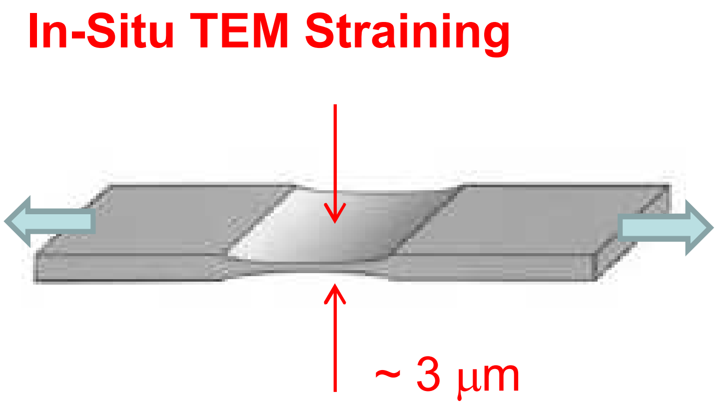
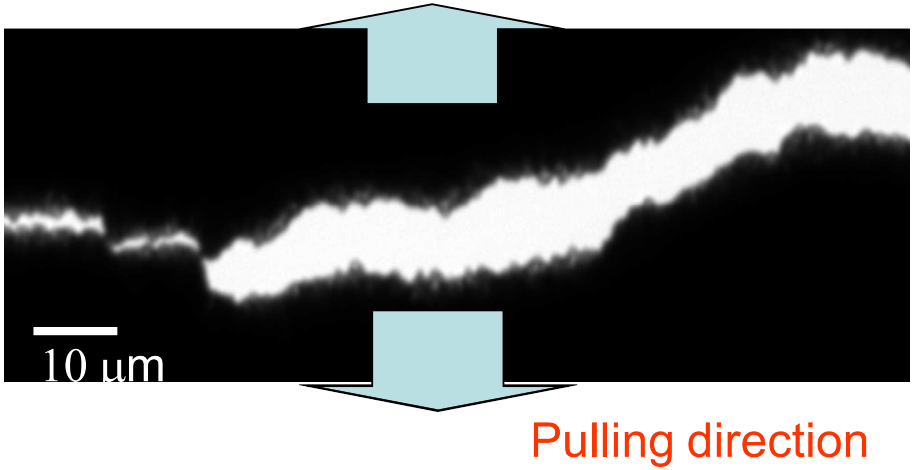
Ta *in-situ* TEM straining experiments

SAND2012-9237P

Ping Lu (Org. 1819)



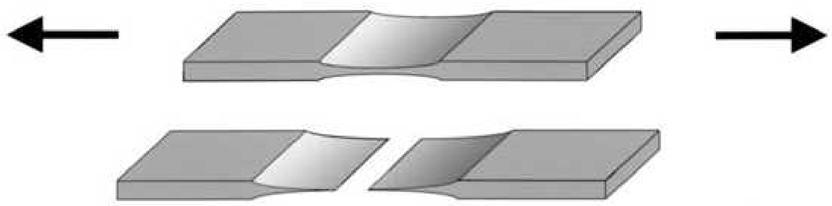


Goals:

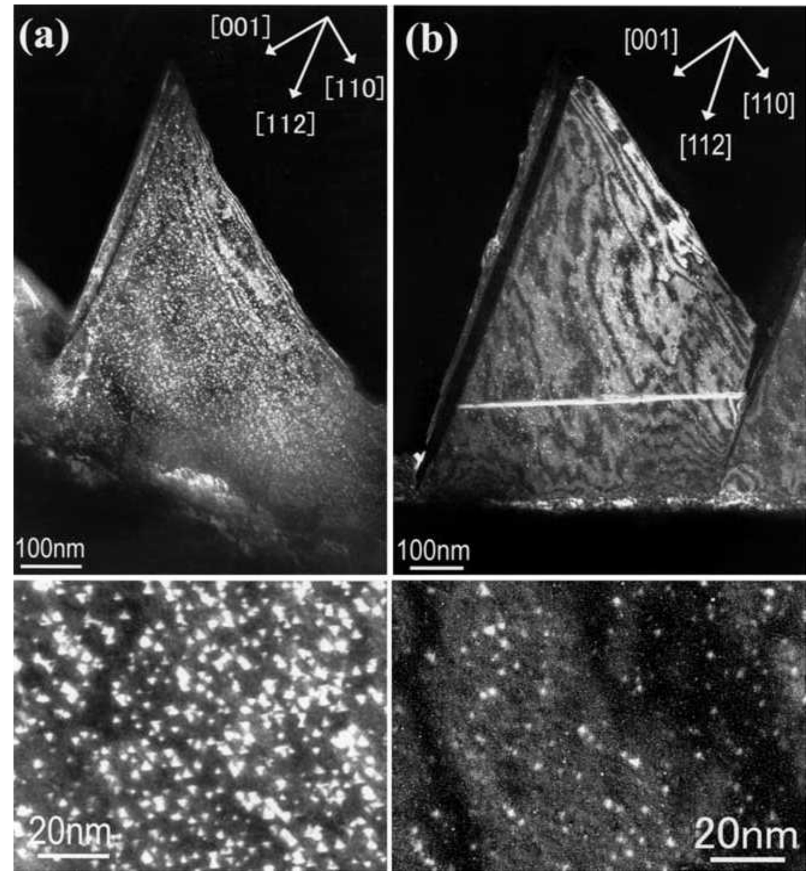
- ✓ To determine defects and defect structures produced by deforming/fracturing in Ta foils;
- ✓ To understand mechanism of defect generation and dynamic of the defect interaction responsible for deformation/fracturing of Ta foils;
- ✓ To understand effect of scaling (bulk, foil, thin film and nanowire) on Ta deformation behavior

In-situ TEM Work on FCC metals (Au, Ni, Cu, Al)

Y. Matsukawa *, K. Yasunaga, M. Komatsu, M. Kiritani



- **Unusual deformation microstructure:**
  1. no dislocations;
  2. vacancy-type point defect clusters (stacking fault tetrahedra (SFTs)) - at extraordinarily high density.



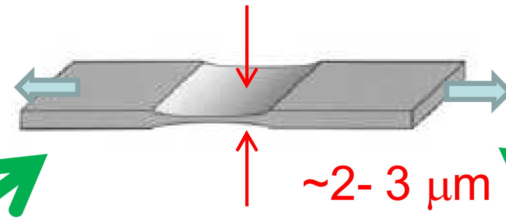
Strain rate $=10^{6}$/s, Strain rate $=10^{-1}$/s,

(1) Materials Science and Engineering A350 (2003) 8-16
(2) Materials Science and Engineering A350 (2003) 17-24

How are Ta *in-situ* straining experiments related?

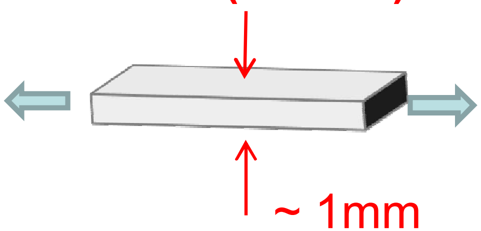
In-Situ TEM Straining

- Foils (~2-3 μm)



In-Situ SEM Straining/EBSD

- Bulks (~ 1mm)

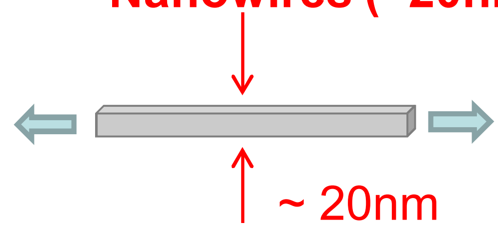


Brad Boyce/Blythe Clark
Task II

Ping Lu
Task I

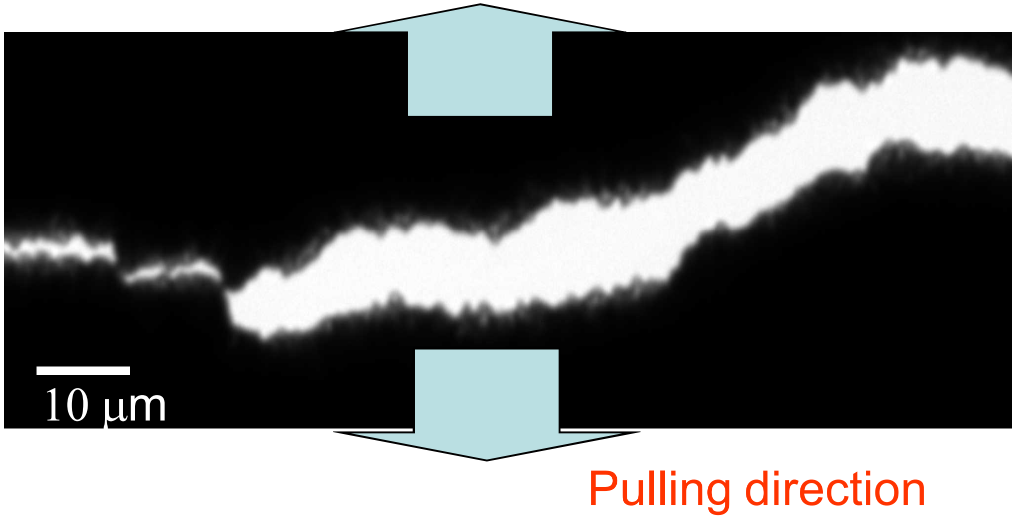
In-Situ TEM Straining

- **Thin film (~20nm)**
- **Nanowires (~20nm)**



- **Effect of scaling (bulk, foil, thin film and nanowire) on Ta deformation behavior**

In-Situ TEM Results from FY11

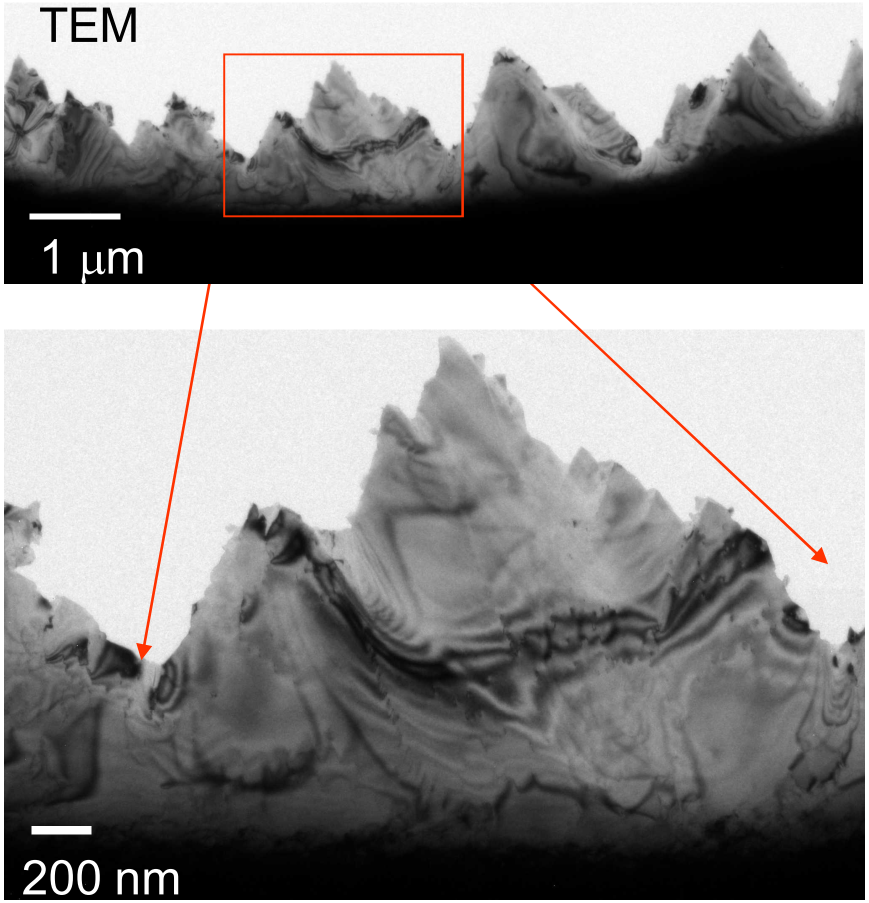


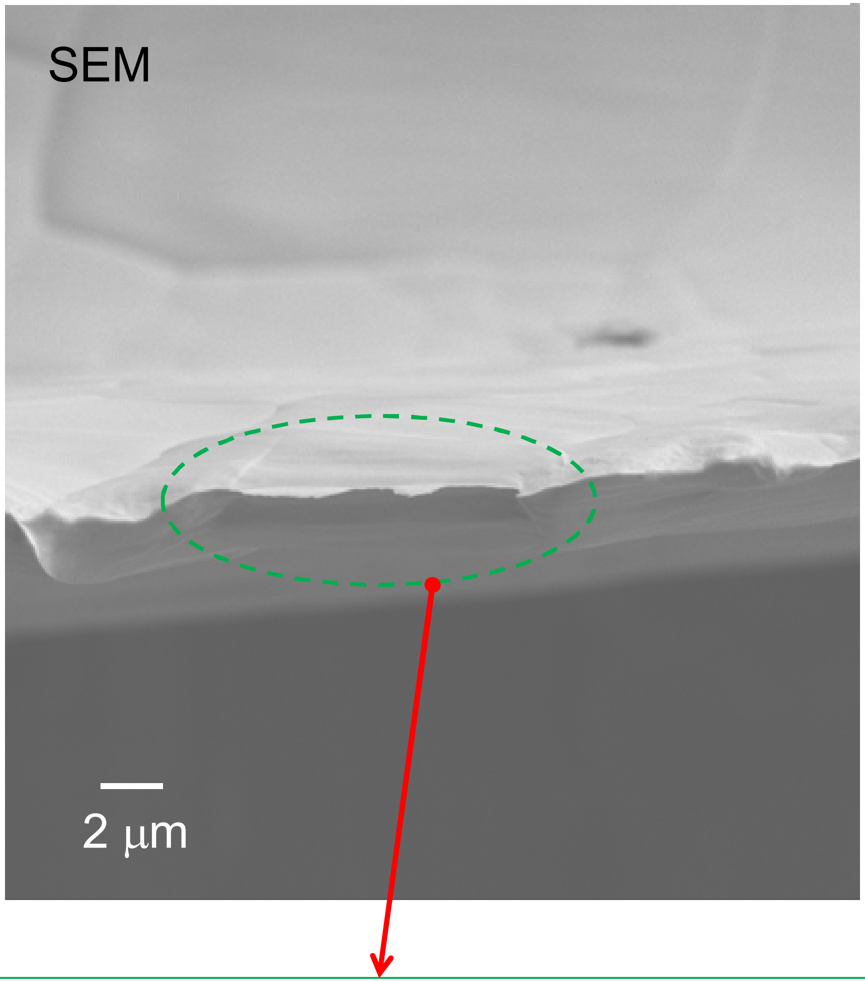
- Pre-thinned by Ar ion-milling to a thickness of about 3 microns.
- Annealed at 1800°C for 2hrs.
- Strained in-situ in TEM until the fracture takes place;
- The areas near the fractured surface were observed by TEM.

<u>Summary of main results:</u>

- **Saw-tooth structure is commonly observed at the fracture front which contains a high density of dislocations;**
- **Most of the dislocations are screw dislocations – {110}<111> slip; Other type dislocations (screw-edge mixed type) may also exist;**
- **No <u>nanosize</u> voids are present in the fracture tips.**

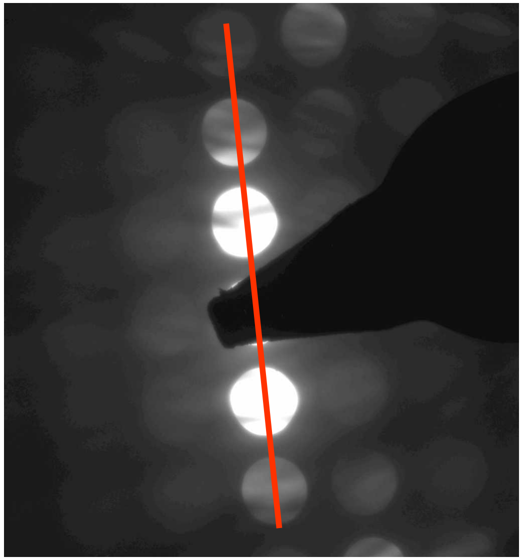
Morphology - Saw-tooth structure at the fracture tips



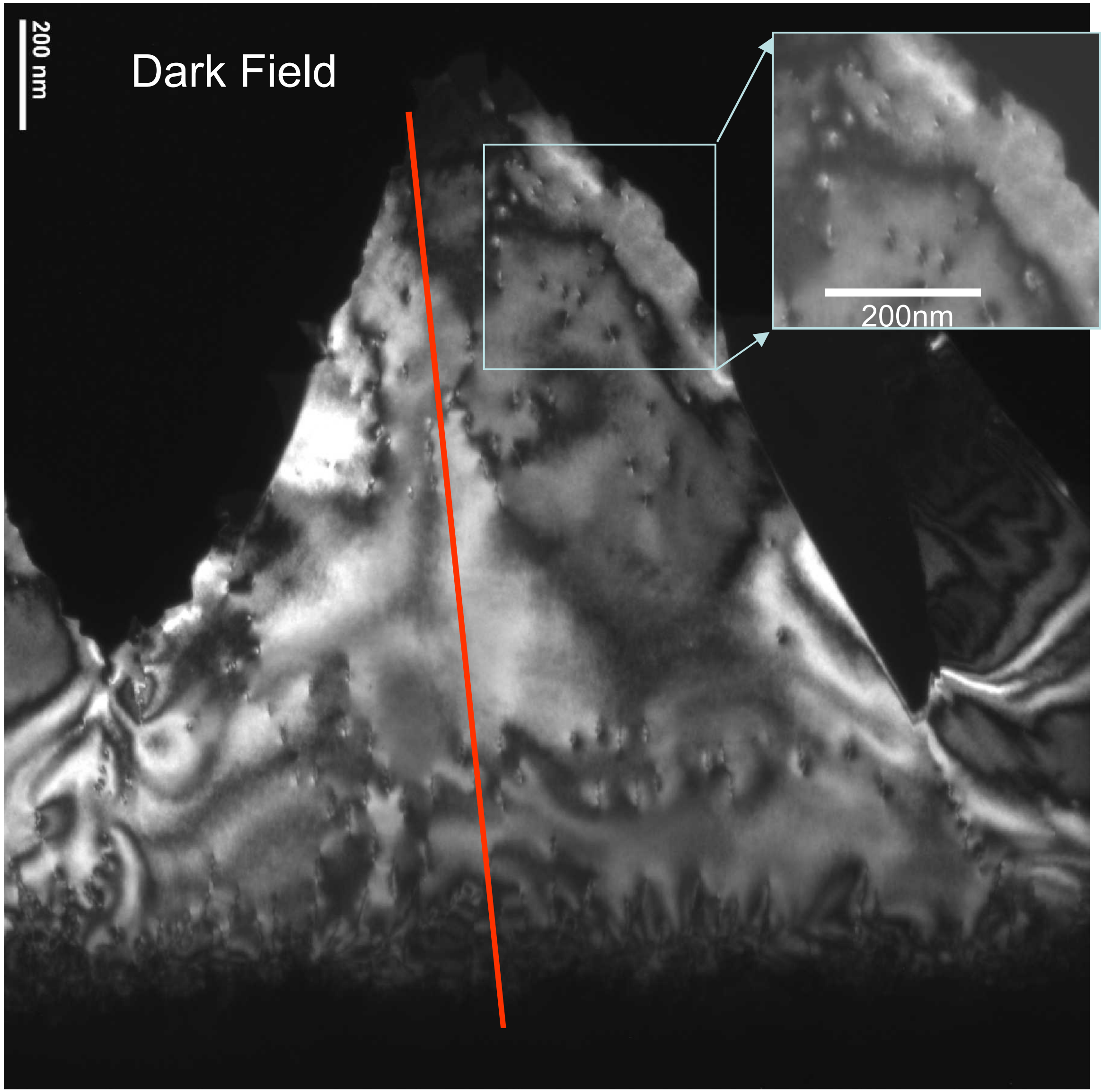


Similar structure is observed locally in in-situ SEM strained samples

The fractured tips contain a high density of dislocations

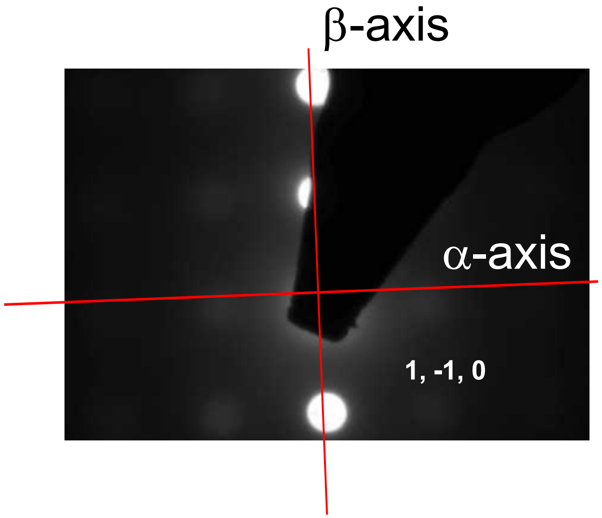


g = (1,-1, 0)

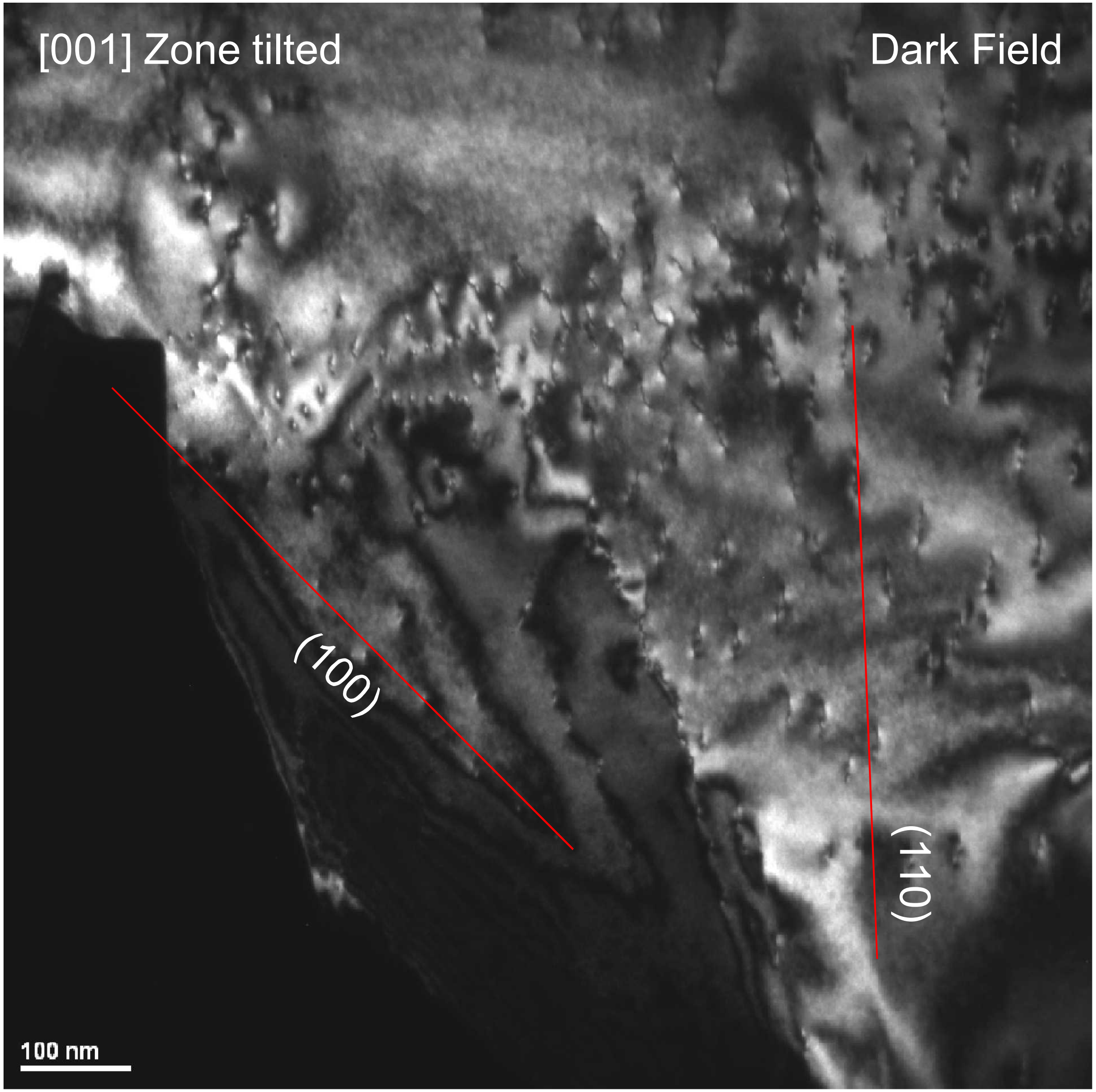


Controlled TEM tilting experiments

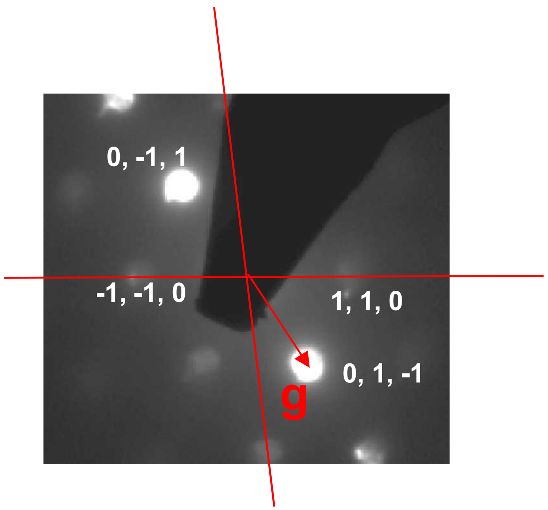
Most of the dislocations are screw dislocations – Other type dislocations (screw-edge mixed type) may also exist.



g = (1, -1, 0)

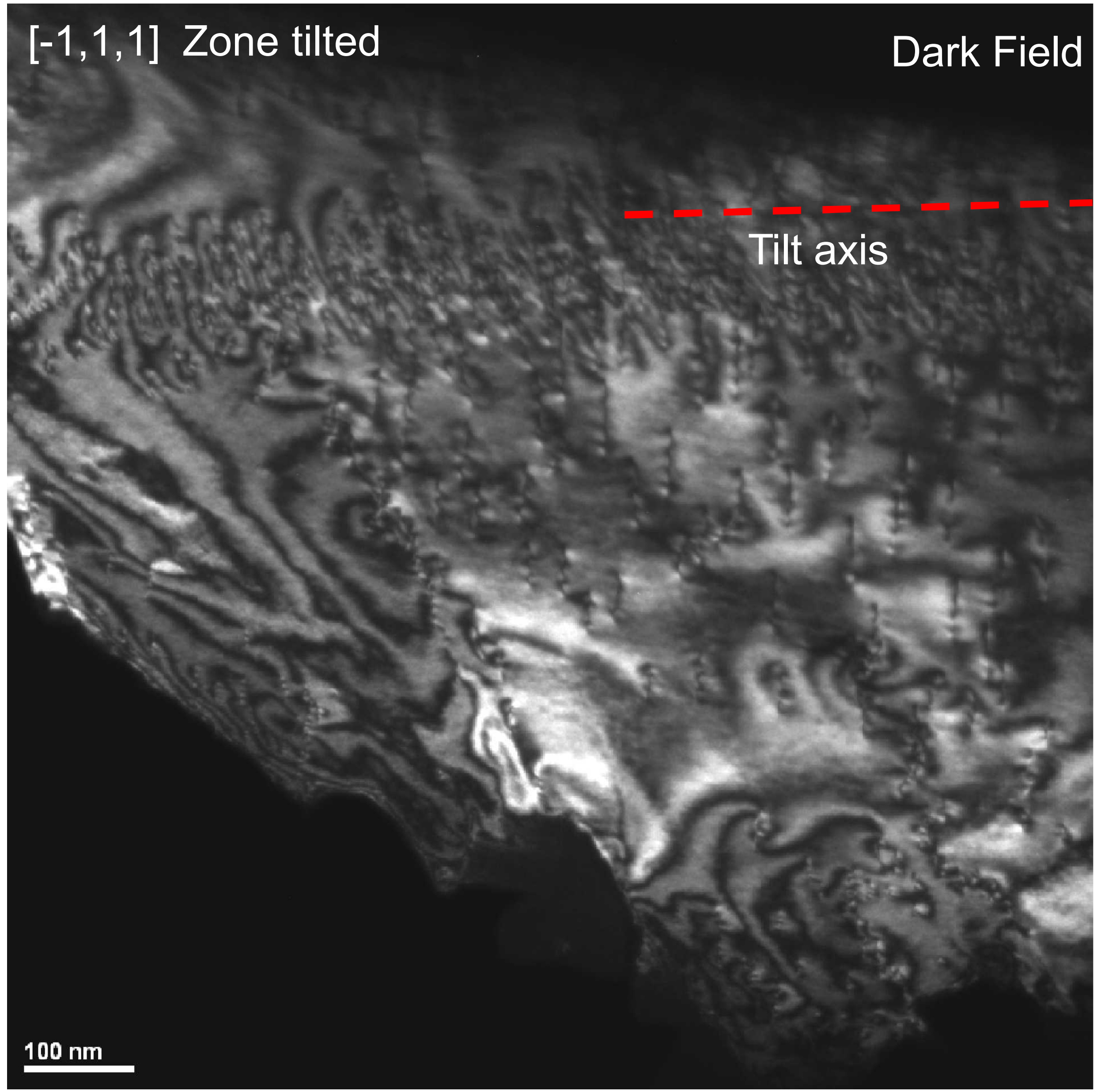


Rotated 54.7° about α axis to [-1,1,1] Zone

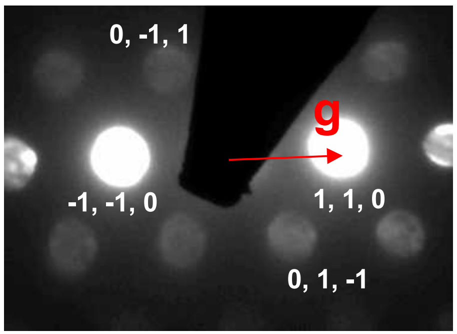


g= [0, 1,-1]

$g.b \neq 0$



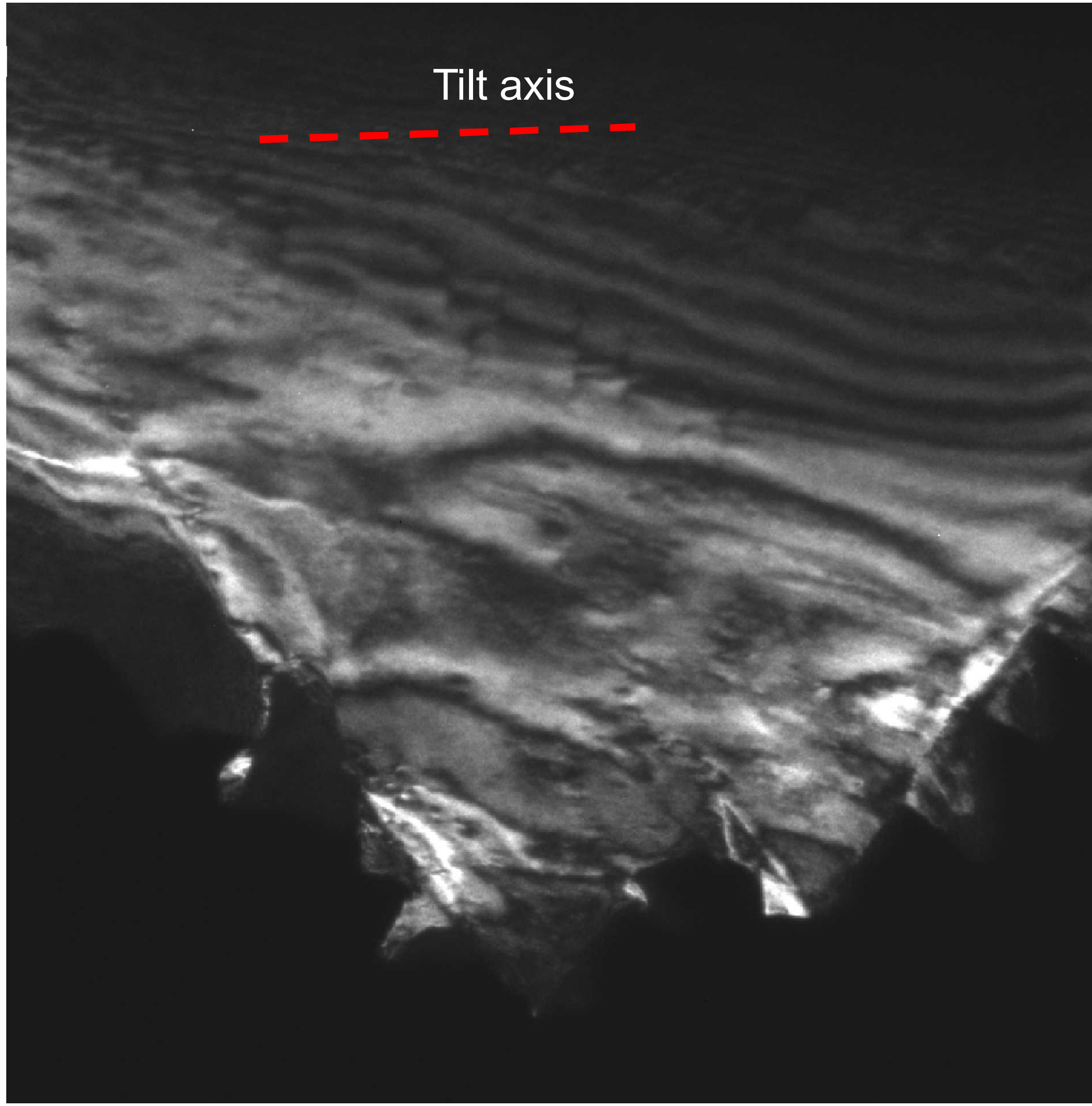
Rotated 54.7° about α axis to [-1,1,1] Zone



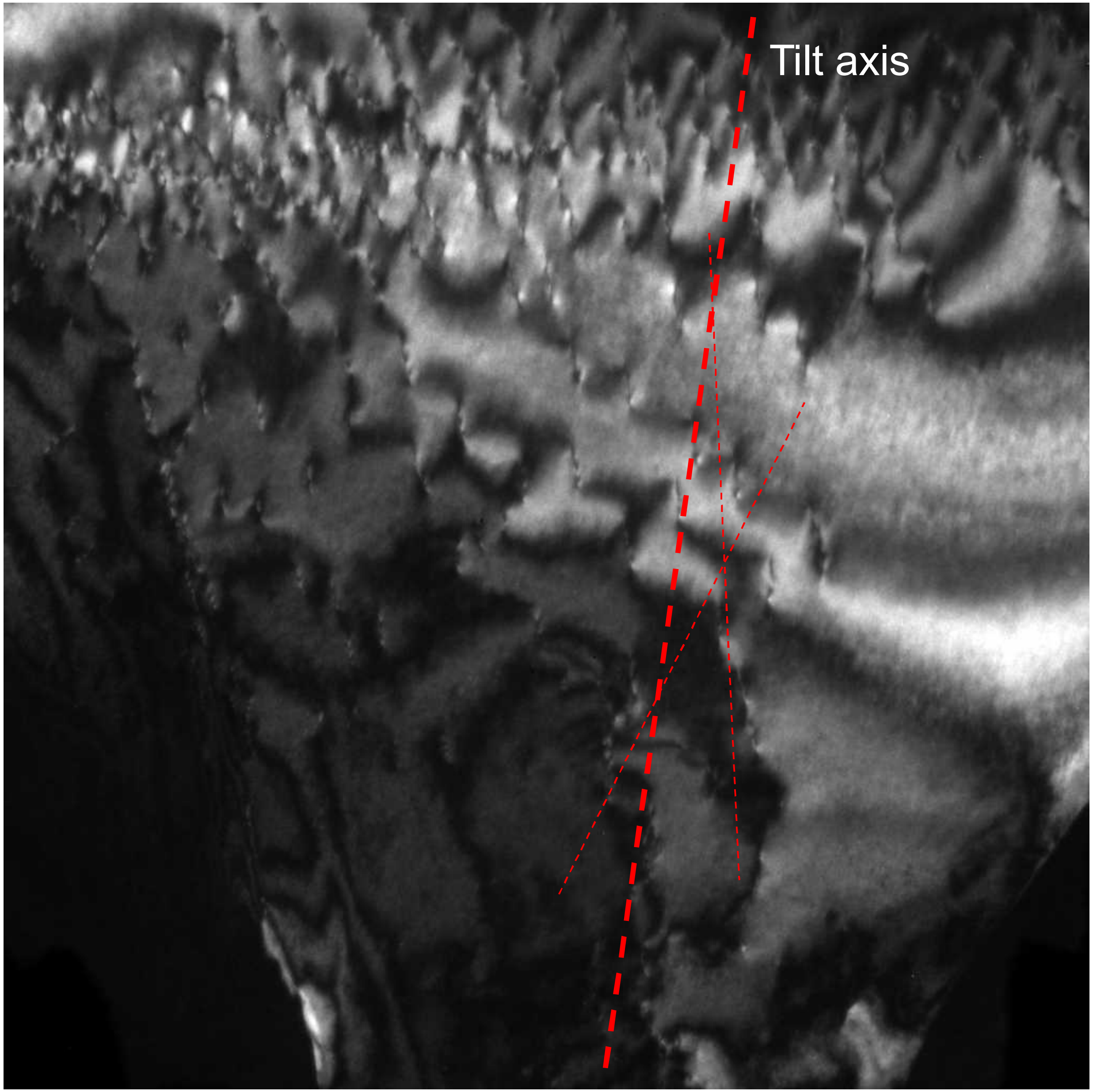
g= [1,1,0]

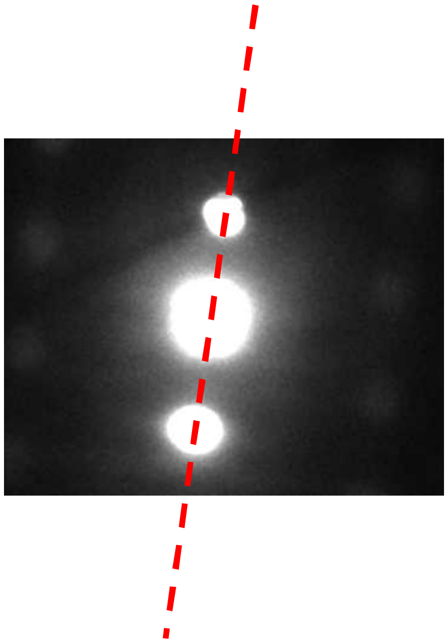
g.b = 0

Extinction condition



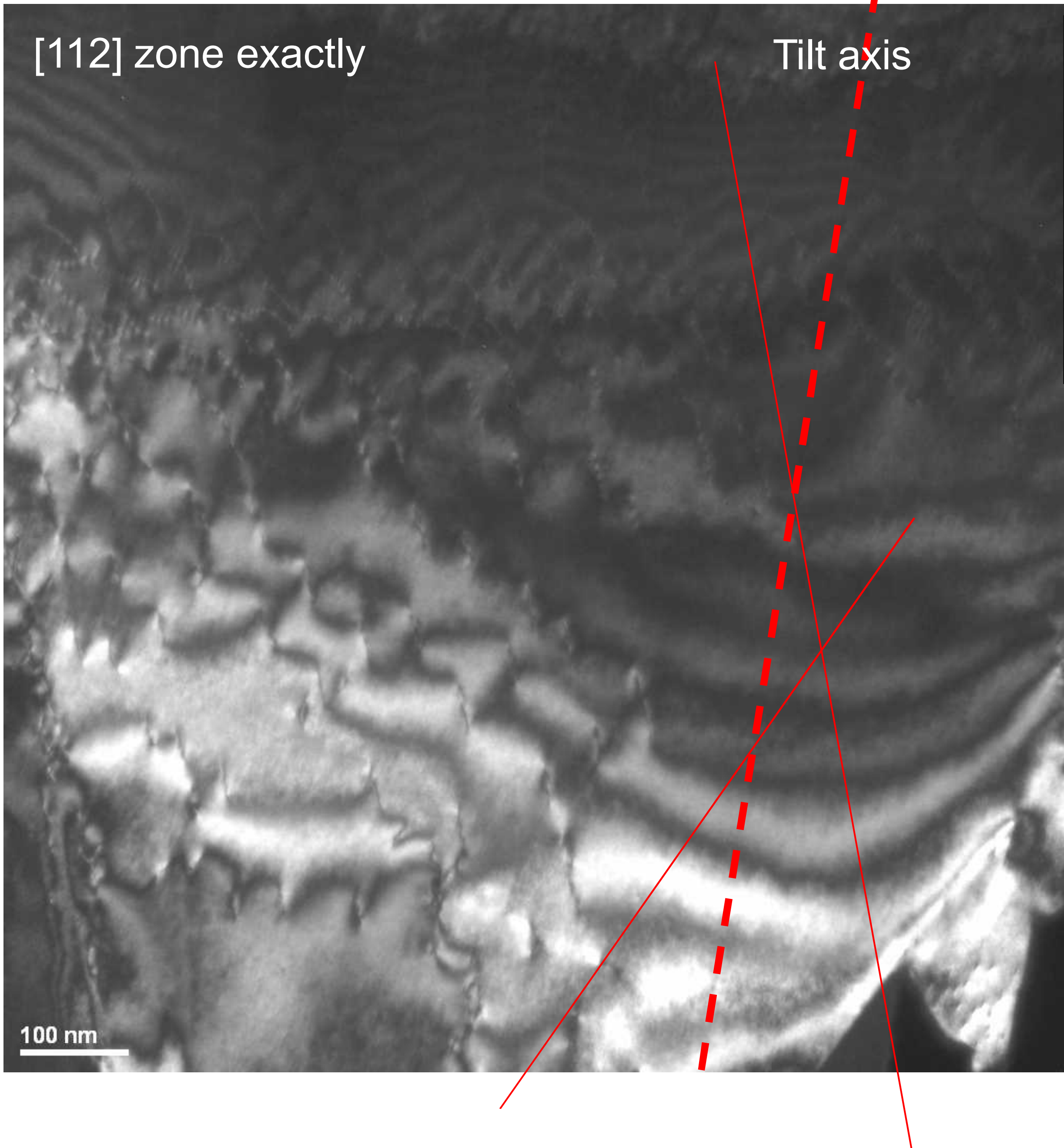
Rotated 25.2° about β axis to [113] Zone

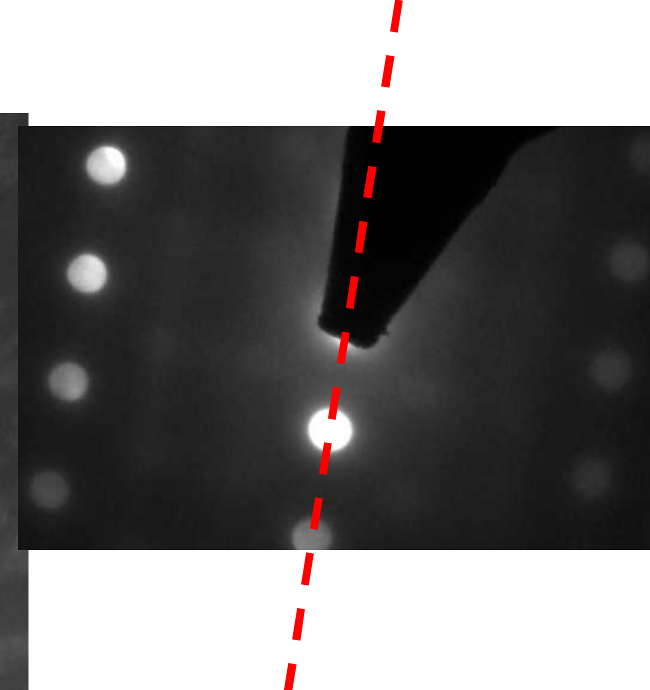




g = (1, -1, 0)

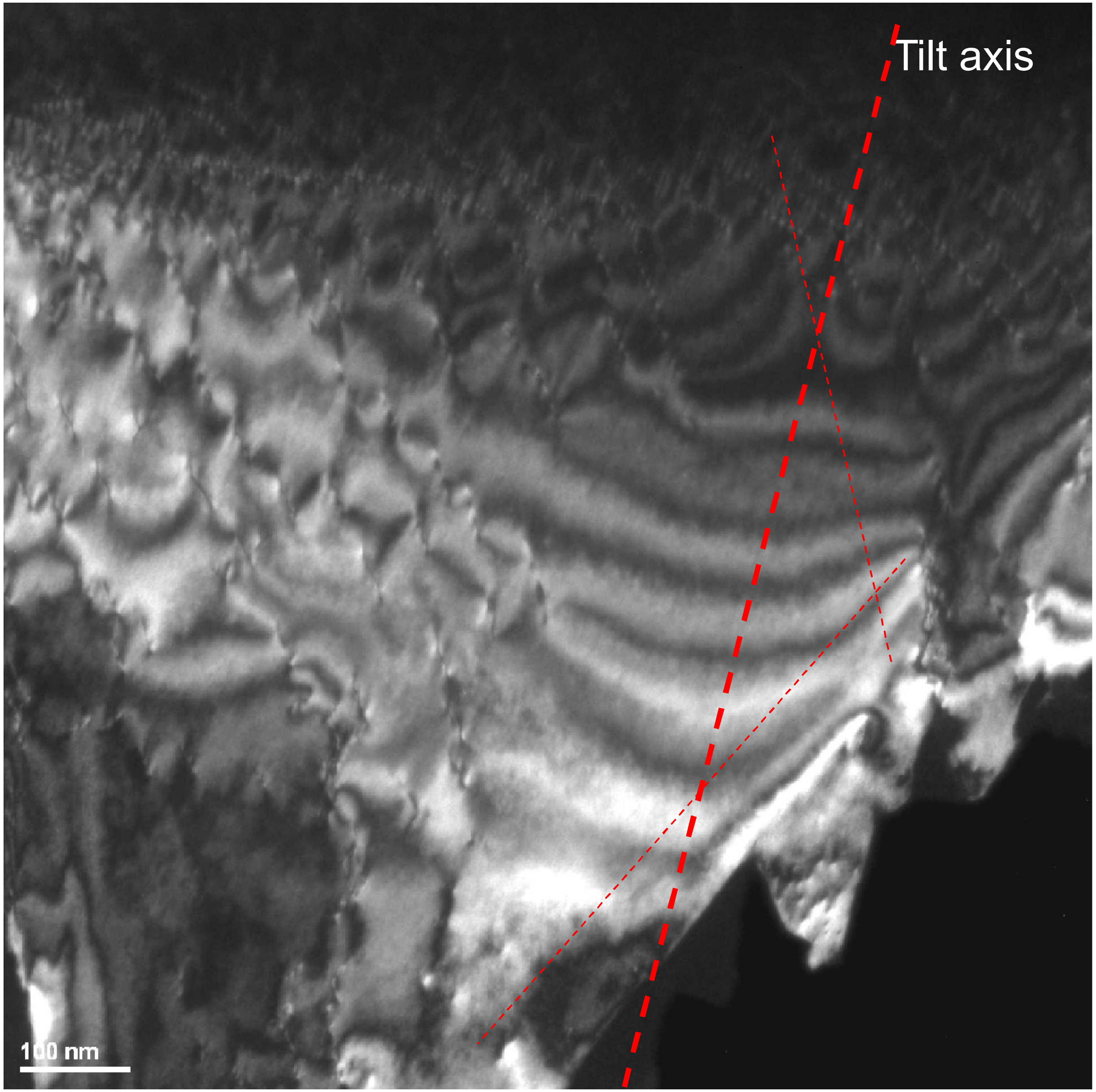
Rotated 35.7° about β axis to [112] Zone

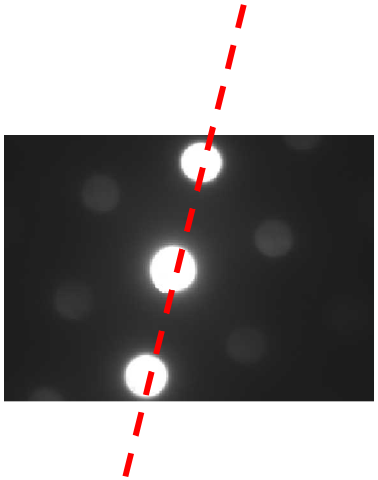




g = (1, -1, 0)

Rotated 54.7° about β axis to [111] Zone





g = (1, -1, 0)

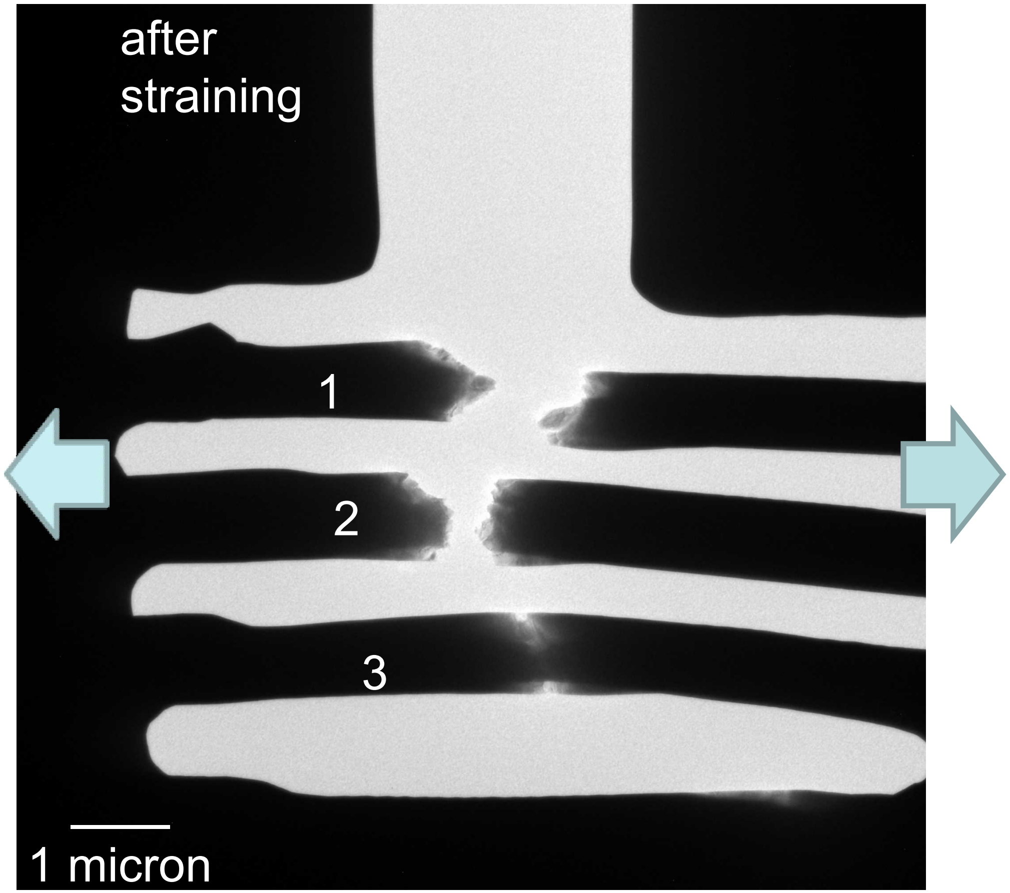
Summary of the results:

- Saw-tooth structure is commonly observed at the fracture front which contains a high density of dislocations;

- Most of the dislocations are screw dislocations – {110}<111> slip; Other type dislocations (screw-edge mixed type) may also exist;

- No nanosize voids are present in the fracture tips.

In-situ dynamics TEM experiments (FY 12)



Low mag. image showing the microbars after straining that has led to fracturing of bars 1 and 2.

Experimental:

- Micron bars, created by ion milling and FIB, will be used for in-situ TEM straining

The design allows for:

(1) controlled fracture of Ta micron bar one by one, and

(2) observation of the defects and defect interaction involved in fracture processes.

A bright field image recording, showing the dislocations (short, black, near horizontal lines) moving up and down during the fracturing process.

C_Ta_straining_Bar1_17kx3_nearend_BF_2times.avi

If you freeze a frame and draw a straight line connecting tips of the dislocations, the line is along <100> direction, since the dislocation is in <111> direction, cross-product of the two vectors gives <110>, normal of the planes the dislocations are in. The dislocations are slipping in {110} planes!

Video frames , showing the movement of a dislocation in a {110} plane. The line of dislocations is in <111> direction.

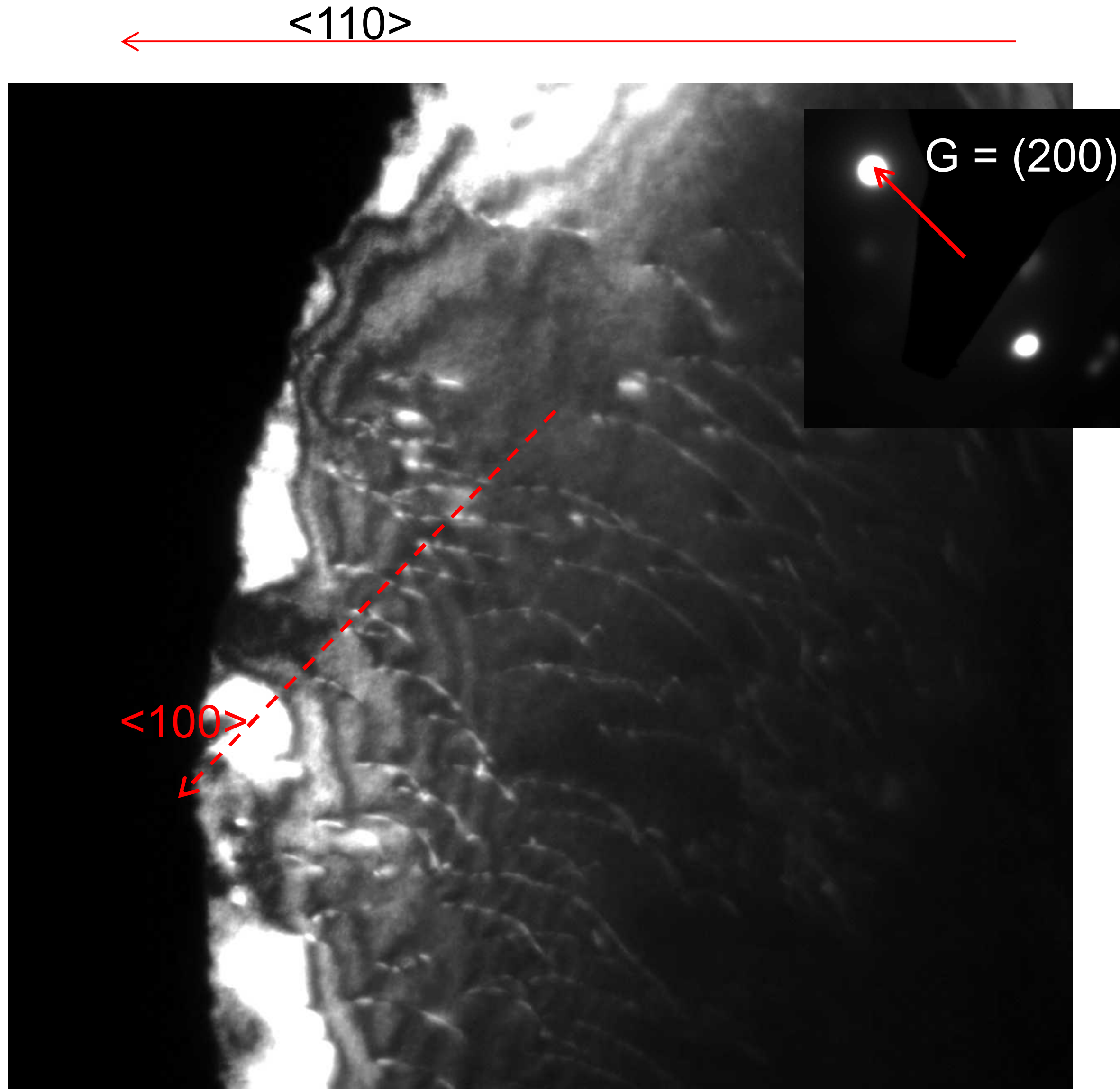
Frame #10 Frame #24 Frame #36

A_Ta_straining_Bar2_13.5kx_DislocationSlip_DF_1time.avi

B_Ta_strainingBar2_13.5_21kxBar2_DF_2times.avi

This area was seen a lot of dislocation generation and movement during the *in-situ* experiment. This image is recorded after the crack passing the area and the specimen re-tilted to get better dark-field contrast.

The dislocations seen are along <111>, and sets of dislocations are in {110} planes.



Formation of large voids in front of the crack

Ta_straining_bulkPart2_BF_21kx_2times.avi

Summary of the results

- The primary defects are of screw types with line direction along <111>.
- The fracturing process involves repeated process of dislocation generation and movement (in direction perpendicular or near perpendicular to the tensile axis), leading to thickness reduction and local rotation of the grain.
- The dislocation movement captured by CCD camera (at rate of 10 frame/second) shows primary active slip system is of {110}<111> type.
- No nano-sized voids are observed near the fracture tip.