HIGH TEMPERATURE TESTING OF CERAMICS

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Summer School
August 28, 2007
Outline

Introduction
• Advanced ceramics vs. advanced structural ceramics
• Market with the advanced ceramics – an overview
• Applications of advanced ceramics
• Requirements to HT structural ceramics in CGT

Testing at HT – mechanical tests
  Geometry (bending, compression, tension)
  Strength, fracture toughness, creep
  Tensile creep testing
  Creep mechanisms – techniques of their determination
  Corrosion resistance

• Conclusions
ADVANCED CERAMICS VS. ADVANCED STRUCTURAL CERAMICS

• Traditional ceramics
  - building (bricks, concrete, glass..).
  - pottery & home tools
  - refractories (blast furnaces, etc...)

• Advanced ceramics
  - electronic (wafers, chips, etc...)
  - structural ceramics (for heat-, wear- and corrosion- resistant components
  - others (e.g., coatings, bioceramics... )
OLDER & NEW “TRADITIONAL” CERAMICS
Market in Advanced Structural Ceramics in North America by Industry, 2005 and 2010 ($ Millions)

Source: BCC Research, 2006
Summary of the advanced ceramic market in US

- The North American advanced structural ceramic market in 2005 ~ $2.3 billion (65%)($3 bill. by 2010)
- Its average annual growth rate (AAGR) of 5.8%

- Bioceramics (2005) ~ $1.5 billion, AAGR of 4.6%
- Wear-resistant and cutting tool inserts ~ 17% of the total market.
- Ceramic armors represent about 13% of the total market.

Energy cost escalations, => more use as components for heat exchangers, recuperators and as solid oxide fuel cell elements.

Source: BCC Research, 2006
APPLICATIONS OF ADVANCED STRUCTURAL CERAMICS AND CERAMIC MATRIX COMPOSITES

- Cutting Tools
- Bearings
- Parts of Engine/Turbine
- Automotive/Aircraft Components
- Wear and Heat Resistant Components
- Piezo/Electroceramics
- Bioceramics
- Glasses...
CUTTING INSERTS AND BEARINGS

High hardness and wear resistance – silicon nitride, alumina, sialon

60% lighter as steel => less energy and 80% higher speed silicon nitride
CERAMICS IN AUTOMOBILES

AUTOMOTIVE MAGNET APPLICATIONS

- Windshield Wiper Motors
- Cruise Control
- Speakers
- Antenna Lift Motor
- Defogger Fan Motor
- Tailgate or Trunk Latch
- Door Lock
- Window Lift Motor
- Fuel Pump Motor
- Seat Positioning Motors
- Cooling Fan Motor
- Automatic Temperature Control
- Sun Roof Motor
- Tape Drive
- Door Lock
- Economy and Pollution Control Devices
- Heating and Air Conditioning Motor
- Liquid Level Indicators
- Windshield Washer Pump
- Air Pump/Suspension
- Ignition
- Anti-Lock Brake Speed Sensors
- Speedometer, Gauges and Digital Clock
- Starter Motor
STRUCTURAL CERAMICS IN AUTOMOBILES

Alumina, zirconia, silicon nitride, silicon carbide
Diesel fuel-injection timing plunger

Cam follower rollers

Valves

Ceramic-metal
HEAT RESISTANT TILES

33000 ceramic (C-C composite with SiC and SiO₂ overcoats) tiles; $T_{\text{MAX}} \approx 1260$ °C
AIRFRAME COMPONENTS

Eurofighter Typhoon.

airframe surface:
70% - CFCs
15% Al- and Ti- alloys
12% Glass Reinforced Plastics (GRP)
15% metals
3% other materials

www.eurofighter.com/et_mp_ma_cf.asp
AIRFRAME COMPONENTS

Airbus A380

25 wt. % - Composite materials Carbon-fibre reinforced plastic, glass-fibre reinforced plastic, and quartz-fibre reinforced plastic (wings, fuselage sections, tail surfaces, doors) Thermoplastics (leading edges). GLARE (Glass-reinforced fibre Al laminate) (upper fuselage and on the stabilizers' leading edges).
**AIRFRAME COMPONENTS**

Boeing 787 Dreamliner

- 50% (body and wing) - composite materials
- 20% - Al alloys
- 15% - Ti alloys
- 5% - high strength steel.
EFFICIENCY vs. GT SIZE

Source: http://www.khi.co.jp/gasturbine/english/rd_e/rd1.html
WHY CERAMICS IN GAS TURBINES ???

Basic thermodynamics of gasses ...

Carnot cycle

Thermal efficiency = \( \frac{(T_h - T_c)}{T_h} \)

Higher operating temperature necessary!!!
BETTER GAS TURBINES.....

HOT ZONE –
HIGHER TURBINE INLET TEMPERATURE (TIT)
IR Power Works Frame 3

Ceramic rotor

Unchanged balance of plant

400 °F

Recuperator

Compressor

Power Turbine

Gearbox

Generator

Electric Power To User

Utility Power

Metallic power turbine (IN713LC)

All new and improved aero-components (compressor + 2 turbines)
U.S. CERAMIC GAS TURBINE PROGRAMS - History

Brittle Material Design – HTT/ Ford Motor Co. (1971-78)

AGT – Allison vs. Garrett Co. / AGT 100 vs. AGT 101
(1979-87) 85 h at 1204°C

ATTAP - Allied Signal - 331 APU (commercially used in the MD 80 and A320 aircrafts since late 90-s)
- Hybrid Vehicle Turbine Engine Technology -1992
- Allison AGT 5 engine (5000 h at <1385°C

- Ceramic Vane Effort
- Materials and Manufacturing

Distributed Energy - (2000 -2006) IGT (relatively big - 4-5 MW turbines)
- Microturbines (relatively small <500 kW turbines)
Subtask: Ceramic Turbine Risk Reduction (Jan 05 –Mar 09)
Solar turbine  Centaur with silicon nitride rotor blades, tested in CA since 1997
4.5 MW turbine for energy cogeneration; retrofit,  TIT ~1000 °C; around 50 000 h accumulated; failure due to FOD
DISTRIBUTED ENERGY PROGRAM (DoE)

UTC Engine Applications – Axial Configuration Machines

- Engines for Unmanned Air Vehicles (UAVs)
  - Ceramics enable engine size and weight reduction

- Microturbines for Cooling, Heating, and Power (CHP) Systems
  - Ceramics provide increased efficiency
  - Waste heat to Chiller, ORC, Hot Water

- Auxiliary Power Units (APUs)
  - Ceramics provide engine weight and operating cost reduction

- Industrial Gas Turbines
  - Ceramics enable increased efficiency and reduced emissions

- UTC has several potential gas turbine engine applications that could employ ceramic turbine components for enhanced customer value
- Primary value achieved through higher temperature/efficiency and weight reduction
SILICON NITRIDE COMPONENTS IN MICROTURBINE PROGRAM

Source: J. Kesseli, Ingersoll-Rand Energy Systems
CGT IN JAPAN

NEDO Program: R&D on CGT (300 kW class)
PEC Program: CGT for automobiles (100 kW)

Thermal efficiency of 42% at 1350 C achieved
Problems with the reliability and lifetime

Source: www.khi.co.jp/gasturbine
EUROPE and CGT (SWEDEN)

Development of ceramic turbine components -1981
Turbine wheel (ASEA CERAMA) tested in a car 1982
GT110 demonstrated reliable at 1250 °C - 1987

European AGATA project developed
Ceramic radial turbine
Ceramic recuperator
Ceramic catalytic combustor

KTT MK II / GT110
• Developed 1985-1987
• Automotive GT for cars
• Power 115 kW (155 hp)
• 2-shaft with ceramic HP turbine
• Low emission combustor (LPP)

Source: Lars Sundin, Volvo Aero Presentation, July 2003, Brussels
CGT – success or failure???

Silicon Nitride ceramic rotor technology is adaptable at moderate temperatures and stresses (up to 20,000 /mo turbine rotors were produced (2003) for the turbocharger industry)

**Main benefits:**
- Increase in thermal efficiency (C200/ORC system - 38% electrical efficiency (Mar 2005))
- Slightly better fuel economy (7%)
- Higher output power
- Significant NO\textsubscript{x} reduction

**Main problems:**
- Water vapor corrosion at higher temperatures
- Long term reliability
- FOD
- Component price
CRITICAL CERAMIC COMPONENTS

- Vane ring
- Stator nozzles
- Radial Turbine Wheels
- Rotor blades/blisk
- Combustor (SiC, C-C)
- Recuperator

Static load

SILICON NITRIDE – $\text{Si}_3\text{N}_4$

100 000 rpm at >1000 -1400°C

Radial Turbine Wheels
Requirements to structural ceramics in CGT

TIT > 1300°C [Licht]:

• Flexural strength at RT and at 1300°C - >950 MPa and > 600 MPa, respectively;
• Weibull modulus >12
• Fracture toughness > 6.5 MPa. m$^{1/2}$
• Creep rate $< 2 \times 10^{-8}$ s$^{-1}$ at 1250°C and 130 MPa.
• Oxidation and corrosion resistance up to 1250°C
M modes of mechanical testing

- Tension
  - Strain = relative size (length) change

- Compression

- Bending

Strain = relative size (length) change
What is STRENGTH?

4-point bending

Brittle behavior

Ductile behavior

Slope = Young’s modulus

> 10 samples (tests) to get the strength... at each temperature
STRENGTH and FAILURE

Tension - the largest flaw propagates (after reaching critical stress)

Compression - many flaws propagate => general crushing
STRENGTH

\[ \sigma = \frac{K_{IC}}{(\pi a)^{1/2}} \]

- \( K_{IC} \) – fracture toughness; \( a \) – flaw size

High strength ceramics require:

- small defects (ceramics - several um, in metals – tens of mm)
- higher fracture toughness (in ceramics <10 MPa.m\(^{1/2}\), metals ->50 MPa.m\(^{1/2}\))
STRENGTH STATISTICS

Strength of metals = deterministic

strength of ceramics = probabilistic

Chalk - \( P_f = 0.3 \)

 Cutting tool - \( P_f = 10^{-2} \)

Protective tile of space shuttle - \( 10^{-8} \)
FAILURE PROBABILITY IN BRITTLE MATERIALS

VOLUME DEPENDENCE OF STRENGTH

Large sample fails at a lower stresses
Weibull: survival probability $P_s(V_0) = \text{fraction of identical samples with the volume } V_0, \text{ which survive loading to a tensile stress of } \sigma$. 

$$P_s(V_0) = \exp\{-(\sigma/\sigma_0)^m\},$$

$\sigma_0$ is the characteristic stress that allows 37% of the samples to survive;

$m$ - Weibull modulus shows the variability (scatter) of the strength.
COMPARISON OF FAILURE PROBABILITIES

Higher $m$: - technology defect elimination
  - materials with higher fracture toughness

Careful design – no stress concentration
FRACTURE TOUGHNESS

Definition

\[ K_I = Y \cdot \sigma \cdot \sqrt{a} \quad [\text{MPa} \cdot \text{m}^{1/2}] \]

- \( K_I \) – fracture toughness (stress intensity factor)
- \( \sigma \) - outer fibre bending stress
- \( Y = Y(a/W) \) - geometry function

Pre-defined defect – crack is necessary
FRACTURE TOUGHNESS MEASUREMENT METHODS

SENB, SEPB, SEVNB, IF, DT ...

Chevron Notch

Vickers indentation on tensile surface
INDENTATION FRACTURE TOUGHNESS

Specimen with Vickers (Berkovich, Knoop) Indentation Crack

\[ K_{IC} \sim H(a)^{1/2}(E/H)^{1/2}(c/a)^{-3/2} \]
STRENGTH vs. TEMPERATURE and TIME

Possible failure modes:
1. Fast failure
2. Slow crack growth
3. Creep fracture

Source: G.D. Queen, 1990
What is CREEP?

4-point bending creep

Creep is time dependent plastic deformation

CREEP
Bending
Tension
Compression
TENSILE CREEP TESTING

- geometry unbiased values experimentally troublesome:
  - serious alignment problems
difficult elongation measurement
- expensive

Hot grips vs. Cold grips

+ geometry unbiased values

- experimentally troublesome:
Creep behavior of SN 88 silicon nitride
(Yb/Y-based additives)

1400°C/ 150 MPa

Minimum strain rate
No. 3: 5.70 h, 0.51 %
No. 4: 24.0 h, 1.20%
No. 5: 70.0 h, 2.30 %
No. 6: 85.06 h, 2.70% creep fracture
CREEP RESISTANCE IN COMMERCIAL Si$_3$N$_4$ CERAMICS

![Graph showing creep rate vs. temperature for different Si$_3$N$_4$ compositions under 150 MPa stress.]

- Pure SN 75 MPa
- AS 950 (SiC part.)
- SN 88 (Yb$_2$O$_3$)
- NT154 (Y$_2$O$_3$)
- SN281 (Lu$_2$O$_3$)
- SN 282

1% after 10,000 h
CREEP MECHANISMS IN VITREOUS BONDED CERAMICS

Creep Mechanisms:
1. Mutual sliding of grains
2. Viscous flow of glassy phase
3. Grain shape change
4. Cavitation

CAVITATION is crucial for creep behavior of ceramics
CAVITATION – TECHNIQUES OF THEIR MEASUREMENT

DESTRUCTIVE  NON-DESTRUCTIVE

Density change  Tensile – compressive strain
A-USAXS  Ultrasonic velocity
TEM/SEM  SLAM
Densitometry (Sink-Float)

Density of TMF solution linearly changes with temperature.

- Standard 1
- Grip sample
- Gauge sample
- Standard 2

Water solution of thalium malonate formate \( \rho \sim 3.55 \text{ g/cm}^3 \)

Water heater/cooling for temperature control.

H_2O
\[ \Delta \rho/\rho_0 = (\rho_{\text{gage}} - \rho_{\text{grip}})/\rho_{\text{grip}} = -f_v \]

relative density change = - volume fraction of cavities

SN 88 silicon nitride
1400°C - 1500°C

Creep strain

Cavitation strain

slope 0.85

Tensile Strain
ULTRASOUND VELOCITY MEASUREMENT

\[ V_{l,sh} = \frac{2d}{\Delta t_{l,sh}} \]
Ultrasound velocity vs. Strain

SN88, 1400°C, 150 MPa

\[ v_{l0} = 10390 \pm 35 \text{ ms}^{-1} \]

\[ \varepsilon_{th} < 0.001 \]

Average Rate of Longitudinal Waves, [m/s]

Tensile Strain

specimen A - interrupted test
gauges

specimen B - interrupted test

9 interrupted creep tests
ELASTIC MODULI VS. CAVITATION

Relative Change of the Elastic Moduli

\[ \frac{(X - X_0)}{X_0} = b_x \cdot f_v \]

Volume Fraction of Cavities (= Porosity), \( f_v \)

SN 88, 1400°C/ 150 MPa
CAVITATION BY SLAM

4-point bending creep, experimental silicon nitride

A – compressive strain zone
B – neutral axis zone
C – tensile strain zone
CAVITATION BY SLAM

High attenuation zone development in tensile stress zone
OXIDATION RELATED MICROSTRUCTURE CHANGES

Layer formation during HT creep in air

ST1 silicon nitride

1400°C, 50 MPa, 263 h

1400°C, 70 MPa, 270 h, 15.4 % strain
High temperature fracture of Si$_3$N$_4$

Polished section of SN88 tested at 850 °C
At 0.003 MPa/s

Secondary phase transformation from oxidation leading to the large volume change, residual tensile stress and damage zone formation
CONCLUSIONS

Structural ceramics reached their maturity and found wide applications at room temperature.

High temperature ceramics have limited applications up to now and further development is necessary.

Main problems for HT ceramics include water vapor corrosion, long term reliability and cost.

Problems with HT testing of ceramics:

- Temperatures 1200 – 1600°C
- Stresses – up to 600 MPa in tension/400 MPa for long term tests (up to 100 000 h)
- Difficulties with the elongation measurement
- Role of environment