Micro-Mechanical Testing For Nuclear Applications

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Introduction

Introduction

Why Micro-mechanical Testing?

Basics

FIB Machining

Nanoindentation

Micro-mechanical Testing Case Studies

Stress – Strain Responses and Size Effects

Measuring Properties of Grain Boundaries

Conclusions

The People

Steve Roberts
Angus Wilkinson
Davide Di Maio
Jicheng Gong
Ben Britton
Fiona Haliday
Mike Rogers
Will Herbert
Lawrence Whyatt
James Robinson
Ele Grieveson
James Gibson

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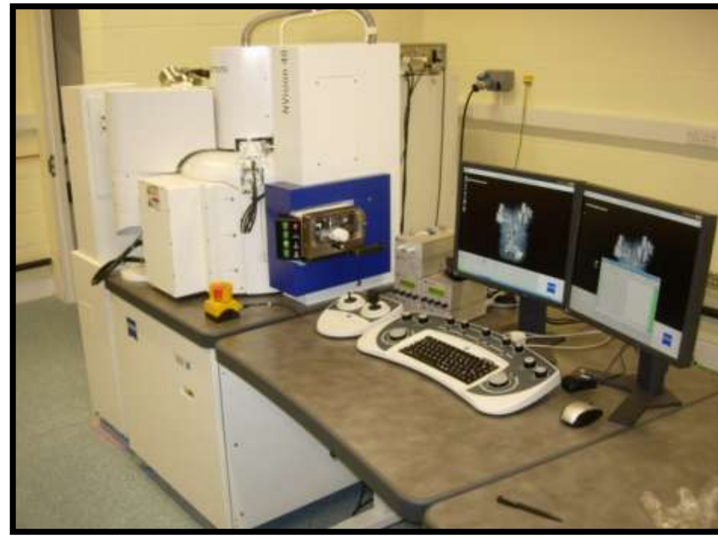
Micro-mechanical testing

- **Recently developed testing techniques**
- **Utilizes Focused Ion Beam (FIB) machining and nanoindentation**
- **Allows manufacture of samples with well-defined stress states**
- **Allows fracture properties, yield strengths and elastic properties to be measured**
- **Temperature variation now available**



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Why use micro-mechanical testing?

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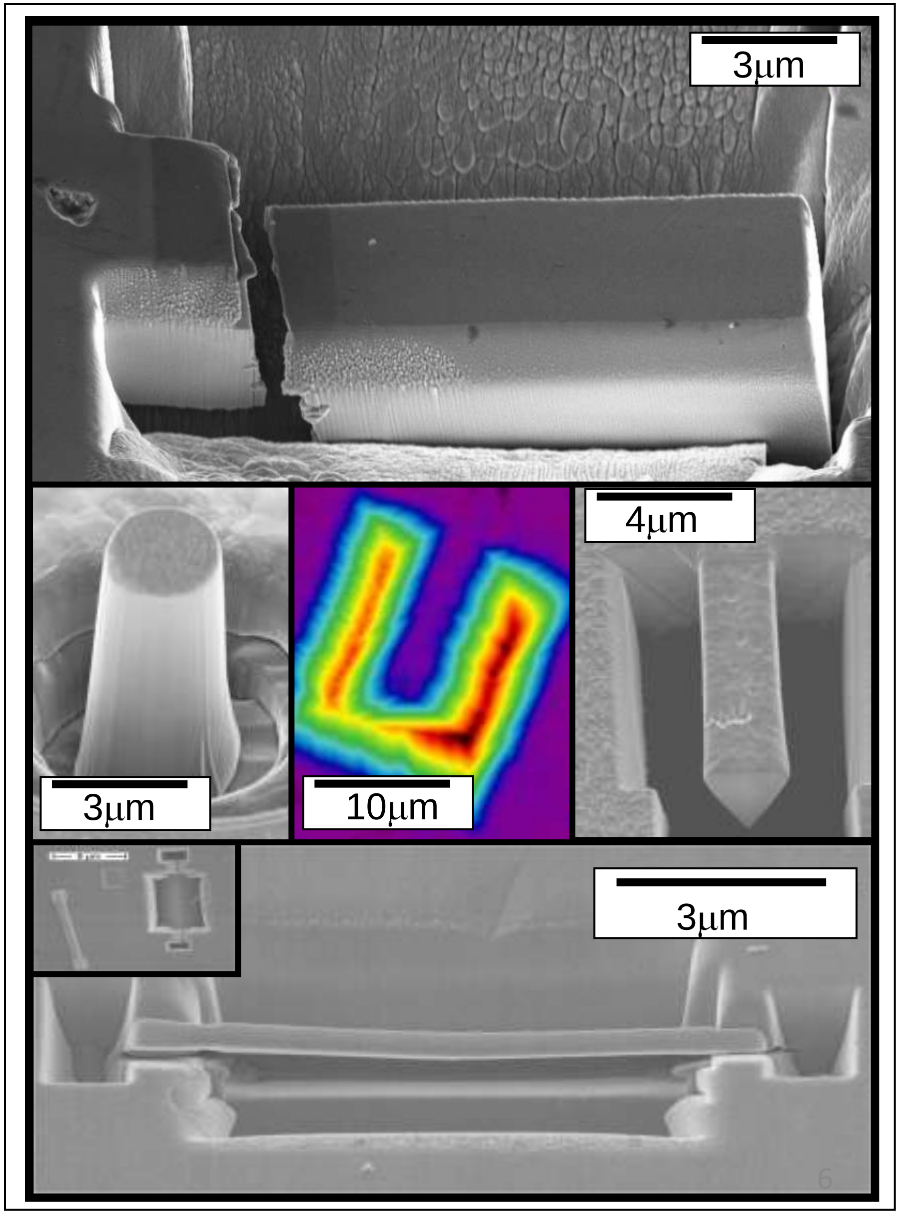
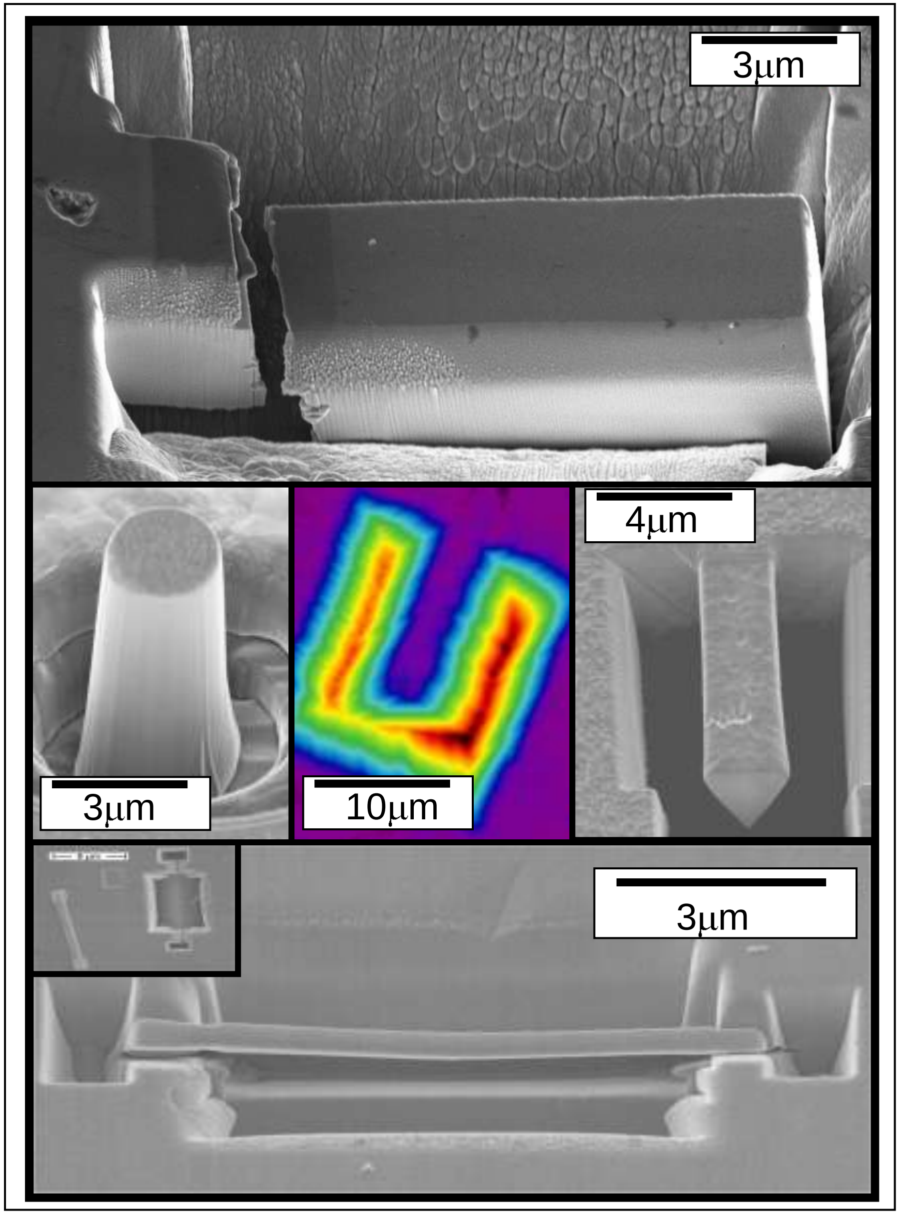
- Useful where only small samples are available
  - Cost
  - Processing
- Need for a sample design that can be machined in surface of bulk samples
- Suitable for measuring individual microstructural features
- Samples that can be manufactured quickly and reproducibly



Types of micro-mechanical testing?

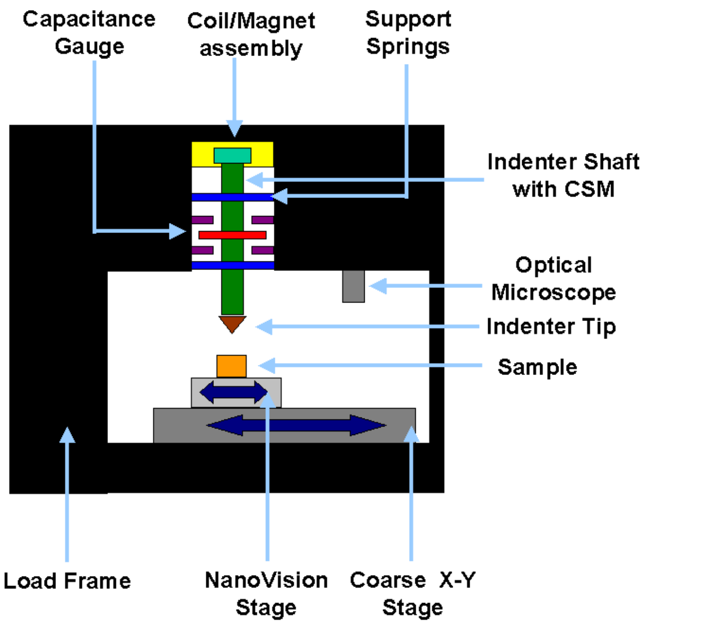
- Electro-deposition
- Selectively etched
- FIB machined

- Compression
- Tension
- Three Point Bend
- Cantilever bending



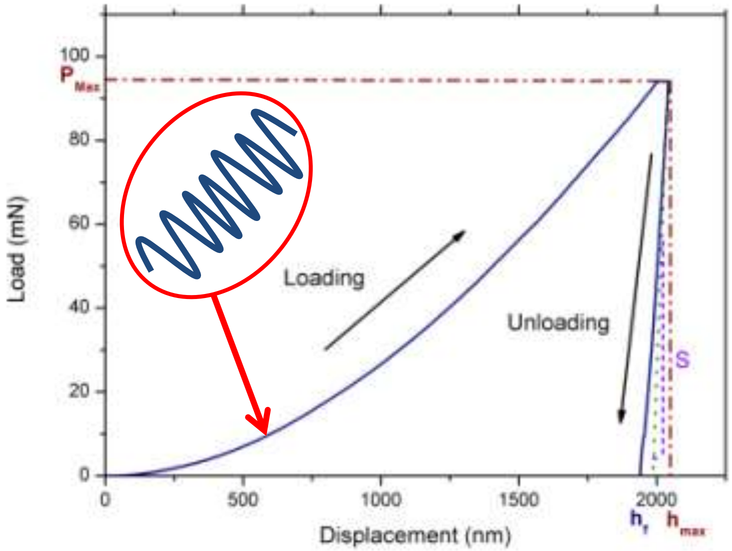
Nanoindentation

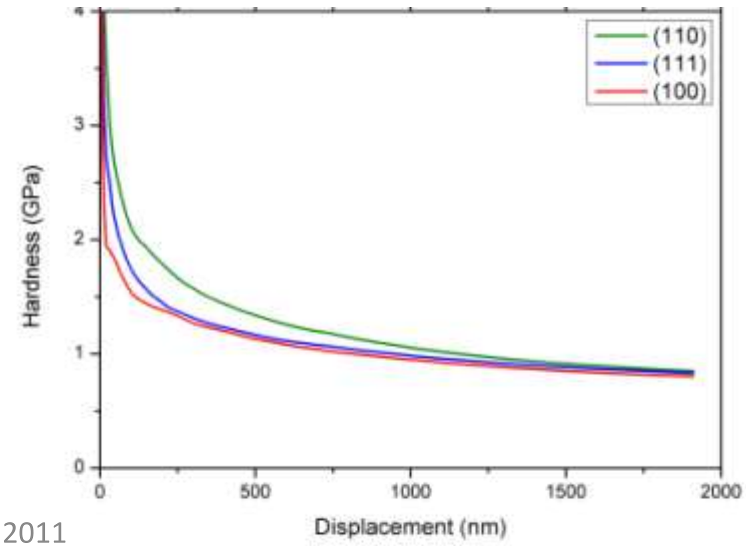
- Nanoindentation mechanical probe which allows local hardness and modulus to be measured
- A sharp diamond is driven into the surface with a known force
- Displacement is measured using a capacitance gauge
- Sharp tip can also be used as a surface profilometer tool
- Also very useful to deform and test specimens



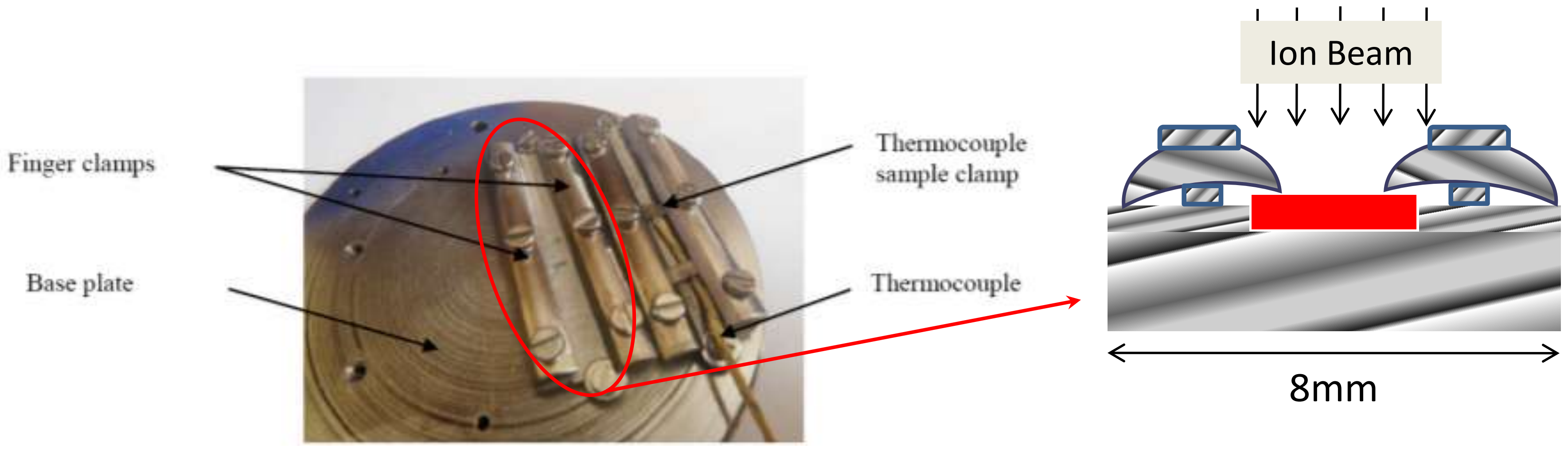
Nanoindentation

- **By knowing the contact area between sample and indenter hardness and modulus from unload can be calculated**
- **A small ac sinusoidal can also be placed on the load**
- **This Continuous Stiffness Measurement (CSM) allows the modulus and hardness to be continually measured as a function of depth**





Nanoindentation



See talk on Wednesday for selected results on nanoindentation of ion implanted surfaces in tungsten and tungsten alloys

Focused Ion Beam Machining

- **FIB uses gallium ion (Ga+) to “knock” atoms out of the sample being machined**
- **Ions focused on surface of sample using electromagnetic lens (similar to SEM)**
- **Beam currents from 1pA to 45nA allow features as small as 5nm to be machined**
- **Also allows deposition of Pt/W/C**

Focused Ion Beam Machining

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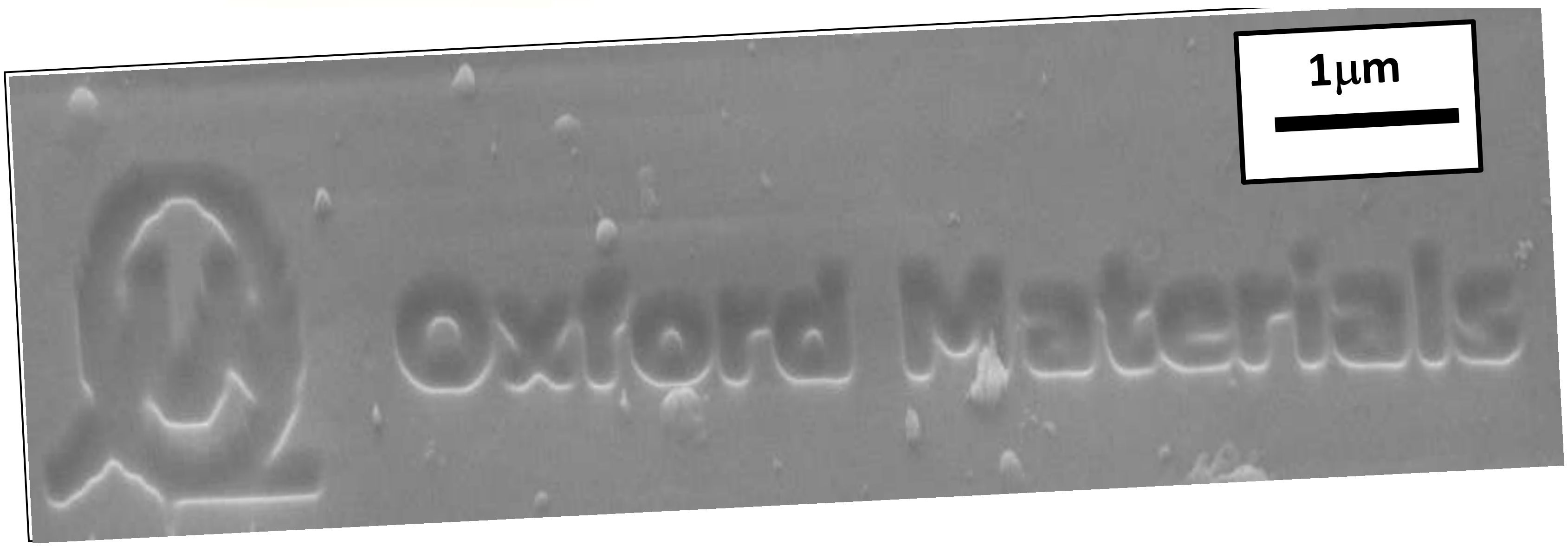

Microcantilever Manufacture

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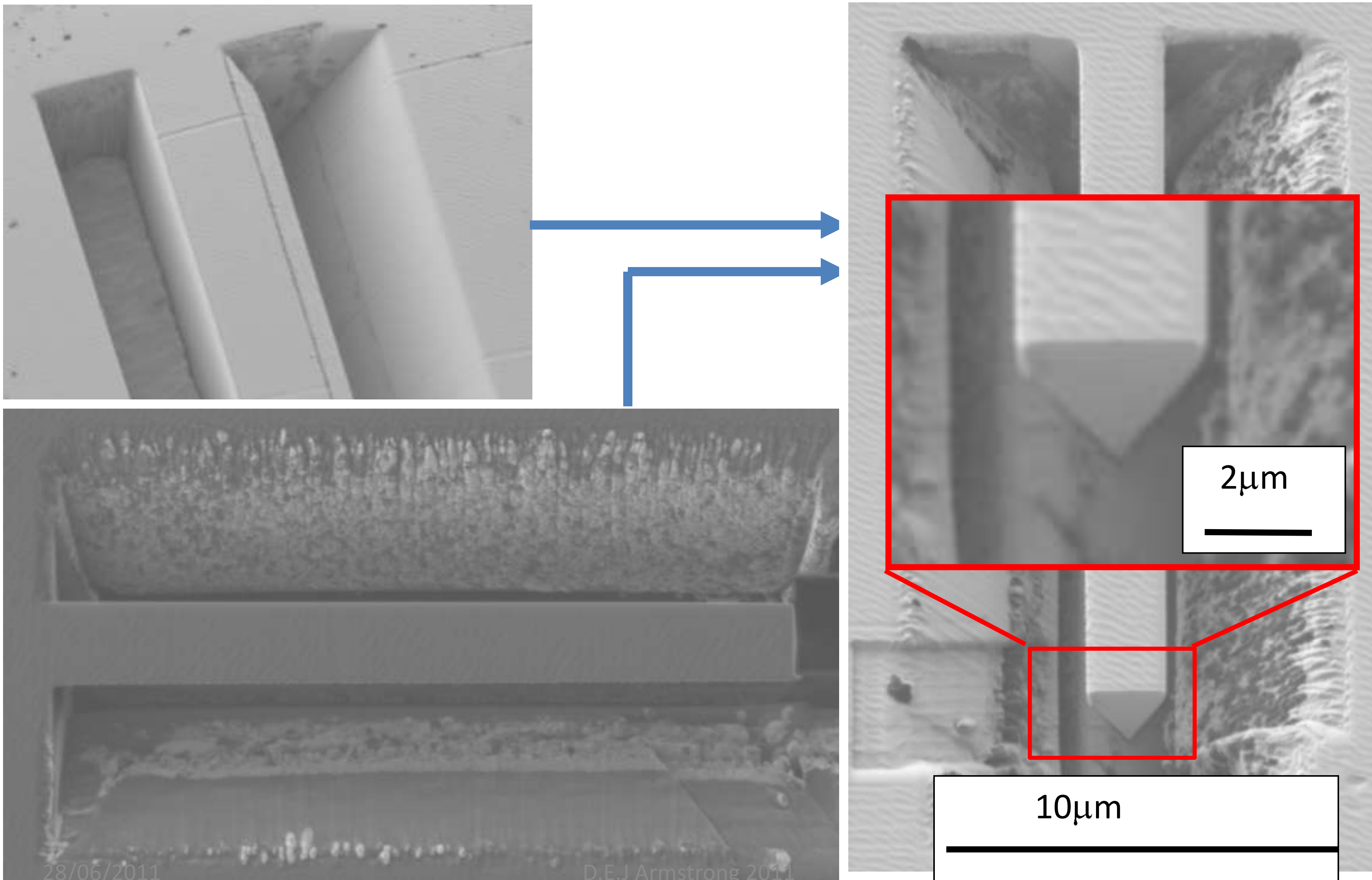
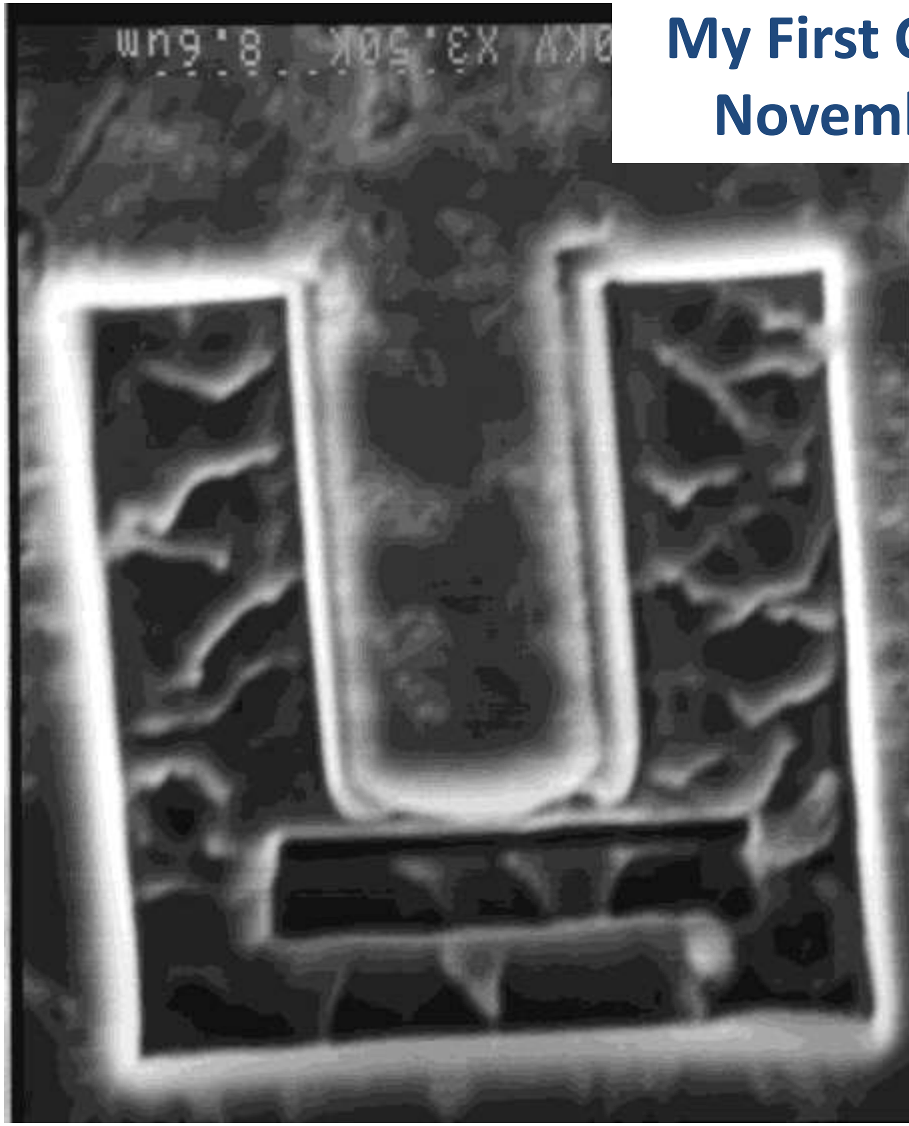
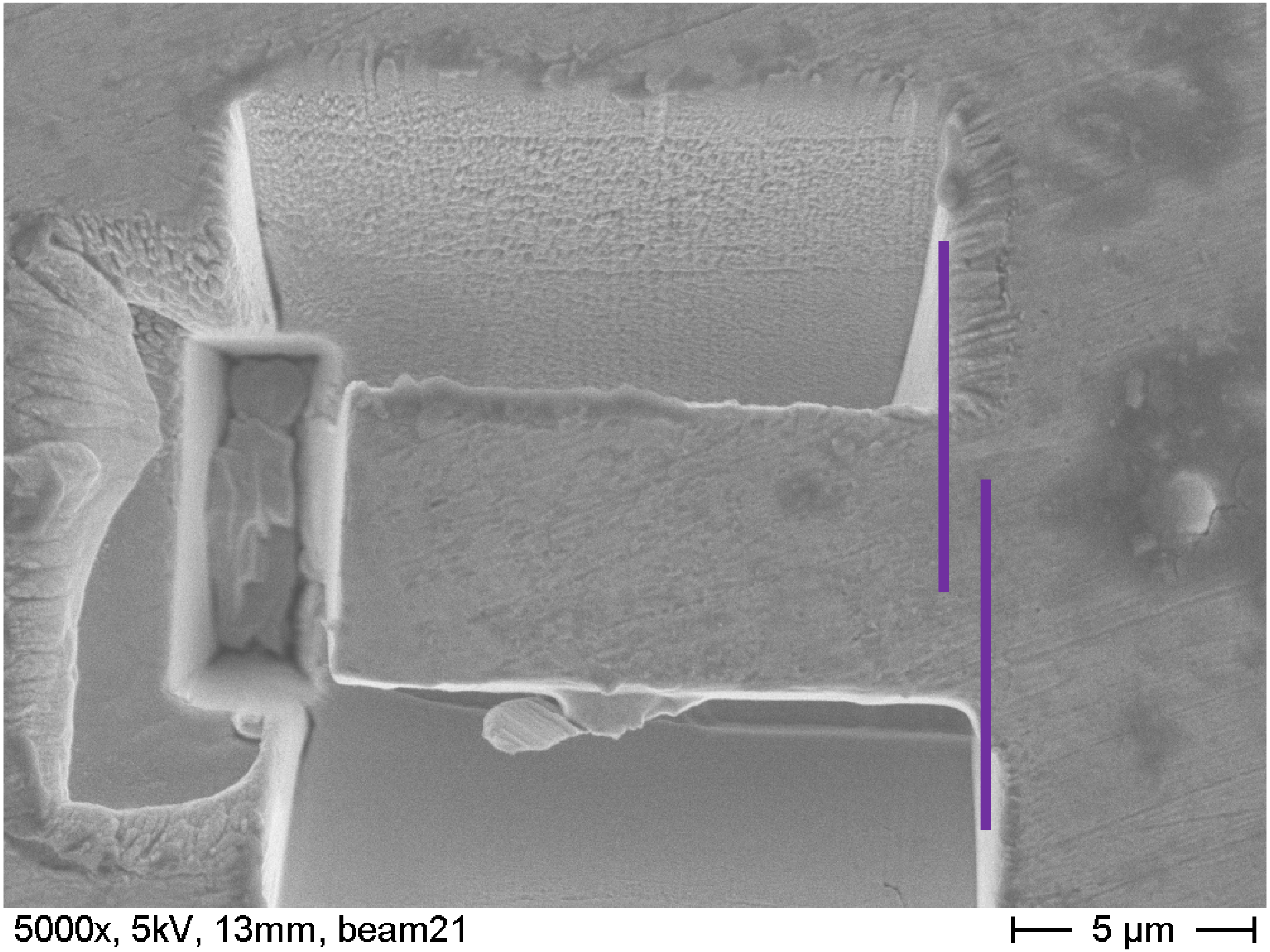


When FIB goes wrong

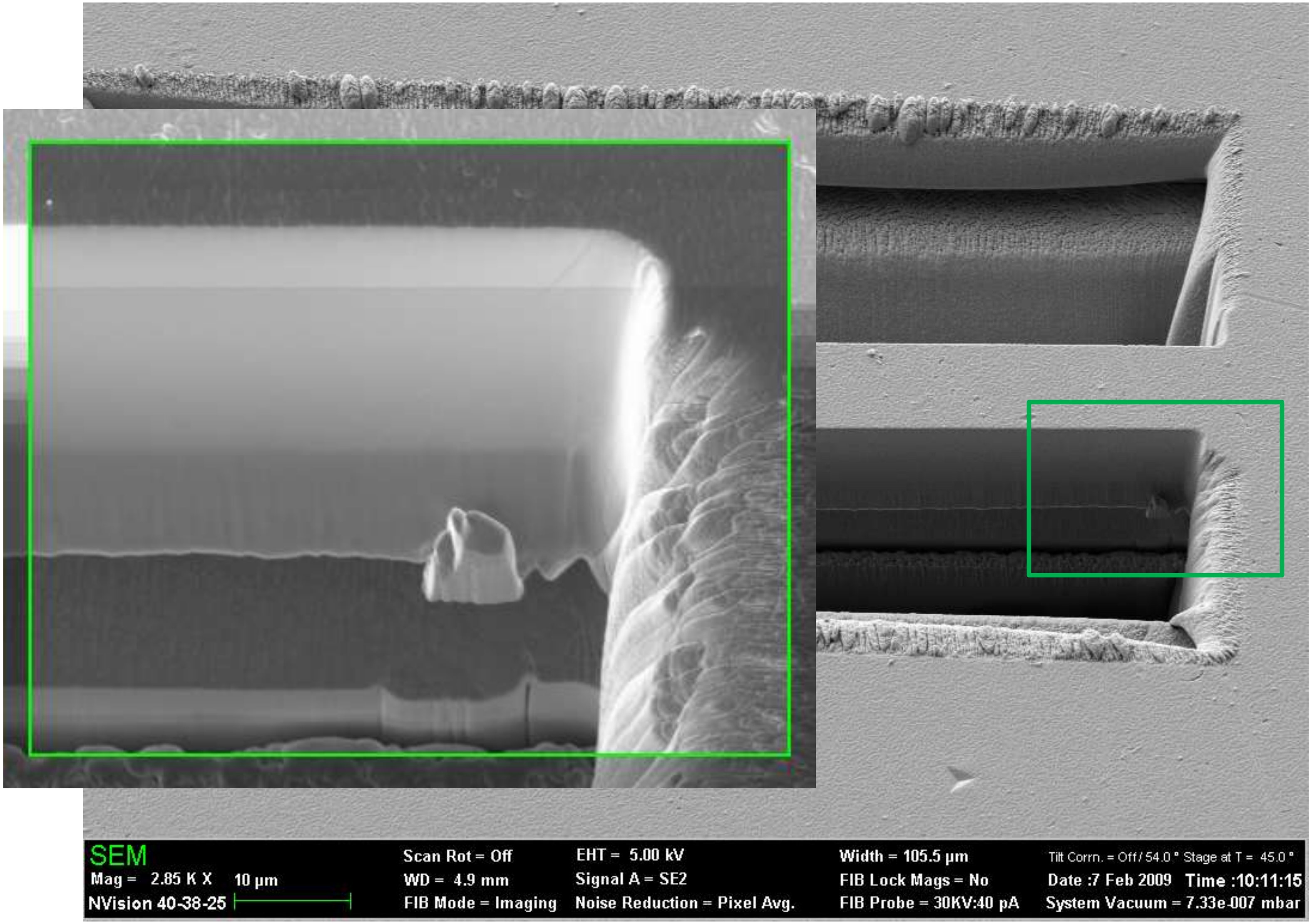
My First Cantilever-
November 2004



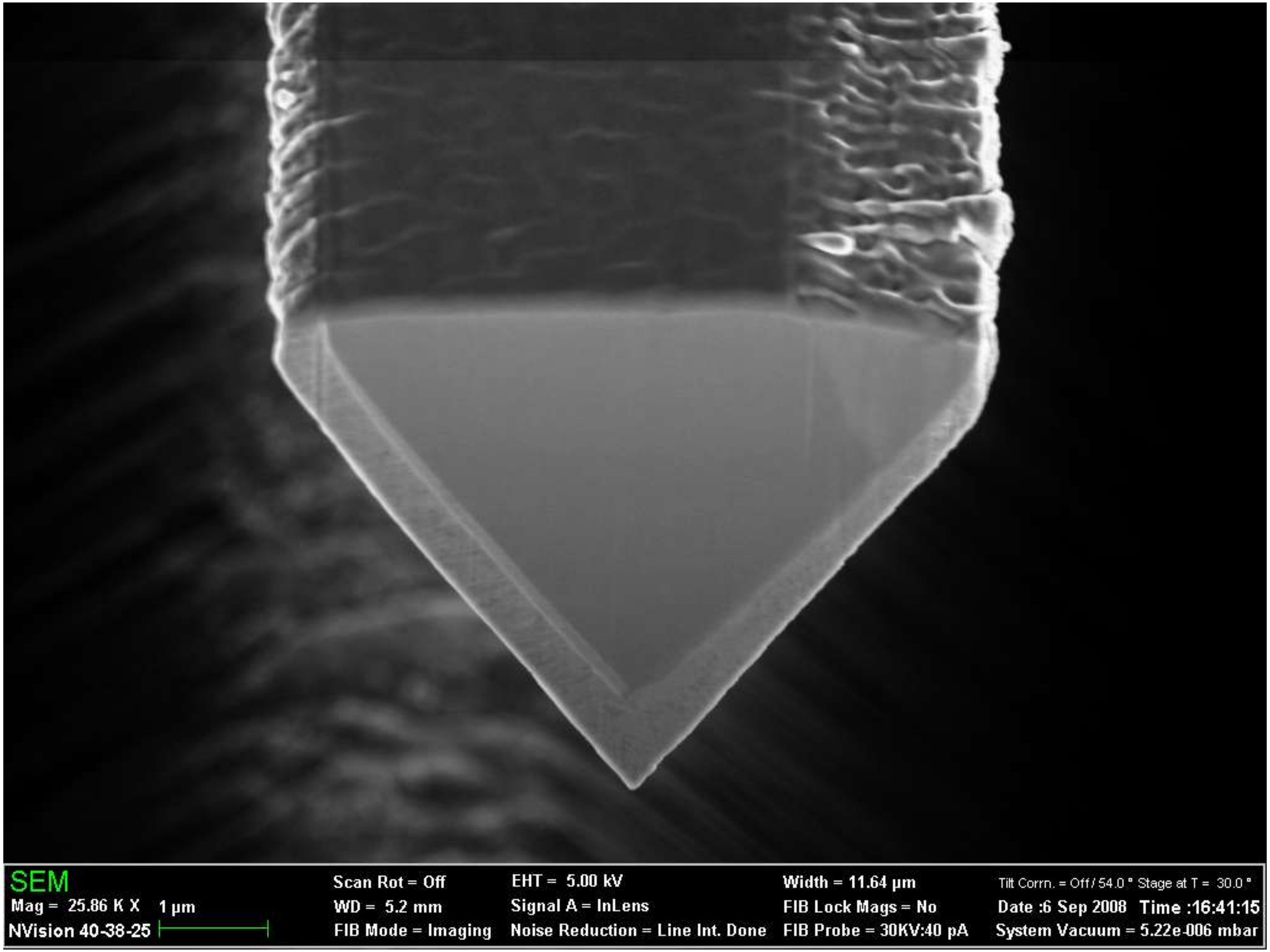
When FIB goes wrong



When FIB goes wrong



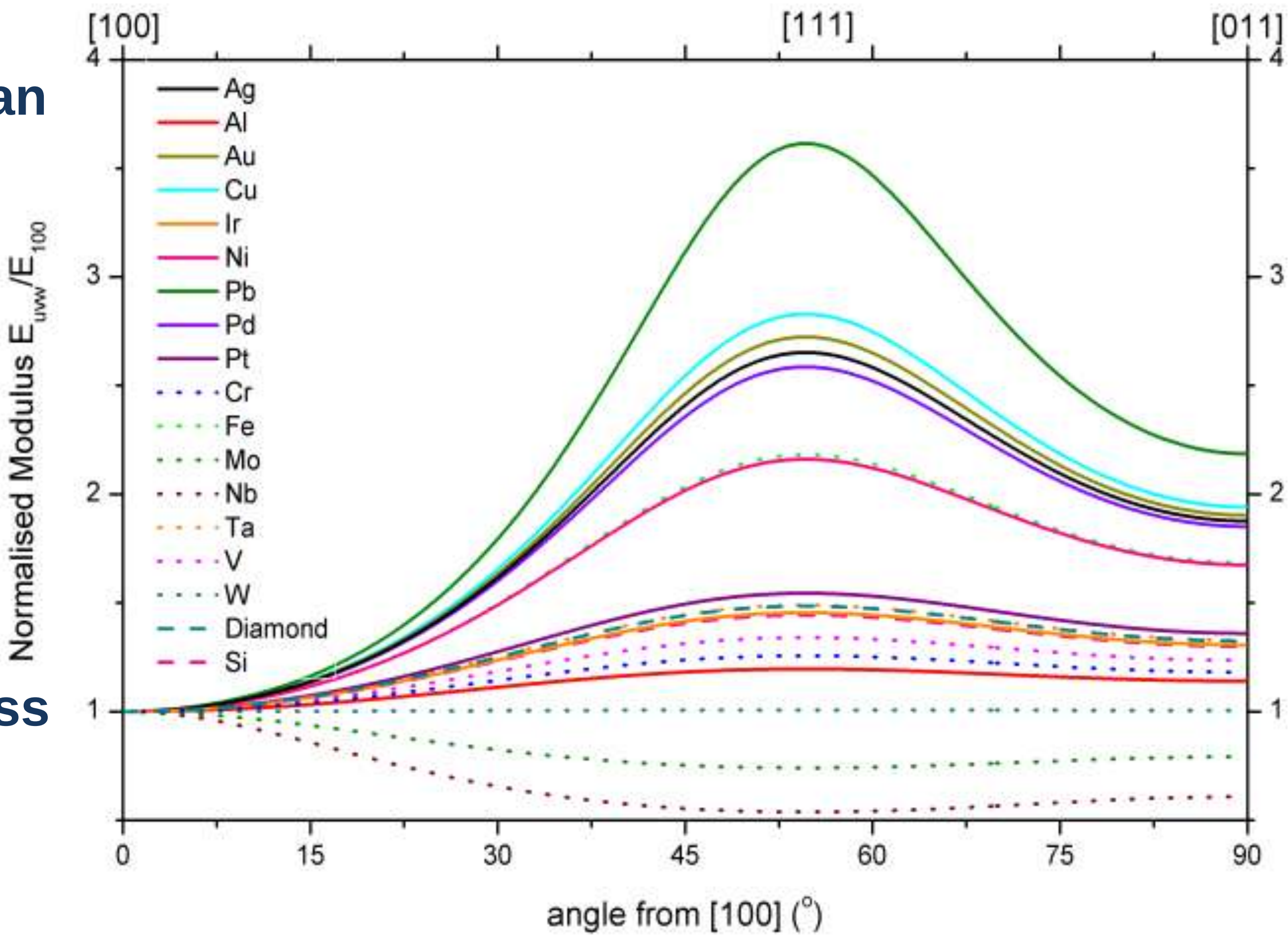
When FIB goes wrong



Case Study One: Elastic Anisotropy

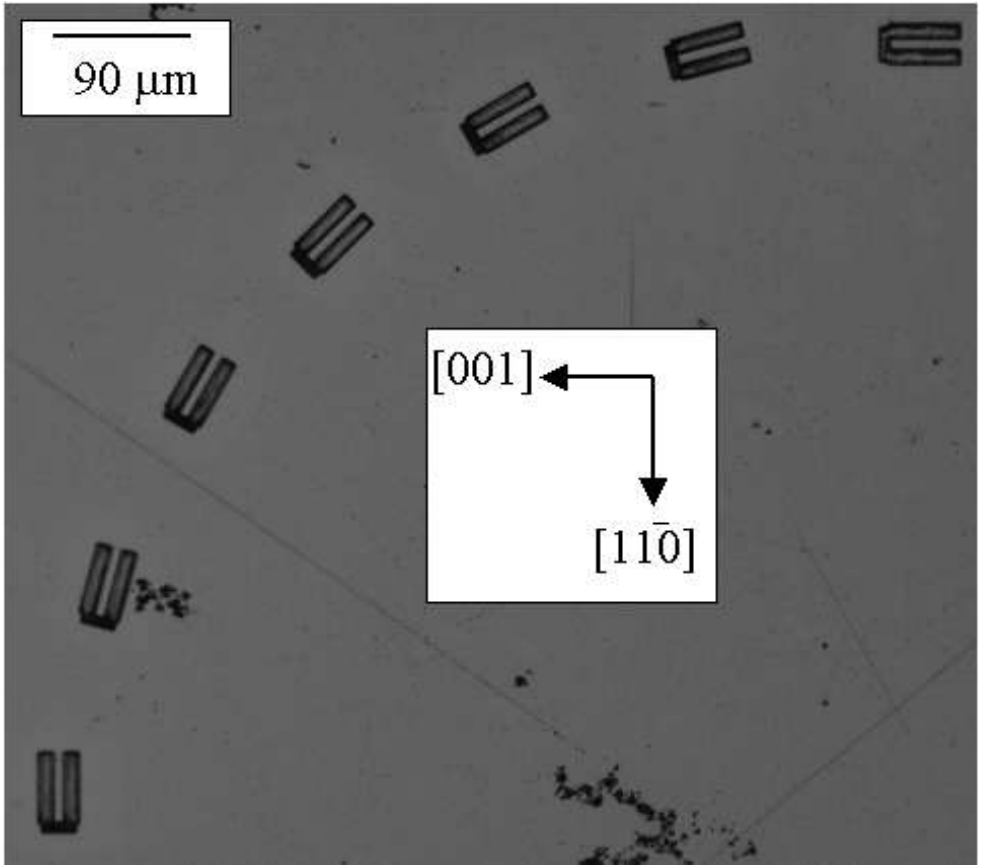
Measuring Elastic Anisotropy

- **Elastic properties can control deformation processes and important for engineering design**
- **Most materials display elastic anisotropy**
- **Difficult to measure experimentally unless large single crystal available**
- **Traditional techniques – static or dynamic require large (mm to cm) samples**



Elastic Anisotropy In Copper

- **Copper: highly anisotropic well characterised material**
- **Should be an "easy" starting material**
- **Cantilevers manufactured in single crystal sample at 15º intervals between [100] and [110] directions**
- **Cantilevers scanned using "nano-vision" stage to produce topographical image**



Multiple loading method

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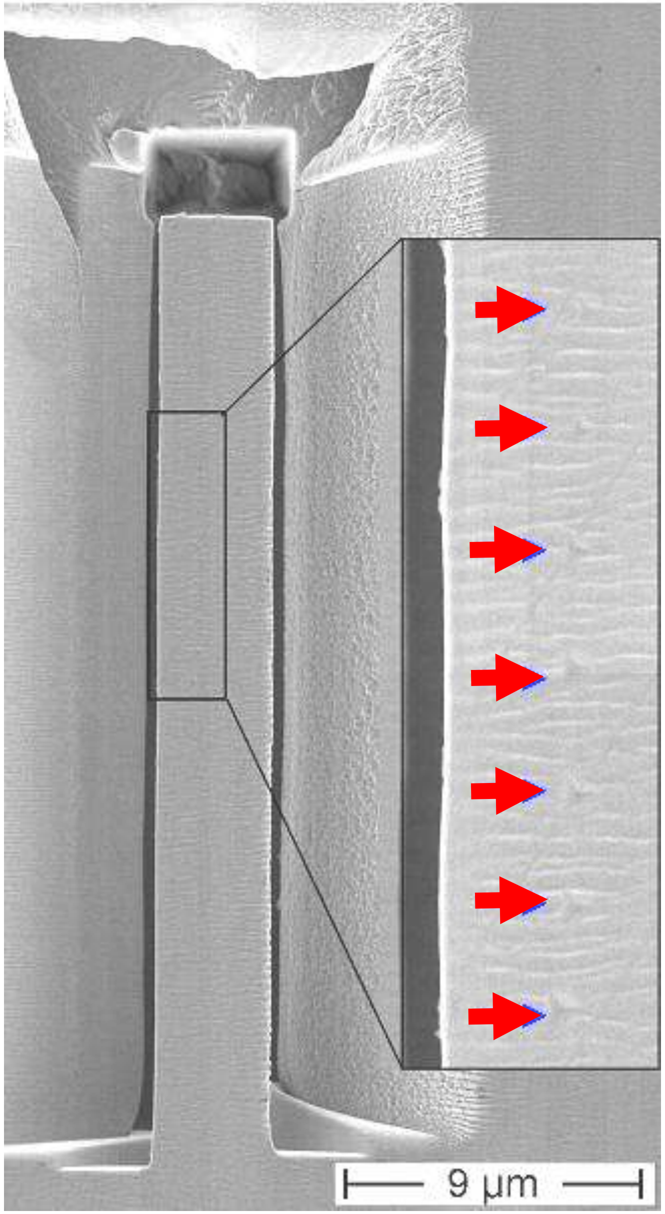
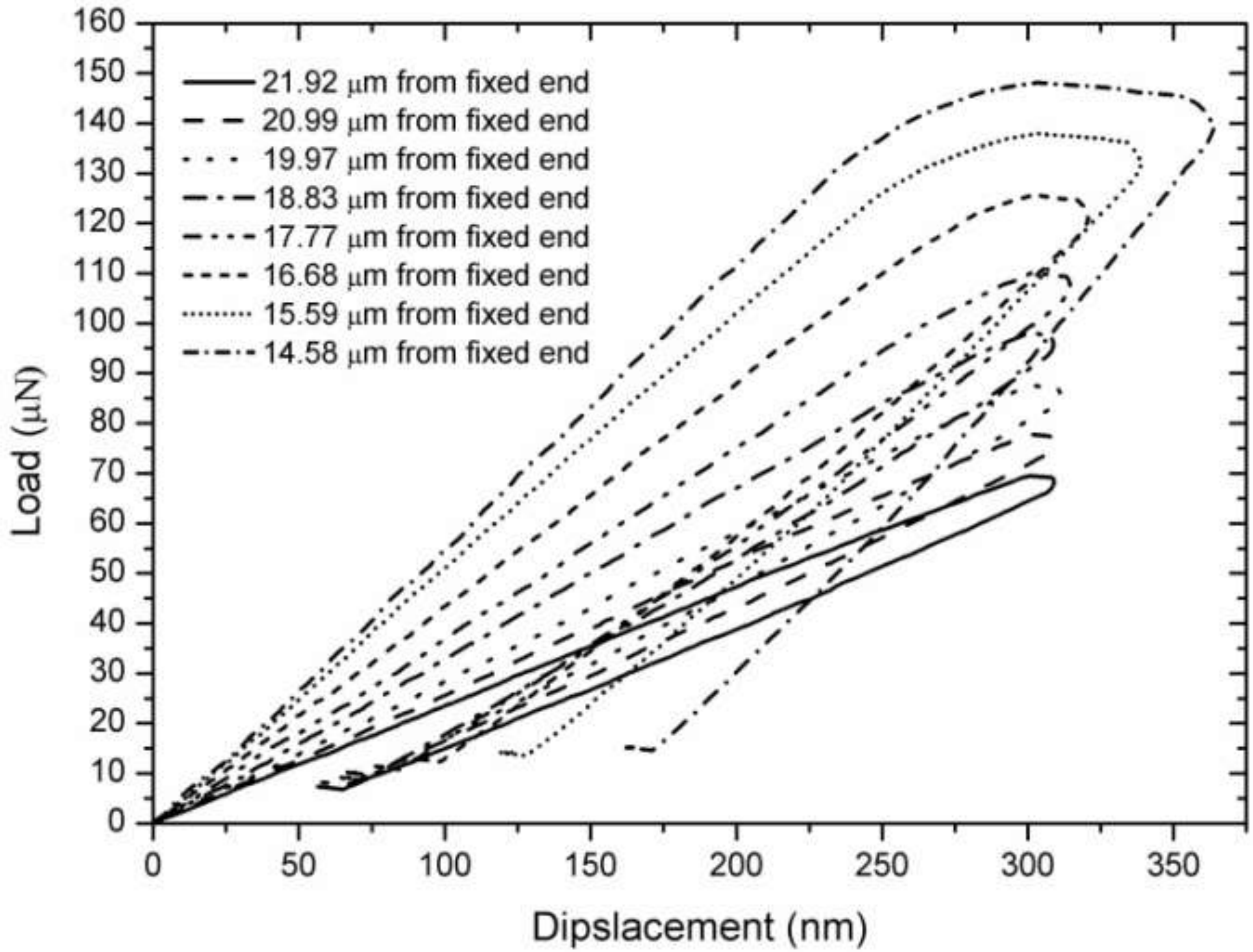
- Longer, thinner cantilevers
- Cantilever loaded using nanoindenter close to free end
- Each loading to 200nm (no yield)
- Indenter moved 700nm towards fixed end and cantilever loaded
- Repeated between 5 and 13 times
- Use unload data



Elastic Anisotropy In Copper



Analysis of Elastic Properties 1

- **From simple beam theory:**

$$\boxed{S = \frac{L^3}{3EI}}$$

- **But due to non-fixed end there is extra deflection at the fixed end:**

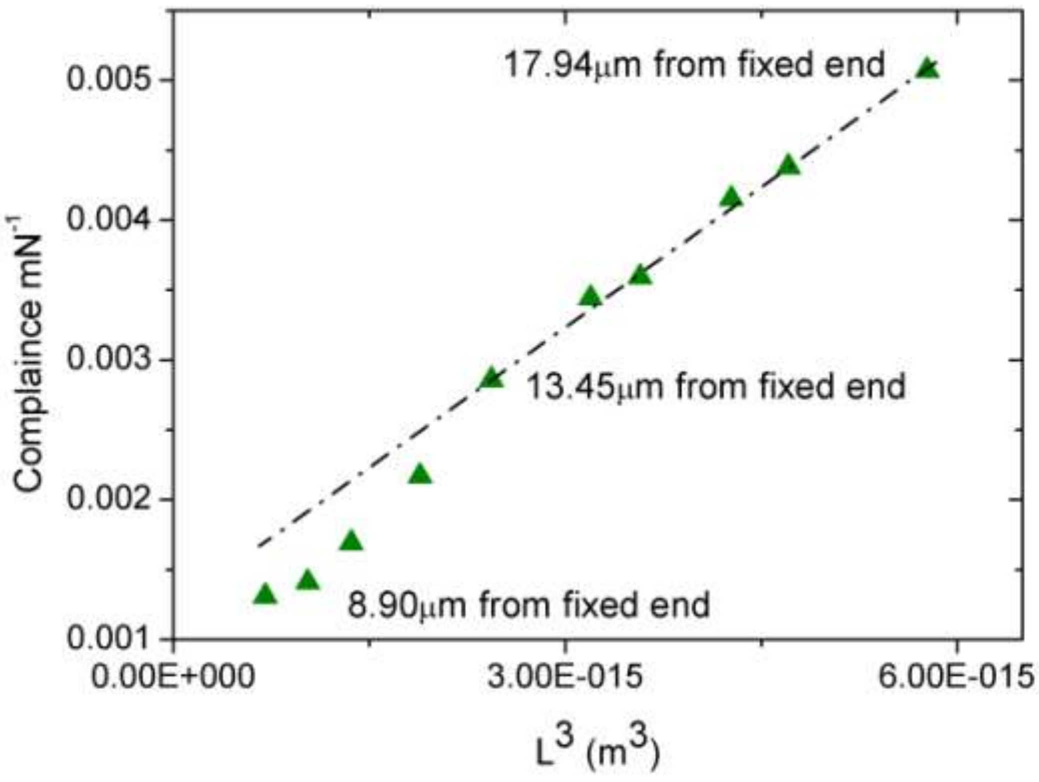
$$\boxed{\delta_L = \frac{PL^3}{3EI} + \theta_0 L + \delta_0}$$

- **Ignoring lower order terms beam compliance can be written as:**

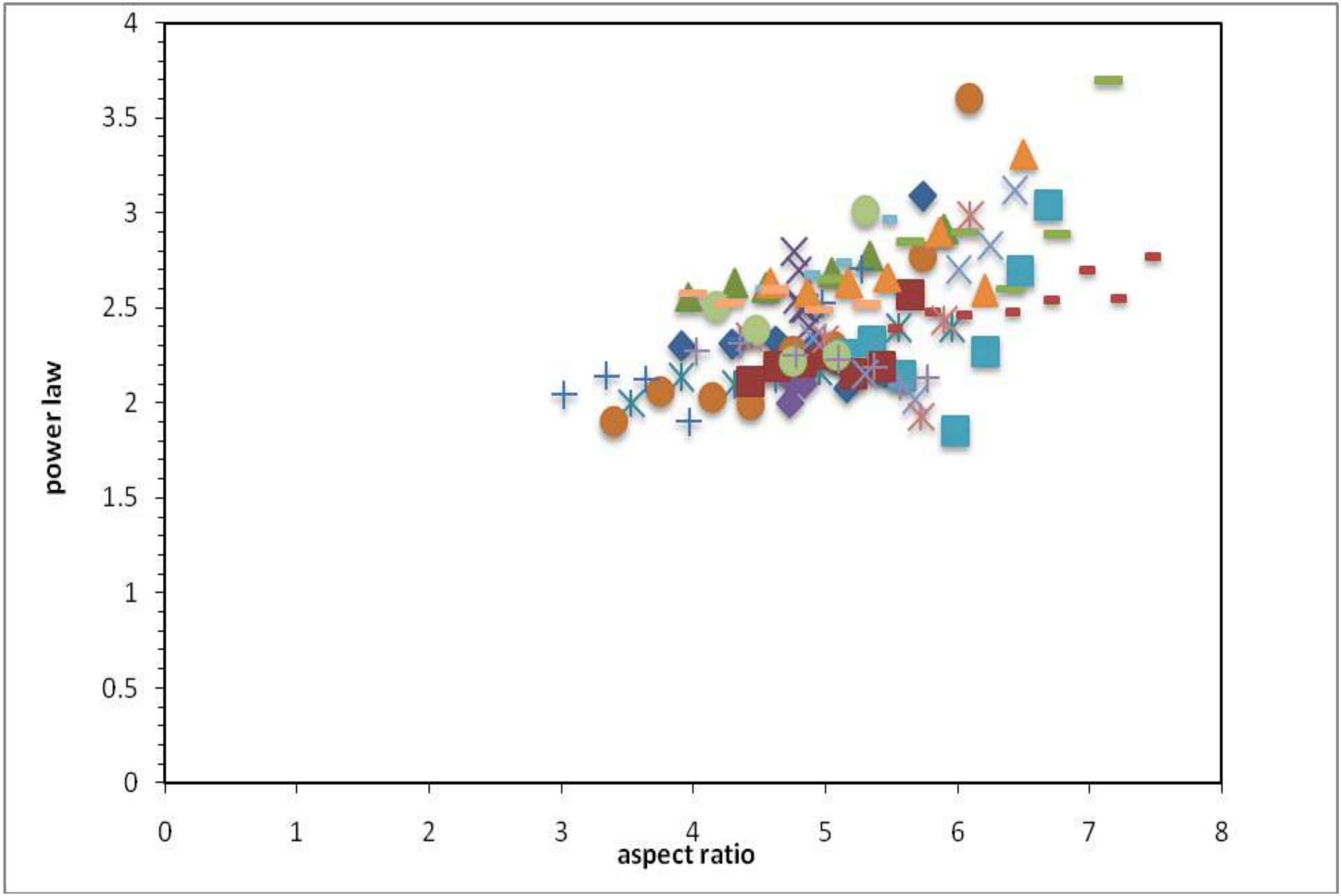
$$\boxed{S = \frac{1}{3EI}L^3 + \theta_{m0}L^2}$$

Analysis of Elastic Properties 2

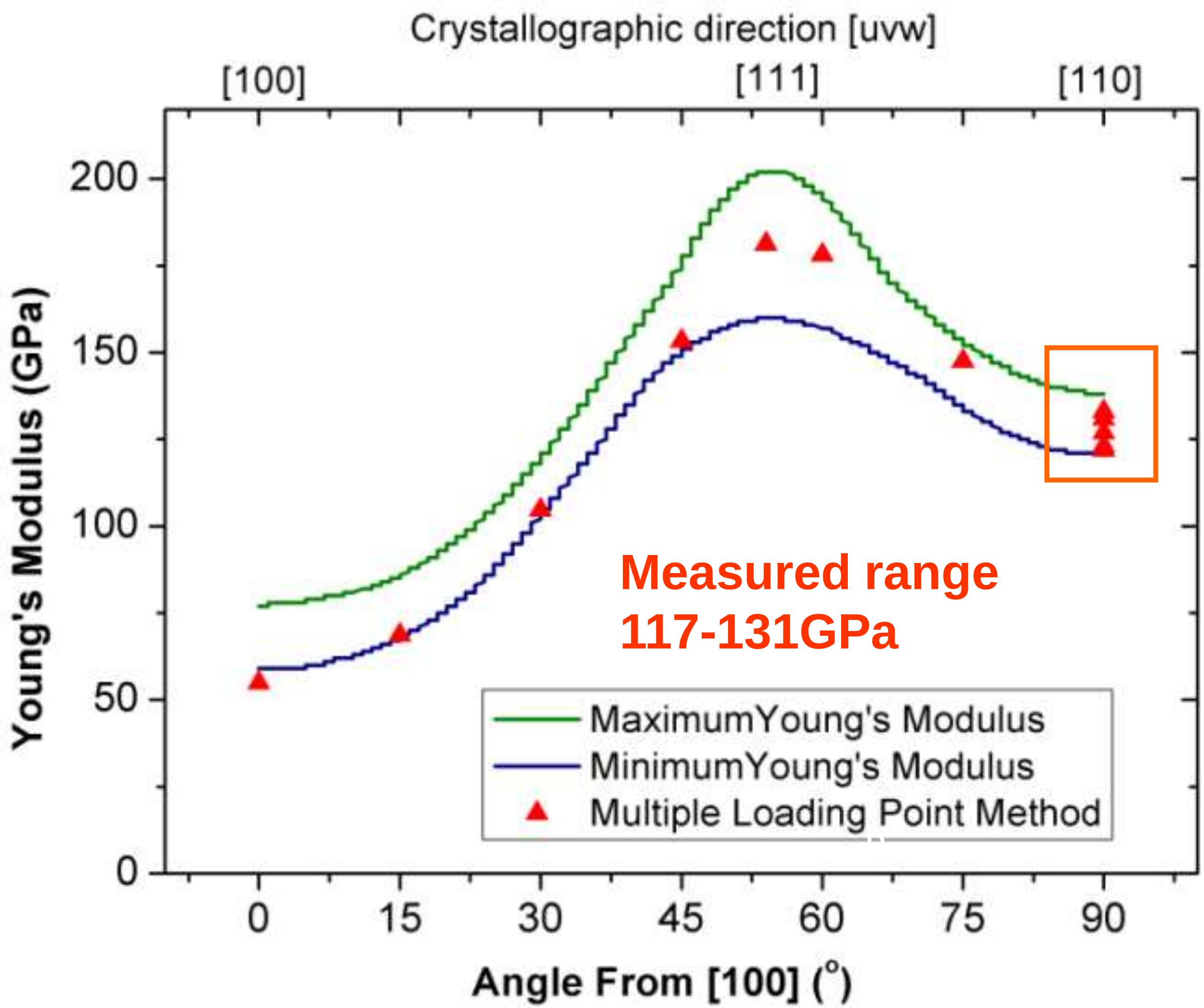
- **Plot of S versus L^3 shows linear relationship at larger values of L**
- **The gradient of this linear region can be used to find Young's modulus**
- **Analysis carried out on cantilevers at 15º intervals between [100] and [110]**
- **Found to give good results for aspect ratio greater than 6**

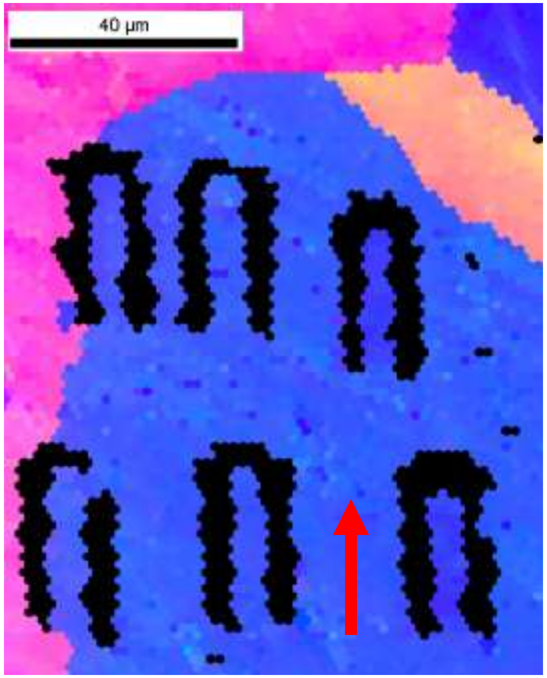


Analysis of Elastic Properties 3

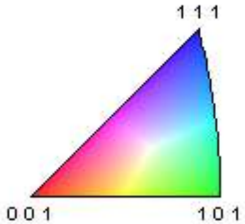


Elastic Anisotropy in Copper



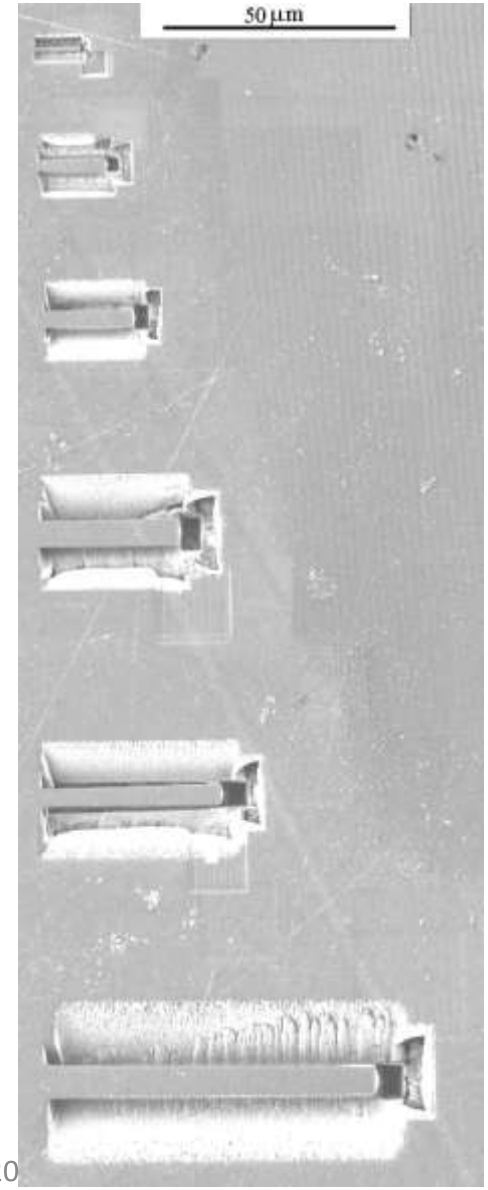


(7 8 9):[15 12 1]

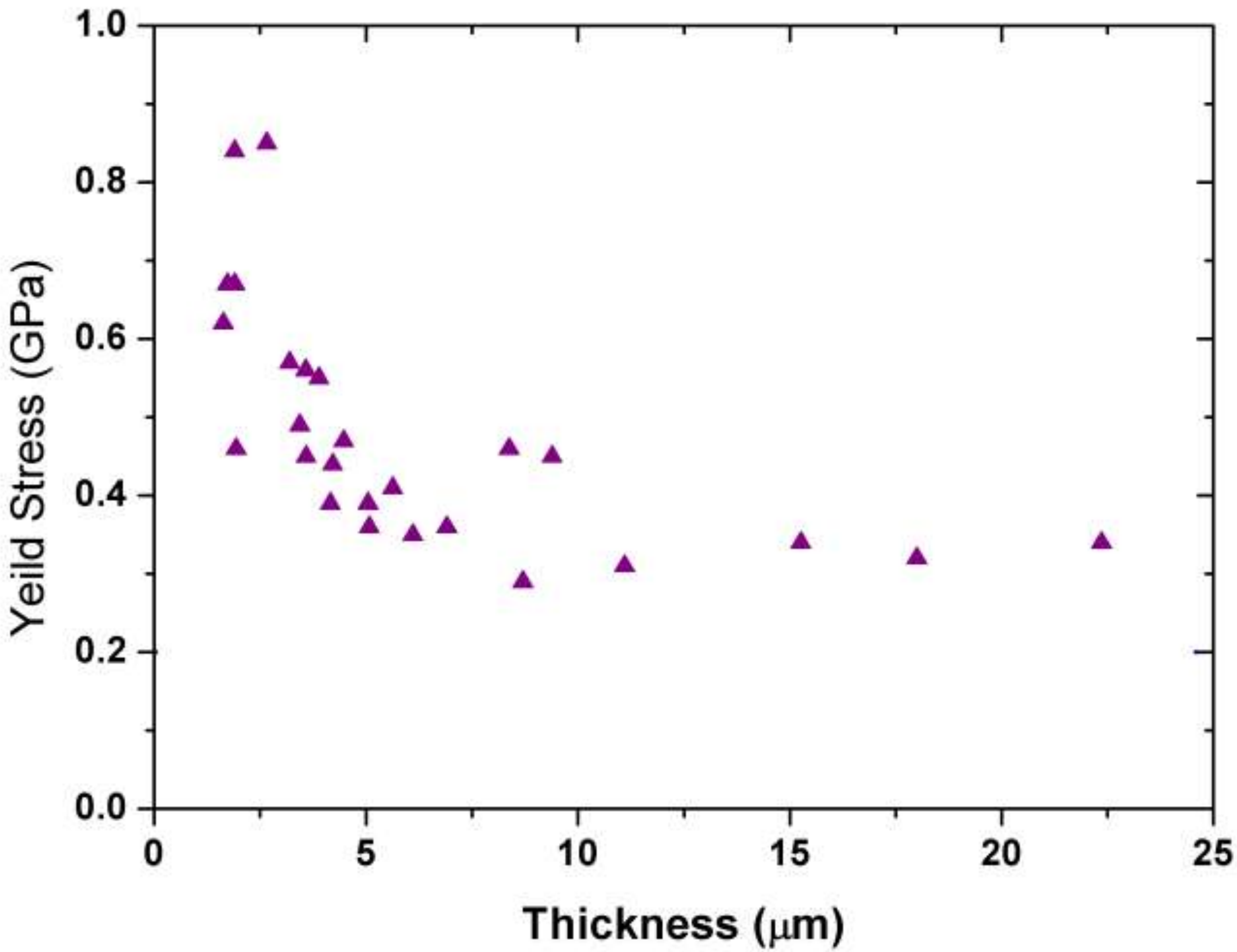


Size Effects on Yield Stress

- Well known that as specimen size decreased yield stress increases
- Exact form of this relationship in triangular microcantilevers unknown
- Cantilevers machined in single crystal copper with long axis in [110] direction
- Range in size from 1μm thick and 10μm long to 18μm thick and 100μm long
- Tested at constant displacement rate of 5nm/s
- Only smallest cantilevers can be used to study ion implanted layers - difficult



Size Effects on Yield Stress



FeCr Micro Pillars

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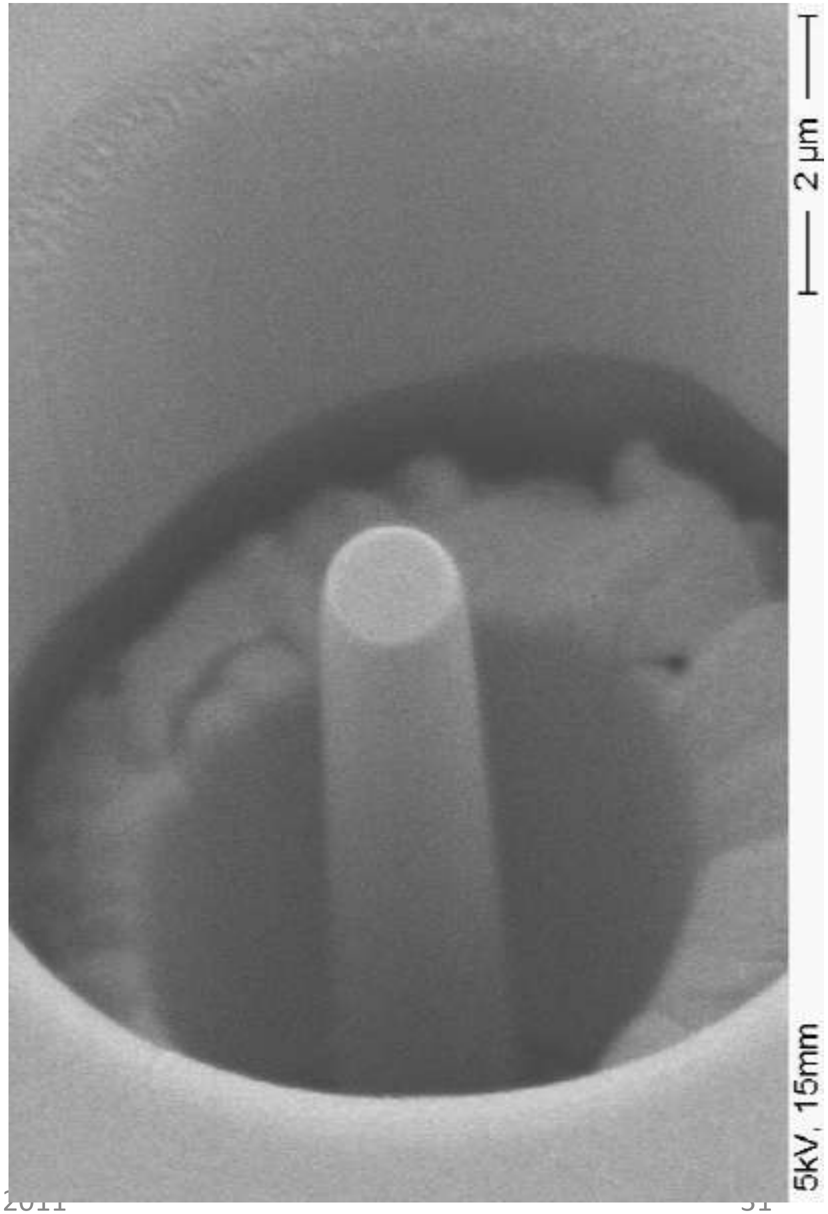
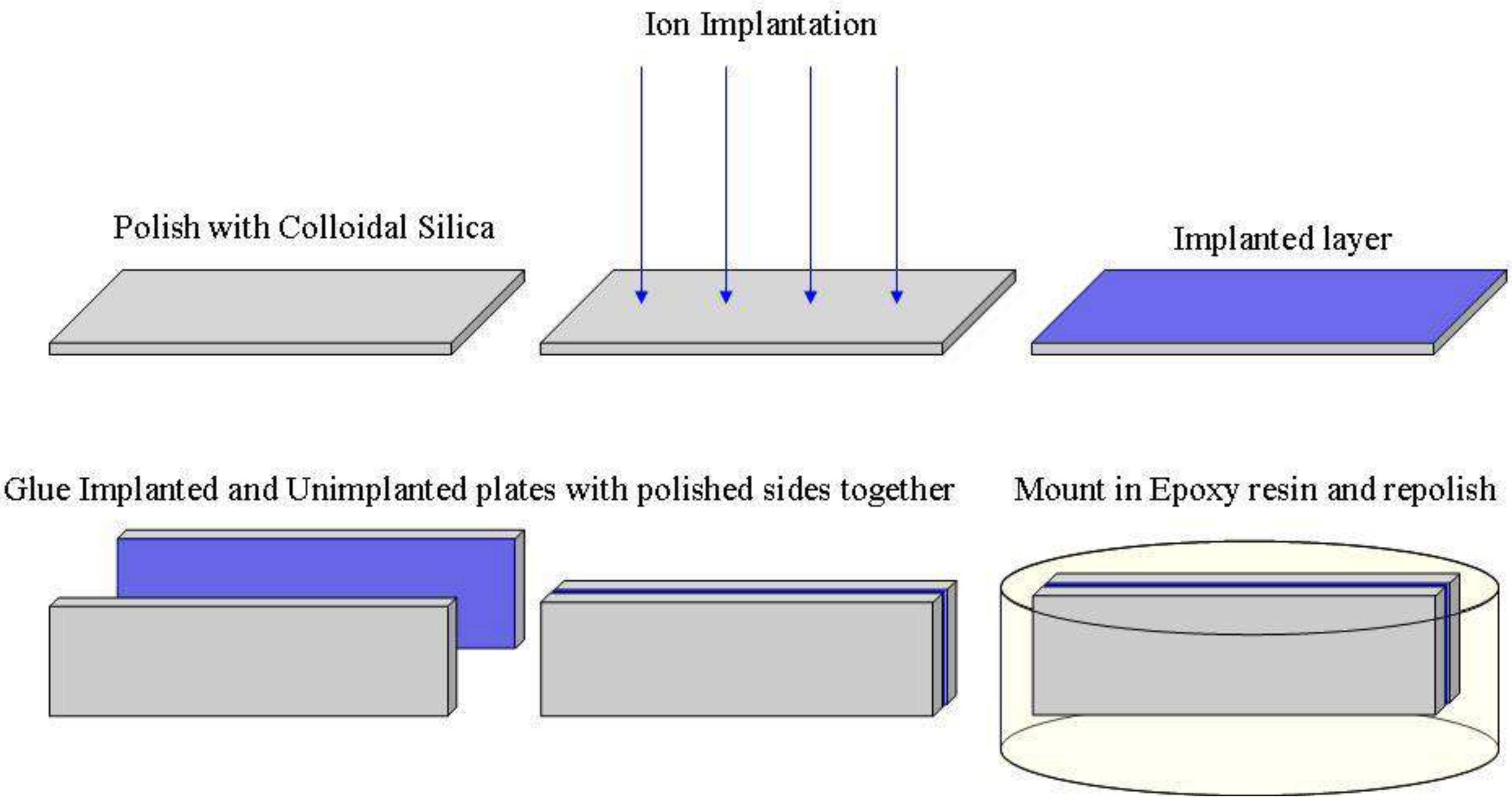
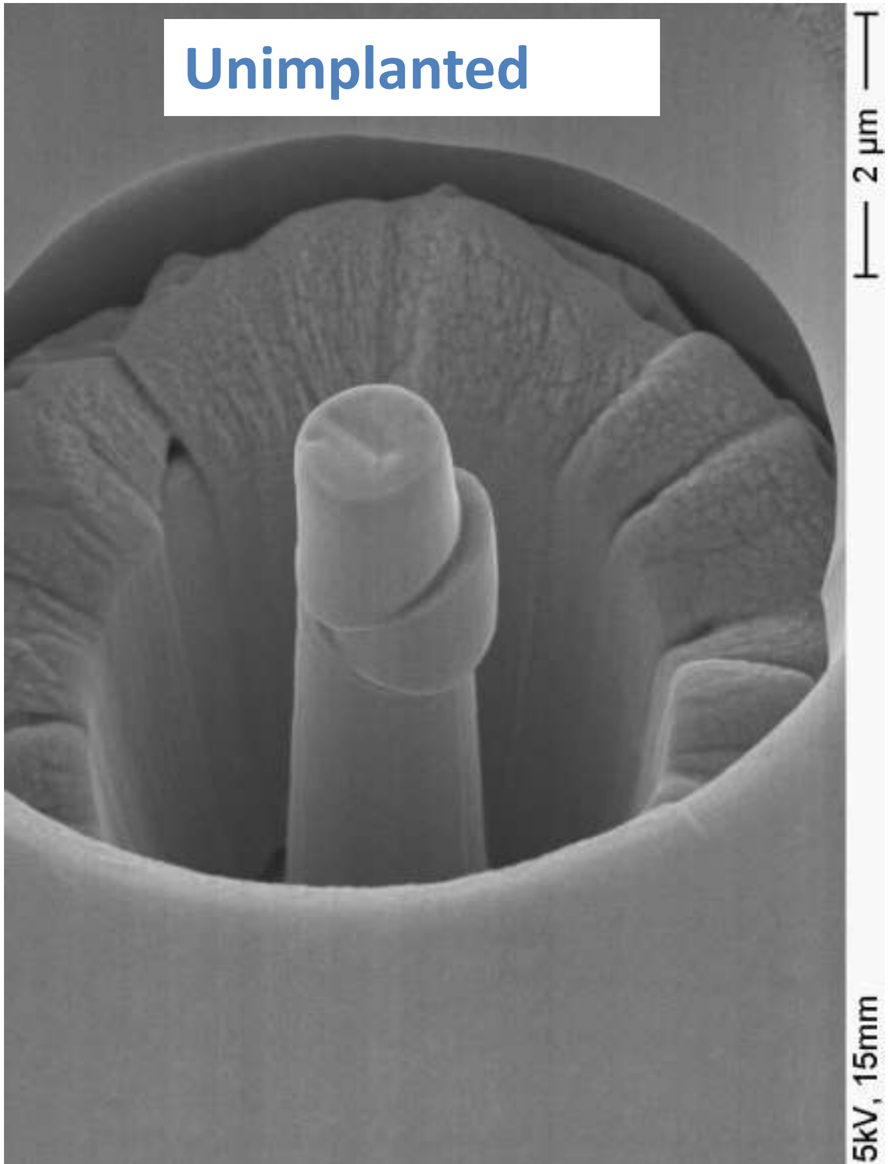
- **Pillars machined into the ion implanted layers, using multi stage approach**
- **Width approx 500nm**
- **Height 3μm**
- **Flat Punch type nanoindenter tip used to compress the pillars**

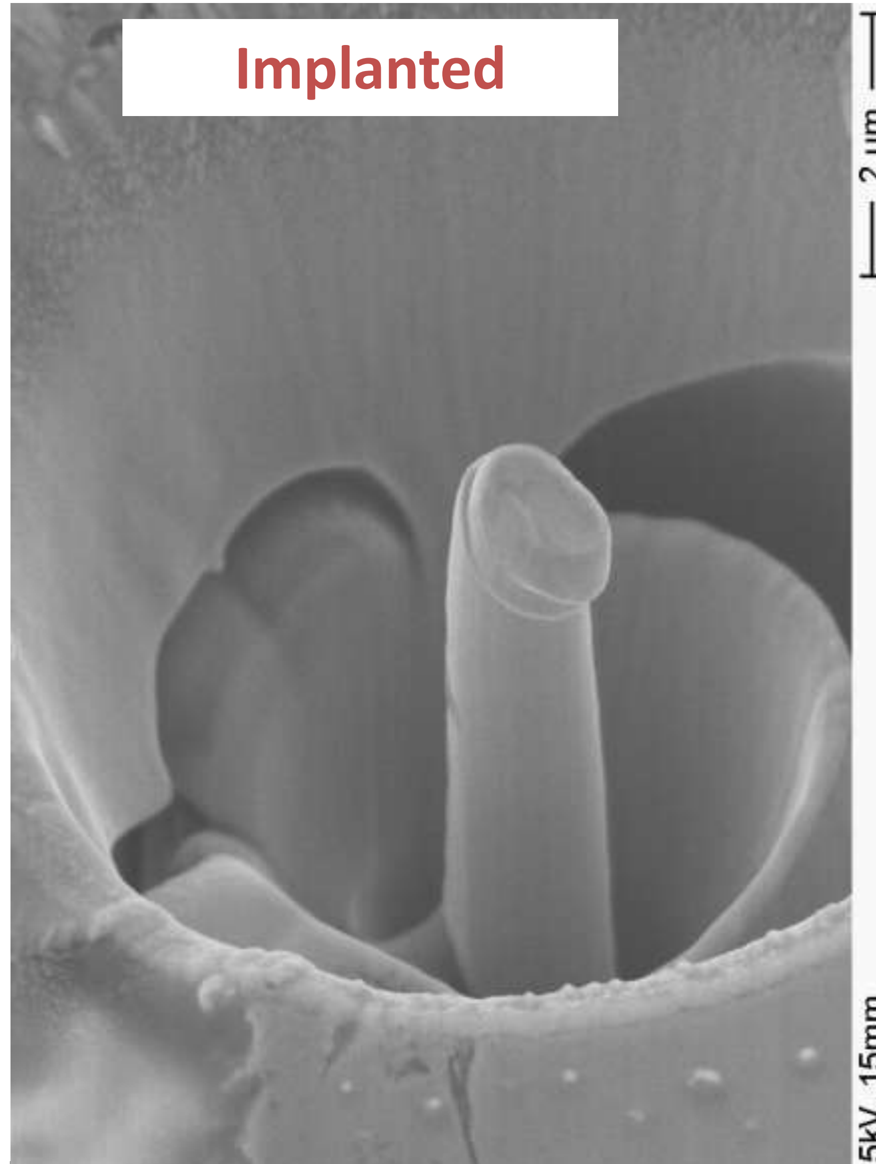


Yield Properties in FeCr Alloys

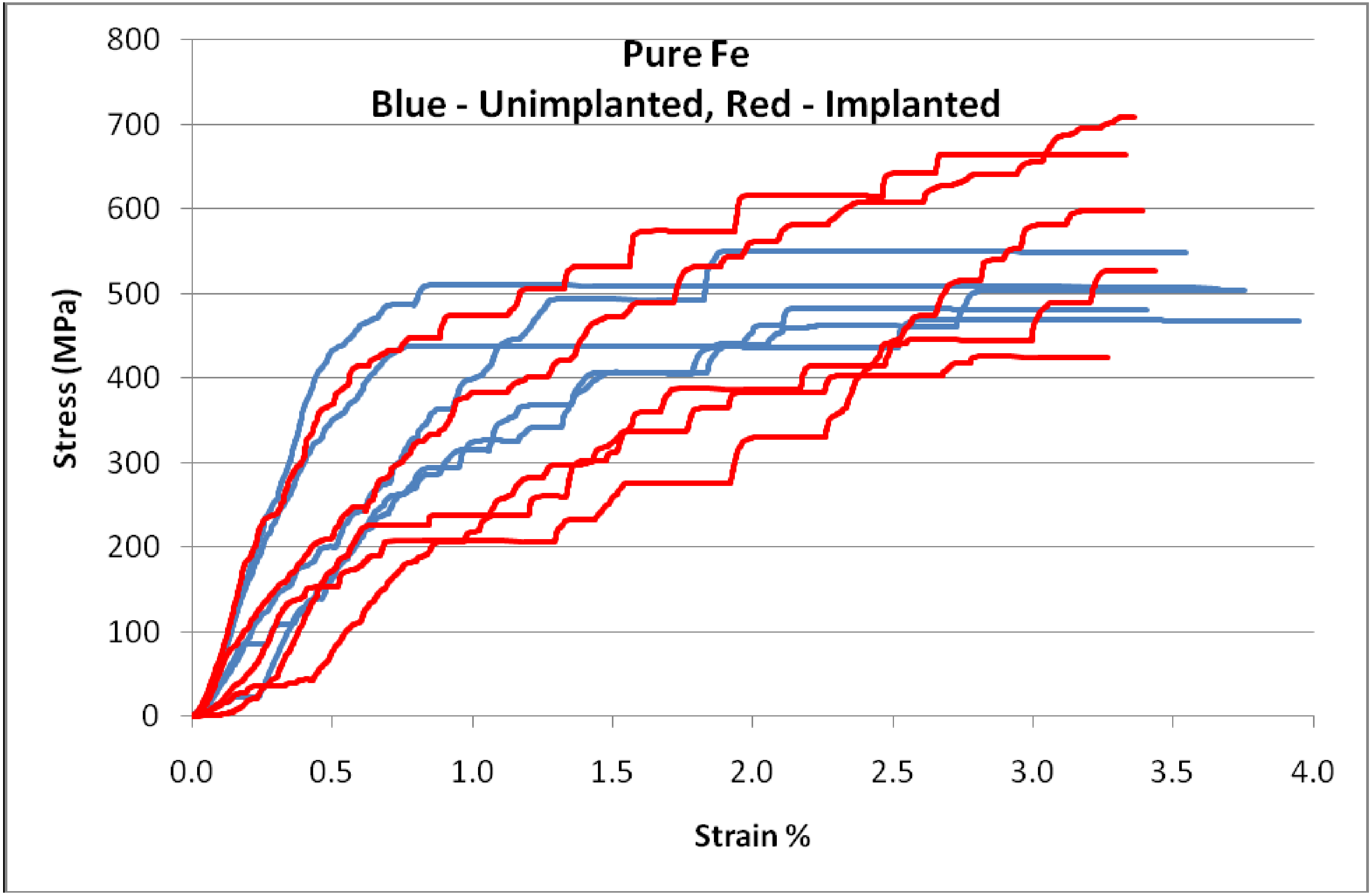


FeCr Micro Pillars





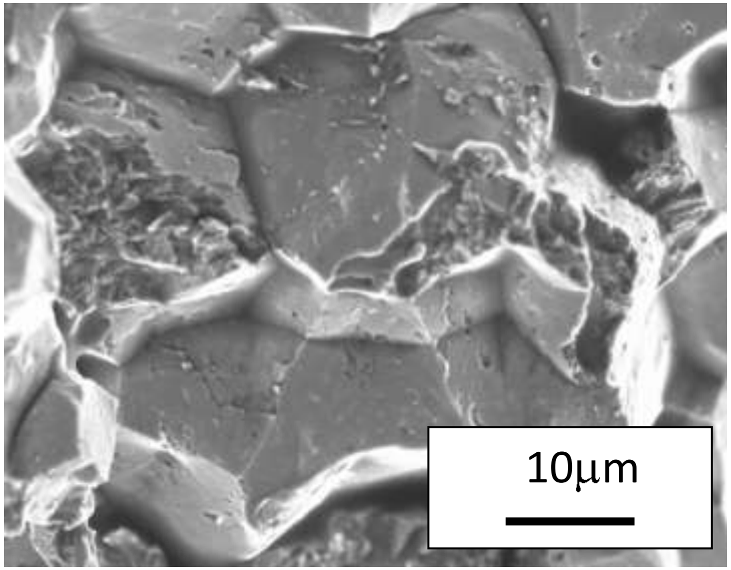
FeCr Micro Pillars

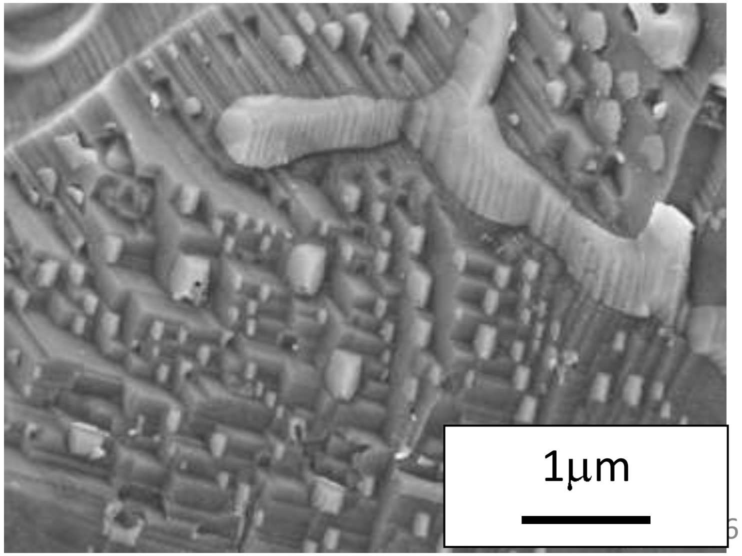


Case Study Two: Fracture of Grain Boundries

Measuring grain boundary fracture toughness

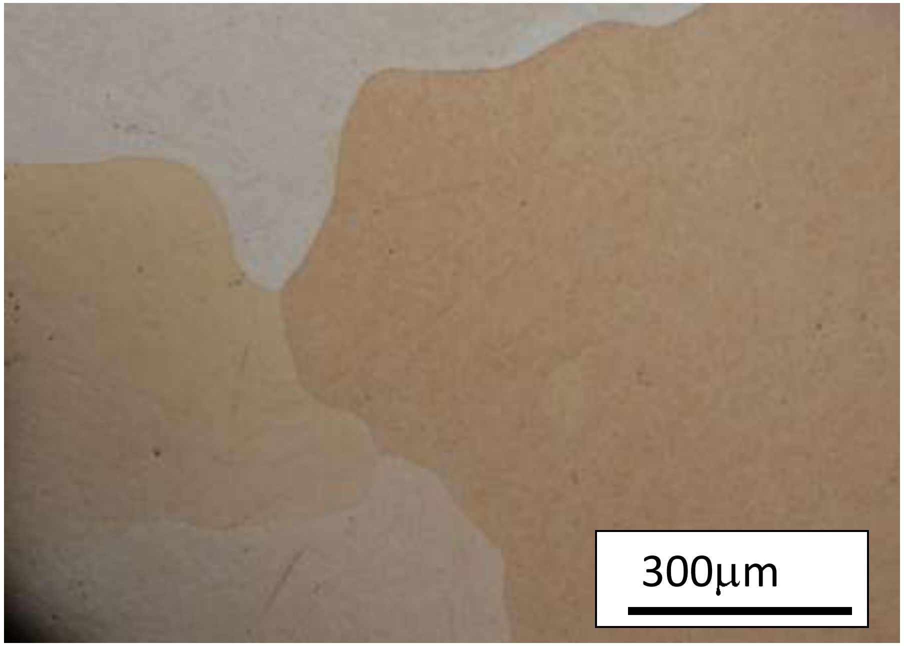
- **Polycrystalline material properties often controlled by grain boundaries**
- **Measurement of single boundaries difficult/expensive**
- **Bi-crystals may only contain “special” boundaries**
- **Need to be able to compare local chemistry with mechanical properties – especially after irradiation**





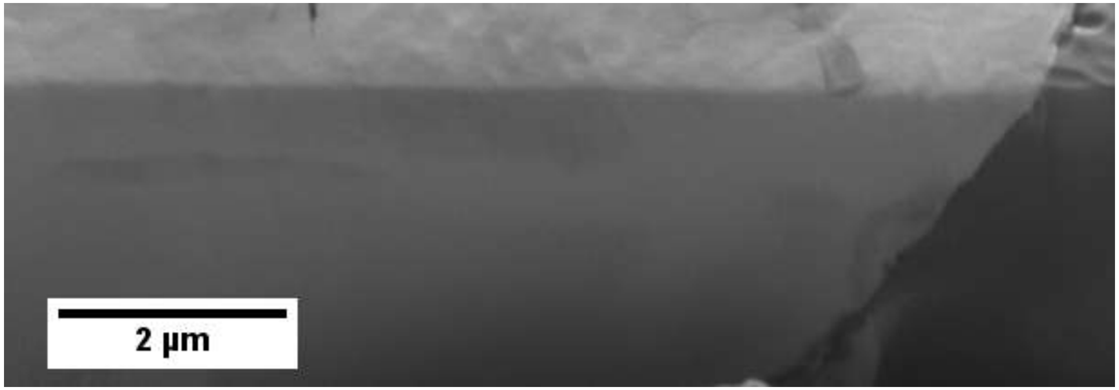
Sample manufacture

- Copper bismuth well known for GB fracture at room temperature
- Mechanism and anisotropy of embrittlement not well understood
- Sample contains 0.02wt%Bi (60ppm)
- Cast in vacuum inside quartz tubes @1374K - slow cooled
- Samples sectioned into bars and discs for testing
- Large grains with no visible precipitates



Cantilever manufacture

- Only grain boundaries running normal to surface tested
- Cantilevers have pentagonal cross-section
- Sharp notch milled at grain boundary to act as fracture initiation site



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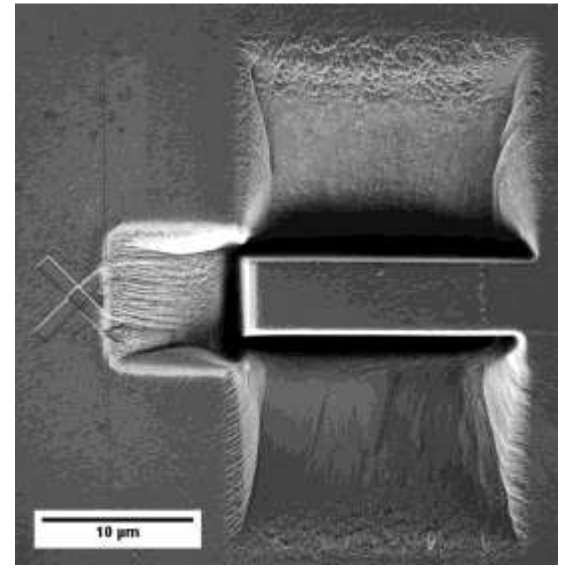
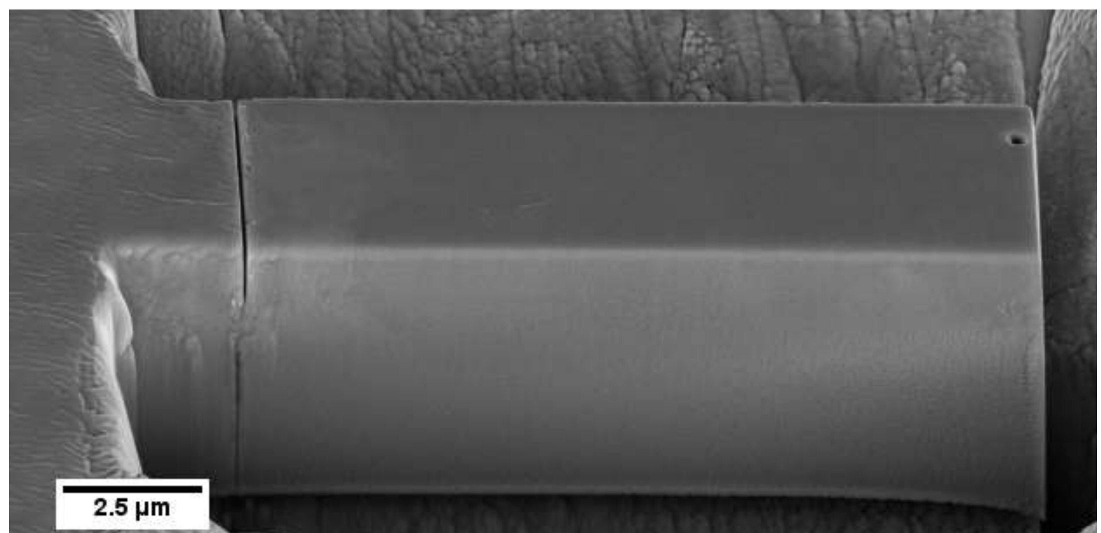




EBSD

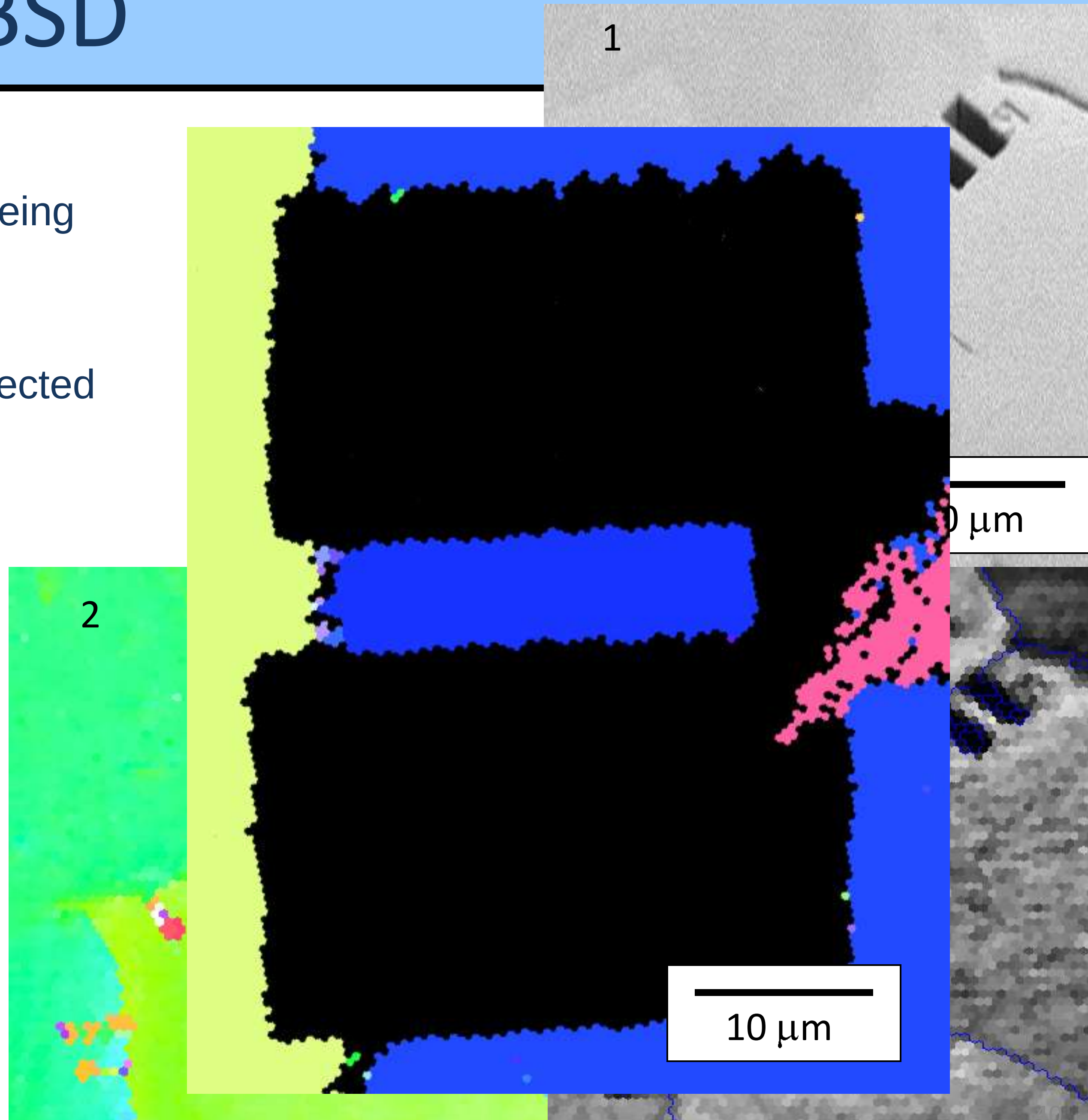
Used to characterise misorientation at g.bs being tested

Allows g.bs of specific misorientation to be selected for testing

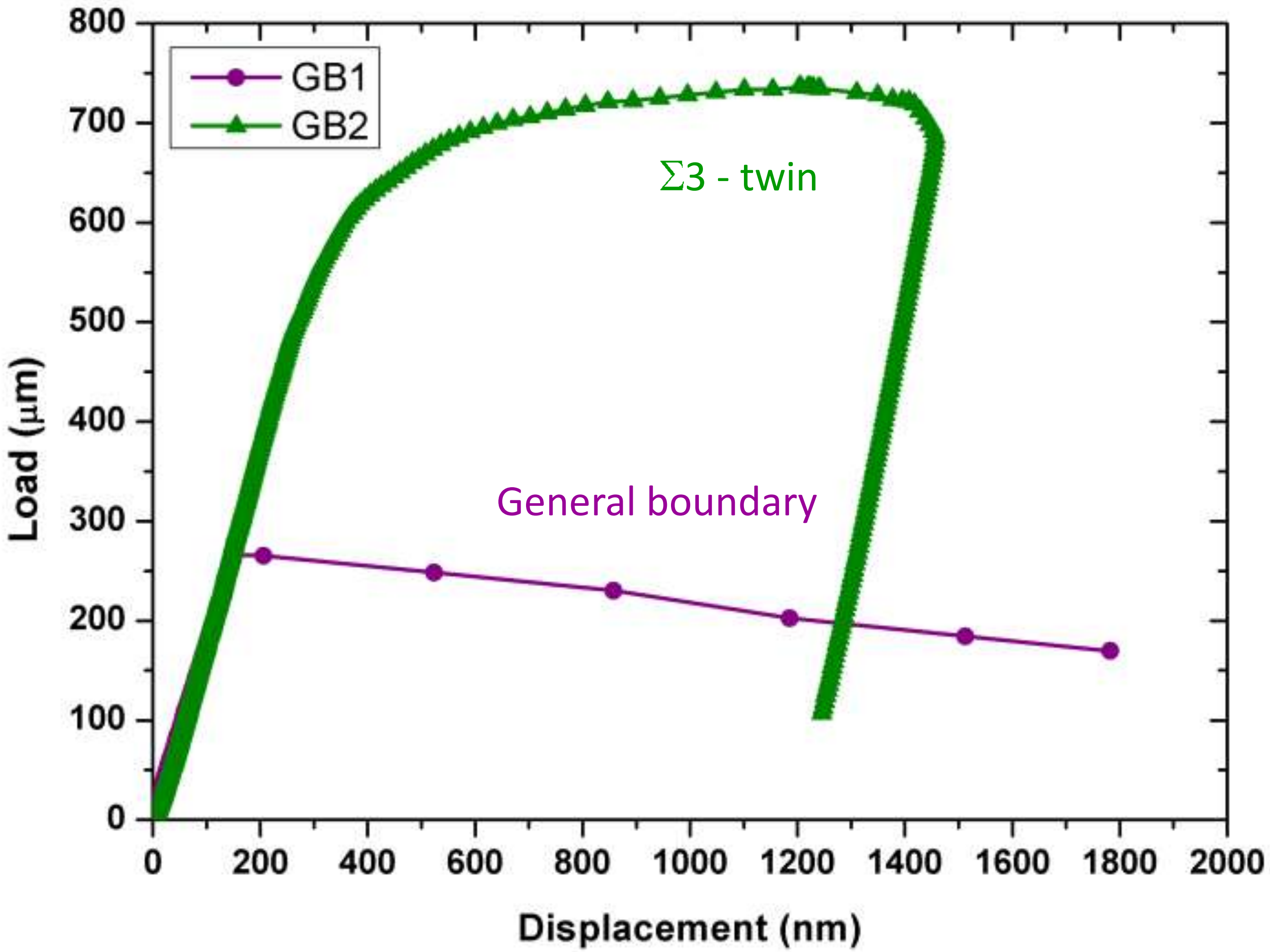
1-SEM image

2- Grain orientations (normal IPF map)

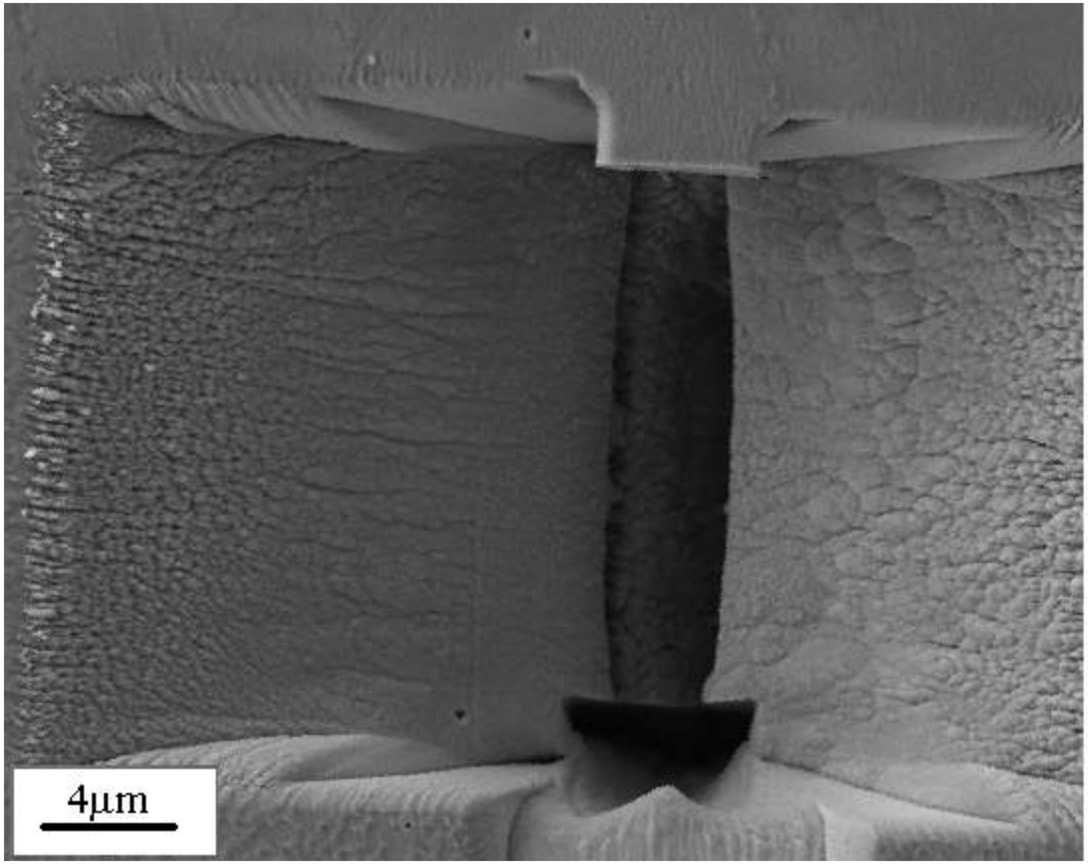
3-Grain boundaries of greater than 5^o misorientation

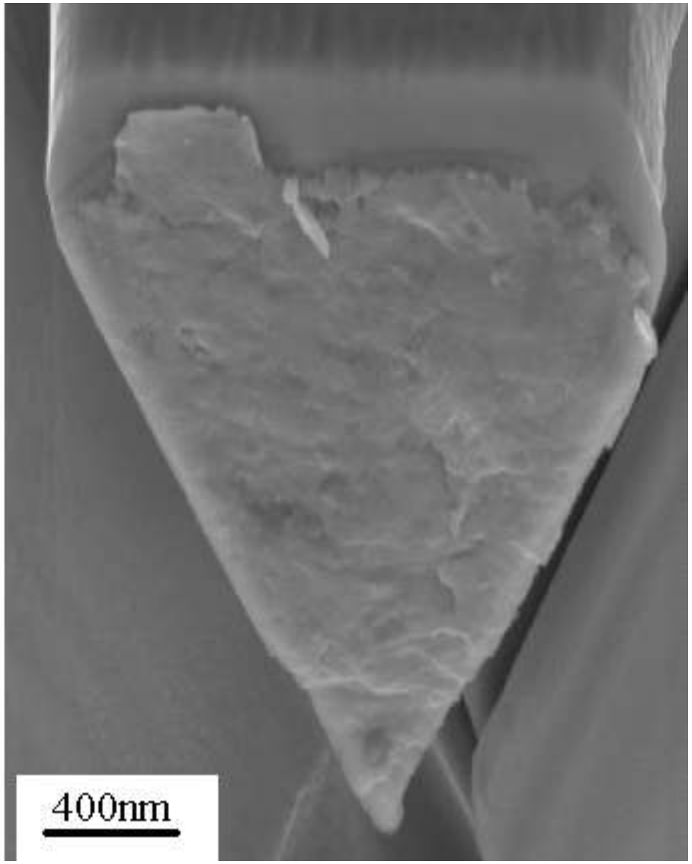


Testing of micro-cantilevers

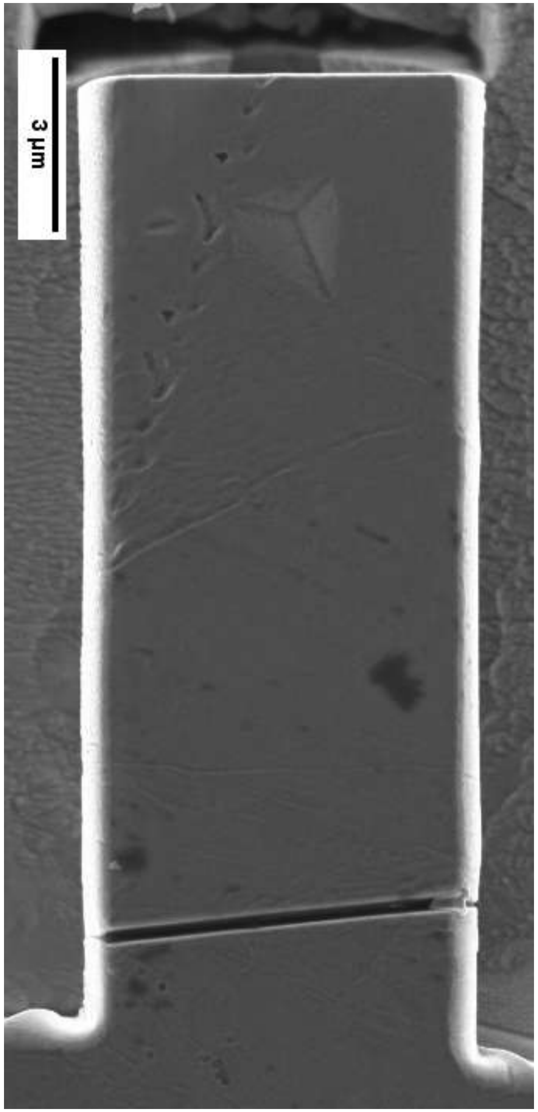


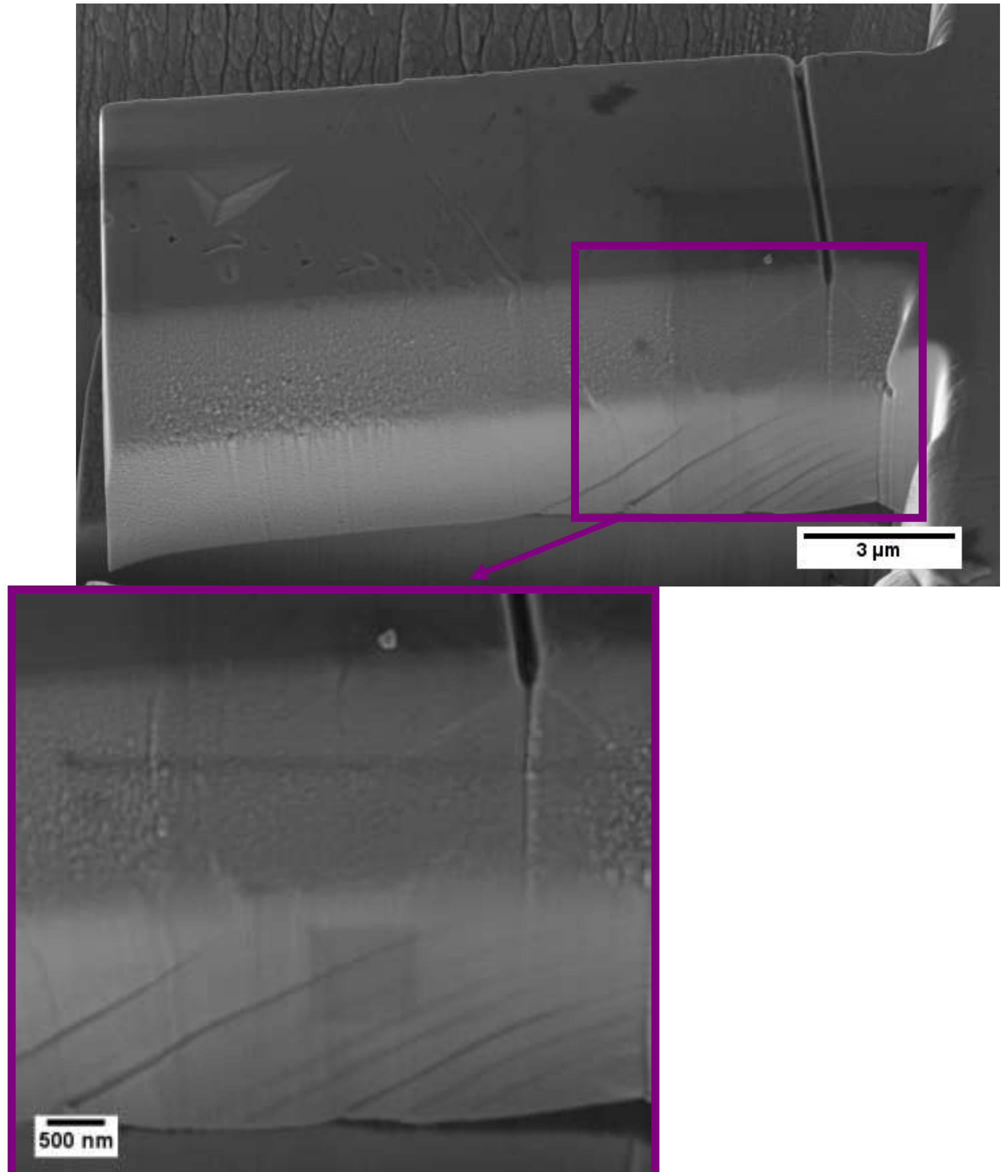
Tested - Fracture





Tested - No Fracture





Calculation of GB fracture toughness

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$$K_{1c} = \sigma_c \sqrt{\pi a} F(\frac{a}{b})$$

- p=load at fracture
- w=width
- b=beam depth
- a=crack depth
- L=length

$$\sigma = \frac{p_c L y}{I}$$

$$I = \frac{wb^3}{12} + (y - \frac{b}{2})^2 bw + \frac{w^4}{288} + \left[\frac{b}{6} + (b - y)\right]^2 \frac{w^2}{4}$$

$$F(\frac{a}{b}) = 1 + 2.53(\frac{a}{b}) - 14.5(\frac{a}{b})^2 + 35.57(\frac{a}{b})^3 - 22(\frac{a}{b})^4$$

This allows the fracture toughness for pentagonal beams to be calculated from the load displacement data and beam dimensions.

Do we have all dimensions?

p=load at fracture

w=width

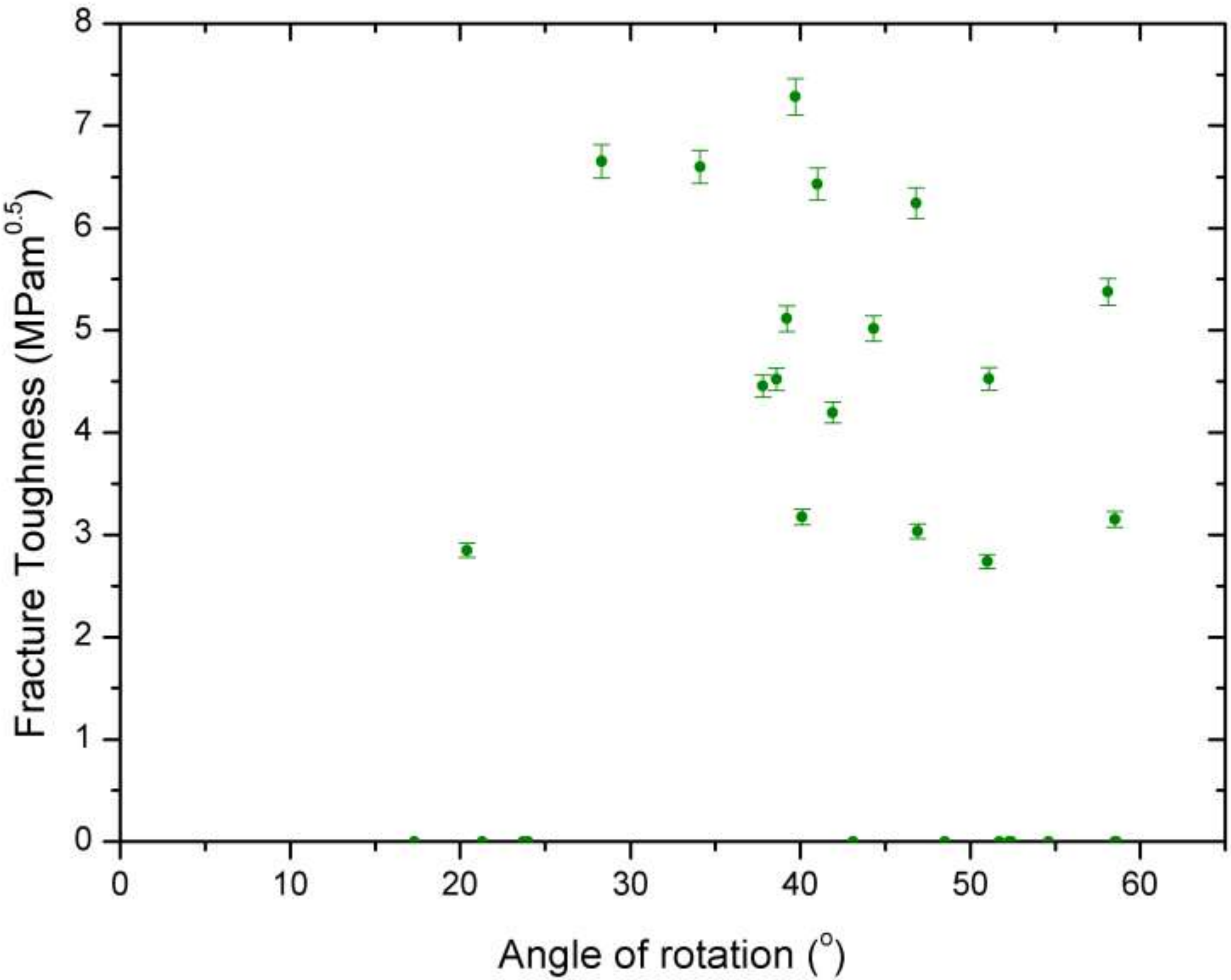
b=beam depth

a=crack depth

L=length

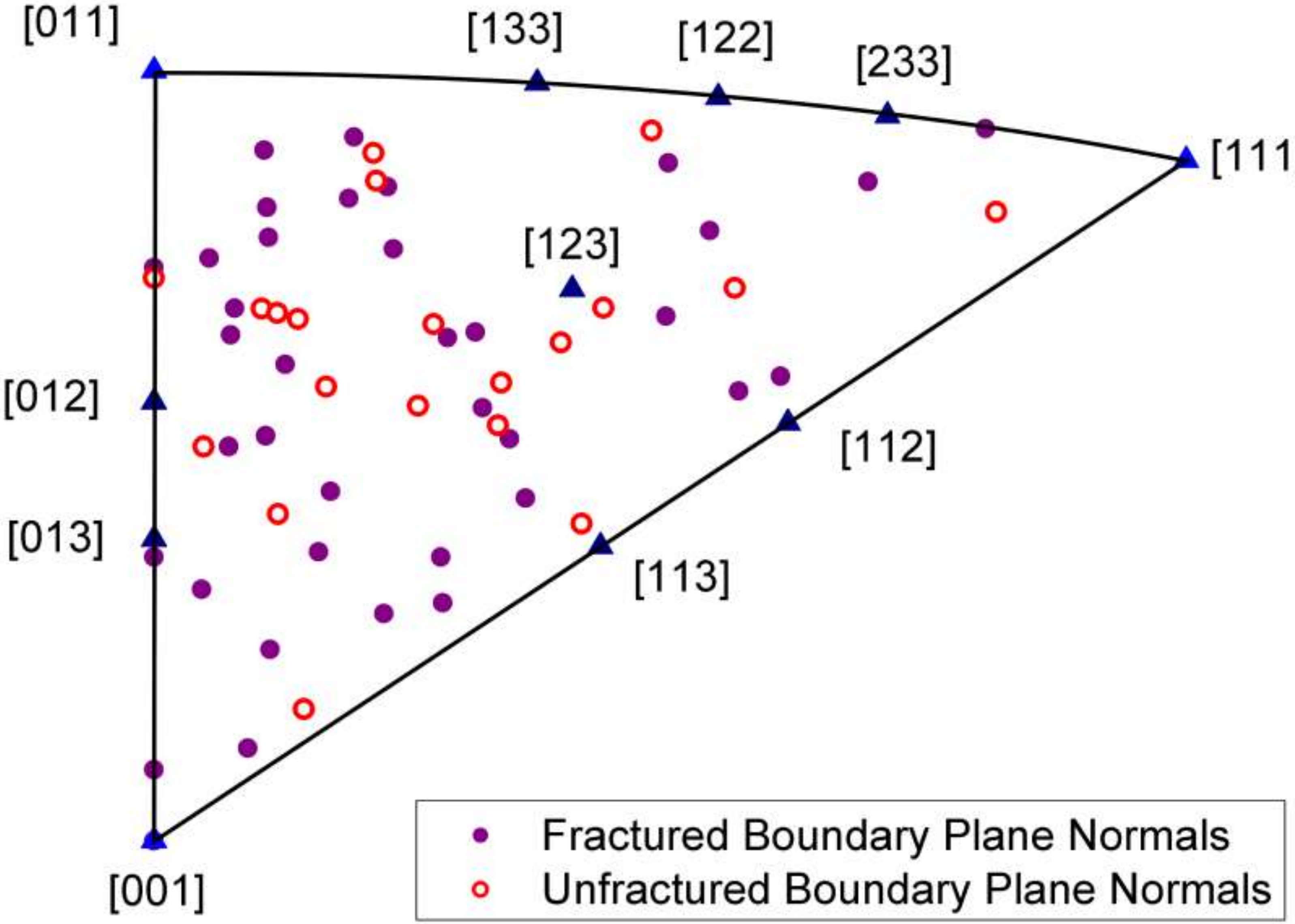
Load – Easy. From Nanoindenter
Width – Easy. From SEM images pre test
Depth – Medium. From SEM image, more difficult than W as sample must be tilted and only end can be measured
Crack depth- Hard. Can estimate before testing but MUST be measured post testing as reproducibility is poor
Length – Hard. Can't be directly measured on fractured specimens. Can be measured using AFM scan

Results

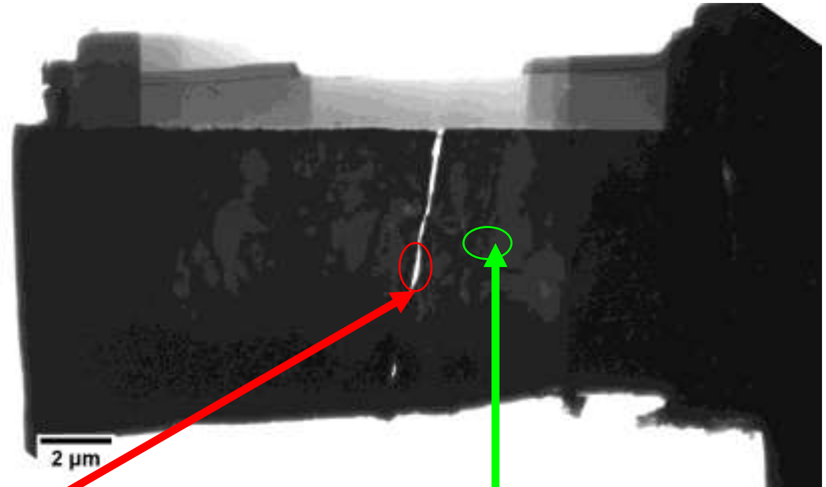


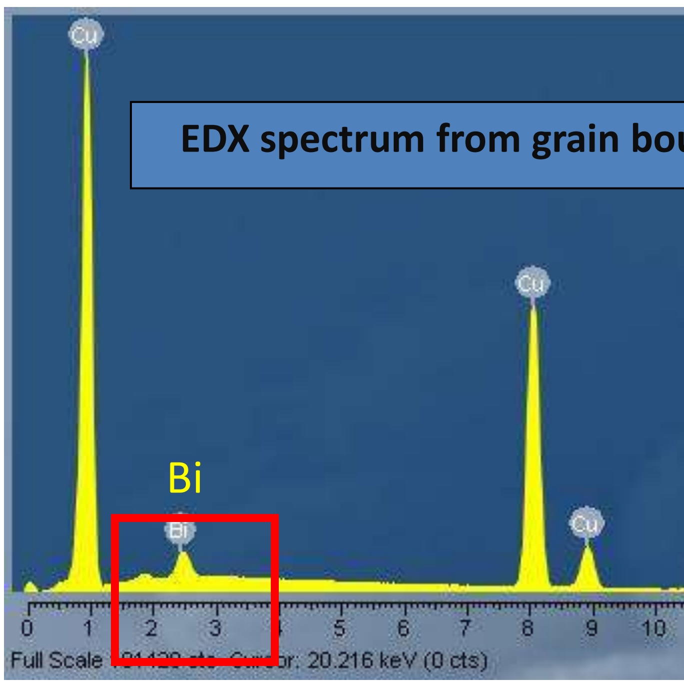
Fracture Toughness (MPam$^{0.5}$)
Angle of rotation (^{o})
0
1
2
3
4
5
6
7
8
0
10
20
30
40
50
60

Results

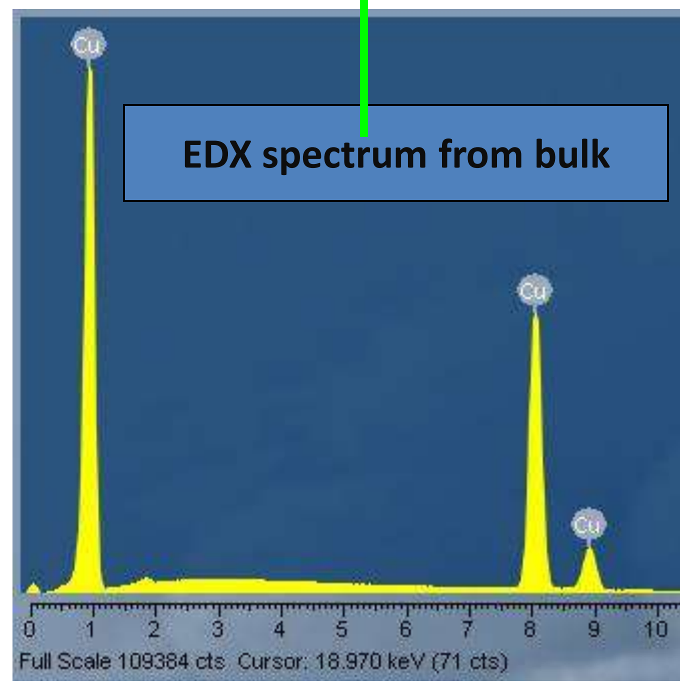

[011]
[133]
[122]
[233]
[111]
[123]
[012]
[112]
[013]
[113]
[001]
Fractured Boundary Plane Normals
Unfractured Boundary Plane Normals

TEM EDX – FIB lift-out





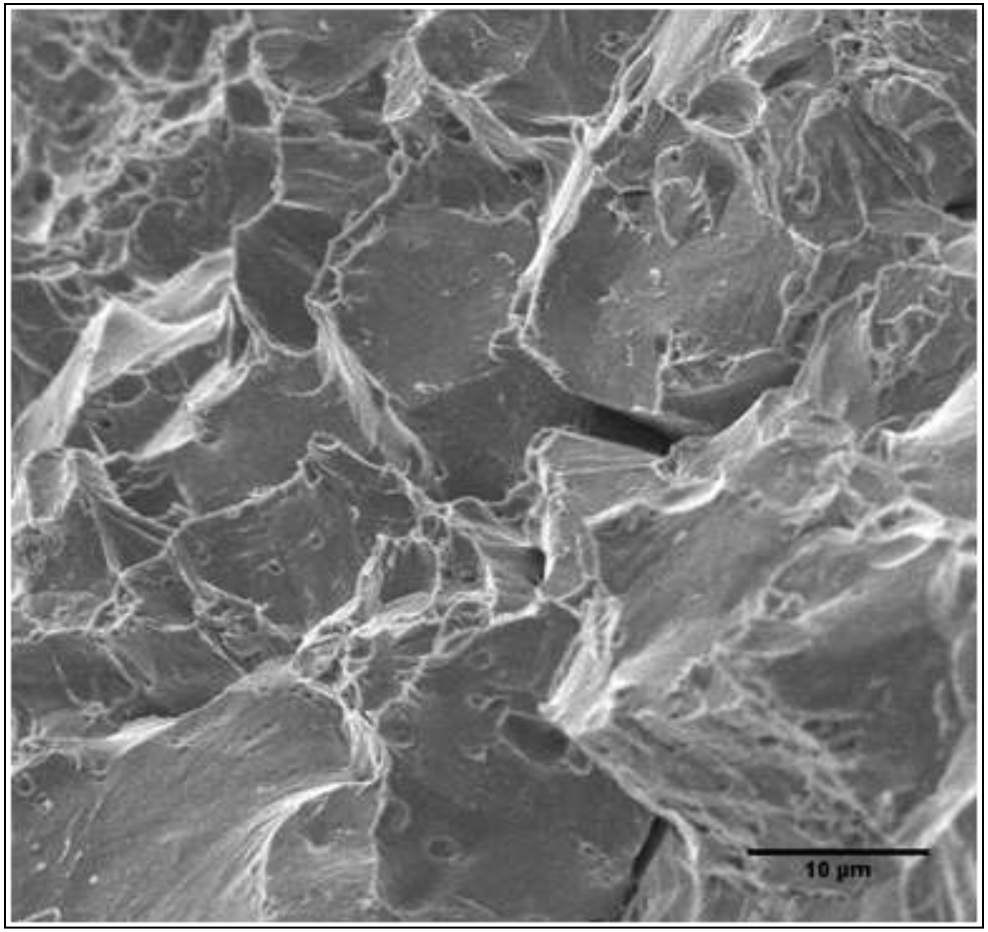
EDX spectrum from grain boundary



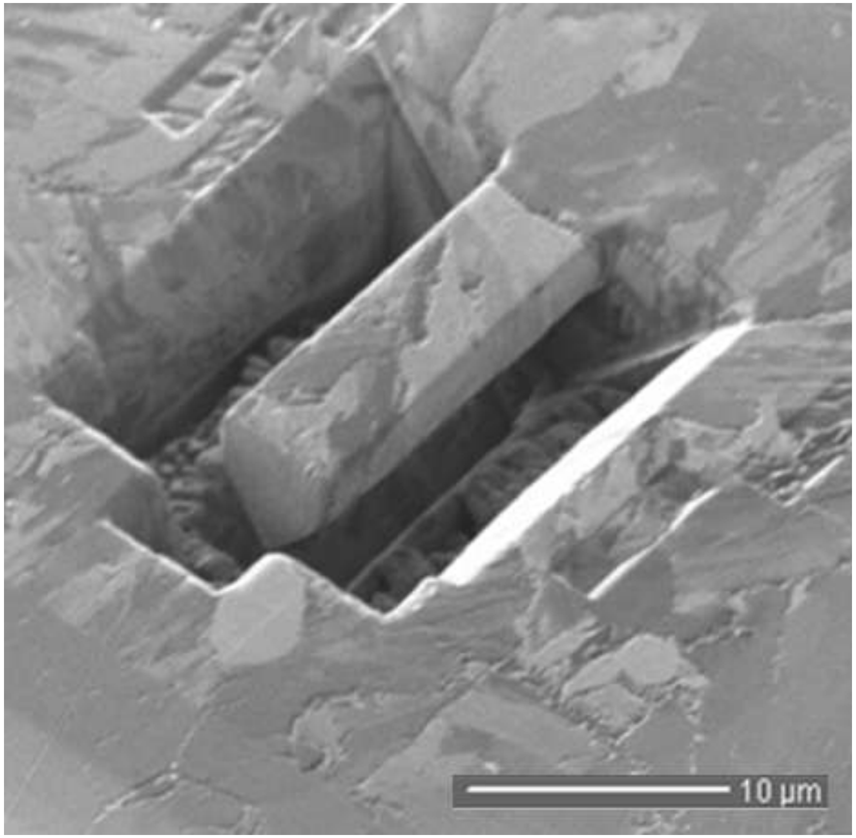
EDX spectrum from bulk

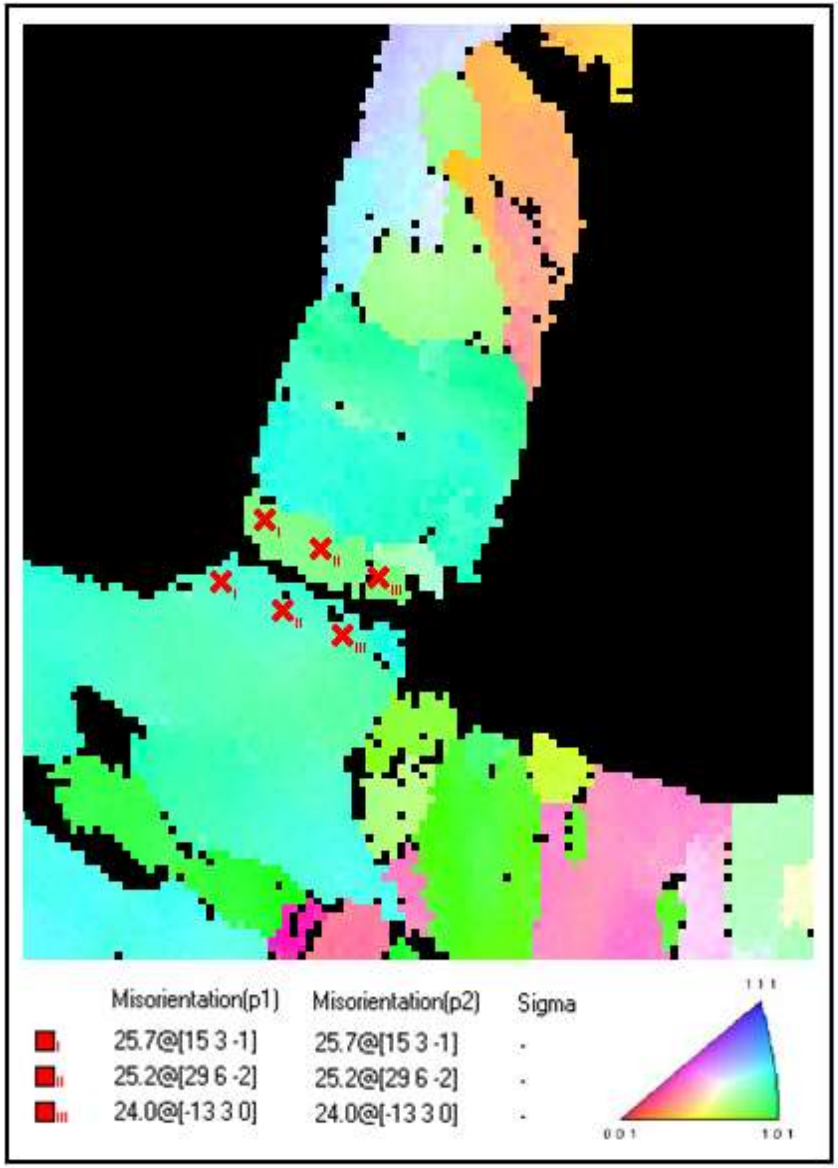
Applied to real systems

- Copper-bismuth is not of engineering use
- Many important nuclear materials are brittle
  - steels, under the right conditions
  - tungsten
  - ceramics
- Investigation into GB fracture in temper-embrittled steels

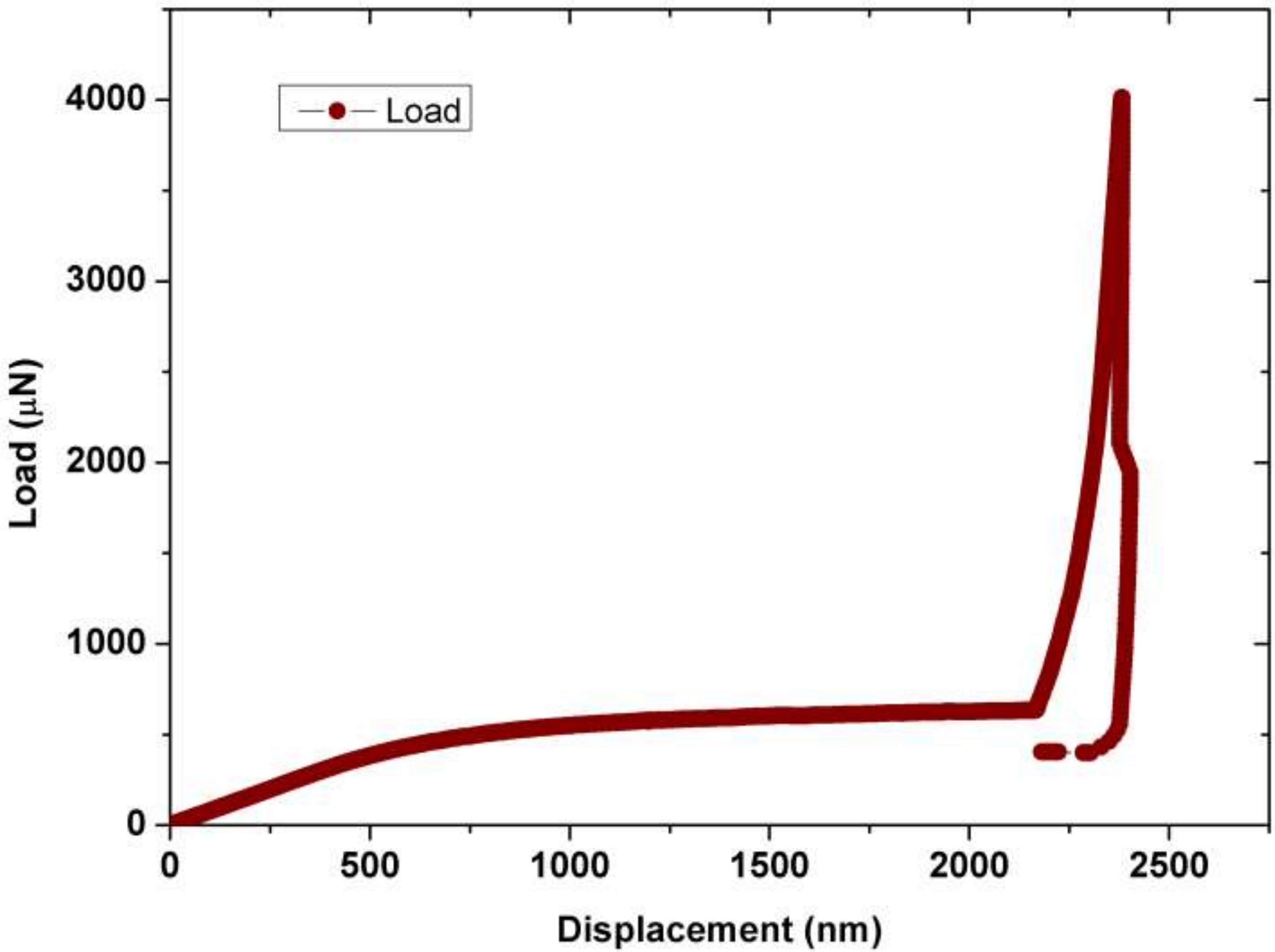


Temper-embrittled Steel





Load-displacement data



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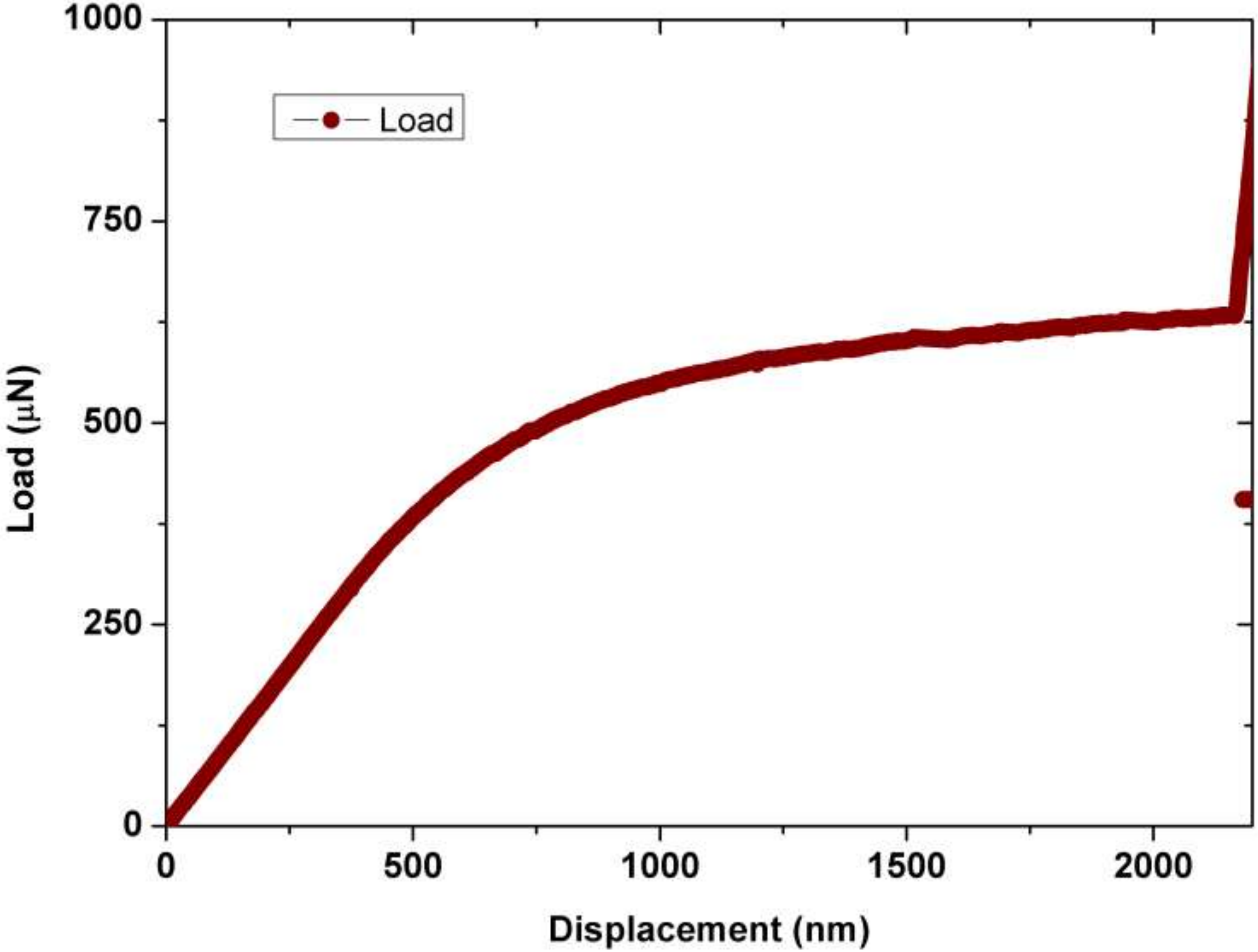
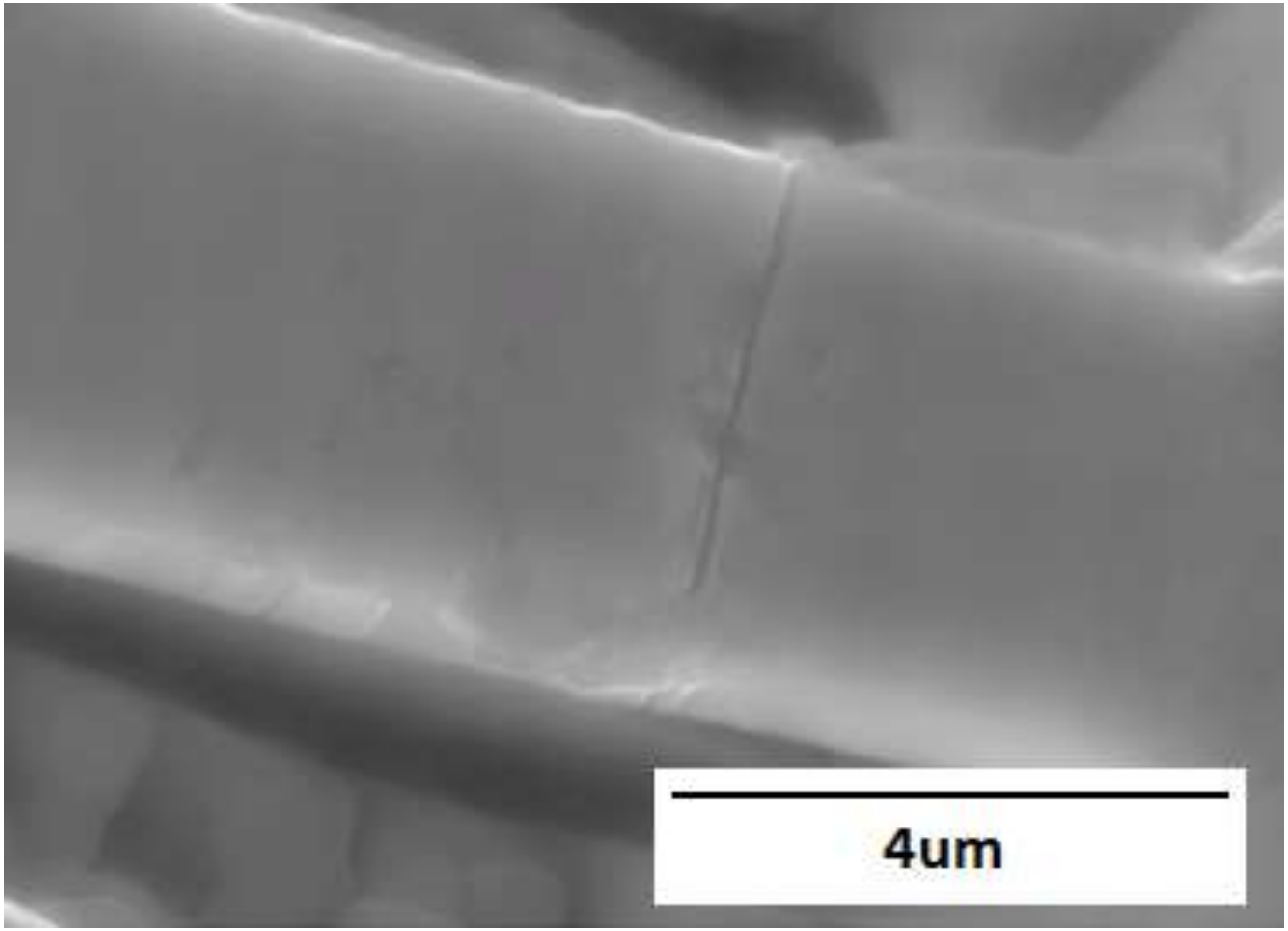
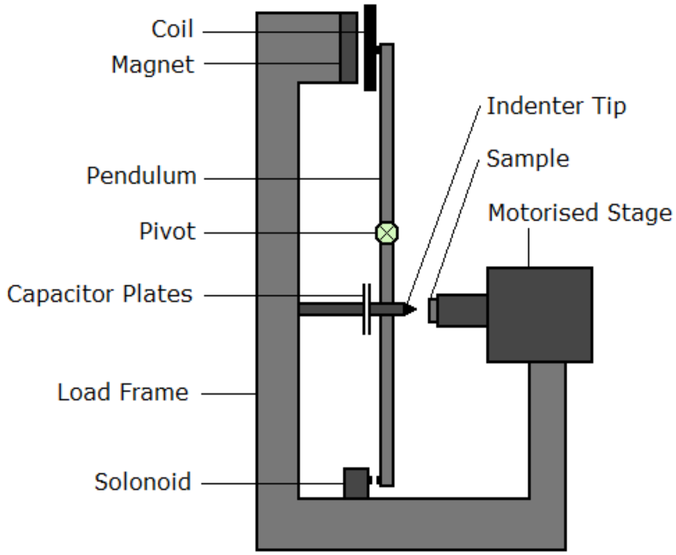
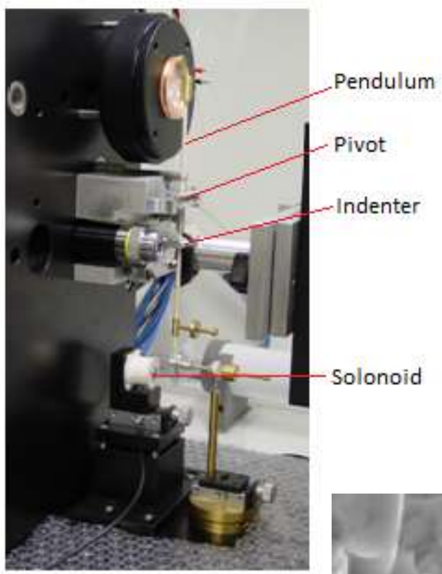


Cantilever after testing

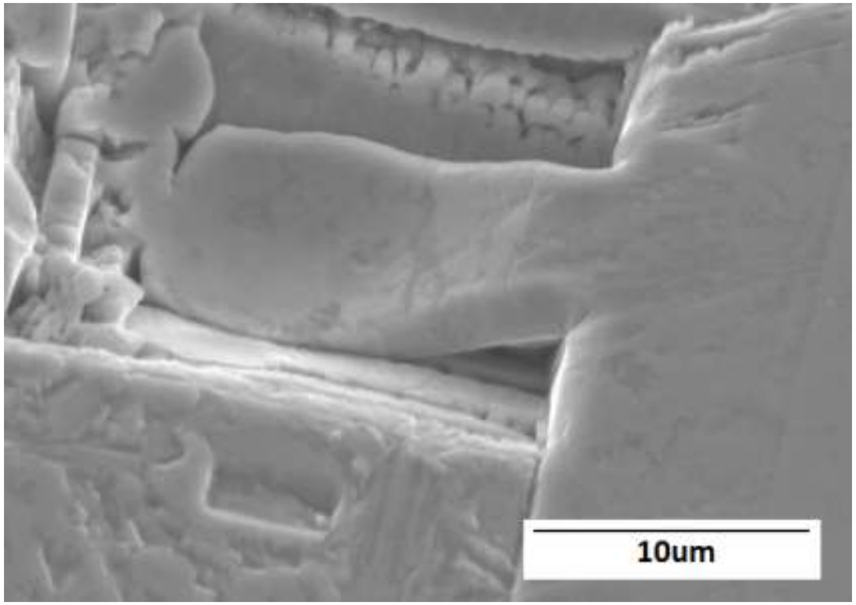


High strain rate testing





MML nanotest platform

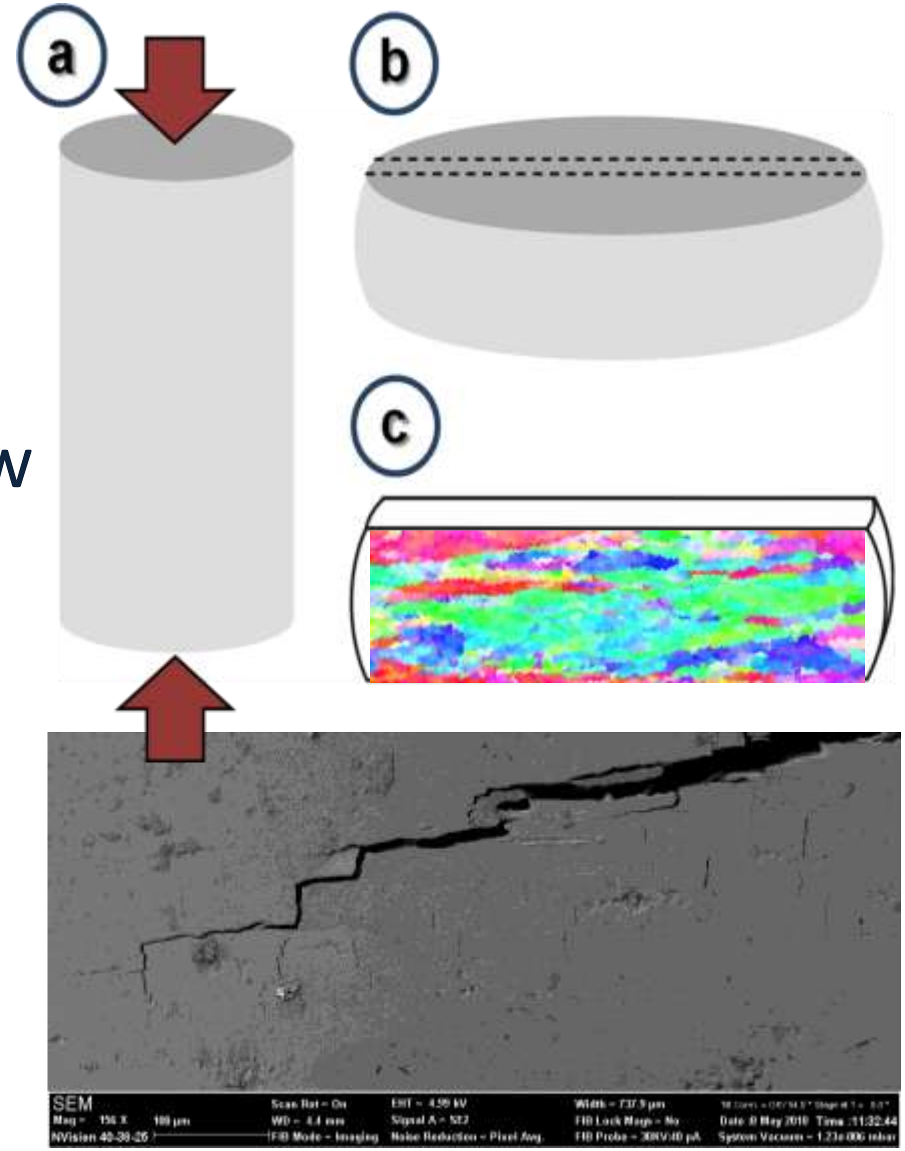


Failure

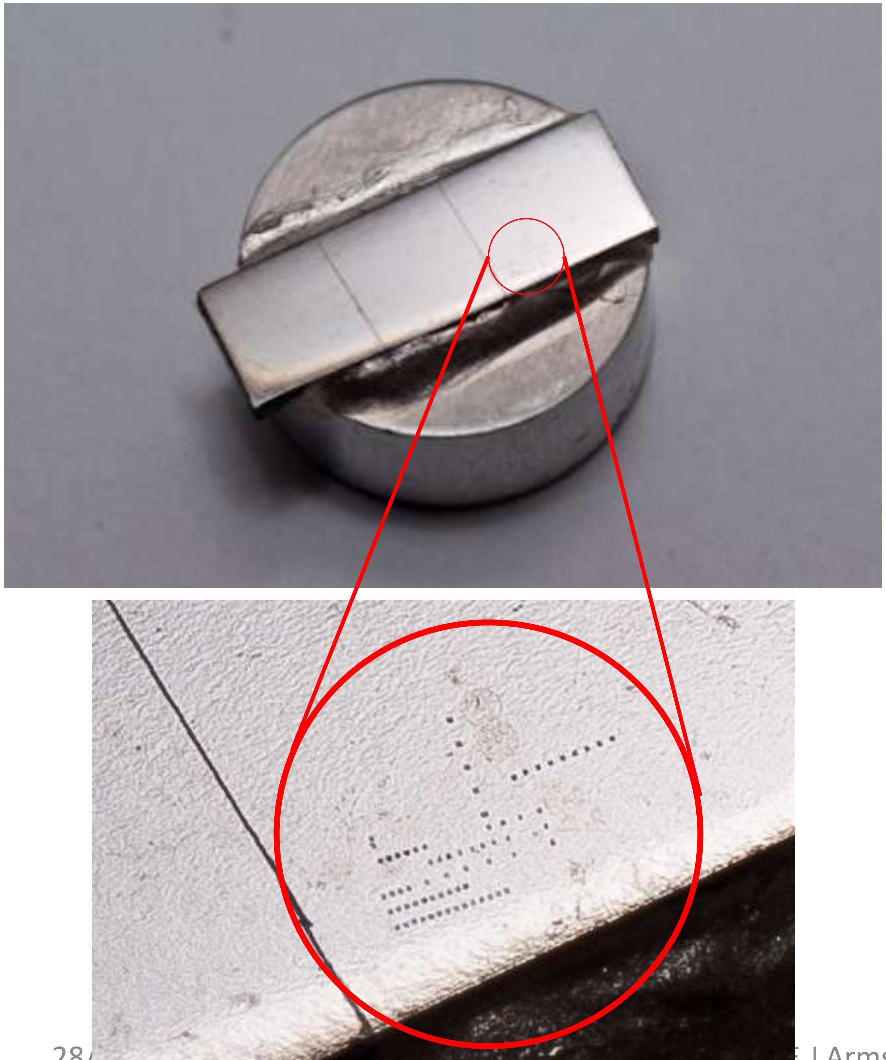
- It was not possible to achieve brittle fracture in temper embrittled S80 steel
- Although it is brittle the macro-fracture toughness is estimated to be 20 $MPam^{0.5}$
- The plastic zone around the crack tip is large
- For a micro-scale specimen to be fractured would need to be ≈10mm (Not very micro!!!)
- But James did write up a very good thesis!!

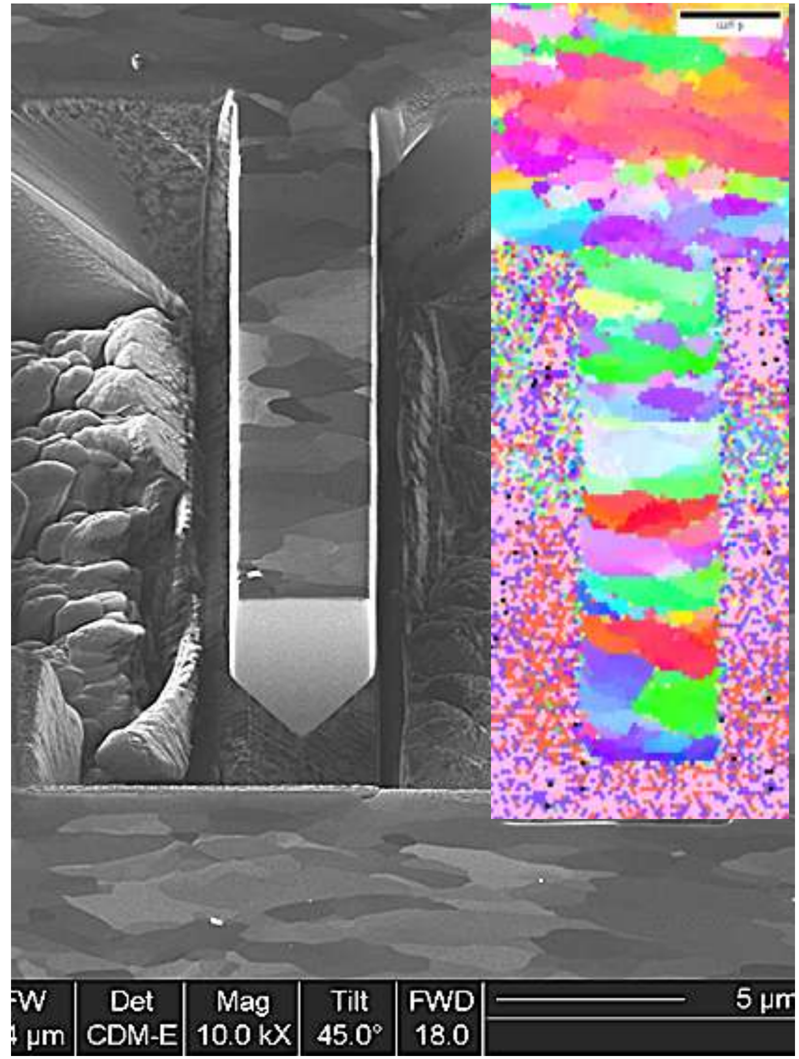
Applications to Tungsten

- Tungsten is brittle ($5MPam^{0.5}$)
- Important for nuclear fusion applications
- Need to understand how to control brittle behaviour
- Tests now being used to characterize brittle boundaries (James Gibson)

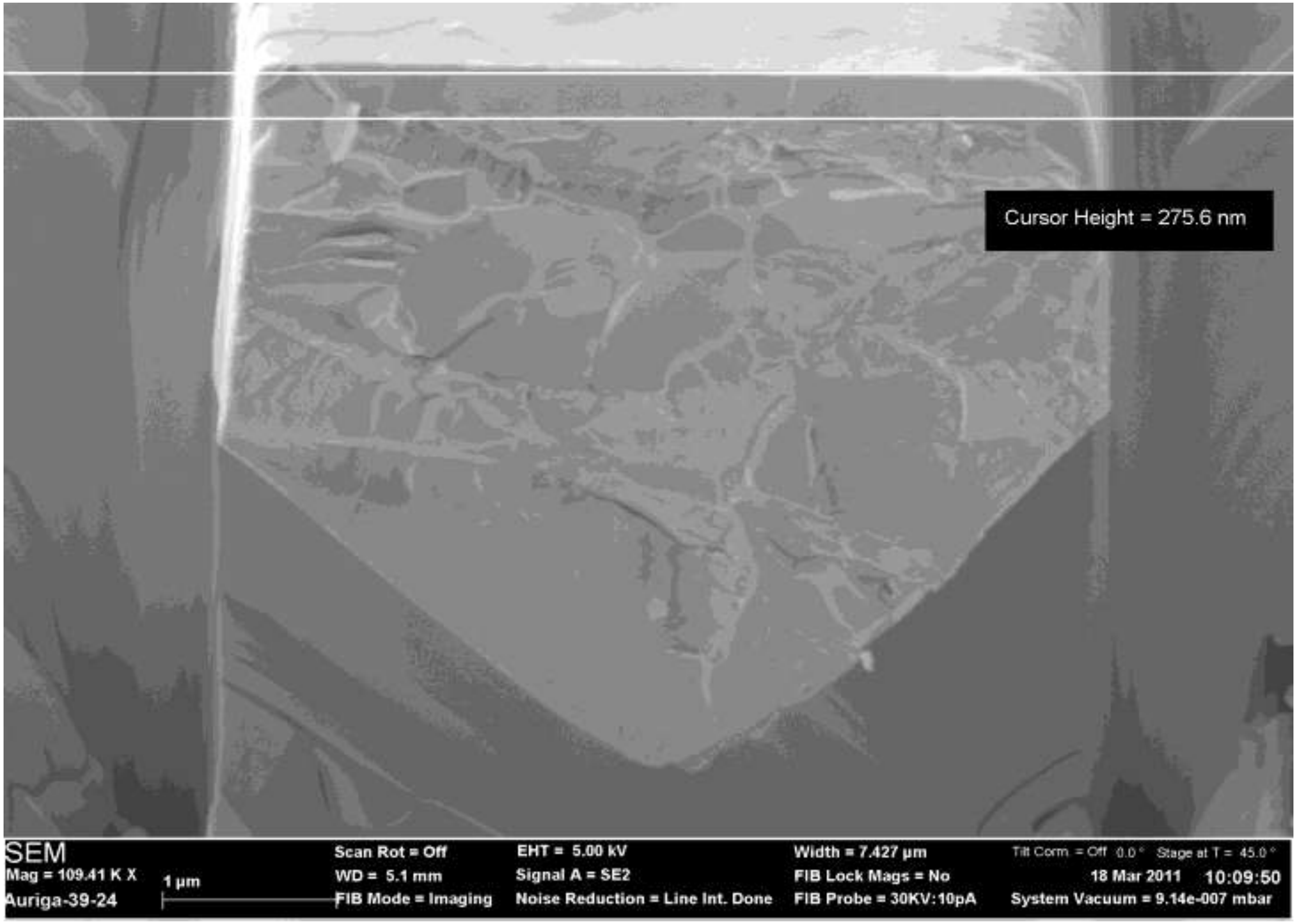


Grain Boundaries in Tungsten

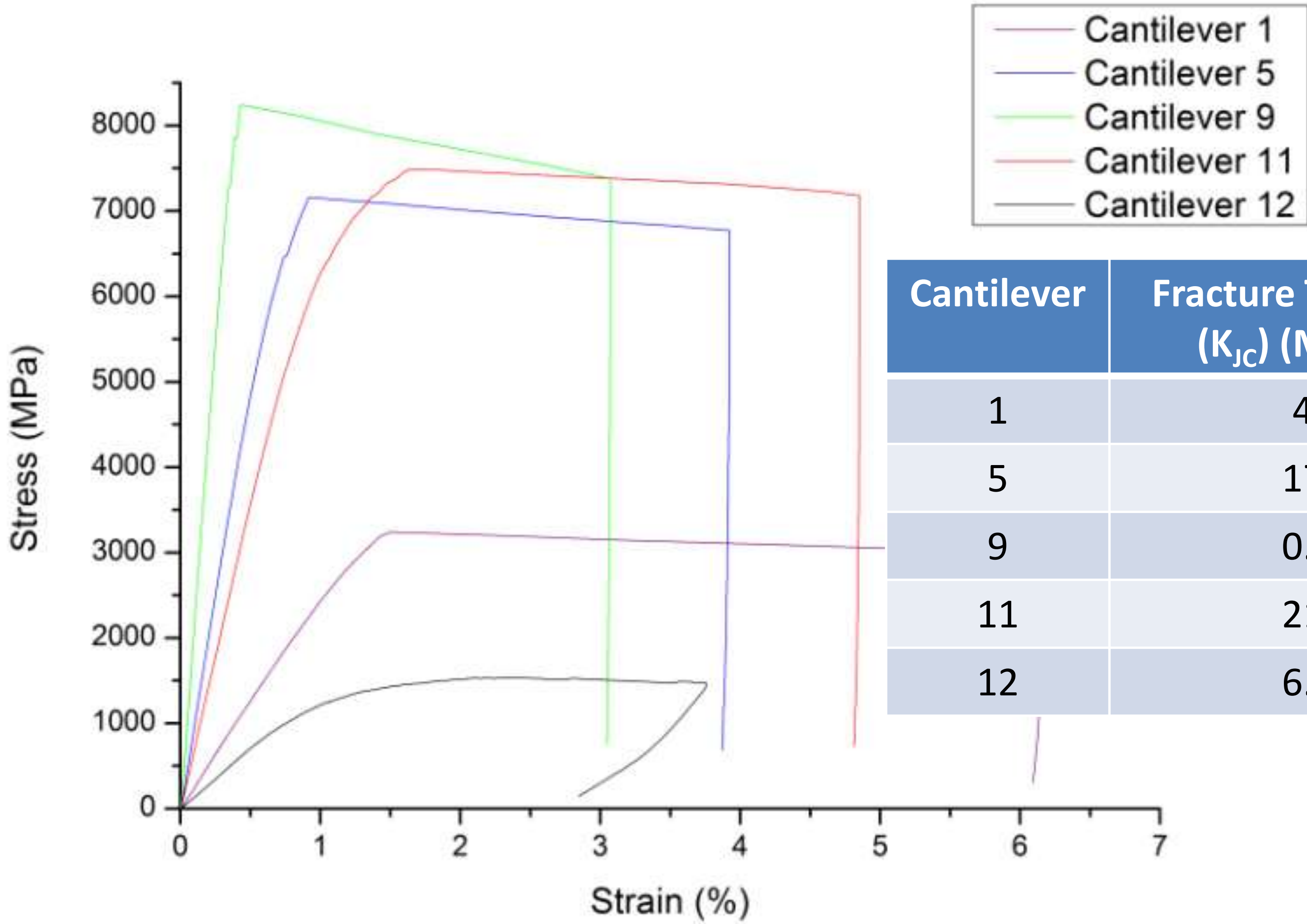




Grain Boundaries in Tungsten



Grain Boundaries in Tungsten



Cantilever	Fracture Toughness (K_{JC}) (MPapm)
1	4.9
5	17.9
9	0.53
11	21.2
12	6.52

Summary

- Micro-cantilever tests allow us to measure a range of material properties
- Effect of single grain boundaries can be measured
- Small volumes of materials needed for many results
- Allows results which are not obtainable using conventional tests

Summary and Future Questions

- Micro-cantilever tests allow us to measure a range of material properties
- Effect of single grain boundaries can be measured
- Small volumes of materials needed for many results
- Allows results which are not obtainable using conventional tests

- Problems in working in such small specimens?
- Are the results representative of bulk samples?
- How do Ga+ ions damage the specimens?
- Can modelling explain size effects?
- Can tests be performed at high temperature?

Thanks To

- Steve Roberts
- Angus Wilkinson
- Michael Rieth
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