Study on solute-defects interaction in iron-based model alloys under ion irradiation

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Challenges for Materials in Nuclear Power System

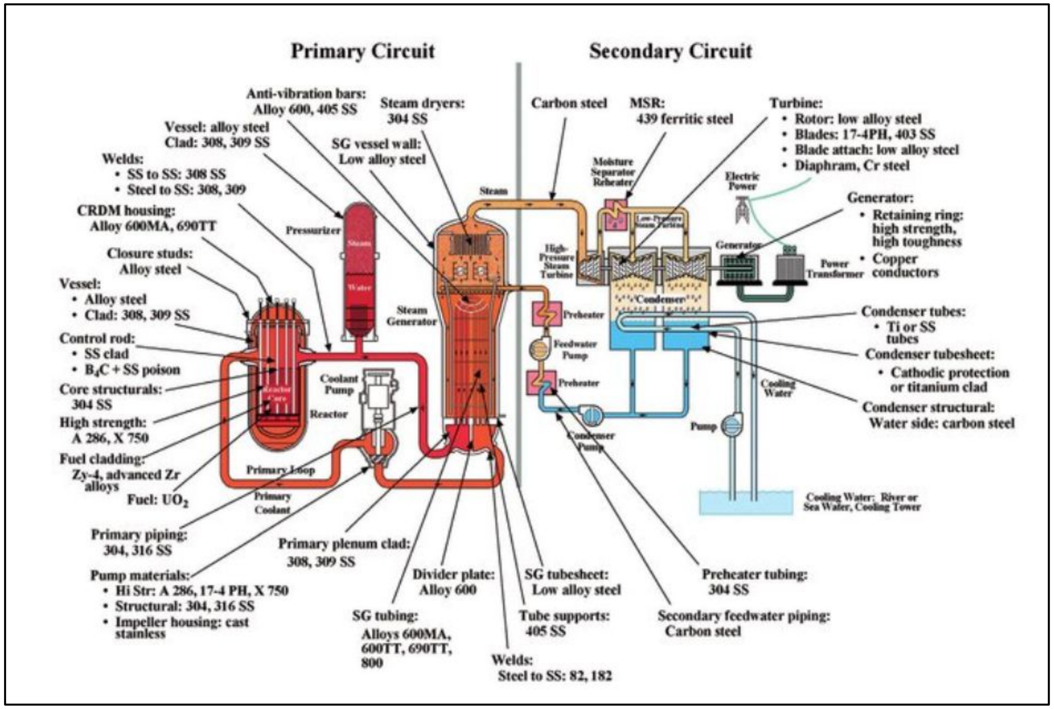
The goal of the nuclear power plant owners and/or operating organizations is to operate for as long as this is economically feasible and safety can be maintained.

One of the issues for plant life management for long term operation is ageing degradation of Systems, Structures and Components (SSC).

Materials degradation in a NPP is extremely complex due to the harsh environment. Components within a reactor core are exposure to high temperature water, stress, vibration, an intense field of high-energy neutrons, or gradients in temperature.

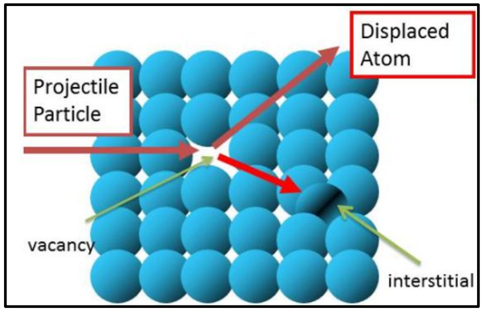
Reactor pressure vessel made of low alloy steel is selected as the research target

- Irreplaceable component
- Irradiation embrittlement
- Degradation behavior is sensitive to small difference in the material



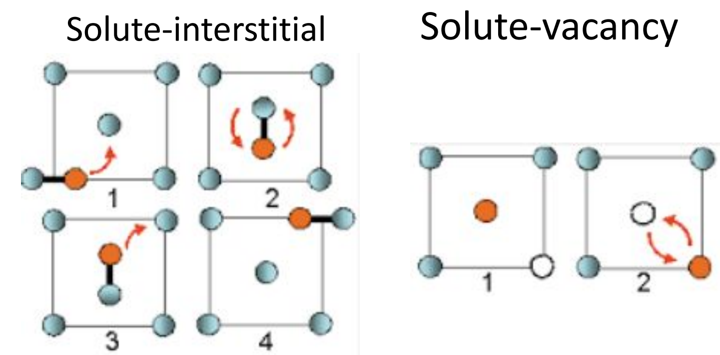
Radiation Damage in Crystalline Materials

An **elastic collision** can eject an atom from its normal lattice position



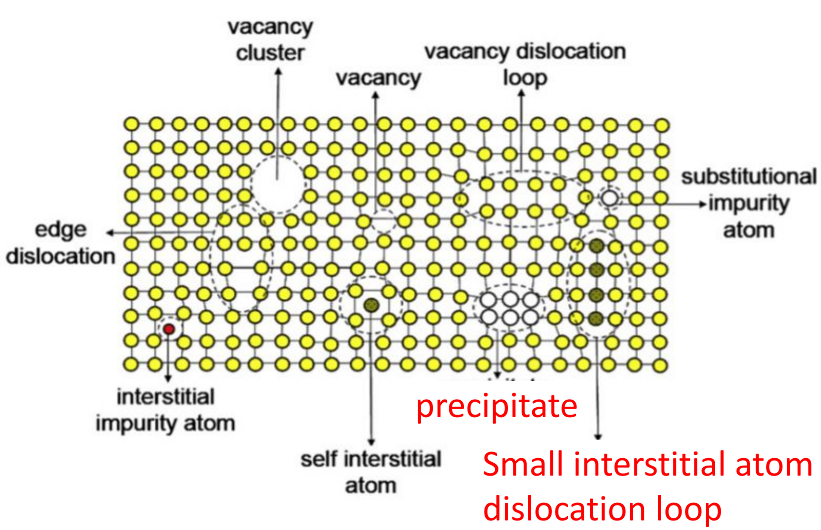
Depends on the major elements and projectile

Diffusion of defects with solute in solid



Sensitive to slight difference in the material

Forms various **nanostructures**



Degradation

- Void swelling (shape change)
- Accelerated corrosion
- Stress corrosion cracking
- Toughness reduction
- Heat conductivity redaction

Objectives

- Development of unique evaluation method of solute-defects interaction in metals using ion accelerator

Considering the solute elements interaction with radiation defects such as vacancy and interstitial and lead to hardening, the research objectives are categorized.

(1) to develop instant technique to evaluate irradiation effect by hardening measurement.

(2) to develop the methodology for evaluation of solute-vacancy interaction in metals using He ion irradiation.

(3) to evaluate solute-interstitial interaction in metal using Fe ion irradiation.

Experimental Design

Experiment (1)

proportional

proportional

Toughness ← Yield Stress ← Instrumentation indentation hardness

Nanostructures → Yield Stress

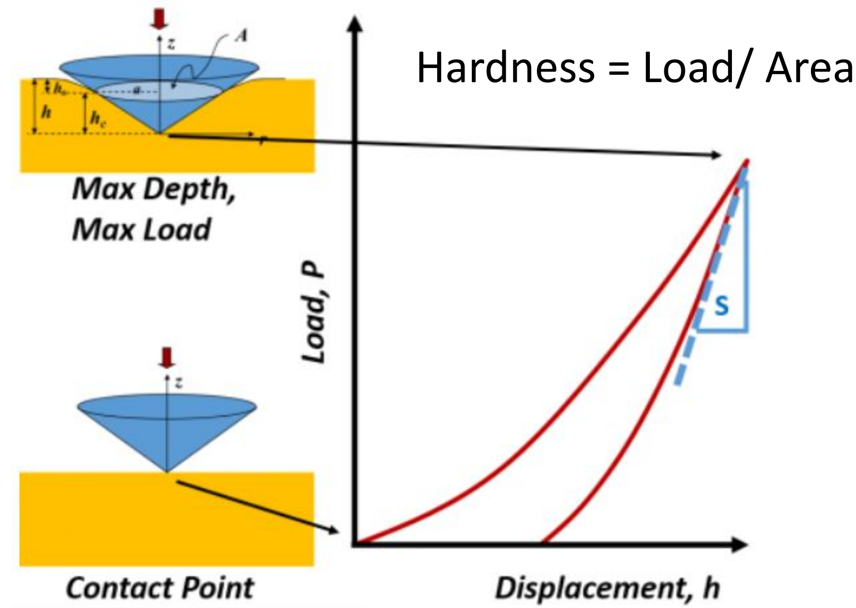
The classic Orowan model

Hardening coefficient is calculated

$$\boxed{\Delta\sigma_k = \alpha_k M \mu b (N_k \, d_k)^{1/2}}$$

Measured yield strength change contributed by defect *k*

Measured number density & average size of defect *k*

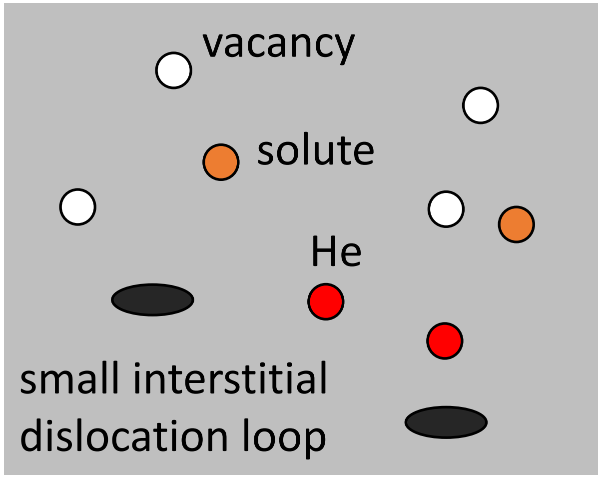


$$h_c = h - \varepsilon \frac{P}{S} \qquad A = f(h_c)$$

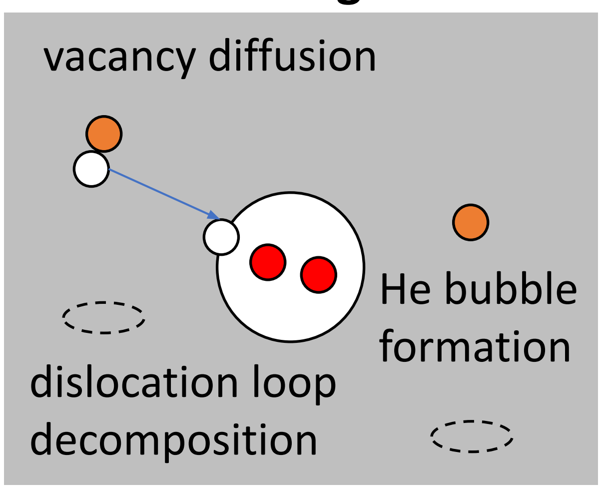
$$E = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \qquad H = \frac{P}{A}$$

Experiment (2)

As Irradiation at RT



After Annealing



As Irradiation:

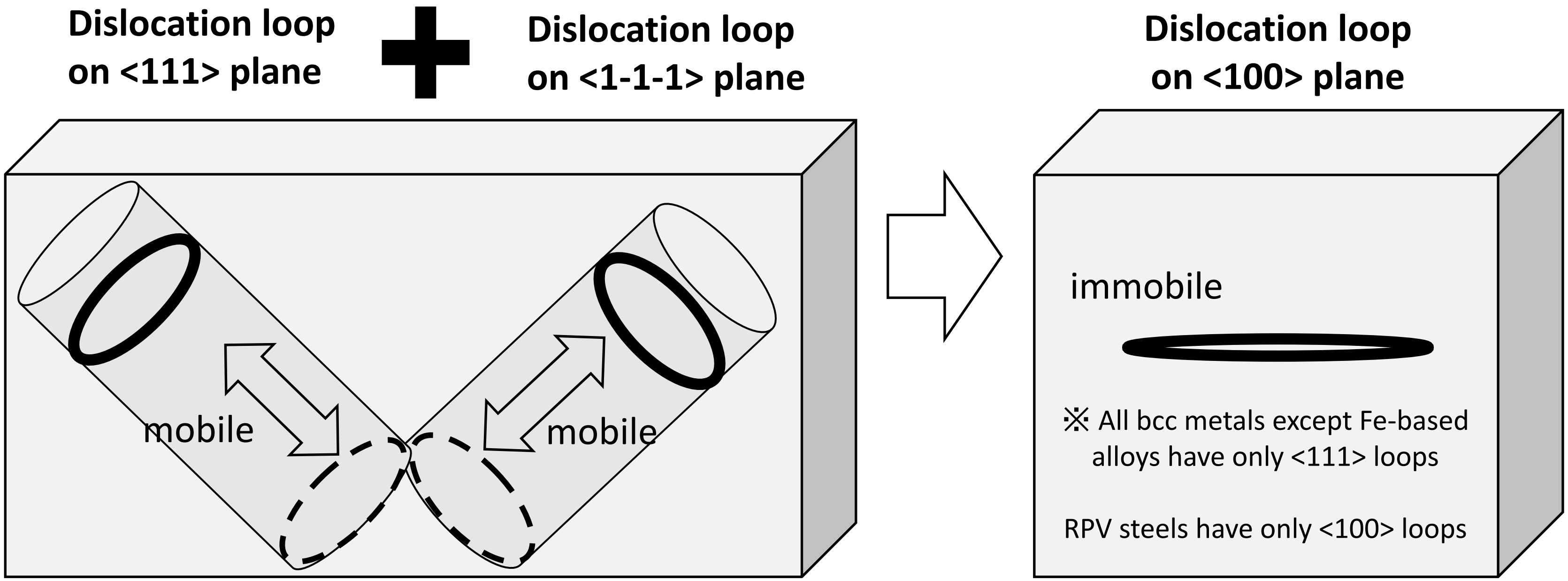
- Vacancies, small interstitial atomic dislocation loops, and helium atoms are dispersed in the iron matrix by irradiation.

After annealing:

- A vacancy start diffusing and react with solute atoms in the iron.
- Vacancies start clustering and form a bubble, and helium atoms stabilize the bubble.
- Dislocation loops start decomposed.

Size and number density of helium bubbles can be indicators for **the solute atoms effect on diffusion coefficient of vacancies**

Experiment (3)



Possible Effect on solute element for stabilizing <100> loops

- Mixed dumbbell migration of solute atoms with interstitials
- Irradiation-induced segregation of solutes on loops
- Reduction of mobility of <111> loops
- Change the share energy of dislocations

Methodology

Chemical composition of materials

Model alloys based on the specification of A508 class 3 nuclear grade steel
The alloys were common in all experiments

	C	Si	Mn	P	S	Ni	Cr	Cu	O	N
A508 class 3 Nuclear Grade	<0.25	<0.40	1.20 ~1.50	<0.008	<0.004	0.40 ~1.00	<0.25	<0.05		
Fe	0.0065	<0.01	<0.01	<0.003	0.0021	<0.02	<0.02	<0.02	0.0087	0.0006
Fe-0.3Si	0.0085	0.30	<0.01	<0.003	0.0019	<0.02	<0.02	<0.02	0.0018	0.0006
Fe-0.6Ni	0.0077	<0.01	<0.01	<0.003	0.0017	0.59	<0.02	<0.02	0.0034	0.0006
Fe-1.4Mn	0.0094	<0.01	1.49	<0.003	0.0022	<0.02	<0.02	<0.02	0.0021	0.0009
Fe-0.6Ni-1.4Mn	0.0094	<0.01	1.46	<0.003	0.0022	0.58	<0.02	<0.02	0.0012	0.0009

Specimens Preparation

Annealing Process

Cutting

Polishing

Irradiation

Ion-beam irradiation techniques

Analysis

Heating at 1000 ^{0}C for 1 hr
Heating at 800 ^{0}C for 1 hr
Quenching with water
Heating for 600 ^{0}C for 24 hrs

Rough polishing
Bath polishing
Electro-chemical polishing

Nanostructure evaluation

FIB
TEM

Irradiation-induced hardening

Nanoindentation

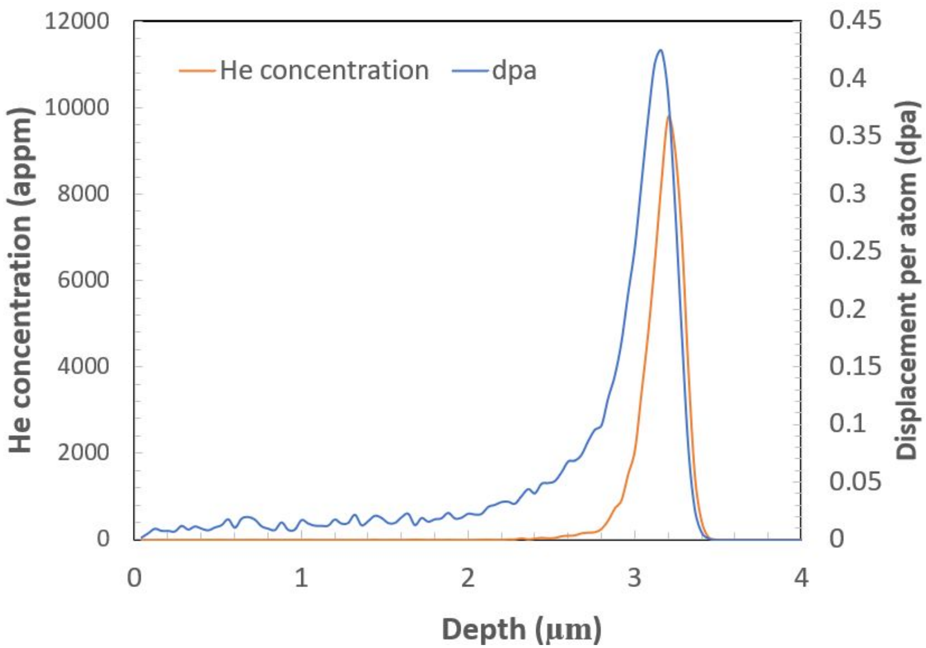
Results and discussion:

Irradiation-induced Hardening

Depth profile of irradiation damage

Stopping and Ranges of Ions in Matter (SRIM) simulation

SRIM is the Monte Carlo simulation code used for calculating ion deposition profiles in materials by ion implantation. SRIM has the capability to compute the radiation damage exposure which is in the unit of displacements per atom (dpa). dpa is a standard measurement for primary radiation damage production. It is possible to simulate the effects of neutron irradiation by calculating dpa through ion irradiation.

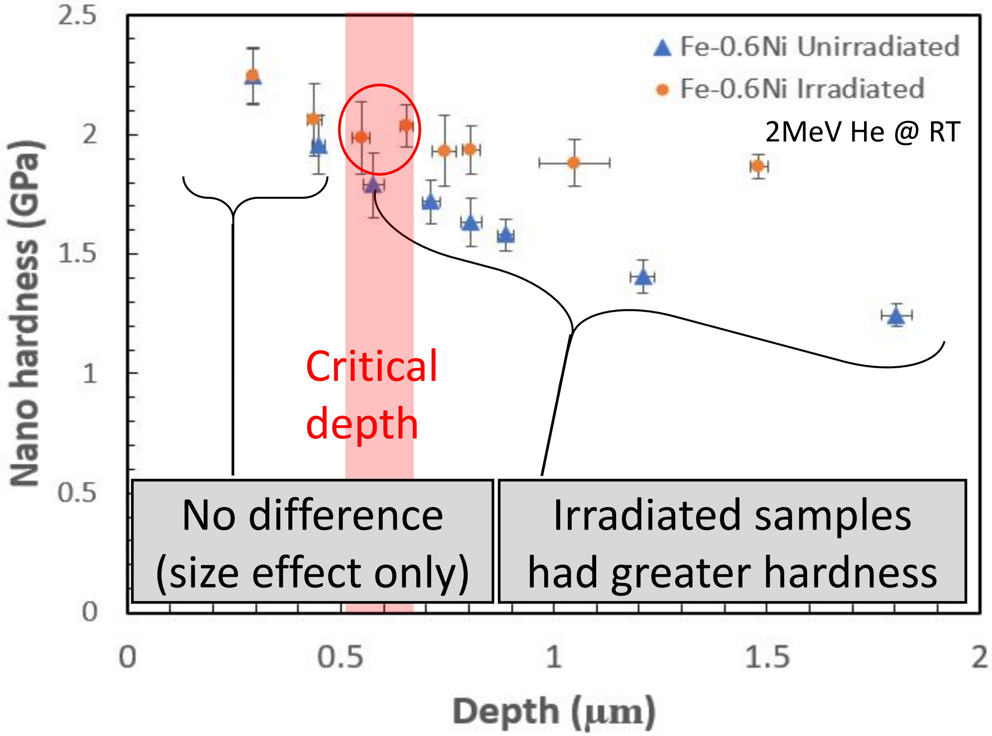


Ion: Helium
Materials: Iron
Irradiation Energy: 2MeV
Fluence: 2.11 x 10^{16} ions/cm^2

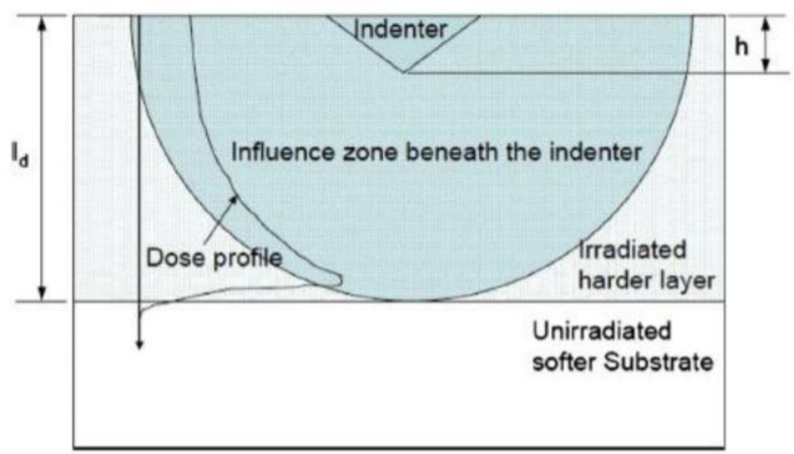
Irradiation depth: ~3.4 μm
He concentration at peak: 10000 appm
Displacement per atom at peak: 0.43 dpa

Depth Dependence on Hardening in He-irradiated Fe alloys following 400 °C annealing

Hardness was measured with varying loads after annealing at 400 °C



Critical depth was 0.65～0.7 µm

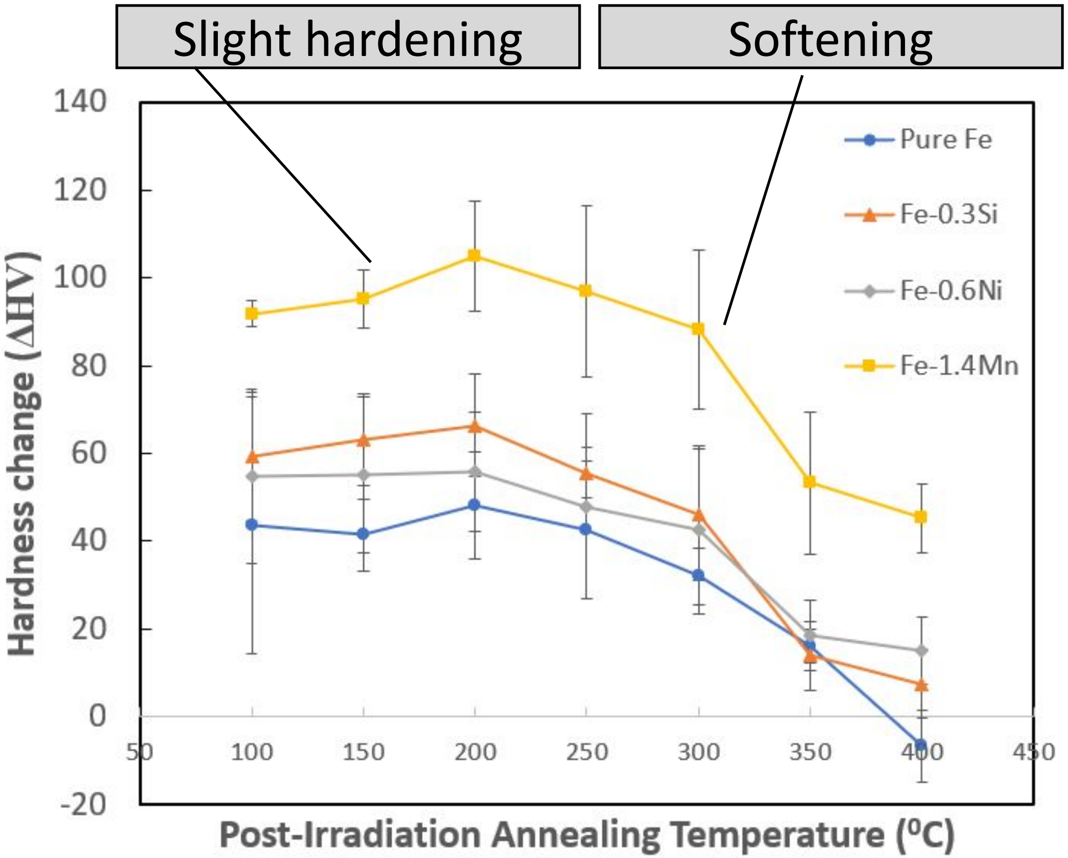


- ✔ **Hardness recover was only observed near the surface**
- ✔ Residual irradiation defects contributed to the hardness deeper than the critical depth

Results and discussion:

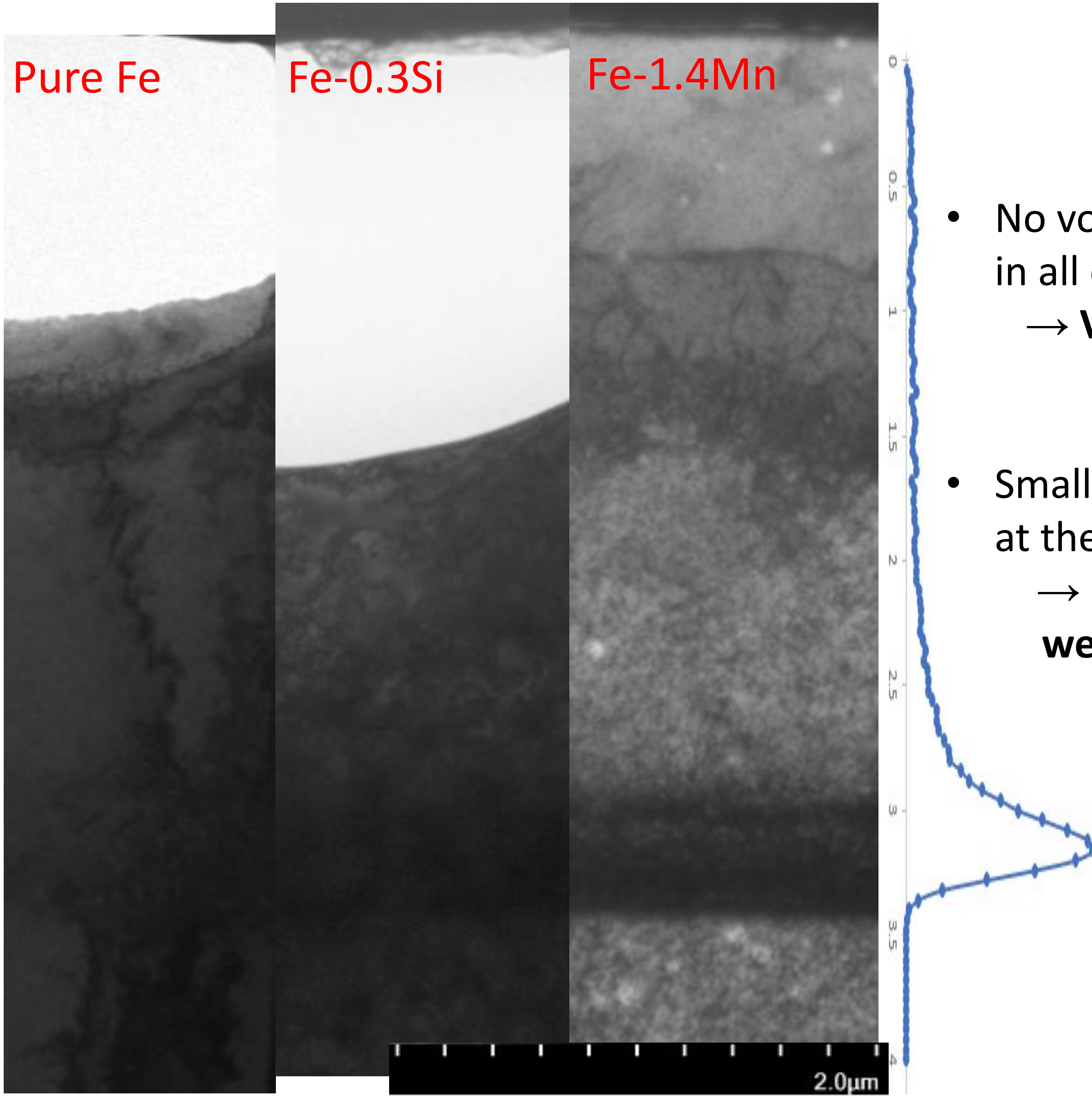
Vacancy diffusion in Fe-based alloys under He ion irradiation

Post-irradiation annealing induced hardening



- Annealing up to 200 °C leads hardening, especially in Fe-1.4 Mn
  → **Growth of nanostructure**
- Hardness recovery was observed in all specimens at 250 °C or higher.
- Hardness was almost recovered in pure iron at 400 °C
  → **Decomposition of nanostructure**

Damage distribution following annealing



- No void/bubble was detected in all conditions
  → **Vacancies were recovered without clustering**
- Small defects are concentrated at the damage peak regions
  → **Interstitial atoms clusters were not fully decomposed, but restructured**

Results and discussion:

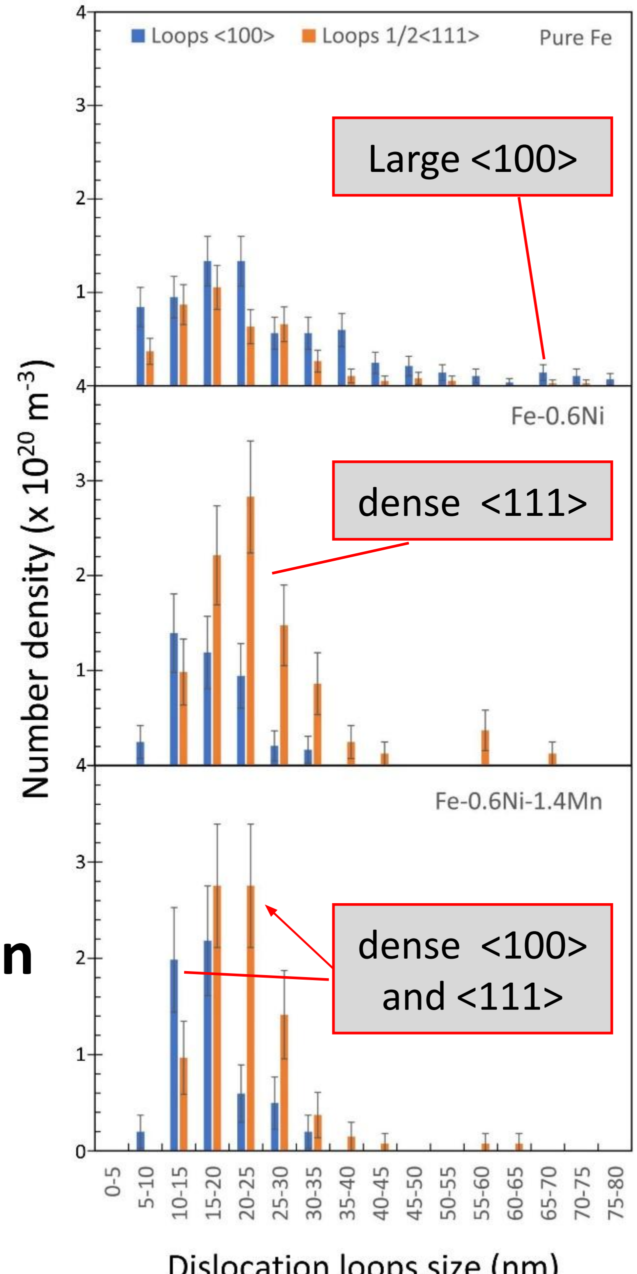
Dislocation loops evolution in Fe-based alloys under Fe ion irradiation

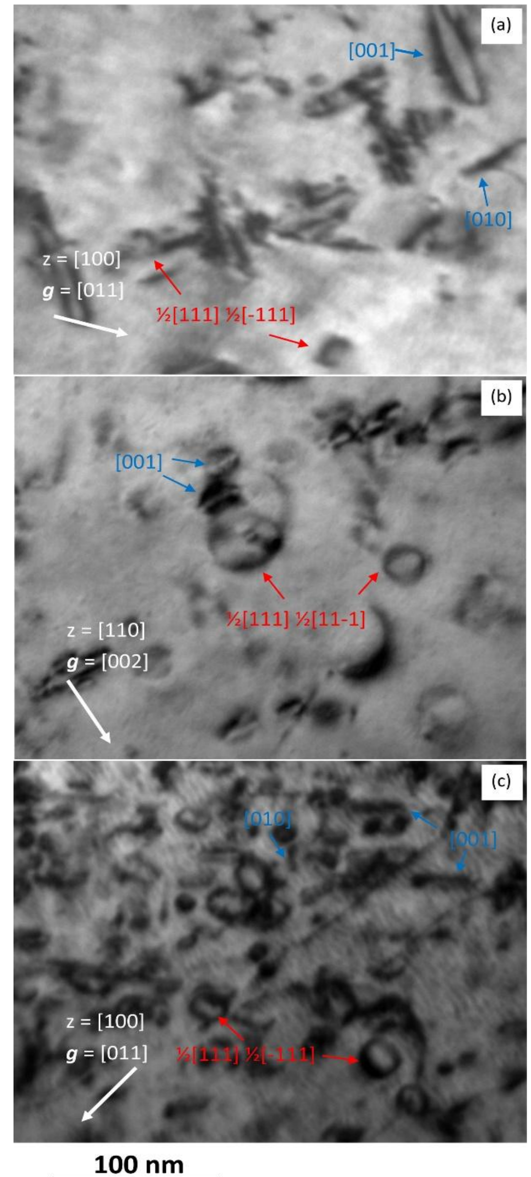
Dislocation loops at 700~900 nm depth

Pure Iron

Fe-0.6Ni

Fe-0.6Ni-1.4Mn



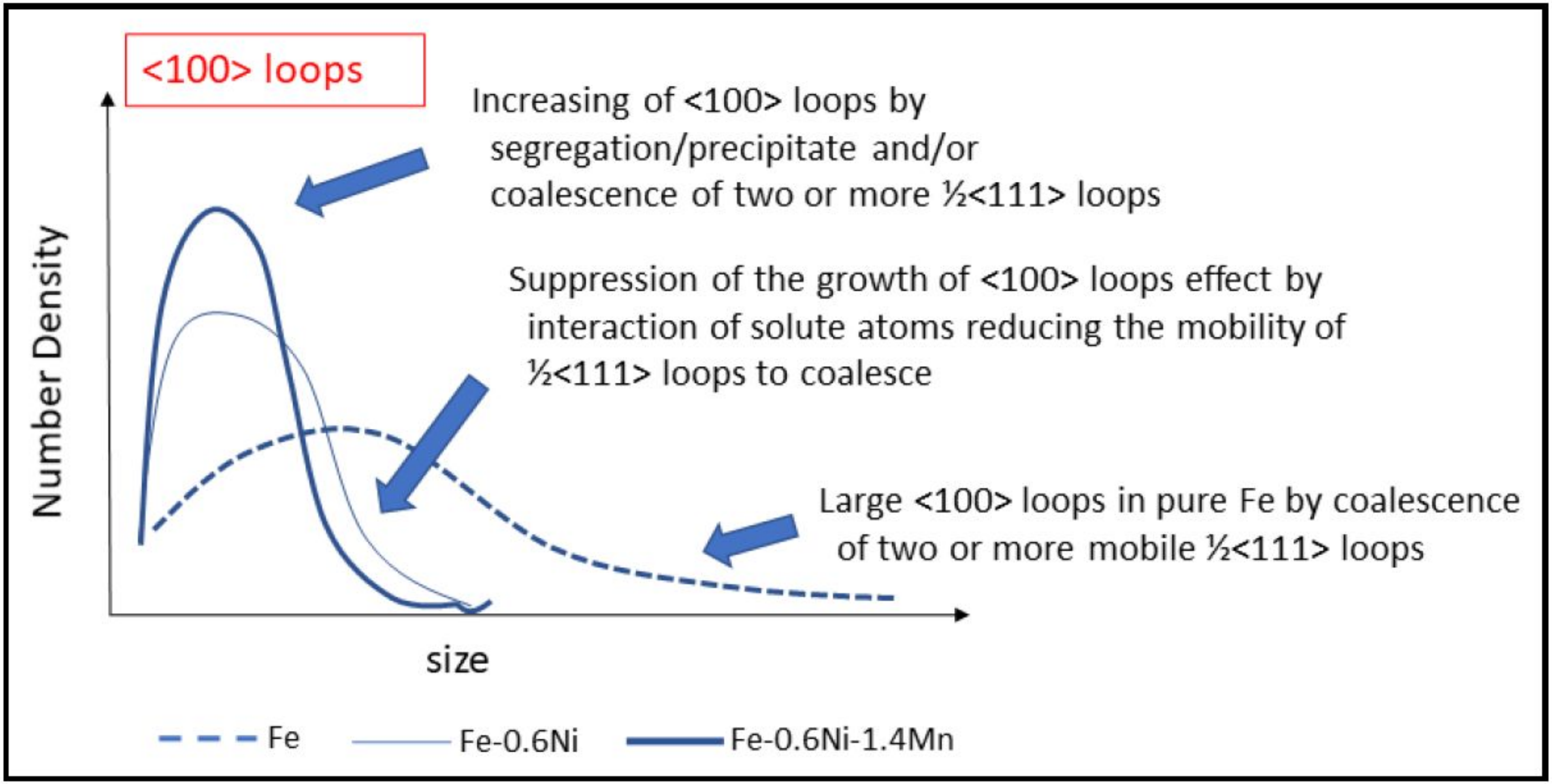


Dislocation loops

	Number density (m^{-3})	Average size (nm)	Fraction of loops (%)	
			½<111>	<100>
Pure-Fe	1.14E+21	24	36.66	63.34
Fe-0.6Ni	1.34E+21	21	69.02	30.98
Fe-0.6Ni-1.4Mn	2.01E+21	20	60.42	39.58

- The number density of dislocation loops increased with the adding of solute elements into Fe, while average size of loops is otherwise.
- The population was dominated by <100> loops in pure Fe.
- ½<111> loops were more dominant than <100> loops in Fe-based alloys

Summary



<100> loops
Increasing of <100> loops by segregation/precipitate and/or coalescence of two or more ½<111> loops
Suppression of the growth of <100> loops effect by interaction of solute atoms reducing the mobility of ½<111> loops to coalesce
Large <100> loops in pure Fe by coalescence of two or more mobile ½<111> loops
Number Density
size
Fe
Fe-0.6Ni
Fe-0.6Ni-1.4Mn

Research Limitation

Helium ions irradiation experiments can be conducted only at room temperature due to limitations in the accelerator design. Therefore, the vacancy or He bubble interaction phenomena are not clearly observed.

Benefit

Myself

- New knowledge in this field.
- Developing my skill in conducting high technology equipment.

Agency

- The expertise in this field can be applied to the current research reactor (RTP) for long-term safe operation.
- Improving maintenance program for RTP.
- Collaboration with universities (inside and outside of the country).

Country

- Enhance competency and capability in nuclear technology.

Implications of study on Government Policy

KEMENTERIAN SAINS, TEKNOLOGI DAN INOVASI

Dasar Sains, Teknologi dan Inovasi Negara (DSTIN)

DSTIN memberi fokus kepada 6 teras strategik

Teras strategik 1: Memajukan Bidang-bidang Penyelidikan dan Pembangunan Saintifik dan Sosial, dan Pengkomersialan (R,D&C);

3. Mengukuhkan perkhidmatan, keupayaan dan kapasiti STI

Teras Strategik 2: Membangun, Memupuk dan Menggilap Bakat

8. Meningkatkan bilangan tenaga kerja teknikal yang mahir dan cekap untuk mengurus, mengendali dan menyelenggara peralatan dan infrastruktur yang khusus

***Dasar Teknologi Nuklear Negara (DTNN)**

Thank you very much