective CFCC components for industrial applications. Successful development of a CFCC industry in the United States could have substantial impacts—not just on industrial energy efficiency, but on the environmental quality and economic health of the United States.

ENERGY, ENVIRONMENTAL, AND ECONOMIC BENEFITS OF CFCCs

Role of Ceramics in Overcoming Materials Limitations

U.S. industry has a critical need for materials that are lighter, stronger, more corrosion resistant, and capable of performing in elevated-temperature environments. Many opportunities for improvements in energy efficiency, productivity, and waste utilization are not pursued because of the lack of availability of materials capable of handling the severe operating conditions often present in industrial processes. A number of advanced materials have been developed with varying potential to meet these requirements, such as metal-matrix composites, carbon composites, polymers and polymer composites, and ceramics. While many of these materials compete with each other under certain operating conditions, ceramics offer the greatest potential where a combination of reduced weight, high-temperature strength, and environmental stability is needed.

Although many ceramics perform well at considerably higher temperatures than conventional metal alloys, they have a tendency to brittleness and as a result can undergo catastrophic failure in service. Failure occurs when the part is subjected to sufficient stress to propagate the microscopic flaws or cracks that are always present in ceramic parts. Even with the most careful processing and fabrication methods, a statistical distribution of flaws will always exist. Consequently, uncertainty over part reliability remains a key limitation in the use of ceramics for some applications. The failure of a component in an industrial process can result not only in costly downtimes (with major portions of a process or a plant shut down while awaiting repair or the arrival of replacement parts), it can result in loss of product (losses can reach into the hundreds of thousands of dollars if the failure of a forging furnace component were to cause a reject load of aircraft forgings).

Considerable effort has been expended in the United States, Europe, and Japan to resolve the problem of brittle failure through research to increase the fracture toughness of ceramics. Better processing to control flaws and toughening of the ceramic through the incorporation of whiskers (small single crystals) have resulted in some material improvements. However, fracture toughness values for both monolithic ceramics and whisker-toughened ceramic composites remain low, and the potential for catastrophic failure persists.

Why Continuous Fiber Ceramic Composites?

The concept of ceramic composites, despite some limitations, has permitted greater flexibility in developing materials that exhibit strength combined with increased toughness. By incorporating second-phase constituents (such as whiskers, multifilament yarns, or large-diameter monofilaments) toughness can be improved substantially. In the case of CFCCs, a long fiber, such as carbon, glass, or ceramic fiber, is embedded in a matrix of monolithic ceramic material. The matrix material contributes chemical and thermal stability while the fiber contributes high-temperature strength and toughness. Like a traditional ceramic, the matrix fails in a brittle manner through crack propagation at localized flaw sites. The fibers provide toughness by arresting cracks, bridging cracks, and a phenomenon called "pull-out," shown in Figure 2. When a crack reaches a fiber, it must divert around the fiber, a process which consumes more energy than linear crack growth. Typically there is not sufficient energy for the crack to change direction and bypass the fiber, and crack growth will be arrested at the point of contact. If the crack is propagated by sufficient energy to deflect around the fiber, the fiber can bridge the crack and thus hold the matrix together. Finally, if the forces upon the composite material are sufficient to fail the matrix, the fibers must be pulled out before the composite can separate. This "pull-out" of fibers from the matrix requires additional energy, and as the fibers continue to carry the load, a non-catastrophic failure mode or metal-like behavior results.

Figure 2 Fiber pull-out imparts increased fracture toughness to ceramics.

The arresting and bridging of cracks and fiber pull-out are the principal mechanisms responsible for the increased fracture toughness and markedly decreased sensitivity to

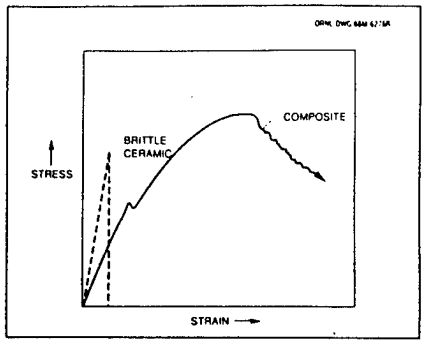

Figure 3 CFCCs exhibit greater fracture toughness when compared with monolithic ceramics.

flaws exhibited by CFCCs (see Figure 3). Fracture toughness values for CFCCs from 15 to 25 MPa $m^{1/2}$ have been measured, representing a considerable increase over that of monolithic ceramics (3 to 6 MPa $m^{1/2}$) and whisker/particulate-toughened ceramics (8 to 12 MPa $m^{1/2}$). For designers concerned with the reliability of ceramic parts, the increased toughness of CFCCs should provide added assurance when considering the incorporation of such materials into equipment design.

The defense and aerospace industries have already recognized the potential of CFCCs and have undertaken development efforts to use these composites in high-performance aircraft engines. As part of the CFCC Program strategy, a primary applications-and-needs assessment was conducted to identify opportunities for CFCC use in industry. Based on this assessment, a list of industrial applications was generated for potential development under the CFCC Program. These applications, summarized in Table 1, are likely to produce considerable improvements in energy efficiency and help to reduce the environmental impacts associated with industrial energy use and waste streams. The potential energy, environmental, and economic benefits of several CFCC applications are shown in Table 2. These applications alone could save more than 1 quadrillion Btus of energy per year and reduce NO_x emissions by 600,000 tons per year. Numerous other environmental benefits could accrue from the use of CFCCs in high-temperature incineration systems (e.g., municipal and industrial waste streams) and pollution abatement technology.

Technical and Economic Limitations of CFCCs

Although CFCC research conducted by ceramic suppliers, universities, and government laboratories is encouraging, for the most part it has not addressed material systems and fabrication processes targeted to industry applications. Cost competitiveness and product reliability are the primary concerns of the private sector in pursuing CFCCs for commercial use. The development of processing technology for industrial applications and the related needs for design, standardization, joining, and prototype demonstration will be necessary to ensure that this promising material reaches its market potential. The CFCC Program Plan stresses the importance of U.S. leadership in the development of CFCC processing and related technologies and is committed to building the required technology base.

Maintaining a Competitive Position

CFCCs offer significant potential for overcoming a number of material design limitations that currently restrict the development of advanced industrial technologies. Before this potential can be realized, however, a sustained R&D effort will be needed to establish the capability to analyze, design, and process CFCC materials. Progress in ceramics development over the past decade has prompted the design of new equipment that takes advantage of the unique properties of ceramics, such as high surface hardness, resistance to corrosive environments, and strength retention at high temperature. The CFCCs add a new dimension, that of toughness, to these unique properties. The near availability of these materials has led to the design of advanced systems with increased thermal efficiencies made possible by operation at elevated temperatures and pressures. One such system, termed a high-pressure heat exchange system (HiPHES), is under development by Solar Turbines, Inc. It is expected to lead to significant performance increases in industrial hazardous waste disposal.

Although the ceramics industry, its materials suppliers, and end-users recognize the potential of CFCCs, industry involvement in CFCC development is currently discouraged by the lack of a technology base and the expectation of an extended gestation period preceding market maturation. In addition to modest Department of Energy (DOE)-supported R&D efforts in this area, research in the United States on CFCCs has to date consisted primarily of limited research conducted by the National Aeronautics and Space Administration and the Department of Defense to develop advanced materials for military and aerospace applications. For the most part, the focus of this research has not been directed toward material systems best suited for industrial applications. The CFCC development activities of these programs represent about $17 million per year of Federal spending.[5] Defense and aerospace industries in the United States are also spending about $60 to $70 million per year of private funding on CFCC development.

Several other countries have also embarked on significant government-supported CFCC development efforts. The governments of our principal trading partners and industrial competitors (including Japan, France, Germany, and the United Kingdom) are currently spending between $5 and $35 million per year to develop CFCC materials.[6] Efforts in Japan focus on industrial applications, while those

TABLE 1

Recommended Industrial Applications for the CFCC Program

CFCC Product Area/Typical Examples	Likely Industrial Markets
Stationary engines Combustors, liners, wear parts	Primarily high-temperature gas turbines (especially combustors); possibly adiabatic diesels, S-I engines
Heat recovery equipment internals Air preheaters, recuperators	Any indirect heating uses: energy-intensive industrial processes (aluminum remelters, steel reheaters, glass melters)
Burners and combustors Radiant tube burners	Potentially any indirect-fired, high-temperature and/or controlled-atmosphere heating/melting/heat-treating use
Burners and combustors Catathermal combustors	Low-NO_x clean fuel heating applications—including gas turbine combustors, industrial process heat
Burners and combustors Low-temperature radiant combustors	Low-NO_x clean fuel heating applications—including small scale (space heating) and large scale (industrial processes)
Process equipment Reformers, reactors, high-pressure heat exchangers	Chemical process industry; petroleum refining; corrosives handling and storage
Waste incineration systems Handling equipment, furnace internals, clean-up	Conventional MSW/RDF facilities; advanced toxic or hazardous waste facilities; with/without energy recovery
Separation/filtration systems Filters, substrates, centrifuges	Gas turbine, combined cycle, and IGCC configurations; diesel exhaust particle traps; molten-metal filters; sewage treatment
Refractories and related products Furnace linings, crucibles, racks, kiln furniture	High-temperature industrial heating/melting/heat-treating processes (primarily retrofit applications)
Structural components Beams, panels, decking, containers	Possible niche uses for EMI shielding, corrosive/abrasive environments, fire or missile protection; infrastructure repair

TABLE 2

Potential Impact of Several CFCC Applications

Selected Examples	Energy Savings	Environmental Benefit	Economic Impact
Gas turbine power generators	0.4 quad/yr in 2010	0.3 million ton/yr NO_x emission reduction	$1.3 billion/yr energy savings
Mature technology generating 350 billion kWh/yr of electrical energy	13% fuel savings for 135 million kW capacity additions 2000-2009 (DOE baseline predictions)	75% reduction in NO_x over conventional technology	$12.7 billion savings projected for 2000-2014
High-pressure heat-exchanger systems Power generation/Steam reforming/ Heat recovery	0.6 quad/yr	0.1 million ton/yr NO_x emission reduction from reduced fuel use	$2.3 billion/yr energy savings
Radiant fire tubes Metal heat drying/coated coil drying	0.06 quad/yr 2% reduction of 0.235 quad for ferrous heat drying and coil drying		$0.2 billion/yr energy savings
Aluminum Remelting Heat recovery	0.3 quad/yr 50% reduction of 0.5 quad for aluminum recycle		$0.1 billion/yr energy savings
Radiant burners		0.2 million ton/yr NO_x emission reduction 25% reduction of NO_x emissions for 6 quads of industrial fuel consumption	

in France, the Federal Republic of Germany, and the United Kingdom are aimed at the development of military and aerospace components.

France has exhibited the strongest commitment to CFCC development. It is currently supporting strong development efforts in ceramic fibers, processing, characterization, and high-temperature behavior, primarily for military and aerospace applications. France is considered the leader in the use of chemical vapor infiltration techniques to form continuous fiber composites and has licensed this technology in the United States. Japan is considered the world leader in the development of ceramic fibers, and R&D in that country is directed for the most part at opening new markets based on fiber technology. Japan aggressively pursues industrial applications to create new markets for its fibers and has recent patent applications that cite CFCCs for a variety of biomedical, gas turbine, refractory, catalyst carrier, and compressor part applications.

Foreign activity in CFCC development could have serious implications for the United States. Japan currently produces the best fibers in the world—and the best component fabrication and processing technologies could follow. Foreign efforts to develop both CFCCs and ceramics could provide those countries with the capability to dominate CFCC markets into the 21st century. Foreign control of CFCC markets would almost certainly have a negative impact on the U.S. balance of trade, since the biggest markets for ceramic fibers, as well as for ceramics and ceramic composites, are in the United States.

The market for industrial applications of CFCCs will be developing over the next five to ten years; therefore, at this time, the benefits of developing a strong industrial composite technology base are difficult to project and quantify to a substantial impact number. To maintain a competitive edge, however, it is vital that the capability to manufacture CFCCs is developed *now*, before the needs and benefits are fully perceived. Five to ten years from now, when the potential of these materials has been proven and it is time to produce components on a commercial scale, the technologies must be in place to fabricate CFCC components, to test their reliability for in-service performance, and to adapt processes for new applications as needed.

Of particular importance is the ability to produce a CFCC component in "near net shape." Because of the very high hardness of ceramics, any post-process forming is expensive and time consuming (i.e., diamond bit tools are typically used to machine ceramic surfaces). Therefore, the CFCC component fabrication process becomes more economic and time-efficient the nearer it approximates the desired end shape. Near net shape is desirable in that it saves the end-user both time and money; however, it also requires that the component designer accurately specify the dimensions and design tolerances of the part to the fabricator. If the United States does not develop a domestic, competitive capability to produce CFCC components to near net shape, U.S. component designers will be forced to provide the descriptions of their proprietary designs to foreign ceramic companies. Particularly in Japan, with its system of large multi-company combines (referred to as *keiretsu*), there is no guarantee that those designs will remain proprietary. Through part specification, technology transfer to foreign competitors will have taken place. If the United States does not develop the capability for reliable, cost-effective processing of CFCCs, then that capability will almost certainly be developed by foreign competitors. At this point, the possible future technologies that could be lost to foreign competitors may not even be on the drawing boards, and the impact of that loss cannot be projected. Supporting a strong CFCC R&D effort in the United States *now* will accelerate the establishment of the necessary domestic technology base, will reduce the risks to industry in developing markets for these composites, and will increase the probability of U.S. leadership in this area. This premise is a key strategy of OIT's CFCC Program: By striking now, before markets are fully identified and the need is obvious, the United States will be assured of the competitive lead.

CFCC PROGRAM STRATEGY

Drawing on the Collective Capabilities of Industry, the National Laboratories, and Academia

The threat of foreign competition, the desire to achieve and maintain international preeminence in ceramic composite technology, and the opportunity to enable the development of advanced industrial technologies are all strong incentives for future CFCC R&D efforts. These critical concerns can be addressed by implementing a program that judiciously takes advantage of the combined capabilities of industry, academia, and government.

Industry's role in CFCC development is vital. In recognition of this, a crucial element of the CFCC Program will be the strategic involvement of industry in guiding technology development in directions that are conducive to commercialization and practical application. As a basic premise, this involvement will require that R&D work be performed *in industry* to ensure successful technology transfer and the development of a competitive U.S. capability to produce CFCCs. Substantial participation by industry will also position the private sector to set a research agenda based primarily on economic and performance criteria. Industry facilities for simulated and in-service testing will provide feedback on the level of improvements achieved and define remaining areas of deficiency. Pilot-scale dem-

onstration will be used to assess scalability, manufacturability, and production economics. The national laboratories and universities will play a responsive role—as industry develops the composite technology, national labs and universities will provide critical technical support (e.g., composite design, material characterization, life prediction, and test methodologies).

Role of Industry Cost-Sharing

Industry is expected to make a substantial commitment to the efforts undertaken by the CFCC Program. However, the extent of industry's ability to cost-share will vary. The Federal role in providing support for CFCC R&D is already well established. The role of industry in providing cost-sharing is more complex, being a function of the perceived investment risks, the nearness of the technology to commercialization, and the ability of the industry to accommodate the costs associated with long-term R&D. Cost-sharing is viewed by the Federal Government, however, as a sound indication of industry intent. Participants in the CFCC Program will be expected to contribute some of the costs of performing the R&D (at least 10 percent, initially). As technical feasibility becomes better established, the prospects for successful commercialization will increase and so will expectations for industry cost-sharing (50 percent or more).

Establishing Industry Alliances

An important factor in the success of the CFCC Program will be the early development of a strong industry consortia to provide essential input for program definition. The informal alliance between U.S. ceramic composites companies and the U.S. Advanced Ceramics Association is an important first step. Cohesiveness and cooperation among fiber suppliers, composite manufacturers, and end-users will also be necessary to understand and meet the technical needs for practical applications of CFCCs. This interaction is a reflection of modern industry perspective, where the need to maintain contact with the customer at the end of the product delivery line is recognized as an important element of success. This has become particularly important for industries that are always one step removed from the end product (such as the raw chemicals manufacturer who produces feedstocks for other manufacturers, who eventually produce final products).

To maintain the critical alliance between all the participants in the chain, from fiber producer to composite manufacturer to end-user, the process development work for both near- and far-term applications will be conducted by heterogeneous R&D teams. These teams will be comprised of a composite manufacturer, in most cases the prime contractor, plus one or more fiber manufacturers, fiber coaters, preform makers (who wind, weave, or braid the fiber to a specific architecture), and end-users. Supported by universities and national labs, these teams will provide the comprehensive approach necessary to ensure the success of the program.

As part of its strategy to encourage strong industry involvement, DOE will be supporting the development of information dissemination tools designed to raise the level of communication between DOE and CFCC researchers, manufacturers, and end-users. A computerized information reporting system is being planned to effectively expedite the flow of information between DOE and CFCC Program R&D participants. The system, which will allow project data to be transmitted directly to DOE, will establish a route to facilitate effective and timely response to R&D progress. A computerized bulletin board will also be developed and made available for use by interested parties from the government, industry, and academia. The bulletin board will provide a vehicle for keeping the ceramics/composites community apprised of the progress of DOE-supported CFCC research. It will also help to raise the general level of communication on the topic of CFCCs by keeping industry abreast of workshops, technology-transfer activities, and other areas where CFCC R&D may have an impact.

Beyond the Federal Role

It is important to recognize that the provision of Federal funding has boundaries—it can serve as the catalyst for an extended research and development effort, but it cannot guarantee that the end result will be a new, commercially viable technology. In short, the successful completion of a sustained, long-term research effort requires more than just financial support—it also demands stamina, flexibility, and a great deal of foresight. From the earliest stage, the program must be more than a research effort—it must include the factors that, from an end-user's perspective, will ensure commercialization farther down the road. A strong reliance on performance requirements and a sharp focus on applications will provide successful guidance in targeting R&D. This approach will ensure that the technology base is being built to support CFCC development all the way to the marketplace. A 10-year program is visualized for the successful development of CFCCs. As progress is made, it is hoped that the technical limitations preventing the widespread use of these materials will be eliminated.

STATUS OF OIT CFCC PROGRAM ACTIVITIES

Proposals from industry to develop CFCC processing technologies are currently under review at the DOE Field Office, Chicago. Industry research initiatives resulting from these proposals are expected to be under contract by the end of the calendar year. Research to provide generic and supporting technolo-

gies to the CFCC industry is already under way at the Oak Ridge National Laboratory and Pennsylvania State University. Additional supporting research is being planned at the Argonne National Laboratory, the Idaho National Laboratory, and the National Institute of Science and Technology.

CONCLUSION

CFCCs have been recognized as a new class of ceramic materials with the high-temperature stability, corrosion resistance, and toughness necessary for a wide range of new and challenging applications. The energy, environmental, and economic benefits that could be realized from the successful development of markets for CFCCs are strong incentives for both government and industry to conduct a major R&D effort. In addition to the DOE CFCC Program, organized support for advanced ceramic research is being provided by trade groups like the Aerospace Industries Association (AIA) and the U.S. Advanced Ceramics Association (USACA). An effective CFCC development effort with participants from government, academia, and the private sector will encourage U.S. industry to take advantage of the many industrial, automotive, and aerospace applications of this innovative new material.

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DOE NEWS

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FOR IMMEDIATE RELEASE
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DOE/35 COMPANIES, UNIVERSITIES AND NATIONAL LABS TO IMPROVE CERAMIC COMPOSITE TECHNOLOGY

New materials that could significantly improve the efficiency and productivity of industrial manufacturing processes are the focus of a new 10-year, $100 million program of the U.S. Department of Energy (DOE). DOE has announced plans to work with 10 research teams, encompassing 35 companies, universities and national laboratories, to develop continuous fiber ceramic composites (CFCCs).

DOE's Assistant Secretary for Conservation and Renewable Energy, J. Michael Davis said, "U.S. industry has a critical need for materials that are lighter, stronger, more corrosion resistant, and capable of performing at very high temperatures. Ceramic composites may be the answer.

"The CFCC program is another example of a joint effort that draws on the combined capabilities of industry, academia and government," Davis added. "Successful development of these materials would have a positive impact, not only on industrial energy efficiency, but on U.S. competitiveness and job creation.

(MORE)

R-92-126

Deputy Assistant Secretary for Industrial Technologies, Alan J. Streb, pointed out that industrial applications for the CFCC program range from heat exchangers to critical components for high-temperature gas turbine engines. Streb said the potential for annual energy savings runs in the billions of dollars.

The 7.6 million dollars in contracts will be let to the 10 industry teams for Phase One of the CFCC program. To last from 15 to 24 months, Phase One will assess the feasibility of the composite process, establish process development goals and set performance targets.

(Attached is the list of the ten industry team leaders).

- DOE -

R-92-126

INDUSTRY TEAM LEADERS FOR CFCC PHASE ONE

Allied Signal (Garrett Ceramic Components)
Torrance, CA

Alzeta Corp.
Santa Clara, CA

Amercom, Inc. (an Atlantic Research Corp. Company)
Chatsworth, CA

Babcock and Wilcox
Lynchburg, VA

The Dow Chemical Company
Midland, MI

Dow Corning Corp.
Midland, MI

Dupont-Lanxide Composites, Inc.
Newark, DE

E.I. Dupont de Nemours and Company, Inc.
Wilmington, DE

General Electric Corporate Research Division
Schenectady, NY

Textron Specialty Materials
Lowell, MA

STRATEGIC DEFENSE INITIATIVE OFFICE INNOVATIVE SCIENCE AND TECHNOLOGY PROGRAMS IN STRUCTURAL CERAMICS

DR. WILLIAM HONG

Fiber-Reinforced Glass and Glass-Ceramic Matrix Composites for Strategic Defense Applications

United Technologies Research Center

OBJECTIVE

- TO DEVELOP THE CONCEPTS AND SCIENTIFIC UNDERSTANDING NECESSARY TO ENHANCE THE READINESS OF FIBER REINFORCED GLASS AND GLASS-CERAMIC MATRIX COMPOSITES FOR STRATEGIC DEFENSE APPLICATIONS

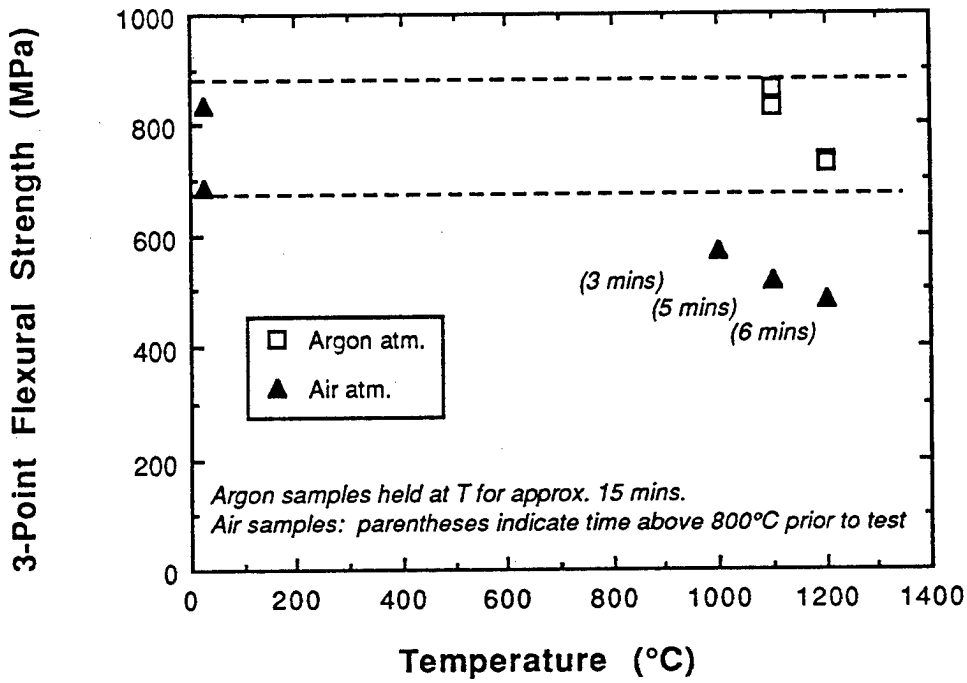
FUTURE EMPHASES-

- IMPROVED OXIDATION RESISTANCE AT TEMPERATURE (USE OF CERAMIC FIBERS, IMPROVED INTERFACES)

RESULTS/HIGHLIGHTS

- C-fiber reinforced BMAS glass-ceramic matrix composites evaluated to 1200°C
- High thermal conductivity C fibers successfully incorporated into glass and glass-ceramic matrices
- Tensile fatigue of various C fiber/glass specimens evaluated; peak fatigue stresses ≈75-80% of UTS demonstrated for up to 1M cycles
- C/glass tubes tested in compression to evaluate for space truss applications; comparable to C/C materials

UTRC



The flexural strength of a unidirectionally reinforced FT700/BMAS composite was assessed in both air and argon at temperatures up to 1200°C. When tested in argon, the composite exhibited no drop in strength up to 1200°C and showed no signs of any plastic deformation in the matrix, indicating that the structural integrity of the matrix at these temperatures is excellent. (Limited flexural creep testing has indicated that these materials can maintain a load of at least 240 MPa at 1150°C for one hour without any evidence of creep.) Samples tested in argon were held at temperature for approximately 15 minutes prior to testing.

Samples evaluated in air were inserted into a hot furnace (800°C) and then ramped to the test temperature at ~50°C/min. The load was applied once temperature was reached. The numbers in parentheses indicate the approximate time that the sample was above 800°C prior to the load being applied. It is clear that strength degradation due to oxidation of the carbon fiber begins to occur rapidly under these conditions. However, samples maintained greater than 70% of their strength even at the maximum test temperature of 1200°C. This suggests that these materials may be suitable for one-time short-term applications such as missile components.

STRATEGIC DEFENSE INITIATIVE OFFICE
INNOVATIVE SCIENCE AND TECHNOLOGY
PROGRAMS IN STRUCTURAL CERAMICS

Reaction-Formed Silicon Carbide Composites

Massachusetts Institute of Technology

OBJECTIVE

- TO INVESTIGATE LIQUID-PHASE REACTION PROCESSING OF SiC CERAMIC COMPOSITES AND EXAMINE METHODS TO IMPROVE THEIR MECHANICAL PROPERTIES AND ULTIMATE USE TEMPERATURE

APPROACH:

- CARBONACEOUS PREFORMS ARE INFILTRATED WITH LIQUID Si ALLOYS AND REACTED TO FORM SiC
- SUPPRESSION OF TEMPERATURE-DEPENDENT C-Si REACTION RATES NECESSARY TO MINIMIZE DAMAGE TO REINFORCEMENTS

RESULTS/HIGHLIGHTS

- Microstructure evolution of polymer-derived C preforms characterized; goal is refinement of final composite microstructure through control of preform porosity and morphology
- Additives being investigated to control amount of residual Si by formation of refractory silicides; additions of Mo and B also found to significantly lower melt infiltration temperature
- Composite fabrication using these approaches and their characterization is underway

SDIO INNOVATIVE SCIENCE AND TECHNOLOGY
FUNDING FOR STRUCTURAL CERAMICS PROGRAMS

	FY 91	FY 92	FY 93
UTRC	400k	400k	400k
MIT	130k	130k	130k