Sintering and Deformation Mechanisms in Plasma-Sprayed 7 wt.% Y₂O₃-ZrO₂ Thermal Barrier Coatings

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Thermal Barrier Coatings

A 200 μm thick YSZ topcoat provide a 200°C drop → engine efficiency increases by ~ 6 - 12%

Typical bondcoat materials: MCrAlY, PtAl
Typical topcoat material: 7 wt% Y₂O₃-ZrO₂ (YSZ) – focus of current research

R. Stevens, “An Introduction to Zirconia”, Publication of Magnesium Elektron Inc.
Overview of Plasma Spray Process

- External Powder Feed
- Lamellae
- Substrate
- Plasma Plume (10,000-20,000K)
- Arc Gas Passes Here
- Power Supply
- Anode, -
- Cathode, +

www.swunited.com/plasma/pages/placap.htm
Plasma-Sprayed YSZ Microstructure

- Lamellae are observed
- Interlamellar pores
- Intralamellar cracks
- Columnar grains
- Porosity of 15-20%
Focus of Our Research: Link the microstructure, before and after high temperature exposure, to the thermal and mechanical properties of the YSZ coating off-of-the-substrate

Outline

• Introduction

• Experimental Procedure

• Results and Discussion
  - Room temperature mechanical behavior
  - Sintering behavior
  - Stress relaxation behavior

• Conclusions
Experimental Procedure

Processing of Stand-Alone Coating Samples

⇒ Plasma plume
⇒ Seven axis robot
⇒ YSZ Powder
⇒ Cool air
⇒ Melted Powder
⇒ Alumina Rod
⇒ Turntable
⇒ Cut

⇒ Etching Al in HCl
⇒ To remove alumina core

YSZ stand-alone coating
Transmission Electron Microscopy Procedures

- Bright-field TEM analysis
- Cross-section and plan-view –
- Tripod polishing technique
- Gatan® ion-mill
- JEOL 2000FX
Overview of YSZ Stand-Alone Coatings

Sample Characteristics

Diameter = 13 mm
Height = 12-15 mm
Thickness = 200-300 µm
Porosity = 13-18%

Yttria rich, non-transformable
$t$-ZrO$_2$ coating
In-Situ Observation of Crack Behavior in Plasma-Sprayed Coatings

- Two distinct slopes in stress-strain data
- Significant cracking “heard” with acoustic emission techniques during loading

ESEM with Load Frame

- Electro Scan 2020 ESEM
- Operated at 25 keV
- Working distance of 27 mm
Close-up of Load Frame

- Strain gages attached to sides of tube
- View outer surface of the coating
Procedures Followed for Compression Testing of Tubes in ESEM

- A region was located which exhibited cracks oriented in various directions with respect to the applied stress.

- The sample was then incrementally stressed, with strain data and micrographs taken at each stress increment (approximately 10 MPa per step).
Stress-Strain Results

- Distinct change in modulus with stress
Effect of Stress on Intralamellar Cracks

Micrographs of top surface

- Cracks formed during cooling
Intralamellar Cracks Investigated in Present Study

- Chose 5 cracks to investigate

- Measured change in crack width as a function of applied stress
Results of Crack Measurements

- Vertically oriented cracks open, horizontally oriented cracks close

- Seems plausible that the increase in E with stress is due to partial closure of horizontally oriented cracks
Crack Nucleation and Propagation

- These images show a crack nucleating and propagating from an existing angled crack
- Significant crack propagation prior to failure
- Consistent with acoustic emission results on PS alumina

Crack Initiation in Angled Micro-Cracks – Ashby and Hallam Results* on PMMA

Sintering Behavior of PS YSZ

Observed Microstructural Changes

- Cont. sintering/some m-ZrO₂
- Partial closure of interlamellar pores
- Full closure of intralamellar cracks
- Closure of intralamellar cracks

Dilatometry Measurements

- Linear resolution: 1 µm (~ 0.02% for a 15mm coating)
- Dilatometer data are deconvoluted in order to isolate the shrinkage behavior of the YSZ coating.
- Temperatures investigated: 800°C thru 1400°C, simple ramps and 10-hr isothermal tests
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Effect of Multiple Heating/Cooling Cycles

- Subtracted out the expansion of apparatus and coating
- Measurable shrinkage, detected at 950°C
Effect of 10-hr Isotherms on Linear Shrinkage of YSZ Tubes

The graph shows the linear shrinkage, expressed as a percentage, of YSZ tubes subjected to isotherms at various temperatures ranging from 800°C to 1400°C over a period of 10 hours. The shrinkage is plotted against time in minutes, with the x-axis representing the time at the indicated temperature and the y-axis representing the linear shrinkage percentage. The graph indicates the sintering behavior and shrinkage characteristics of YSZ tubes under these conditions.
Effect of Isothermal Temperature on Shrinkage Rate

- Significant shrinkage still occurring after 10-hrs at 1300°C, 1400°C
Link Shrinkage Behavior with Microstructural Changes

After 10-hrs @ 800°C
Cross-sectional view

• Narrow intralamellar microcracks observed

After 10-hrs @ 900°C
Plan view
Microstructural Changes with Temperature

After 10-hrs @ 1000° C

- No narrow intralamellar microcracks were observed.
- Pores and wide intralamellar cracks remain.
Microstructural Changes with Temperature
After 10-hrs @ 1200°C

- Interlamellar lenticular pores are breaking down into channels of isolated globular pores by pinching between adjacent lamellae.
Stress Relaxation Experiments on Stand-Alone YSZ Coatings

- Apply constant strain, monitor load
- Test temps: 1000°C, 1050°C, 1100°C, 1200°C
Stress State in the YSZ During Heat-up

- Effective biaxial compressive stress applied within the plane of the coating
Stress Relaxation Behavior as a Function of Temperature

<table>
<thead>
<tr>
<th>Temp. °C</th>
<th>Time to 0 MPa, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>600</td>
</tr>
<tr>
<td>1050</td>
<td>400</td>
</tr>
<tr>
<td>1100</td>
<td>130</td>
</tr>
<tr>
<td>1200</td>
<td>80</td>
</tr>
</tbody>
</table>

Original Stress Applied was 20 MPa
Stress Relaxation Behavior As a Function of Beginning Stress Level

- Modeling behavior of the material
- TEM experiments
Summary

• The stress-strain curve for the YSZ tube showed an increasing modulus with two distinct regions of differing modulus – linked to crack closure.

• Micrographs taken using an ESEM showed that cracks oriented parallel to the applied stress opened and cracks oriented perpendicularly to the applied stress closed.
Summary

• The shrinkage and shrinkage rate of PS YSZ increased with temperature and decreased with time. PS YSZ is still shrinking after 10 hours at 1300°C and 1400°C.

• Narrow intralamellar cracks are healing around 1000°C. All cracks have healed after 10 hours at 1000°C.

• Interlamellar pores were found to close at temperatures > 1000°C. This process seems to be triggered by pinching between two adjacent grains as they assume an equilibrium shape.