Mechanical and physical properties of ZnSe windows to be used with the FEANICS (Flow Enclosure Accommodating Novel Investigations in Combustion of Solids) experiments were measured in order to determine design allowables. In addition, the literature on crack growth properties was summarized. The average Young’s modulus, Poisson’s ratio, equibiaxial fracture strength, flaw size, grain size, Knoop hardness, Vicker’s hardness, and branching constant were 74.3 ± 0.1 GPa, 0.31, 57.8 ± 6.5 MPa, 21 ± 4 mm, 43 ± 9 um, 0.97 ± 0.02 GPa, 0.97 ± 0.02 GPa, and 1.0 ± 0.1 MPam^0.5, respectively. The properties of current ZnSe made by chemical vapor deposition are in good agreement with those measured in the 1970’s. The hardness of CVD ZnSe windows is about one twentieth of the sapphire window being replaced, and about one-sixth of that of window glass. Thus the ZnSe window must be handled with great care. The large grain size relative to the inherent crack size implies the need to use single crystal crack growth properties in the design process. In order to determine the local failure stresses in one of the test specimens, a solution for the stresses between the support ring and the edge of a circular plate load between concentric rings was derived.
Mechanical Properties of ZnSe for the FEANICS Module

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2006 Electromagnetic Windows Symposium

May 1 - 4, 2006
OUTLINE

- Background; FEANICS Mission
- General properties of ZnSe
- Estimation of crack growth parameters
- Grain size effects
- Test results
- Summary
BACKGROUND

NASA missions require the safe-use of light weight brittle materials:

- Specialty Windows
- Solar concentrators
- Electronic substrates
- Combustor liners
- Bearing balls
- Leading edges

\{ Single Crystal Applications \\
Polycrystalline & Composite Applications \}
Mission - support study combustion of thick and thin solid fuels in microgravity:

- Forced Ignition and Spread Test
- Solid Inflammability Boundary At Low-Speed

FEANICS
Flow Enclosure Accommodating Novel Investigations in Combustion of Solids

Pressurized combustion chamber
ZnSe windows
Window assembly
Gasket
O-rings
2.25"
OBJECTIVES

- Review literature on fracture toughness, strength and crack growth properties for CVD ZnSe.
- Identify design values for room temperature, high humidity environment: applied stress of 10 MPa for 10 years.
- Run necessary tests to compliment the literature data.
- Most papers did not give full parameter set for design => Additional objective: reanalyze existing data sets.
CVD ZnSe Literature

Open Literature:


Closed Literature:

Miles: “incipient flaw size is comparable to the grains size. The effective material strength depends only weakly on the volume under stress........only slightly on the degree of perfection of the surface polish.” => weak Weibull effect........Effect for a grain size?

ZnSe is soft: Scratches extend length of grains causing single crystal, low energy fracture.
Microstructure of CVD ZnSe

- Microstructure varies widely, exhibits heavy twinning: $l = 42 \pm 2$ um perpendicular and $47 \pm 3$ um parallel (95% confidence). Grains as large as 150 um are apparent.
Window Strength
(Ring-on-Ring Loading)

Thin windows:
Strength of CVD ZnSe

For well polished ZnSe, strength is \(~55\) MPa.
Weibull scaling not apparent (study needed), but
Need similitude of flaw population.
Weibull Distributions
(Ball-on-3 Ball Loading)

- Weibull modulus of ~5, nominal strength of >50 MPa, but strength depends on environment – SCG.
Weibull moduli of $\sim 9$, nominal strength of $\sim 60$ MPa. Agrees with AFML data (139 test, $m = 9.2$, $\sigma_f = 54$ MPa).
Fracture Toughness Methods

Double Torsion

Double Cantilever Beam
Fracture Toughness Methods

\[ \alpha = \frac{a_1 + a_2 + a_3}{3W} \]

\[ \alpha_o = \frac{a_0}{W} \]

\[ \alpha = \frac{a_1 + a_2 + a_3}{3W} \]

Remove 4.5h to 5h

SEPB

CNB

SCF or IF
Fracture Toughness

- Fracture toughness increases with crack size and lower humidity: 0.3 MPa√m for small cracks and 0.9 MPa√m for large cracks in dry environments. ASTM C1421 CNB – 0.43 MPa√m.
Macro crack fracture toughness is substantially larger than small crack or single crystal values. Grain sampling vs. gain bridging => R-curve....
Slow Crack Growth

\( (\nu = AK_I^n) \)

- Three types of SCG data on ZnSe:
  - constant stress rate testing (Dynamic Fatigue)
  - constant stress testing (Static Fatigue)
  - fracture mechanics testing (DCB and DT)

- Main issues with the data:
  - small number of tests
  - significant scatter
  - poor reporting

- Macro crack stress intensities are substantially larger than those expected for failures from small cracks – Need similitude between the test data and component.
Slow Crack Growth
(Constant stress rate or dynamic fatigue, water)

- 18 data points; poor regression coefficient and large standard deviations.

Fracture Stress, MPa

Stress Rate, MPa
Slow Crack Growth
( Constant stress or static fatigue, air)

- 29 data points; better regression coefficient and standard deviation; five run outs.
Slow Crack Growth
(DT and DCB in air)

- Two data regimes macro and small or single crystal.
- No mathematical explanation of the shift or the assumptions used.
Estimation of Small Crack Parameters from Macro Crack Data

\[ v = AK^n_I \]

\( v_{terminal} \)

\( K_{I.poly} \)

\( K_{I.small} \)

More realistic model:

More grains encountered

\[ v = AK^n_I \]

Need observation of actual, small crack growth rates.
Estimation of Small Crack Parameters from Macro Crack Data

\[ v = AK^n_I \]

- Assume equivalent terminal velocity:
  \[ v = A_{Poly} K_{IcPoly}^n = A_{Single} K_{IcSingle}^n \]

- Assume identical slopes \((n)\):
  \[ A_{Single} = A_{Poly} \left( \frac{K_{IcPoly}}{K_{IcSingle}} \right)^n \]
Slow Crack Growth
(DT, DCB, constant stress in air)

- Two data regimes: Macro and small or single crystal.
- Reasonable agreement between small crack and shifted macro crack curves.
Slow Crack Growth (DT and DCB in water)

- Consistent DT data, scattered DCB data.
Slow Crack Growth
(DT and DCB, water)

- DCB water data has many low velocity, high stress intensity values – crack pinned - truncate.
- Nonlinear fits give smaller parameters.
Slow Crack Growth
(DT, DCB, Constant Stress Rate in Water)

- Macro crack data and constant stress rate data agree when a macro fracture toughness is used.
- Shifted macro crack curve and small crack curve agree.
# Slow Crack Growth
## (Parameter Summary)

<table>
<thead>
<tr>
<th>Data Source</th>
<th>Test Method (number of tests)</th>
<th>$n \pm SD_n$</th>
<th>$A_{n}$ m/s (MPa√m)-$^{n}$</th>
<th>$A_{Small}$ m/s (MPa√m)-$^{n}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Evans and Johnson</td>
<td>DT and DCB, Line</td>
<td>86</td>
<td>$1.11 \times 10^{13}$</td>
<td>$5.05 \times 10^{50}$</td>
</tr>
<tr>
<td>“ “ “ “</td>
<td>B-O-3B, Dyn. (18)</td>
<td>$50 \pm 32$</td>
<td>$9.98 \times 10^{3}$</td>
<td>$1.08 \times 10^{25}$</td>
</tr>
<tr>
<td>“ “ “ “</td>
<td>DT (25)</td>
<td>$40 \pm 2$</td>
<td>$1.09 \times 10^{3}$</td>
<td>$1.97 \times 10^{20}$</td>
</tr>
<tr>
<td>Freiman et al.</td>
<td>DCB (21)</td>
<td>$12 \pm 5$</td>
<td>$5.0 \times 10^{-3}$</td>
<td>$8.61 \times 10^{2}$</td>
</tr>
<tr>
<td>“ “ “ “</td>
<td>DCB (9), Truncated</td>
<td>25</td>
<td>282</td>
<td>$1.46 \times 10^{13}$</td>
</tr>
</tbody>
</table>

| Air                    |                               |              |                             |                               |
| Freiman et al.         | DCB (11)                      | $26 \pm 6$   | $6.2 \times 10^{-3}$       | $3.7 \times 10^{8}$           |
| McKinney et al. Air    | 4-point Static (28)           | $21 \pm 6$   | $4.8 \times 10^{-6}$       | $6.5 \times 10^{3}$           |

- Likely values are $n = 40$, $A_{Poly} = 1000$, $A_{single} = 10^{20}$
Crack Branching Constant

Branching constant $A_b = 1 \pm 0.1 \text{ MPa} \sqrt{\text{m}}$, residual stress $= 9 \text{ MPa}$. 
SUMMARY

- Based on 1970’s data, the single crystal or small crack SCG parameters for CVD ZnSe in water are \( n < 40 \), and \( A_{\text{single}} > 10^{20} \), \( K_{Ic} = 0.33 \text{ MPa } \sqrt{\text{m}} \).

- The macro-crack parameters are much different: \( 25 < n < 80 \) and \( A_{\text{Poly}} = 10^3 \), \( K_{Ic} > 0.60 \text{ MPa } \sqrt{\text{m}} \).

- Strength is \( \sim 55 \text{ MPa} \) with a Weibull modulus of \( \sim 6 \) to 9.

- Small crack parameters estimated for macro-crack data agree reasonably with parameters estimated from strength data.

- But, we really need a SCG model that incorporates R-curve behavior, and observation of actual small crack growth rates.

- Many of the existing data sets contain much scatter and too few data points.