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 $\pm$ 0.1 GPa, 0.31, 57.8 $\pm$ 6.5 MPa, 21 $\pm$ 4 mm, 43 $\pm$ 9 um, 0.97 $\pm$ 0.02 GPa, 0.97 $\pm$ 0.02 GPa, and 1.0 $\pm$ 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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Cleveland, Ohio

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

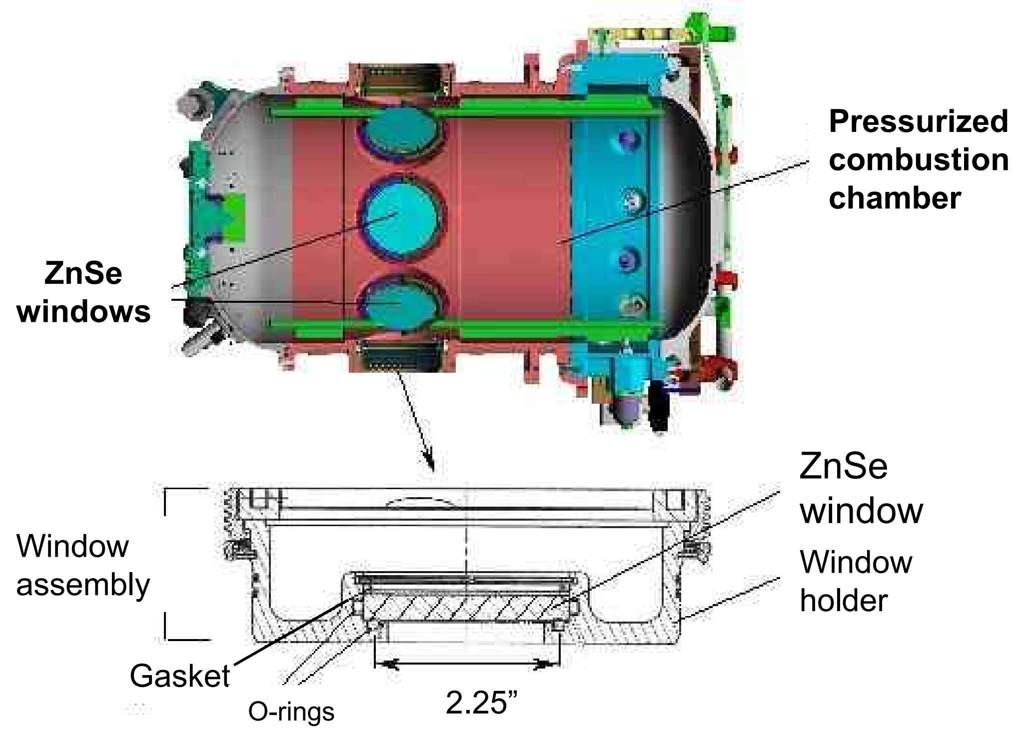
FEANICS

Flow Enclosure Accommodating Novel Investigations in Combustion of Solids

- Mission - support study combustion of thick and thin solid fuels in microgravity:

Forced Ignition and Spread Test

Solid Inflammability Boundary At Low-Speed



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

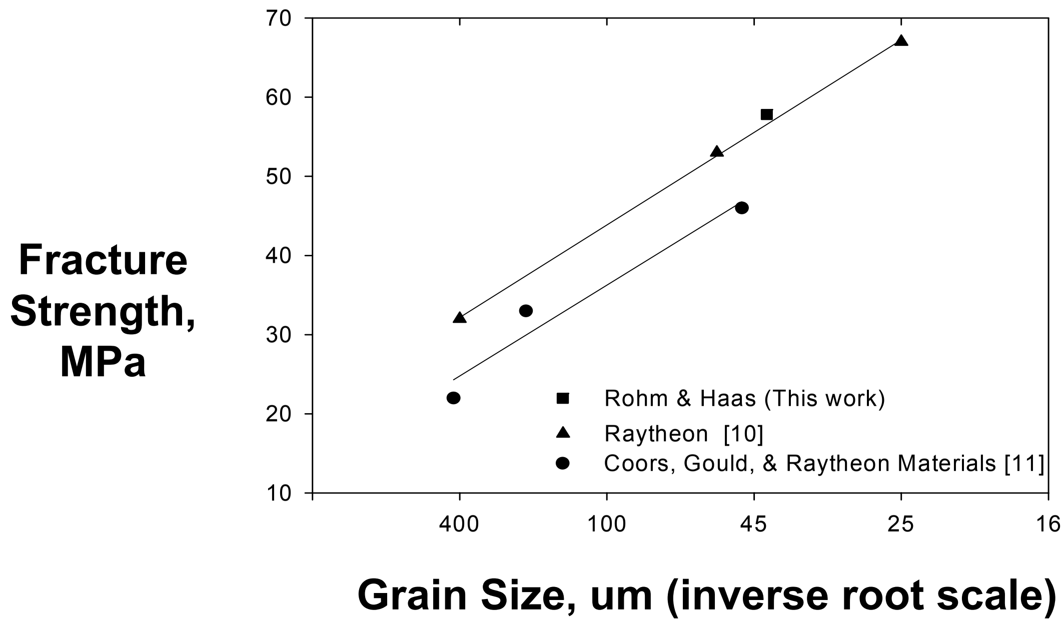
Open Literature:

1. A.G. Evans and H. Johnson, "A Fracture-Mechanics Study of ZnSe for Laser Window Applications," J. Am. Ceram. Soc. Vol. 58, No. 5-6, pp. 244-249 (1975)
2. S.W. Freiman, J.J. Mecholsky, Jr., R.W. Rice, and J.C. Wurst, "Influence of Microstructure on Crack Propagation in ZnSe," J. Am. Ceram. Soc., vol. 58, No. 9-10, pp. 406 – 409 (1975)
3. P. Miles, "High Transparency Infrared Materials – A Technology Update," *Opt. Eng.,* Vol. 15, No. 5, pp. 451-459 (1976).
4. K.R. McKinney, J.J. Mecholsky, Jr., and S.W. Freiman, "Delayed Failure in Chemically Deposited ZnSe," *J. Am. Ceramic Soc.,* Vol. 62, No. 7-8, pp. 336-340, (1979)
5. R.W. Rice, S.W. Freiman, and J.J. Mecholsky, Jr., "The Dependence of Strength-Controlling Fracture Energy on the Flaw-Size to Grain-Size Ratio," *J. Am. Ceram. Soc.,* Vol. 63, No. 3-4, pp. 129-136 (1980).
6. N. Ferneilus, G, Graves, and W. Knect, "Characterization of Candidate Laser Window Materials," pp. 188-195 in SPIE Vol. 297 Emerging Optical Materials, (1981)
7. R.L. Taylor and J.S. Goela, "Specification of Infrared Optical Materials for Laser Applications," pp. 22-35 in SPIE Vol. 607 Optical Component Specifications for Laser Based Systems and Other Modern Optical Systems, (1986).
8. "Estimation of ZnSe Crack Growth Properties for Design of the FEANICS (Flow Enclosure Accommodating Novel Investigations in Combustion of Solids) Windows," J.A. Salem, NASA TM 213359, 2005.
9. "Mechanical Characterization of ZnSe Windows for use with the Flow Enclosure Accommodating Novel Investigations in Combustion of Solids (FEANICS) Module," J.A. Salem, NASA TM 214100, 2006.

Closed Literature:

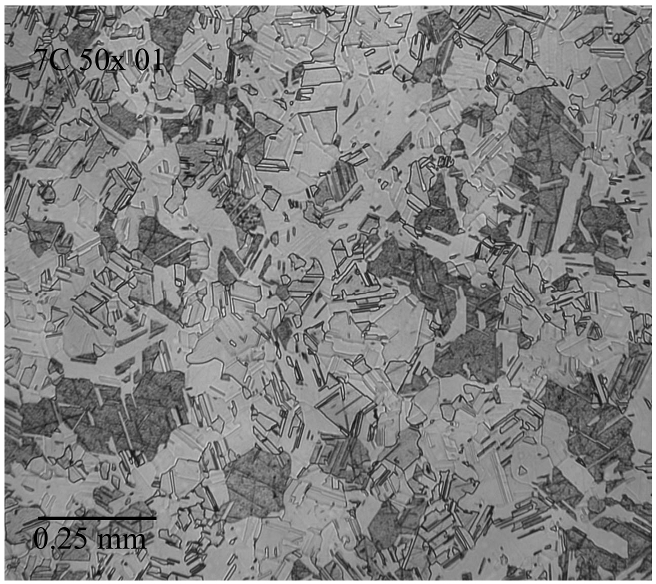
1. J.C. Wurst and T.P Graham, "Thermal, Electrical, and Physical Measurements of Laser Windows," University of Dayton Research Institute, Ohio, AFML-TR-75-28, April 1975.
2. G.A. Graves, D.E. McCullum, and J.M. Wimmer, "Investigation of the Thermal, Electrical, Mechanical, and Physical Properties of Infrared Laser Window and IT Transmitting Materials," AFML-TR-77-23, UDRI-TR-77-06, April 1977.
3. D.J. Iden, J.A. Detrio, J. Fox, "Optical Window Mechanical Strength and Reliability,' University of Dayton Research Institute, Ohio, Technical Report No. UDR-TR-83-73, June 1983.

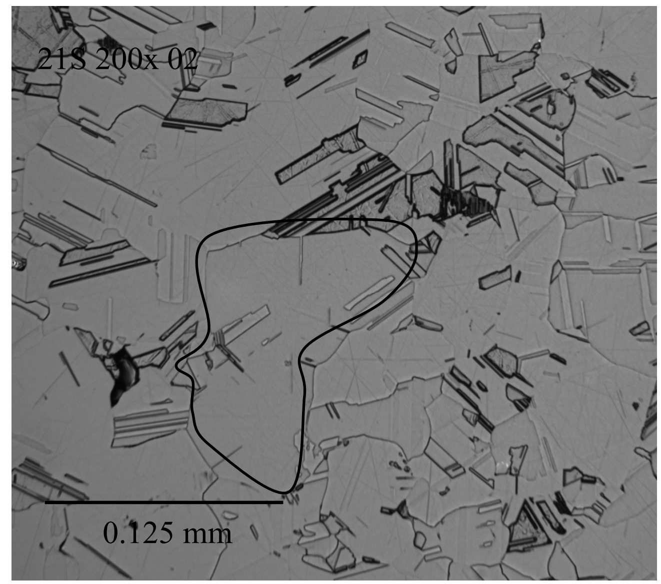
Strength of ZnSe



- 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.

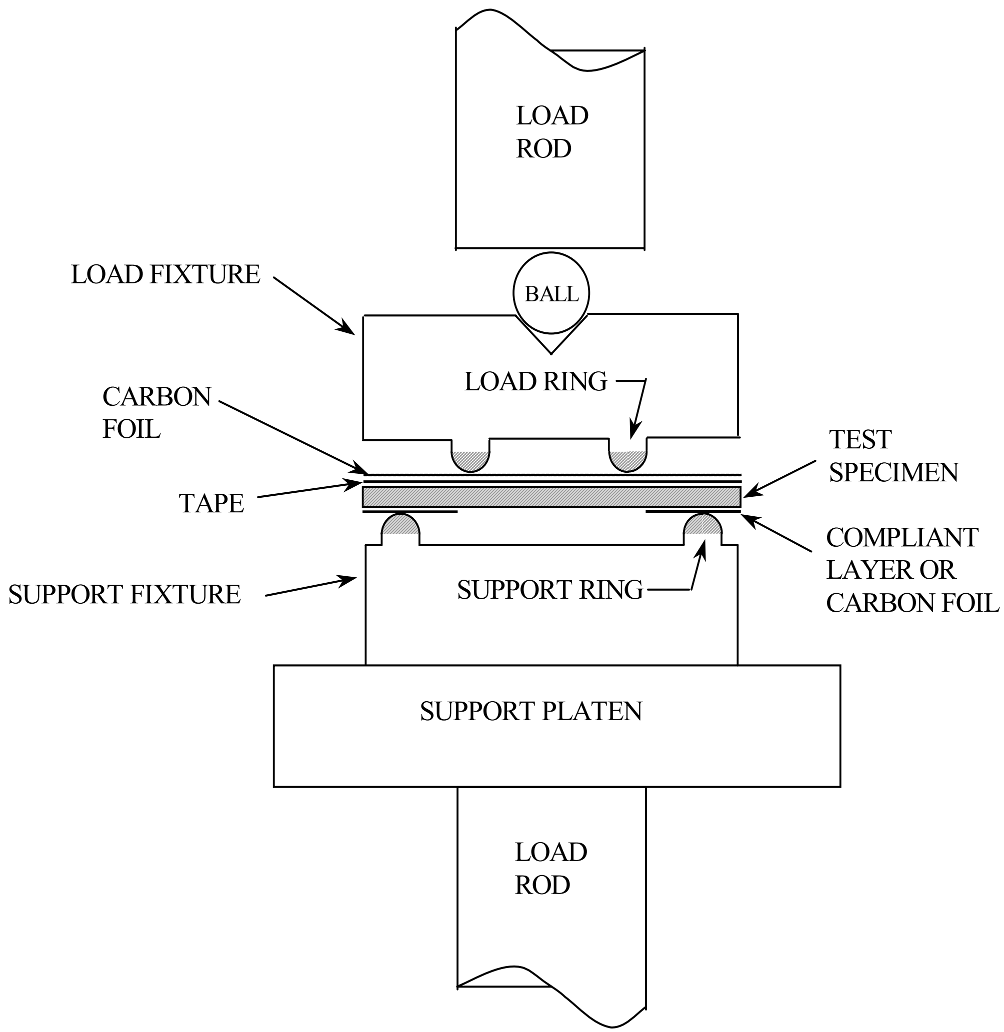
Microstructure of CVD ZnSe



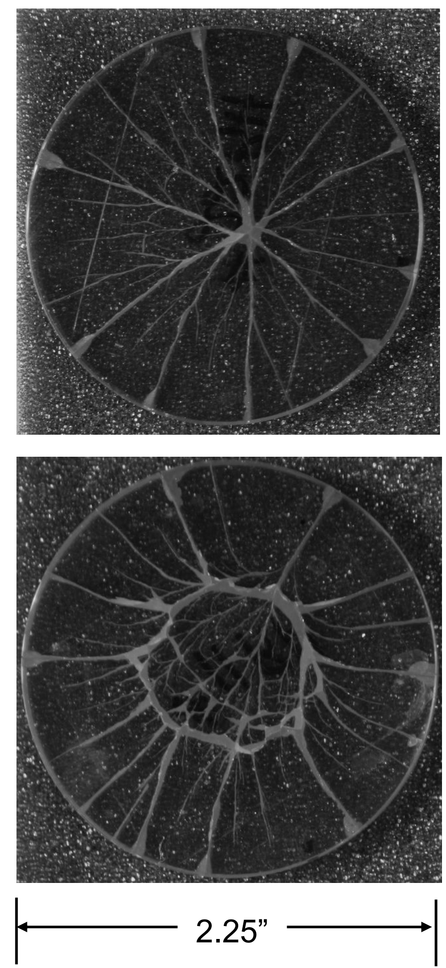


- Microstructure varies widely, exhibits heavy twinning: l = 42±2 um perpendicular and 47±3 um parallel (95% confidence). Grains as large as 150 um are apparent.

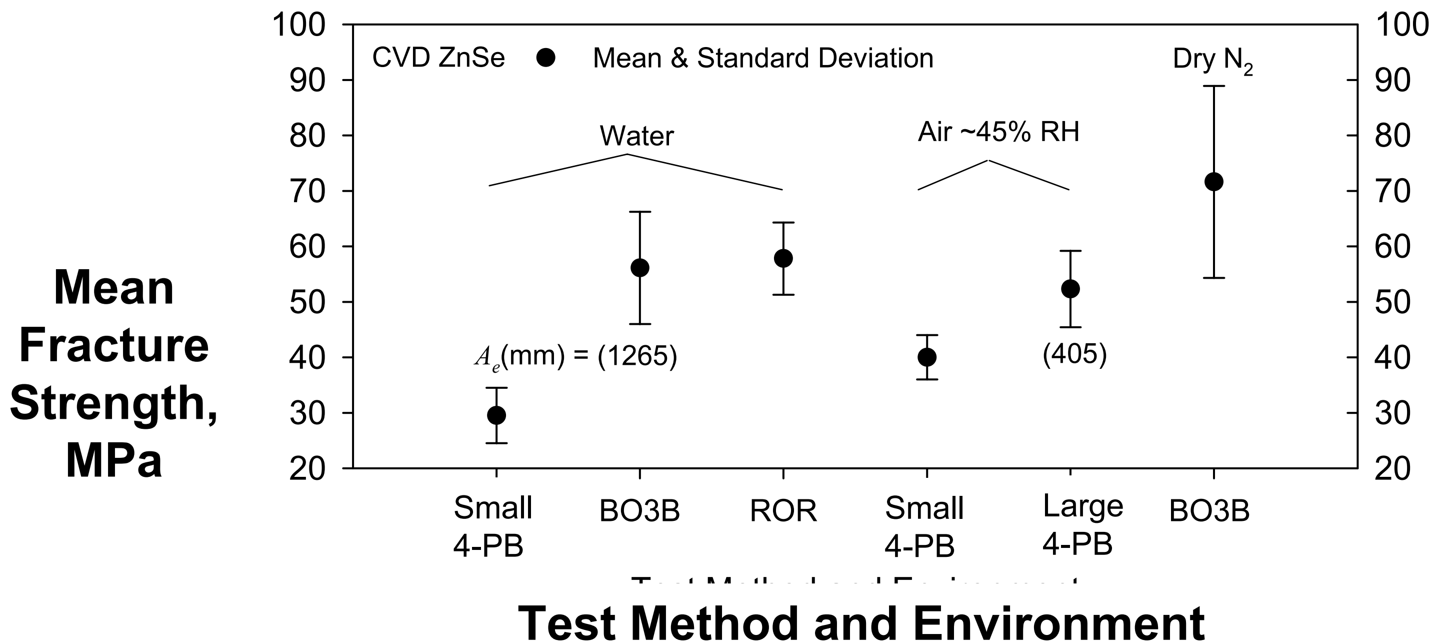
Window Strength
(Ring-on-Ring Loading)



Thin windows:

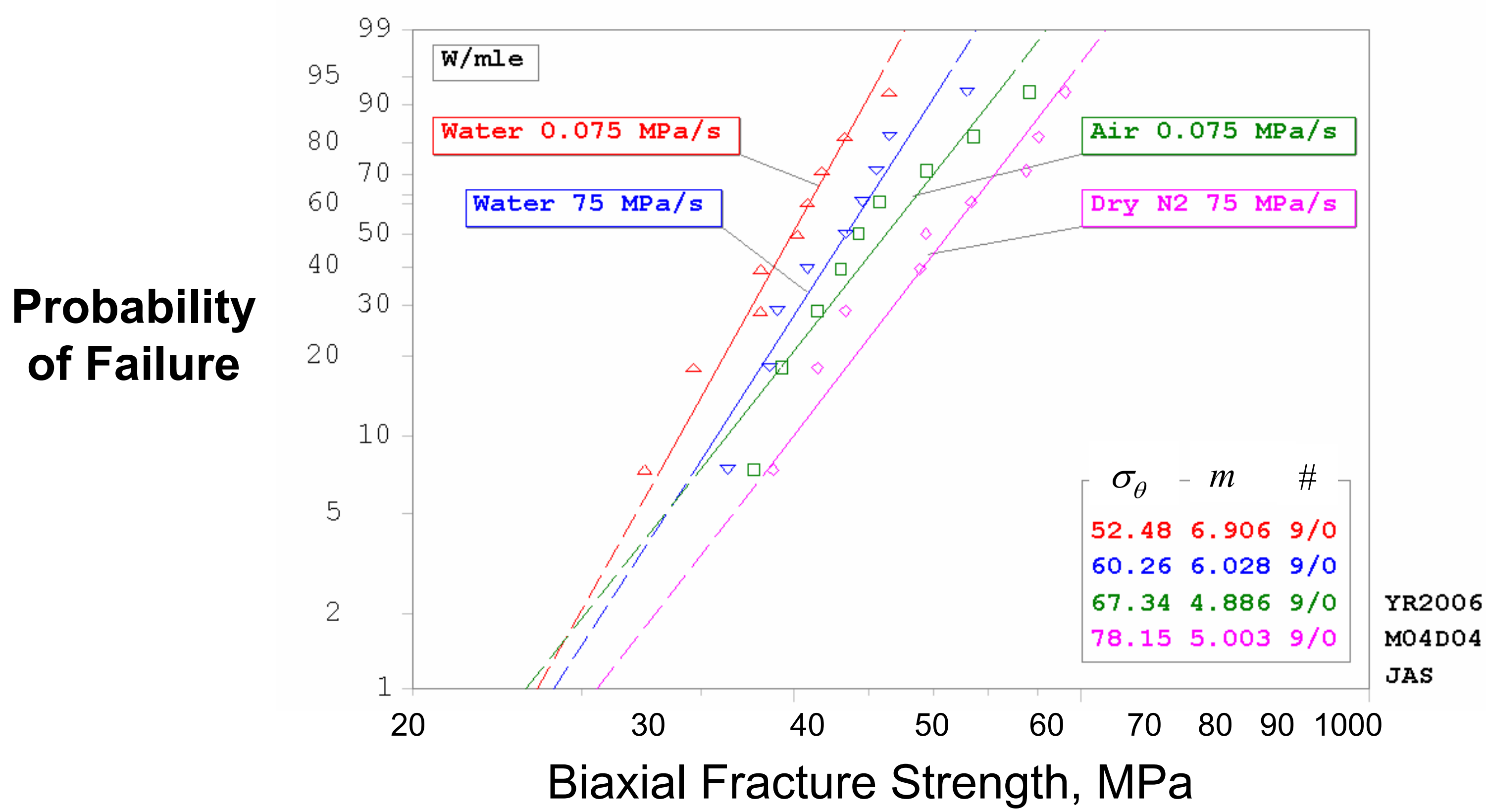


Strength of CVD ZnSe



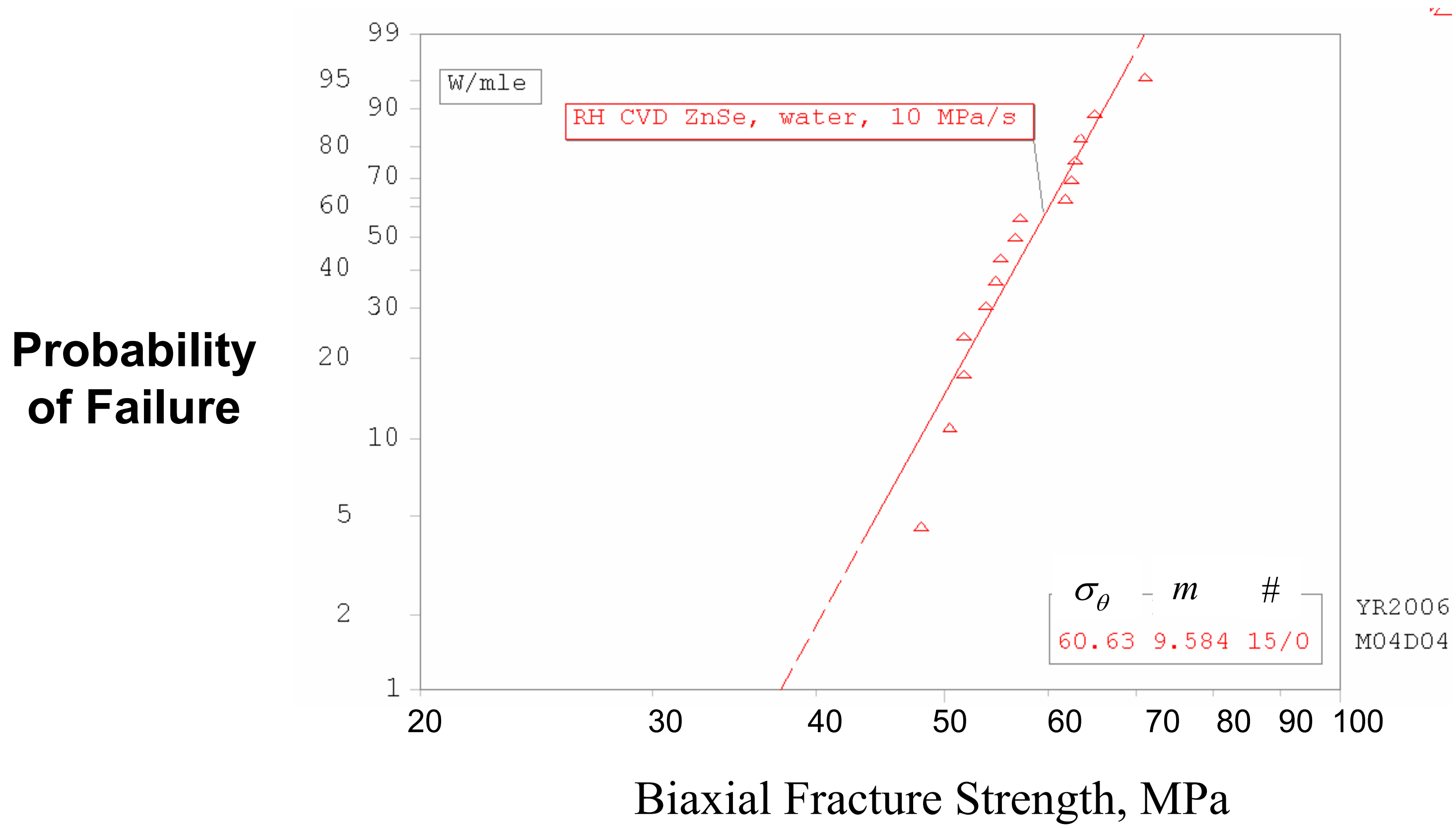
- ❑ For well polished ZnSe, strength is ~55 MPa.
- ❑ Weibull scaling not apparent (study needed), but Need similitude of flaw population.

Weibull Distributions
(Ball-on-3 Ball Loading)



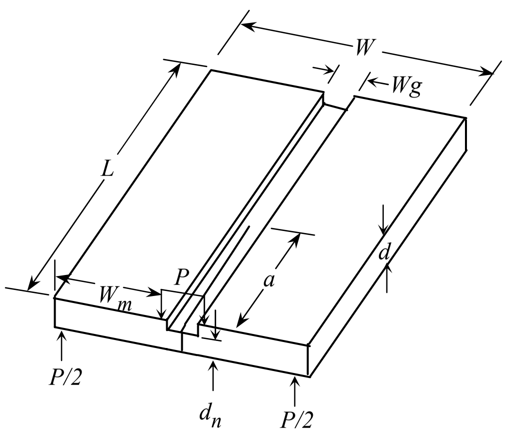
❑ Weibull modulus of ~5, nominal strength of >50 MPa, but strength depends on environment – SCG.

Weibull Distributions
(Ring-on-Ring Loading)

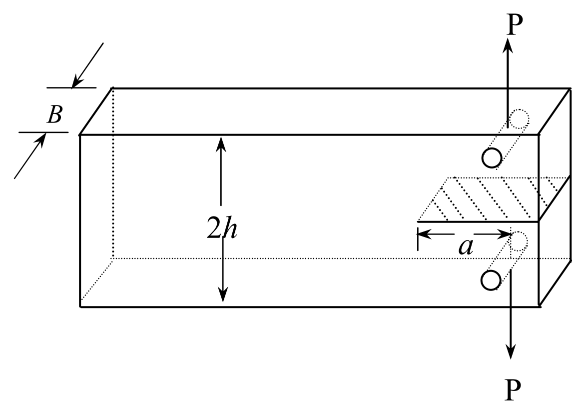


- ❑ Weibull modulus of ~9, nominal strength of ~60 MPa. Agrees with AFML data (139 test, m = 9.2, σ_f = 54 MPa).

Fracture Toughness Methods

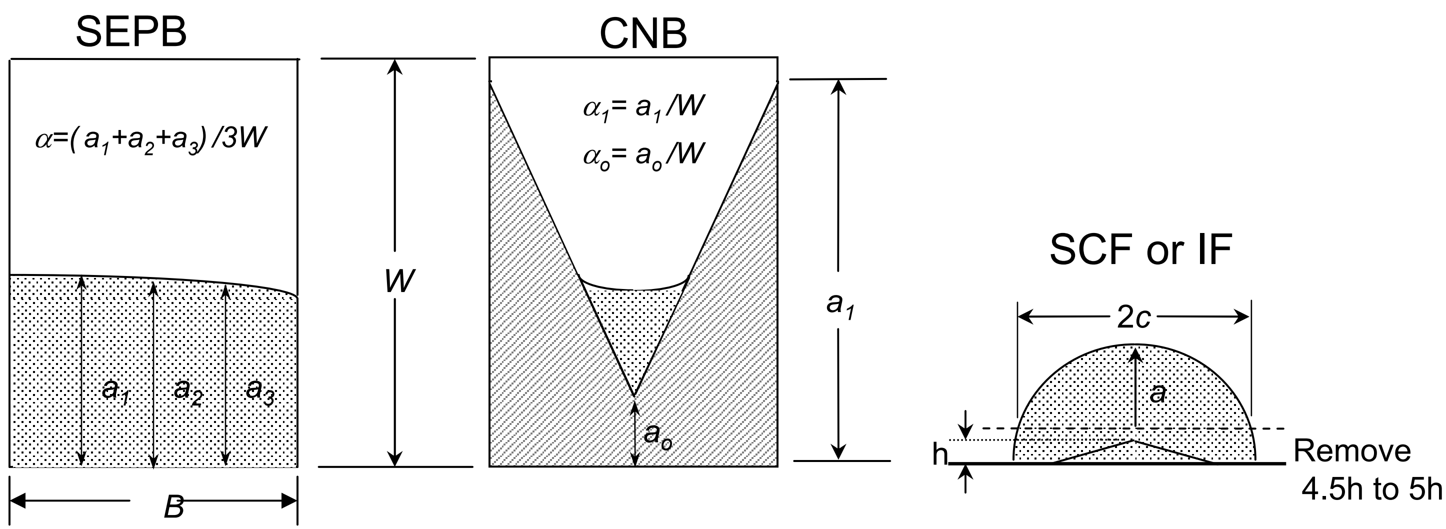


Double Torsion

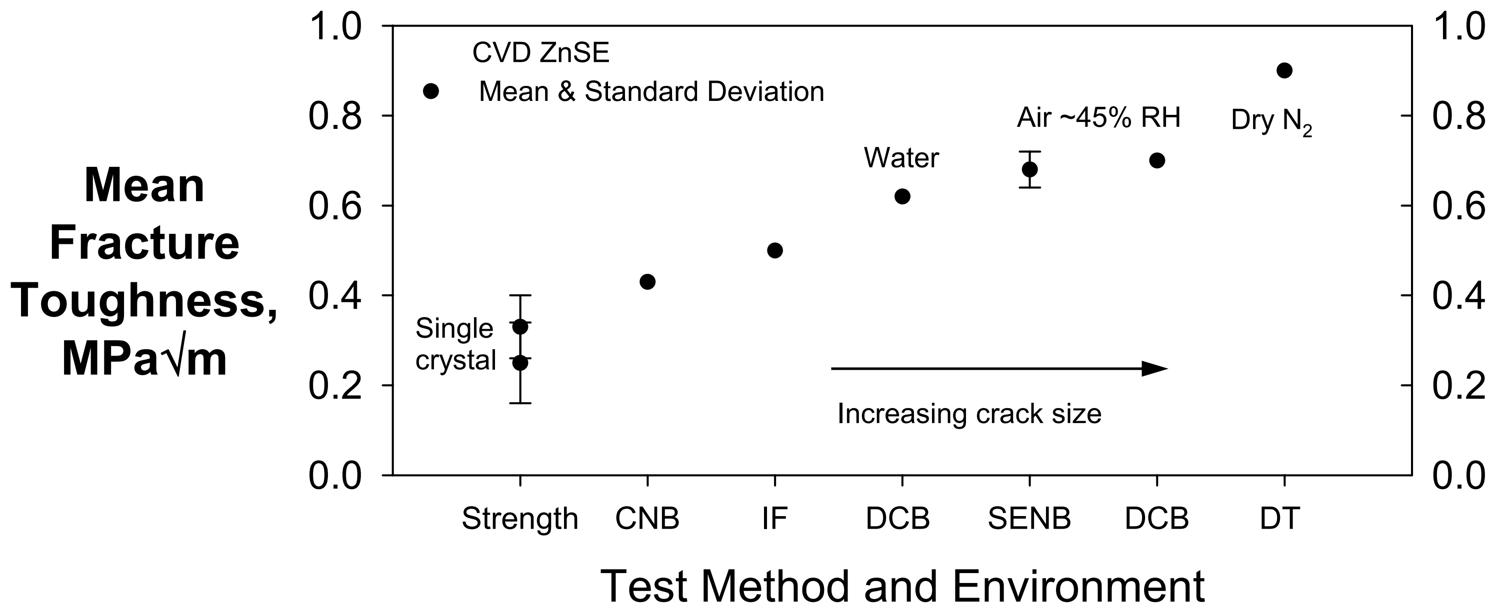


Double Cantilever Beam

Fracture Toughness Methods

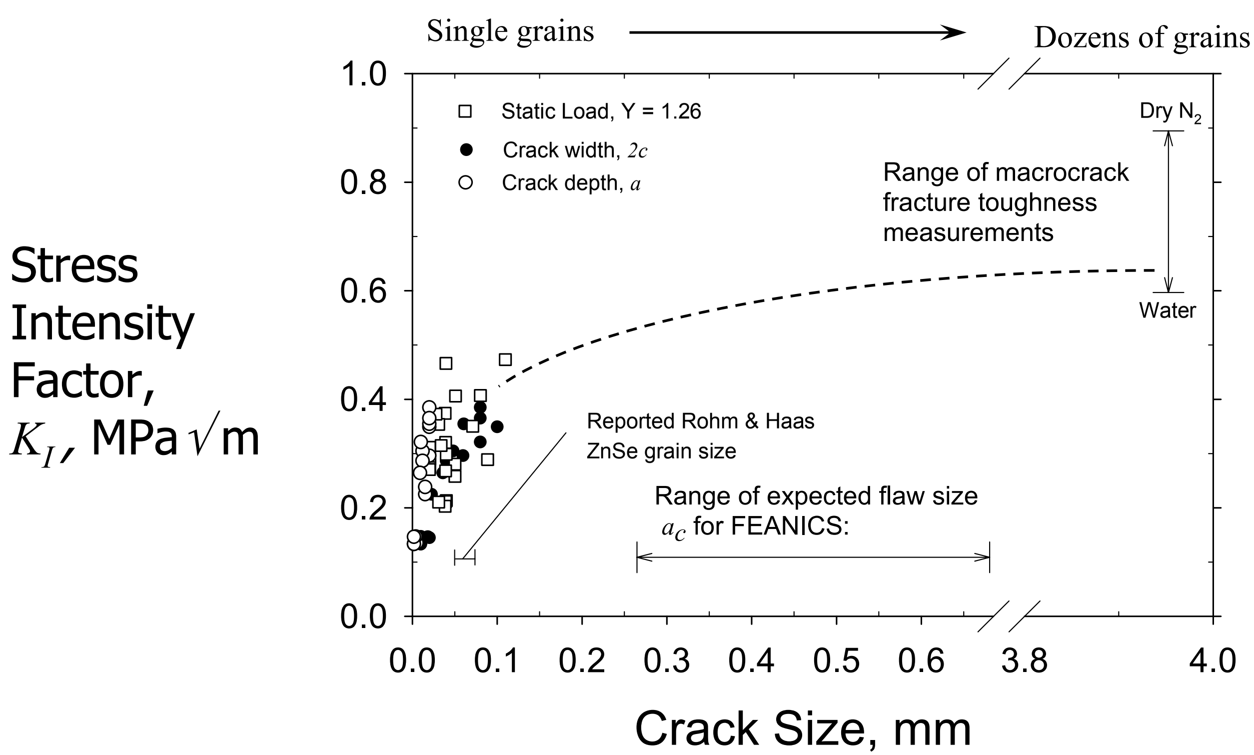


Fracture Toughness



- 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.

R-curve Behavior



- Macro crack fracture toughness is substantially larger than small crack or single crystal values. Grain sampling vs. gain bridging =>R-curve….

Slow Crack Growth

$$(v = AK_I^n)$$

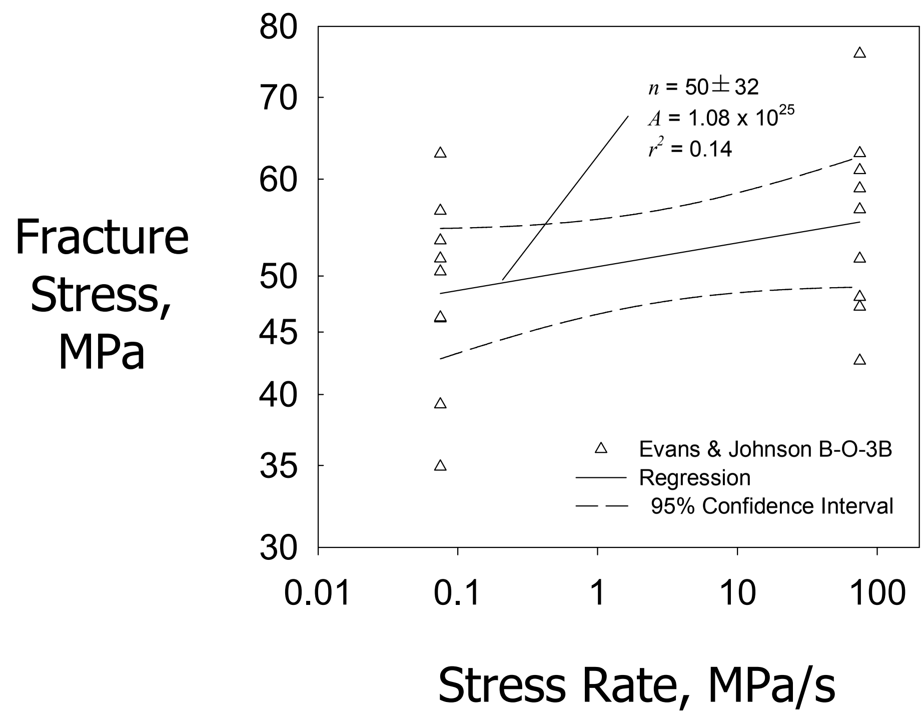
- Three types of SCG data on ZnSe:
  - constant stress rate testing (Dynamic Fatigue)
  - constant stress testing (Static Fatigue)
  - fracture mechanics testing (DCB and DT)

- 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.

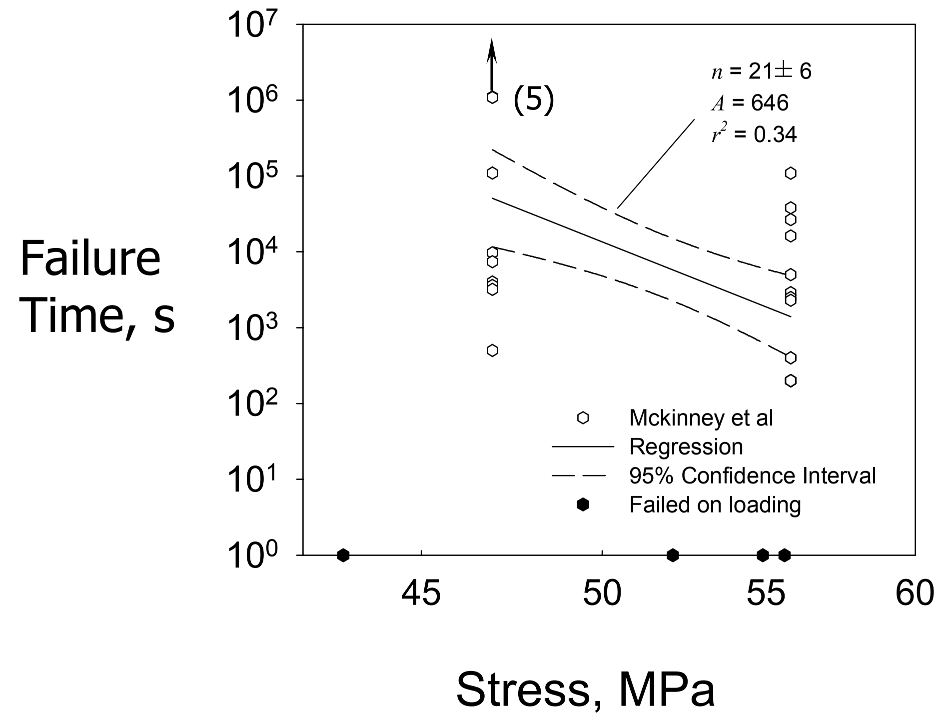
Slow Crack Growth

(Constant stress rate or dynamic fatigue, water)



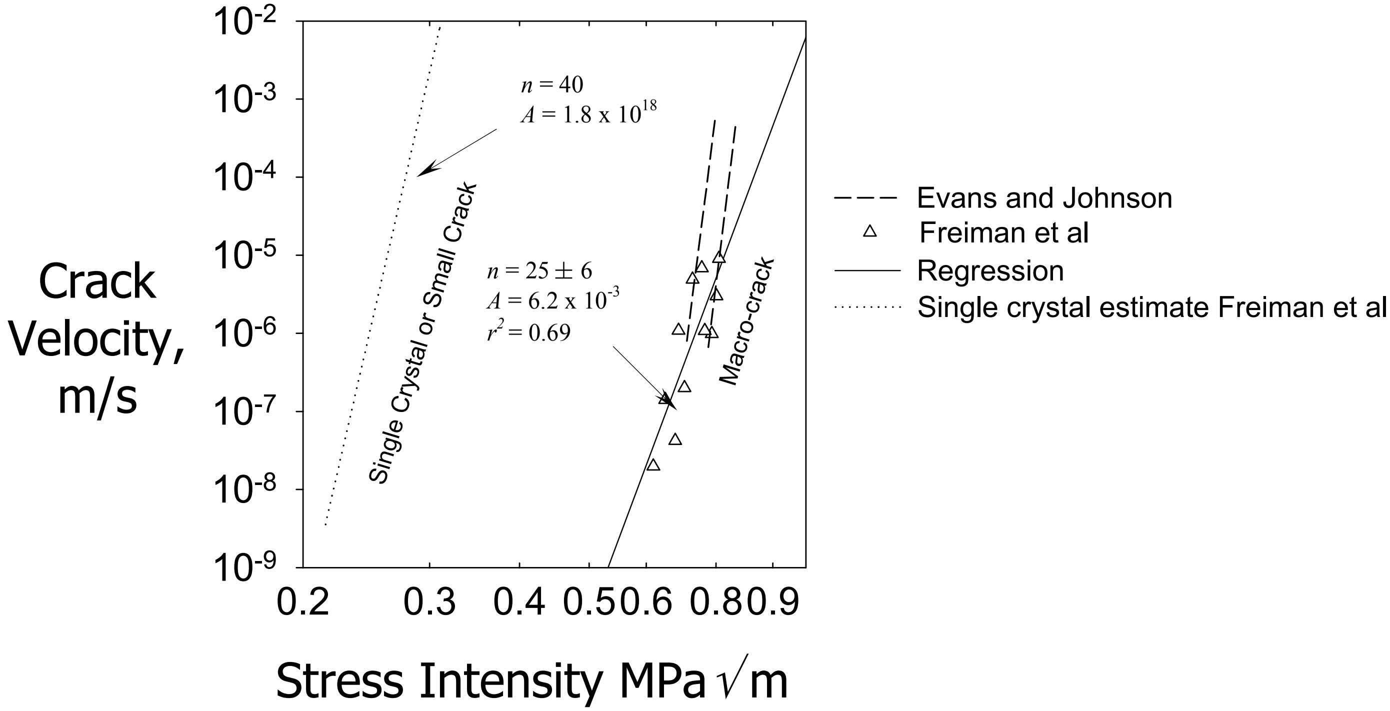
- 18 data points; poor regression coefficient and large standard deviations.

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



- ❑ 29 data points; better regression coefficient and standard deviation; five run outs.

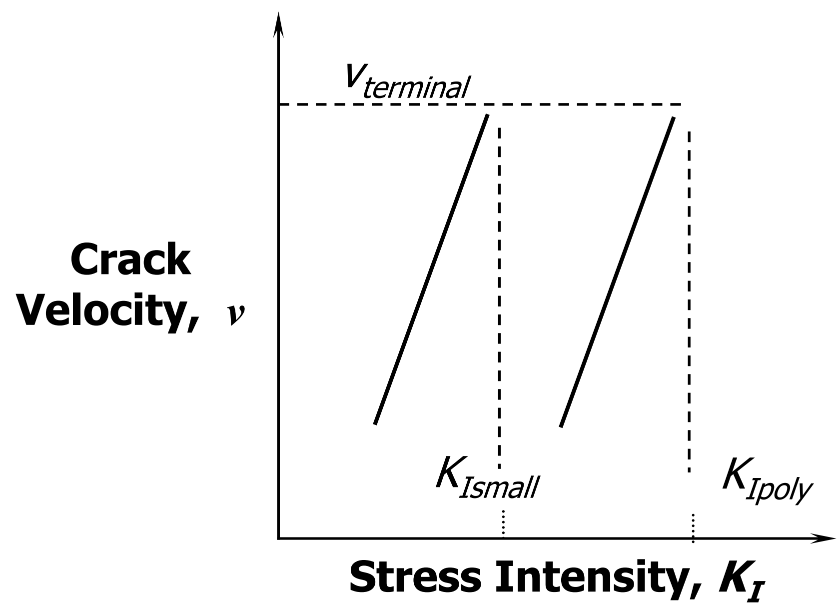
Slow Crack Growth
(DT and DCB in air)



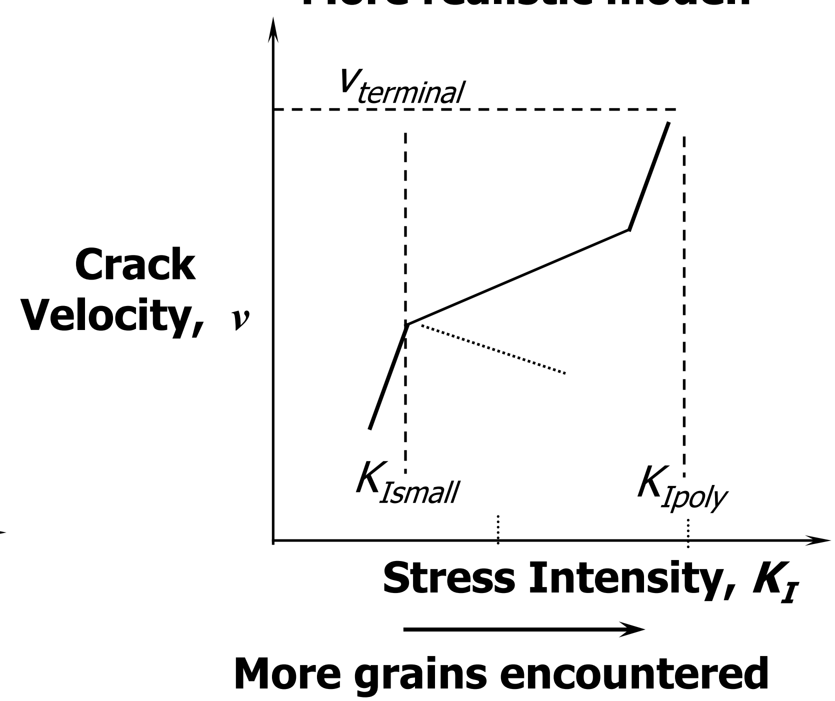
- ❑ Two data regimes macro and small or single crystal.
- ❑ No mathematical explanation of the shift or the assumptions used.

Estimation of Small Crack Parameters from Macro Crack Data

$$v = AK_I^n$$



More realistic model:

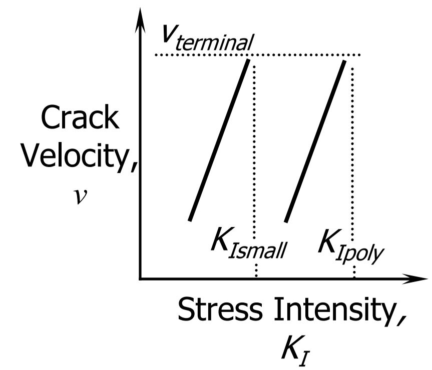


- ❑ Need observation of actual, small crack growth rates.

Estimation of Small Crack Parameters from Macro Crack Data

$$v = AK_I^n$$

- Assume equivalent terminal velocity:

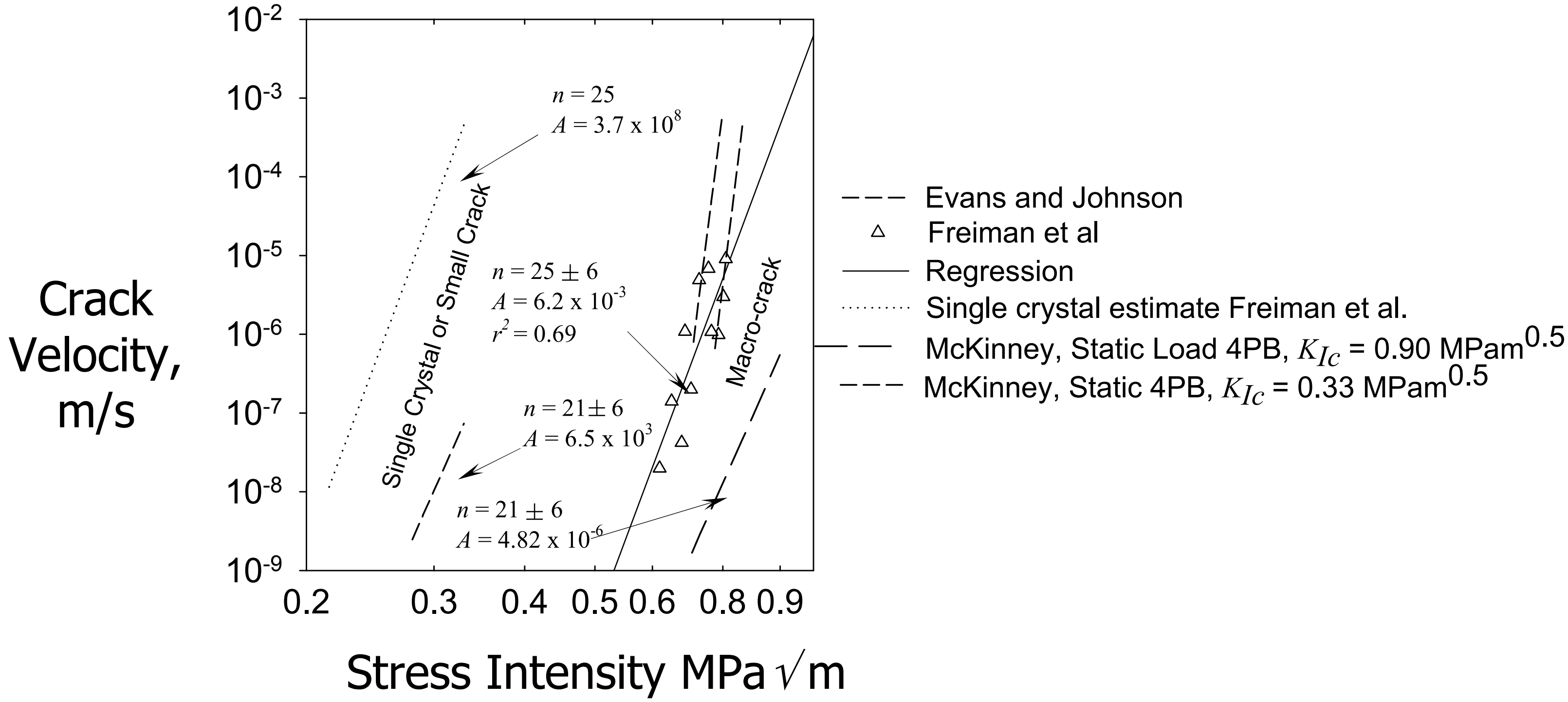


$$v = A_{Poly} K_{IcPoly}^n = A_{Single} K_{IcSingle}^n$$

- Assume identical slopes (n):

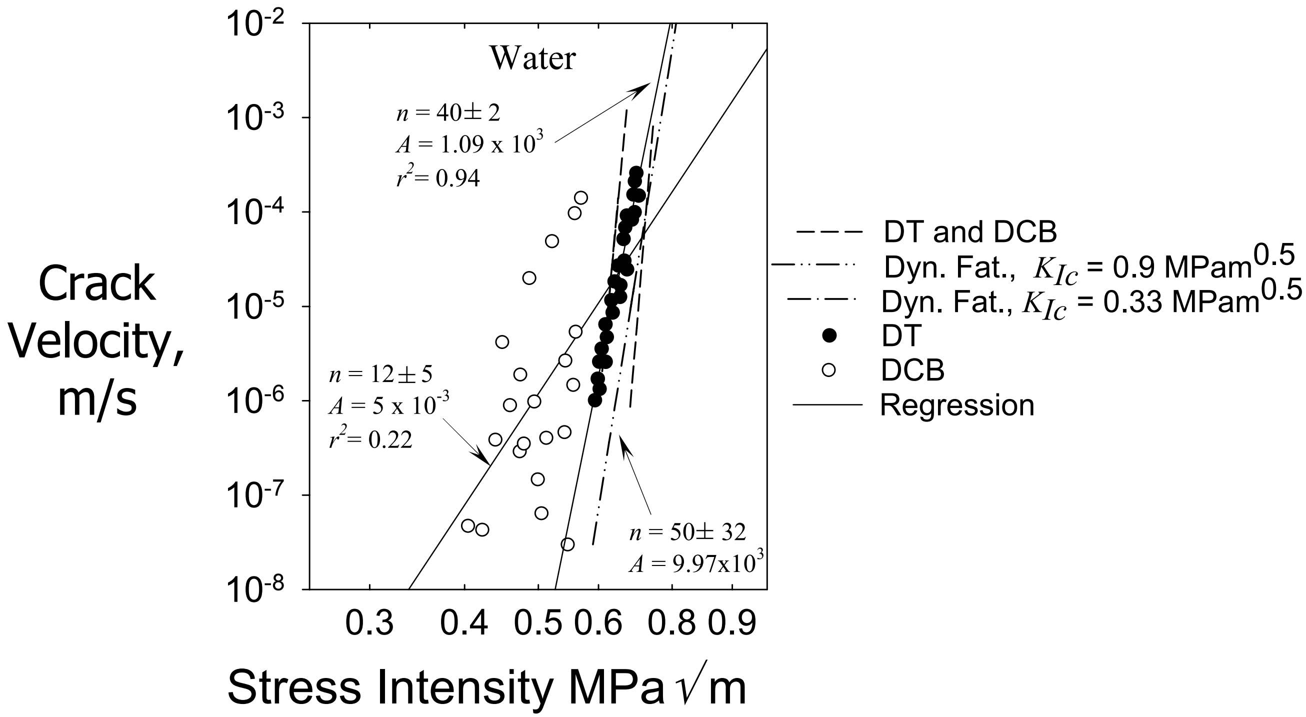
$$A_{Single} = A_{Poly} \left(\frac{K_{IcPoly}}{K_{IcSingle}} \right)^n$$

Slow Crack Growth
(DT, DCB, constant stress in air)



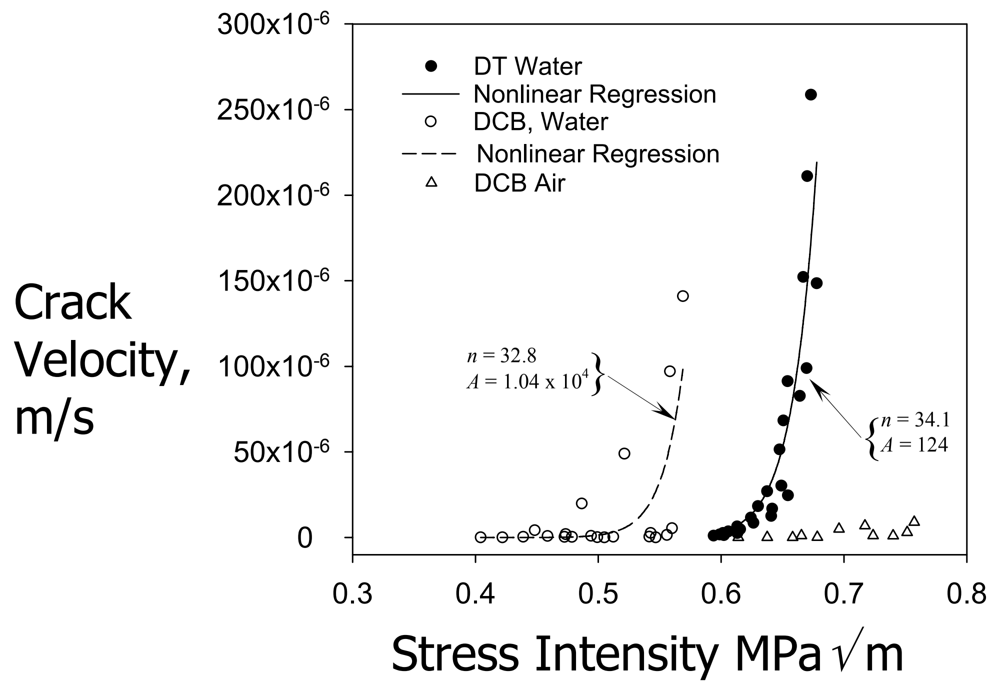
- ❑ Two data regimes: Macro and small or single crystal.
- ❑ Reasonable agreement between small crack and shifted macro crack curves.

Slow Crack Growth
(DT and DCB in water)



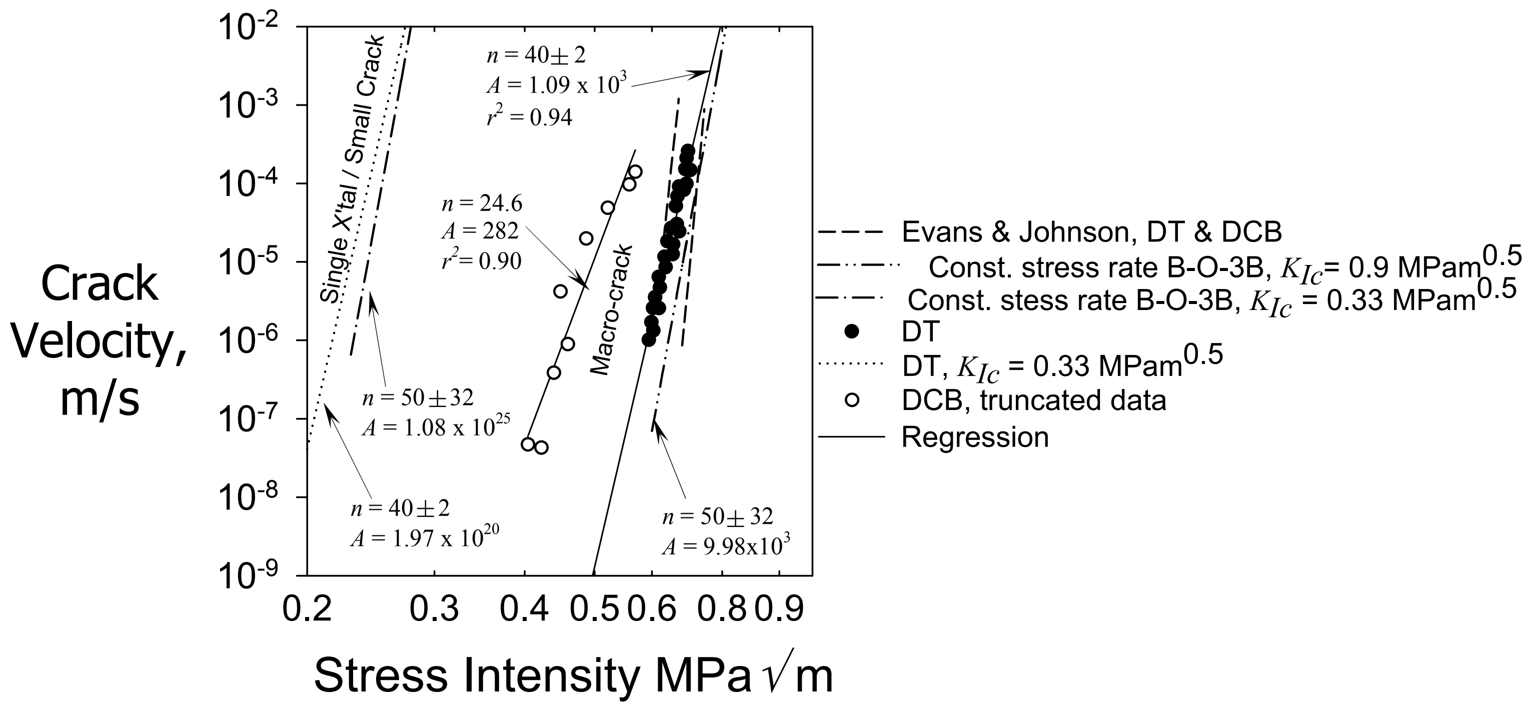
- Consistent DT data, scattered DCB data.

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)



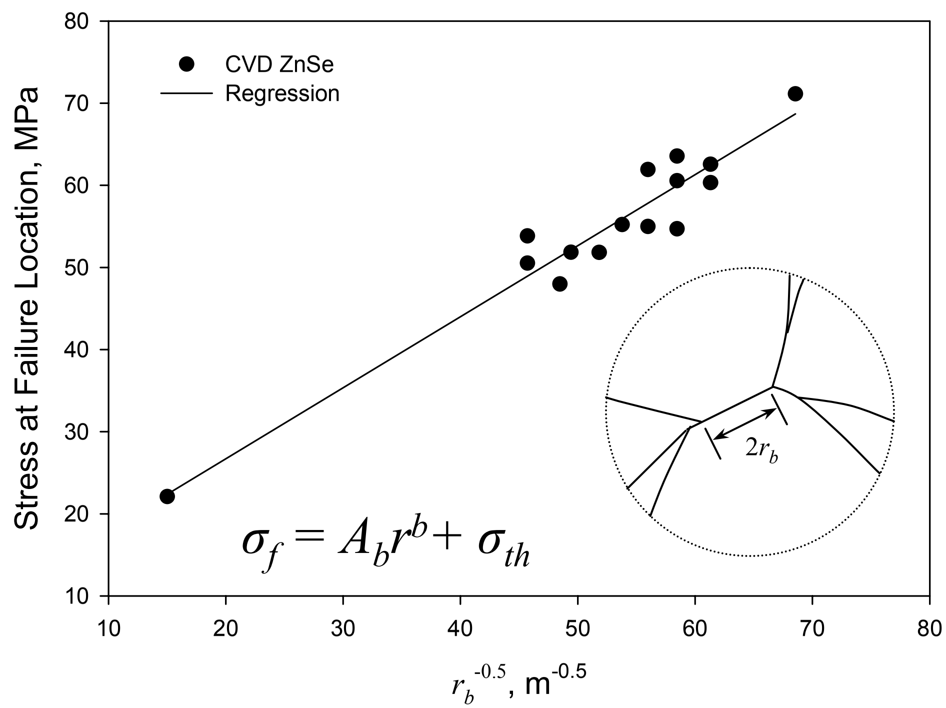
- ❑ Macro crack data and constant stress rate data agree when a maco fracture toughness is used.
- ❑ Shifted macro crack curve and small crack curve agree.

Slow Crack Growth
(Parameter Summary)

Data Source	Test Method (number of tests)	$n \pm SD_n$	A m/s (MPa√m)-n	A_{Small} m/s (MPa√m)-n
Evans and Johnson	DT and DCB, Line	86	1.11×10^{13}	5.05×10^{50}
“ “ “	B-O-3B, Dyn. (18)	50 ± 32	9.98×10^{3}	1.08×10^{25}
“ “ “	DT (25)	**40 ± 2**	$\mathbf{1.09 \times 10^{3}}$	$\mathbf{1.97 \times 10^{20}}$
Freiman et al.	DCB (21)	12 ± 5	5.0×10^{-3}	8.61×10^{2}
“ “ “	DCB (9), Truncated	25	282	1.46×10^{13}
Air				
Freiman et al.	DCB (11)	26 ± 6	6.2×10^{-3}	3.7×10^{8}
McKinney et al. Air	4-point Static (28)	21 ± 6	4.8×10^{-6}	6.5×10^{3}

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

Crack Branching Constant



- Branching constant A_b = 1 ± 0.1 MPa√m, residual stress = 9 MPa.

SUMMARY

- Based on 1970's data, the single crystal or small crack SCG parameters for CVD ZnSe in water are $n < 40$, and $A_{single} > 10^{20}$, $K_{Ic} = 0.33$ MPa$\sqrt{m}$.
- The macro-crack parameters are much different: $25 < n < 80$ and $A_{Poly} = 10^3$, $K_{Ic} > 0.60$ MPa$\sqrt{m}$.
- Strength is ~ 55 MPa with a Weibull modulus of ~ 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.