NATIONAL EDUCATORS' WORKSHOP
NEW: Update 91

STRUCTURAL CERAMICS

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Oak Ridge National Laboratory

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PERSPECTIVES ON WHERE MATERIALS ARE GOING


IMPACT OF MATERIALS

IN 1982, DAMAGES DUE TO MATERIAL FAILURE WERE GREATER THAN $200 BILLION ANNUALLY - THIS APPROACHES THE ANNUAL FEDERAL DEFICIT. (M. Cohen in Advanced Materials Research, NAS/NAE, 1987)

COMPETITIVENESS AND QUALITY OF LIFE IN THIS DECADE AND 21st CENTURY WILL DEPEND UPON ADVANCES IN MATERIALS. (paraphrase of R. Chianelli, MRS Bulletin, August, 1990)

LEADERS IN MATERIALS TECHNOLOGY WILL DOMINATE THE MARKETPLACE IN THE 21st CENTURY. (Panel discussion at Int'l Symp. Basic Technologies for Future Industries, Kobe, Japan, March 1989)
EVOLUTION OF MATERIALS SCIENCE AND ENGINEERING

DATE

10,000 BC  5000 BC  0  1000  1500  1800  1900  1940  1960  1980  1990  2000  2010  2020

GOLD  COPPER  BRONZE  IRON  METALS  CAST IRON  STEELS  ALLOY STEELS  HIGH PERFORMANCE ALLOYS  MICROALLOYED STEELS  INTERMETALLIC COMPOUNDS

POLYMERS
WOOD  SKINS  FIBERS  PAPER  GLUES  RUBBER  LIGHT ALLOYS  SUPER ALLOYS  TITANIUM ZIRCONIUM ETC.

COMPOSITES
BONE  STRAW-BRICK  STONE  FLINT  POTTERY  GLASS  CEMENT  BAKELITE  NYLON  HIGH TEMPERATURE POLYMERS  HIGH STRENGTH POLYMERS  CERAMIC COMPOSITES

CERAMICS
REFRACTORIES  PORTLAND CEMENT  FUSED SILICA  CERAMIC CORNING TOUGH ENGINEERING CERAMICS

CONDUCTING POLYMERS  HIGH PERFORMANCE POLYMERS  METAL-MATRIX COMPOSITES  ACRYLIC PLASTICS  POLYESTER EPOXIES  KEVLAR
COMMODITY METALS AND PLASTICS HAVE PASSED DEMAND PEAK AND USAGE IS DECLINING
THERE IS A CHANGE IN BASIC MATERIAL USE

- **SUBSTITUTION OF ONE MATERIAL FOR ANOTHER HAS SLOWED THE GROWTH OF DEMAND FOR PARTICULAR MATERIALS**

- **DESIGN CHANGES HAVE INCREASED THE EFFICIENCY OF MATERIALS USE**

- **SATURATION OF MARKETS WHICH WERE PREVIOUSLY EXPANDING HAS OCCURRED**

- **LOW MATERIALS CONTENT IN PRODUCTS FOR NEW MARKETS, PARTLY BECAUSE OF THE COST OF HIGHER PERFORMANCE MATERIALS**
MARKET DEMAND IS NOW LIGHTER, STRONGER, MORE DURABLE MATERIALS

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<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
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(Materials Science and Engineering for the 1990s)
MODERN MATERIALS 50X HIGHER STRENGTH-TO-DENSITY RATIO THAN CAST IRON
MODERN MATERIALS ENABLE HIGHER ENGINE OPERATING TEMPERATURES
MATERIALS ENABLED EXPONENTIAL INCREASE IN CUTTING TOOL SPEED
THE PORTFOLIO OF ADVANCED MATERIALS

- CERAMICS
- POLYMERS
- METALS
- MMC
- PMC
- CMC

REINFORCEMENTS
STRUCTURAL CERAMICS ARE LEADING CANDIDATES FOR MANY APPLICATIONS

CERAMICS
Inorganic, nonmetallic materials processed or consolidated at high temperatures

TRADITIONAL
Based primarily on natural raw materials of clay and silicates
- Clay products
- Glass
- Cement

ADVANCED*
Include artificial raw materials, exhibit specialized properties, require more sophisticated processing
- Structural
- Electronic
- Optical

* Fine ceramics, Engineered ceramics, New ceramics, Value-added ceramics
THE APPEAL OF STRUCTURAL CERAMICS IS EASY TO UNDERSTAND

<table>
<thead>
<tr>
<th>Desired Characteristic</th>
<th>Light with compressive strength &gt; or = metals</th>
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<tr>
<td>Light / strong</td>
<td>Withstand extreme temperatures</td>
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<tr>
<td>High temp resist.</td>
<td>Chemically inert, hard, abrasion resistant</td>
</tr>
<tr>
<td>Corrosion resist.</td>
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<tr>
<td>Efficient processing</td>
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STRUCTURAL CERAMICS ARE A RESULT OF A FEW KEY DEVELOPMENTS

<table>
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<tr>
<th>MATERIALS</th>
<th>TRADITIONAL + DEVELOPMENT = ADVANCED</th>
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<tbody>
<tr>
<td>ANALYSIS</td>
<td>NATURAL SYNTHETIC MATERIALS PREPARATION SYNTHETIC (CONTRIVED)</td>
</tr>
<tr>
<td>PROCESSING</td>
<td>OPTICAL, MACRO XRD, ELECTRON MICROSCOPY MICRO-ANALYSIS</td>
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<tr>
<td></td>
<td>HISTORIC, TOLERANT RELATIONS OF PROCESSING &amp; PROPERTIES UNIFORM CHEMISTRY &amp; STRUCTURE</td>
</tr>
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</table>

1800 1930 1950 1990
SOPHISTICATED MICROSCOPY ALLOWS DETERMINATION OF MICROSTRUCTURE
MECHANICAL PROPERTIES CAN BE DETERMINED AT THE MICROSTRUCTURAL LEVEL
TENSILE TESTER—SPECIALIZED EQUIPMENT FOR DETERMINING CERAMIC MECHANICAL PROPERTIES
ADVANCES IN CERAMICS WILL BE BASED ON ATOMISTIC MODELING, TAILORED MICROSTRUCTURE AND SOPHISTICATED PROCESSING
TOUGHENING IS REQUIRED TO MAKE CERAMICS VIALBE FOR STRUCTURAL APPLICATIONS

- **Metals**
  - $K_{IC} = 25 \text{ MPa} \sqrt{\text{m}}$
  - $\sigma_f = 987 \text{ MPa}$ (141 ksi)

- **Toughened Ceramics**
  - $K_{IC} = 10 \text{ MPa} \sqrt{\text{m}}$
  - $\sigma_f = 395 \text{ MPa}$ (55 ksi)

- **Current Ceramics**
  - $K_{IC} = 4 \text{ MPa} \sqrt{\text{m}}$
  - $\sigma_f = 158 \text{ MPa}$ (25 ksi)

- **For $C = 400 \mu\text{m}$**

**Graph 2:**
- **Whisker and Fiber Reinforcement**
- **Partially Stabilized Zirconia**
- **Monolithic Ceramics**

**Temperature (°C):**
- 0
- 200
- 400
- 600
- 800
- 1000

**Toughness (MPa\sqrt{m}):**
- 0
- 5
- 10
- 15
- 25

**Flexure Strength (MPa):**
- $10^1$
- $10^2$
- $10^3$
- 5
- 2

**Maximum Flaw Size ($\times 10^{-6} \text{ m}$):**
- 1
- 2
- 5
- 10
- 50

**Maximum Flaw Size ($\times 10^{-3} \text{ in.}$):**
- 1
- 2
- 5
- 10
- 50
MICROSTRUCTURE TAILORING & NOVEL COMPOSITE DESIGN ADDRESS INHERENT BRITTLENESS OF CERAMICS

FRACTURE TOUGHNESS

GLASSES
OXIDES
NON-OXIDES
WHISKER REINFORCED
TRANSFORMATION TOUGHENING
FIBER REINFORCED

COMPARED TO METALS WITH TYPICAL FRACTURE TOUGHNESS OF 15-200
FRACTURE RESISTANCE IS INCREASED BY REINFORCING CERAMICS WITH STRONG MICROSCOPIC WHISKERS

Ceramic Whiskers and Human Hair

Ceramic Composite Fracture Surface

Toughness Model

\[
dK = d^w \left( \frac{V_r}{6(1-V^2)} \right) \frac{E_c}{E^w} \frac{G_M}{G^w}
\]

- \( d^w \) = Fracture strength whisker
- \( V \) = Vol. fracture whiskers
- \( r \) = Whisker radius
- \( E \) = Young's modulus
- \( G \) = Fracture Energy
R&D ADDRESSING EFFECTS OF WHISKER COATINGS ON COMPOSITE PROPERTIES

Alumina-SiC whisker interface

Carbon coated whisker-alumina interface
FIBER-REINFORCED CERAMIC COMPOSITES HAVE BEEN FABRICATED USING A FORCED CHEMICAL VAPOR INFILTRATION PROCESS.
EXPERIMENTAL RESULTS SHOW MODIFICATION OF MATRIX/WHISKER INTERFACE IMPROVES COMPOSITE TOUGHNESS
SUCCESSFUL FABRICATION BY FCVI OF ARTICLES WITH COMPLEX SHAPES AND FIBER ARCHITECTURES HAS BEEN DEMONSTRATED

ANGLE-WOUND TUBE

REPRESENTATIVE ROTOR (cloth layup)

3-D ORTHOGONAL WOVEN DISK

THICK-TO-THIN DISK (cloth layup)
CERAMICS TESTED IN AEROSPACE APPLICATIONS
ADDITION OF ZrO$_2$ TO BRITTLE CERAMICS INCREASES RESISTANCE TO CRACK PROPAGATION

- Toughening is derived from stress-induced martensitic transformation in ZrO$_2$ particles
  - Transformation consumes energy needed for further propagation
  - Increased volume of new phase contributes compressive forces resisting further propagation
MICROWAVE SINTERING HAS SIGNIFICANT IMPLICATIONS FOR NEW MATERIALS APPLICATIONS

Accelerated Kinetics:
- Sintering occurs at lower temperatures

Significant Potential Benefits
- Lower temperature processing
- Finer microstructures
- Better mechanical properties

Advanced Materials Applications:
- Composites from incompatible materials
- Self-lubricating high-temperature bearings
- Electrical materials
- Engine components
MICROWAVE FACILITY FOR DENSIFYING CERAMICS
Microstructure of Si$_3$N$_4$-6% Y$_2$O$_3$-2% Al$_2$O$_3$ After Annealing at 1200°C for 20 hours

Conventional Heating

Microwave Heating
MICROSTRUCTURE MODIFICATION SIGNIFICANTLY IMPROVES CREEP RESISTANCE

SN-6Y2A
ANNEALING CONDITIONS: 1200°C/20 h
TEST TEMPERATURE: 1260°C
Tensile creep of NTX-154 and NT-164 at 1370°C

NTX-154 had a glass layer at most Si$_3$N$_4$/Si$_3$N$_4$ grain boundaries after creep (100 MPa, 1200 h).

No glass layer was found at NT-164 Si$_3$N$_4$/Si$_3$N$_4$ grain boundaries after creep (150 MPa, 959 h).
GELCASTING
A NEW CERAMIC FORMING PROCESS

OBJECTIVE:
Develop low cost, high quality forming process
- Complex shapes
- Large sections
- High yields
- Near-net shape
- Scalable to high volume production

APPROACH:
Gelcasting: A new method of fabricating ceramic-based by mixing polymers and ceramic powders with a suspension of ceramic powder in order to obtain a nonembrittlement casting and solidification process.
- Ceramic powders
- Polymer
- Monomer
- Dispersant

PROCESS FLOW CHART

PROCESS APPLICATIONS
List of materials gelcast:
- Alumina
- Silicon nitride
- Silicon carbide
- Zirconia
- Fused silica
- Sialon
- Composites
- Nicalon fiber reinforced reaction-bonded silicon nitride
- Aluminum oxides

TECHNOLOGY TRANSFER AND COMMERCIALIZATION

Office of Transportation Technologies
Base Technology Development
Automobile Applications

Office of Industrial Technologies
Continuous Gelcasting
Host Ceramic Matrix Applications

Companies interested in Gelcast Commercialization

Applications
- Specialty Products
- Thermal Energy
- Ceramic Materials Technology
- Heat Exchangers
- Products
- Ceramic Products
- Ceramic Rocket Nozzles
- Ceramic Products
- Ceramic Insulators
CERAMIC TURBOCHARGER ROTOR IN PRODUCTION VEHICLE

Nissan Motor Co. Silicon Nitride Turbocharger Rotor
EXTENDED VEHICLE TESTING OF VALVE TRAIN COMPONENTS
SUMMARY

- SPECIALIZED, TECHNOLOGY-INTENSIVE, HIGH-VALUE-ADDED ADVANCED CERAMICS ARE MOVING TOWARD COMMERCIALIZATION TO FILL THE DEMAND FOR LIGHTER, STRONGER, MORE CORROSION RESISTANT MATERIALS

- ADVANCEMENTS WILL RELY MORE AND MORE ON PROCESSING AND MODELING FROM THE ATOMIC SCALE UP WHICH IS MADE POSSIBLE BY ADVANCED ANALYTIC, COMPUTER, AND PROCESSING TECHNIQUES

- SPECIALIZED PROPERTIES AND HIGHER COST WILL PROVIDE BOTH NEW OPPORTUNITIES AND CHALLENGES TO DESIGNERS AND END-USERS
  - MORE RIGOROUS DEFINITION OF COMPONENT REQUIREMENTS
  - COMPONENT REDESIGN TO ACCOMMODATE BRITTLE BEHAVIOR
  - SYSTEM REDESIGN TO MAKE FULL USE OF NEW MATERIALS
  - CLOSER COUPLING BETWEEN DESIGNERS AND MATERIALS DEVELOPERS