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Temperature-dependent deformation behavior of highly nanotwinned Cu

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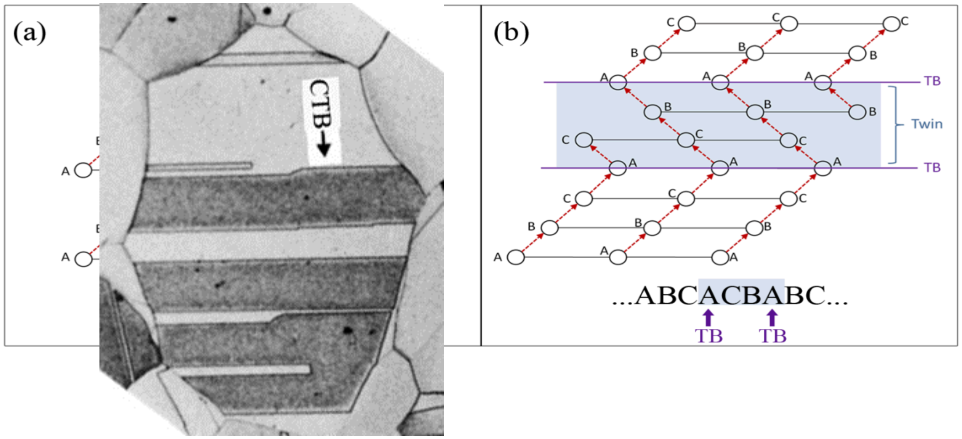
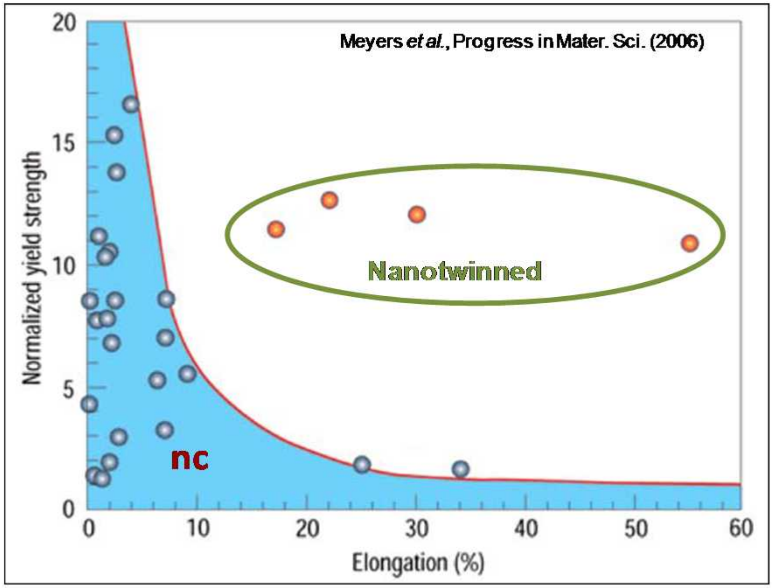
Nanotwinned metals show a potential to simultaneously possess many attractive properties

- A **twin** is a portion of a crystal that takes on a definite, symmetrical orientation relative to the rest of the lattice
- When the twin thicknesses are at the nanoscale (**nanotwin**), unique mechanical properties and plastic responses are observed

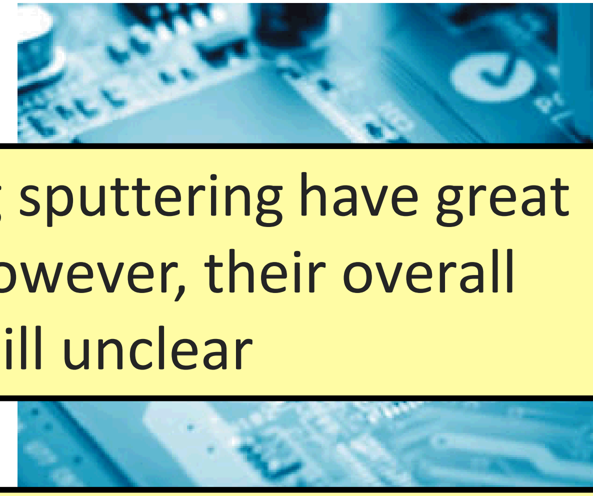




The high density of symmetric and coherent interfaces leads to:

- Ultra-high strengths (similar to nanocrystalline metals)
- Enhanced ductility
- Thermal stability
- Corrosion resistance
- Electrical conductivity

“Growth” nanotwins can be used to develop high-performance metallic coatings



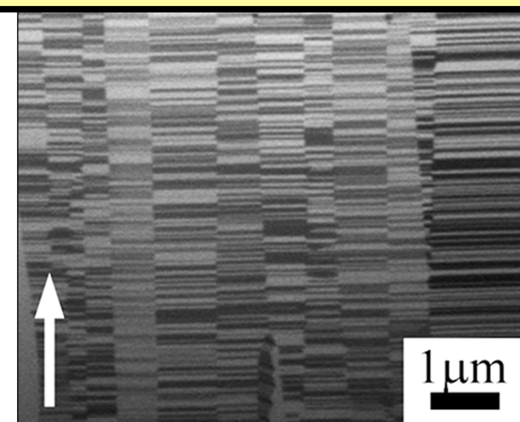
- The unique combination of mechanical, electrical, thermal, and corrosion properties

These distinct structures that form during sputtering have great potential for advanced applications – however, their overall mechanical performance is still unclear

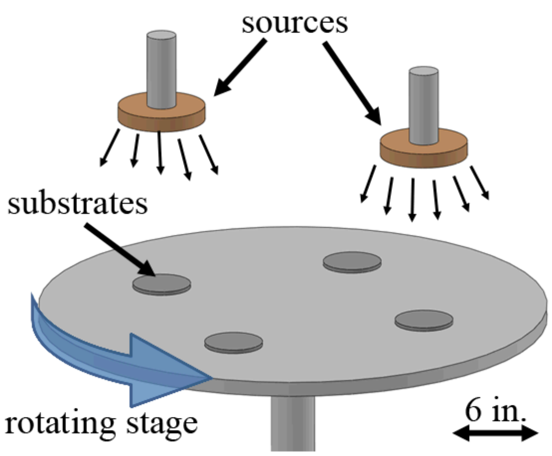
  - MEMS
  - Optical coatings

Understanding the mechanical behaviors of these metals in various environments is critical for their future development

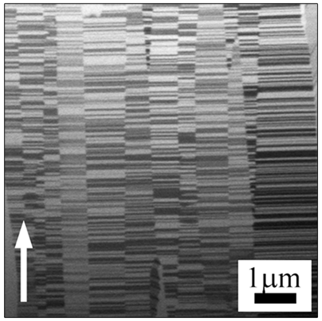
- applications can also be used to synthesize high densities of growth nanotwins
- The typical resulting microstructure:
  - Highly aligned columnar grains
  - High density of nanotwins parallel to substrate

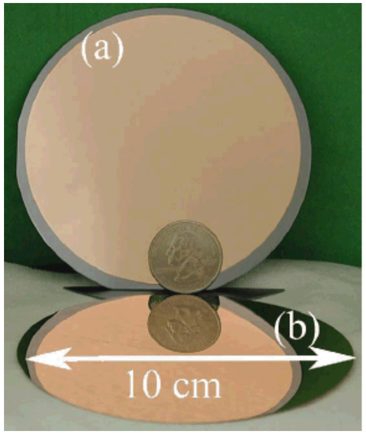


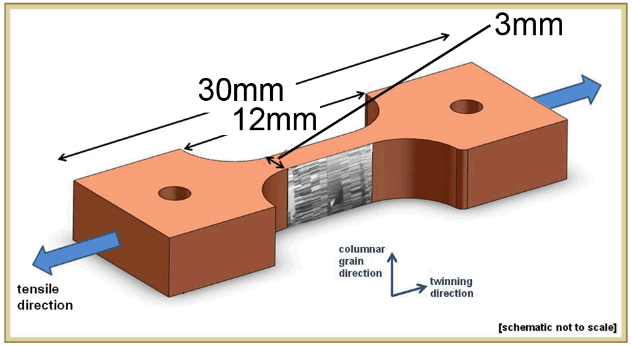
Magnetron sputtering was used to produce highly nanotwinned "thick" foils



- A modified multilayer sputtering approach was developed at LLNL for the synthesis of highly nanotwinned Cu
- Cu-Cu layers (99.99+ % purity) were deposited onto 10 cm diameter Si wafers
- Cu foils (170 µm thick) were removed from the substrates to be free-standing
- Microstructural features:
  - 500-800 nm columnar grain widths
  - 40 nm average twin thickness

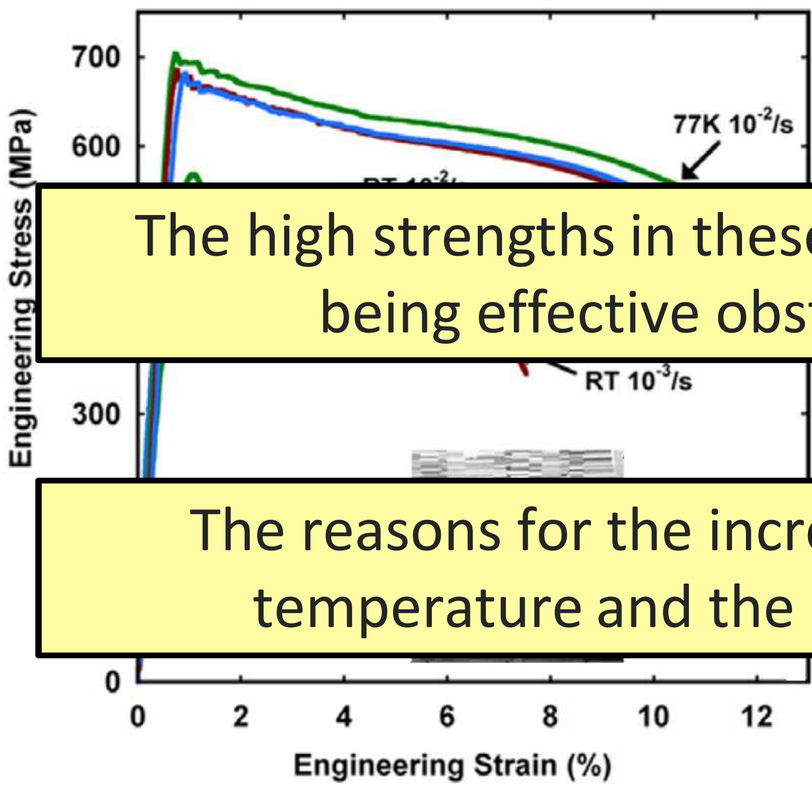






- Tensile dogbones were die-cut
  - Gauge width: 3mm
  - Gauge length: 6mm (measured using non-contact laser extensometer)

Tensile tests performed at RT and 77K revealed atypical stress-strain response



- At both temperatures, strengths are much higher than coarse-grained Cu (~70 MPa) and ultra-fine-grained Cu (~100 MPa)

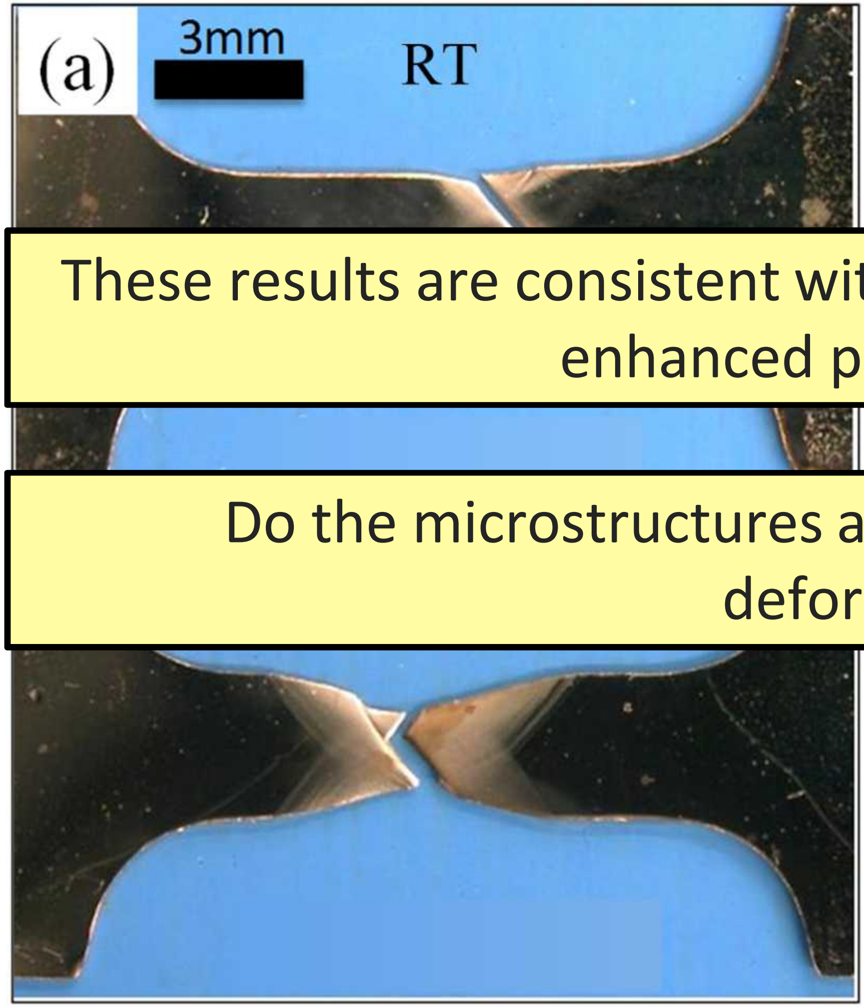
The high strengths in these metals are due to twin boundaries being effective obstacles for dislocation motion

  - One predominant peak at RT
  - Peak followed by serrated stress-strain response at 77K

The reasons for the increased elongation with decreasing temperature and the apparent softening are unclear

- Higher strengths and total plastic strains at 77K

Shear band formation was observed to be the primary deformation mechanism at both temperatures



RT:

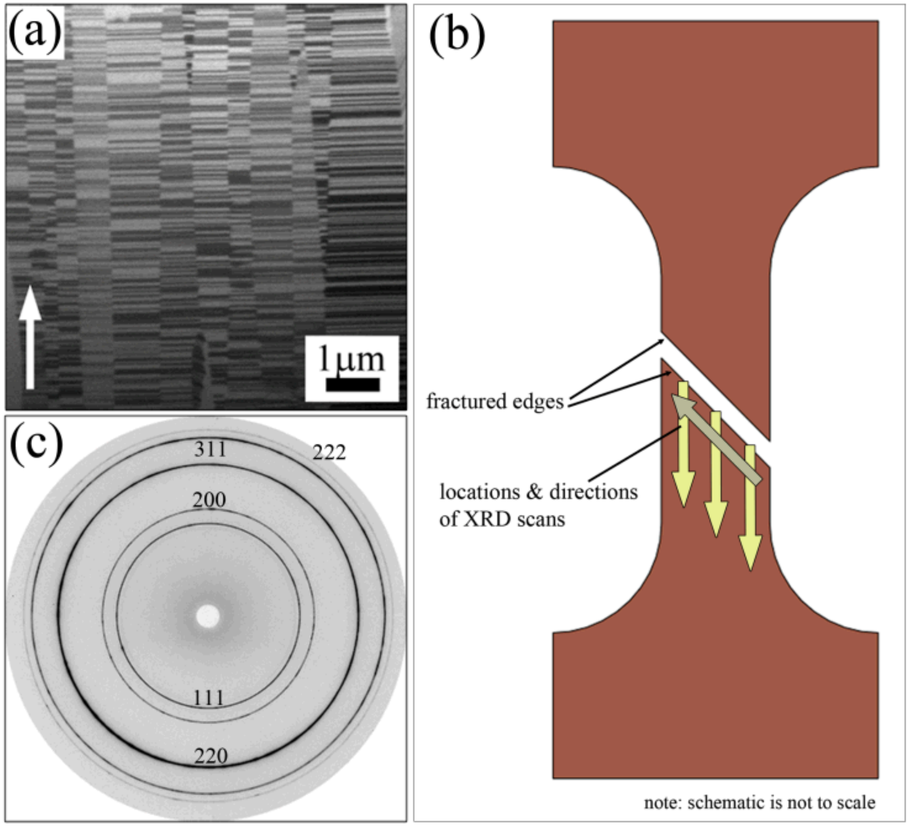
- Two predominant bands with fracture occurring along the primary band (first

These results are consistent with the stress-strain curves showing enhanced plasticity at 77K

Do the microstructures also show evidence of plastic deformation?

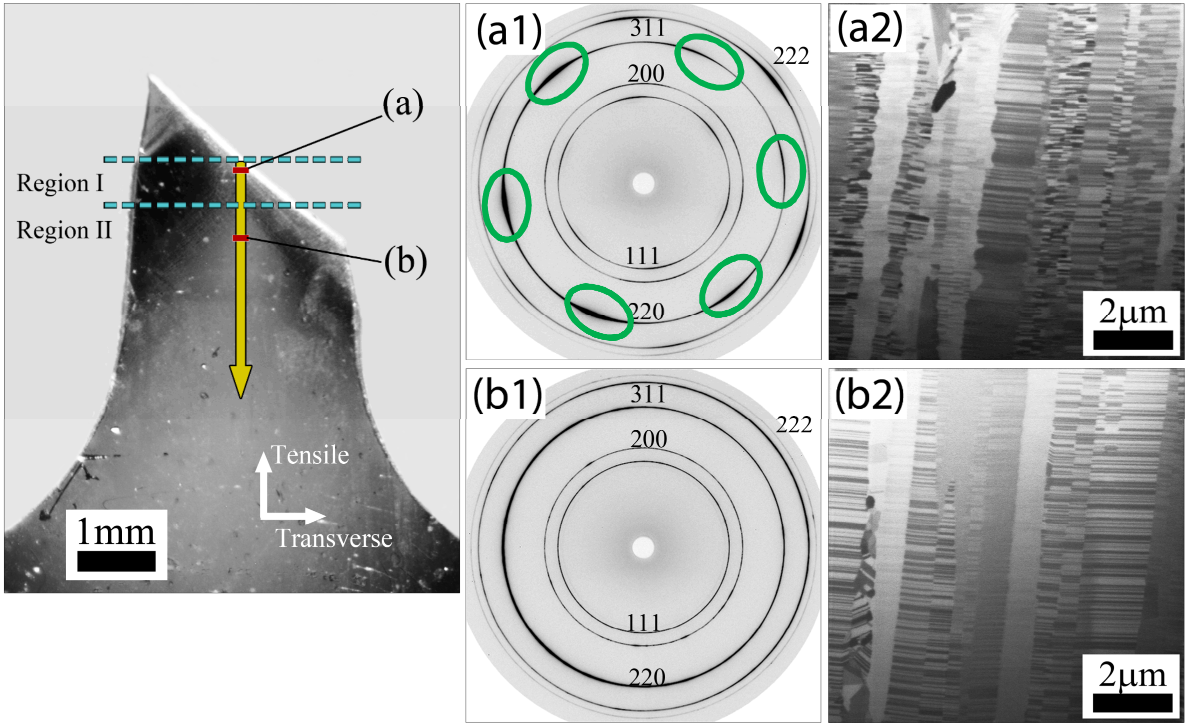
- throughout gauge section
- Much more necking
- Cup-and-cone fracture profile

High energy microdiffraction and focused-ion beam cross-section imaging was used for microstructural investigation



- High Energy Microdiffraction (HEMD) using synchrotron X-ray was used at the ESRF (Grenoble, France)
- High energy (69.7keV) beam was used to transmit the sample thickness and produce full diffraction patterns
- Scans using a microfocused X-ray beam (8 µm x 20 µm) were performed across and along the shear banded regions
- FIB cross-sectional imaging was performed at select locations

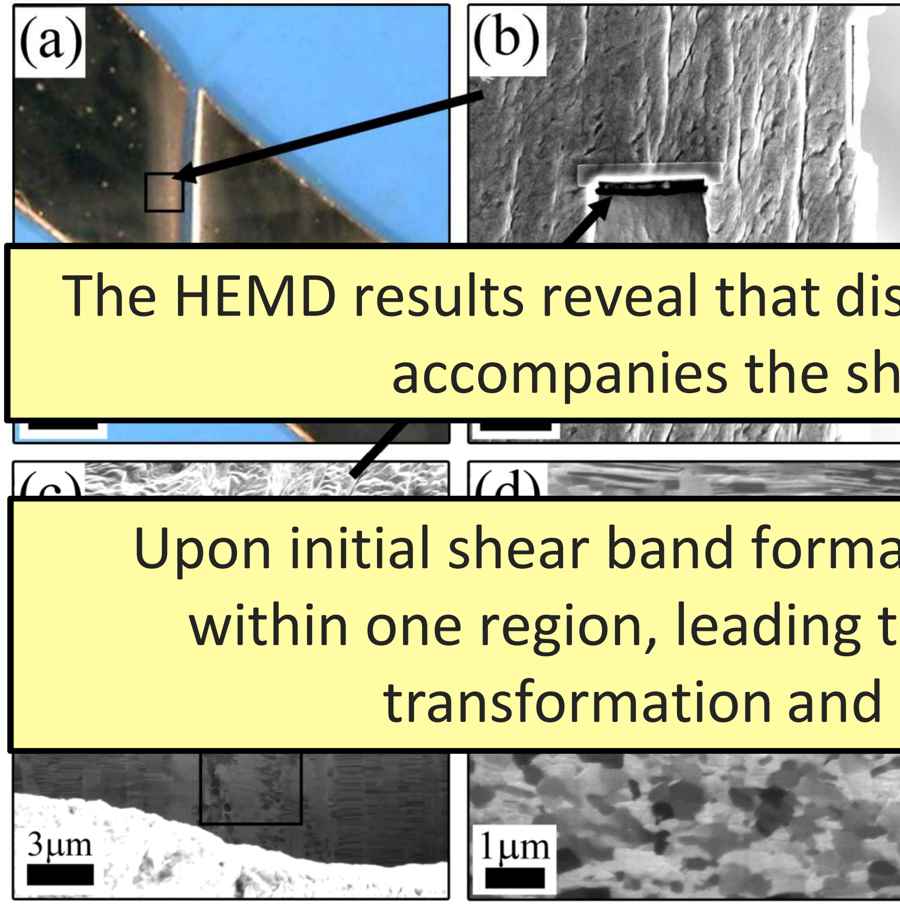
HEMD & FIB of the RT samples highlight grain-rotation and deformation-texturing within shear-banded regions

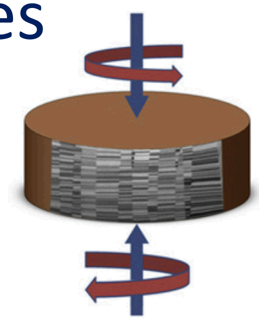


Two distinct regions where the DPs change:

- d < 500 μm : DPs show a deformation texture indicative of in-plane grain rotation (likely dislocation-induced)
- d > 500 μm : DPs and microstructure similar to as-prepared case

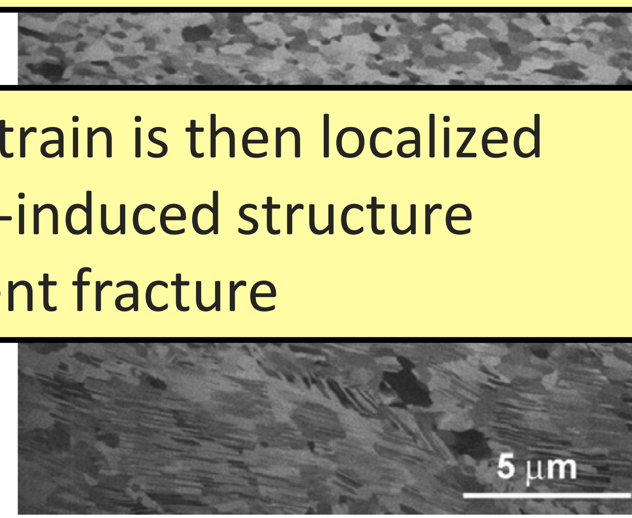
Additional FIB analysis in the RT samples revealed severe detwinning near and along the fracture sites





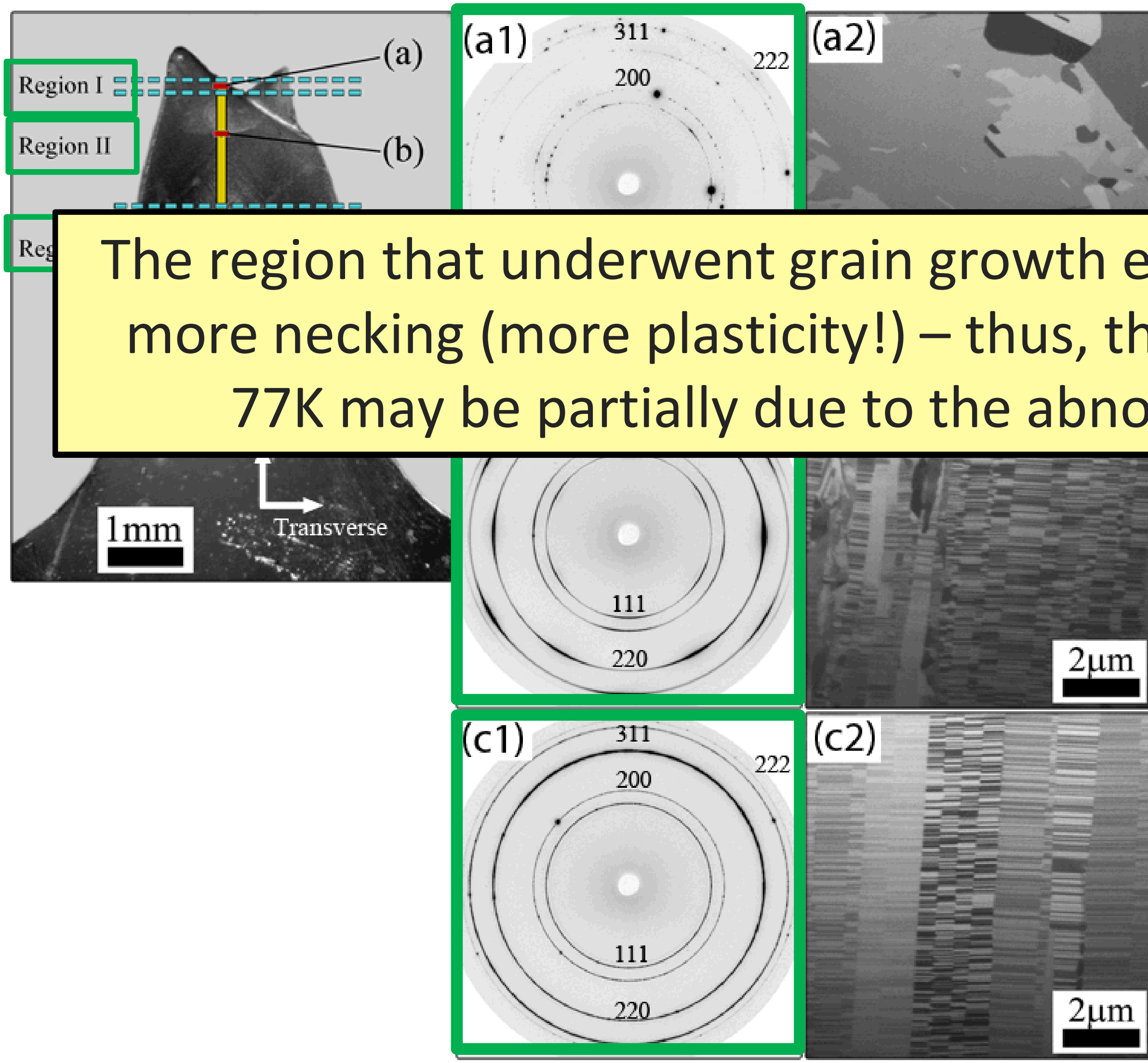
The HEMD results reveal that dislocation-mediated grain rotation accompanies the shear band formation

Upon initial shear band formation, all strain is then localized within one region, leading to a strain-induced structure transformation and subsequent fracture



An complementary high pressure torsion experiment revealed that this structure transformation is **strain-induced** and occurs when **strains are between 0.6 and 1**

77K results showed the same texture within the shear band regions **and** *additional plasticity by abnormal grain growth*



The region that underwent grain growth experienced much more more necking (more plasticity!) – thus, the higher elongation at 77K may be partially due to the abnormal grain growth

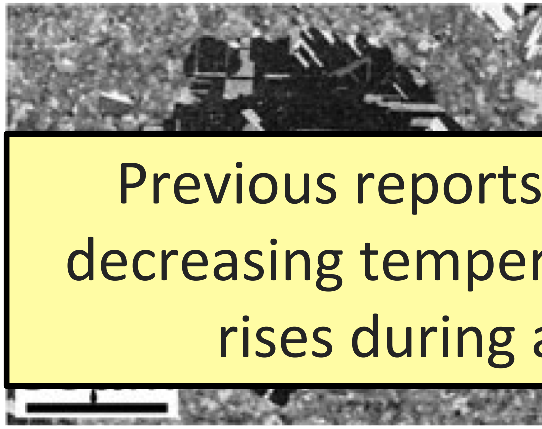
Three distinct regions :

- d < 120 μm : "spotty" DP (cg structured revealed!)

same deformation texture as for the RT sample (in-plane grain rotation)

- d > 1550 μm : DP and microstructure similar to as-prepared case

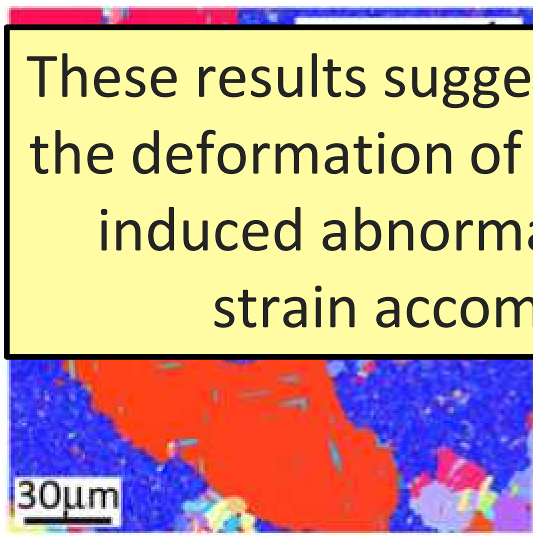
Complementary heat-treatments show abnormal grain growth after ~300K temperature rise



- Heat treatments on these samples revealed abnormal grain growth mechanisms become

Previous reports have shown a decreased heat capacity with decreasing temperature in Cu – this leads to higher temperature rises during adiabatic processes at low temperatures

Adiabatic temperature rise estimates in the sample tested at 77K:



These results suggest that an additional mechanism is active during the deformation of highly nanotwinned Cu at 77K – these adiabatic induced abnormal grain growth processes may lead to further strain accommodation and higher total plastic strains

strain is converted to heat), and all remaining plastic strain after shear band formation is localized to the "necked" region

$$\Delta T = \mathbf{650\ K}$$

Discussion

- Simulations have suggested that when external loads are parallel to the twin boundaries (as in this case), substantial dislocation accumulation occurs at the columnar grain boundaries – these mechanisms are likely responsible for the in-plane grain rotation and texture in the shear banded regions
- The larger total strains at 77K are attributed to:
  - (i) an increased propensity for forming shear bands – multiple shear bands distributed across the gauge section avoids the localization of strain to only one region (as in the RT case), thus delaying fracture
  - (ii) the activation of additional thermo-mechanical mechanisms [i.e. the combination of adiabatic temperature rises and an increased dislocation debris (often found during low-temperature deformation) that promotes abnormal grain growth]
- Fracture in the RT samples occur by first transforming the microstructure into a less mechanically stable detwinned structure – we hypothesize that these are strain-induced grain boundary transport mechanisms, but this requires further work
- The underlying mechanisms leading to the shear-band formation are still unclear – this will likely require large-scale MD simulations to quantitatively capture the dynamics in the deformation process (in particular, the relationship between the grain rotation and shear banding)

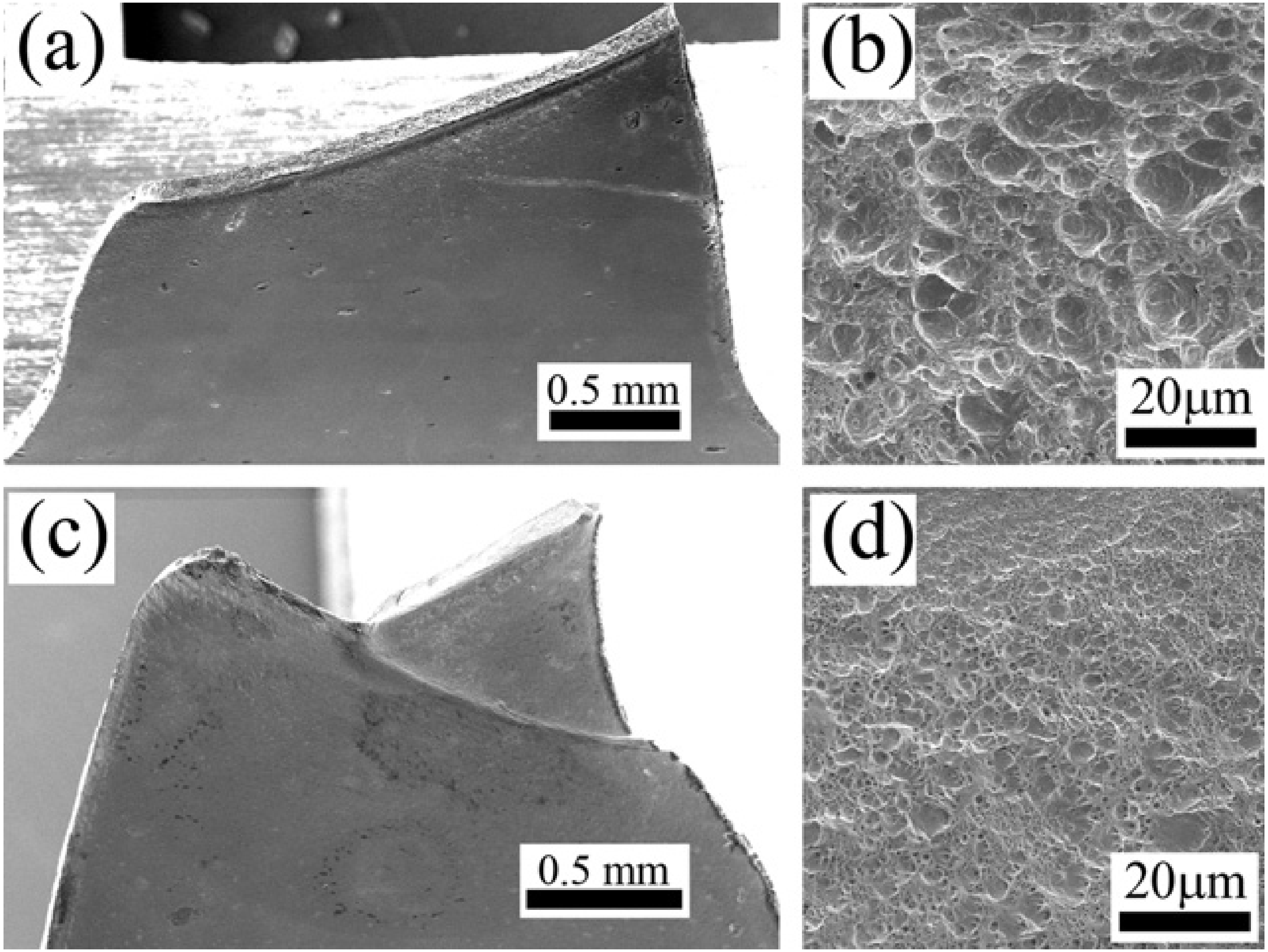
Conclusions

- The unique nanotwinned structures that form during sputtering show impressive strengths (up to 10 times that of coarse grained metals)
- Atypical plastic behavior was observed, including yield peak phenomena and deformation by shear banding
- Dislocation-mediated in-plane grain rotation was shown to accompany the shear banded regions
- An overall increase in plasticity was observed with decreasing temperature – at low temps, additional plastic mechanisms are active, including an increased shear band propensity and abnormal grain growth
- The improved mechanical performance, in particular at low temperatures, will lead to the further develop of these structures for advanced coatings applications

- NSF grant number NSF-DMR-0955338
- LLNL – sample preparation and mechanical testing
- ESRF – synchrotron micro-diffraction

Thank you

Extra Slides


(a)
0.5 mm
(b)
20μm
(c)
0.5 mm
(d)
20μm