

Data Interpretation 4: Mapping

N. Randall

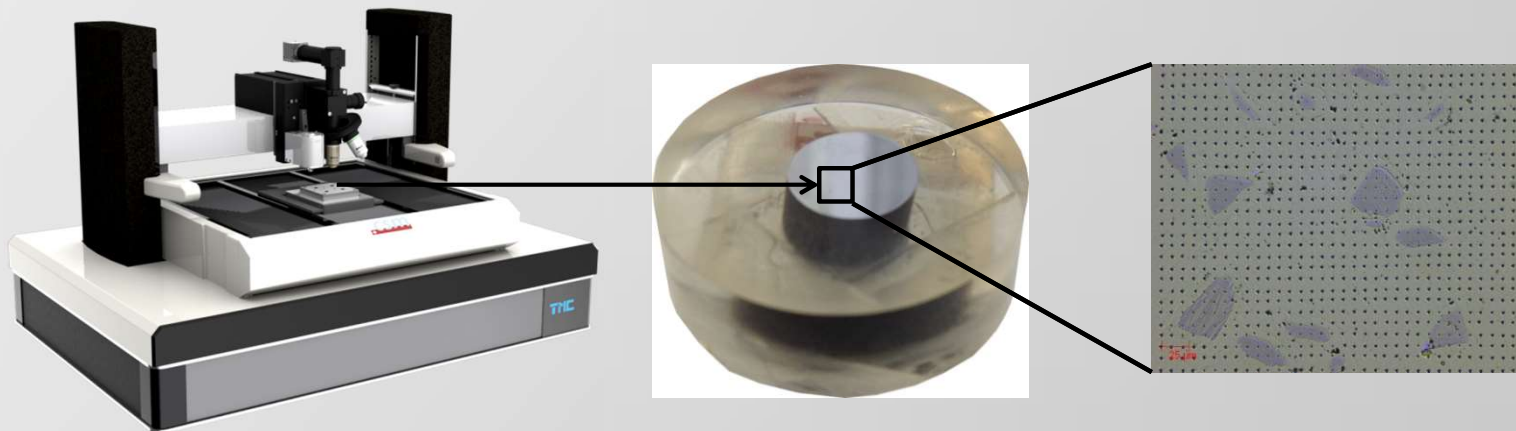
Grid Indentation

> Statistical analysis

- Automated matrix of measurements
- Easy access to average data of Hardness (H_{IT}) & Elastic Modulus (E_{IT})

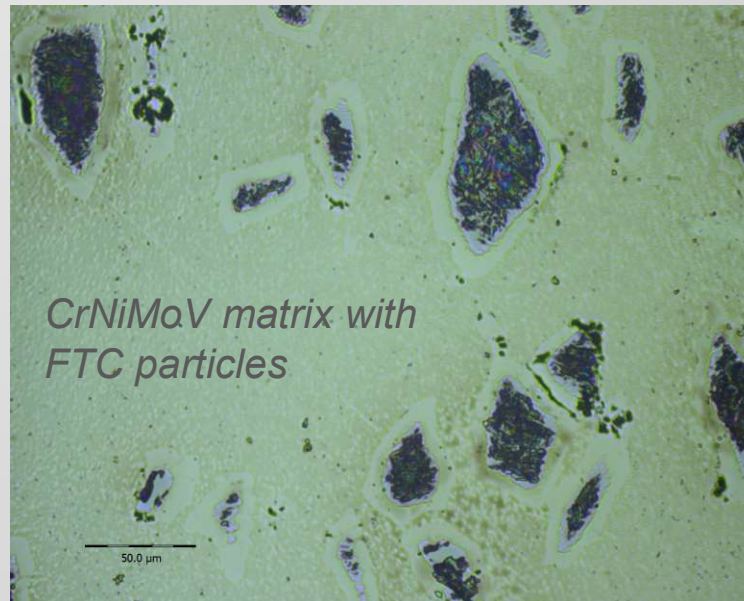
> Grid indentation technique

- A two-dimensional mapping tool for examining the properties of constituent phases independently of each other.
- Large arrays of indentations and statistical analysis of the resulting data

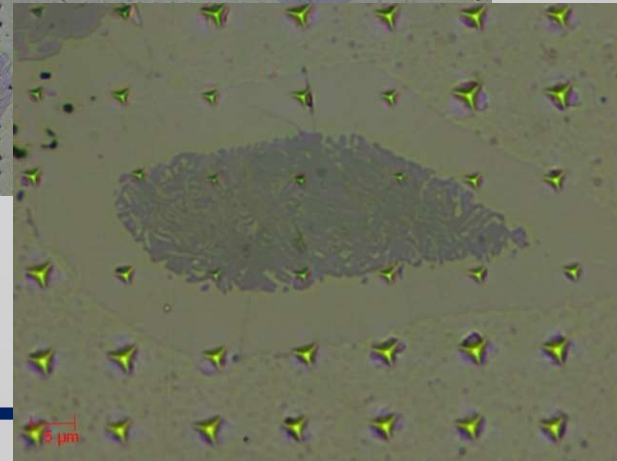
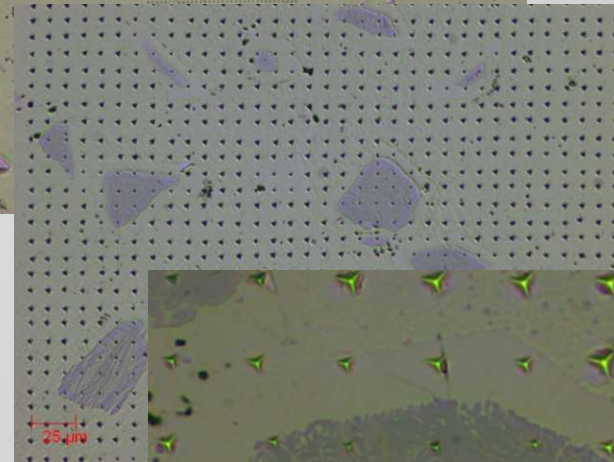
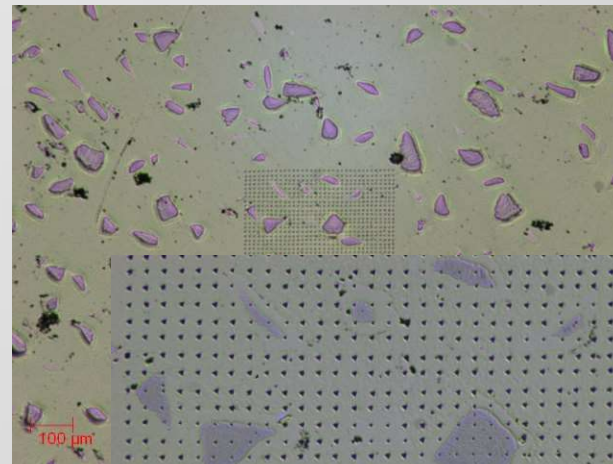
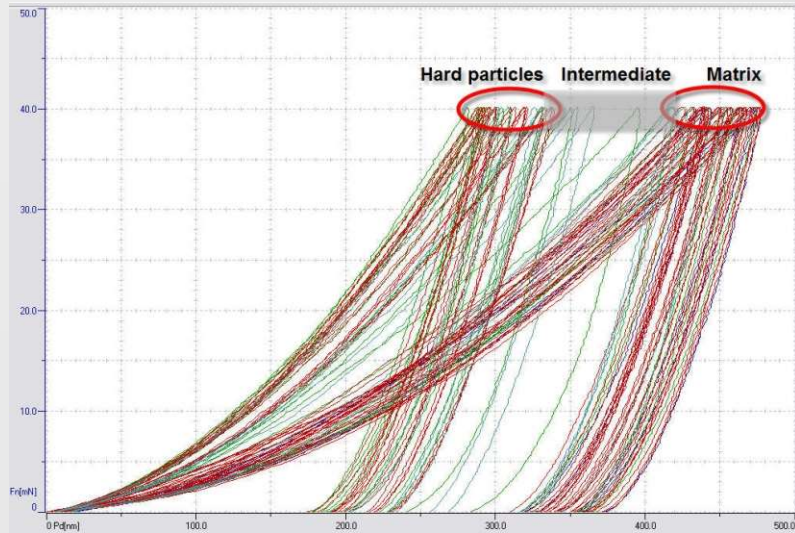


Co-W alloy

Simple Matrix
20 x 20 indents
Applied load 20 mN
Loading rate 40 mN/min.
Pause 15 s
Approach and Retract speeds 5000 nm/min.
Separation: 7 μ m



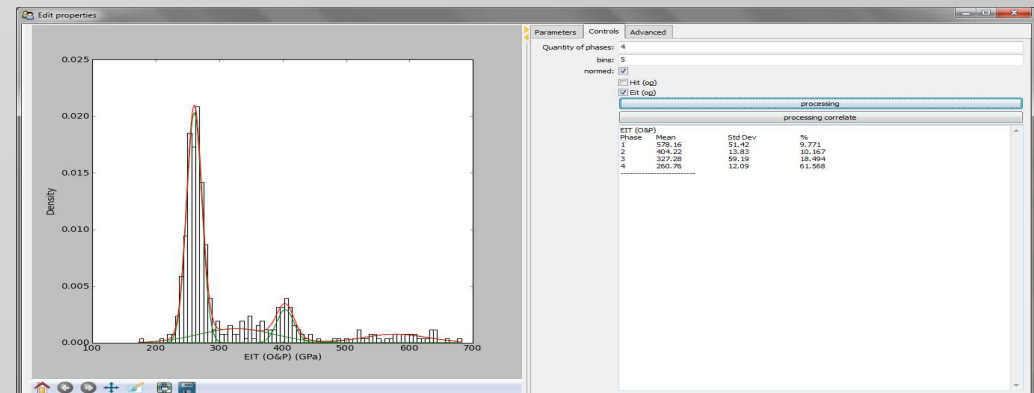
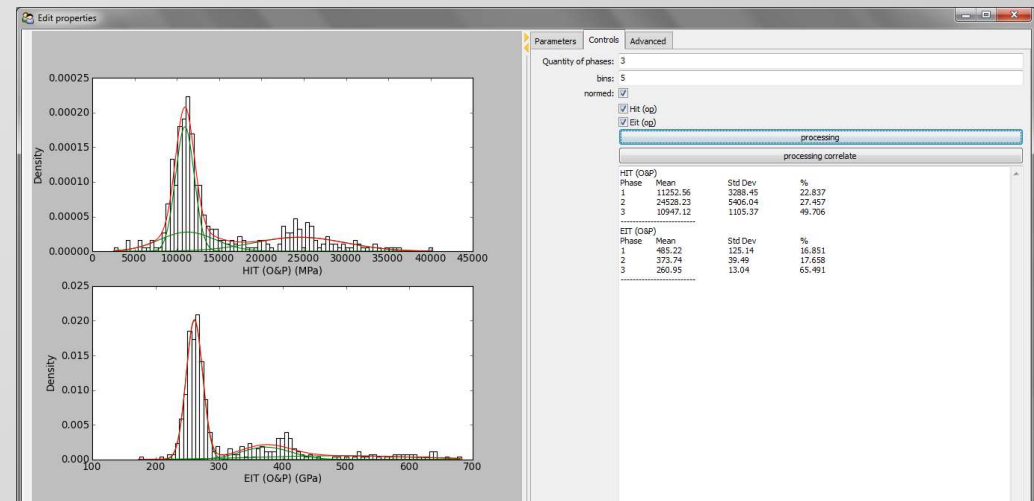
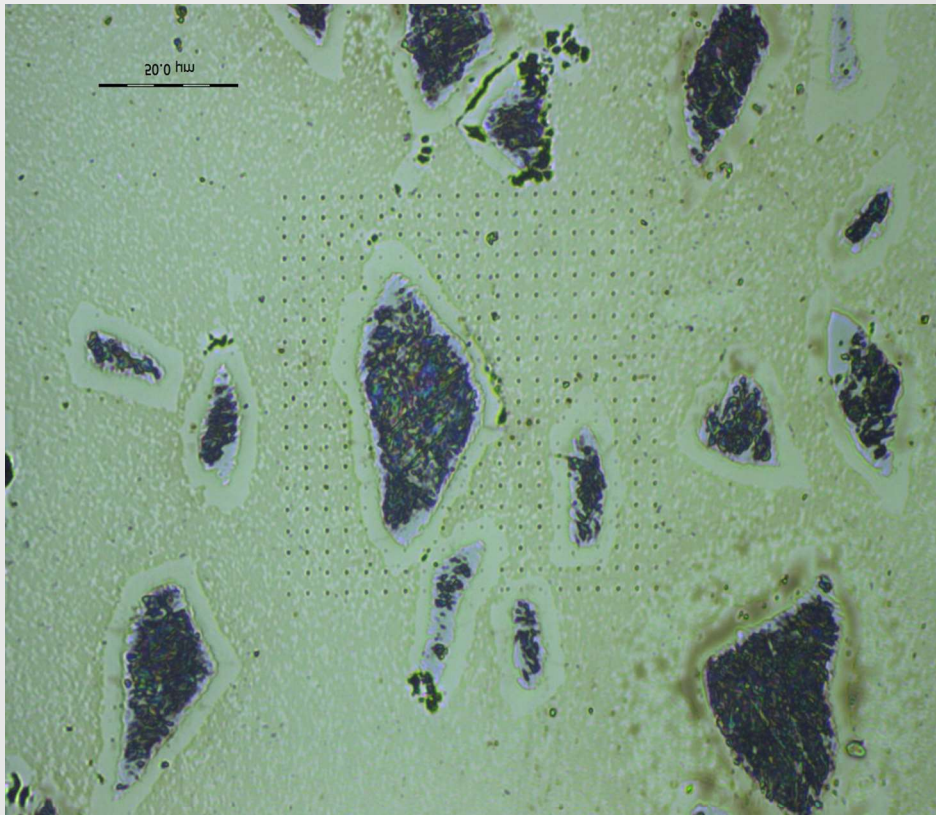
Co-W alloy



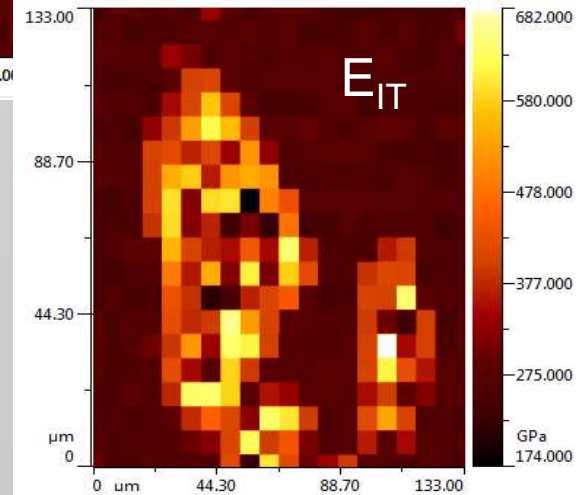
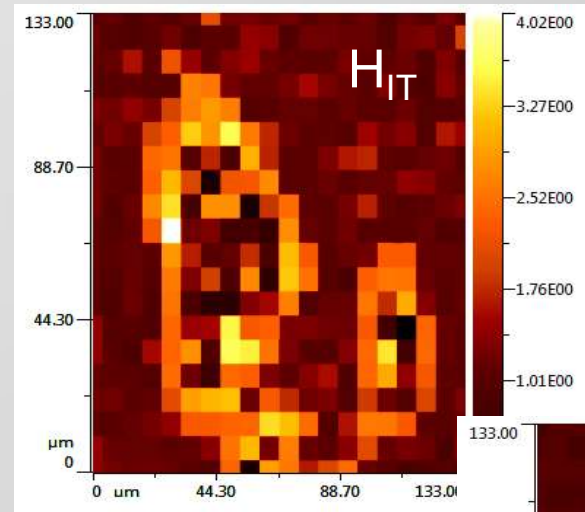
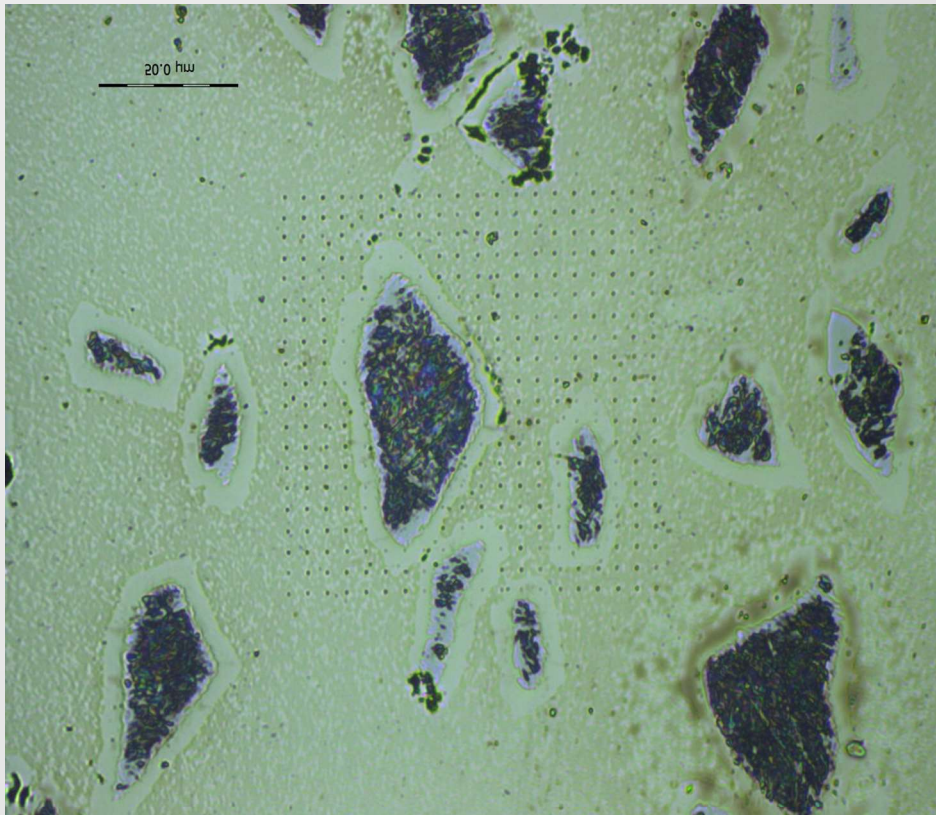
Three types of position of the indentation:

- 1 – in the matrix
- 2 – in the particle
- 3 – on the interface of the matrix and the particle

Co-W alloy



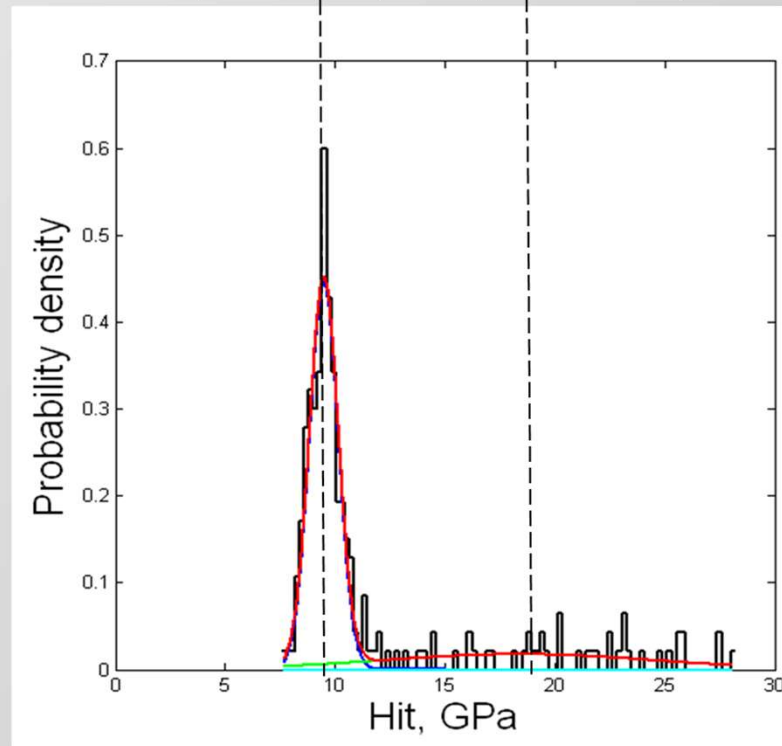
Co-W alloy



Co-W alloy

	Cobalt	Tungsten
Hardness, H_{IT} :	9.5 GPa	18.4 GPa
Volume Fraction :	74%	26%

- Cobalt has very distinct peak
- Tungsten is spread. Why?
- When indenting a hard phase embedded in a softer phase, difficult to have no influence of soft material
- Grain size here is 50 μm and indent depth is 500 nm, so 2 orders of magnitude smaller but there is still influence of the Cobalt...

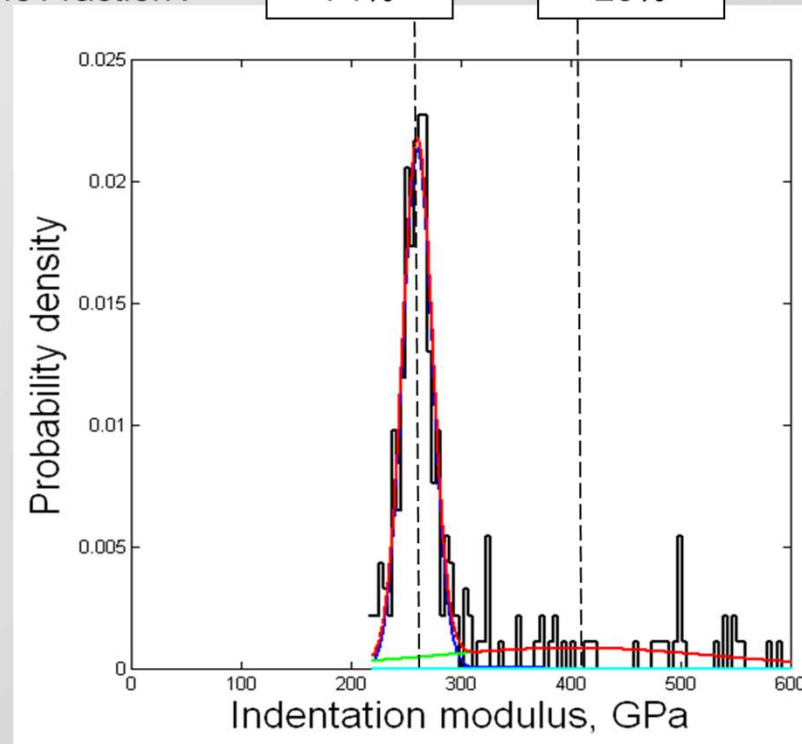


Co-W alloy

Elastic Modulus, E_{IT} :
Volume Fraction :

	Cobalt	Tungsten
Elastic Modulus, E_{IT}	260 GPa	403 GPa
Volume Fraction	74%	26%

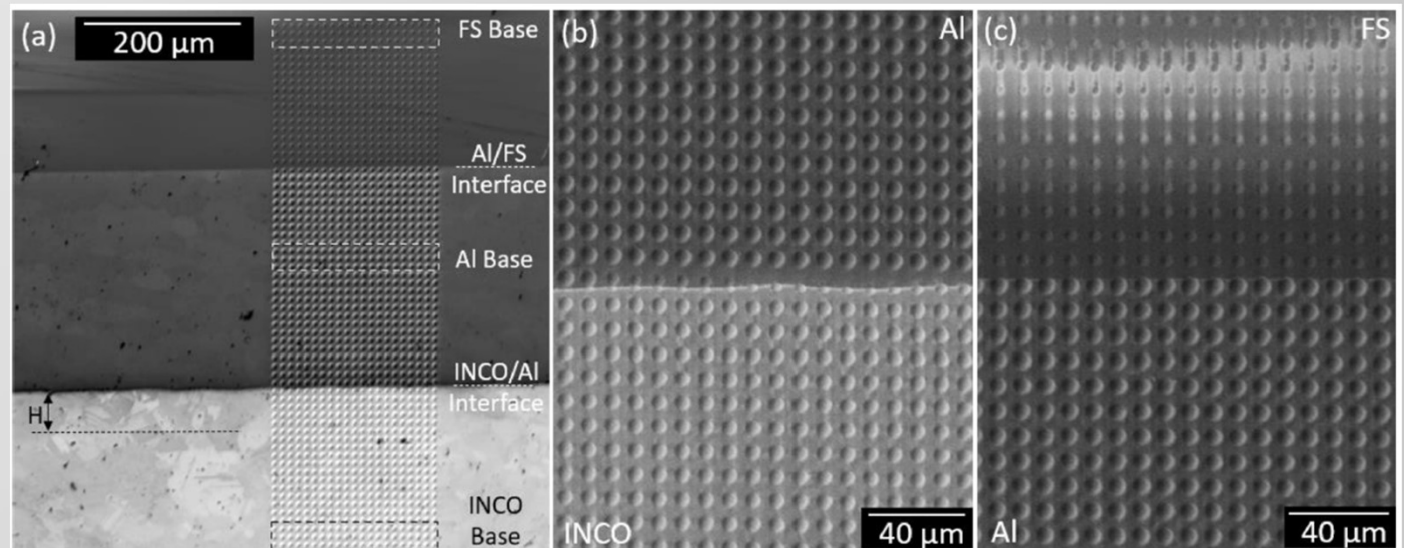
(Macro) $E_{IT} = 288$ GPa



Inconel/Al/Fused Si Diffusion Bonded Joint

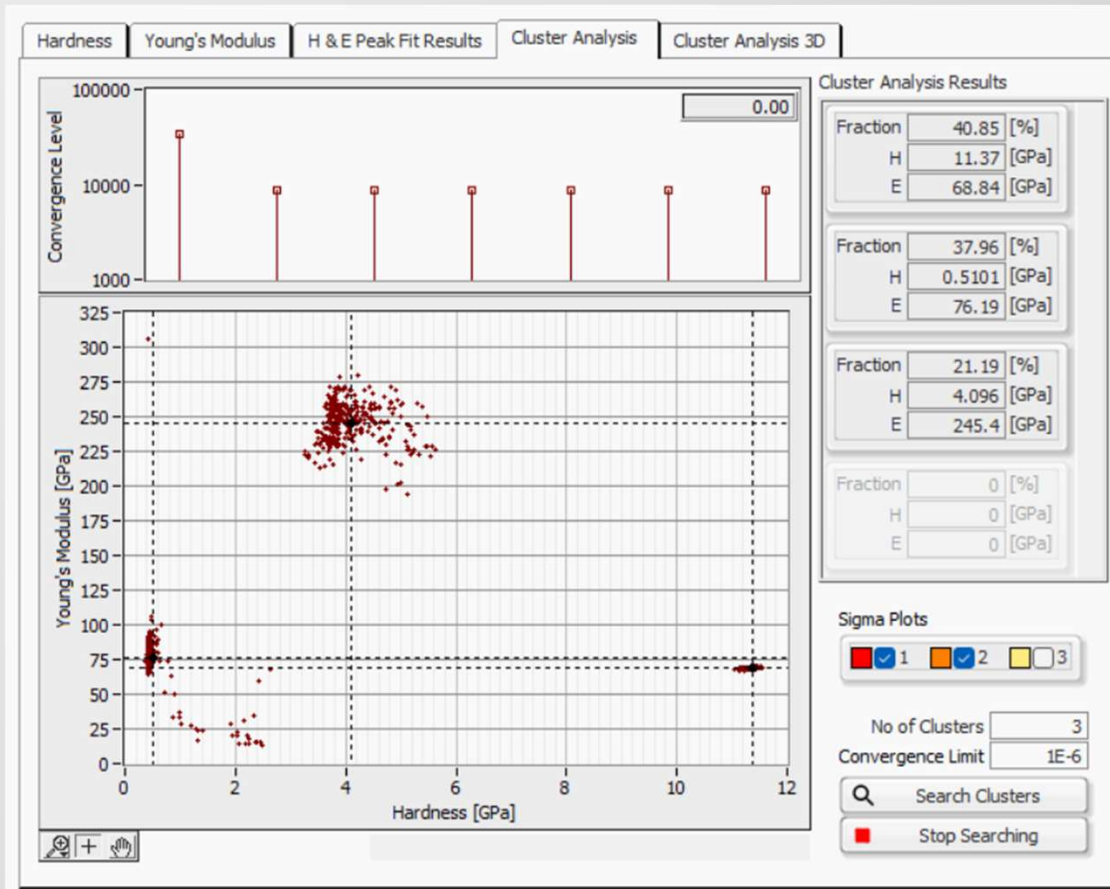


Example of a spherical nanoindentation map (1500 indents)



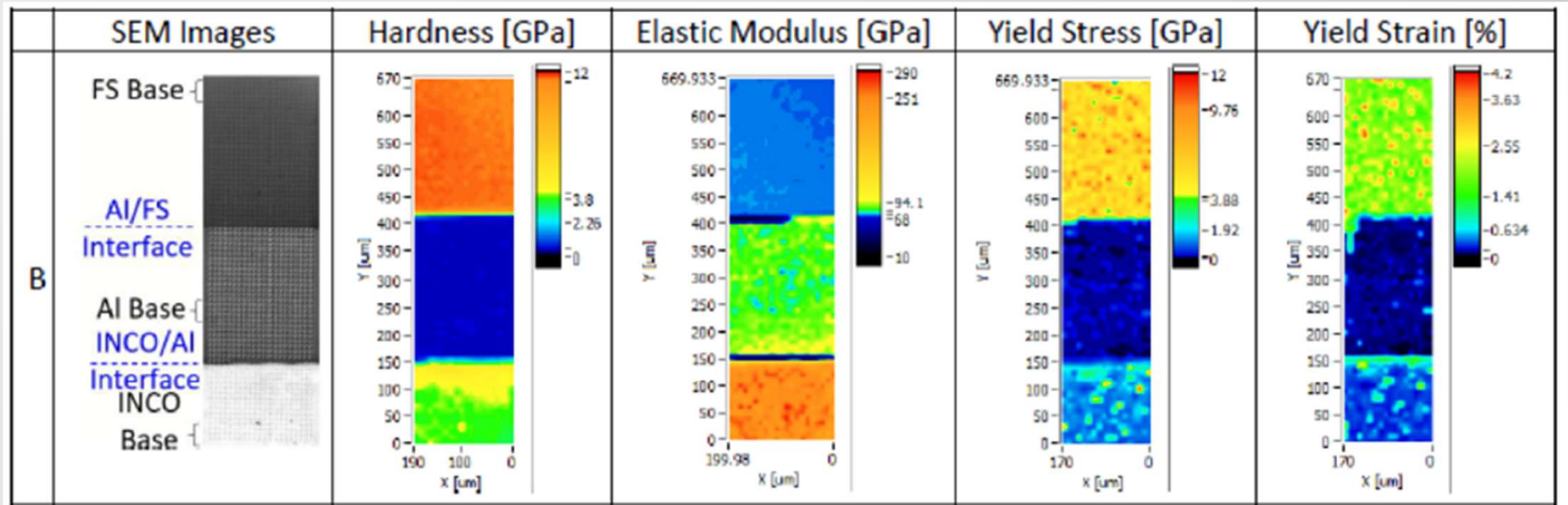
Inconel/Al/Fused Si Diffusion Bonded Joint

Cluster Analysis:



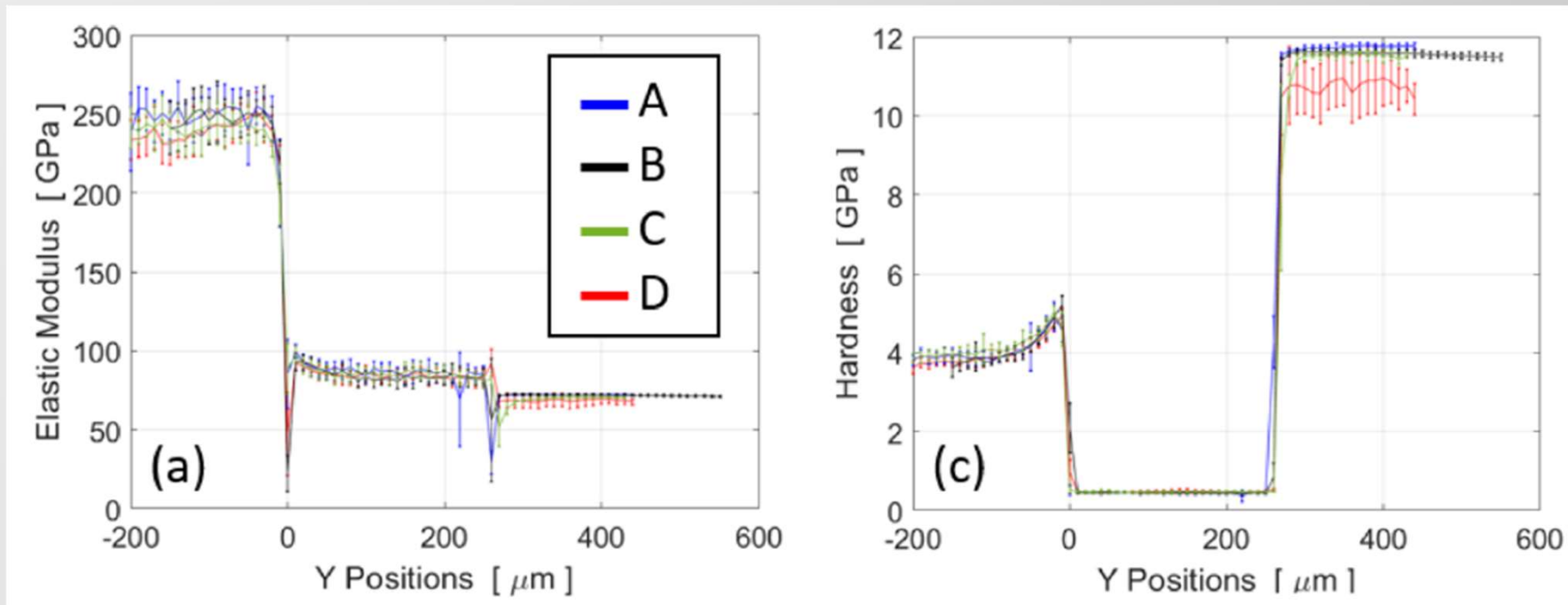
Inconel/Al/Fused Si Diffusion Bonded Joint

Indentation property maps:



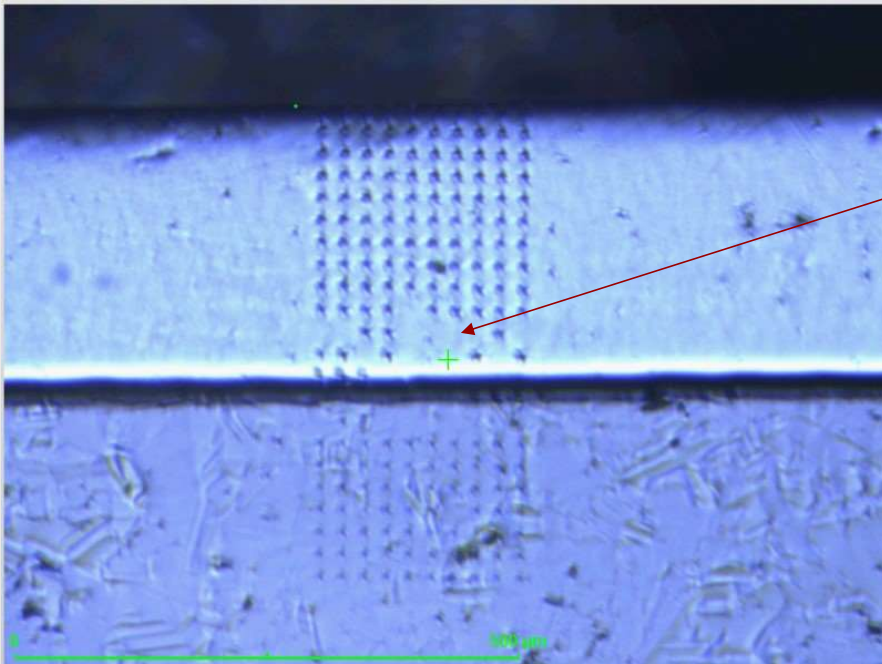
Inconel/Al/Fused Si Diffusion Bonded Joint

H and E typical profiles:

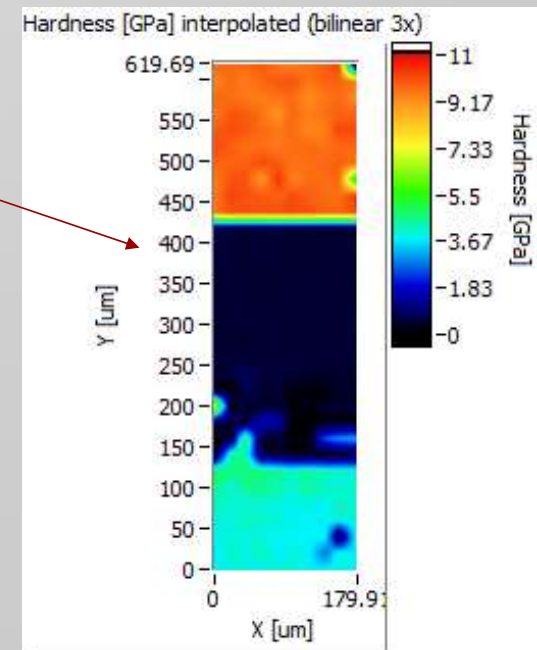


Inconel/Al/Fused Si Diffusion Bonded Joint

Maps do not always go according to plan...! From a previous course..

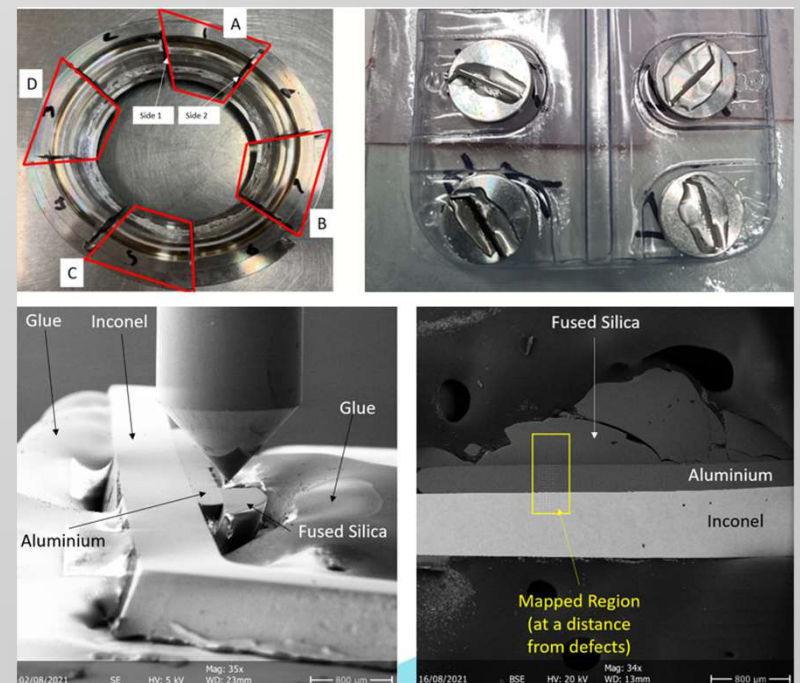
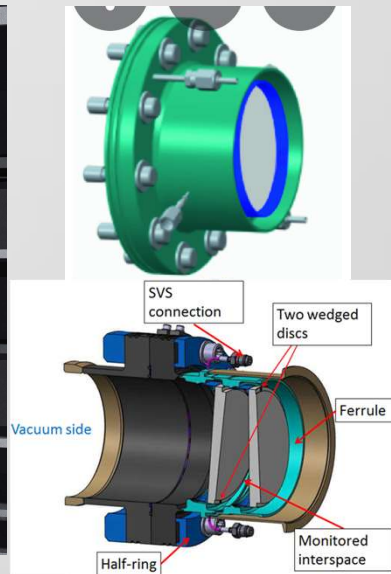
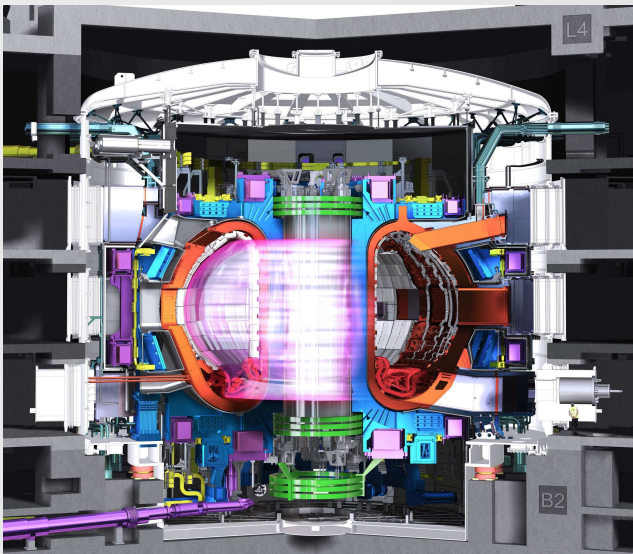


If nanoindentation mapping protocols are not properly designed, some indents may be missing (e.g. typical in noisy environments)



Inconel/Al/Fused Si Diffusion Bonded Joint

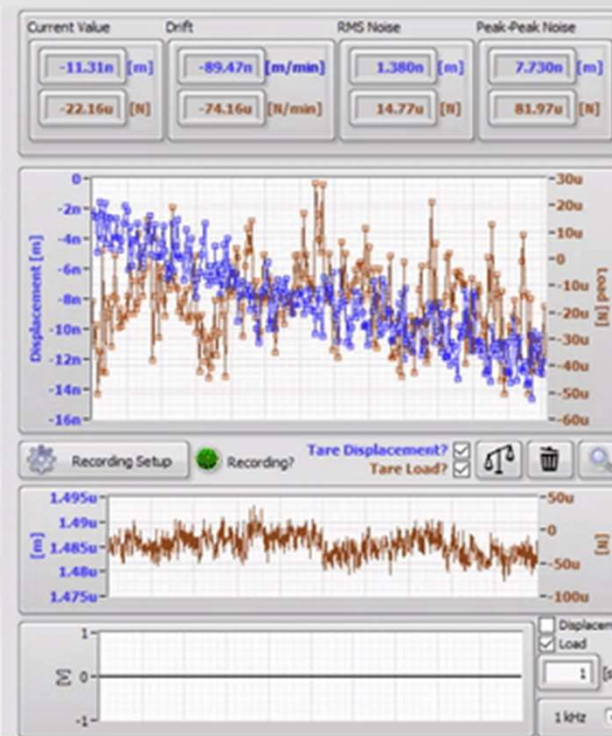
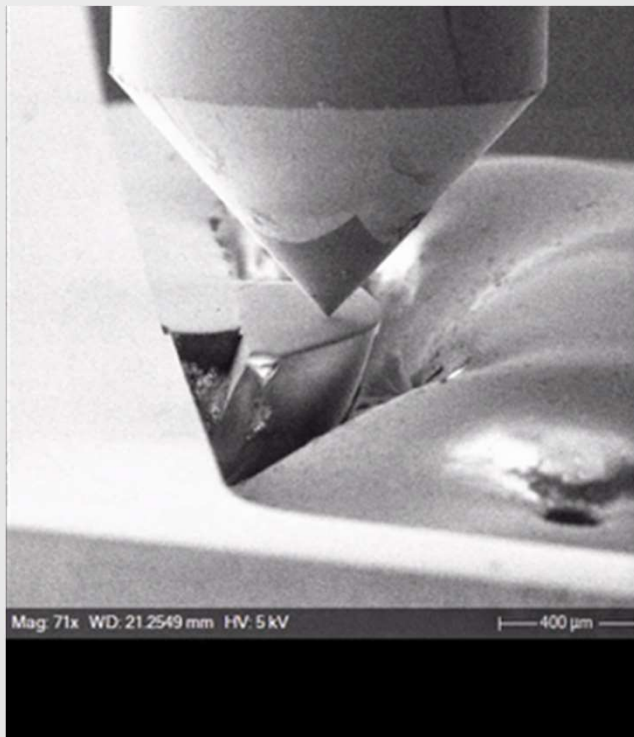
The sample you tested is from the ITER Nuclear Fusion Tokamak reactor...



The ITER tokamak will be the world's largest tokamak, with a plasma radius (R) of 6.2 m and a plasma volume of 840 m³, operating at 150 Million°C and producing 500 MW <https://www.iter.org/mach>

Pero, R., Randall, N., Frey, D., Widmer, R. N., Darby, T., Aucott, L., ... & Bushby, A. (2023). Stress-strain curve mapping by nanoindentation—a technique to qualify diffusion-bonded window assemblies for ITER. *Fusion Engineering and Design*, 196, 113977. <https://doi.org/10.1016/j.fusengdes.2023.113977>

Inconel/Al/Fused Si Diffusion Bonded Joint



2x30 indentation map across the 2 interfaces
32 μm spacing (large spacing)

Load and Displacement vs. Time

Load and Voltage
(applied to the piezostack) vs. Time
At 1 kHz acquisition rate