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Study on the interaction between dislocations and helium bubbles in copper by in situ straining experiments in transmission electron microscopy.

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Objective

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Macro scale
Degradation in mechanical properties

Quantitative estimation
of radiation hardening

⇔

Micro scale
Microstructural evolution
Extended defect clusters
←Obstacles to dislocation movements
Fusion environment
<u>He bubbles</u>

Correlation between mechanical properties and microstructure

Dislocation–defects interaction

<u>In-situ straining observation</u>
Dynamic information
(bow-out angle, velocity etc.)

The objective of the present work is clarify the interaction between dislocations and cavities by performing **<u>in-situ TEM observation</u>**. <u>He bubbles</u> and <u>voids</u> are introduced in <u>pure copper</u> as the obstacles to the dislocation movements.

Does cavity contribute to the hardening ?

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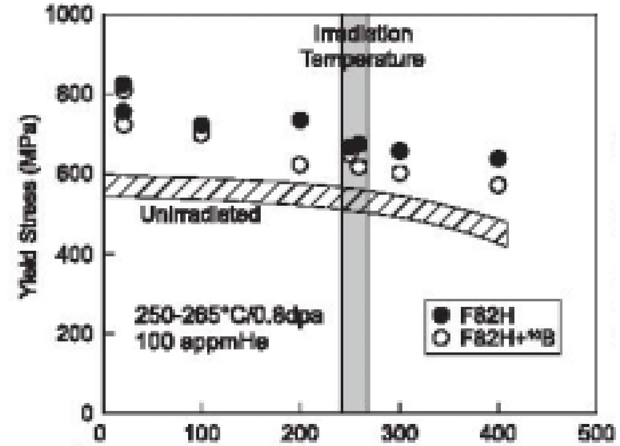
No hardening because both shear modulus and flow stress in cavity are zero ?



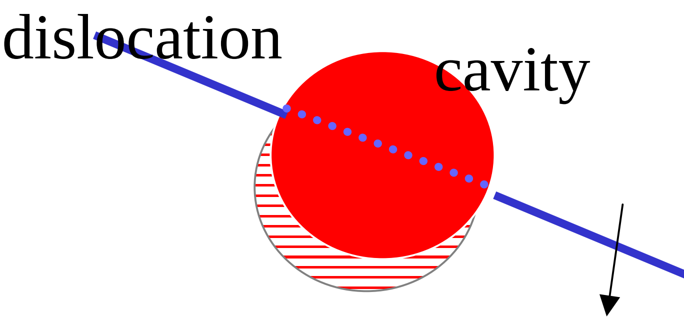
Hardening of voids is smaller than I-loops

Candra Y, Fukumoto K, Kimura A, Matsui H
JOURNAL OF NUCLEAR MATERIALS 272: 301-305 1999

No B-addition effect on yield stress in irradiated F82H

Shiba K, Hishinuma A
JOURNAL OF NUCLEAR MATERIALS 283-287: 474-477 2000

Long range interaction



Modulus effects (difference in matrix and cavity)
Cavity has an attractive interaction to dislocation

Contact interaction

Self energy of dislocation

Self energy of dislocation decrease corresponding to the length of dislocation segment which are disappeared when dislocation cut the cavity.

Interfacial energy

When the cavity was sheared by dislocation, new interfaces should be created on the surfaces of cavity.

Specimens

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Cu (FCC) 99.999%

Cold work rolling 0.1mm R.T.

Fraise machining

Annealing 950℃ (T_m = 1083℃) 2×10^{-4} Pa

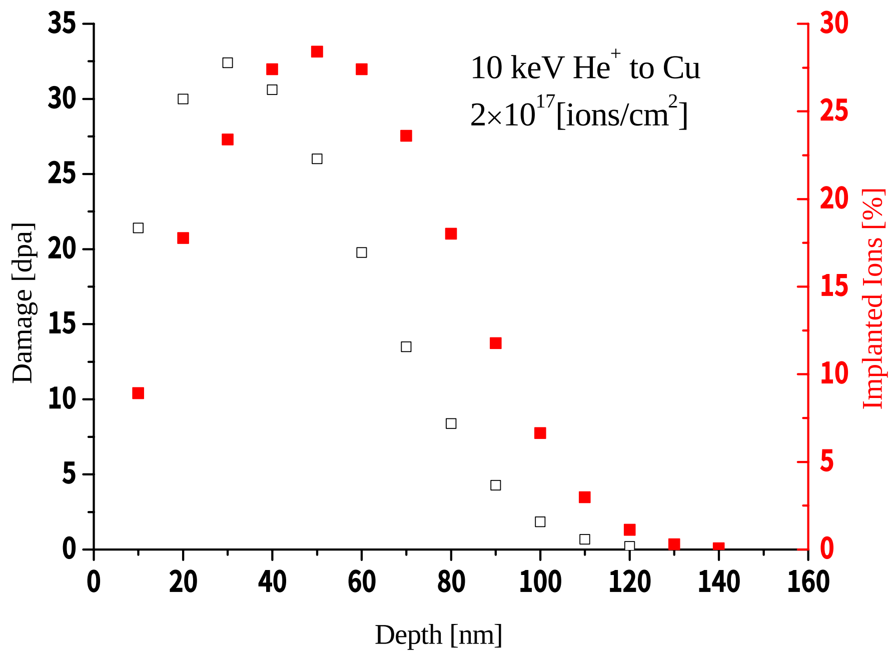
<u>Jet polishing (8 V, 100 mA, –10 ℃)</u>

<u>Flash polishing (8 V, 0.2 sec, –10 ℃)</u>

150 HNO_3
350 Methanol
40 Butyl Cellosolve

Ion irradiations

Ion gun (Coltron)
Sample (3mm disc、In-situ TEM)
Injected ions He+
Energy of ions 10keV
Fluence 2×10^{17} [ions/cm^2]
Damage levels 32 [dpa] (at damage peak)
Damage rate 1×10^{-4}〜1×10^{-3} [dpa/sec]
Temperature R.T.
Vacuum levels 4×10^{-5} [Pa]



Thin foil irradiation (Large fraction of V-type clusters)

Pressure of helium bubbles

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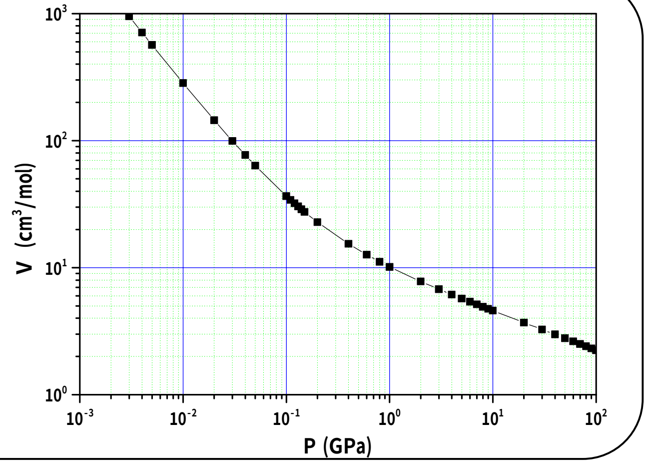
Over pressurized bubbles

MLB-EOS

$$V = (22.575 + 0.00064655T - 7.2645T^{-1/2})P^{-1/3} + (-12.483 - 0.024549T)P^{-2/3} + (1.0596 + 0.10604T - 19.641T^{-1/2} + 189.84T^{-1})P^{-1}$$

V : molar volume (cm^3/mol), P : pressure(kbar), T : temperature (K)



Loop punching limit

$P = (2\gamma + \mu b)/r$

P: pressure (Pa), γ: surface energy (N/m),
μ: shear modulus, b: burgers vector, r: cavity radius

	1x10^{16} /cm^2 300°C	1x10^{16} /cm^2 350°C	2x10^{17} /cm^2 RT	2x10^{17} /cm^2 RT +anneal650C
P (GPa)	4.0	3.6	23.7	0.6

Nano indentation

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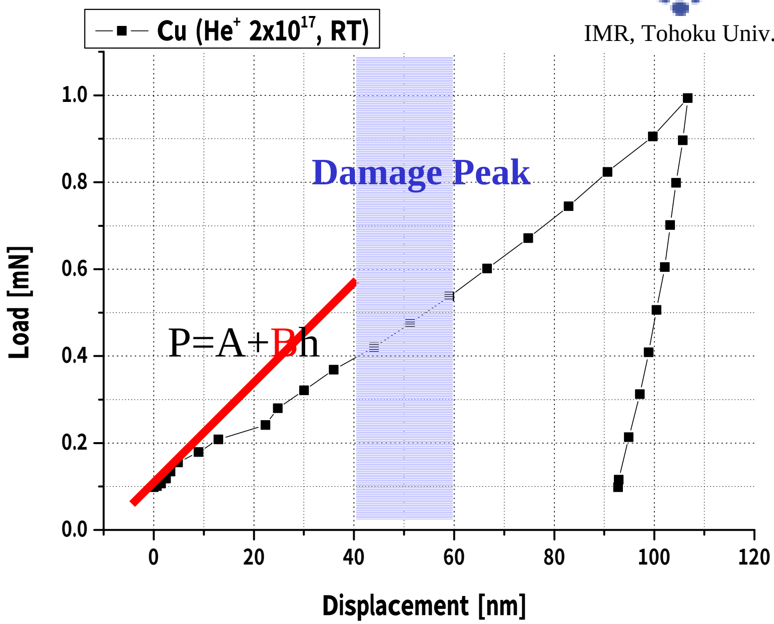
UMIS-2000 (CSIRO)

Indenter Type:	Berkovich
Indentations per line:	5
Control Method:	Force
Initial Contact Force (mN):	0.1
Maximum Force (mN):	1
Force Increment:	Square root
Time for Dwell(sec):	1
Area Function Correction	

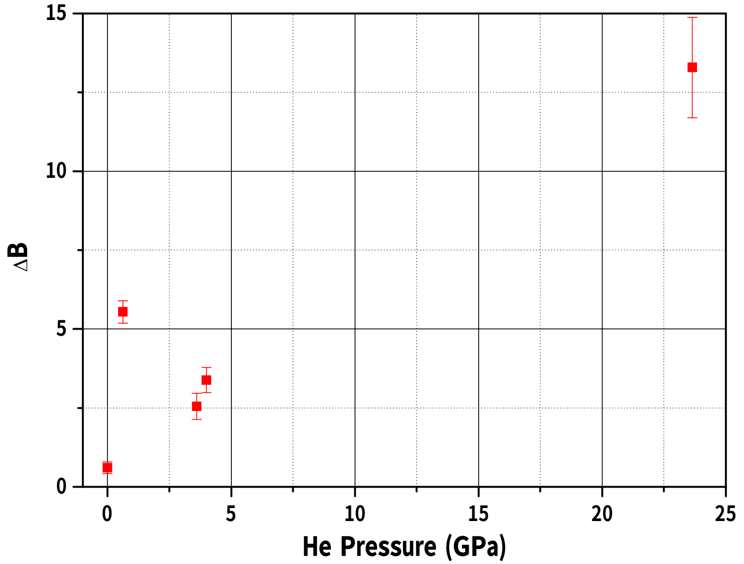


Evaluation of the hardening

Damage region is only at near surface
1st order fitting at the beginning of the curve

$$P=A+Bh$$

Pop-in at 1/10 of the damage peak
(Ref. A.J. Whitehead: Thin Solid Films, 220 (1992) 277)



<u>Hardening increased with bubble pressure</u>

Note; Number density and size of the bubble, other extended defects should be considered

In-situ TEM

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TEM (JEOL 2010)
200kV

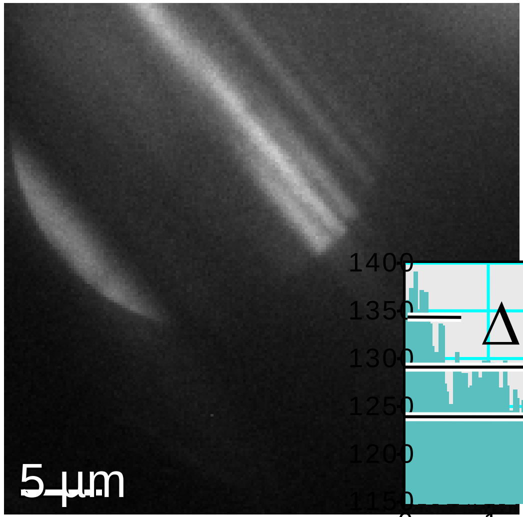
Double tilt holder (JEOL EM-31030)
Single tilt straining holder (Gatan model 671.DH)
strain rate 1.0 [μm/sec]
load 500 [g]

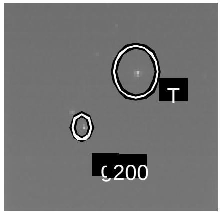
CCD camera (JEOL EM-24230)
sampling rate 30[frames/sec]
connection to TV monitor and PC

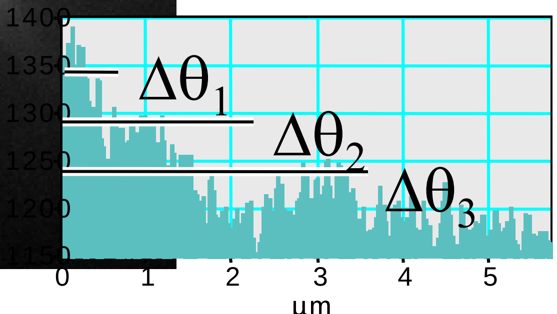
Thickness measurement:
Convergent beam diffraction (CBD)

$$\frac{S_i^2}{n_k^2} + \frac{1}{\xi_g^2 n_k^2} = \frac{1}{t^2}$$

$$s_i = \lambda \frac{\Delta\theta_i}{2\theta_B d^2}$$







Post irradiation annealing at 650 ^{o}C

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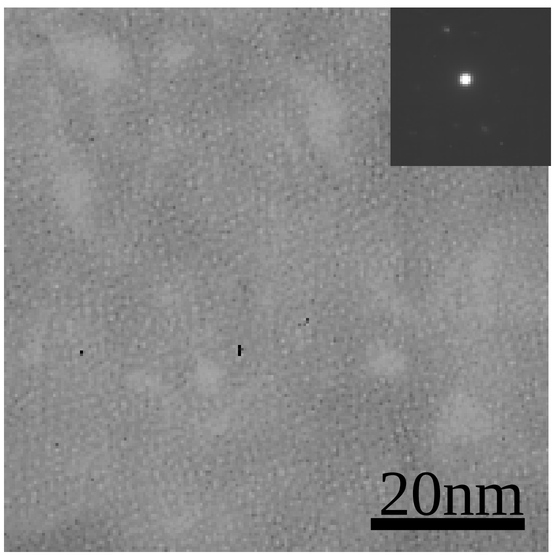
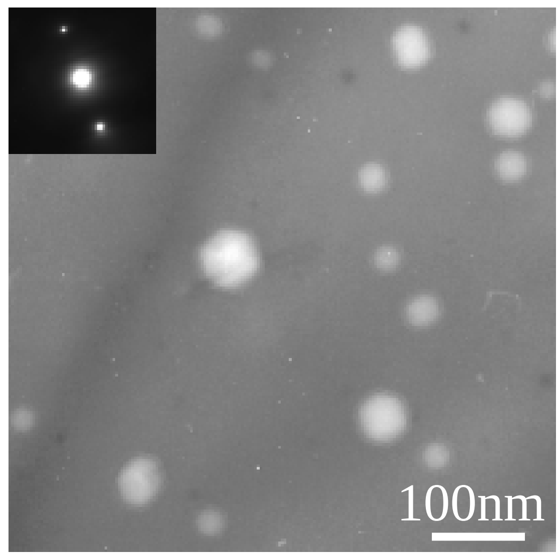
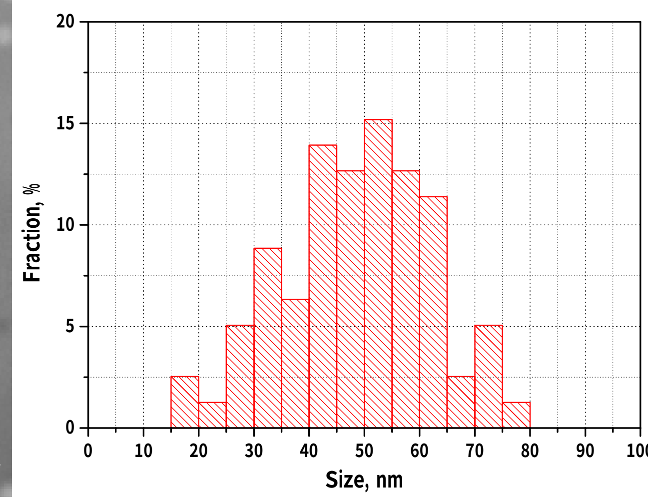
He^{+} irradiation
2×10^{17} ions/cm^2

He^{+} irradiation
2×10^{17} ions/cm^2
+
Annealing
650 ^{o}C 20 min





Size distribution



1.3 nm
7.2×10^{24} /m^2

49 nm
4.3×10^{20} /m^2

In-situ observation

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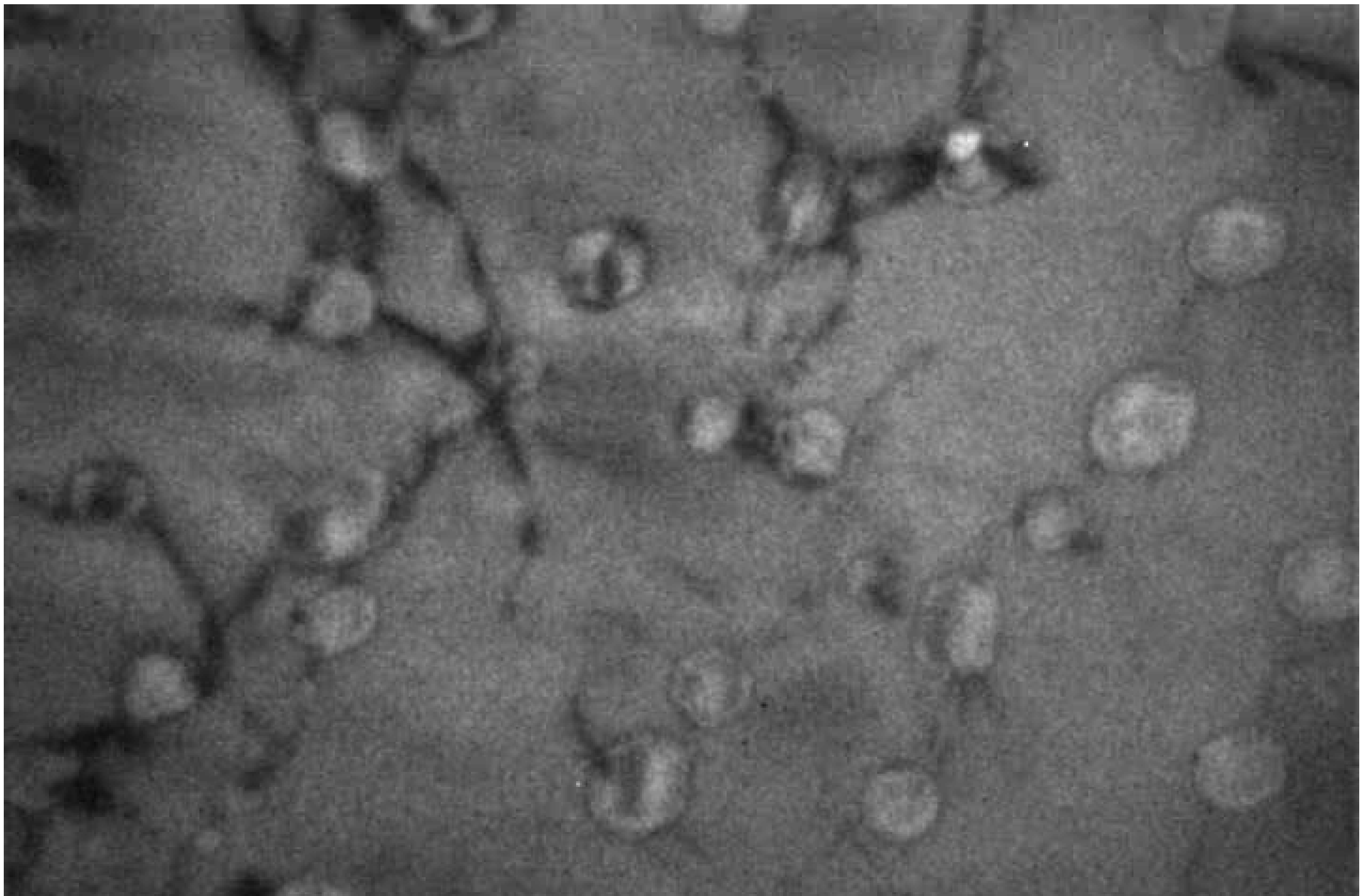
He^+ irradiation
2×10^{17} ions/cm^2

\+

Annealing
650 ^oC 20 min



100nm

Most of dislocations are pinned by cavities→cavities contribute hardening

Bow out and depinning from cavity

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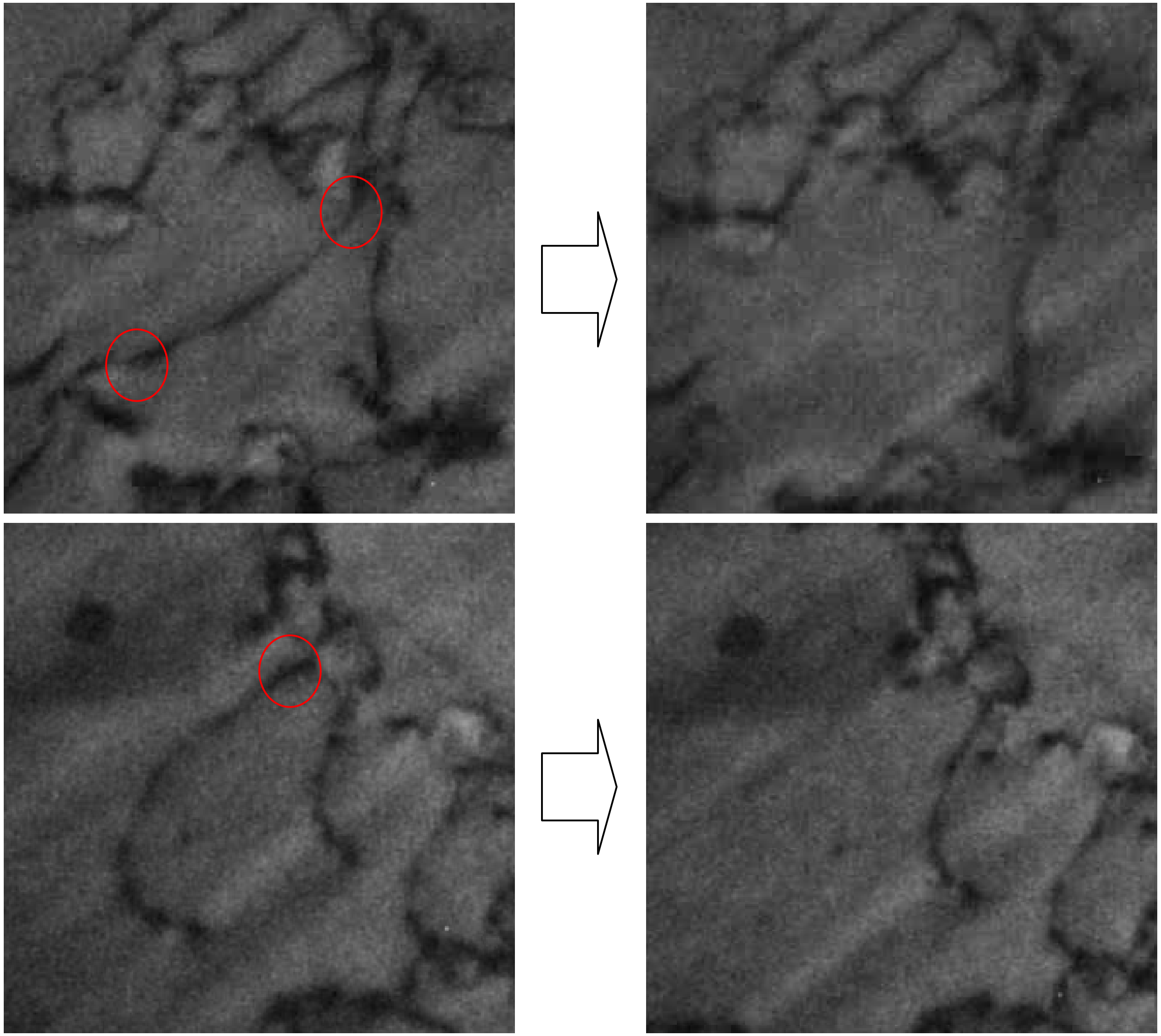
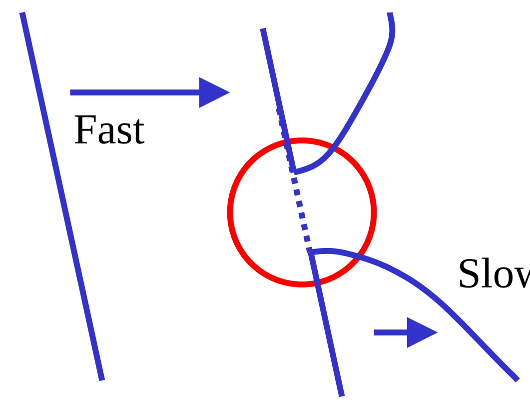


Small bow-out angle

Attractive interaction (modulus effect)

Stable at the center of cavity



Snapshots were taken just before and after the breakaway

Distance from center of cavity to glide plane

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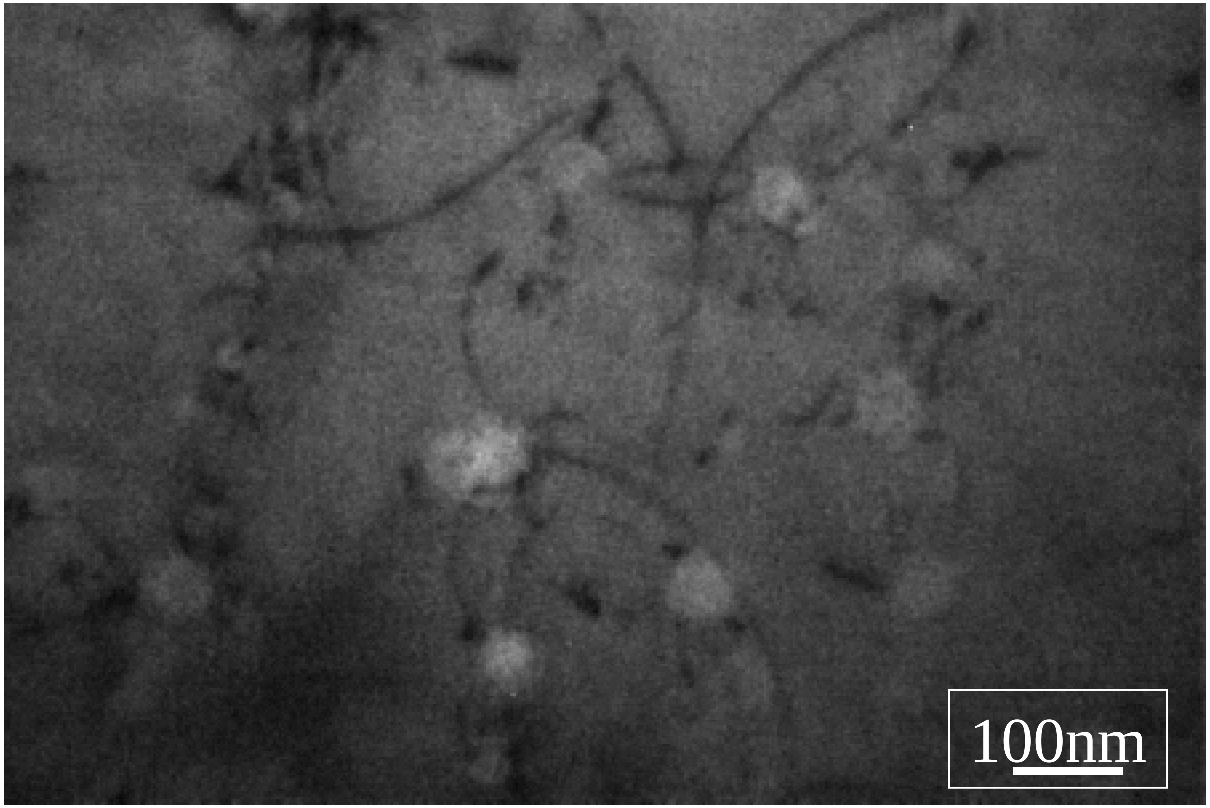
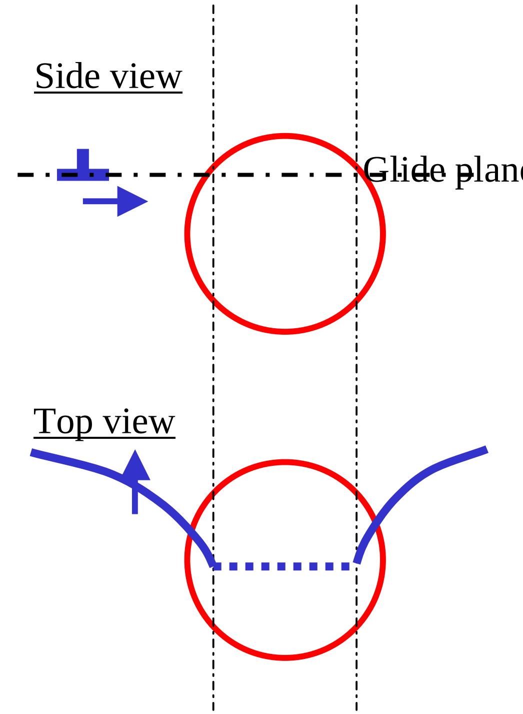




- Dislocations are observed inside cavities because the center of cavities are not always on glide plane (Micrographs are projected images)
- Distance from the center of cavity is important parameter to discuss the obstacle strength

Measurements of bow-out angle and obstacle spacing

Determination of the slip system

(Analyze must be performed on slip plane to discuss the dislocation-defect interaction)

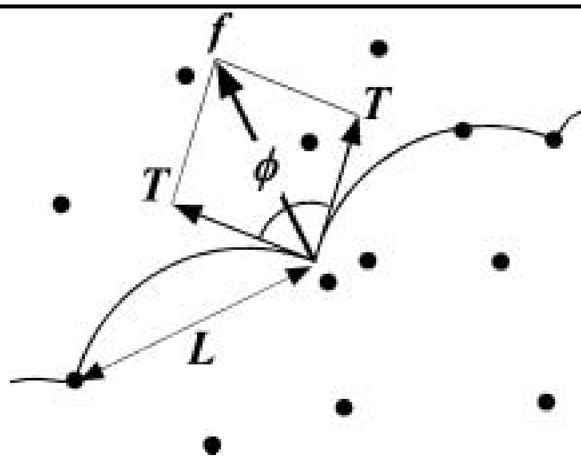
Determined by Diffraction pattern, tensile direction, slip line and Schmid Factor
→ Nature of dislocations (edge or screw)

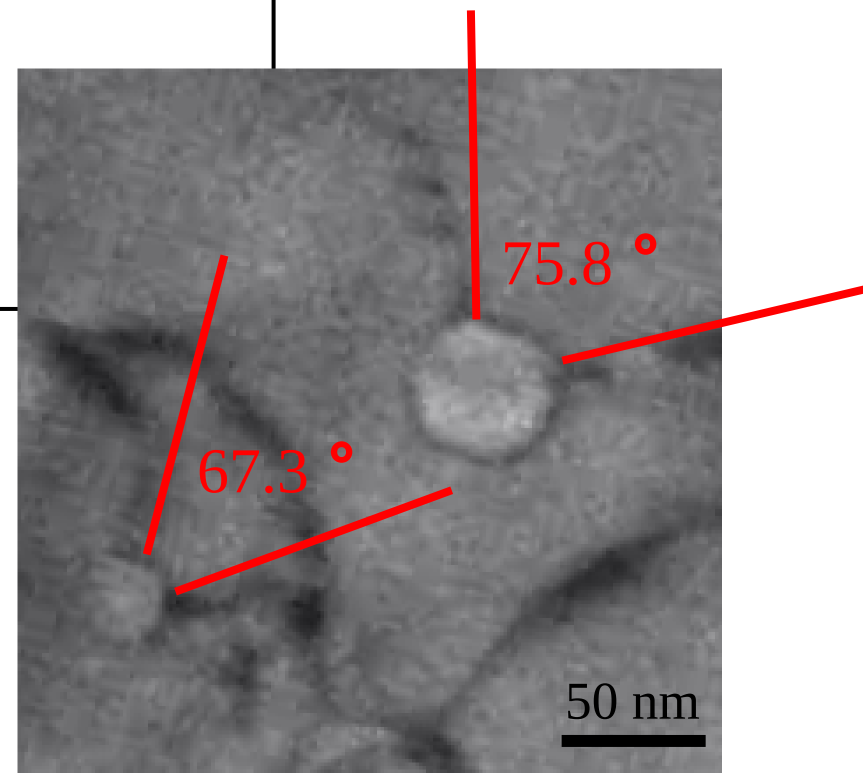
a) Obstacle Strength (bow-out angle), ϕ_c

→ $$f_c = 2T\cos\left(\frac{\phi_c}{2}\right)$$

b) Obstacle Spacing, L

→ Distance between the pinning points





Distributions of obstacle strength and spacing

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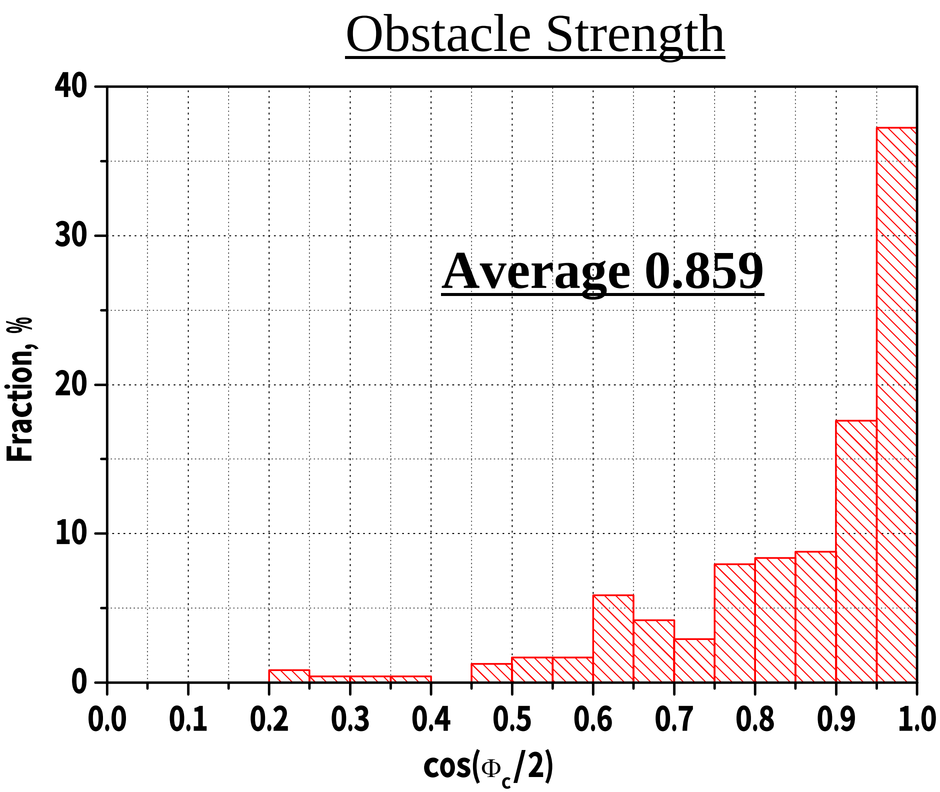

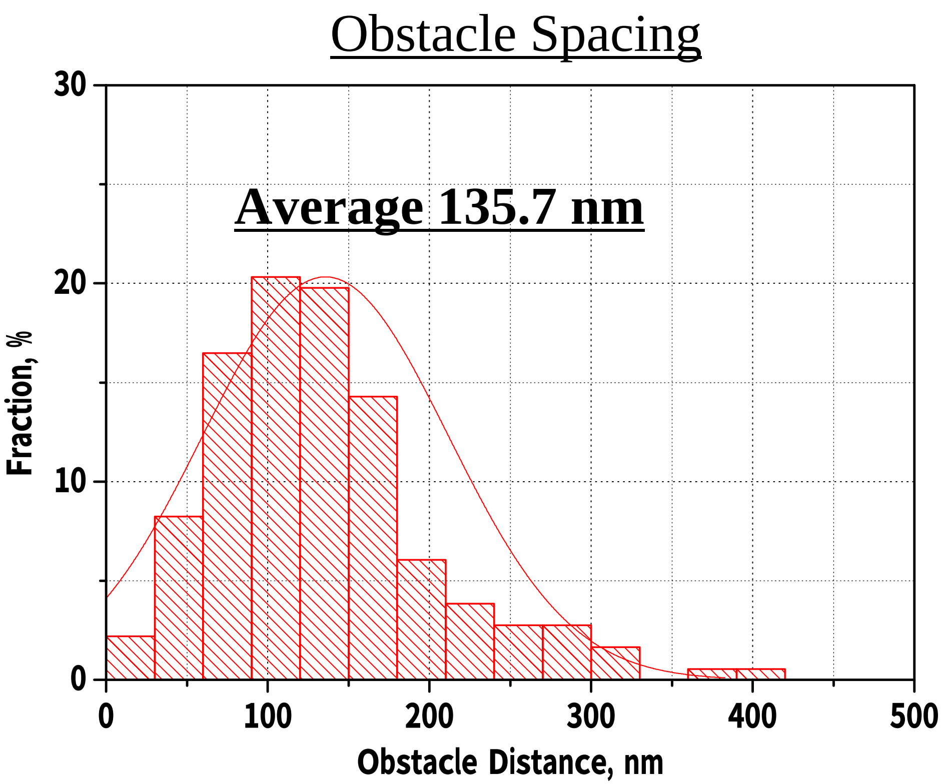

- The bow-out angles are small and the average strength factor ($\cos(\Phi_c/2)$) is 0.859.
- Obstacle spacing along dislocation is comparatively small.

(Dislocations are easily pinned because the large size of cavities)

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Foreman's relationship

$$\Delta\tau = \frac{\mu b}{\overline{L}} \cos\left(\frac{\phi_c}{2}\right)^{\frac{3}{2}} \left(1 - \frac{\phi'}{5\pi}\right)$$

$$(\phi' = \pi - \phi_c)$$

τ: shear stress
μ: shear modulus
b: burgers vector
L: average spacing
ϕ_c : critical angle

Increase in shear stress, $\Delta\tau$

Irradiation 1×10^{16} ions/m^2 350^{o}C

	Void	Bubble
Increase in shear stress, $\Delta\tau$: 59.9 MPa	5.9 MPa	13.2 MPa
Polycrystalline (Taylor Factor 3.06): 183.2 MPa	18.2 MPa	40.5 MPa

$\cos\left(\frac{\phi_c}{2}\right)$	0.86	0.29	0.37
size	47.2 nm	4.1 nm	8.4 nm

Size dependence of the obstacle strength

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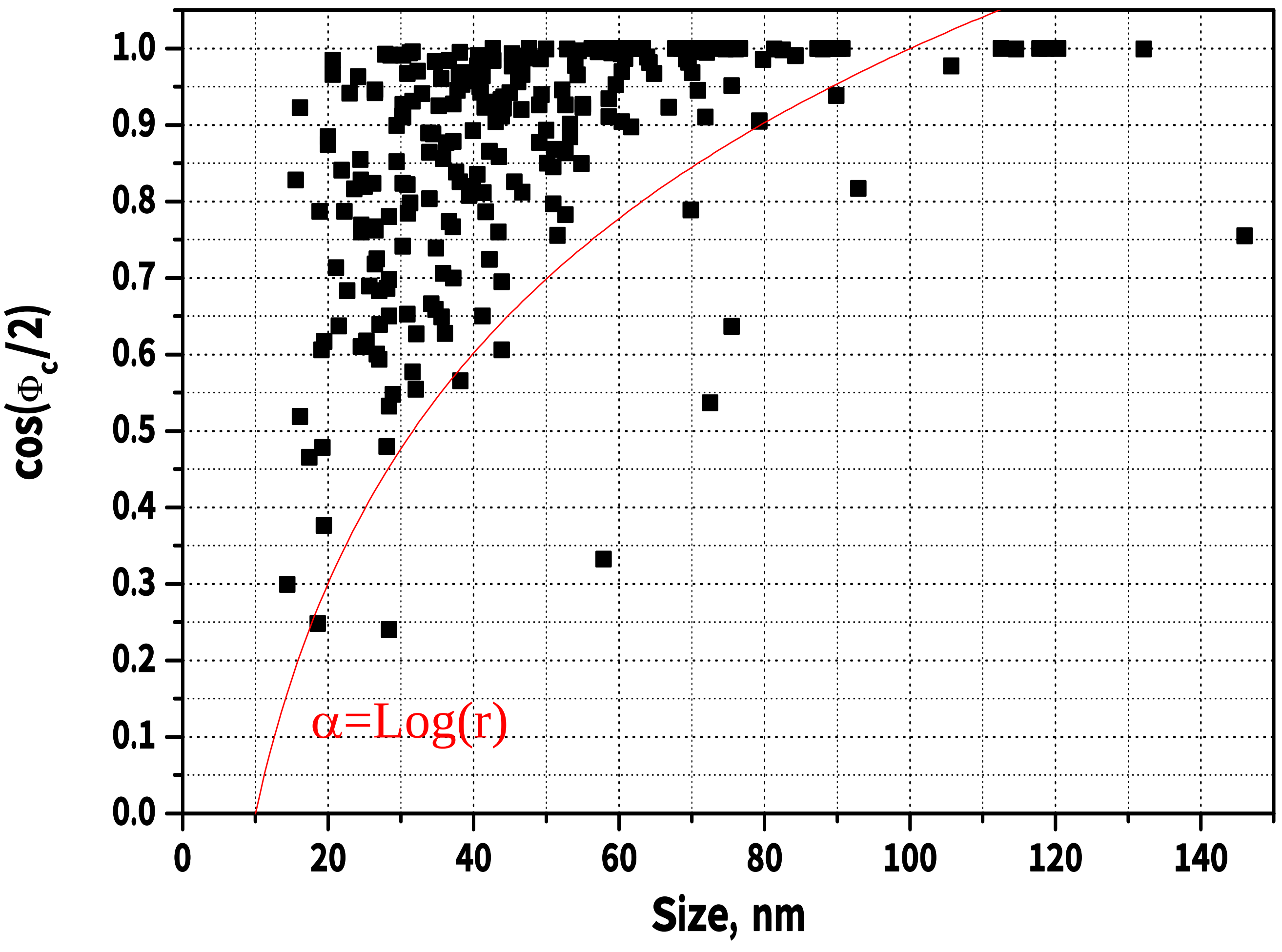

- Obstacle strength increases with the size.
- Strength factor of cavities which are larger than 70nm is almost 1.

Cross slip of screw dislocations at large cavity

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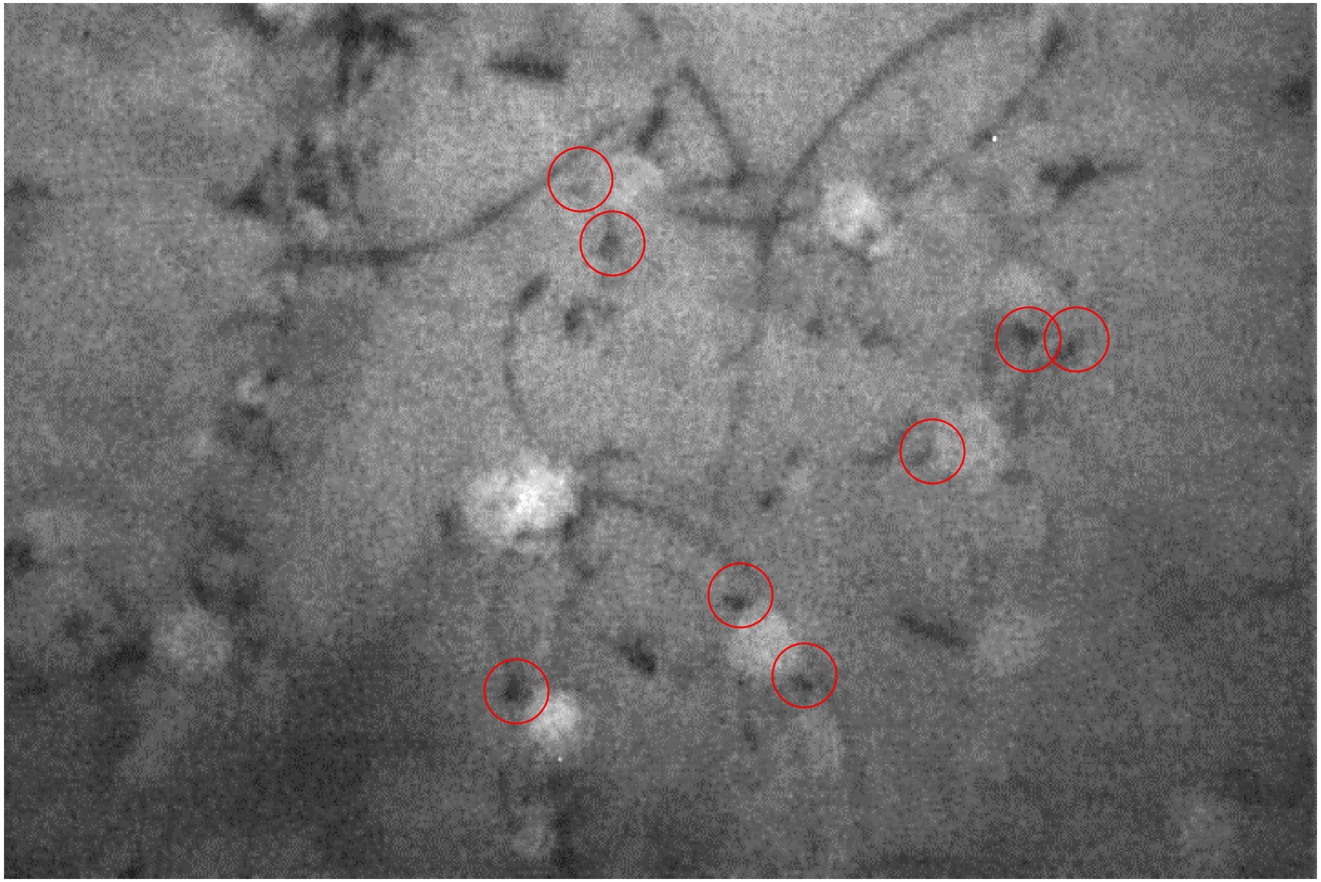
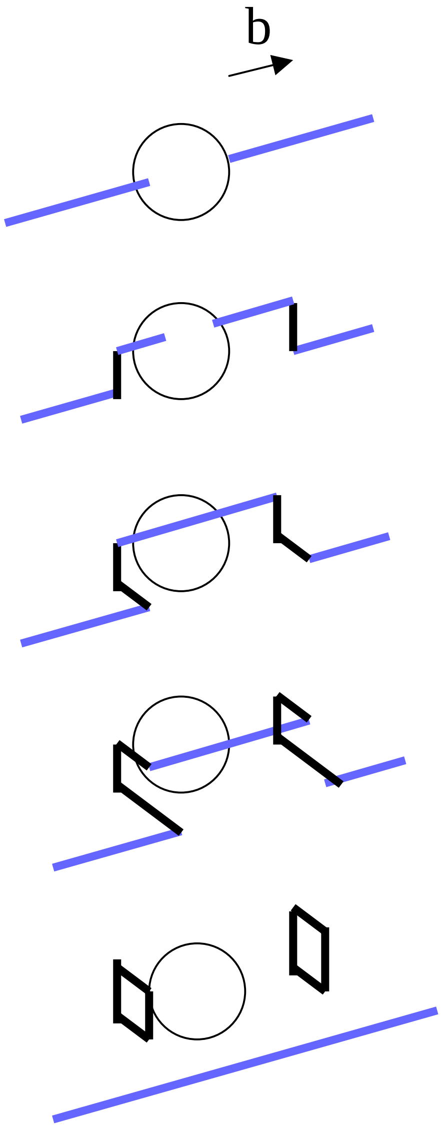
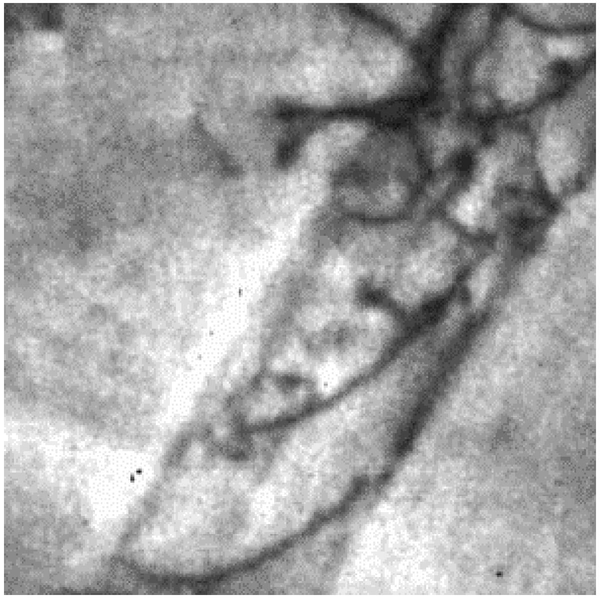


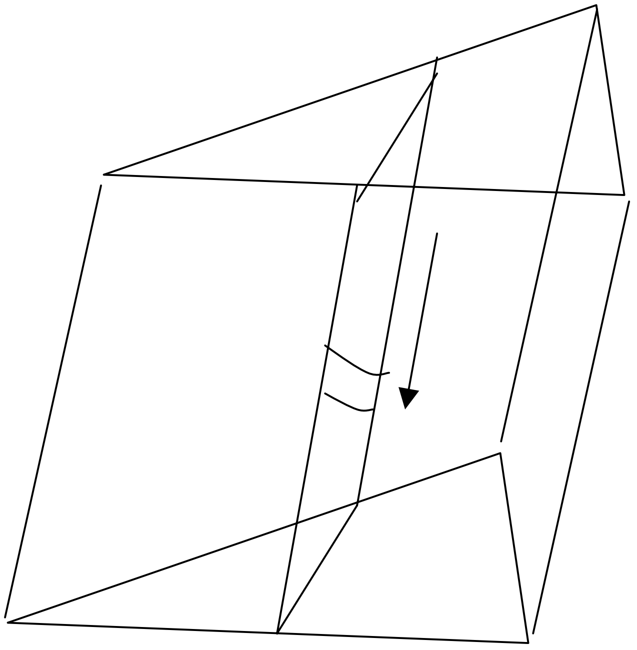


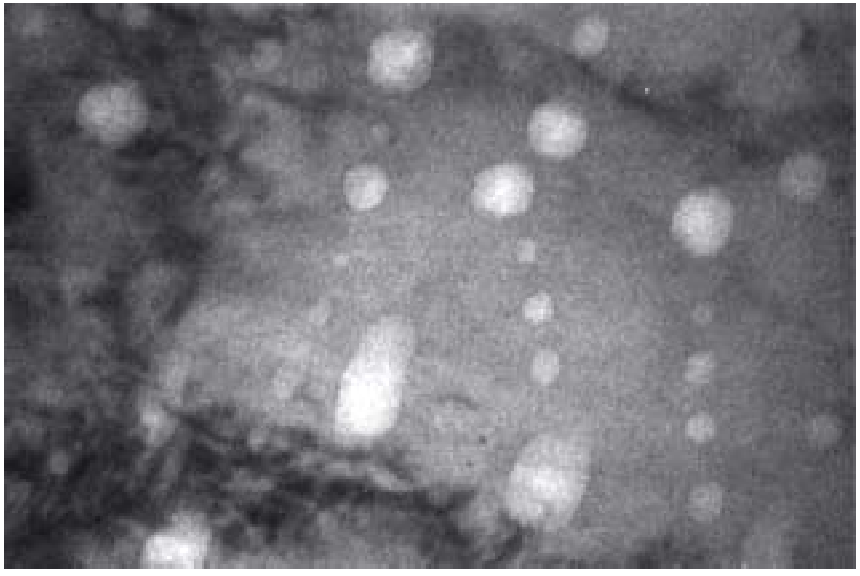
By-pass process of dislocation may occur at large cavity…
Small black dotted contrast left around the cavity.
← Dislocation loops might be left after an double cross slip.

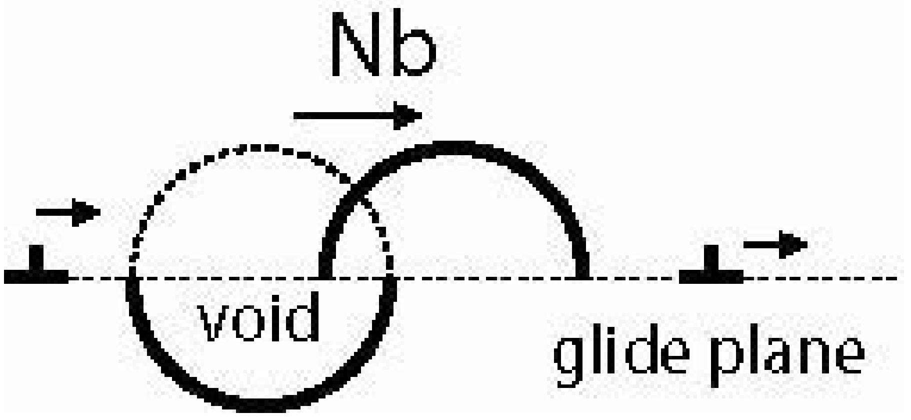
<u>Debris loops will act as new obstacles for subsequent dislocation.</u>

Glide of several dislocations









Stacking fault and partial dislocation

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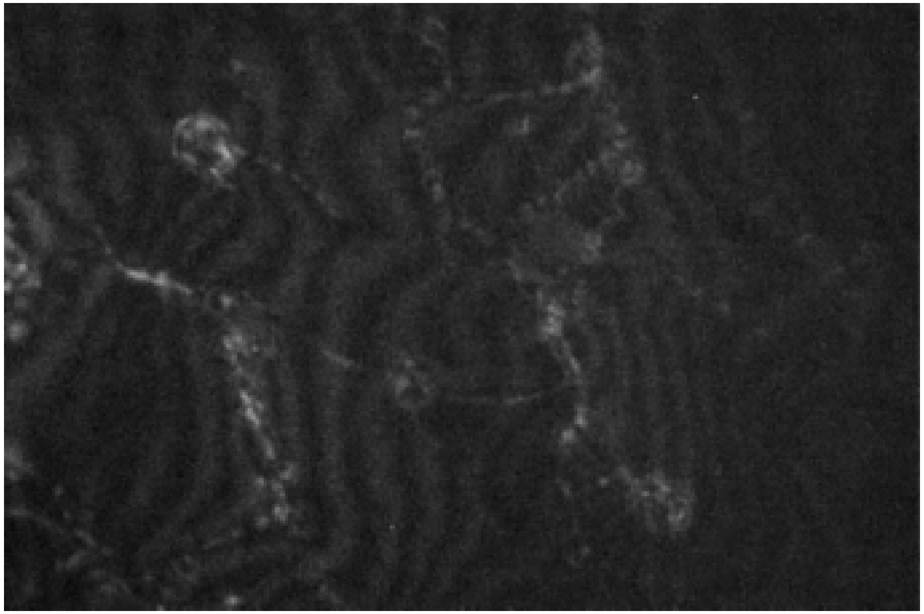
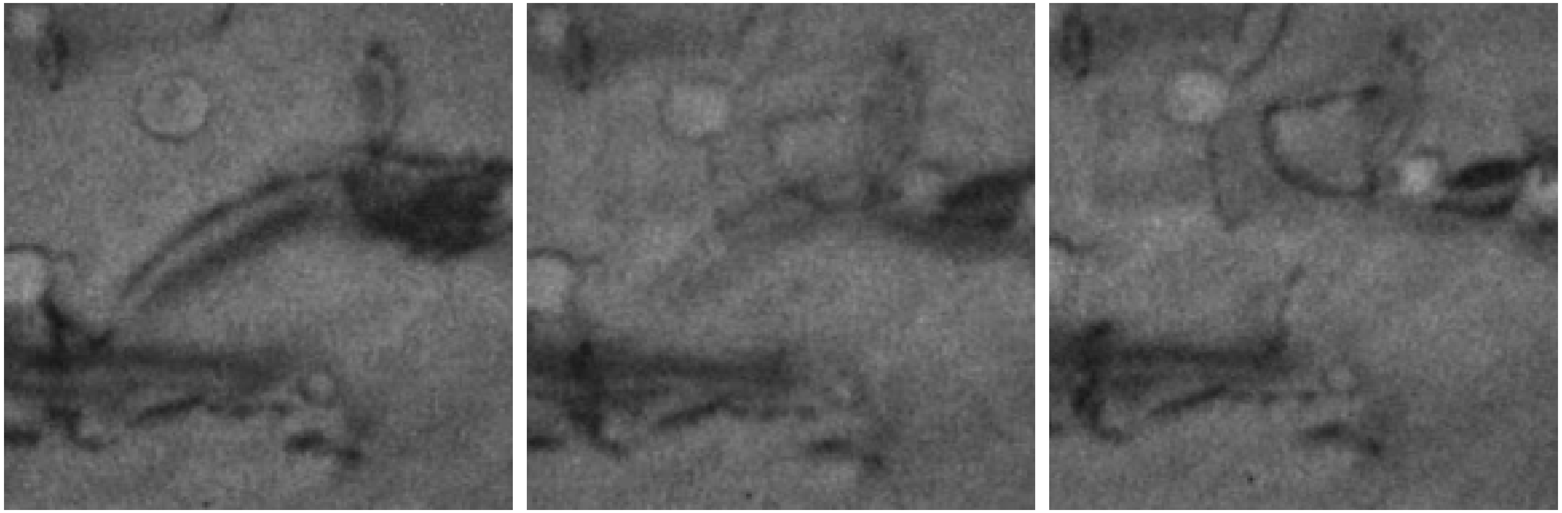


<u>Dark field image</u>

Partial dislocation is not clear

<u>During in-situ straining</u>

Few dislocations are observed as partials with stacking fault



Conclusions

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Interaction between mobile dislocations and cavities were examined by in-situ TEM observations of pure copper irradiated by helium ions.

- Dislocations were pinned at cavities which were randomly dispersed in the matrix.

  →Direct evidence of contribution of cavity to the hardening.

  Mobile dislocation interacted with obstacles were determined as a screw type.
- According to the frame-by-frame analysis, the bow-out angles were small, which indicate that the cavities played as a strong obstacle to the dislocation motion. Strength factor α were 0.86 for bubble (47.2nm).
- Obstacle strength increases with obstacle size. Strength factor of cavities which are larger than 70nm is almost 1.
- Distance from the center of cavity to the glide plane is important parameter to discuss the obstacle strength.
- The attractive interaction between cavity and dislocation was observed by in-situ experiments.
- Hardening increased with bubble pressure, although the number density, size of bubbles and other extended defects should be considered.