Presentation to: Gruppo Frattura Italia

Fracture toughness testing of advanced technical ceramics – standards development

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Overview

- Objectives of facture toughness testing
- Behaviour of cracks
- Fracture mechanics
- Criteria for good test results
- Standardised and non-standard testing methods
- Standardised methods
  - Experimental considerations
  - Validation
- Conclusions/recommendations

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What are we trying to achieve in fracture toughness testing of relatively brittle materials?

- Data for understanding material development directions
- Data for the correct material ranking for applications involving:
  - Wear
  - Impact
  - Chipping resistance
- Data for fractographic investigations
- Data to support subcritical crack growth investigations

But it’s a bit of a minefield…..

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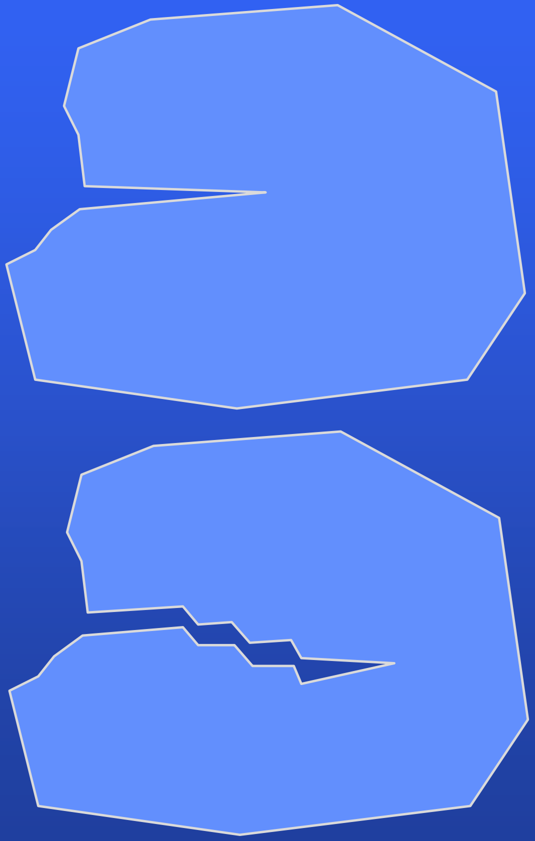
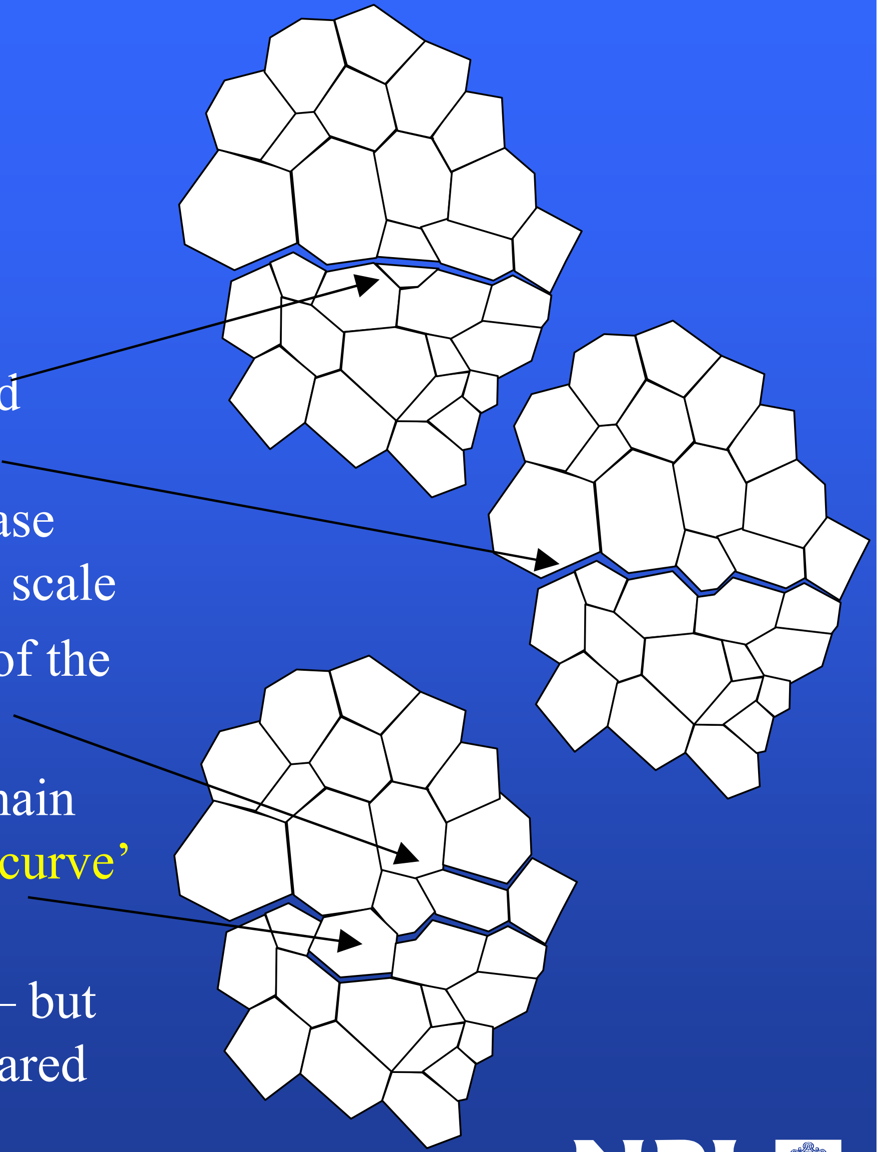
Behaviour of cracks in brittle materials



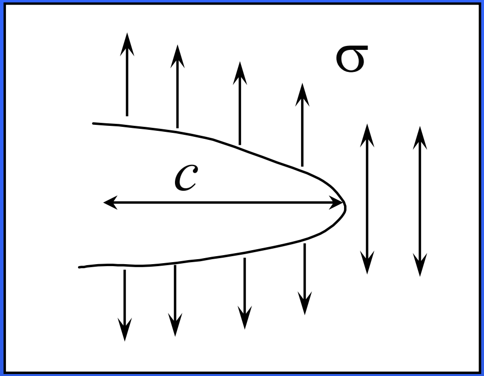
- Glass
  - Homogeneous, isotropic, featureless structure
  - ‘Griffith flaws’
  - ‘atomically sharp’
  - subcritical growth
  - behaviour independent of size of the crack
- Single crystals
  - Crystallographic structure
  - Preferred cleavage directions
  - Controlled by anisotropic elasticity and surface energy

Behaviour of cracks in brittle materials

- Polycrystalline materials
  - Cracks can run through grains (transgranular fracture) or around grains (intergranular fracture) depending on crack velocity, phase composition and microstructural scale
  - Microcracking can occur ahead of the main crack tip (‘process zone’)
  - Wedging can occur behind the main crack tip – leads to so-called ‘R-curve’ behaviour
  - Much more complex behaviour – but generally better toughness compared with single crystals or glass



Fracture mechanics



- Griffith relationship for a through crack in a plate

$$\sigma_f = \sqrt{E\gamma / Ac}$$

- Stress intensity relationship for fast fracture

$$\sigma_f = K_{Ic}/Y\sqrt{c}$$

where Y is a crack shape parameter

So, to get a measure of K_{Ic} we need to measure local stress, crack shape and crack length

Usual assumptions concerning K_{Ic} when dealing with ceramics:

- Linear elastic behaviour
- Crack tip is sharp
- Crack shape is effectively planar (despite roughness of fracture surface)
- No subcritical crack growth
- No effect of environment if we do it fast enough
- No crack face tractions, i.e. no R-curve
- No residual stresses

Therefore:

- Test methods need to achieve these assumptions as closely as possible

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Principal condition for a good test

- Stress distribution about the crack tip is well-defined and calculable

Consequently:

- For ‘proper’ answers, this rules out indentation methods!!

Geometries – main considerations

To achieve reliable results, the test geometry:

- Needs to give an experimentally reproducible outcome
- Should not be too difficult to make, to create a sharp pre-crack in, and to test reliably
- Should not require too much material
- Should allow the straightforward introduction of a sharp crack
- Should develop a well-known stress distribution from simple application of force
- Should have minimal uncertainties in calibration equations

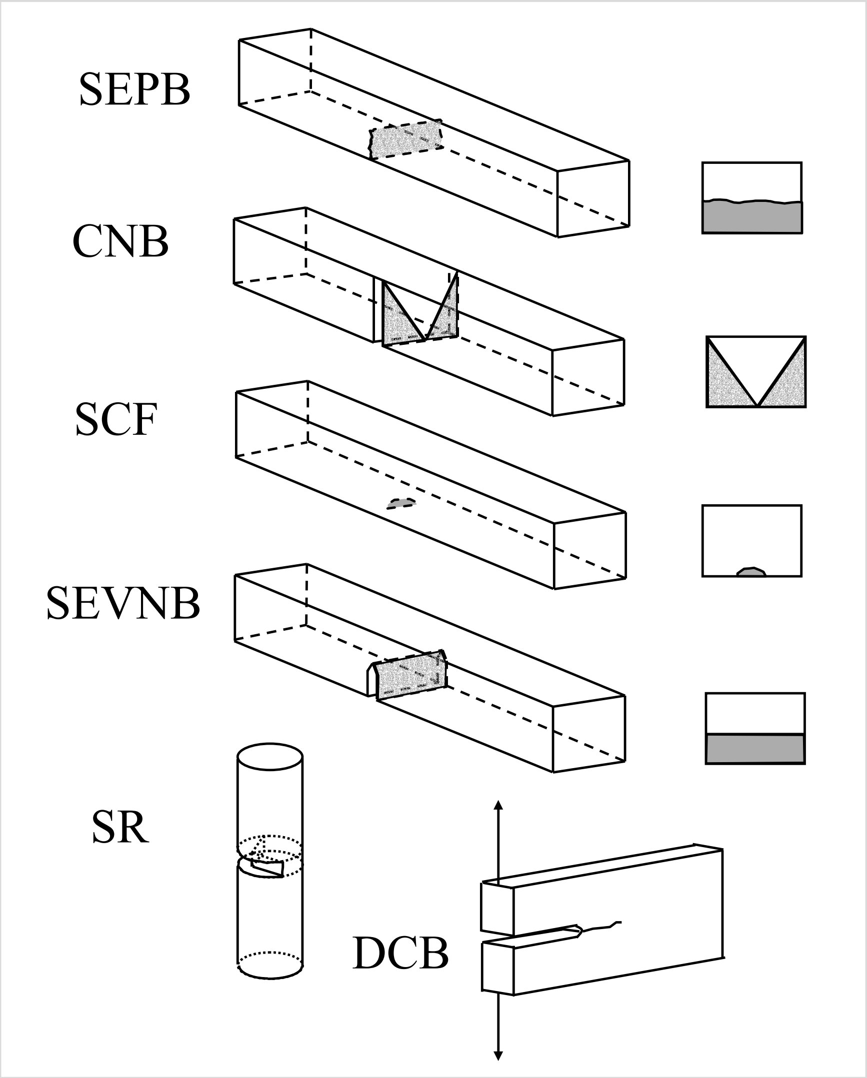
Choice of geometries

‘GOOD’:

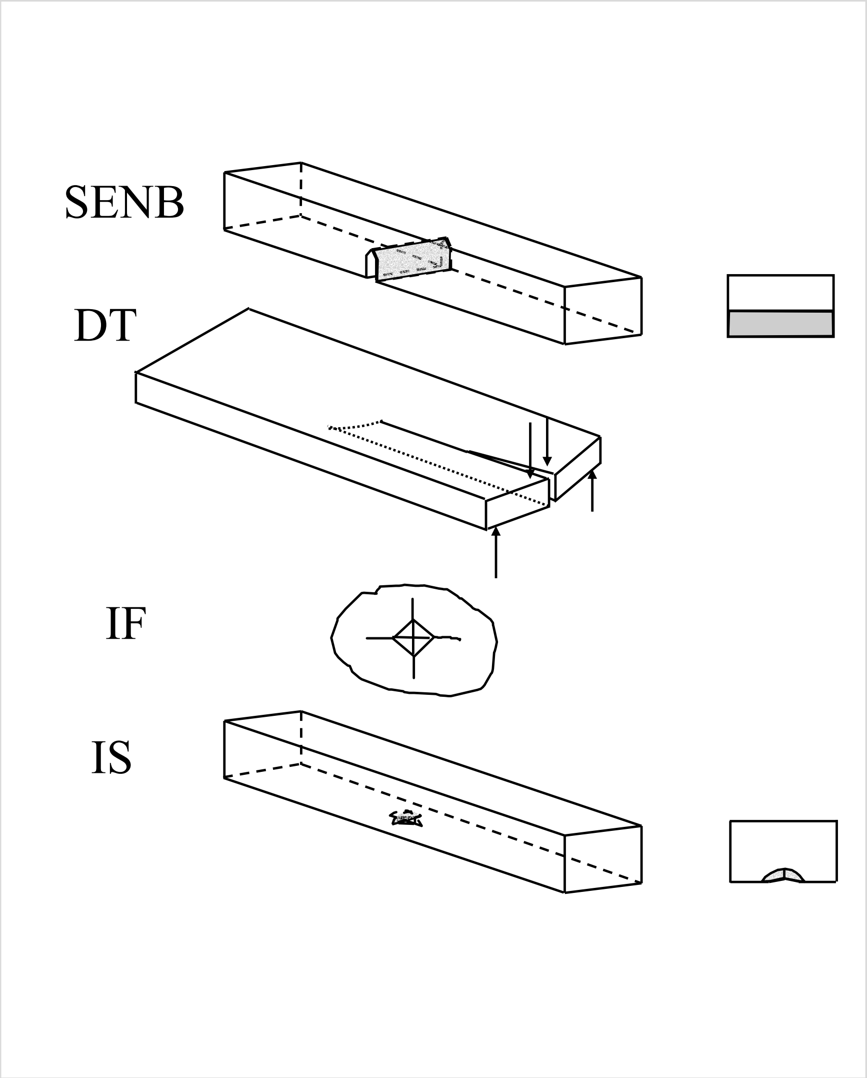
- Single edge pre-cracked beam (SEPB)
- Chevron notch (CNB)
- Surface crack in flexure (SCF)
- Single-edge Vee-notch beam (SEVNB)
- Short chevron notched rod (SR)
- Double cantilever beam (DCB)

Note:

- First four advantageously based on standard flexural strength test-pieces
- Rod and plate more difficult and not generally used



Choice of geometries



'BAD':

- Single edge notched beam (SENB) – not a sharp crack – tends to overestimate toughness
- Double torsion (DT) – uncertainties concerning crack length and mixed mode
- Indentation fracture (IF) – uncertainties concerning residual stress field – result indent load dependent and subjective
- Indentation/strength (IS) – uncertainties concerning residual stresses – result indent load dependent

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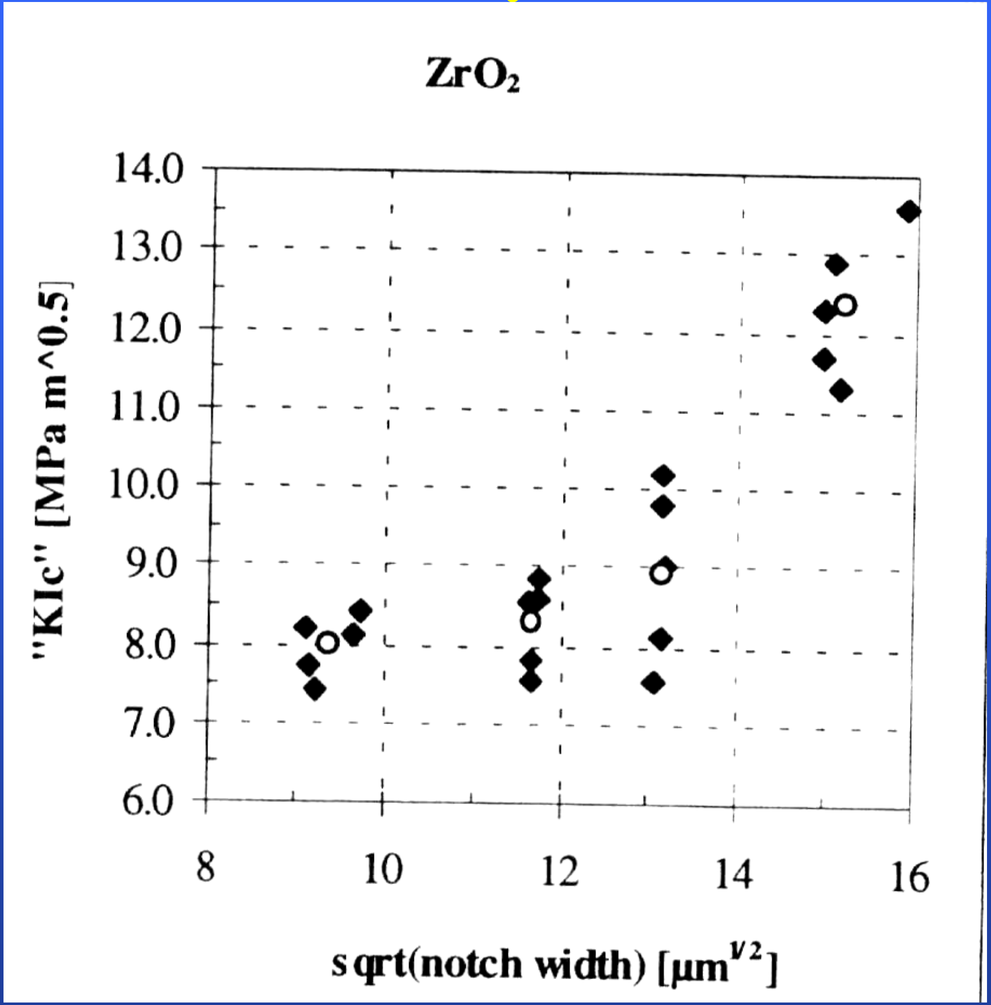
SENB - Notch root radius sensitivity

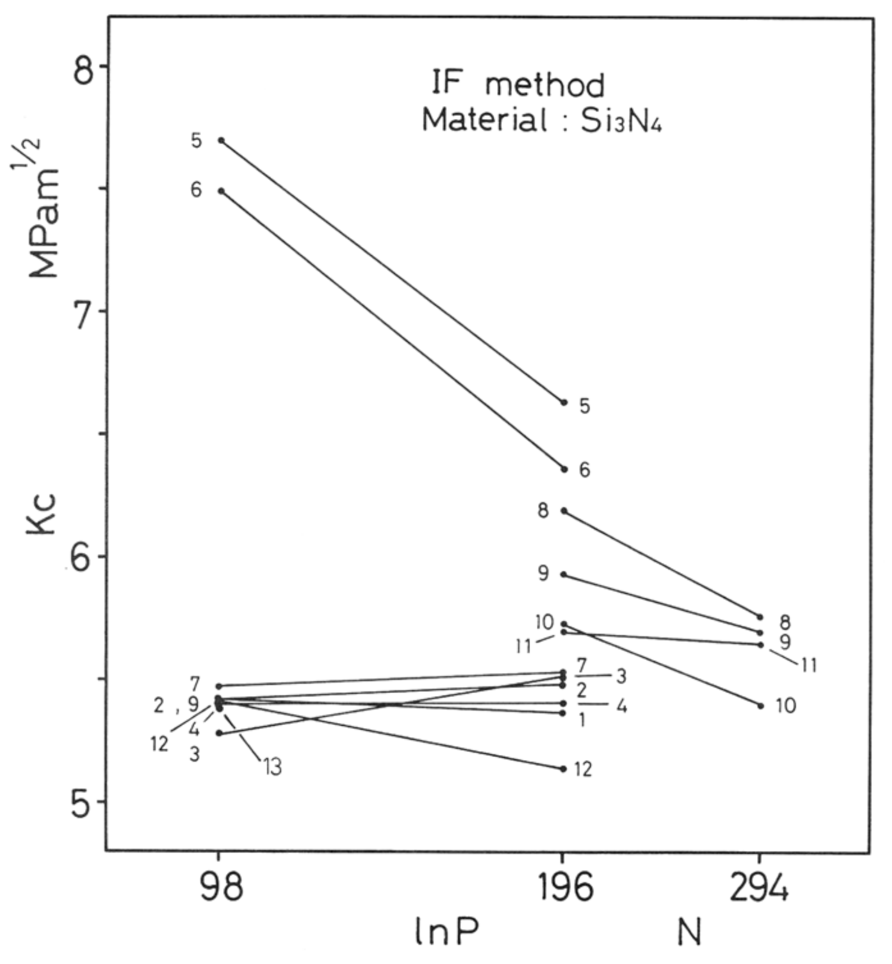
- In materials in which cutting the notch induces high residual compressive stress, SENB results are highly root radius dependent



Source: Primas and Gstrein,
ESIS TC6 RR, October 1995

Indentation Fracture (IF)- indent load dependence

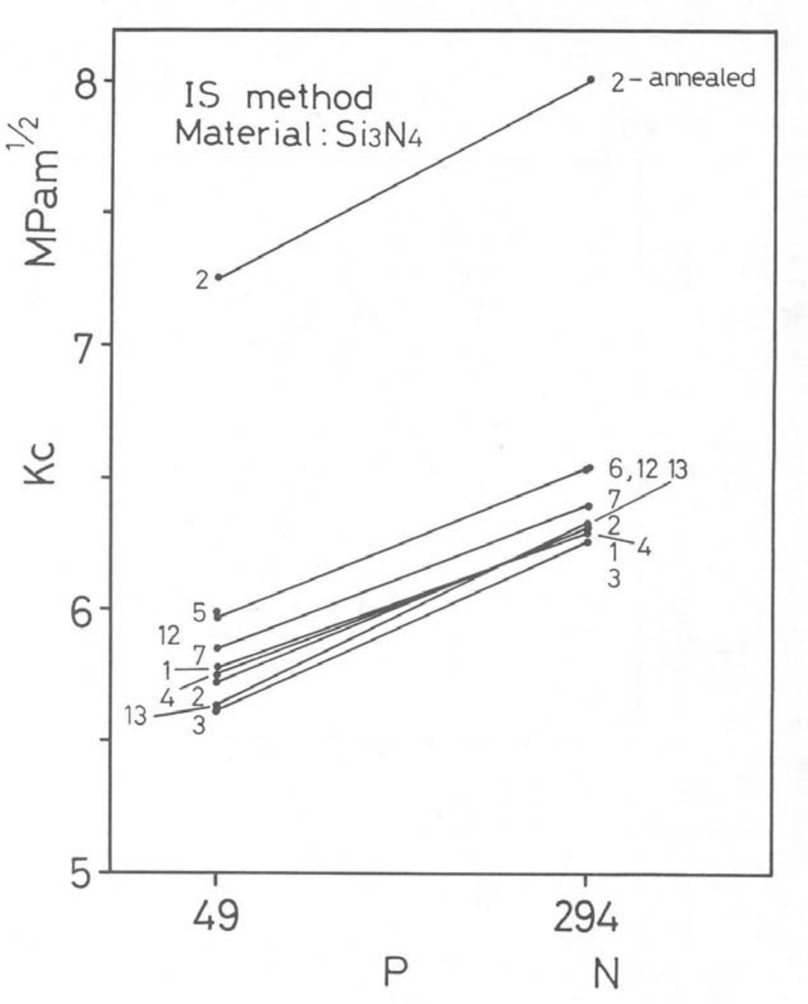
- Equations are based on assumptions about stress fields (many of them!)
- Not a fast fracture method, but often matched to 'true' K_{Ic} of questionable pedigree
- Can be indent force dependent
- High scatter in most materials because crack paths are microstructure dependent



Source: Awaji et al. VAMAS report No. 8, 1990

Indentation Strength (IS) – indent load dependence

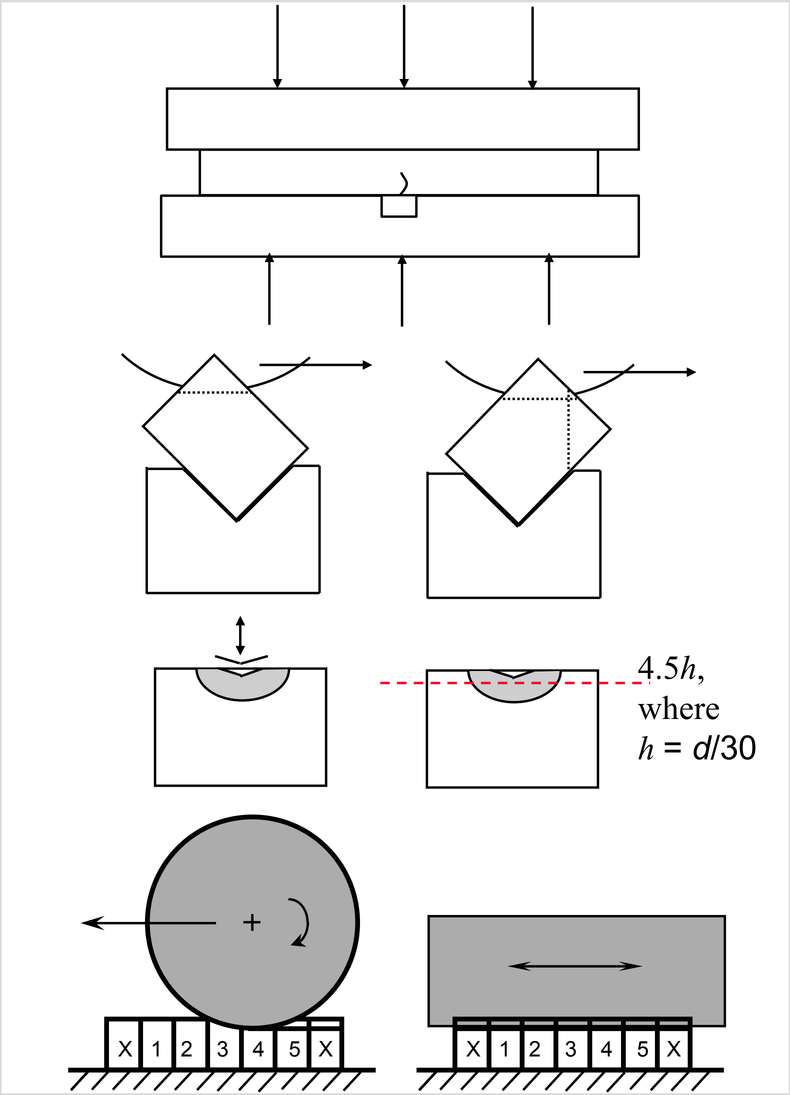
- Assumes half-penny shaped crack around the indent
- Smaller scatter than IF method, but result is indent force dependent
- Requires correlative matching with reliable 'true' K_{Ic} data (most data are of questionable pedigree) to account for residual stresses



Source: Awaji et al. VAMAS report No. 8, 1990

Producing starter cracks

- SEPB – bridge indentation method
  - need a bridge jig
- CNB – chevron with sharp end
  - need accurate sawing of notch
  - advantageous to use a Vee blade
- SCF – Knoop indentation flaw with removal of residual stress zone (4.5 x indent depth)
  - need indenter plus grinding/ polishing
- SEVNB – razor blade and diamond paste honing
  - easier with reciprocating machine



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SEPB – Bridge pre-cracking

- Critical part of process
- Jig must be accurately machined
- Jig should not be too stiff – prevents flexing of test-piece into gap
- The gap may need to be adjusted for different materials
- Use a single indent or a row of three indents to initiate the crack
- Loading alignment must be good to get a straight crack
  - discard test-pieces with >10% variation in crack length across width
- Pre-crack length to be 20-50% of test-piece thickness
- Testing is a simple flexural loading test
- Calculation based on Srawley and Gross notch beam equations adjusted for actual span conditions

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SEPB - calculations

◆ Valid for cracks depth *a* with $0 < \alpha = a/W < 0.6$

$$K_{Ic} = \frac{F}{B\sqrt{W}} . \frac{S_1 - S_2}{W} . \frac{3\sqrt{\alpha}}{2(1-\alpha)^{3/2}} .Y^*$$

$$Y^* = 1.9887 - 1.326\alpha - (3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)(1+\alpha)^{-2}$$

where

F is the fracture force

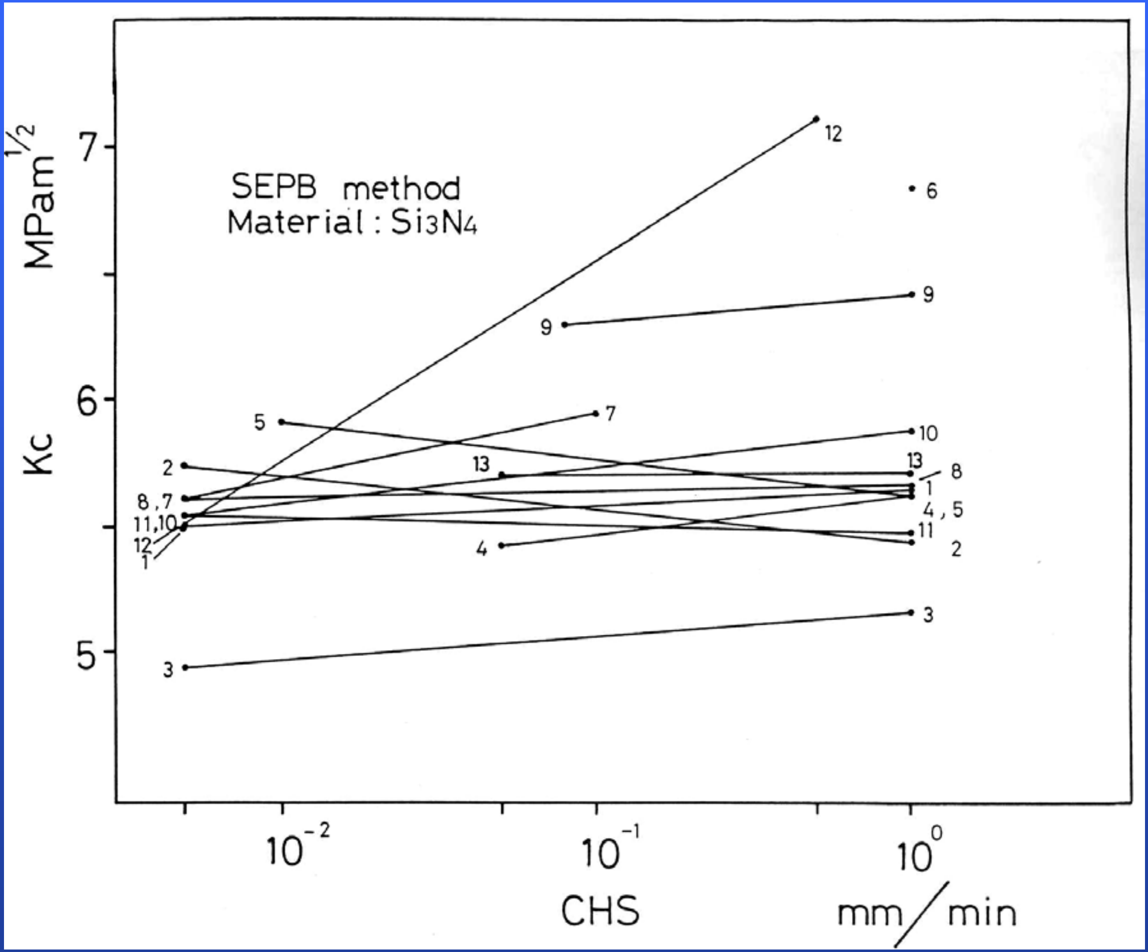
W is test-piece depth

B is the test-piece width

S_1, S_2 are the spans

SEPB - test validation

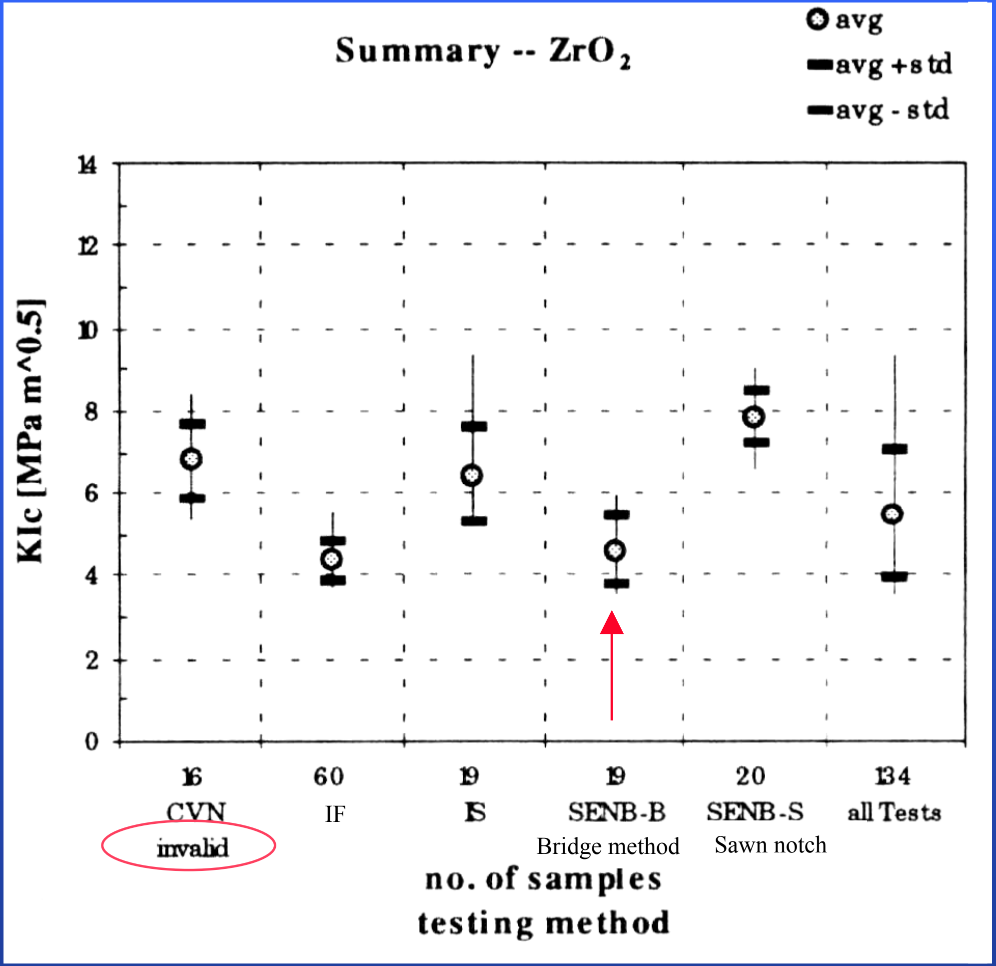
- Most labs in a 1990 RR obtained fairly consistent results
- Main issue is with the construction of the bridge jig and test-piece alignment.



Source: Awaji *et al.* VAMAS report No. 8, 1990

SEPB – test validation

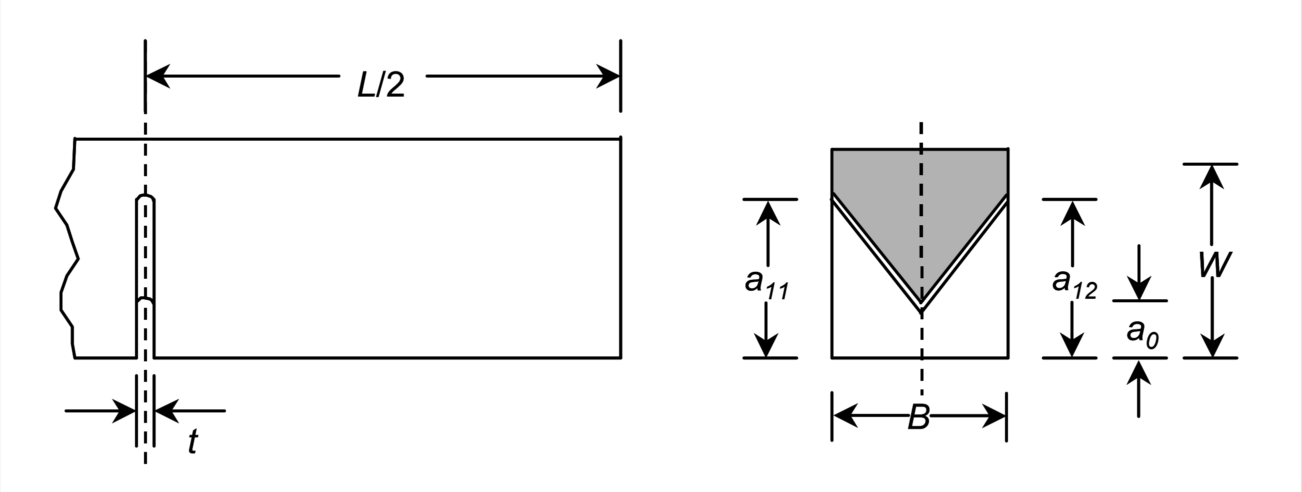
- SEPB (= SENB-B in figure) has lowest toughness consistent with valid sharp crack geometry and minimal residual stress
- Note that CVN tests are invalid, and IS gives high results



Primas and Gstrein,
ESIS TC6 RR, October 1995

CNB – experimental issues

- Two sides of notch to be coplanar and symmetrical
- Ideally, test machine should be stiff to optimise the chances of stable crack growth
- Calibration equations are based on Bluhm slice model, but vary from source to source.
- A valid test is one in which there is a clear progressive peak in fracture force



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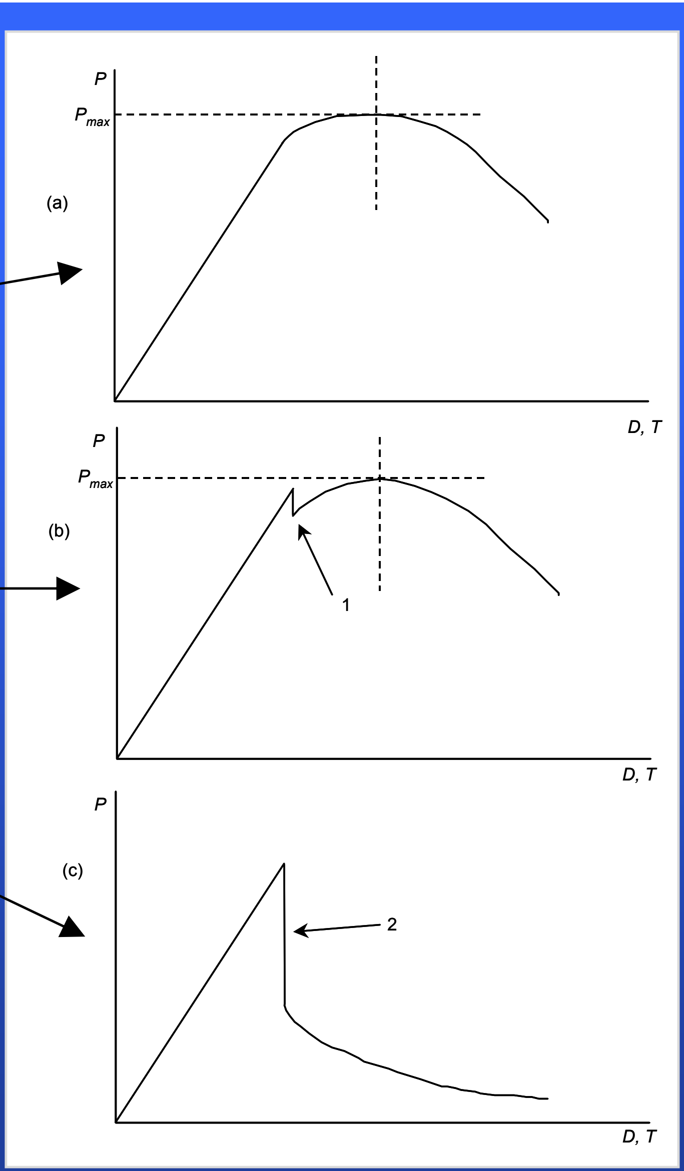
CNB – valid and invalid test behaviour

- Smooth initiation and smooth growth - **valid**
- ‘Pop-in’ initiation and smooth growth - **valid**
- Uncontrolled pop-in and fracture – **invalid**

Invalidity can be caused by lack of system stiffness



CNB - equations

- Valid for notches with $0 < \alpha_0 = a_0/W < 0.1$, $0.95 < \alpha_1 = a_1/W < 1.0$

$$K_{Ic} = \frac{F_{\max}}{B\sqrt{W}} Y$$

$$Y = (3.08 + 5.00\alpha_0 + 8.33\alpha_0^2)\left(1 + 0.007\frac{\sqrt{S_1 S_2}}{W}\right)\left(\frac{\alpha_1 - \alpha_0}{1 - \alpha_0}\right)\left(\frac{S_1 - S_2}{W}\right)$$

Fett and Munz

- Considered accurate to within 4%
- More accurate versions exist for specific cross-sectional geometries (i.e. limited ranges of span and notch sizes - see e.g. ASTM)

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CNB – philosophical issues

- If controlled growth is required, is the *K*-value determined really K_{Ic}?
  - Possibly not for environmentally sensitive materials
- Controlled growth is easier to get with stiff systems, but the will the crack velocity be even lower?
  - Unclear if sufficient research has been done
- Can R-curve behaviour be deconvoluted?
  - Different parts of the crack have propagated different distances, so probably not
- Analysis assumes straight crack front, but experimentally often not the case – does this matter?
  - Probably a manifestation of R-curve effects or cracks running out of the notch root

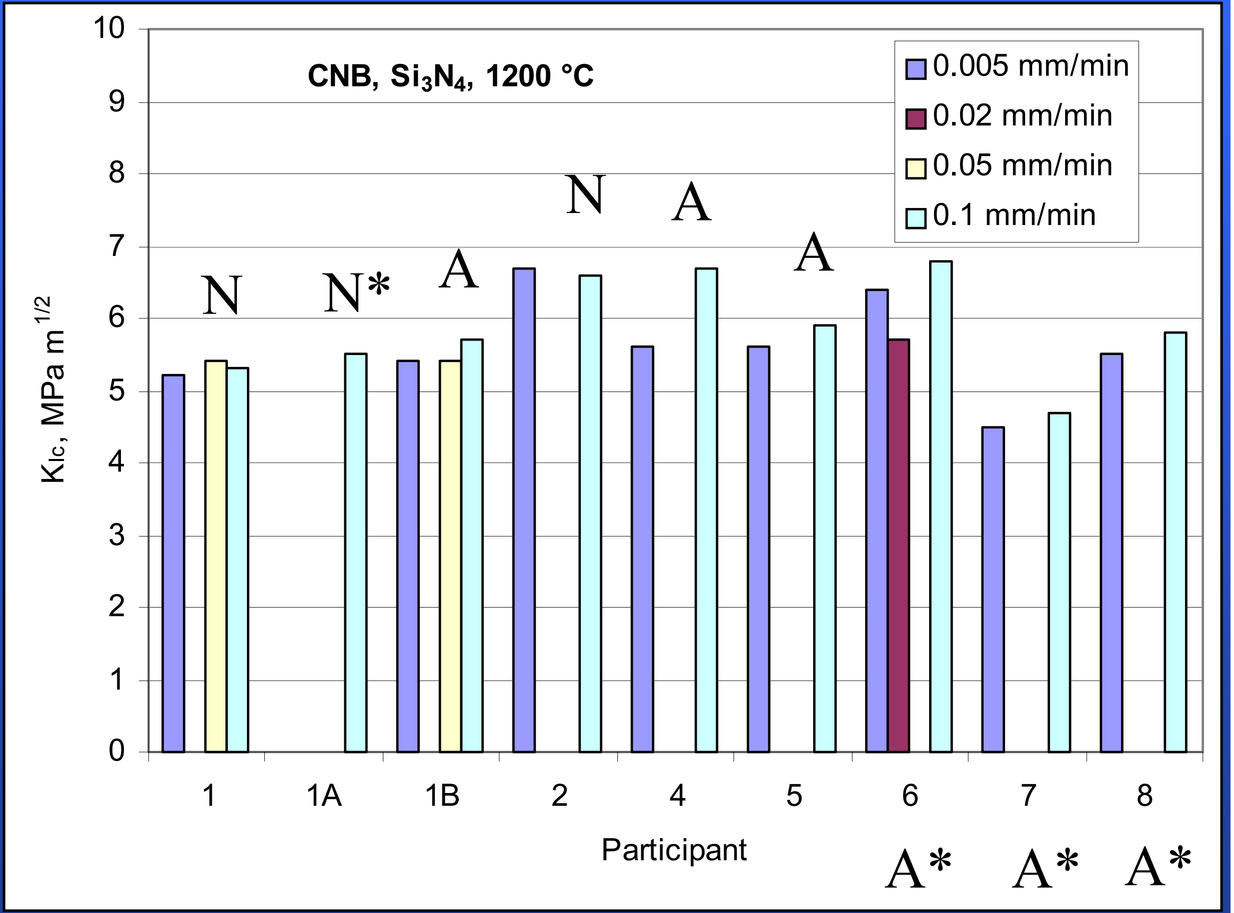
CNB – test validation

A, A* = 30/10, 40/20 mm spans in air

N, N* = 30/10, 40/20 mm spans in N_2

Note: Lab 1 used Vee-shaped notches – low scatter,

<±0.2 MPa $m^{1/2}$



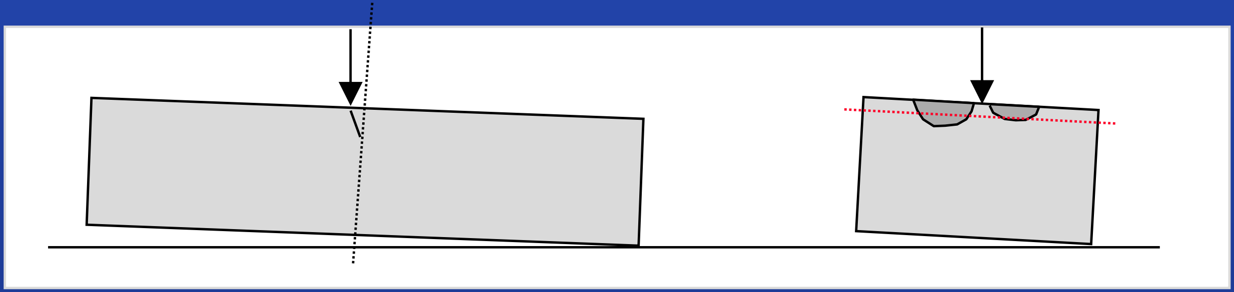
Mizuno and Okada, VAMAS report No.16, 1993

SCF – experimental issues

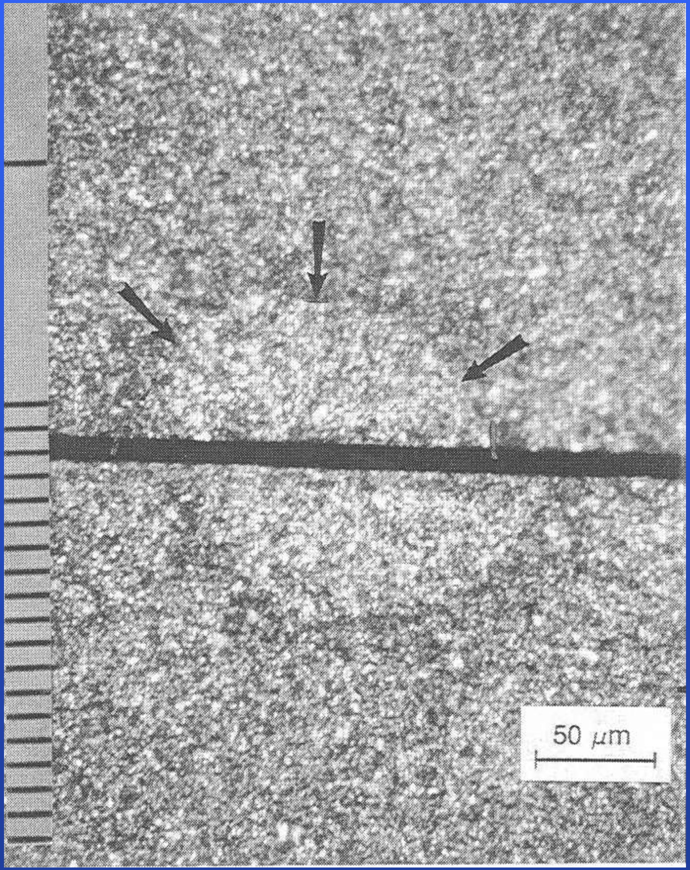
- Assumes indentation will produce half penny shaped cracks
- Assumes successful removal of indent and residual stress zone
- Assumes that after fracture, original crack-line can be detected
  - requires fractographic skills
  - may not work on coarse-grained materials

Experimental tricks:

  - Angle the direction of indentation ~1° away from the normal to make the pre-crack slightly angled compared to fracture plane – makes the pre-crack easier to see – does not seriously affect calibration
  - For Palmqvist cracks (e.g. Y-TZP) tilt the test-piece sideways as well – this exaggerates one lobe of the crack

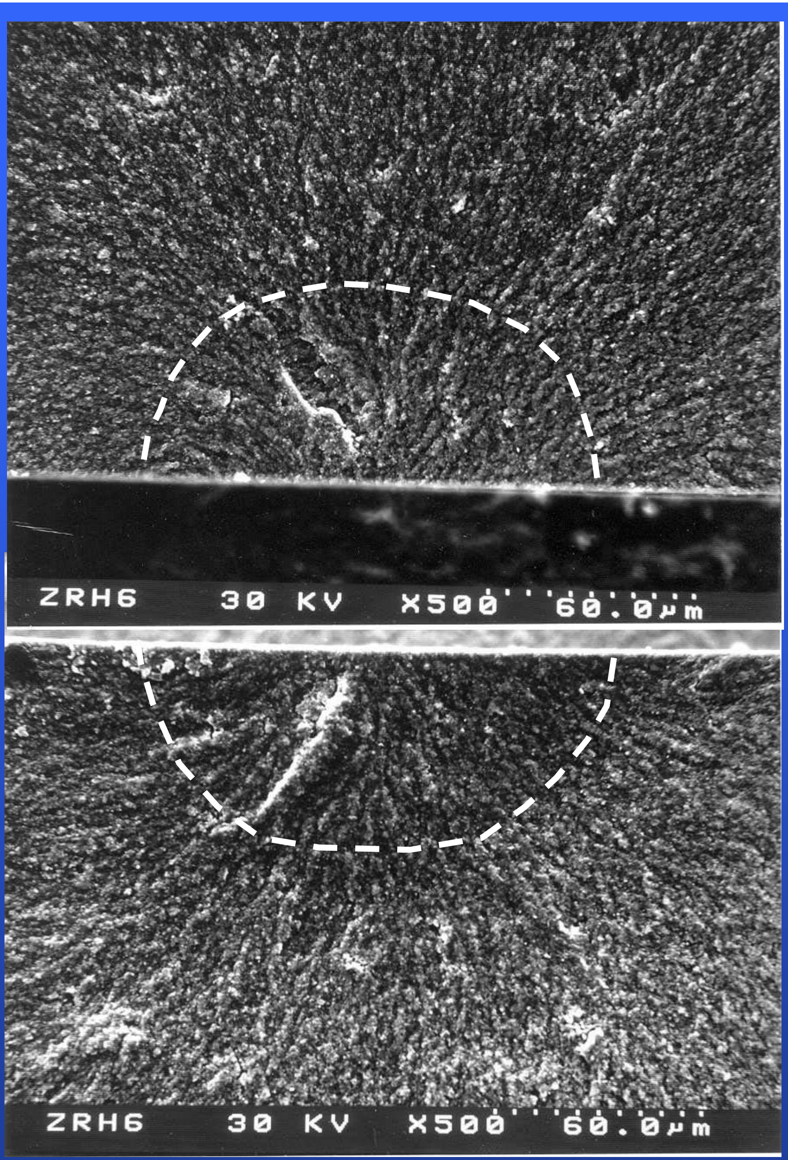


SCF – appearance of pre-cracks



Optical: HPSN

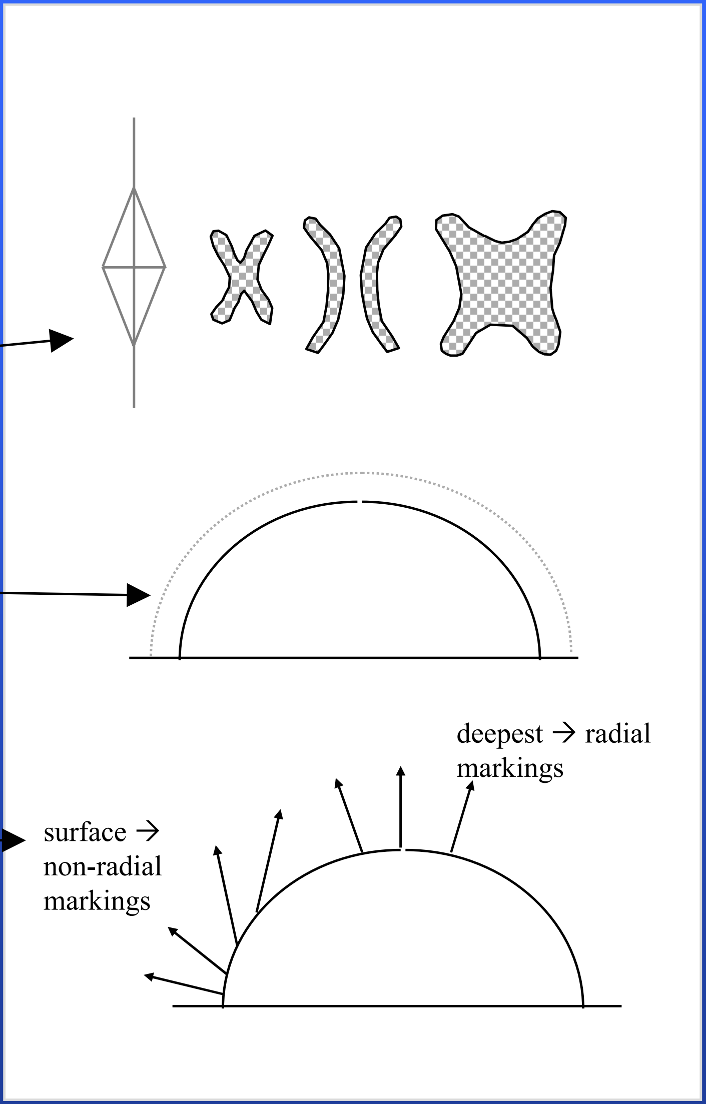
Quinn *et al.*: VAMAS report No. 17, 1993



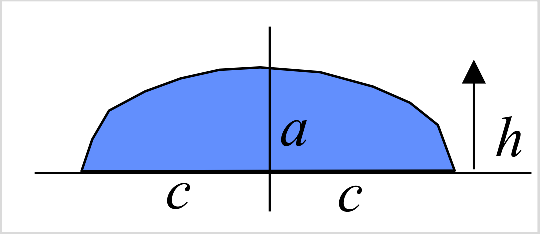
SEM: Y-TZP

SCF – experimental issues

- Look out for remnants of subsurface lateral cracks – remove more material if seen
- Look out for crack growth – must take outer boundary of semielliptical crack
- Crack can initiate from the surface or the deepest part of the crack
  - identify by changes in marking direction at pre-crack boundary
  - compute crack shape parameters for both positions and take lower value if start position is unclear



SCF - equations



$$K_{Ic} = Y\sigma\sqrt{a}$$

where σ is the fracture stress

Fracture from the deepest part: Fracture from the surface:

$$Y_d = (\sqrt{\pi} M H_2)/\sqrt{Q}; \quad Y_s = (\sqrt{\pi} M S H_1)/\sqrt{Q}$$

Where:

$$Q = 1 + 1.464(a/c)^{1.65}; \quad S = (1.1 + 0.35(a/h)^2)\sqrt{(a/c)}$$

$$M = (1.13 - 0.09(a/c)) + [-0.54 + 0.89\{0.2 + (a/c)\}^{-1}](a/h)^2$$
$$+ [0.5 - \{0.65 + (a/c)\}^{-1} + 14\{1 + (a/c)\}^{24}](a/h)^4$$

$$H_1 = 1 - \{0.34 + 0.11(a/c)\}(a/h)$$

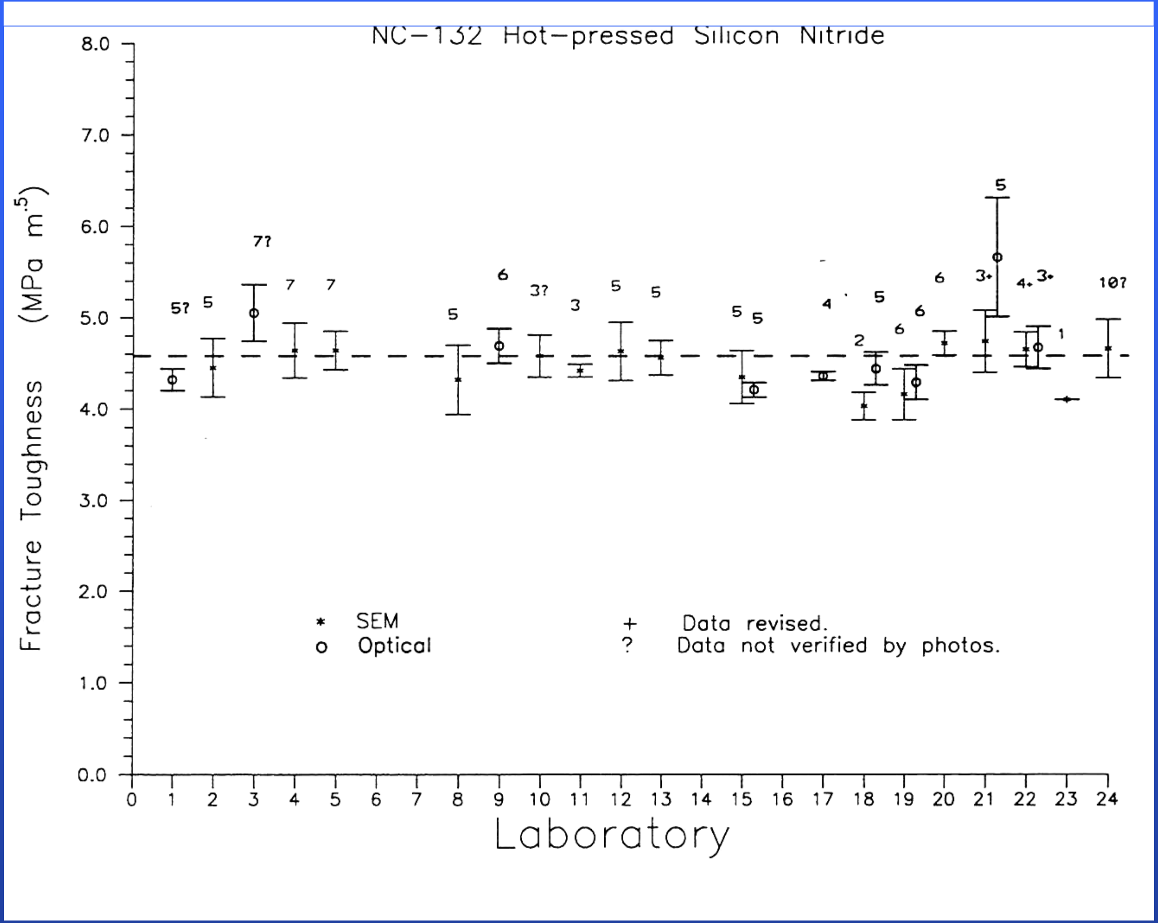
$$H_2 = 1 - \{1.22 + 0.12(a/c)\}(a/h) + \{0.55 - 1.05(a/c)^{0.75} + 0.47(a/c)^{1.5}\}(a/h)^2$$

Based on Newman-Raju analysis

Eng. Fract. Mech. 1981, **15**[1-2], 185-192

SCF – method validation

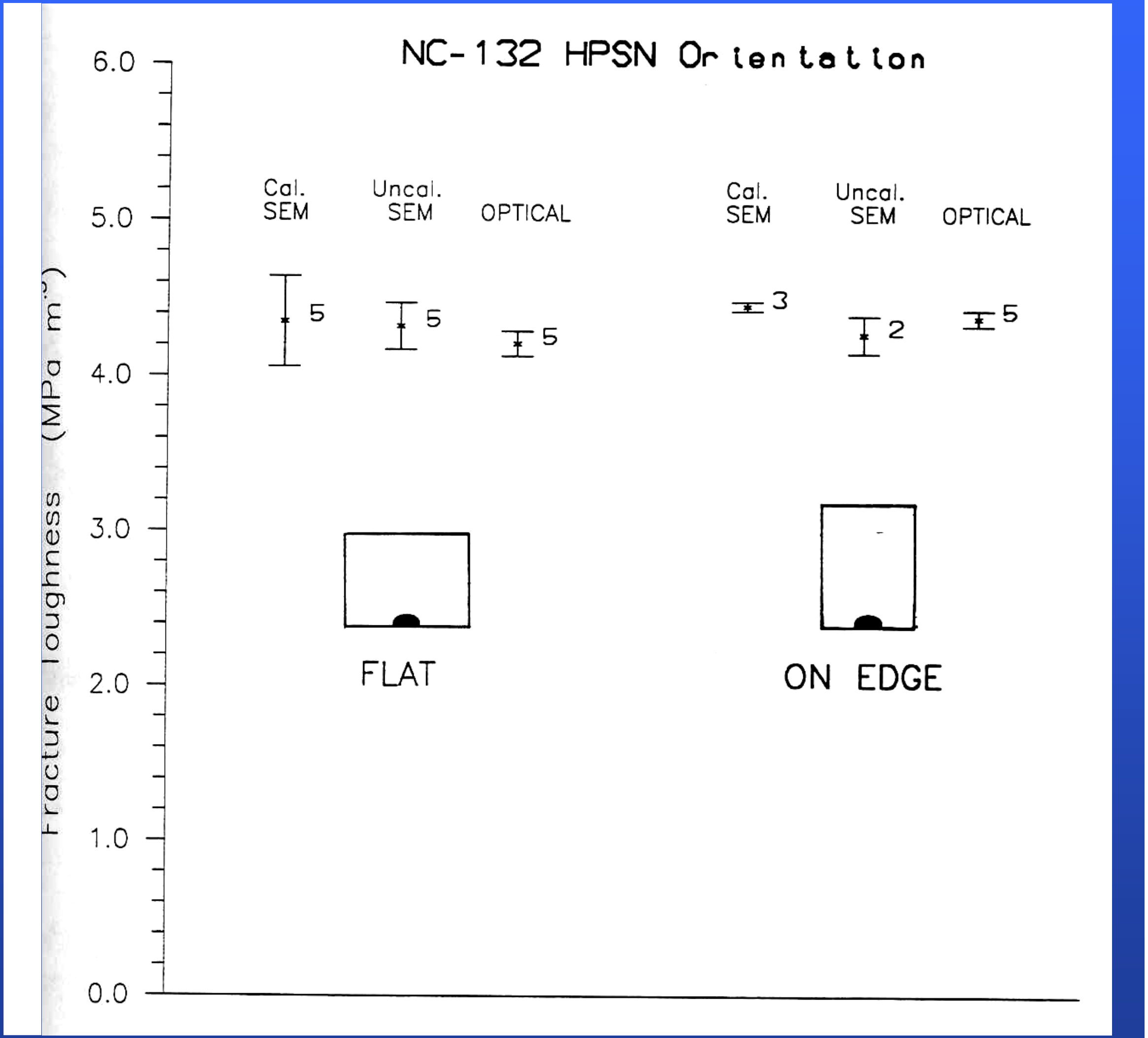
- RR results show most participants obtained consistent results within a narrow band.



Quinn *et al.*: VAMAS report No. 17, 1993

SCF – test validation

- Consistency of measuring flaw size is good
- Optical or SEM can be used
- Accuracy of flaw size measurement not critical



Quinn, VAMAS Report No. 17, 1993

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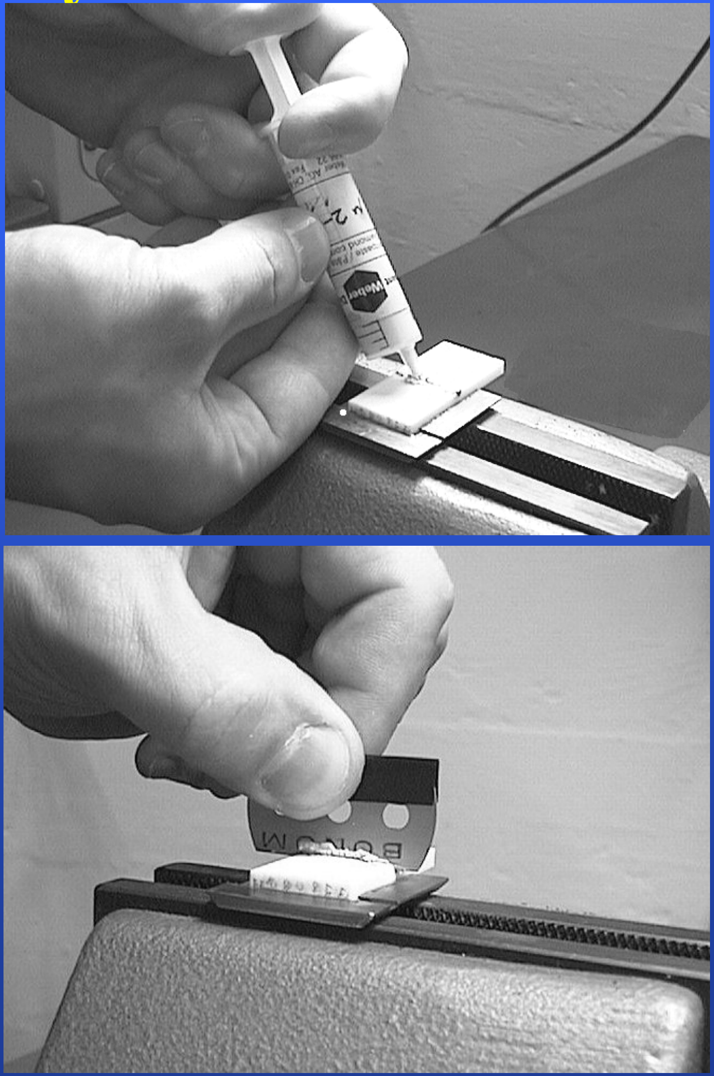
SEVNB – how small does the notch tip radius have to be to represent a crack?

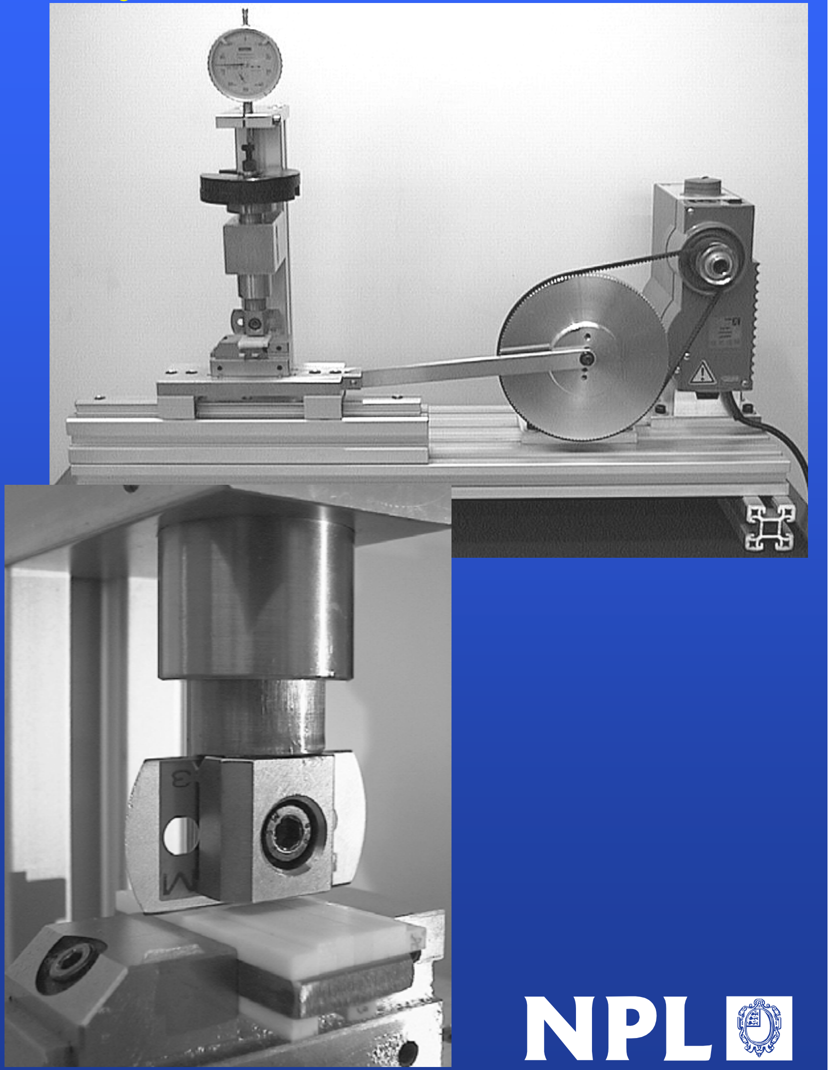
- Generally thought to be of the order the grain size or smaller
  - Not thought to be appropriate for Y-TZP
- Assumed that damage at the notch tip pops in to form a crack during loading
- Can sometimes see this pop-in distance – add this to measured notch depth
- Significant subcritical crack growth can also occur – also need to add this to notch depth

SEVNB – notch honing

By hand:

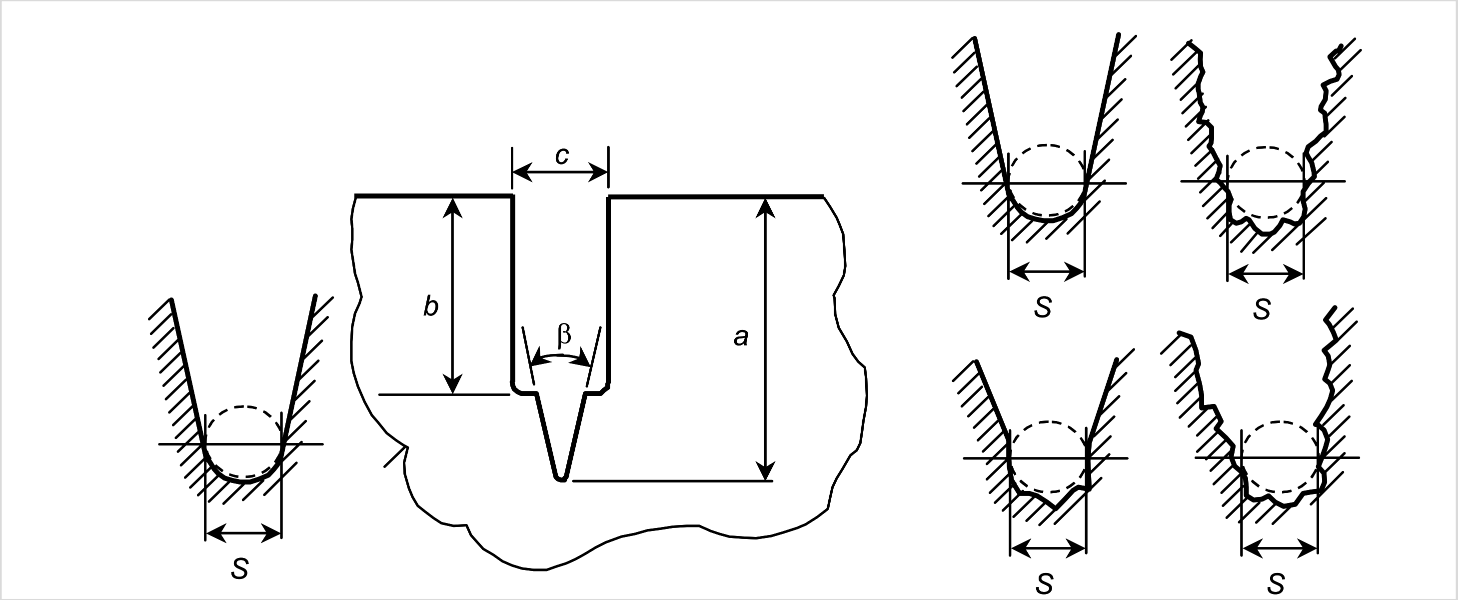


By machine:



SEVNB – notch tip geometry

- In fine grained materials can get a tip radius of ~2 µm with notching machine
- In coarse-grained materials, tip radius determined by grain size
- Tip radius can be examined at test-piece sides



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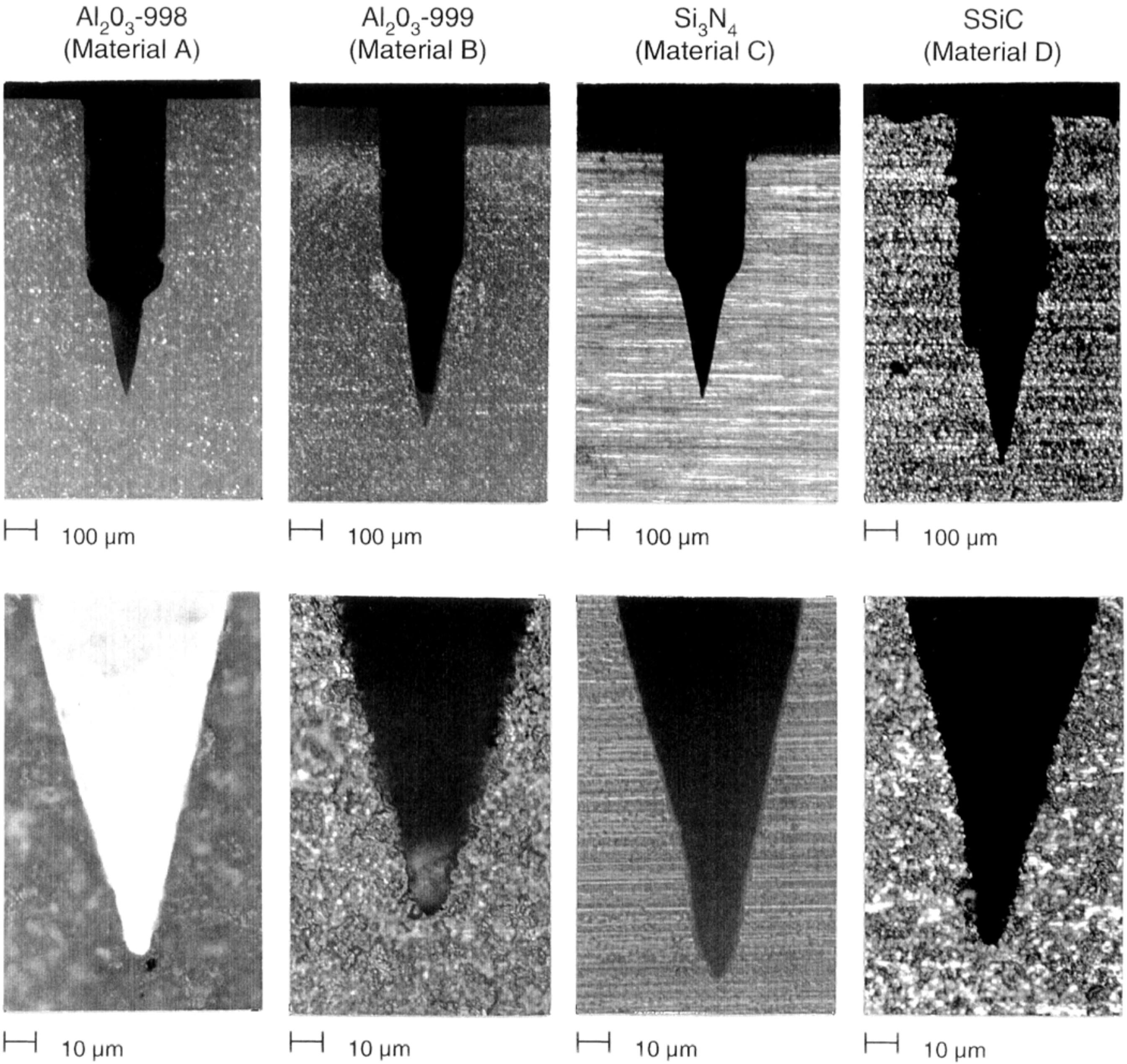
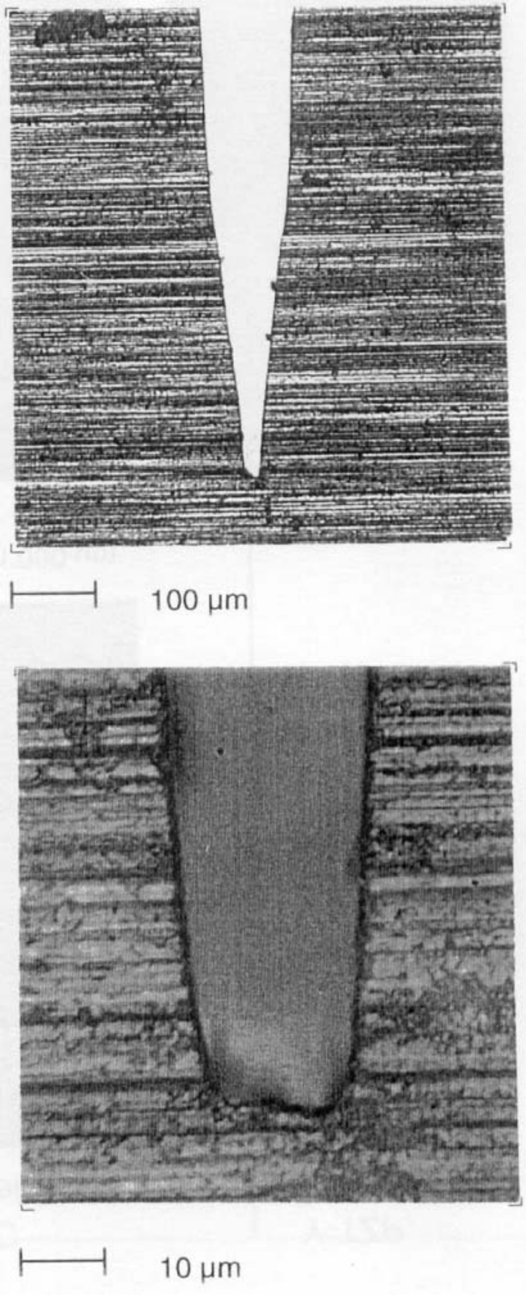
SEVNB notches



Produced in the VAMAS/ ESIS RR

Participant 16
Kübler, VAMAS/ESIS RR, 1999

SEVNB - Notch honing without a sawn pre-notch



- By using a machine with good blade position control, direct sawing of notches can be made, even in tough materials such as silicon nitride

Participant 2, silicon nitride
Kübler, VAMAS/ESIS RR, 1999

SEVNB - Dos and Don’ts of notch honing

By hand:

- starter notch should be just wider than razor blade
- use 6 µm diamond paste and a backed blade for safety
- move blade smoothly in reciprocating motion, keeping it upright, and don’t load too hard
- don’t rock the blade
- finish with a new blade and finer paste

By machine:

- make sure blade is aligned with direction of motion and with pre-sawn notch, if used
- lift blade occasionally and re-charge with abrasive/lubricant
- finish with new blade and finer paste

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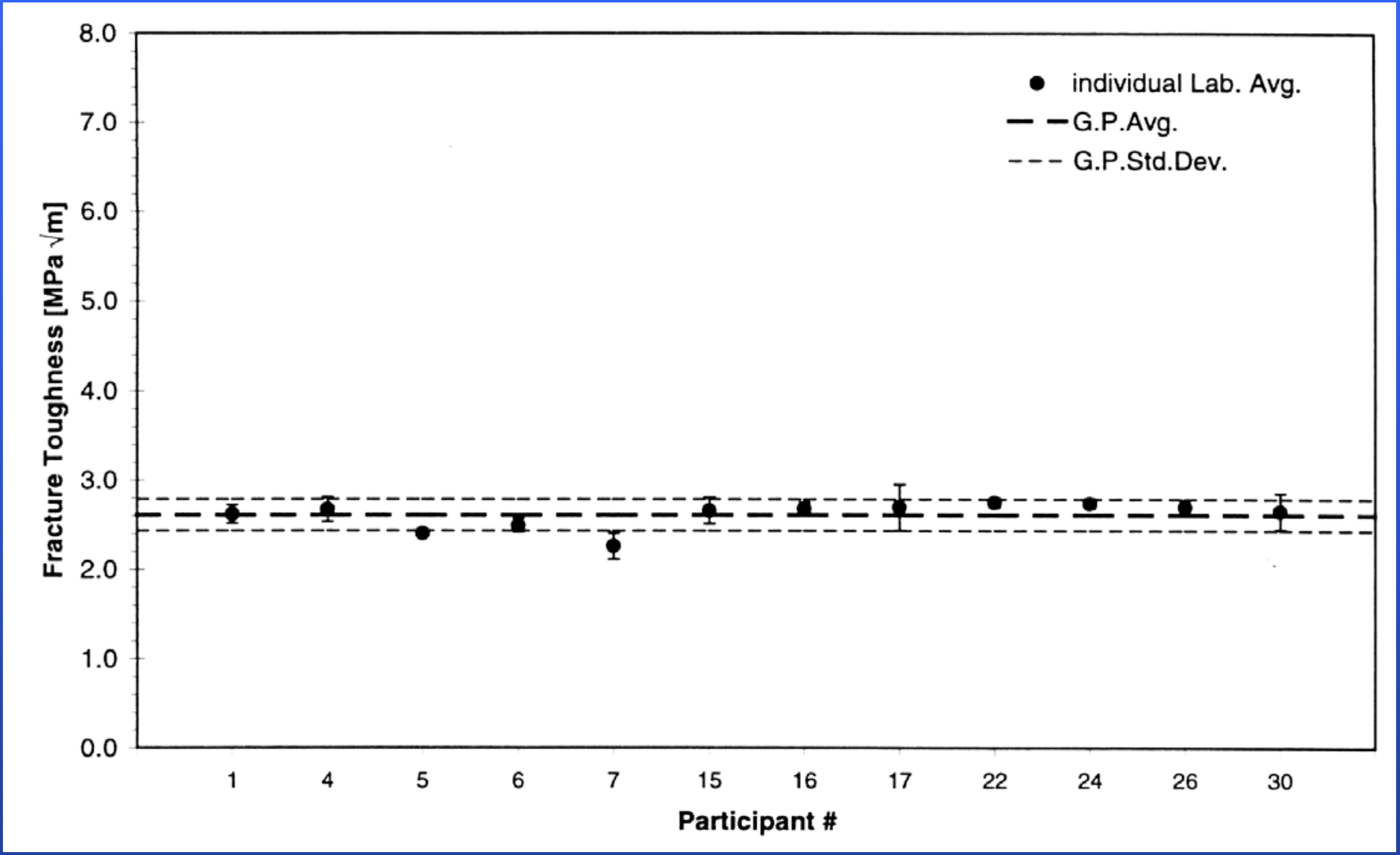
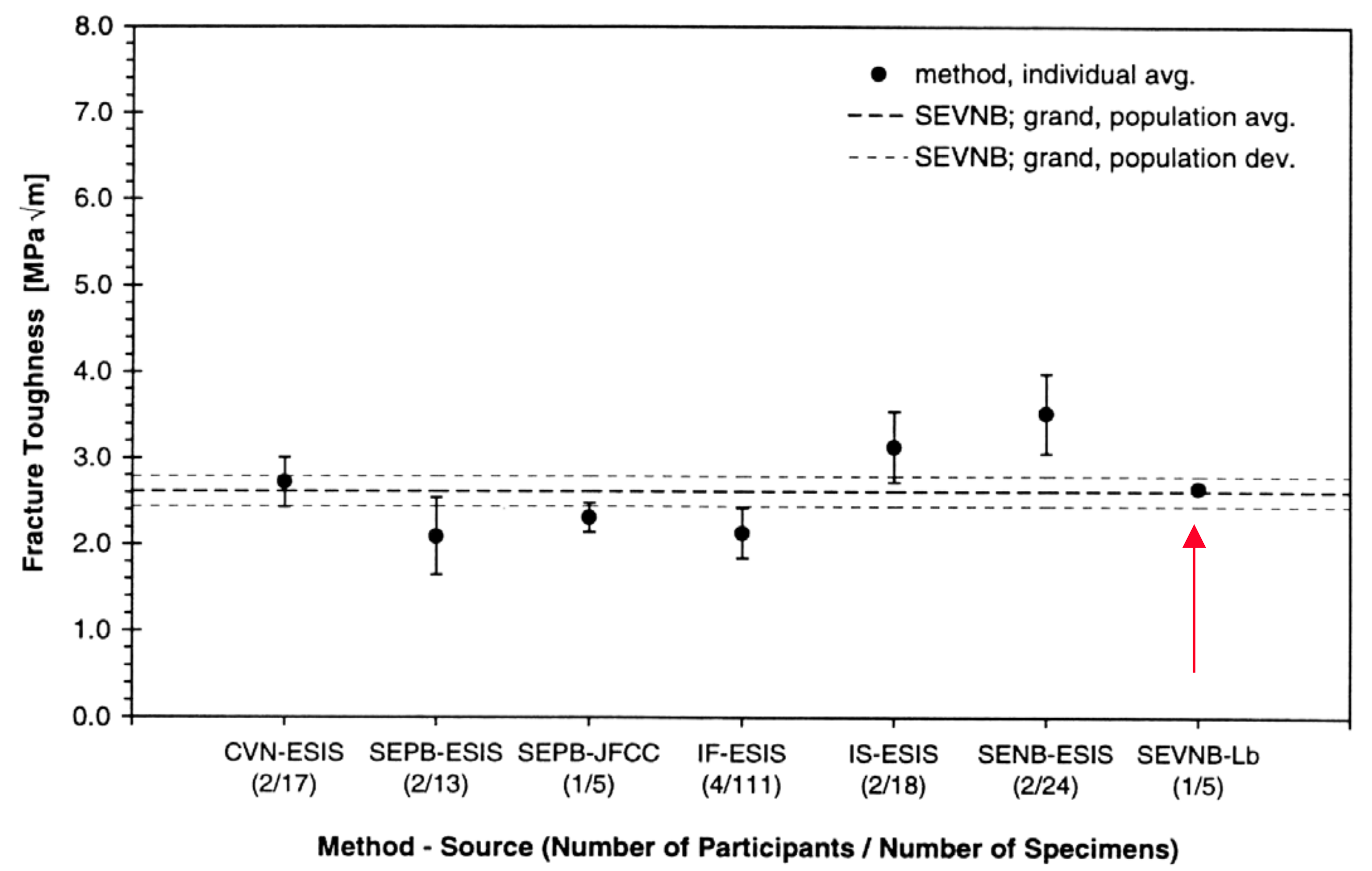
SEVNB test validation – VAMAS/ESIS round robin



- Sintered silicon carbide – interlaboratory consistency

Kübler, VAMAS/ESIS RR, 1999

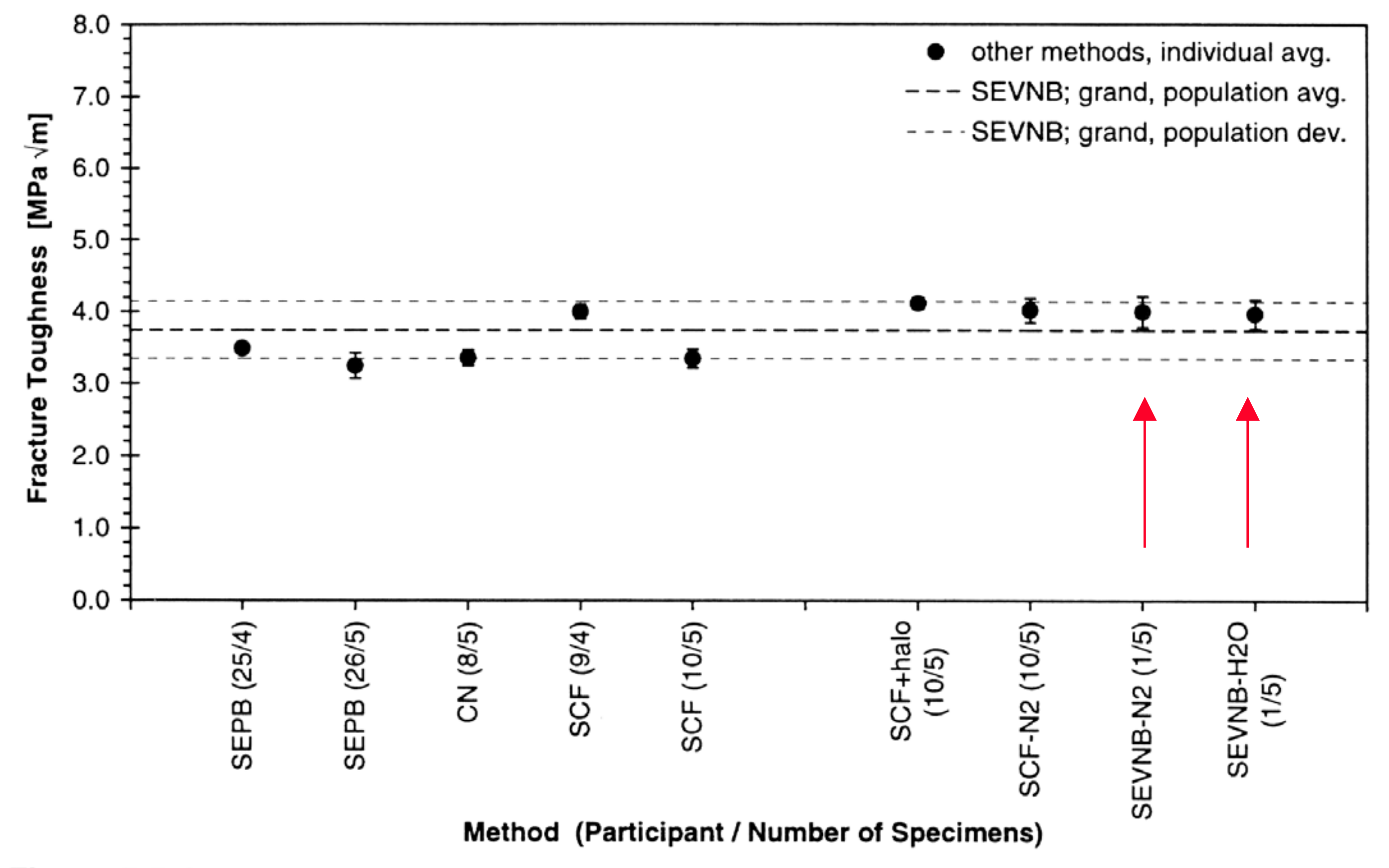
SEVNB test validation – VAMAS/ESIS round robin



- Sintered silicon carbide – inter-method consistency

Kübler, VAMAS/ESIS RR, 1999

SEVNB test validation – VAMAS/ESIS round robin



- AD999 alumina – inter-method consistency

Kübler, VAMAS/ESIS RR, 1999

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SEVNB – test validation

- VAMAS/ESIS round robin organised by EMPA (CH, Kübler)
- Aluminas, silicon nitride, silicon carbide, zirconia
- Narrow band of results for most materials
- Y-TZP gave results higher than sharp crack methods (but grain size smaller than notch tip + phase transformation)

		Total number of		Repeatability (within-lab)		Reproducibility (between-lab)	
Material	Method	Participants	Test pieces	Std.dev. MPa $m^{1/2}$	CV %	Std.dev. MPa $m^{1/2}$	CV %
Alumina-998	SEVNB	28	135	0,17	4,6	0,22	6,1
Alumina-999	SEVNB	21	102	0,23	6,2	0,40	10,7
GPSSN	SEVNB	27	129	0,28	5,3	0,34	6,3
SSiC	SEVNB	12	56	0,12	4,5	0,18	6,8
Hot pressed Si3N4	SCF	19	102	0,24	5,4	0,31	6,8
Hot iso-pressed Si3N4	SCF	15	100	0,38	7,7	0,45	8,9

Fracture toughness standards available

Method	CEN*	ASTM	JIS	ISO
SEPB	(EN 14425-2) = ISO 15732	ASTM C1322	JIS R1607	ISO FDIS 15732
CNB	TS14425-3 ≠ ISO 24370	ASTM C1322	-	ISO FDIS 24370
SCF	(EN 14425-4) = ISO 18756	ASTM C1322	-	ISO FDIS 18756
SEVNB	TS 14425-5	-	-	To be proposed

* CEN TS 14425-1 is a guide to methods

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Which method to choose?

- All methods have pros and cons (see prTS14225-1 – the ‘Guide’)

Recommendation

- SEPB for most materials, also R-curve and crack growth studies
- CNB for most materials although it may be difficult to get valid crack initiation in tough ones
- SCF for all except coarse-grained materials
- SEVNB for all except very fine grained materials

Do not recommend:

- SENB: overestimates toughness in tougher materials
- IF: subjective measurement and poor calibration
- IS: poor calibration – indent load dependent
- DT: mixed mode failure

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Acknowledgements

Those who have organised technique development and VAMAS and ESIS round robins to provide us with reliability data for the different methods, especially:

- H Okada and M Mizuno (JFCC) - SEPB
- J Kübler (EMPA) - SEVNB
- G D Quinn, R Gettings (NIST), J Kübler (EMPA), G. Swab (USARL) – SCF
- R Primas EMPA, R Gstrein, CDLH – method comparison

and all the fracture mechanicists who compute the calibrations….