DAMAGE AT THE INTERFACE BETWEEN NANOTWINNED AND DE-NANOTWINNED REGIONS IN FATIGUED NANOTWINNED Cu

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OUTLINE

BACKGROUND: Mostly nanocrystalline Cu

NANOTWINNED Cu: Description

FATIGUE:

- Effect of fatigue on nanotwinned Cu
- Structural and Strength Changes
- Crack Formation & Interfacial damage

FIRST "PRACTICAL" METHOD OF MAKING NANOCRYSTALLINE SAMPLES, AND THEIR FIRST PROPERTY MEASUREMENTS, WERE ANNOUNCED BY H. GLEITER AT AN ANNUAL RISØ CONFERENCE IN THE 1980s

A FRENZY OF RESEARCH IN THE AREA FOLLOWED

("NANOCRYSTALLINE" MEANS GRAIN SIZE ~ < 100 nm)

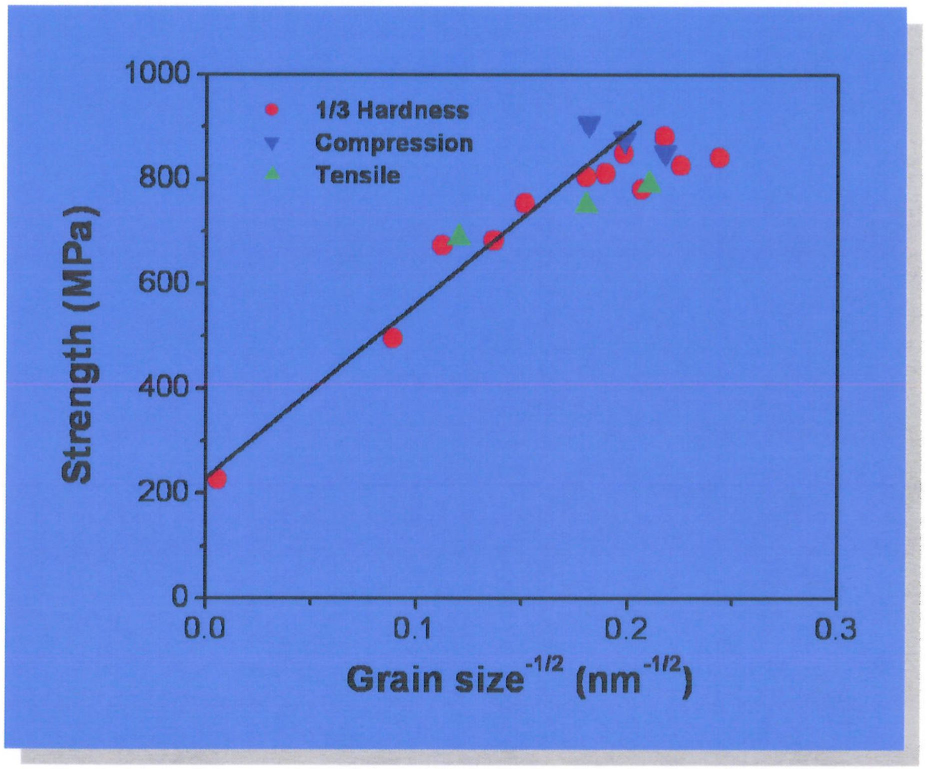
WHY THE GREAT INTEREST IN NANOSTRUCTURED METALS?

- HALL-PETCH PREDICTION

  $$\sigma_y = \sigma_o + k/\sqrt{d}$$

- NEW DEFORMATION MECHANISMS MUST COME INTO PLAY

Hall-Petch Behavior of Various Nanocrystalline Cu Samples

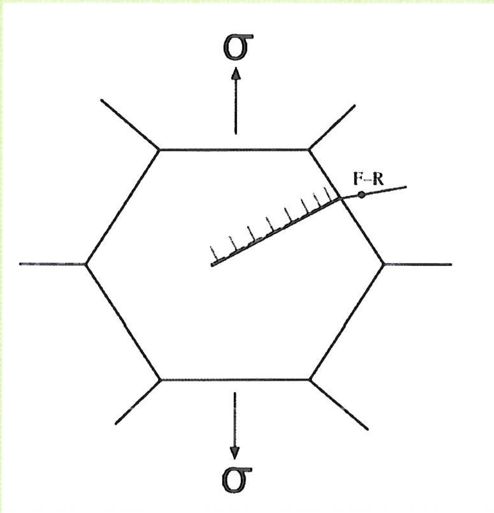


(straight line = extension of coarse-grain data)

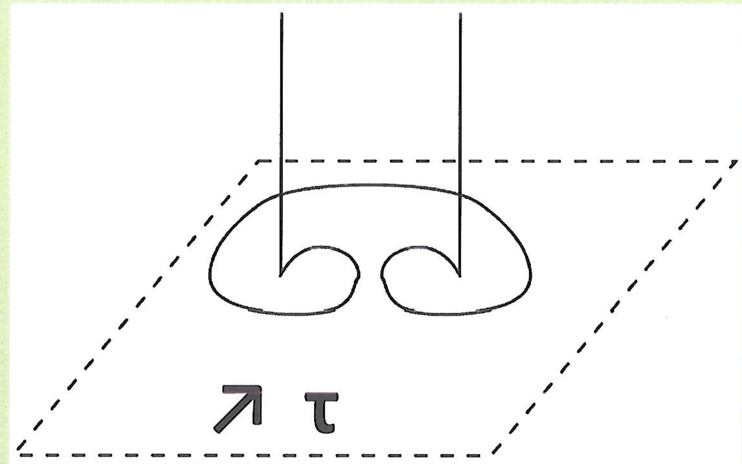
- Summary of different synthesis routes (**ball-milling, surface attrition, & IGC**) and measurement methods (**compression, tension, & hardness**)

Mechanisms For Plastic Deformation

Coarse-Grain Concepts



Cottrell
Hall-Petch Equation



Frank-Read Source

To activate F-R source, shear stress τ ~ 2Gb/d

Contributions of Molecular Dynamics Simulations

- Reveals deformation processes on an atomic scale
- Shows behavior at very small grain sizes (< ~10 nm), where synthesis is difficult
- Computer samples are free from flaws that mask intrinsic behavior in experiments

A major finding: Dislocations are emitted from & absorbed into grain boundaries (even at small grain sizes!)

EXTENSIVE GB ENERGY IN NANO METALS PROMOTES MICROSTRUCTURAL INSTABILITY

*** SYNTHESIS METHODS FOR NANO METALS INVOLVE A "DRIVEN" TECHNIQUE (SCHUH)**

***SPONTANEOUS GRAIN GROWTH SEEN AT RT IN HIGH PURITY NANO METALS†**

†Günther, Kumpmann and Kunze, 1992
Weissmüller, Löffler and Kelber, 1995

OBVIOUSLY, NANOCRYSTALLINE METALS HAVE PROBLEMS!

STRUCTURAL INSTABILITY
EXTREMELY BRITTLE
LOW ELECTRICAL CONDUCTIVITY

NANO-TWINNED METALS, INTERESTING MICROSTRUCTURES

- **HIGH STRENGTH*, ASSOCIATED WITH SPACING BETWEEN TWIN BOUNDARIES**
- **HIGHER DUCTILITY THAN EQUIVALENT 3-D**
- **GOOD STRUCTURAL STABILITY AT HIGH T****
- **HIGH ELECTRICAL CONDUCTIVITY***

UNDER CYCLIC (AND OTHER) STRESSES?

** Lu, Chen, Huang, Lu, Science 323 (2009) 607*

*** Anderoglu, Misra, Wang, Zhang, JAP (2008) 094322*

A SPECIAL NANO-TWINNED MICROSTRUCTURE: COLUMNS OF ALIGNED TWINS

//////////////////
\\\\\\\\\\\\\\\\\\ ↑ GROWTH DIRECTION
////////////////// Σ 3 TWIN BOUNDARIES

PROCESSED USING NANOLAMINATE TECHNOLOGY

- Foils coated on standard silicon wafers by DC magnetron sputtering
- Foil thickness = 170 µm, self-supporting
- Ultrahigh purity Cu targets, argon gas

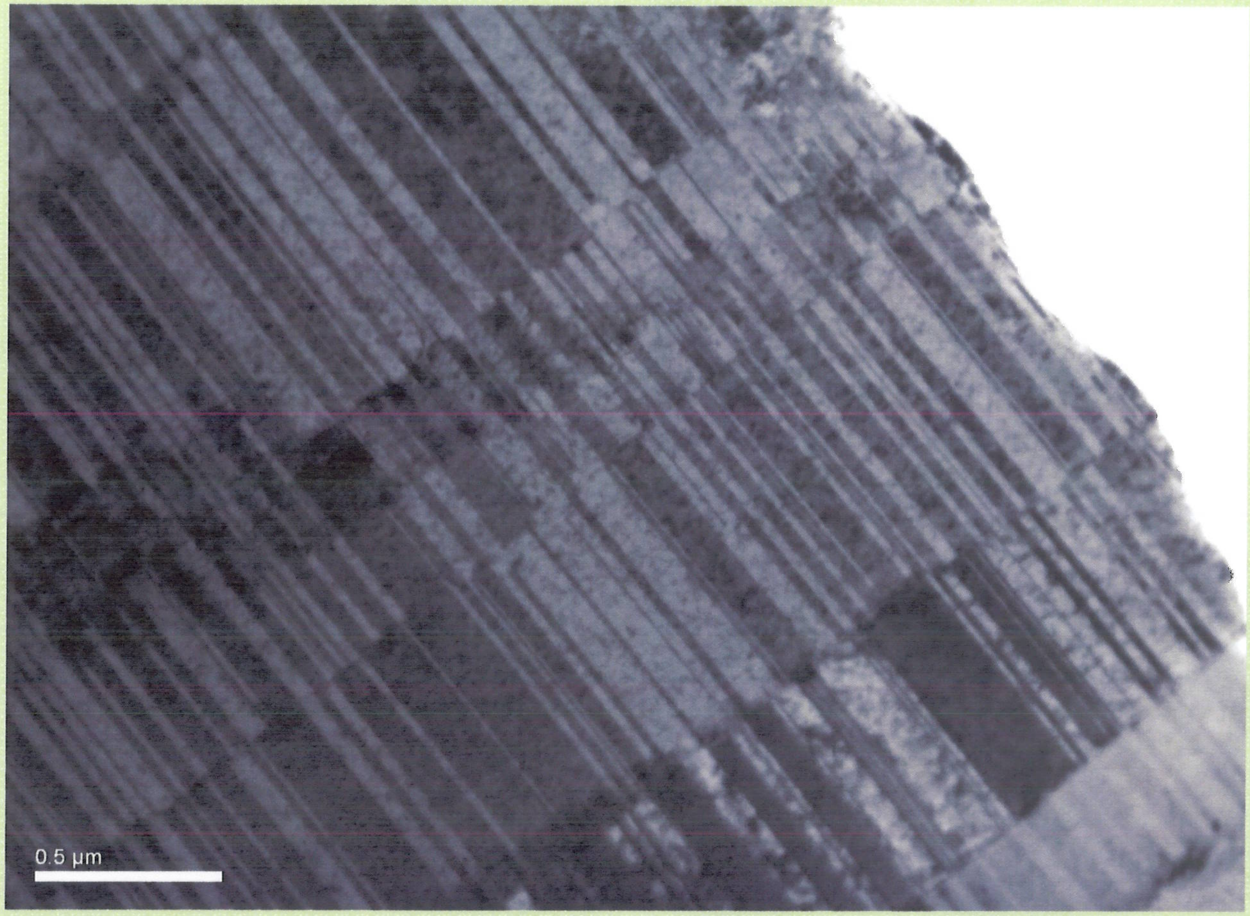
- *(Hodge, Wang and Barbee, MatsSciEngA 2006)*

MODES OF DEFORMATION STUDIED

- TENSION-TENSION FATIGUE, DOGBONE SAMPLES
- COMPRESSION-COMPRESSION FATIGUE, DISKS
- EXTREME COMPRESSION, DISKS
- INDENTATION, COMPLEX STRESS STATE
- HIGH PRESSURE TORSION (HPT)

- TRANSVERSE SECTIONS STUDIED BY FIB + TEM

NANOTWINNED Cu, AS RECEIVED TEM IMAGE



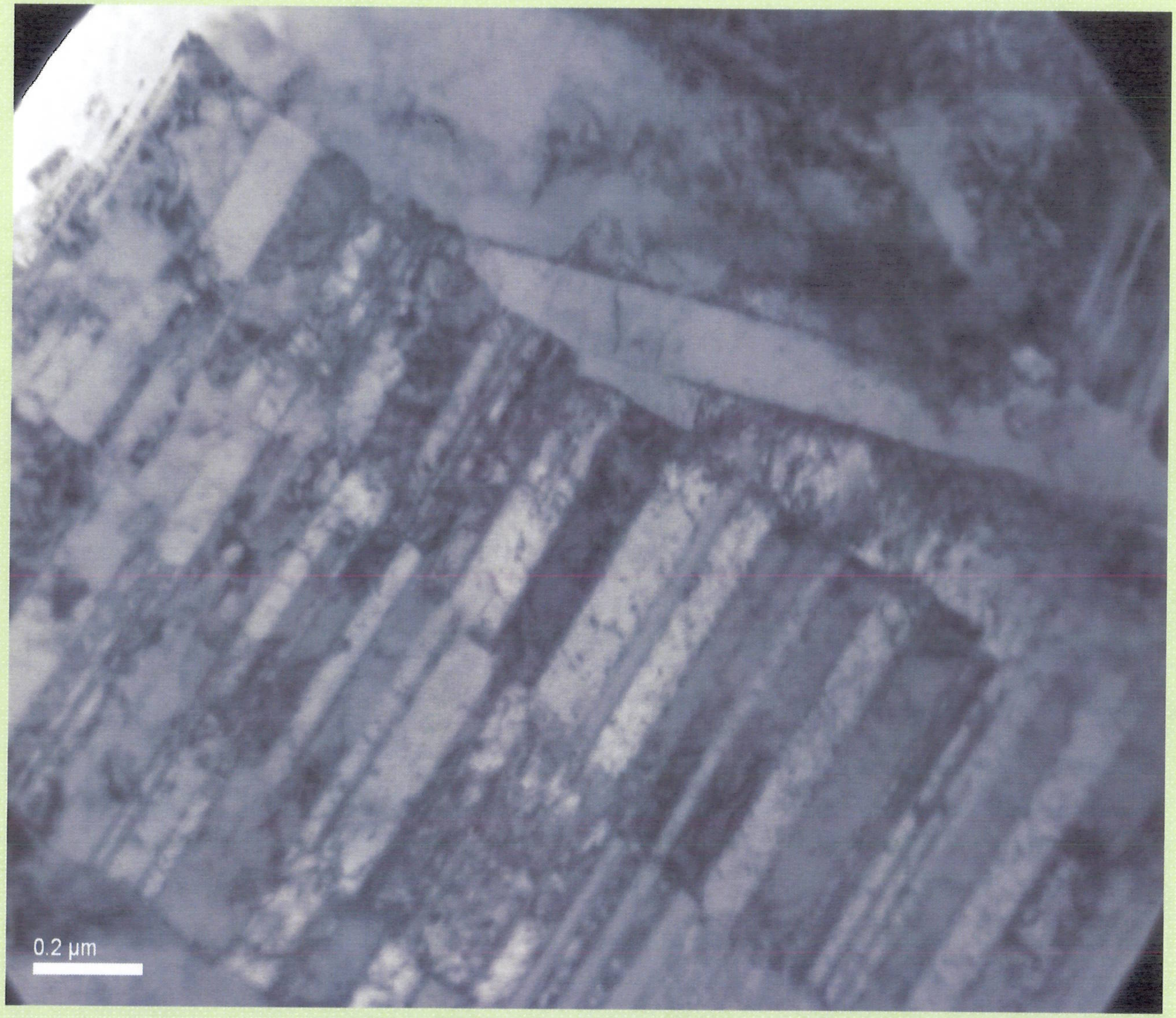
Max Stress = 450 MPa,5195 Cycles

TENSION-TENSION FATIGUE

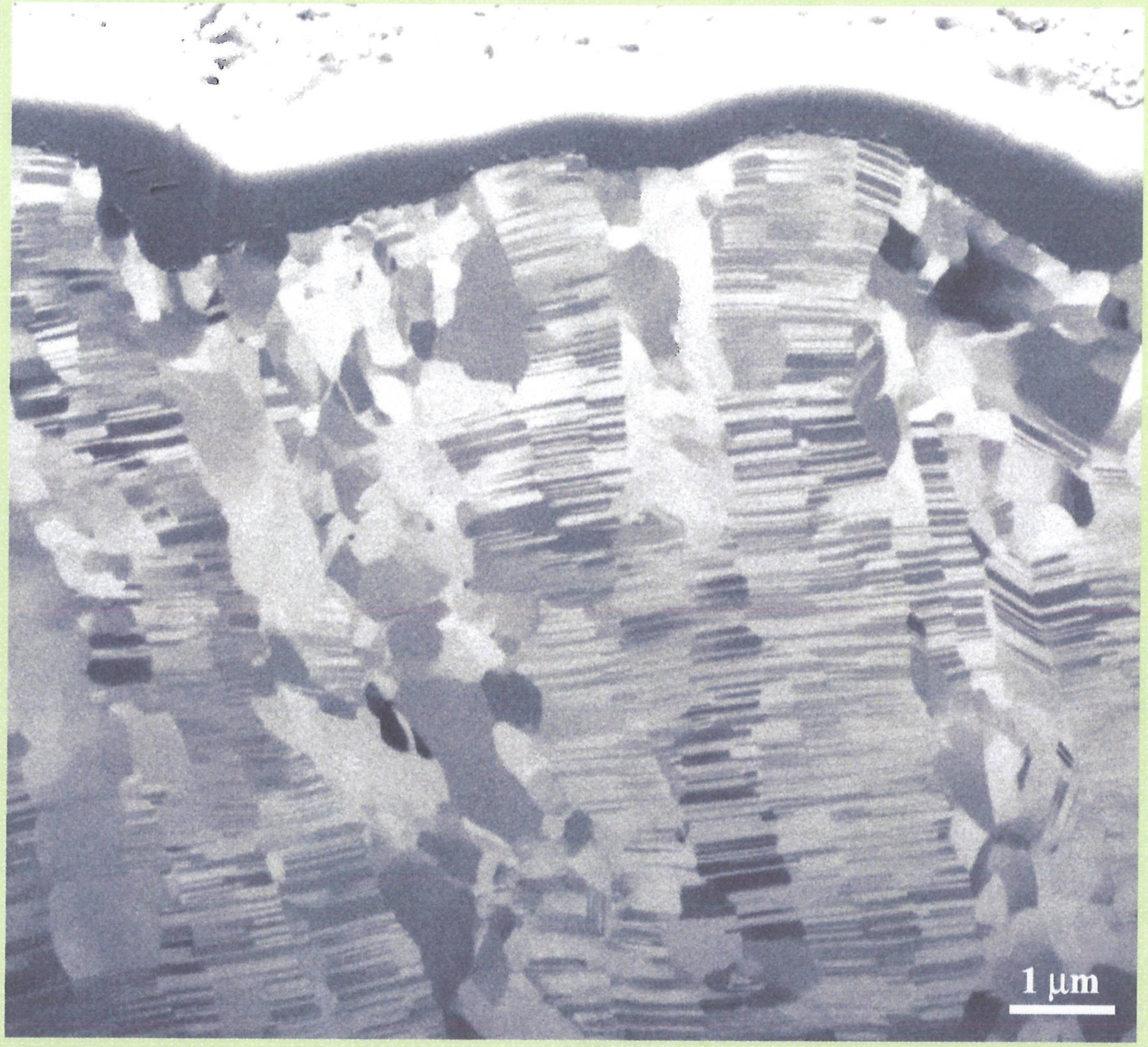
TEM IMAGE

NOTE DISLOCATIONS BETWEEN ADJACENT TWIN BOUNDARIES

Fatigued to failure at 450 MPa max stress

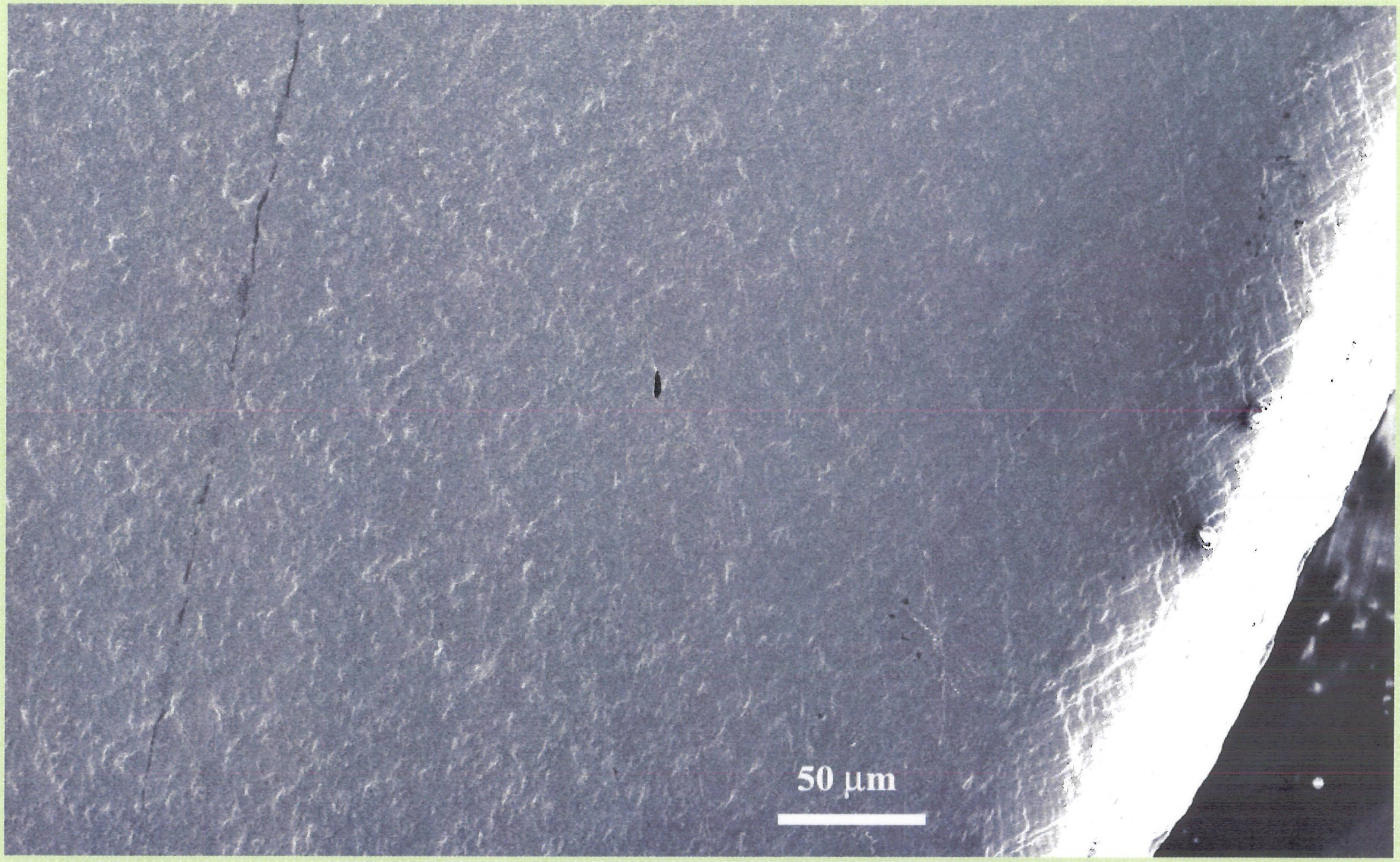


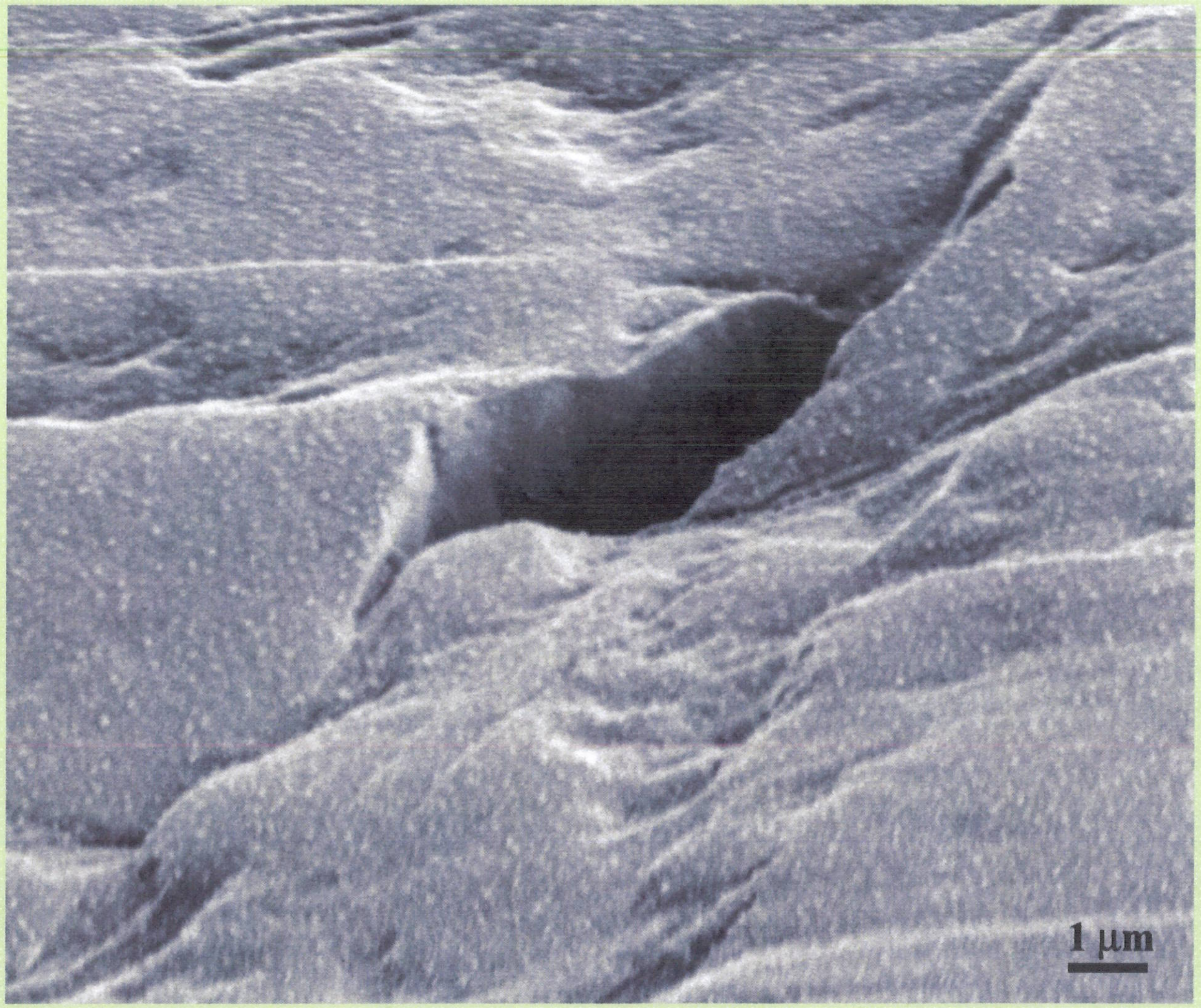
SURFACE DIPS WHERE Cu IS DE-NANOTWINNED



FATIGUED AT 450 MPa MAXIMUM STRESS

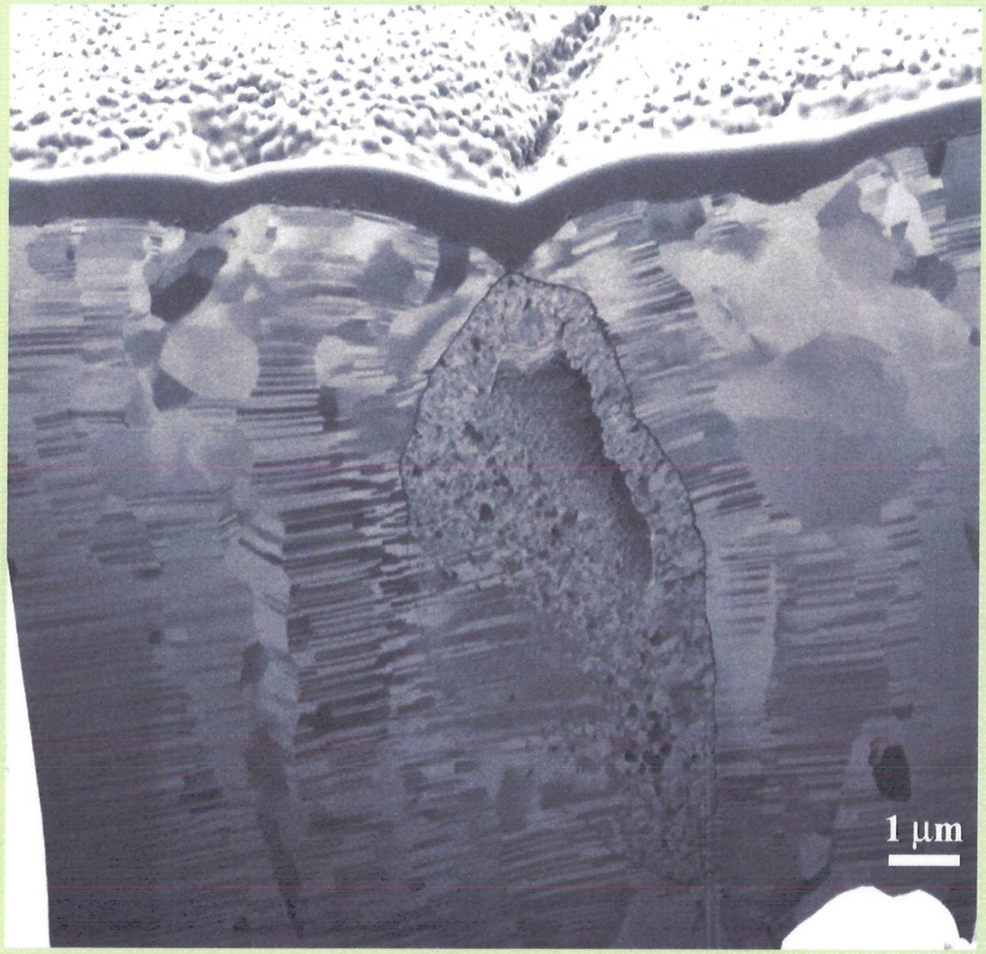
TWO SETS OF PARALLEL SURFACE DEPRESSIONS ON FATIGUED SAMPLE; NOTE SAMPLE EDGE FOR ORIENTATION, CRACK





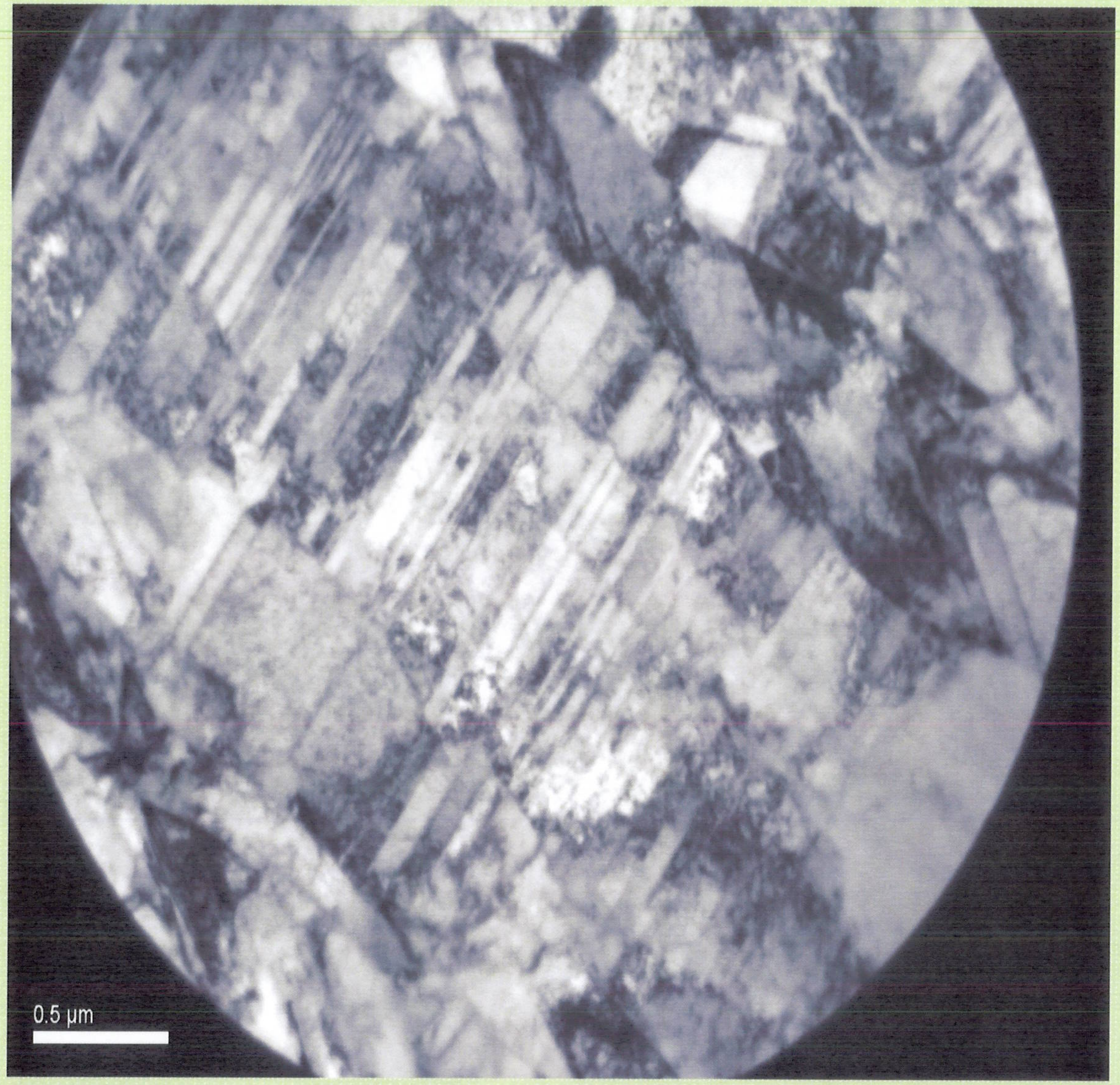
CRACK AT INTERSECTION OF TWO SURFACE DEPRESSIONS IN FATIGUED Cu

HOLE BELOW SURFACE, NOTE DIP AT SURFACE FATIGUED AT 450 MPa MAX STRESS



TRANSVERSE FIB CUT THROUGH SURFACE CRACK IN FATIGUED COPPER

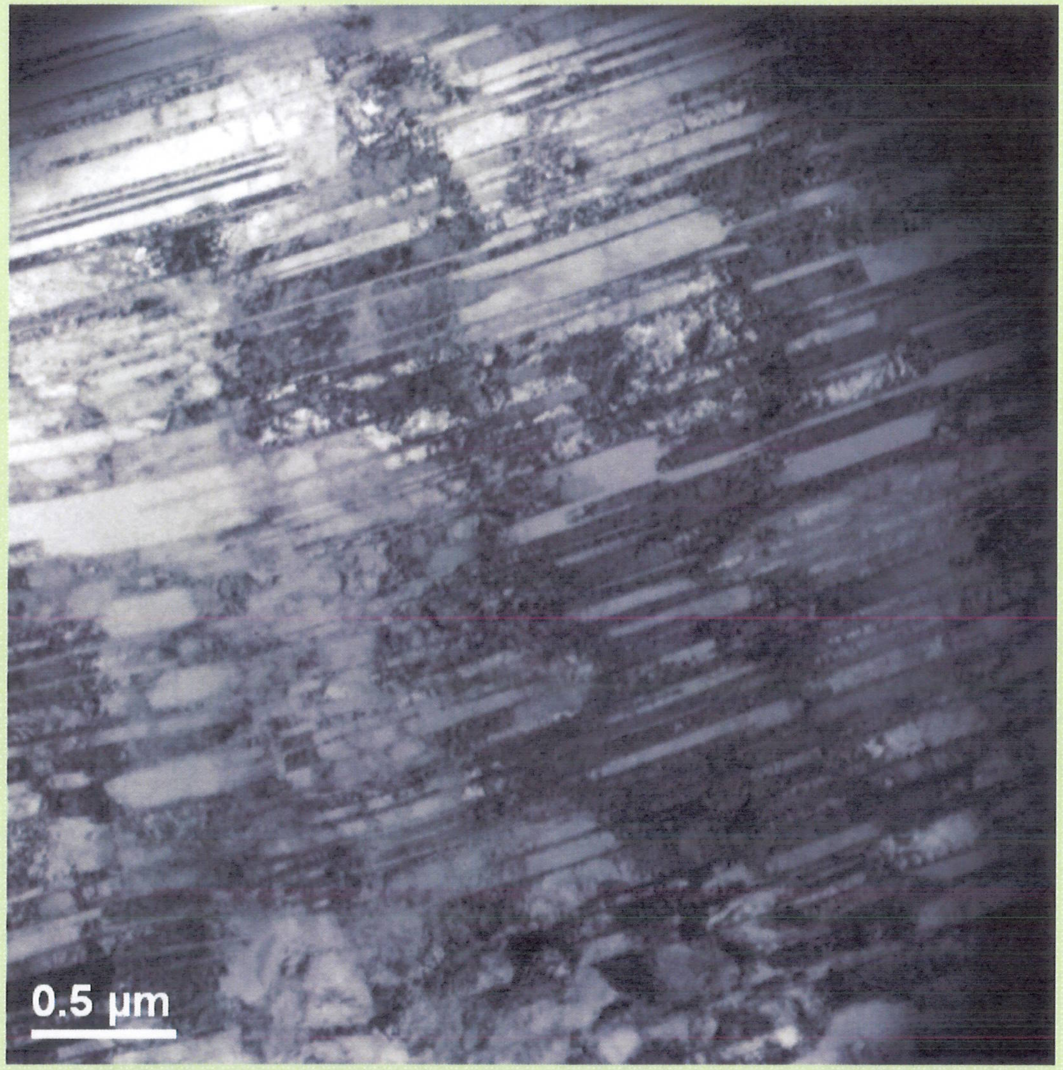
DISLOCATION BUILD UP AT INTERFACES BETWEEN DIFFERENT MODES OF DEFORMATION



Fatigued to failure at 450 MPa max stress

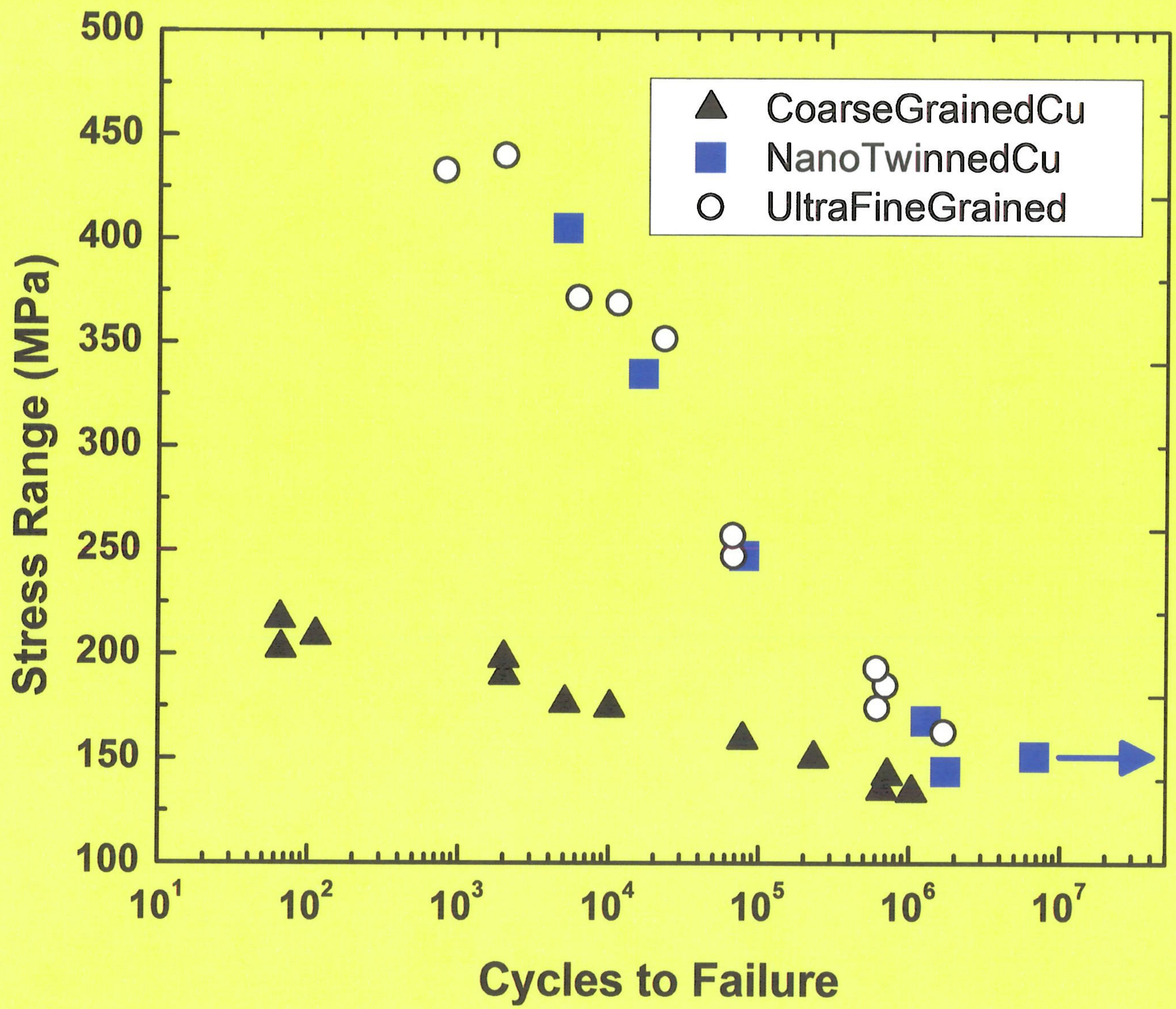
DISLOCATION BUILD UP AT COLUMN BOUNDARIES

Sample fatigued at 450 MPa



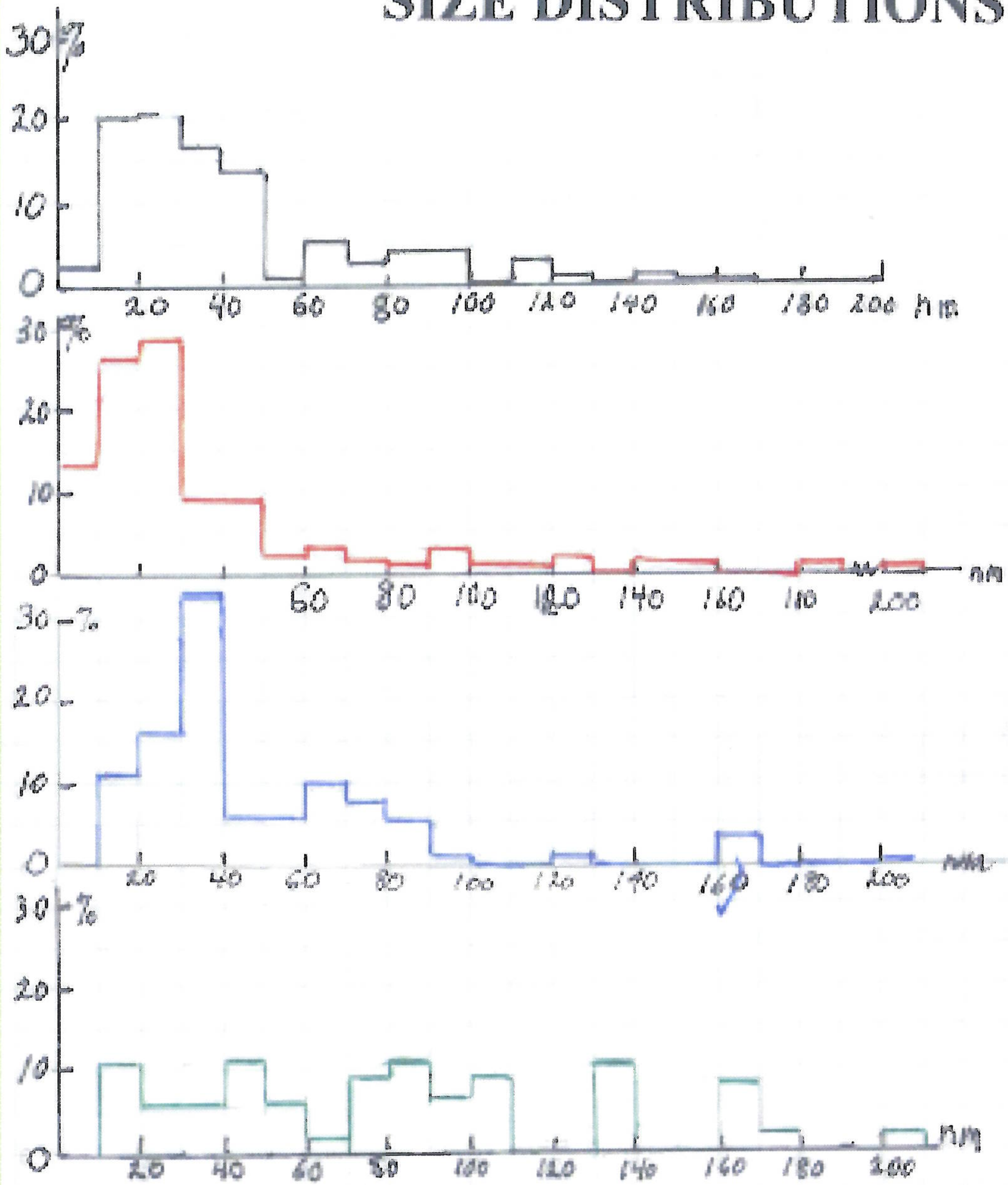
[redacted]OF DEFORMATION ON HARDNESS

	HARDNESS (GPa)
[redacted]ssed	1.8-1.9
[redacted]sed to 3840 MPa	1.8
[redacted]sion fatigue, 5000 cycles	
[redacted] -56 MPa	1.8
[redacted]nsion-tension	
[redacted]37 MPa, 20,000 cycles	1.7-1.8
[redacted]nsion-tension	
[redacted]45 MPa, 5195 cycles	1.7



CoarseGrainedCu
NanoTwinnedCu
UltraFineGrained
Stress Range (MPa)
500
450
400
350
300
250
200
150
100
10^1
10^2
10^3
10^4
10^5
10^6
10^7
Cycles to Failure

SIZE DISTRIBUTIONS OF TB SPACINGS



As received, median TB spacing = ~ 35⁺ nm

Compressed, -25% strain, median spacing ~ 25⁺ nm

Fatigued 450 MPa max stress median spacing ~ 35⁺ nm

Fatigued 450 MPa, near crack median spacing ~ 50 nm

SUMMARY

Nanotwinned Cu combines strength and stability

However, especially under fatigue conditions, de-twinning can occur, leading to deformation incompatibilities and crack nucleation