Fracture & Fatigue of Advanced Metallic Alloys

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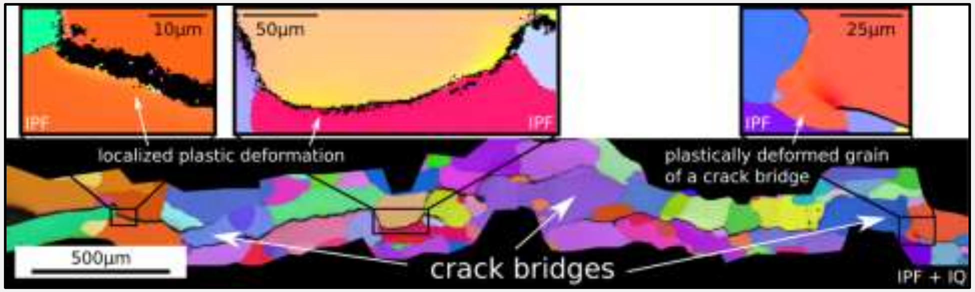
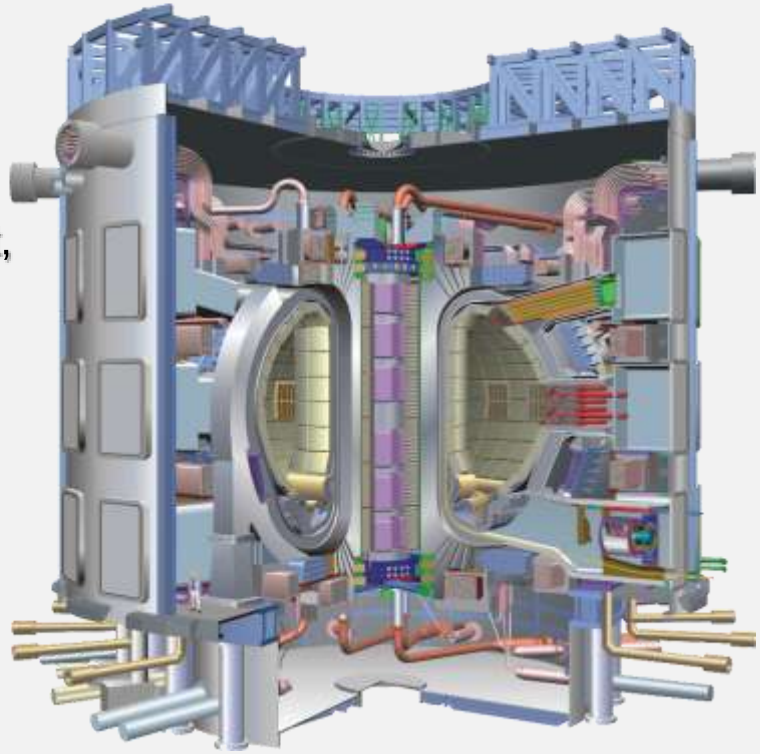
Work supported by the Office of Science (Basic Energy Sciences) of the Department of Energy (Berkeley, Oakridge) and by the National Science Foundation (Caltech).

Mechanical Properties of Structural Materials

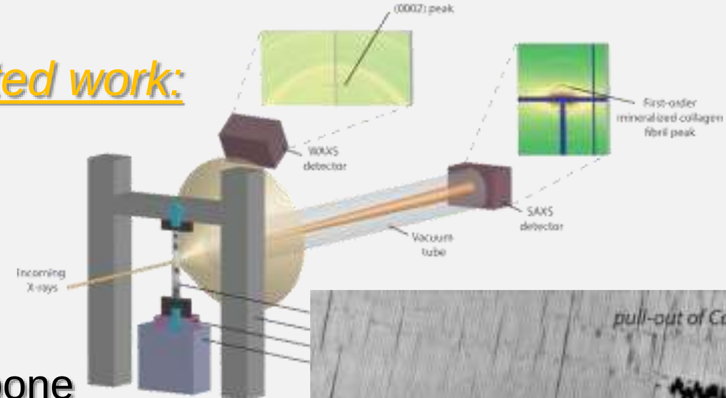
<u>*Past research (Master & PhD theses):*</u>

- Fracture behavior of W-materials
  - understanding mechanisms determining brittle fracture at low temperatures & ductile-to-brittle transition
  - influence of grain size, dislocation density/arrangement, texture & impurities on fracture mechanism
  - fracture behavior in the range RT to 1000C



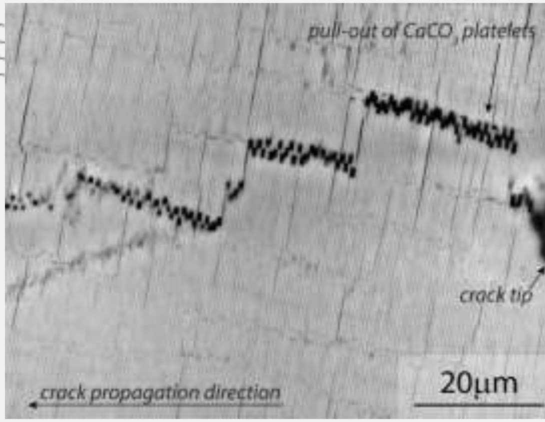


Past and Current Research
Non-metallic materials



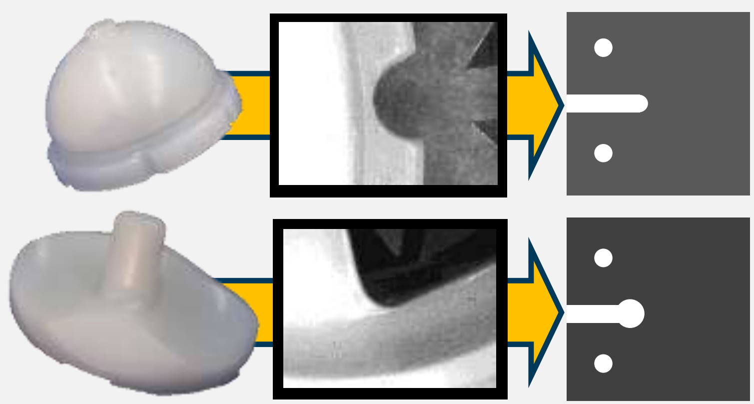
Current research (Post-doctoral work) – bio-related work:

- Mechanical properties of biological & nature-inspired materials
  - using *in-situ* VP-SEM & SAXS/WAXD techniques
  - understanding effects like aging, strain rate, diseases (OI) and drugs on structural integrity of bone
  - investigating natural materials (fish scales, skin, nacre, …) & mimicking natural structures using ceramics/metals/polymers



- Strength of nano-particle reinforced fibers & thin films
  - understanding mechano-optical sensing capabilities
  - verifying usability of block copolymers for lithium metal batteries

- Fatigue behavior of UHMWPE (ultra-high-molecular-weight polyethylene)
  - used in hip and knee replacements
  - evaluation of fatigue crack propagation from various notch geometries



Past and Current Research – Metallic Alloys

Mechanical Properties of Structural Materials

Past research (Master & PhD theses):

- Fracture behavior of W-materials
  - understanding mechanisms determining brittle fracture at low temperatures & ductile-to-brittle transition
  - influence of grain size, dislocation density/arrangement, texture & impurities on fracture mechanism
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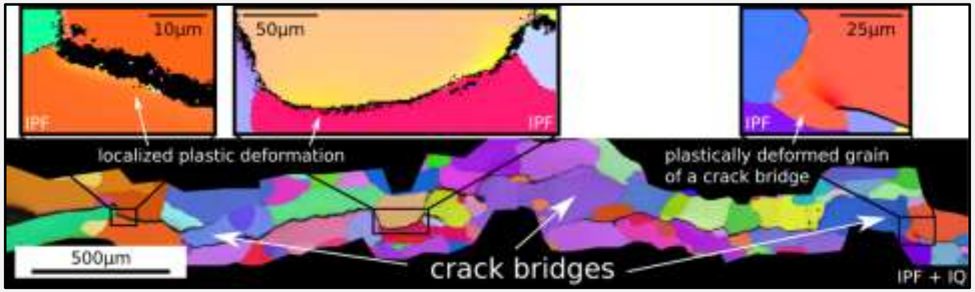

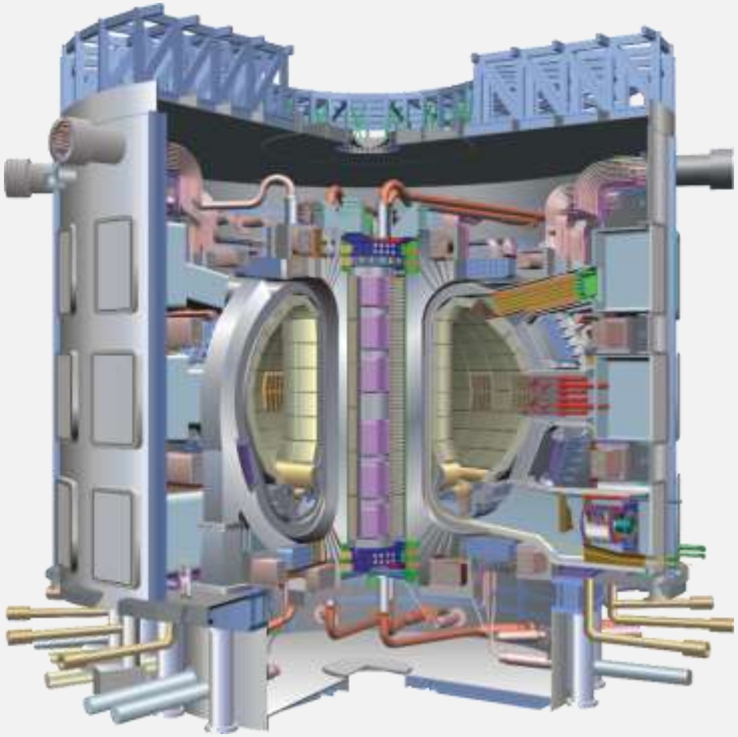

Current research (Post-doctoral work) – engineering materials:

- Strength and toughness of nuclear graphite
  - investigating deformation mechanisms and damage tolerance in the temperature range room temperature to 1000C using synchrotron x-ray tomography
- Fracture & fatigue of bulk-metallic glasses (BMGs)
- Fracture behavior of high-entropy alloys (HEAs)

BERKELEY LAB

EPFL – Seminar, 1st February 2016

Fracture & Fatigue of Multi-Component Alloys

Fracture and fatigue of bulk-metallic glasses (BMGs)

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Mechanical Properties of Metallic Glasses

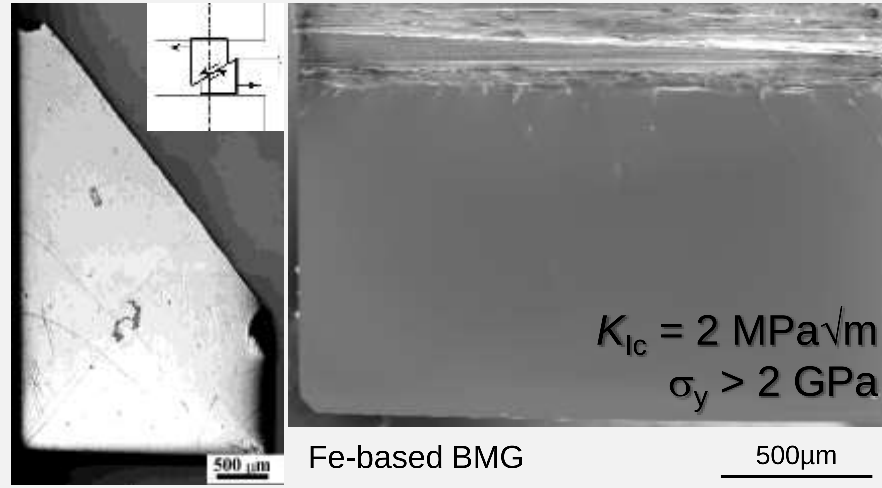
→ high strength (0.5 – 4 (5) GPa)

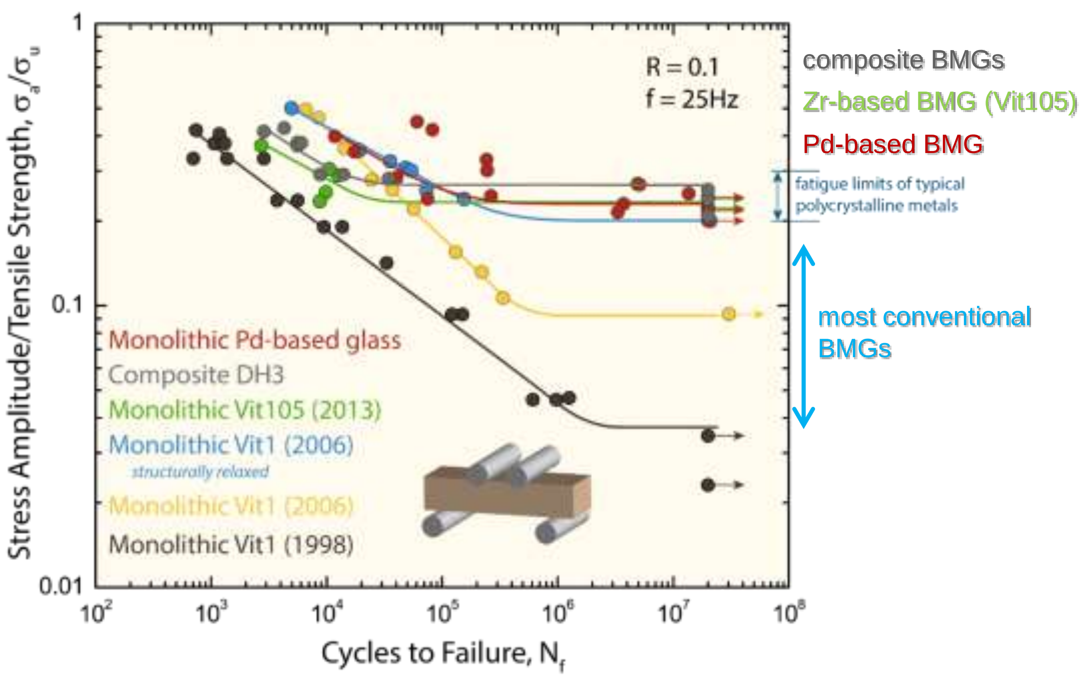
→ low stiffness (<100 GPa)

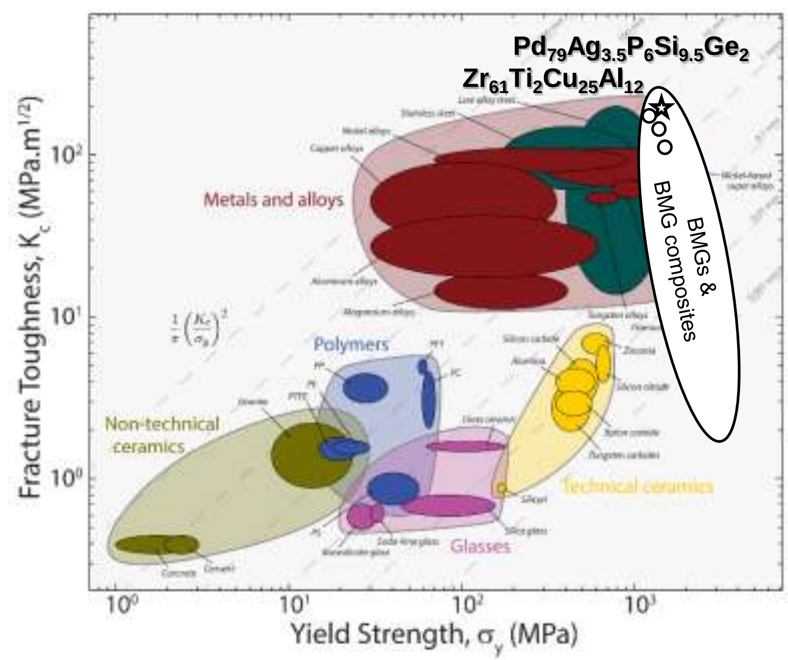
→ limited ductility
(tension: ~ 0% / compression: < 2%)

→ varying fatigue strength
(0.05 – 0.2 (0.3) σ_{UTS})

→ 'OK' fracture properties
(fracture toughness 10 – 100 (200) MPa√m)



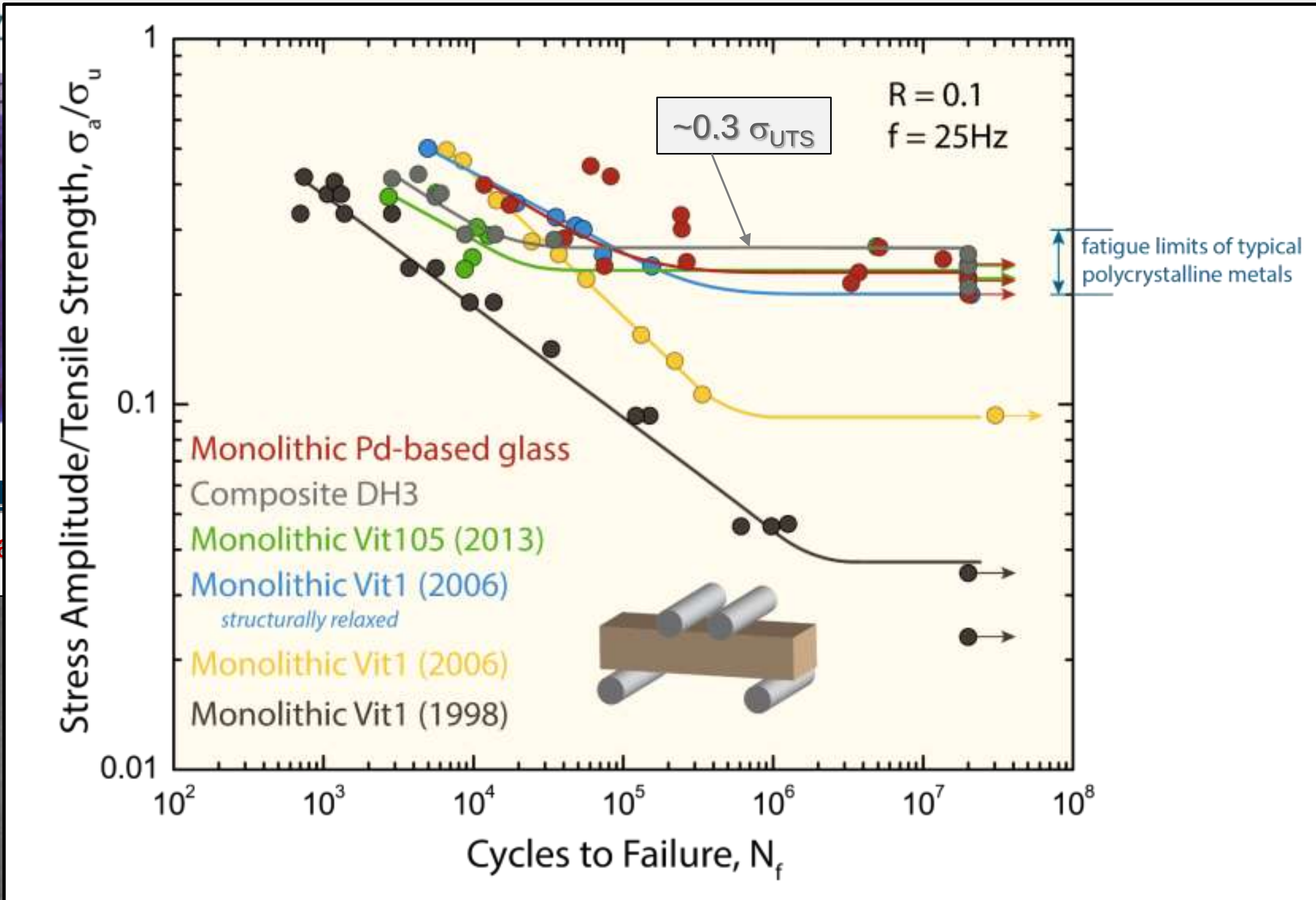




Gludovatz, Demetriou, Floyd, Hohenwarter, Johnson, Ritchie, *PNAS 2013* | Sergueeva, Mara, Kuntz, Lavernia, Mukherjee, *Phil. Mag.*, 2005 | Naleway, Greene, Gludovatz, Dave, Ritchie, Kruzic, *Metal. Mater. Trans. A*, 2013

Bulk-metallic glass matrix composites

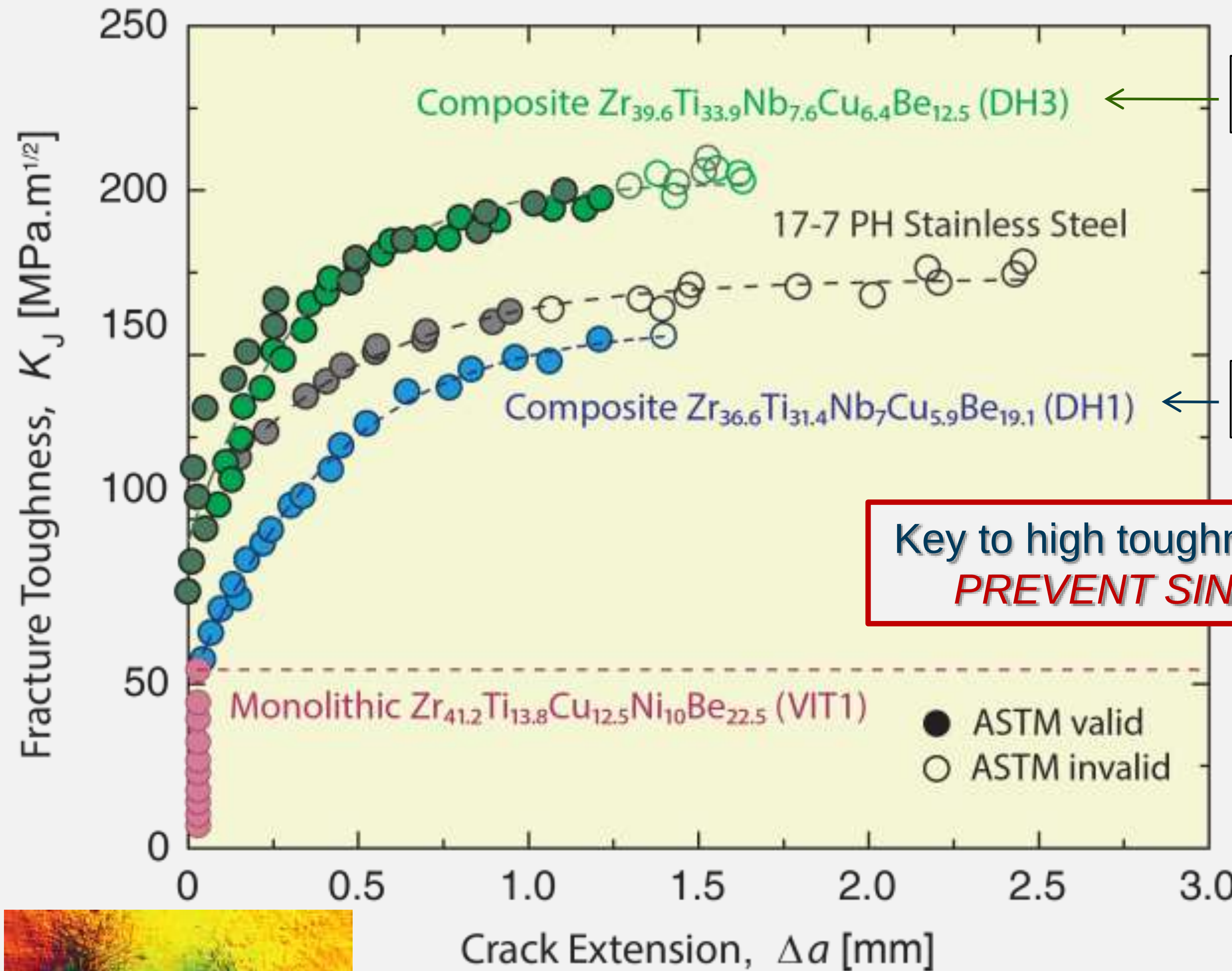
BMG in situ Matrix Composites



Launey, Hofmann, Johnson, Ritchie *PNAS* 2009

Hofmann, Sun, Weist, Duan, Lind, Demetriou, Johnson *Nature* 2008

Toughness of BMG in situ Matrix Composites



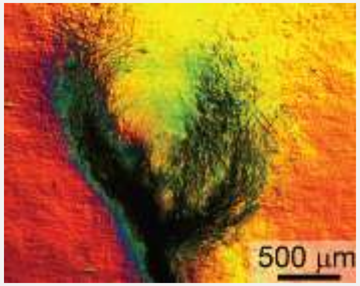
σ_y ~ 1.1 GPa

$$K_{JIc} = (J_{Ic}\, E')^{1/2}$$

σ_y ~ 1.5 GPa

Key to high toughness & excellent fatigue behavior:
PREVENT SINGLE-SHEAR BAND FAILURE

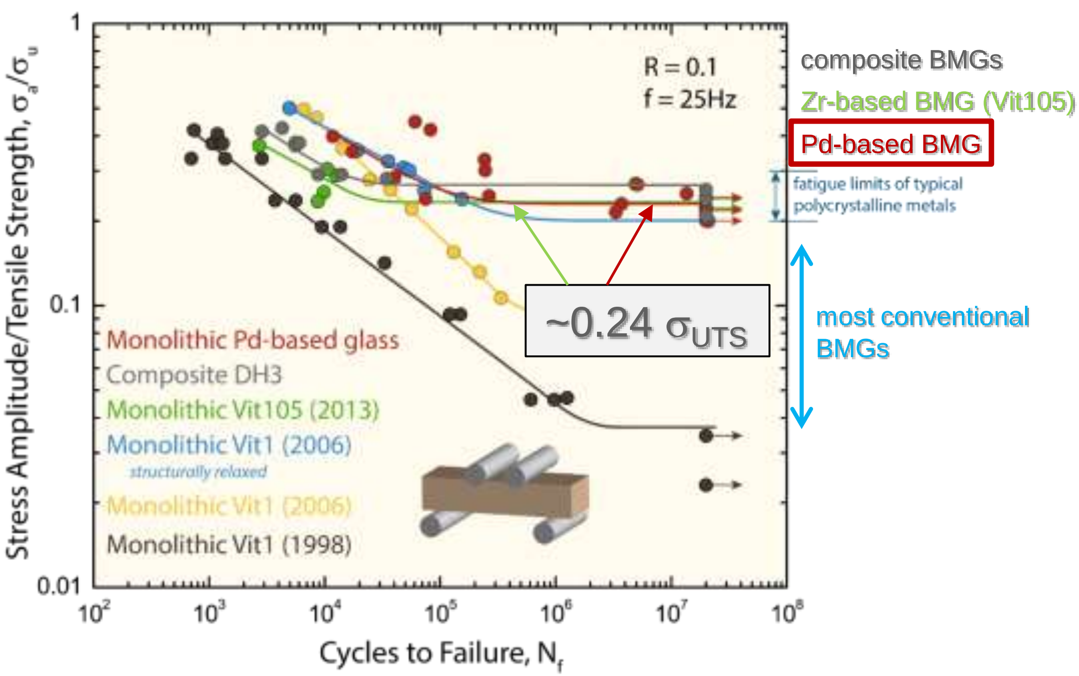
DH3: K_{JC} = 157 MPa.m$^{0.5}$
DH1: K_{JC} = 97 MPa.m$^{0.5}$

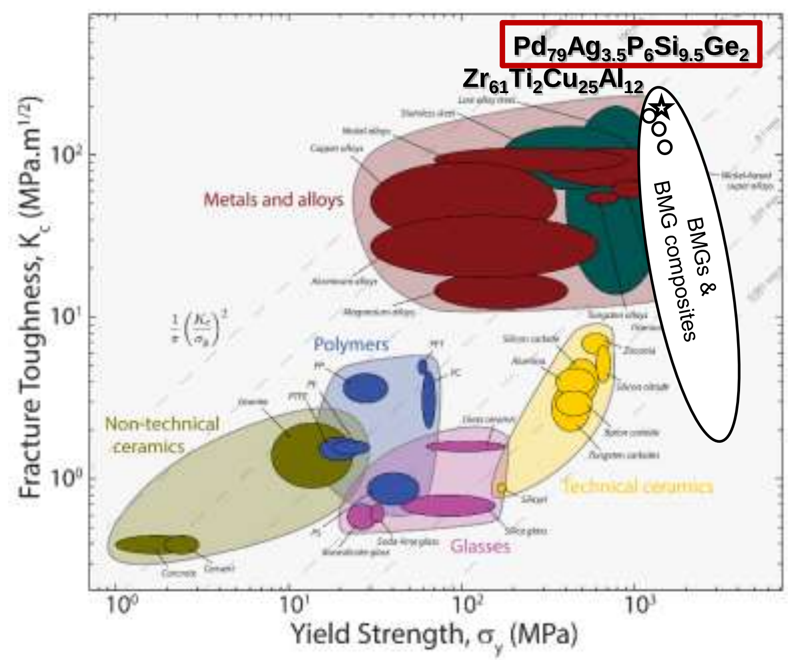


BMG composites display very high damage tolerance

Launey, Hofmann, Suh, Kozachkov, Johnson, Ritchie *Appl. Phys. Lett.* 2009

Fully Amorphous Monolithic Glasses

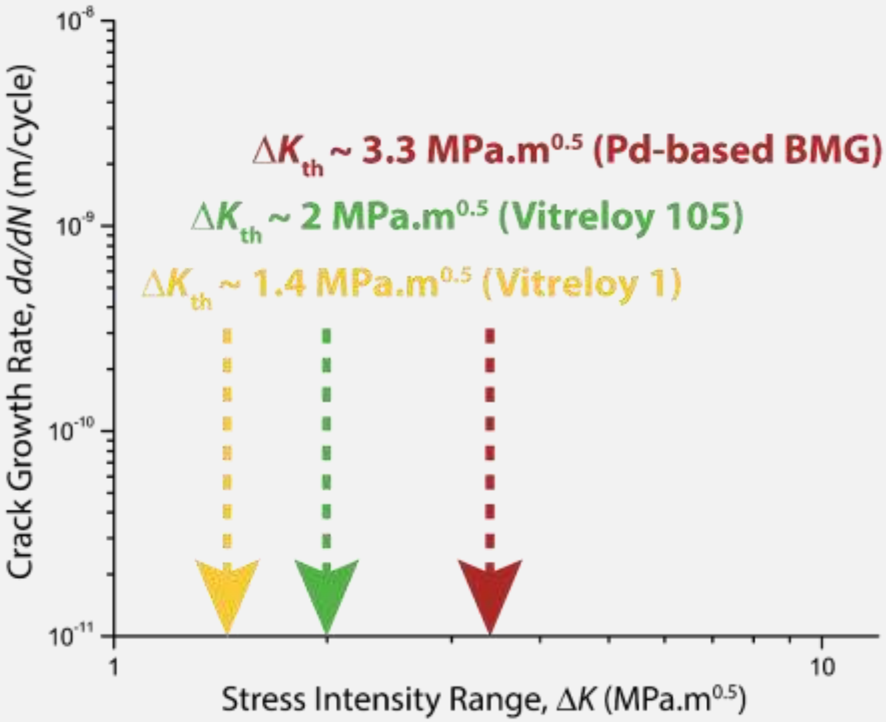


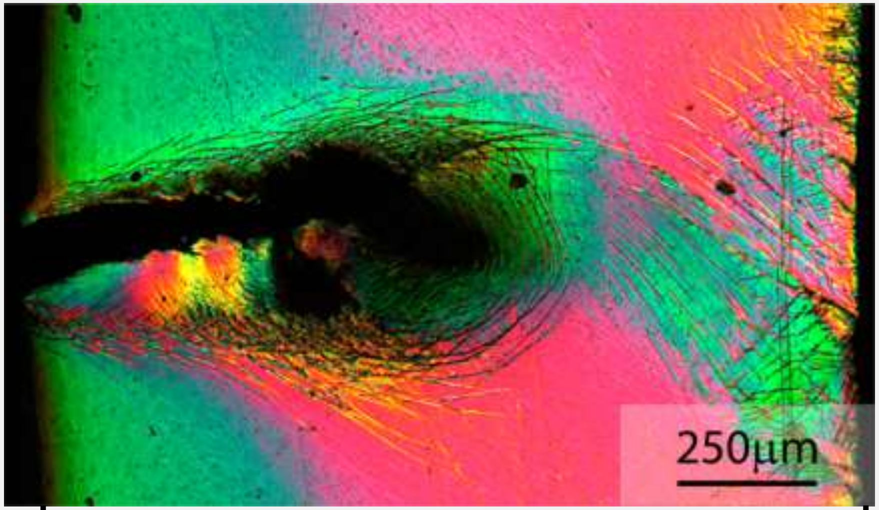


Gludovatz, Demetriou, Floyd, Hohenwarter, Johnson, Ritchie, *PNAS 2013* | Sergueeva, Mara, Kuntz, Lavernia, Mukherjee, *Phil. Mag.*, 2005 | Naleway, Greene, Gludovatz, Dave, Ritchie, Kruzic, *Metal. Mater. Trans. A*, 2013

Pd-based BMG: Threshold & flaw sensitivity

high threshold → high endurance limit





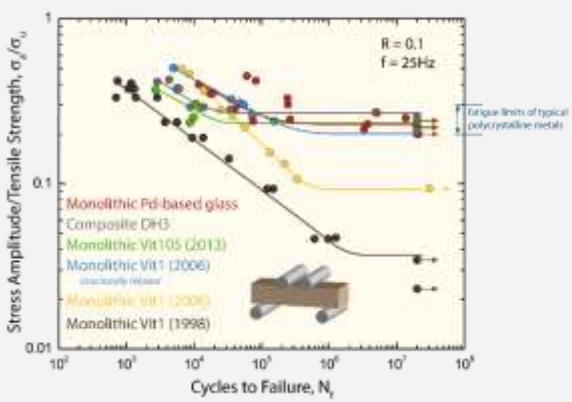
thickness of 4-point bending stress-life (S-N) sample

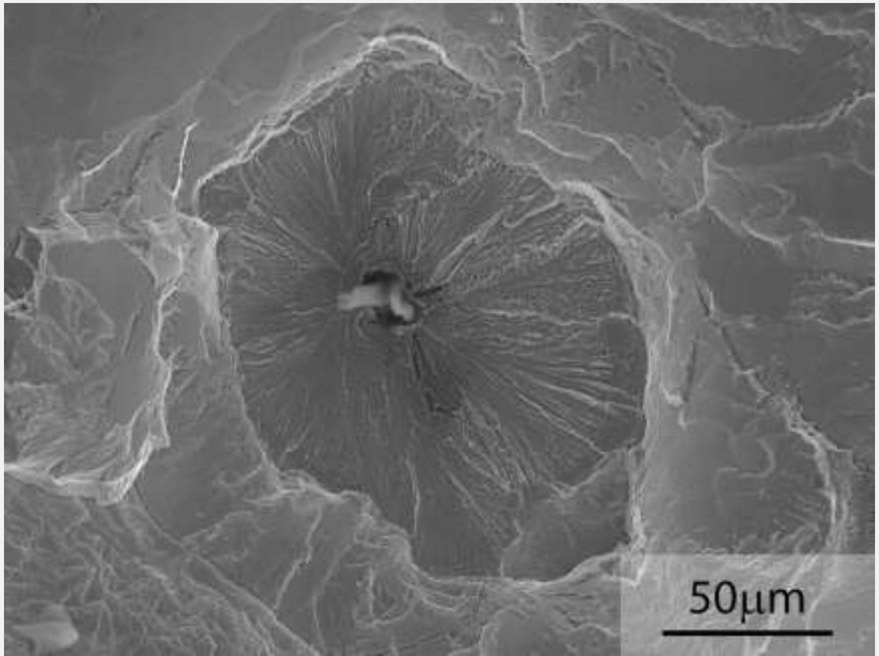
$Pd_{79}Ag_{3.5}P_6Si_{9.5}Ge_2$

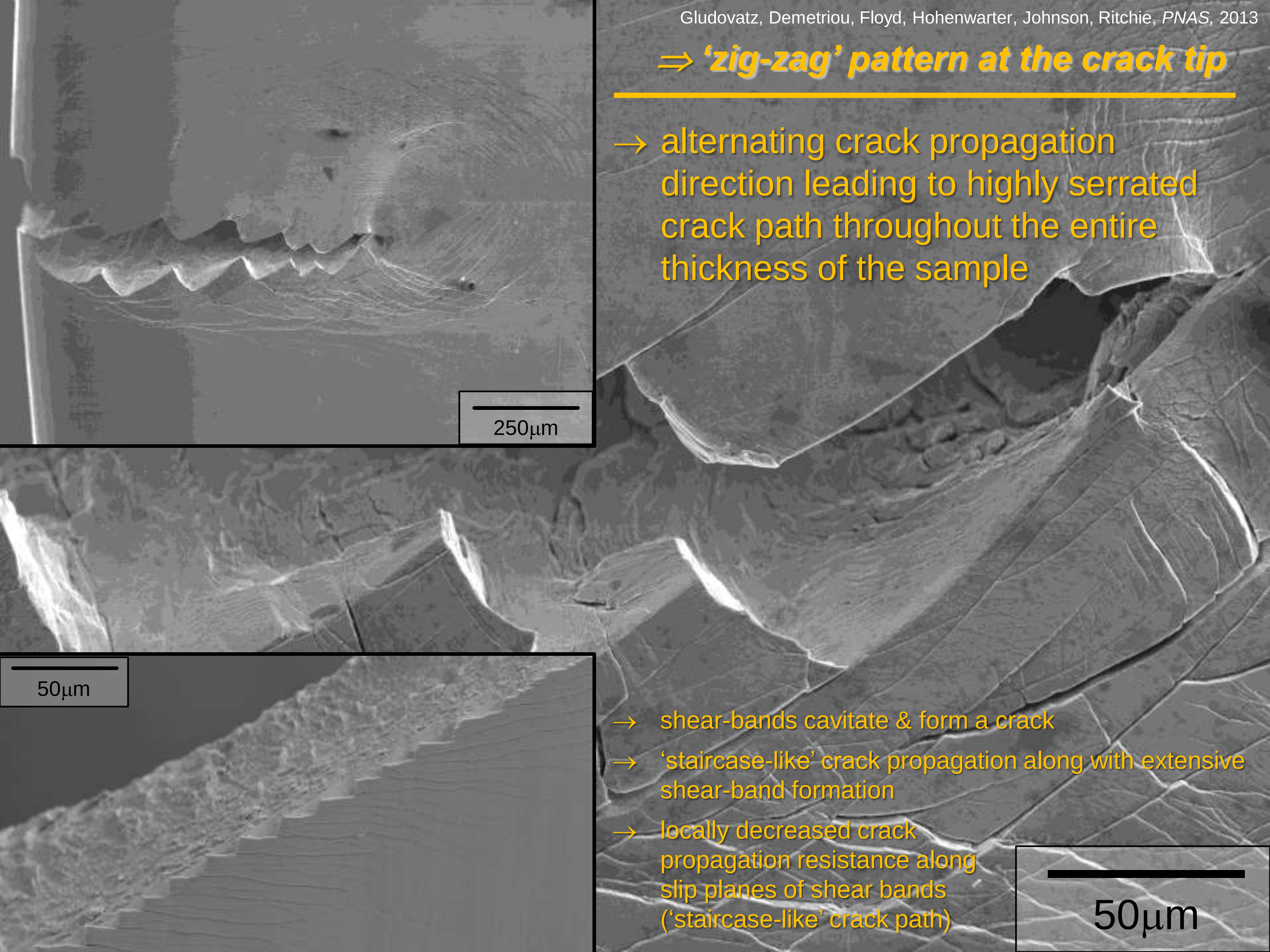
E = 88 GPa

B = 172 GPa

G = 31 GPa

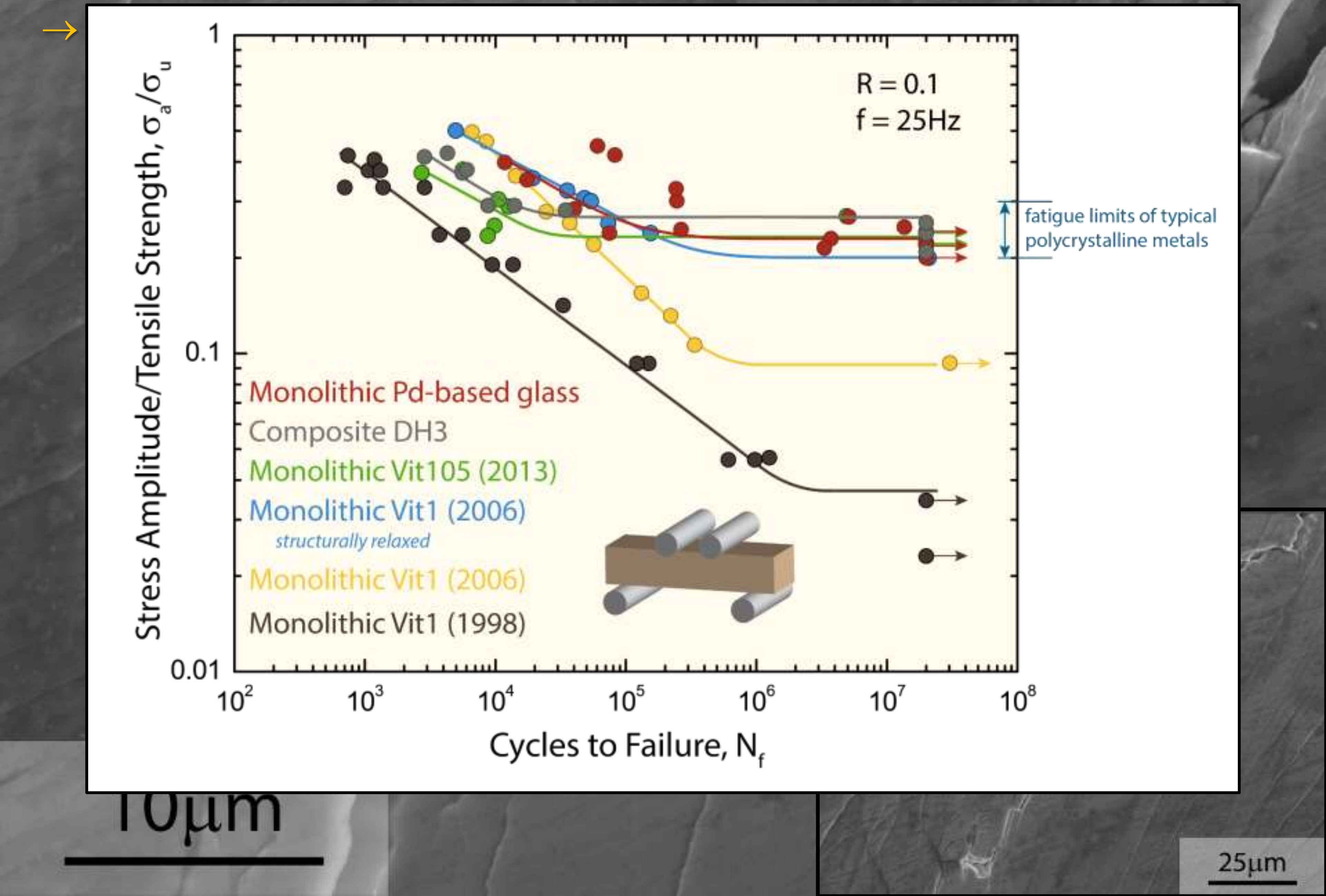




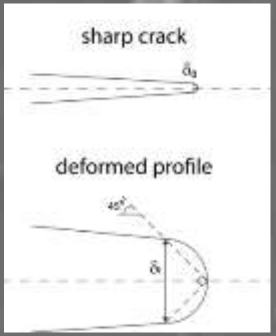

Gludovatz, Demetriou, Floyd, Hohenwarter, Johnson, Ritchie, *PNAS*, 2013
⇒ ‘zig-zag’ pattern at the crack tip
→ alternating crack propagation direction leading to highly serrated crack path throughout the entire thickness of the sample
250μm
50μm
→ shear-bands cavitate & form a crack
→ ‘staircase-like’ crack propagation along with extensive shear-band formation
→ locally decreased crack propagation resistance along slip planes of shear bands (‘staircase-like’ crack path)
50μm

Roughness-induced crack closure

→

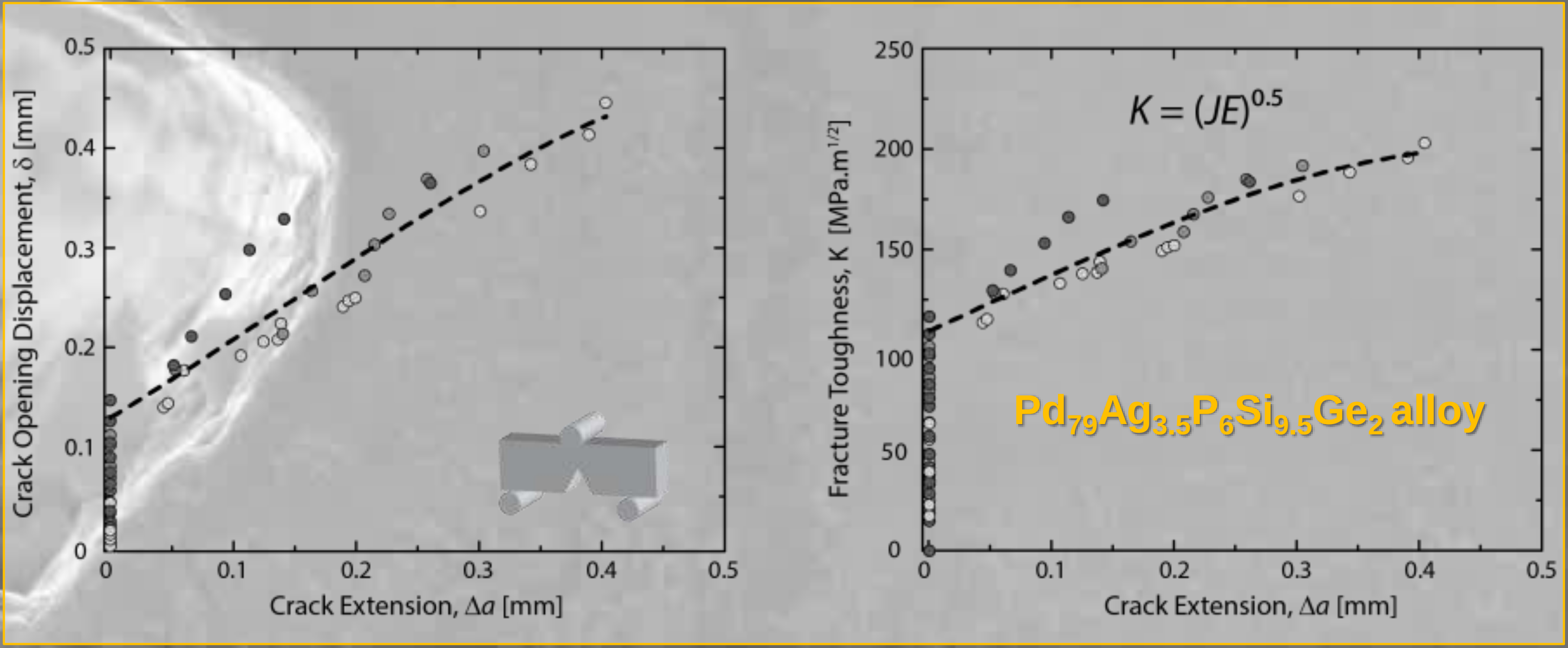


- multiple stable shear-band formation and proliferation
  → excessive crack tip blunting and plastic-zone size formation, prior to initiation and propagation of shear-band cracks



- akin to general yielding and fully ductile fracture in crystalline alloys

- large crack-tip open displacements (CTODs) lead to very high toughness



Pd-based monolithic BMGs

WD16.0mm 15.0kV x3.0k 10um

Demetriou, Launey, Garrett, Schramm, Hofmann, Johnson, Ritchie, Nat Mater, 2011

Current issues with metallic glasses

Major problem: VARIABILITY of mechanical properties

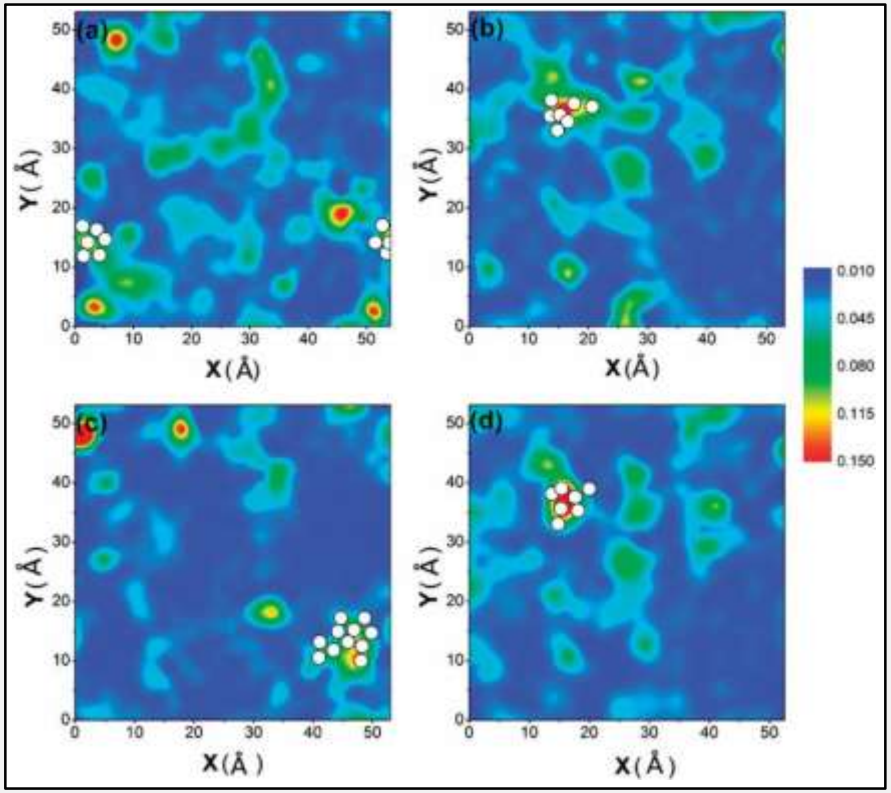
i) Processing
ii) Mechanical testing

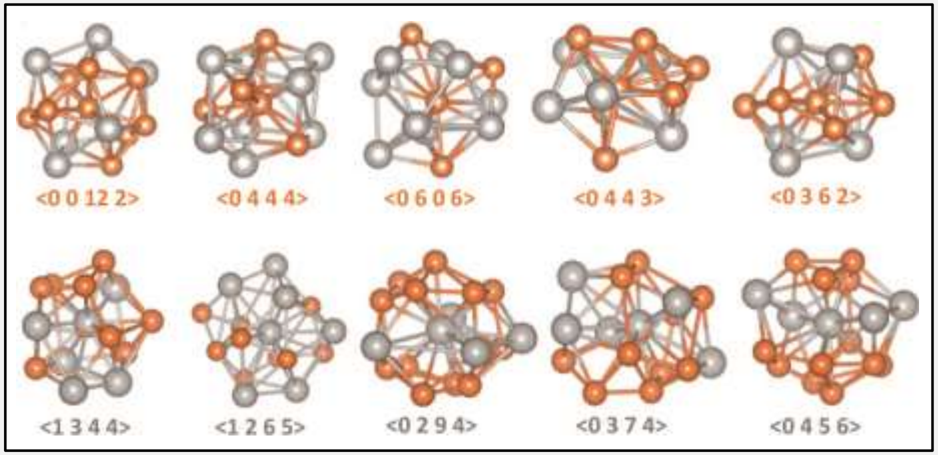
processing & structure-property relation

various processing techniques:
- → suction casting
- → centrifugal casting
- → counter-gravity casting
- → thermo-plastic forming
- → …

limitations:
- → sample size
- → cooling rate
- → shape
- → …

Fecht, Johnson, *Mat Sci Eng A*, 2004





- → variations in local atomic packing structure
- → quasi-localized soft spots
- → strongly influence shear deformation

Ding, Patinet, Falk, Cheng, Ma, *PNAS*, 2014

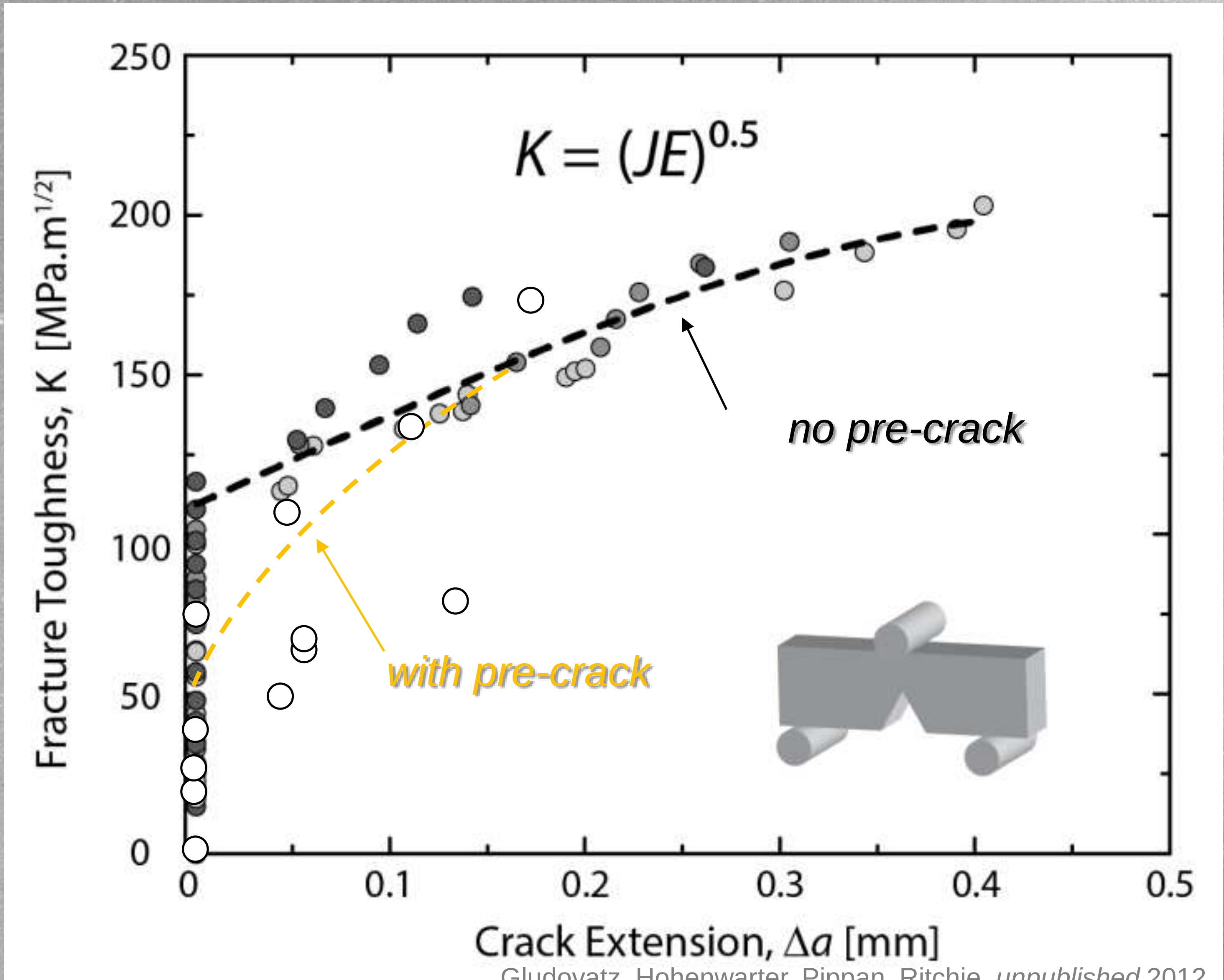
Current issues with metallic glasses

Major problem: VARIABILITY of mechanical properties

i) Processing: - 'structure'-property relation

ii) Mechanical testing: - notch sensitivity

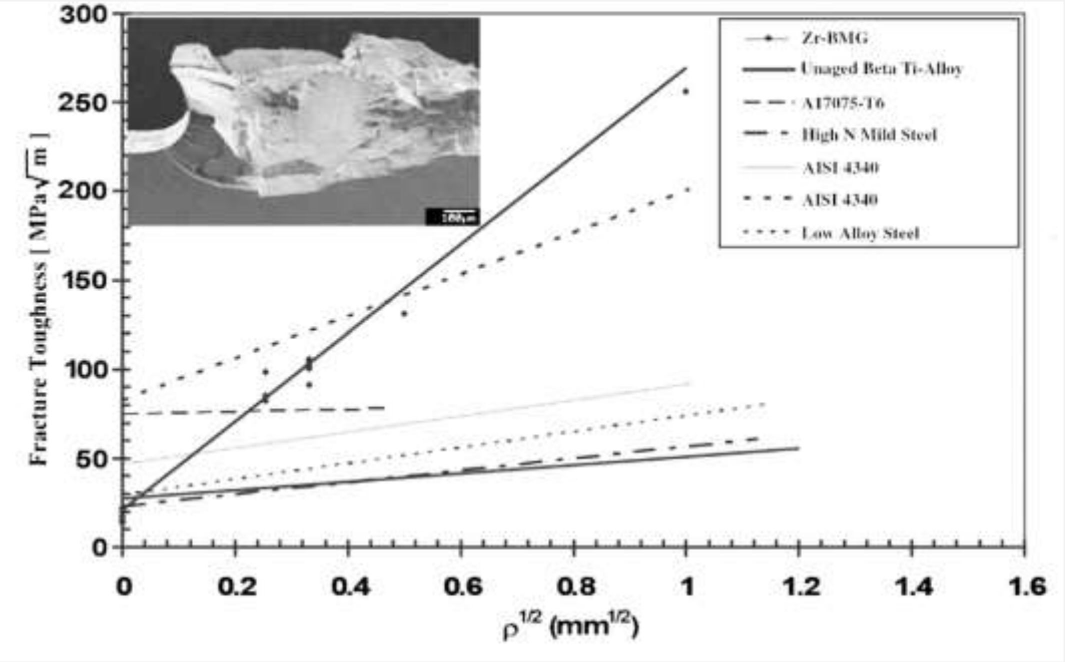
mechanical testing: i) notch sensitivity



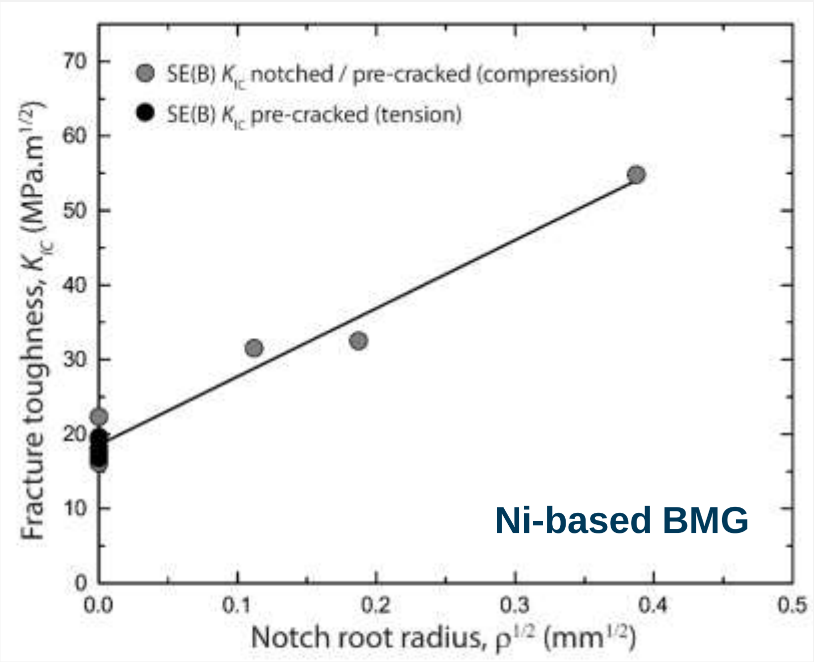
Gludovatz, Hohenwarter, Pippan, Ritchie, *unpublished* 2012

mechanical testing: i) notch sensitivity

- increased notch root radius, $\rho \uparrow \Rightarrow$ **artificially** elevates toughness, $K_Q \uparrow$
- notch root sensitivity varies for different compositions
- effect possibly more pronounced than in crystalline materials
- K_Q values of samples tested with same ρ comparable



Lewandowski, Shazly, Nouri, *Scripta Mater*, 2006



Gludovatz, Demetriou, Johnson, Ritchie, *unpublished*

Current issues with metallic glasses

Major problem: VARIABILITY of mechanical properties

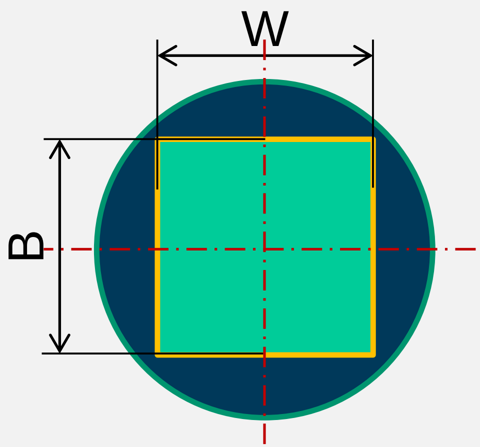
i) Processing: - ‘structure’-property relation

ii) Mechanical testing: - notch sensitivity

- sample size

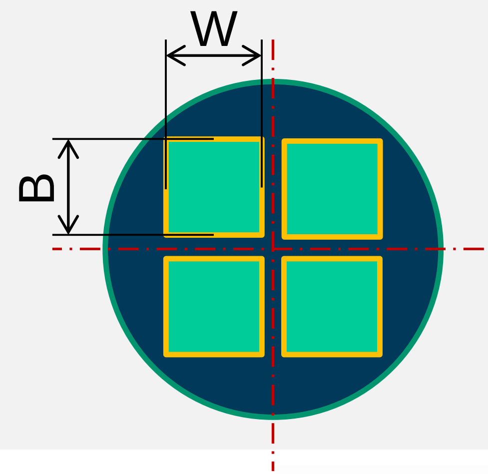
mechanical testing: ii) sample size

SE(B) *without* pre-crack:

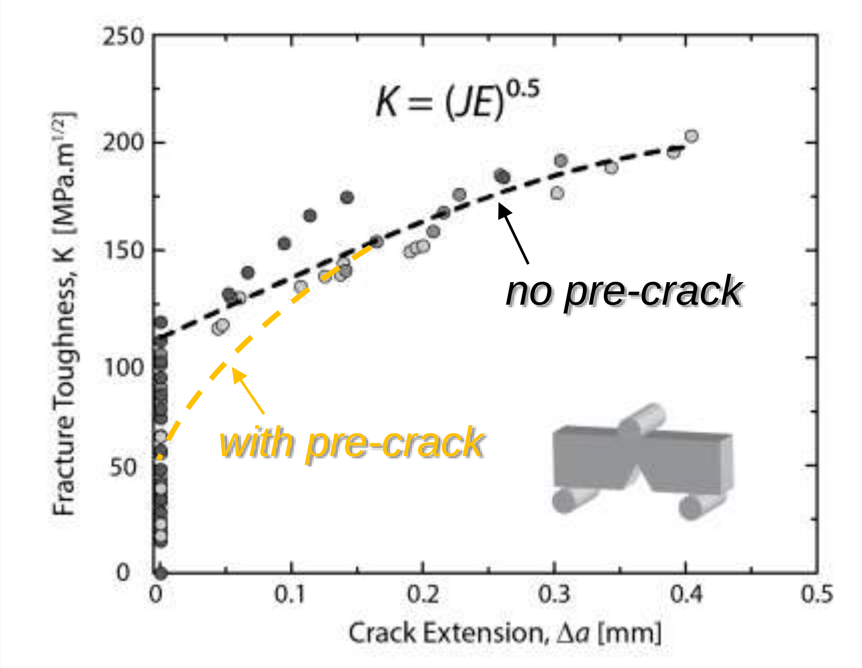


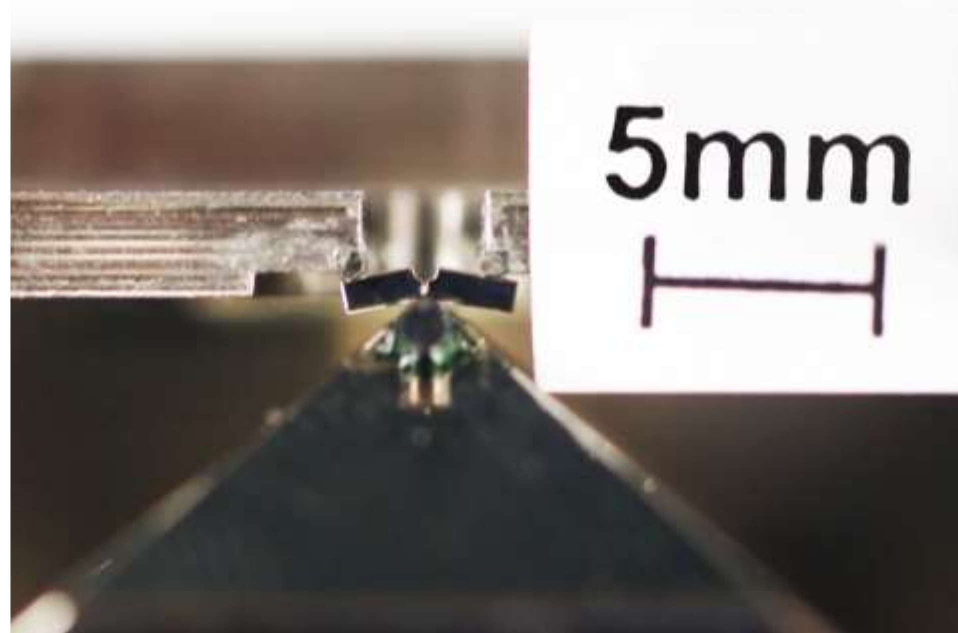
S ~ 15 mm
B ~ 2.1 mm
W ~ 2.1 mm
a ~ 0.5 W
→ b ~ 1 mm

SE(B) *with* pre-crack:



S ~ 2.9 mm
B ~ 0.7 mm
W ~ 0.7 mm
a ~ 0.5 W
→ b ~ 0.35 mm

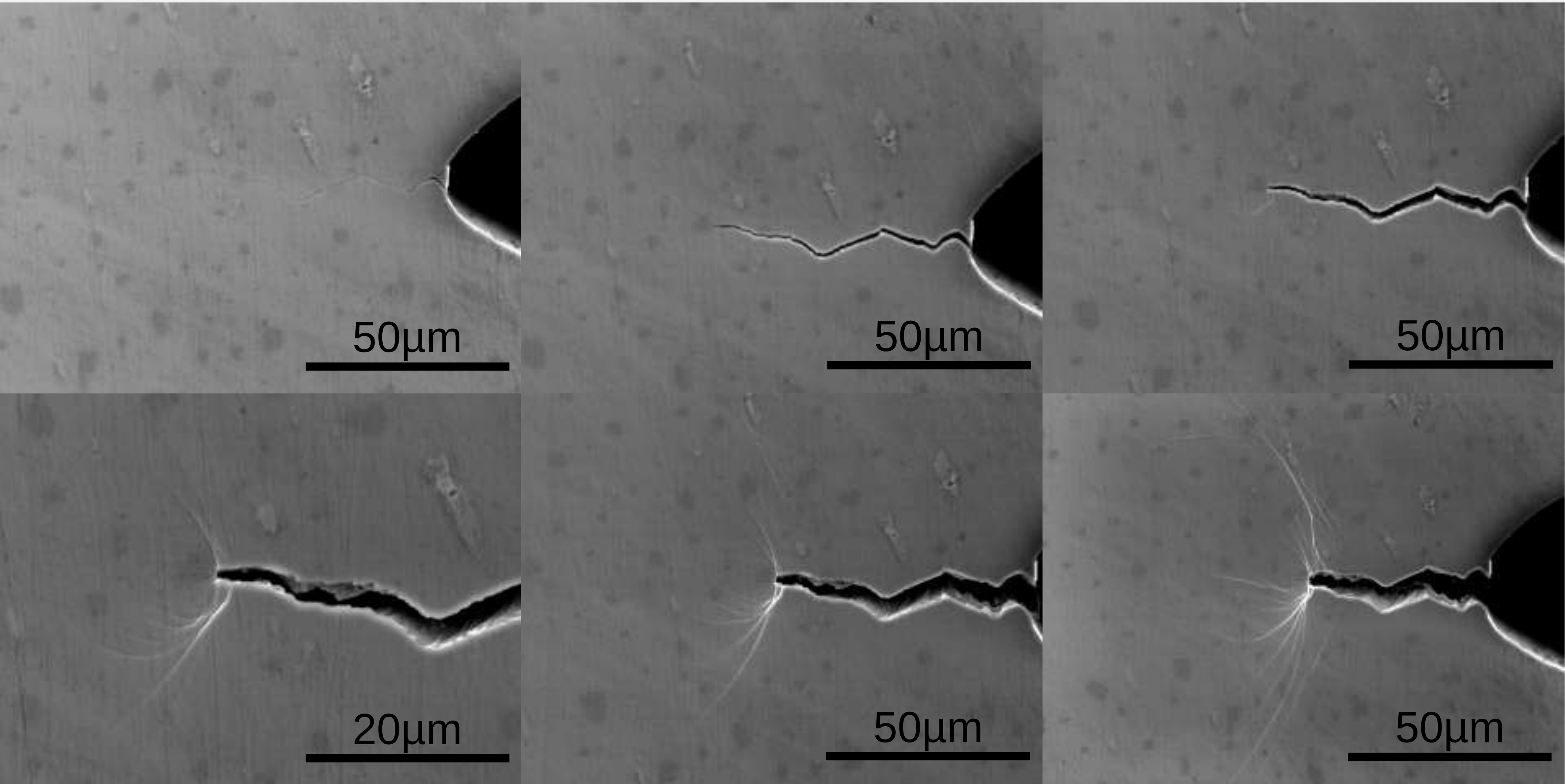




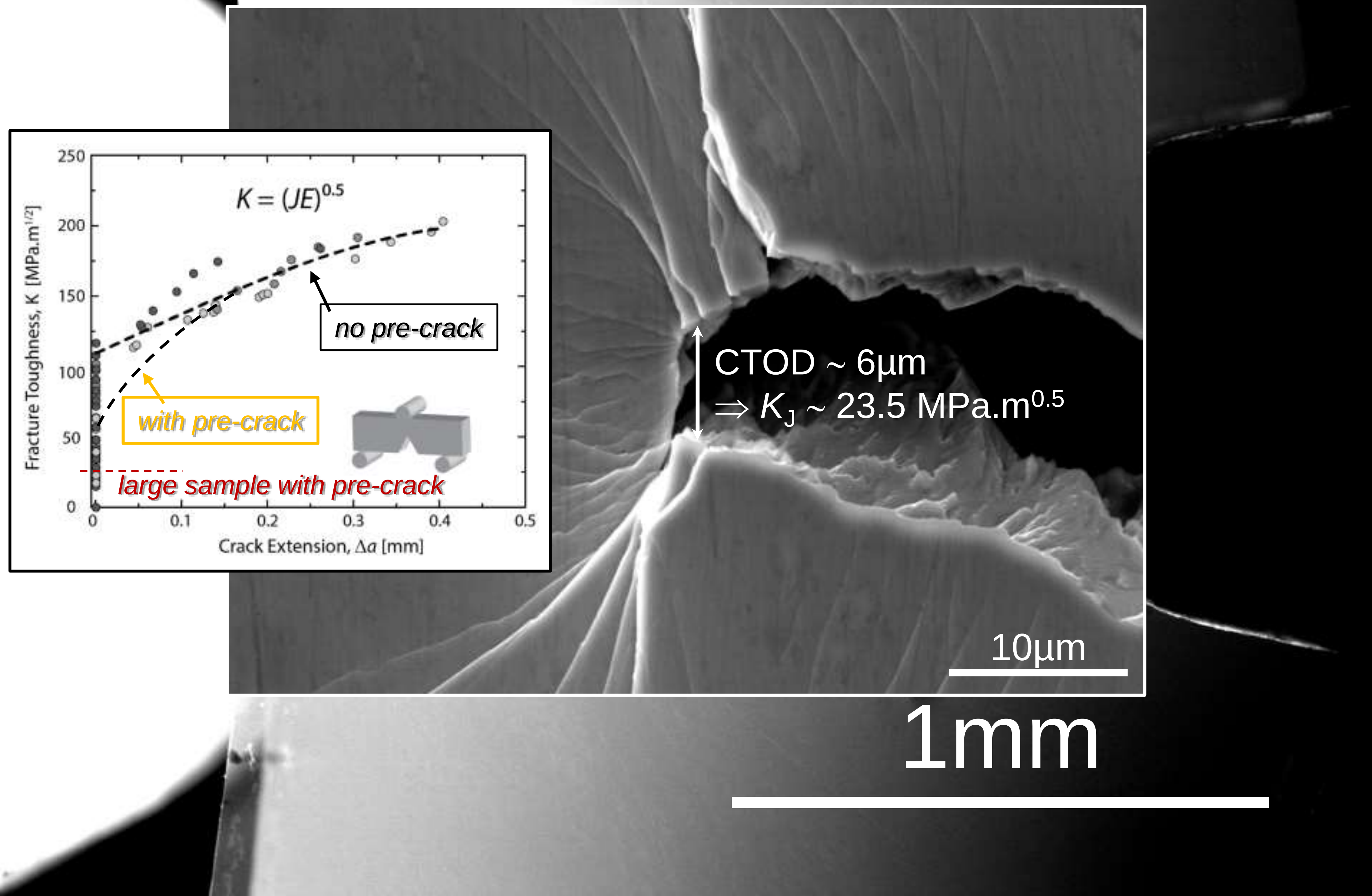
Demetriou, Launey, Garrett, Schramm, Hofmann, Johnson, Ritchie, *Nat Mater*, 2011
Gludovatz, Hohenwarter, Pippan, Ritchie, *unpublished*

SE(B) *with* pre-crack:

(S ~ 8 mm, B ~ 2.1 mm, W ~ 2.1 mm, a ~ 0.5 W, *b ~ 1mm*)



mechanical testing: ii) sample size



Current issues with metallic glasses

Major problem: VARIABILITY of mechanical properties

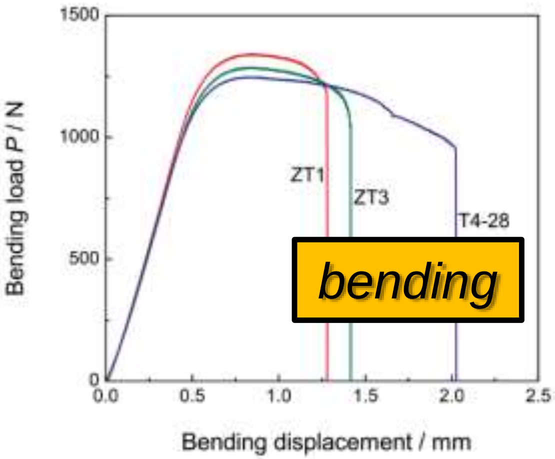
i) Processing: - 'structure'-property relation

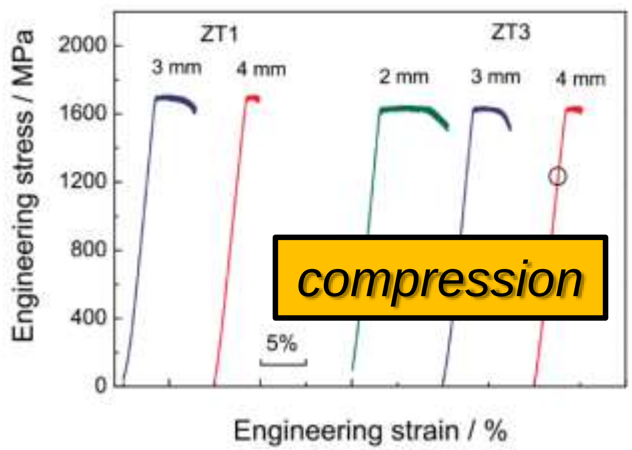
ii) Mechanical testing: - notch sensitivity

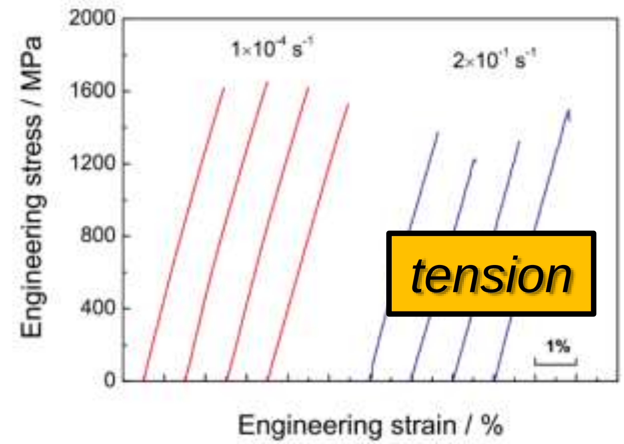
- sample size
- loading condition

mechanical testing: iii) loading condition

BMGs show major differences when being loaded in …







He, Xu, *J Mater Sci Technol*, 2012

<u>SE(B) & SE(T) samples:</u>

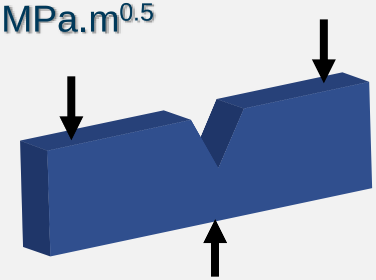
B ~ 3.5 mm / *W* ~ 7 mm / *a* ~ 0.5 *W*

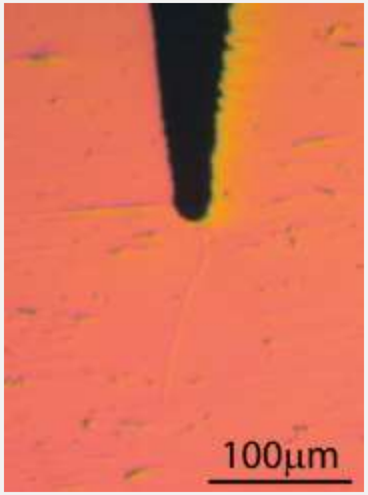
<u>pre-cracking in bending:</u>

ΔK = 5.5 – 7 MPa.m$^{0.5}$

R = 0.1

f = 25 Hz





? K_{Ic} (bending) > K_{Ic} (tension) ?

$Zr_{56}Ni_{25}$Al15Nb_4 (ZNAN) - brittle	
BENDING	*TENSION*
~48 MPa.m$^{0.5}$	~83 MPa.m$^{0.5}$

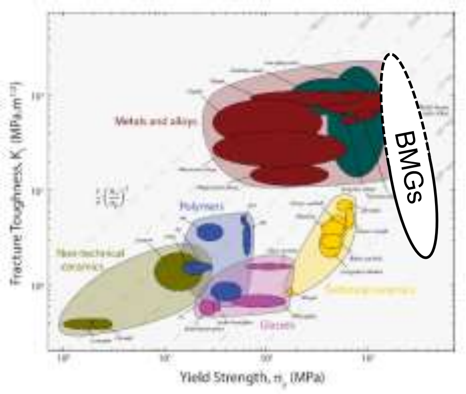
$Zr_{52.5}Cu_{17.9}Ni_{14.6}Al_{10}Ti_5$ (Vit105) - medium	
BENDING	*TENSION*
~59+ MPa.m$^{0.5}$	~88 MPa.m$^{0.5}$

$Zr_{61}Ti_2Cu_{25}Al_{12}$ (ZT1) – ductile	
BENDING	*TENSION*
~64+ MPa.m$^{0.5}$	~129 MPa.m$^{0.5}$

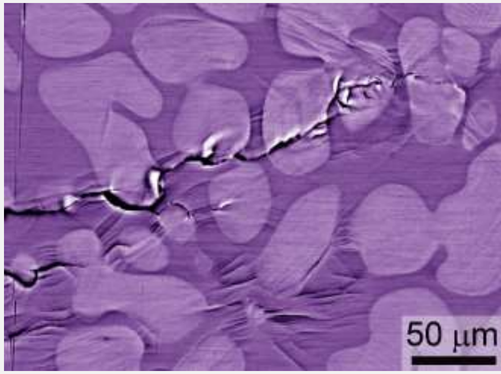
Gludovatz, Garrett, Demetriou, Ritchie, *unpublished*, 2015

Conclusions (part I)

Metallic glasses show good combination
of strength and toughness



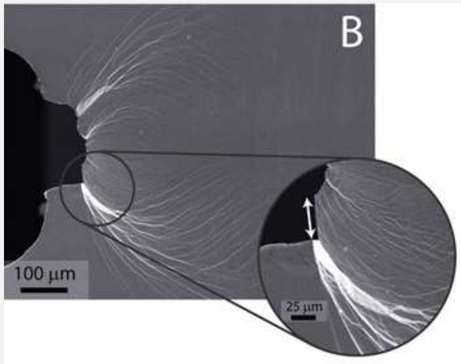
- BMGs … fascinating class of potential structural materials with a decent combination of mechanical properties:
  - low stiffness & high strength (0.5 – 4 GPa)
  - reasonable fracture toughness (10 – 100 MPa.$m^{0.5}$)
  - 'OK' fatigue strength (0.05 – 0.2 σ_{UTS})



- Some glasses/glass-like materials can significantly enhance the fracture/fatigue properties through ductility from "plasticity" via multiple shear-band formation:

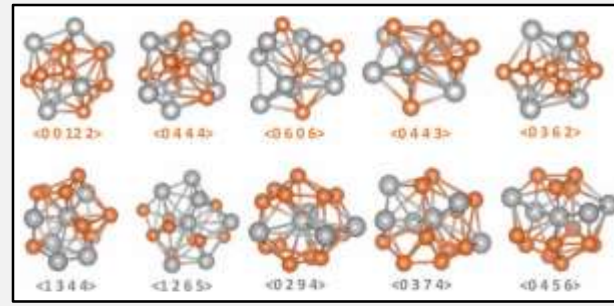
  - ***Glass-composite alloys with second phase dendrites***
    → **ARRESTING** shear bands before they can form cracks
    → microstructural length-scales, $\lambda \leftrightarrow \boldsymbol{a_c}$, mechanical length-scales

  - ***Fully amorphous glasses***
    → ***FORMATION & PROLIFERATION of multiple shear bands***
    … *BEFORE* crack initiation and propagation



⇒ excellent toughness & fatigue behavior!

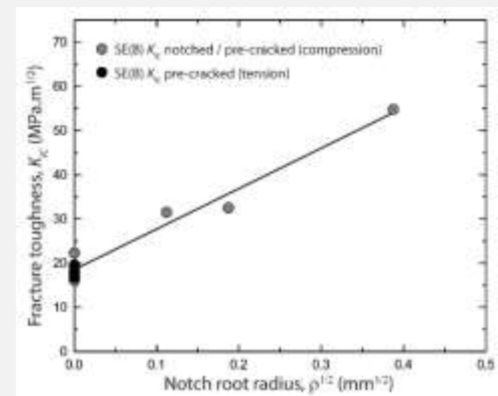
⇒ Major problem: Variability of mechanical properties



Processing

- ***structure-property relationship***
  → local atomic packing structure / quasi-localized soft spots strongly influence shear deformation

Mechanical testing …



- ***notch root radius / flaw sensitivity***
  → larger notch root radii artificially increase the fracture toughness
  → notch root sensitivity varies for different compositions
- ***sample size***
  → smaller samples seem to behave more 'plastic' (K_{Ic}, K_{JIc}, K_Q ↑) & show stable crack propagation rather than catastrophic failure
- ***loading condition***
  → no obvious bending ductility
  → large variation in the results of both bending and tension
  → trend to higher numbers in tension

NEED to understand variability of mechanical properties in BMGs to be able to use them in structural applications

EPFL – Seminar, 1st February 2016

Fracture & Fatigue of Multi-Component Alloys

Fracture toughness of the CrCoFeMnNi high-entropy alloy at cryogenic temperatures

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California Institute of Technology, Pasadena

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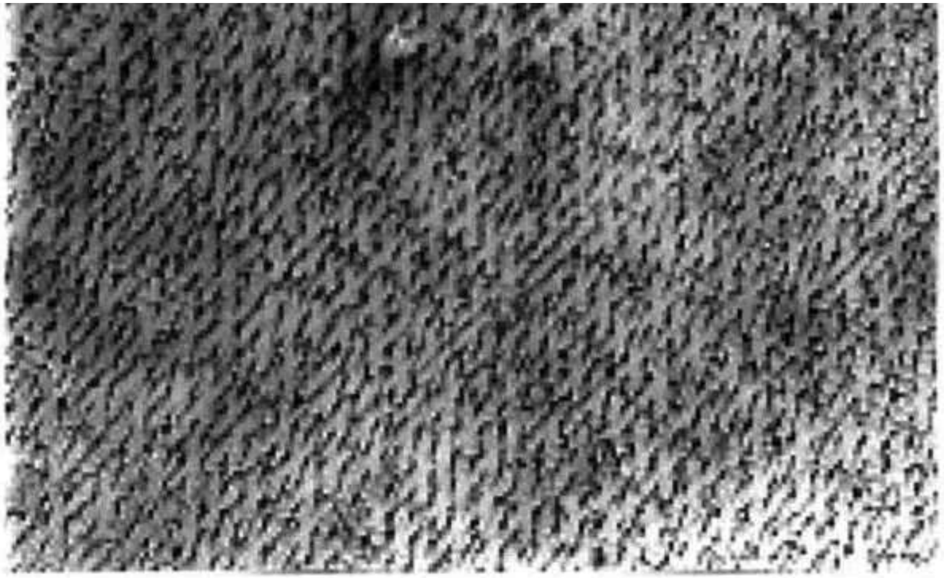
Anton Hohenwarter
University of Leoben, Austria

Work supported by the Office of Science (Basic Energy Sciences) of the Department of Energy (Berkeley, Oakridge) and by the National Science Foundation (Caltech).

A new class of equiatomic alloys …

Cast dendritic structure but single-phase FCC

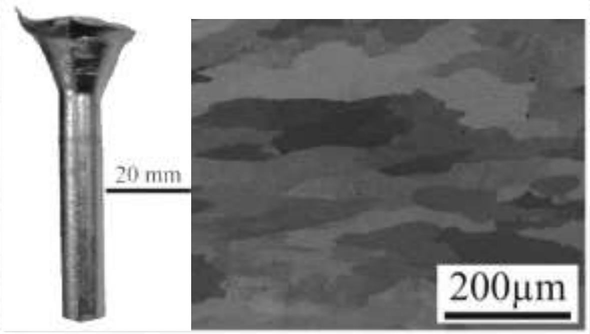


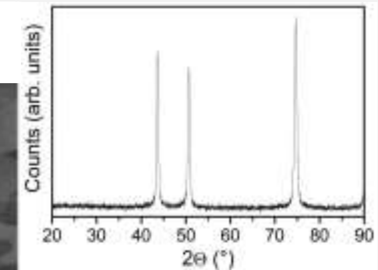
1 mm

First identified 2004 by Cantor et al.

CrCoFeMnNi

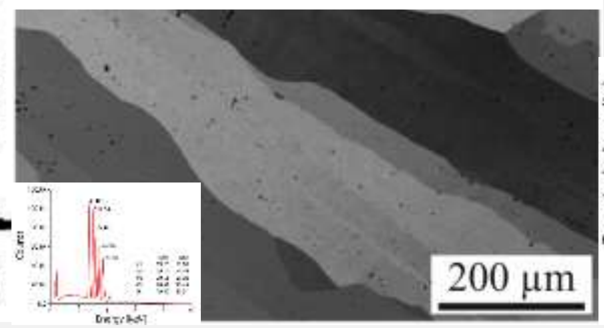
As cast

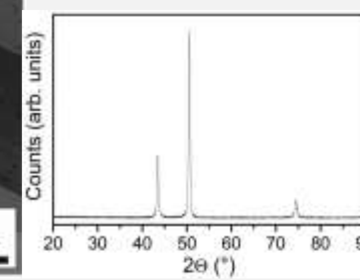




Stable single-phase FCC solid solution

Annealed 72h @ 1000C





Arc-melted and drop-cast alloy is indeed single phase FCC

Why HEA?

Element	Ni	Fe	Cr	Co	Mn
Crystal structure	fcc	bcc	bcc	hcp	A12

⇒ Hume-Rothery rules for solid solubility do ***NOT*** apply

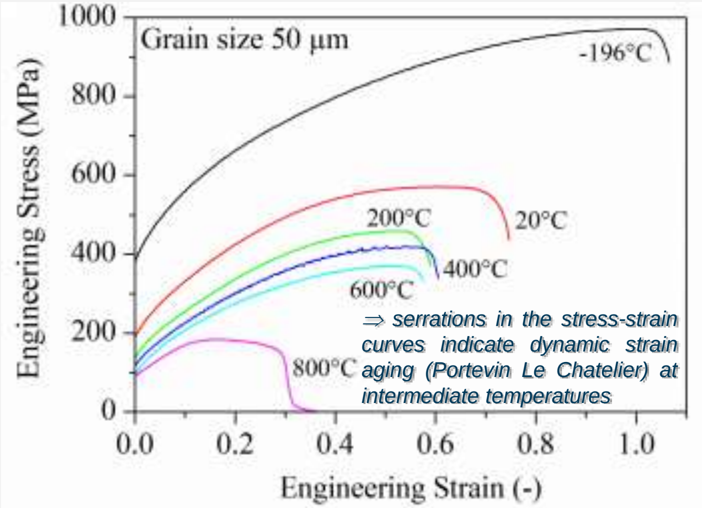
⇒ *Configurational entropy* can stabilize solid solutions (relative to compound/precipitate formation)

⇒ ***High-entropy alloys*** *(number of elements ≥ 5)*

$$\Delta G = \Delta H - T\Delta S$$

Cantor et al., Mater. Sci Eng. 2004
Yeh et al., Adv. Eng. Mater. 2004
Otto et al., Acta Mater. 2013

Tensile stress-strain behavior



⇒ **strong temperature** dependence of strength and ductility

⇒ **highest ducitilites at -196C** *(likely due to prevention of necking)*

⇒ degree of **work hardening** ($\sigma_u - \sigma_y$) **highest at -196C**

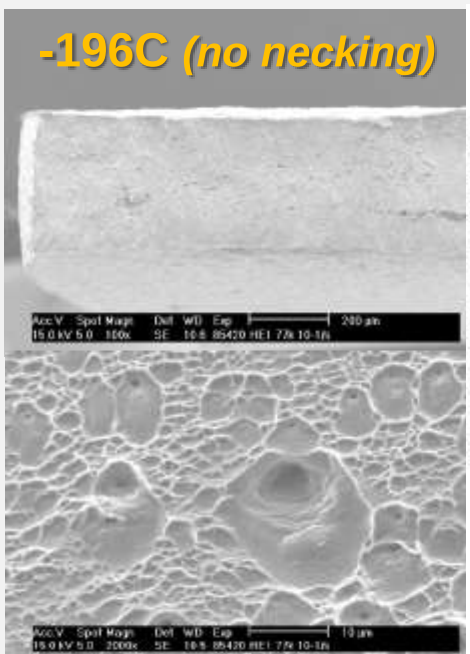
⇒ σ_y approx doubles from RT to -196C (*thermally activated yielding*)

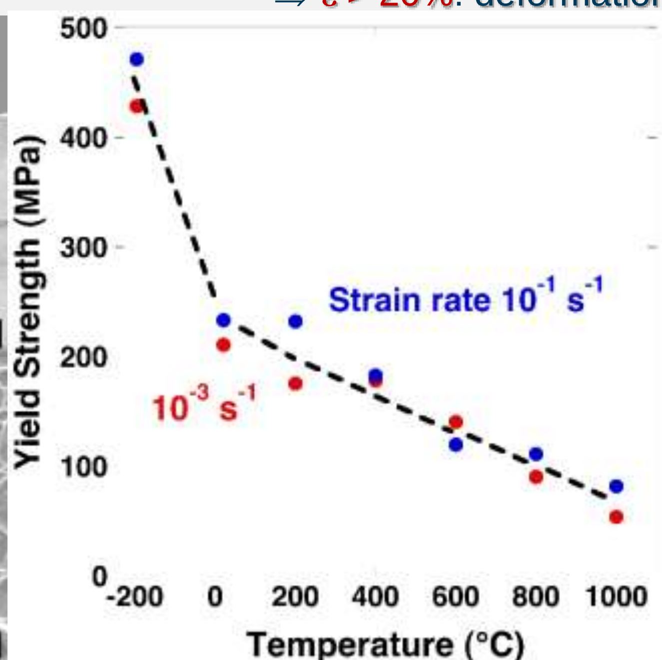
⇒ **σ_y insensitive to strain rate** (unusual for thermally activated yield)

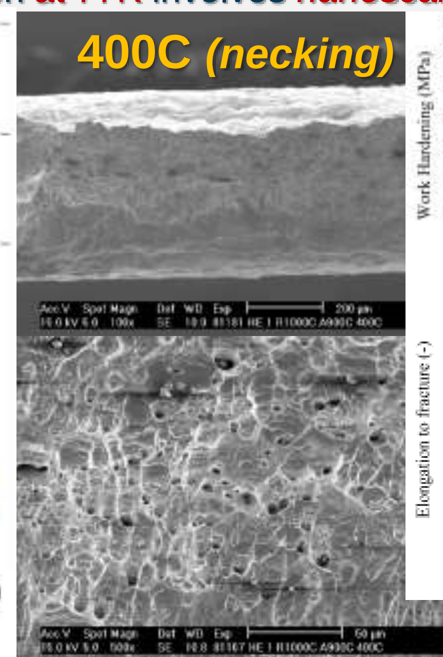
Deformation:

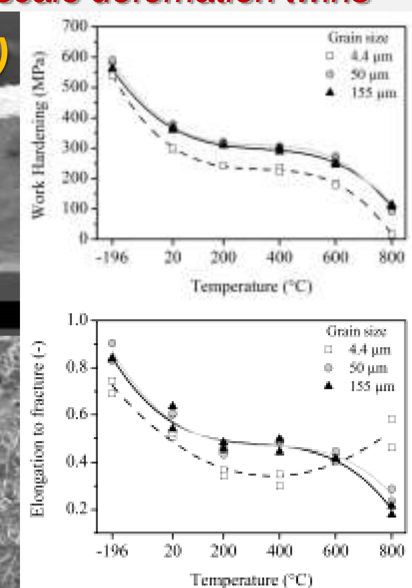
⇒ ε < 2 %: deformation by *planar slip* on {111}<110> (*ALL* temperatures)

⇒ ε > 20%: deformation at 77K involves *nanoscale defomation twins*



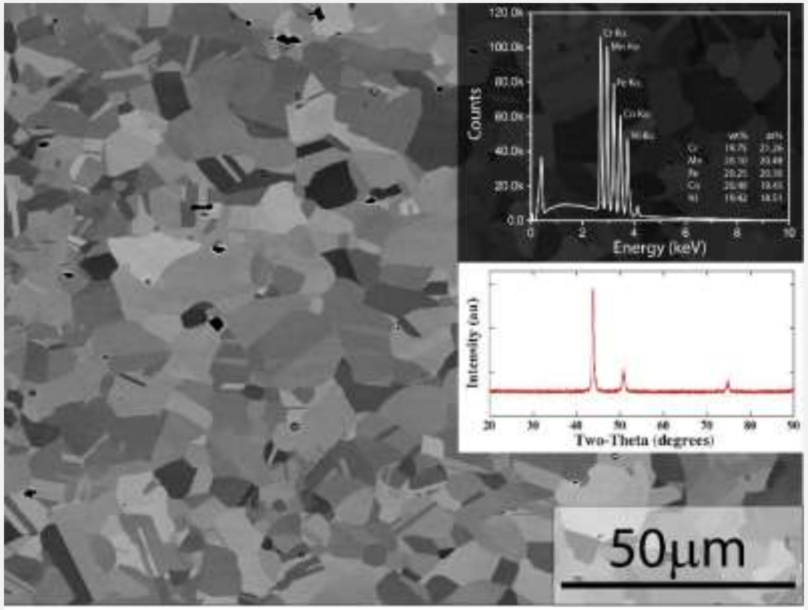


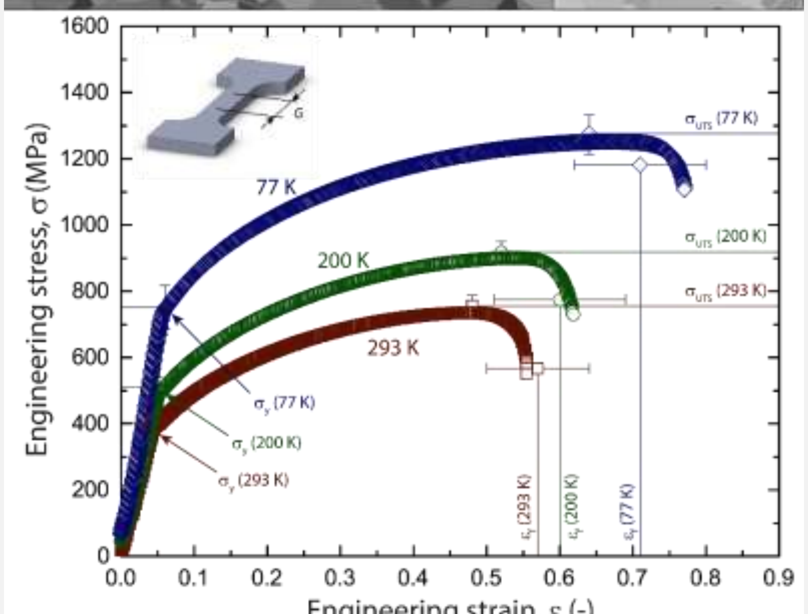




Gali et al., Intermet. 2013
Otto et al., Acta Mater. 2013

Microstructure, elastic & mechanical properties





Microstructure:

- equiatomic, single-phase material
- equiaxed grains
- grain size ~ 6 μm
- numerous recrystallization twins

Elastic properties:

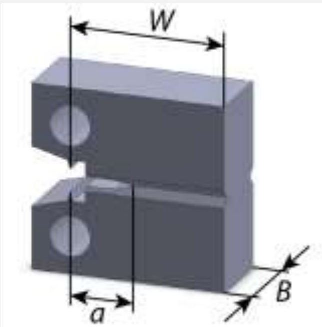
temp (K)	E (GPa)	ν
77	214.5	0.256
200	209	0.263
293	202	0.267

Mechanical properties:

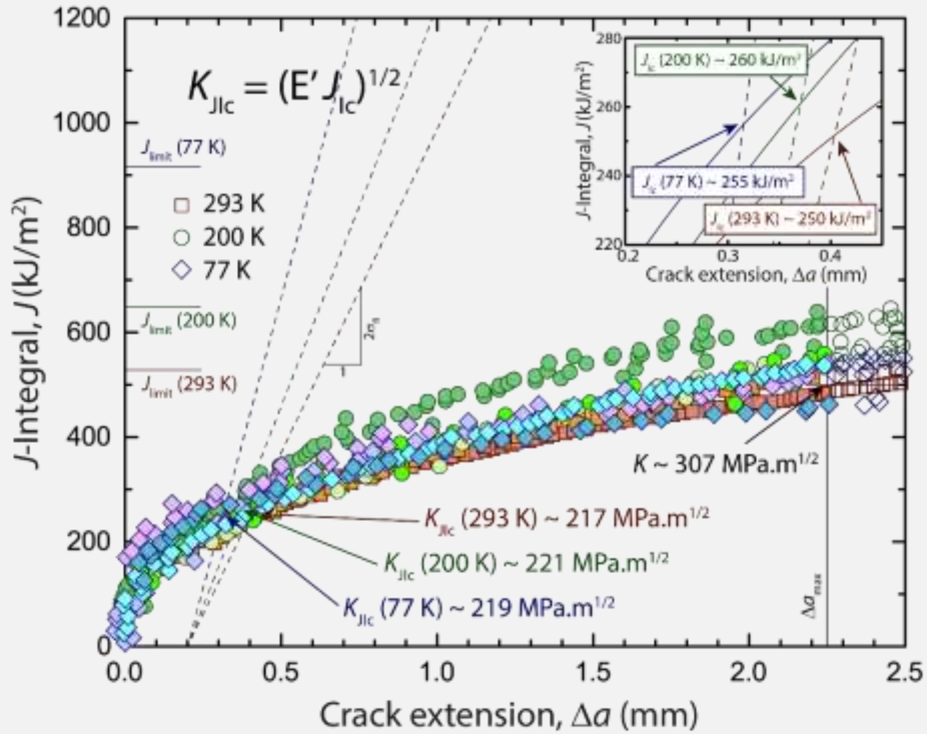
temp (K)	σ_y (MPa)	σ_{UTS} (MPa)	ε_f	n (0.06-0.15)
77	85% increase in σ_y	70% increase in σ_{UTS}	25% increase in ε_f	0.36
200				0.41
293				0.41

Gludovatz, Hohenwarter, Catoor, Chang, George, Ritchie *Science* 2014

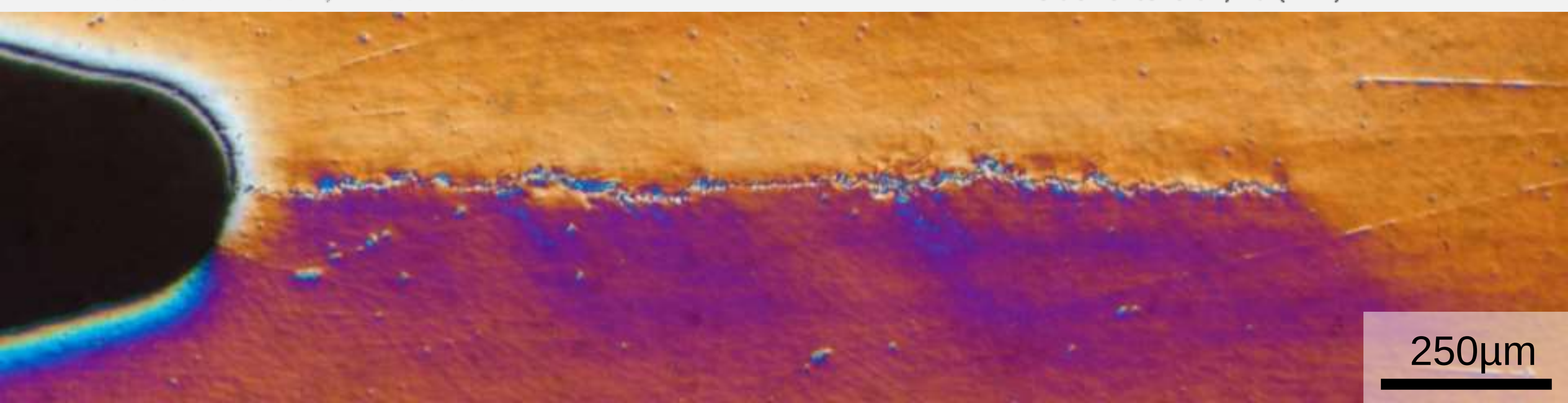
Fracture toughness measurements



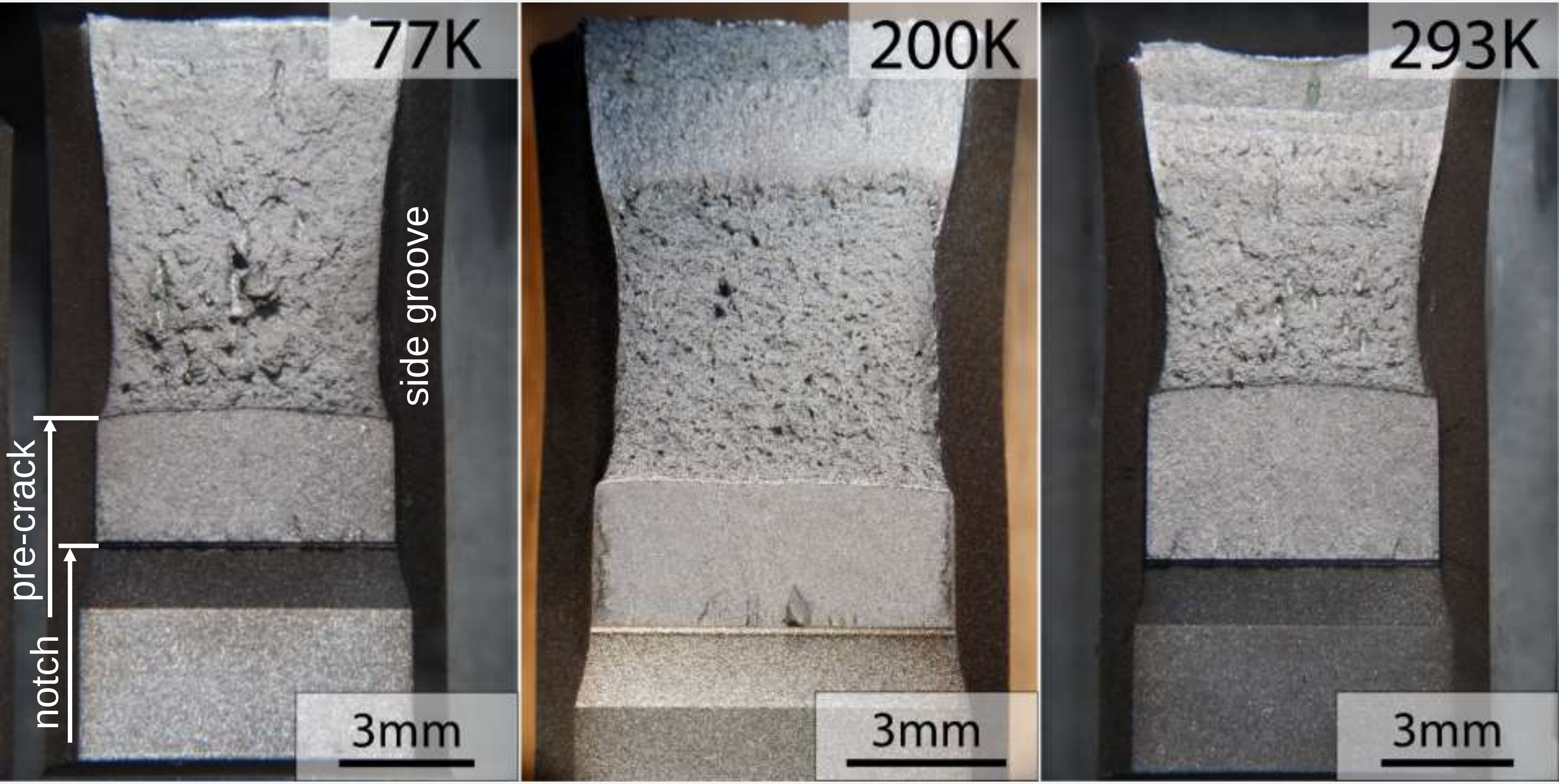
C(T)-samples:
W = 18 mm
B ~ 9 mm / B_N ~ 7 mm



- → samples machined by EDM
- → surfaces polished using SiC-paper
- → pre-cracked in tension
  (R = 0.1, ΔK = 12 – 13 MPa.m$^{1/2}$)
- → side-grooved by EDM
- → tested at 293K, ~200K & ~77K



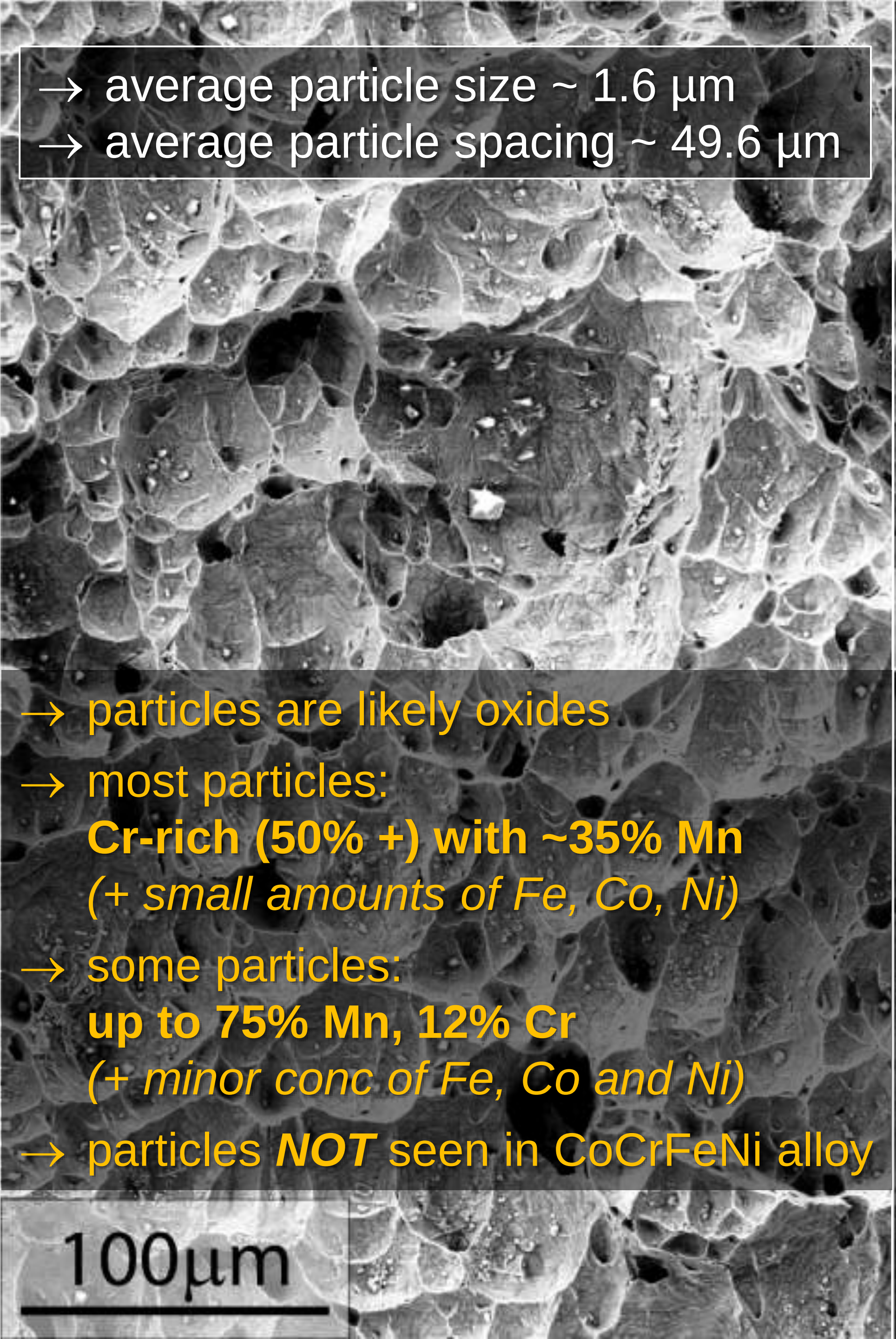
Fractography



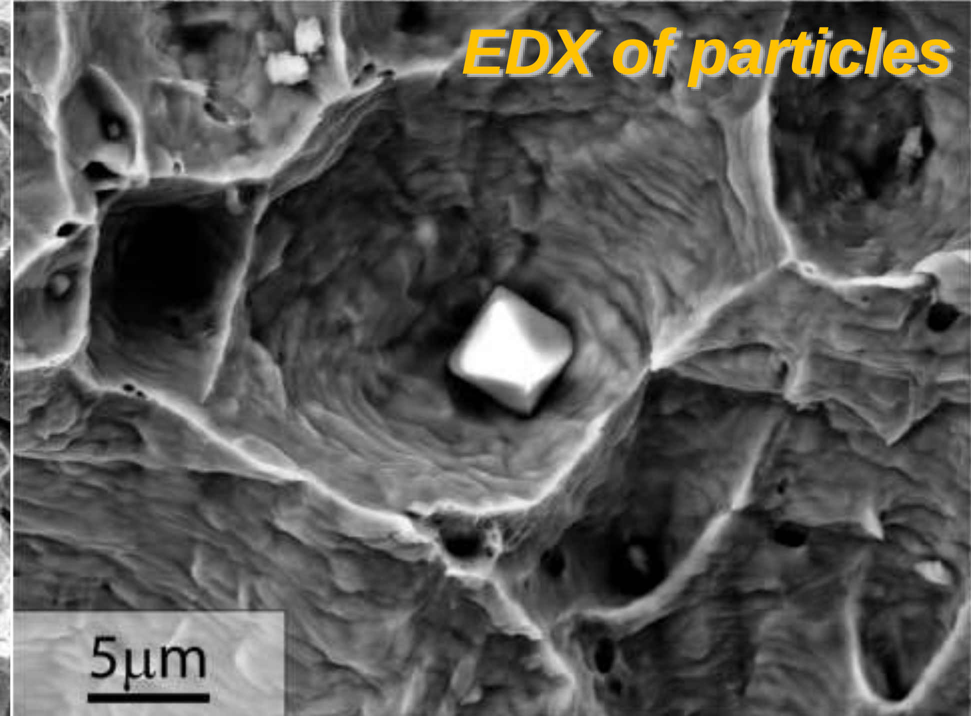
- after the last loading/unloading cycle, one sample of each temperature was fatigued to failure in order to investigate the fracture surfaces

Gludovatz, Hohenwarter, Catoor, Chang, George, Ritchie *Science* 2014

EDX of particles



→ average particle size ~ 1.6 µm
→ average particle spacing ~ 49.6 µm

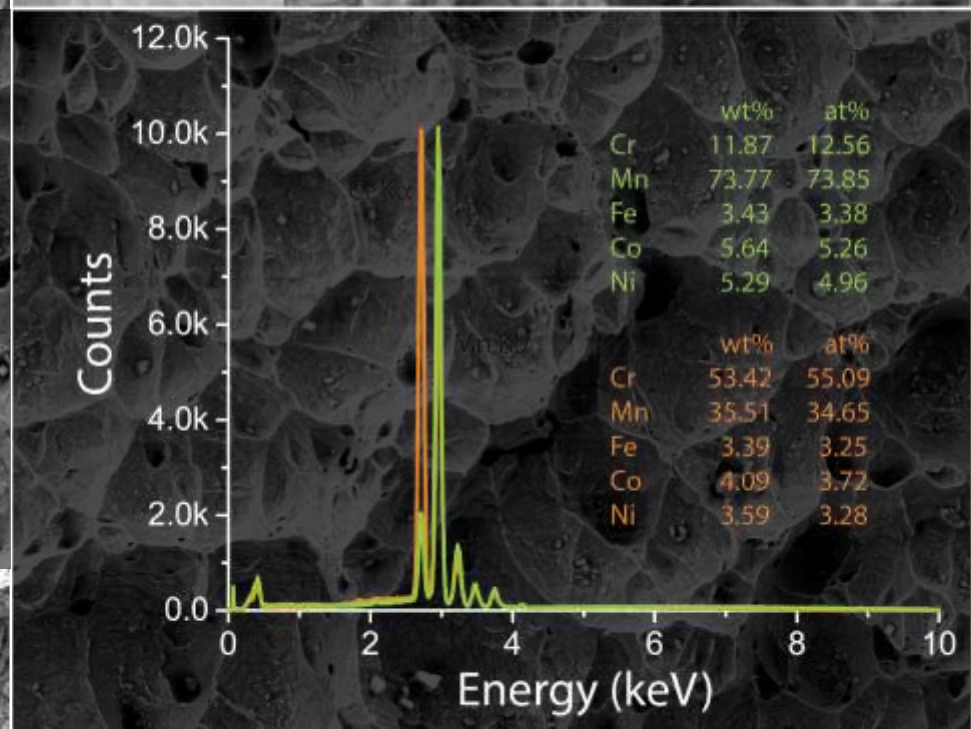


→ particles are likely oxides

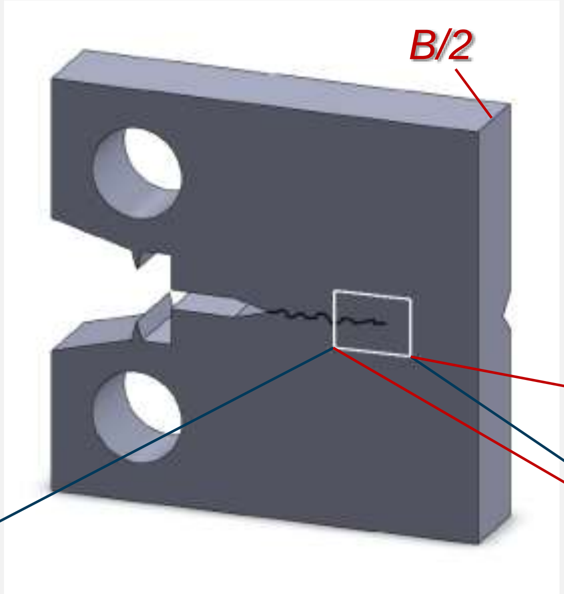
→ most particles:
Cr-rich (50% +) with ~35% Mn
(+ small amounts of Fe, Co, Ni)

→ some particles:
up to 75% Mn, 12% Cr
(+ minor conc of Fe, Co and Ni)

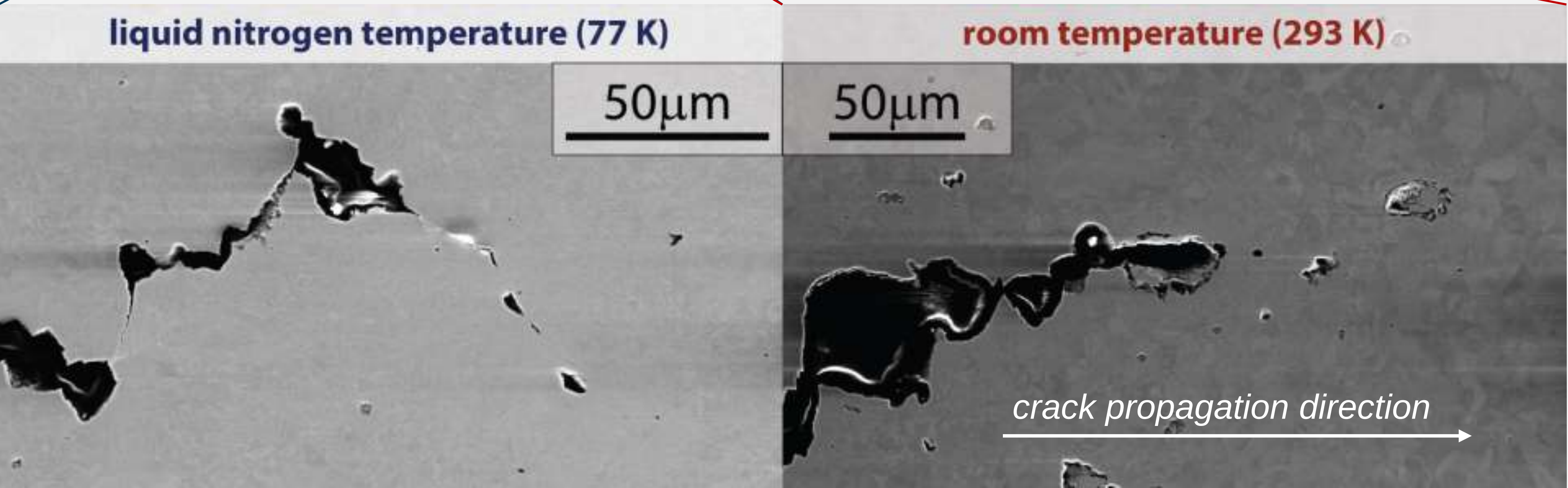
→ particles ***NOT*** seen in CoCrFeNi alloy



Fracture analysis

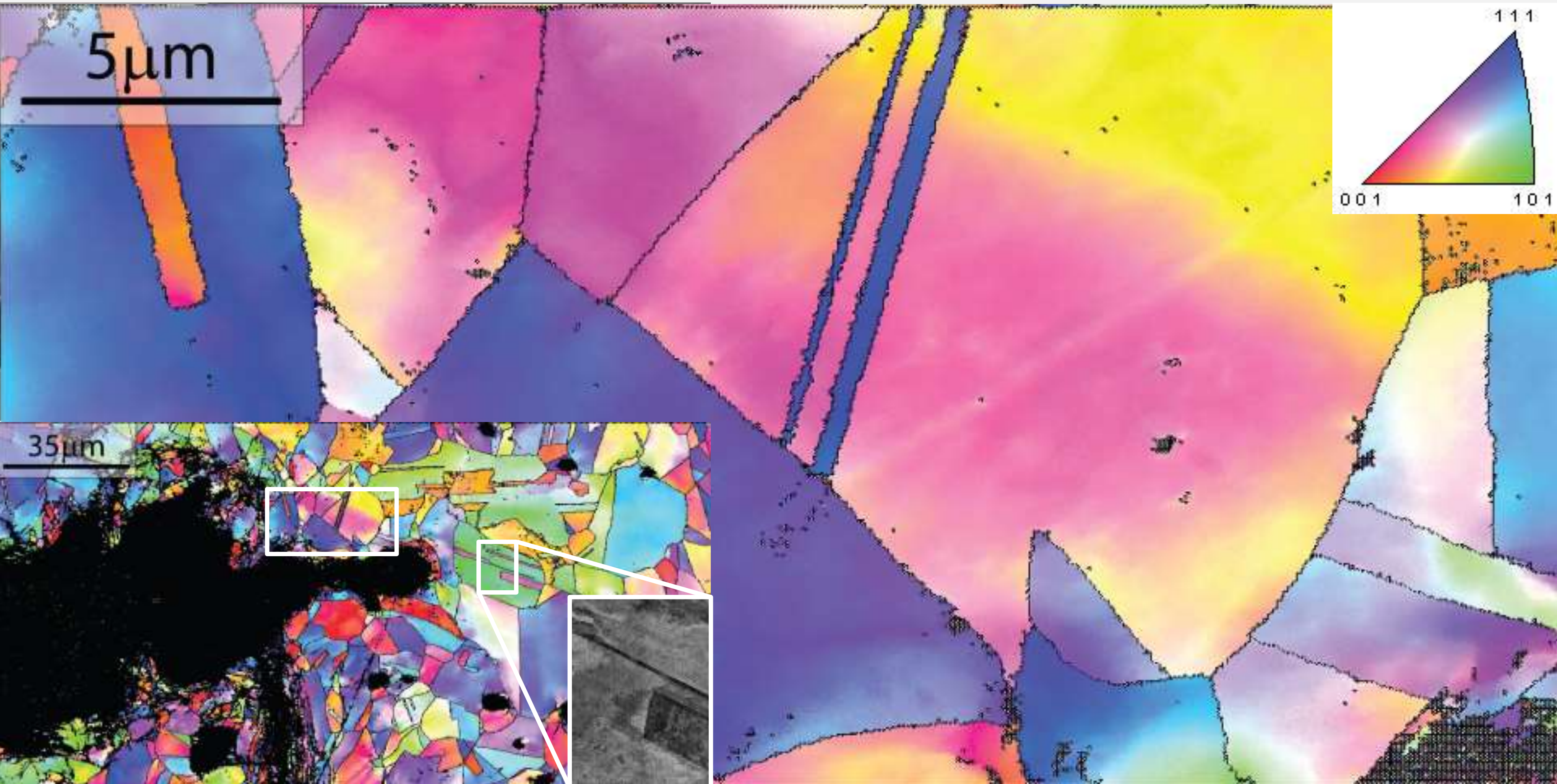


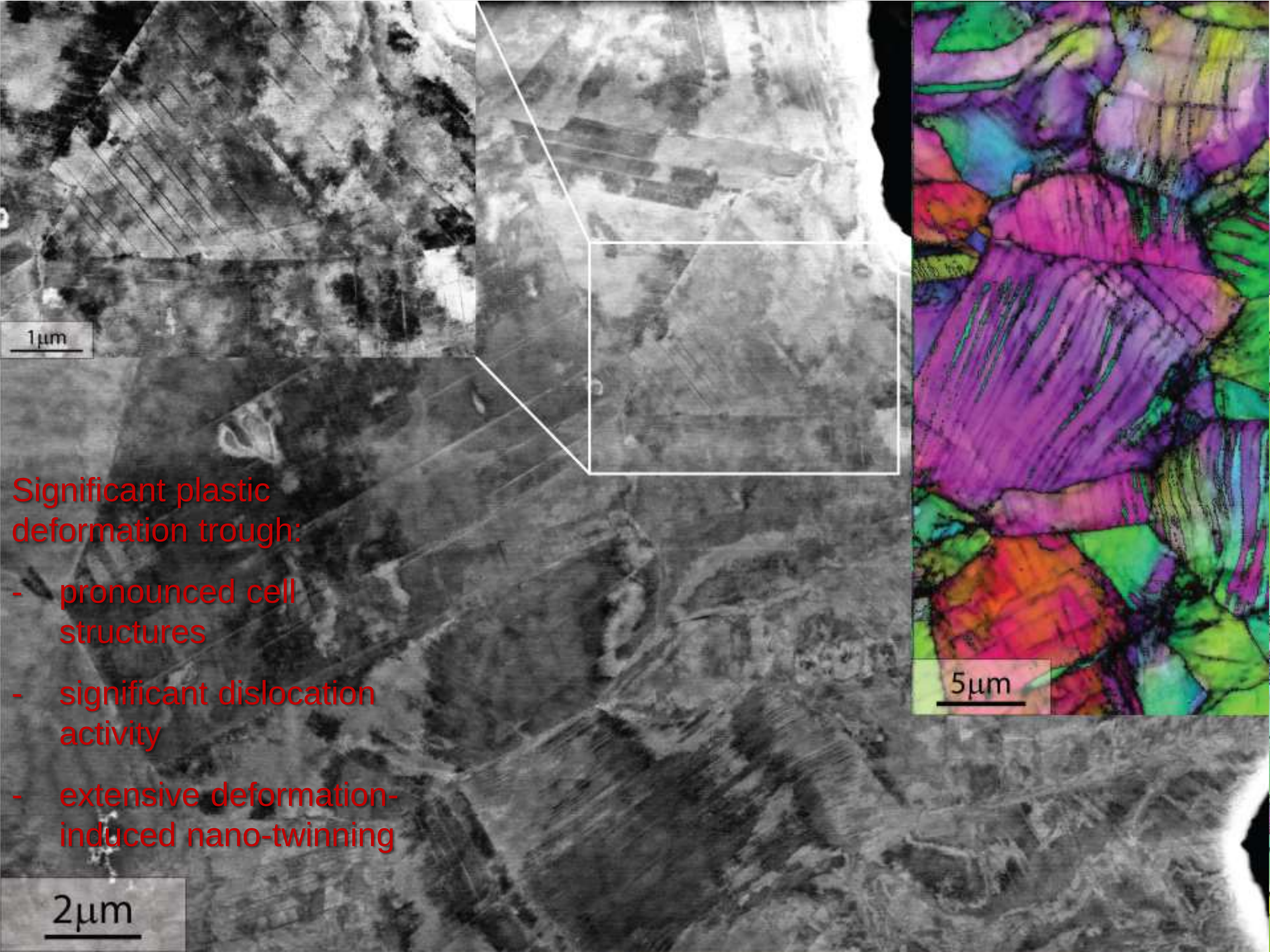
- some samples (293K / 77K) sliced in halves
- each half embedded in conductive / non-conductive resin
- both halves examined using secondary electron (SE) and back-scattered electron (BSE) modes in the SEM



EBSD & BSE at the interior of the crack tip @ 293K

- deformation mainly by dislocation motion (planar slip) → grain misorientations
- no evidence of pronounced deformation-induced nano-twinning
- mainly annealing/recrystallization twins visible





Significant plastic deformation trough:

- pronounced cell structures
- significant dislocation activity
- extensive deformation-induced nano-twinning

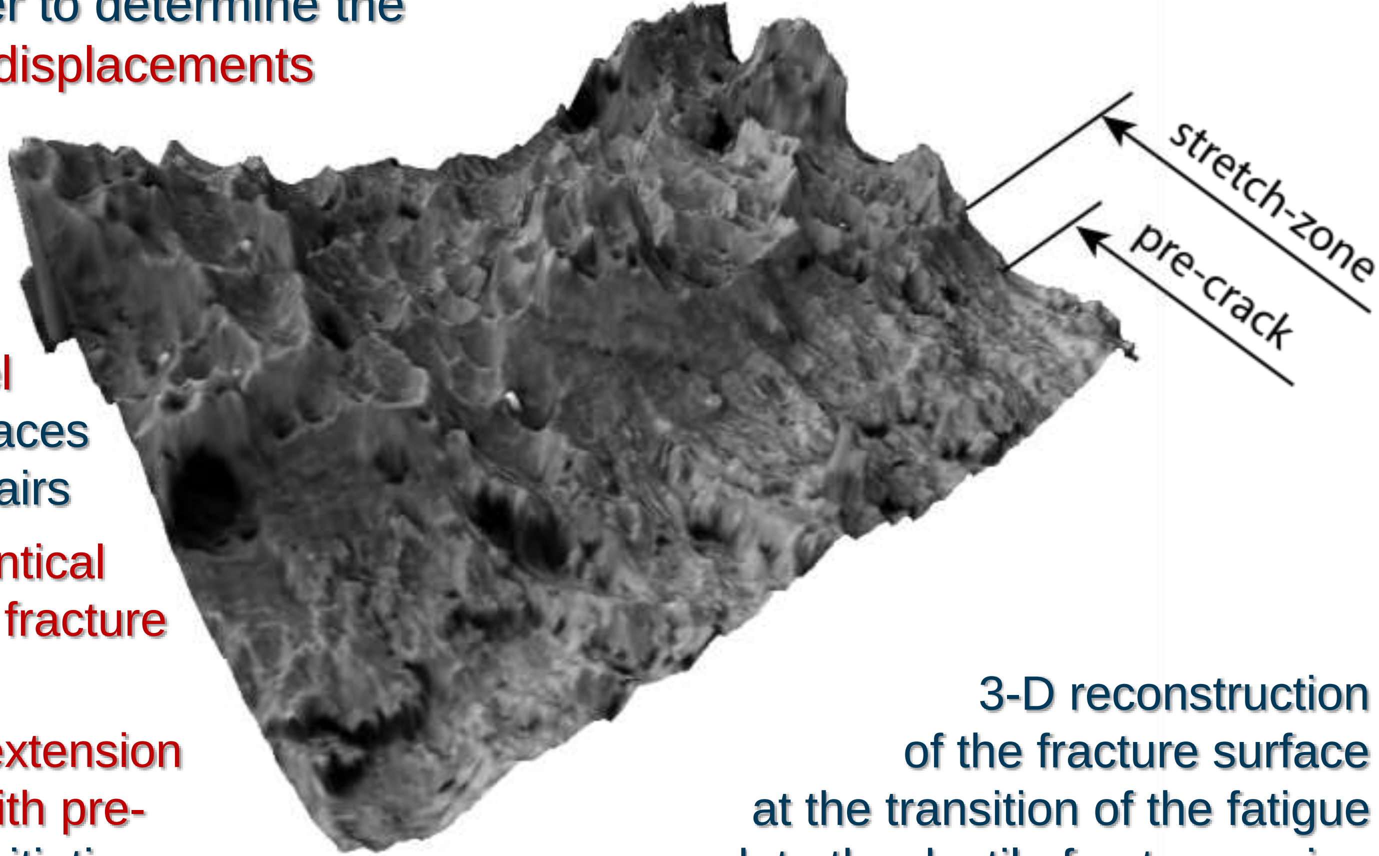
Stereophotogrammetry

- an independent assessment of the toughness can be achieved from the fracture surface
- crack-initiation toughness K_i can also be determined using *stereophotogrammetry* in the SEM in order to determine the crack-tip opening displacements (CTOD) at the onset of crack extension

Global K_{JIc} measurements:

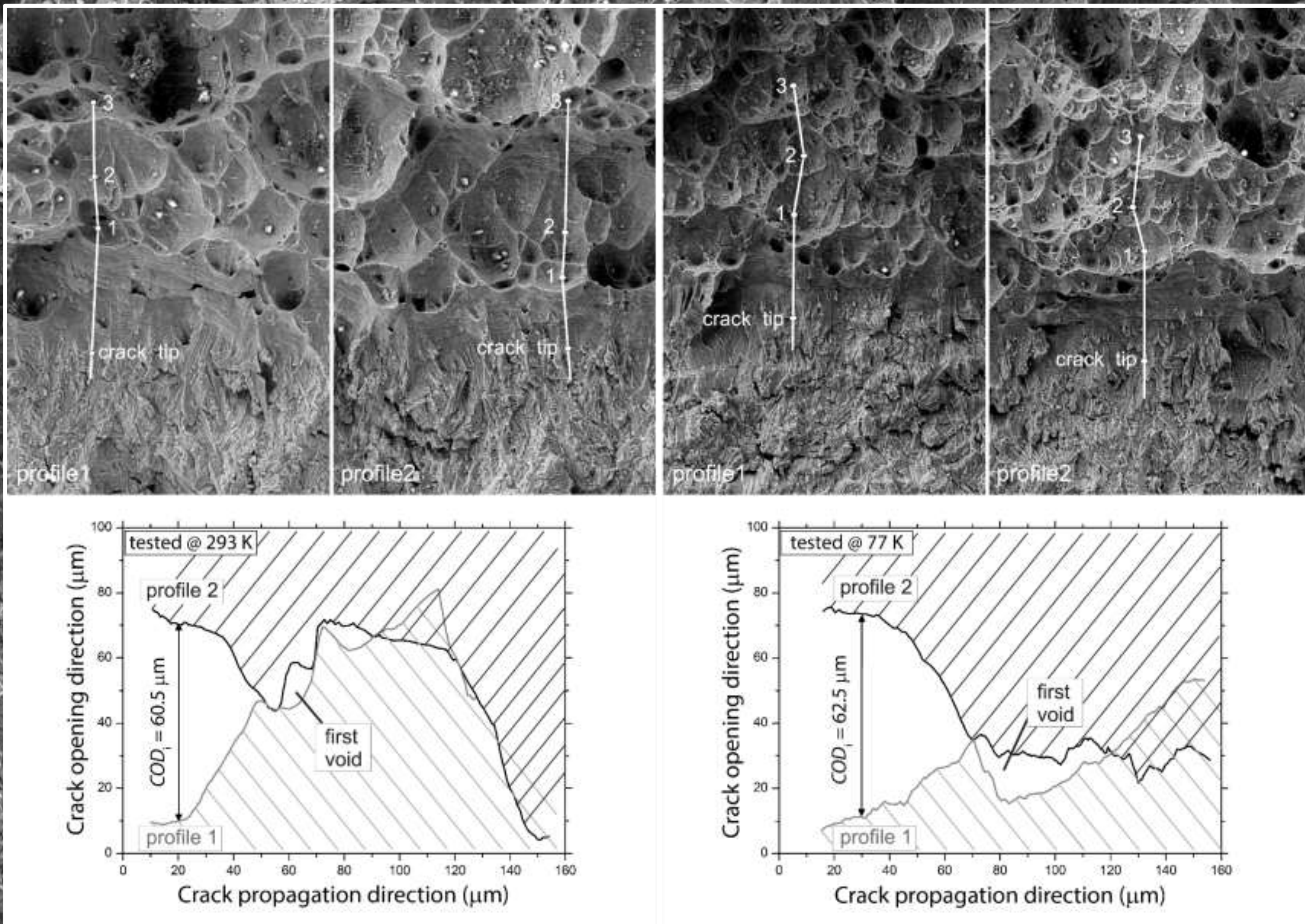
K_{JIc} (293K) ~ 217 MPa√m

K_{JIc} (77K) ~ 219 MPa√m



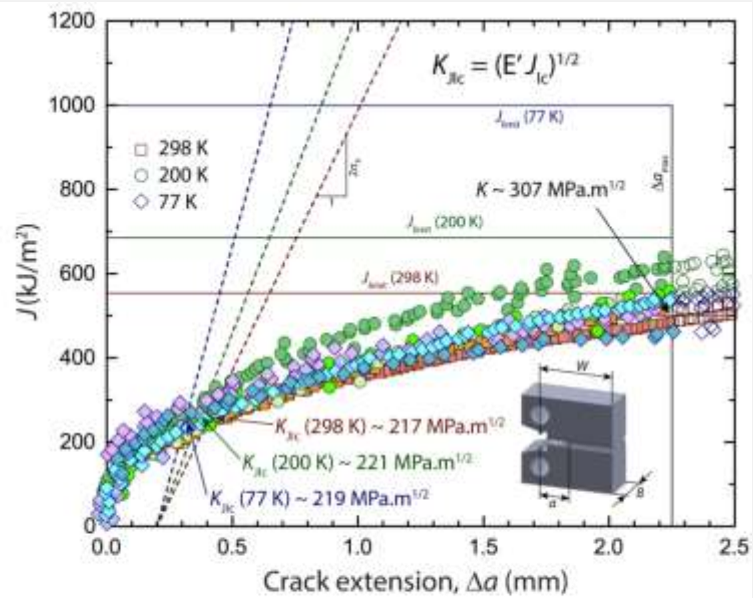
- digital surface model of both fracture surfaces from SEM images pairs
- determination of identical crack paths on both fracture surfaces
- first physical crack extension from coalescence with pre-crack → **$CTOD_i$** at initiation

3-D reconstruction of the fracture surface at the transition of the fatigue pre-crack to the ductile fracture region side 2 (position 1) @ 77K



Profile 3 31.5 µm 61.2 µm 32 µm 59.4 µm

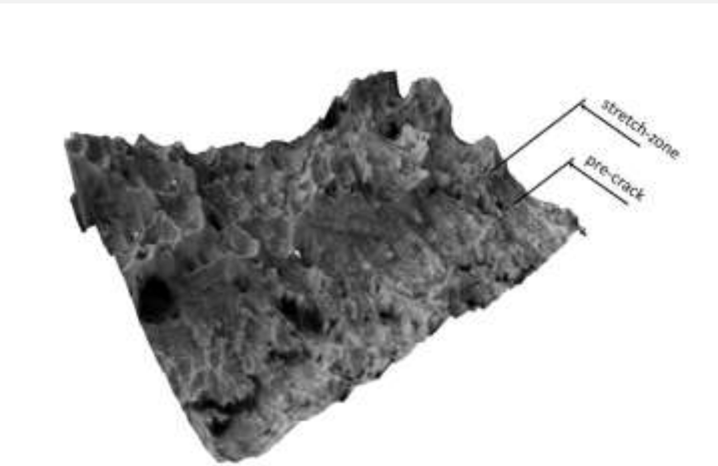
Fracture toughness of CoCrFeMnNi



using ASTM E1820 & stereophotogrammetry

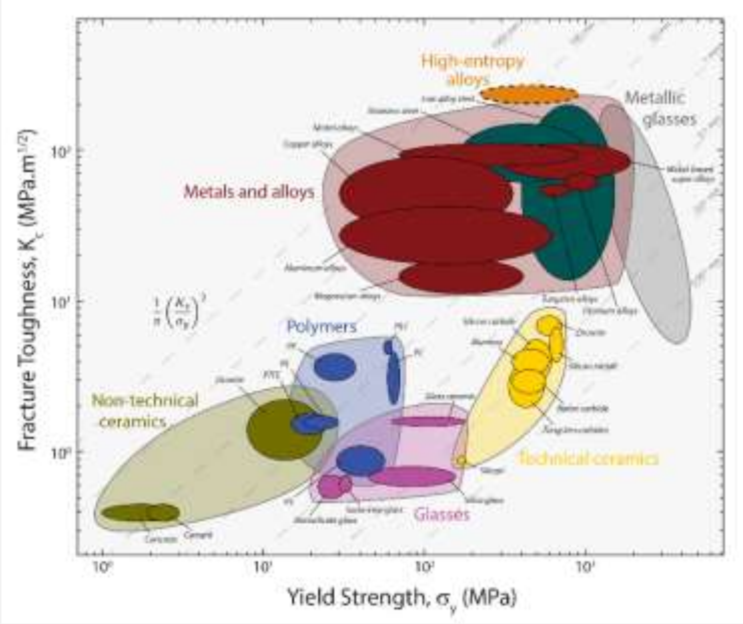
$$J_i = \frac{1}{d_n} \sigma_0 COD_i$$

$$J_{Ic} = K_{JIc}^2 \;/\; E'$$



	293K	**77K**
$CTOD_i$	57 ± 19 μm	49 ± 13 μm
σ_y	410 MPa	759 MPa
σ_{UTS}	763 MPa	1280 MPa
J_i	195 kJ/m²	219 kJ/m²
K_i (Δa → 0)	191 MPa.m$^{1/2}$	203 MPa.m$^{1/2}$
K_{JIc} (Δa = 200 μm)	217 MPa.m$^{1/2}$	219 MPa.m$^{1/2}$
K_{ss} (stable crack growth)	>300 MPa.m$^{1/2}$	>300 MPa.m$^{1/2}$

Ashby map (strength vs. toughness) & Conclusions



- **High-Entropy Alloys**
  → new aspect of metallurgy in the quest for new materials with interesting properties

- **CoCrFeMnNi single-phase fcc alloy**
  → excellent damage-tolerance properties which don't degrade at cryogenic temperatures

- **@ 293K:** σ_{UTS} ~ 763 MPa
  K_{JIc} = 217 MPa.m$^{1/2}$ (K_i = 191 MPa.m$^{1/2}$)
  deformation by *planar dislocation slip*

- **@ 77K:** σ_{UTS} ~ 1280 MPa
  K_{JIc} = 219 MPa.m$^{1/2}$ (K_i = 203 MPa.m$^{1/2}$)
  deformation associated with *nanoscale deformation twinning*

- **Toughness** → associated with continuous steady hardening (n ~ 0.4)
  → suppressing plastic instability & localization
  → appears to be a characteristic of plastic deformation by twinning

- !!! σ_{UTS} ~ 1.3 GPa & K_{JIc} ~ 220 MPa$\sqrt{m}$ @ 77K → extremely damage-tolerant !!!
  (properties exceed those of many materials including many austenitic stainless steels)