

Hardening effect of helium bubbles in ferritic/martensitic steels

Lei Peng^{1,2}, Z. Tong³, Yong Dai⁴

¹ School of Nuclear Science and Technology, USTC, China

² Institute of Plasma Physics, CAS, China

³ China Institute of Atomic Energy, China

⁴ Paul Scherrer Instiut, Switzerland

Outline

Introduction

Experimental

Results

Discussion

Conclusions

Introduction

Helium-induced hardening of RAFMs

The **Reduced Activation Ferritic/Martensitic (RAFM) steels** are presently considered as the primary candidate blanket structural materials for fusion reactors. They have also great potential to be applied to target structures of high power spallation neutron sources.

High displacement damage and helium (He) concentration will be produced in structural materials by intense flux of high energy neutrons. The understanding of helium-induced hardening and embrittlement effects in MF steels is one of key issues in the current fusion and spallation materials programs.

Fusion reactors

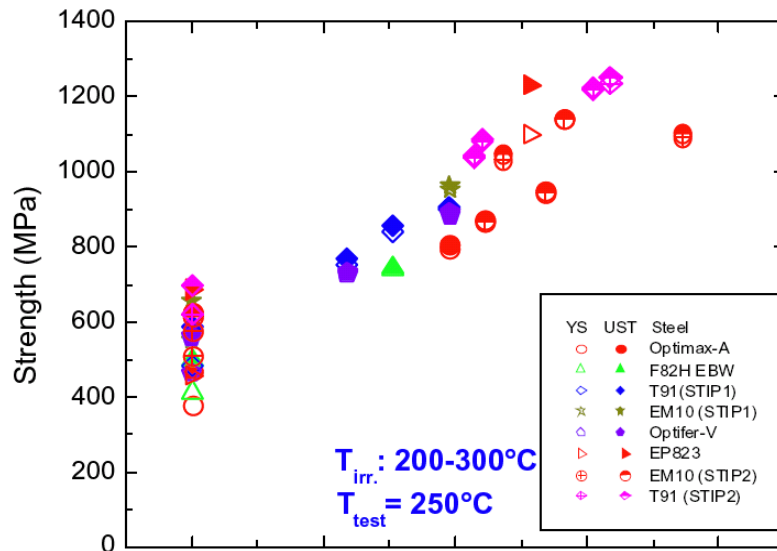
Flux (n/cm²·s)	>10¹⁵
Dose	20-30 (dpa/yr)
He production	10-15 (appm/dpa)

Spallation targets

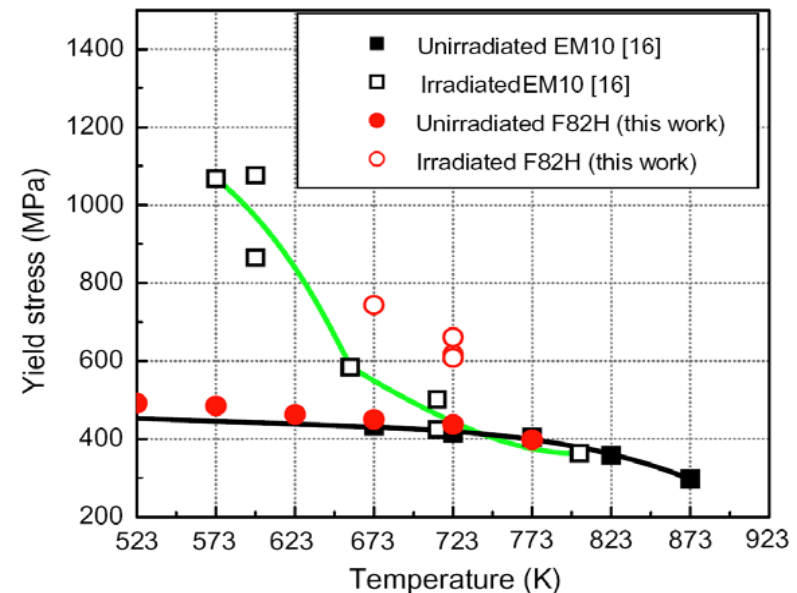
Flux (n/cm²·s)	>10¹⁴
Dose	10-20 (dpa/yr)
He production	< 100 (appm/dpa)

Helium effects on hardening

In recent studies, various RAFM steels were irradiated to 20 dpa / 1800 appm He at Swiss spallation neutron source (SINQ). A series of studies have been conducted on the mechanical properties and microstructure of F82H and Optimax-A steels in SINQ target irradiation program (STIP). The results demonstrate that **He can introduce significant hardening effects**, even at temperatures above 400° C where neutron irradiations don't show any evident changes in strength of RAFM steels .



~70 appm/dpa He, <400°C



~70 appm/dpa He, >400°C

Micro-hardness and microstructure

To investigate the effects of helium on hardening after irradiation, [annealing experiments](#) have been done to recover the irradiation-induced changes in mechanical properties and microstructure.

[Fast and inexpensive](#) hardness testing have been used for rapid overall mechanical property evaluations instead of elaborate tensile testing because of its [non-destructively testing and saving in material](#).

[Hardness and strength have a good correlations.](#)

In the present work, [micro-hardness and microstructure](#) before and after [annealing](#) of RAFM steels, F82H and Optimax-A, after irradiation (4.6~11.3 dpa, 300~1200 appm, <400°C) have been investigated.

Experimental

Materials

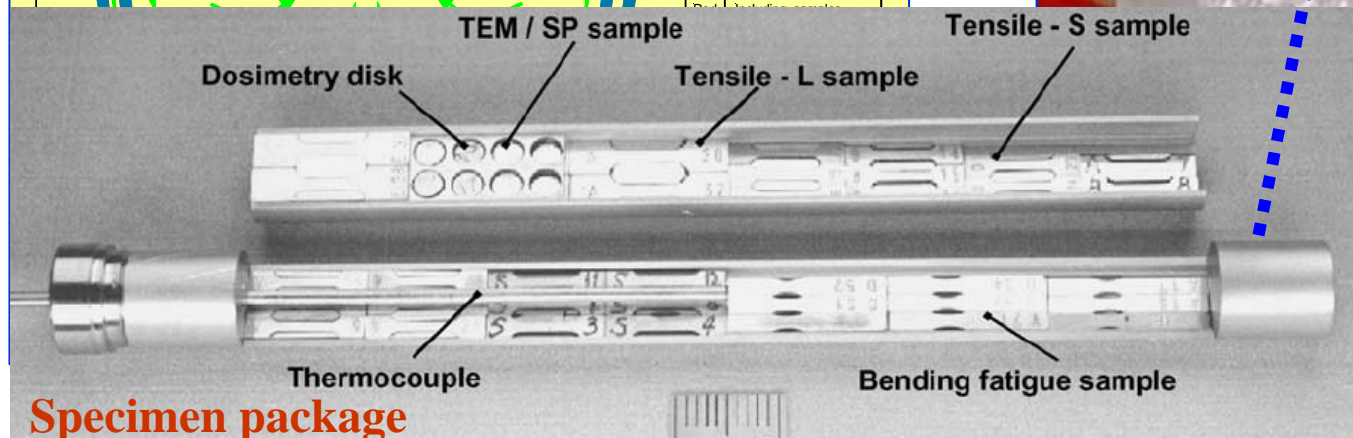
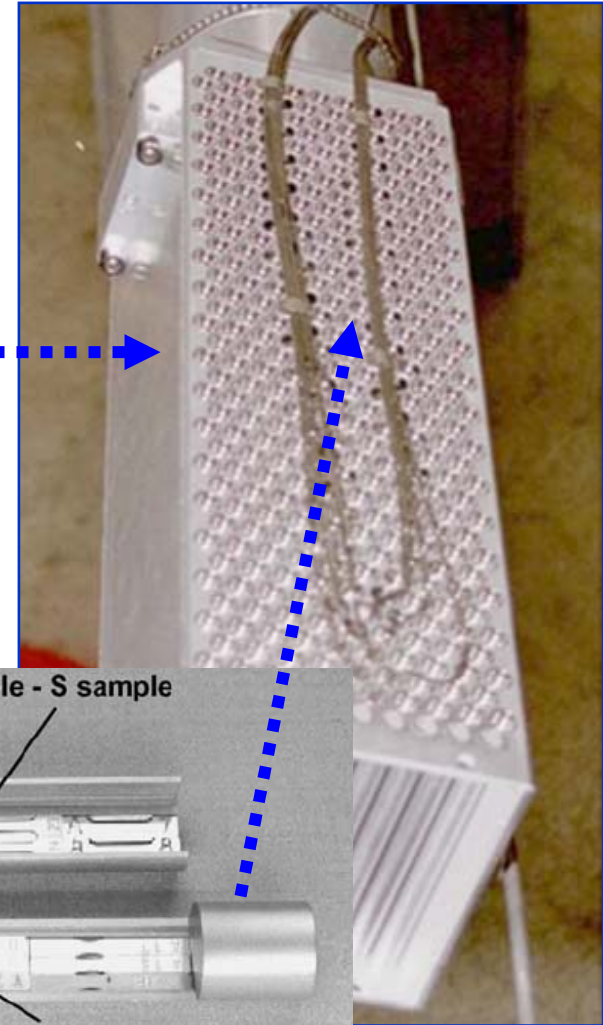
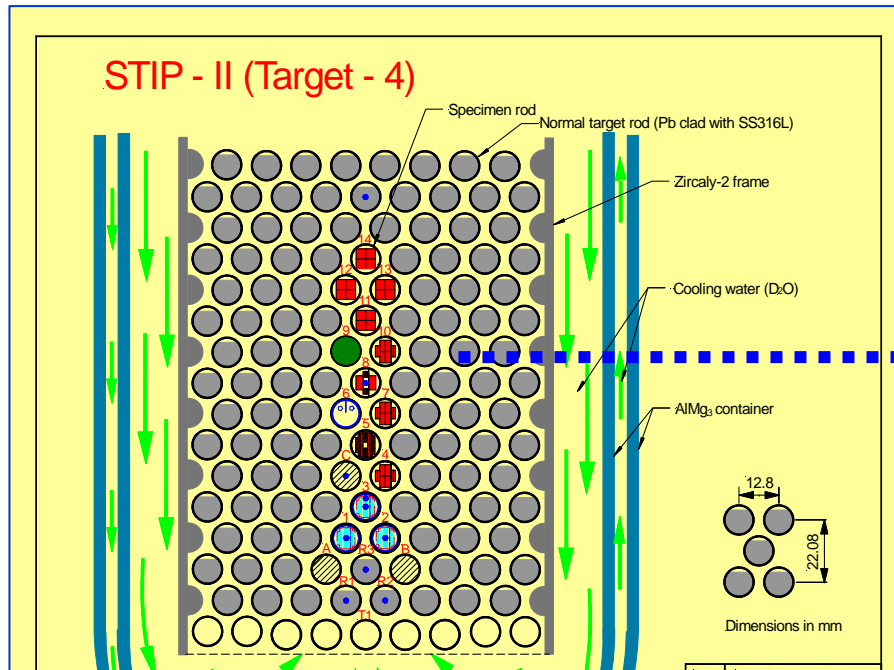
➤ Compositions

RAFM _s	Cr	W	V	Mn	C	Si	Mo	Nb	P
Optimax-A	9.3	0.97	0.24	0.6	0.098	0.02	0.09	<0.01	0.01
F82H	7.87	1.98	0.19	0.1	0.09	0.07	0.003	0.002	0.003

➤ Heat treatments

RAFM _s	Normalizing	Tempering
Optimax-A	1050 °C	750 °C /120min
F82H	1040 °C /30min	750 °C /60min

SINQ Target Irradiation Program - STIP



Irradiation conditions

➤ STIP-I

Miniature type tensile specimens were irradiated in SINQ spallation targets

RAFM _s	Irr. Temp. (°C)	Dose (dpa)	He (appm)
F82H	184	5.3	340
	159	4.64	300
	175	5.3	340
	151	4.64	300
	260	8.96	720
	307	10.49	910
	256	8.96	720
	302	10.49	910
	369	11.32	1173
	369	11.32	1173
	345	11.32	1173
	345	11.32	1173

RAFM _s	Irr. Temp. (°C)	Dose (dpa)	He (appm)
Optima x-A	161	6.27	430
	116	4.64	280
	161	6.27	430
	116	4.64	280
	277	10.49	910
	235	8.96	720
	235	8.96	720
	277	10.49	910

➤ Helium Implanting

560 appm / 0.09 dpa,

1350 appm / 0.22 dpa; 25~70°C.

Sample preparation

➤ Procedure

Cutting (from grip sections of tensile specimen)

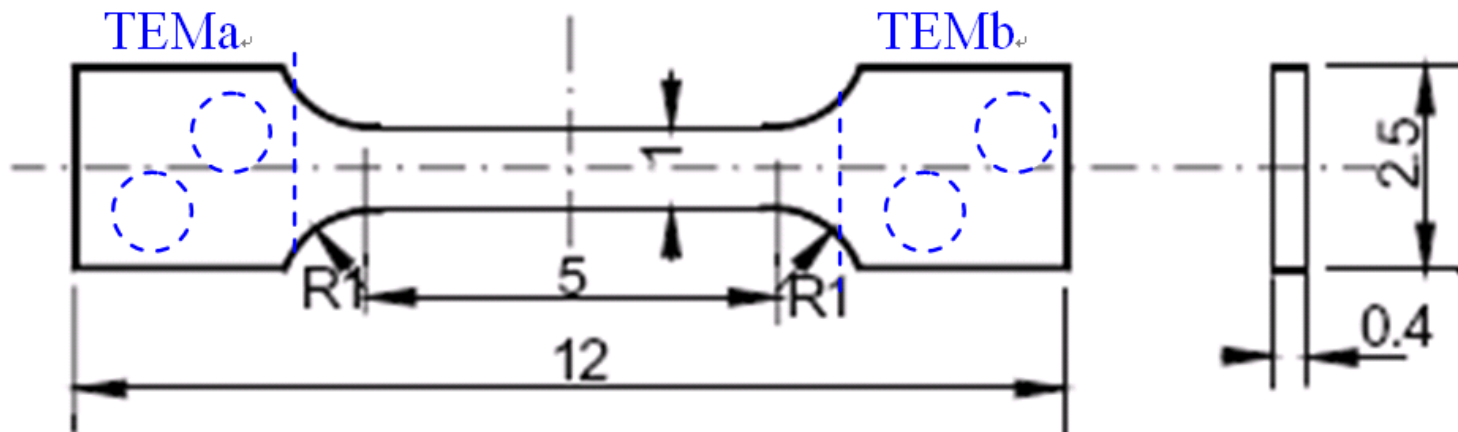
Punching (1 mm disc)

Annealing (500°C / 600°C / 700°C, 2 hours)

Embedding (in 3 mm SS316 ring)

Polish

Electron polish (for TEM observation)



Test methods

➤ Micro-hardness Test

Vickers micro-hardness measurements (HV 0.05) on the grip sections of tensile specimen. (Mean value of 10 indentations)

➤ TEM Observation

The TEM investigation on the microstructure was performed with a JEOL 2010 type microscope. The most often used image conditions were two beam bright field (TBBF) and weak beam dark field (WBDF) at (g, 5g), $g = 110$.

Micrographs of WBDF at (g, 5g), $g = 110$ were used to quantifying the size and number density of defect clusters and dislocation loops for all the samples and micrographs of two beam

TBBF ($g = 110$) were used for quantifying the size and number density of helium bubbles for most samples.

The thickness of observed area of a thin foil was deduced from the number of fringes in micrographs of WBDF at (g, 5g), $g = 110$.

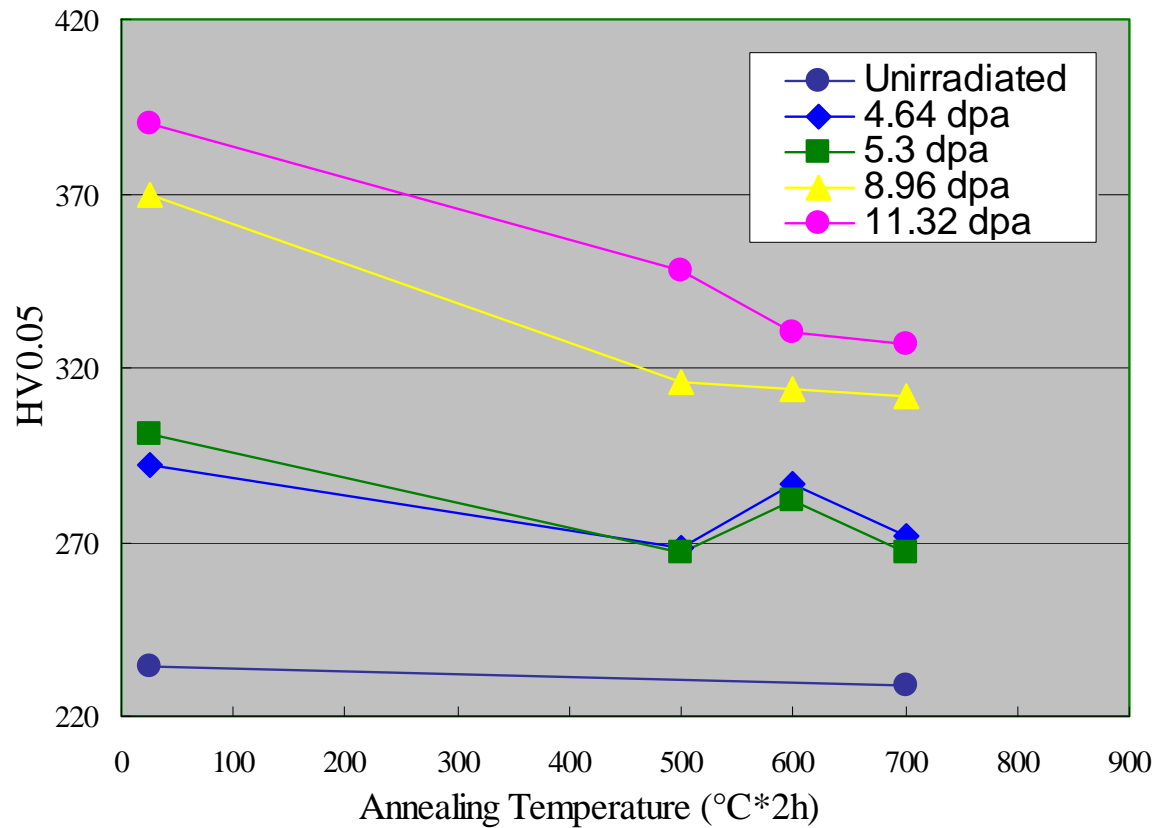
Results

(Micro-hardness and Microstructure)

- ✦ Different temperature annealing
- ✦ 600° C/2h annealing on F82H
- ✦ 600° C/2h annealing on Optimax-A
- ✦ Annealing on F82H after He implanted

Different temperature annealing

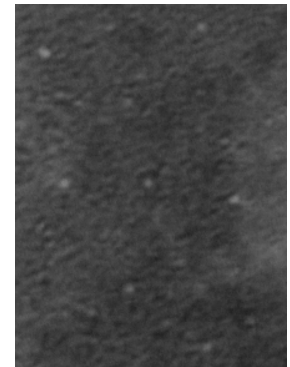
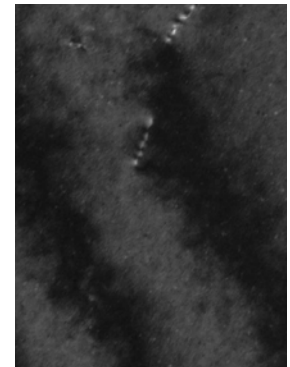
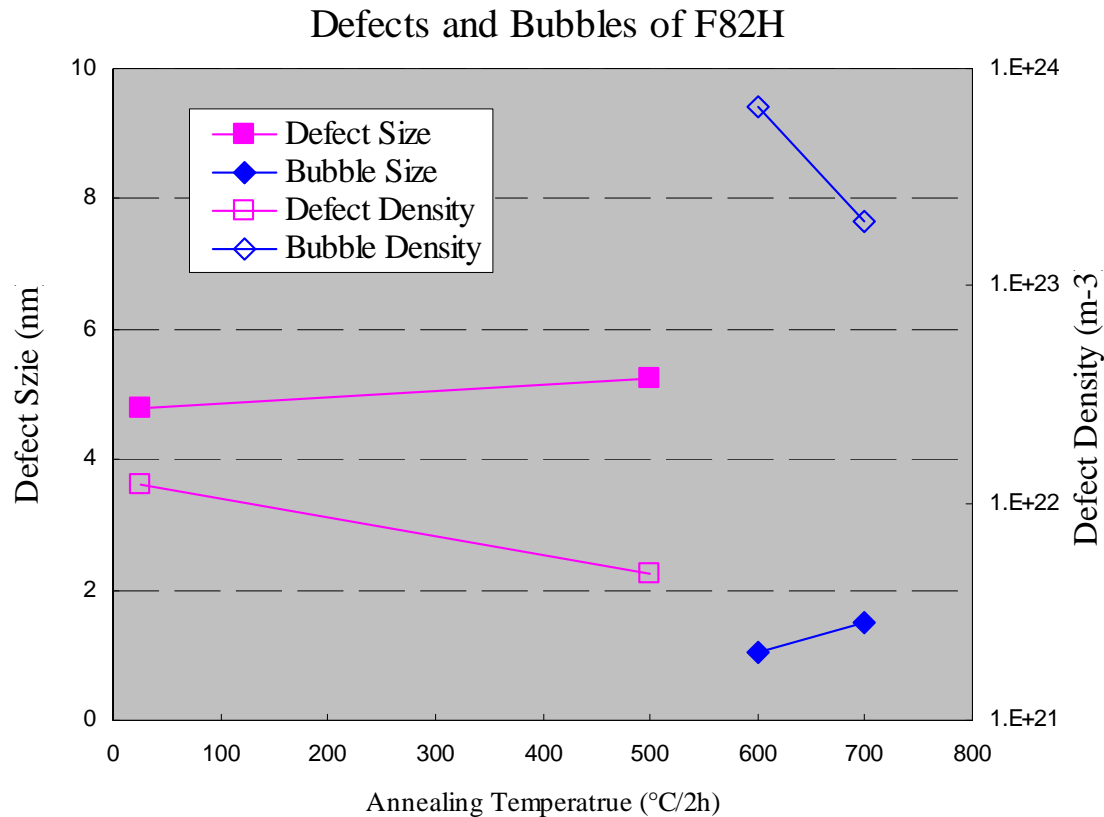
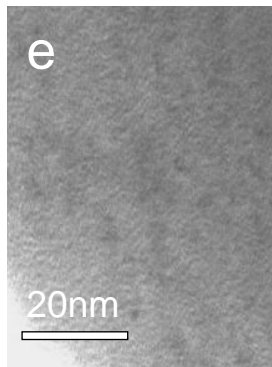
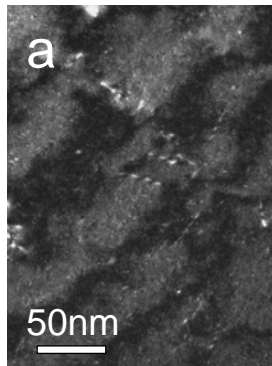
Micro-hardness



Micro-hardness of F82H after different temperature annealing

Different temperature annealing

Microstructure (5.3 dpa/340 appm He/175°C)



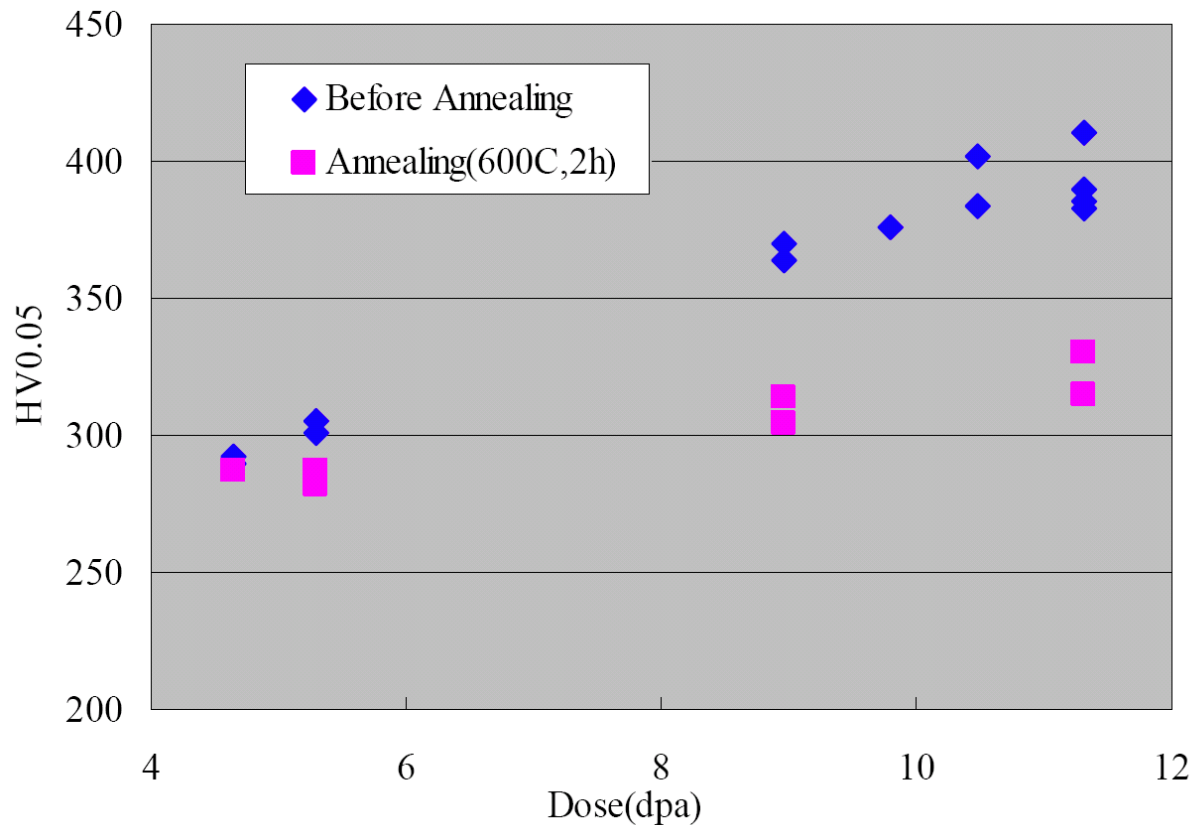
Before Anneal

700°C/2h

Defects are mostly recovered after annealing at 600 °C for 2 hours.

Micro-hardness and Microstructure (F82H)

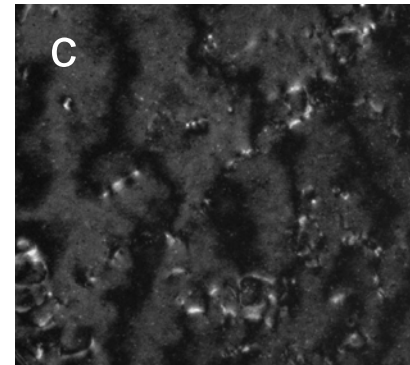
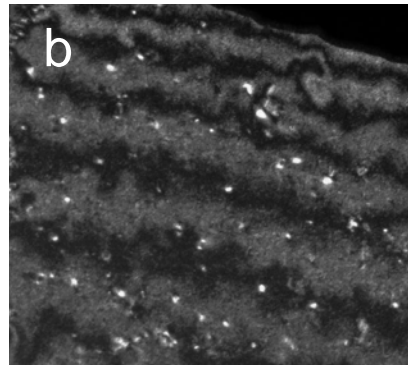
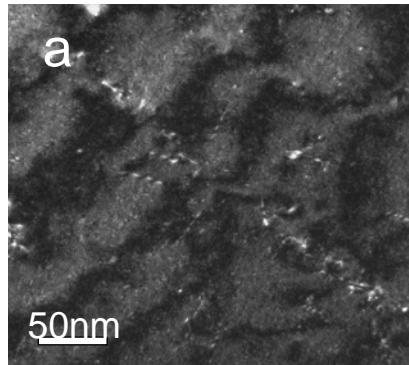
Micro-hardness of F82H after STIP irradiation



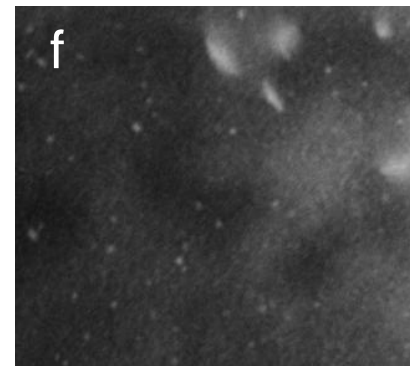
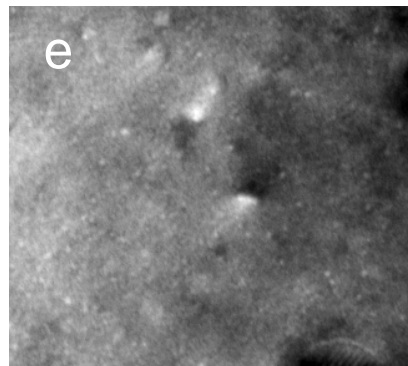
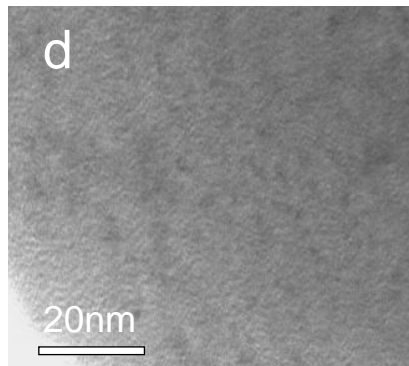
Micro-hardness of F82H before and after 600°C/2h annealing

Micro-hardness and Microstructure (F82H)

Microstructure of F82H before annealing



Defects



Bubbles

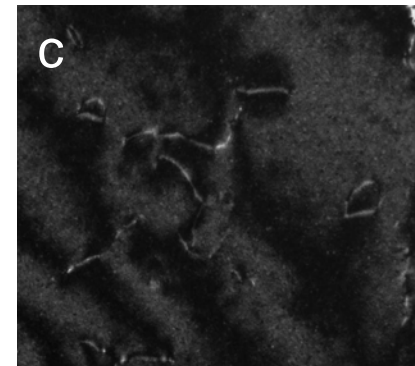
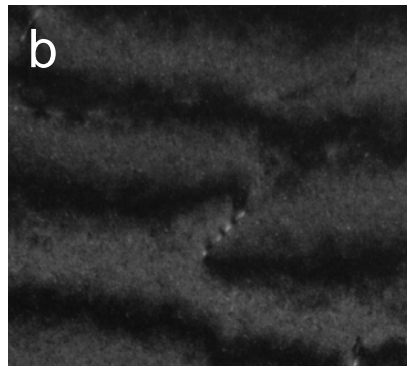
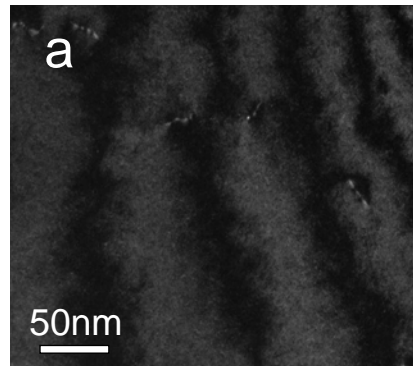
5.3dpa/340appm He

8.96dpa/720appm He

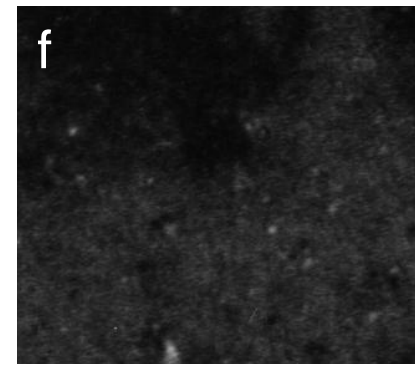
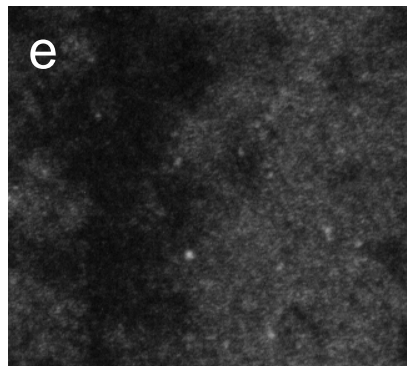
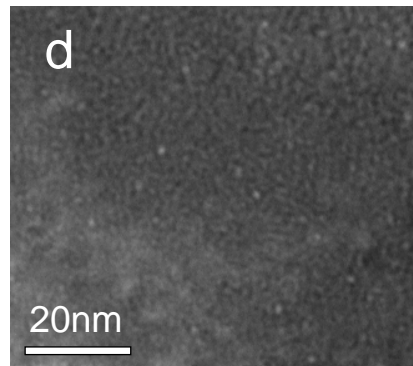
11.32dpa/1173appm He

Micro-hardness and Microstructure (F82H)

Microstructure of F82H after 600°C/2h annealing



Defects



Bubbles

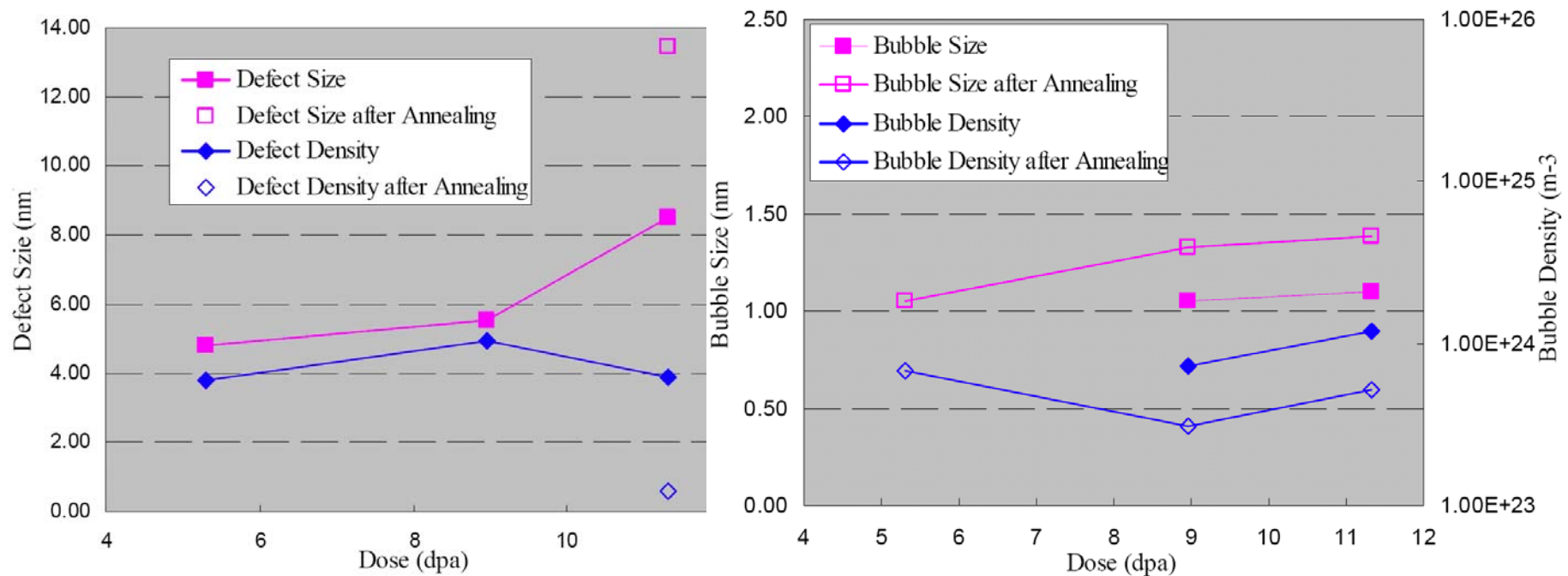
5.3dpa/340appm He

8.96dpa/720appm He

11.32dpa/1173appm He

Micro-hardness and Microstructure (F82H)

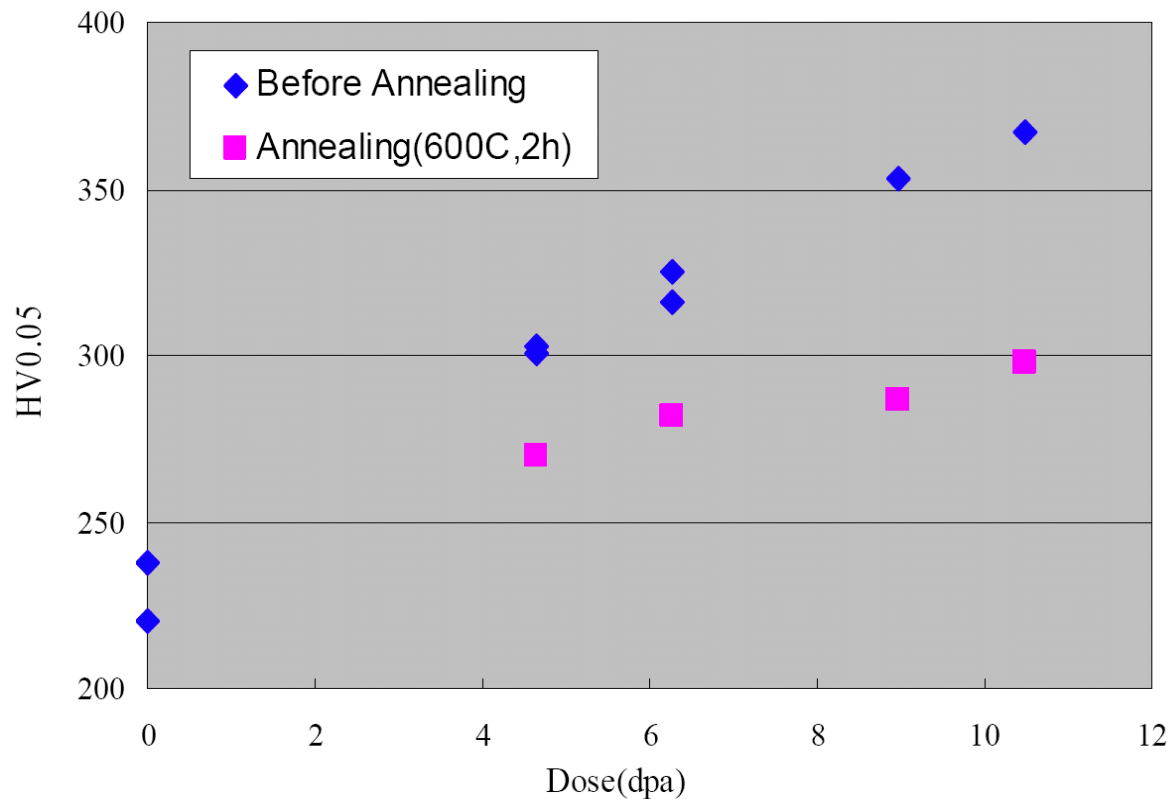
Quantitative analysis of defects and bubbles in F82H after STIP irradiation



Size and density of defects (left) and bubbles (right) of F82H

Micro-hardness and Microstructure (Optimax-A)

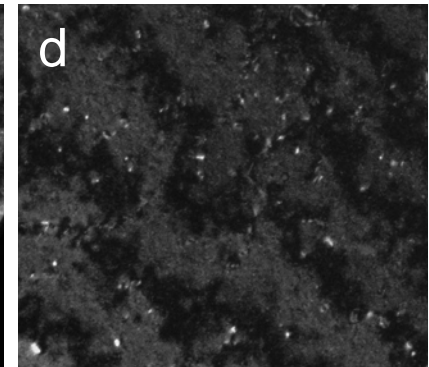
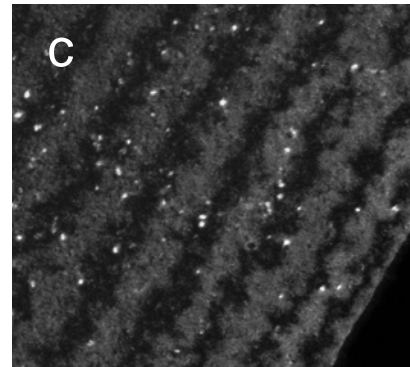
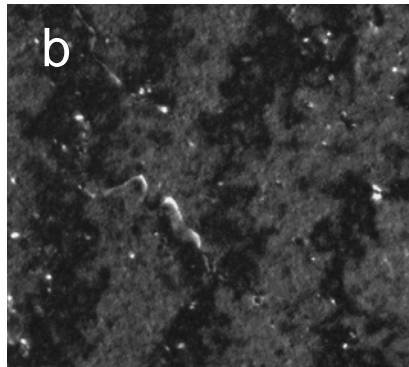
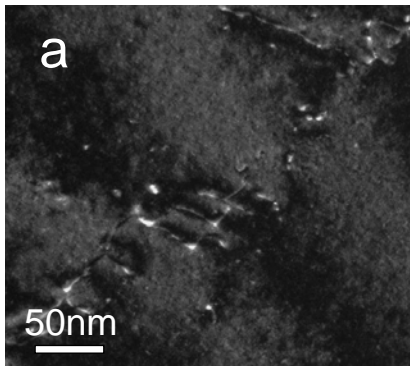
Micro-hardness of Optimax-A after STIP irradiation



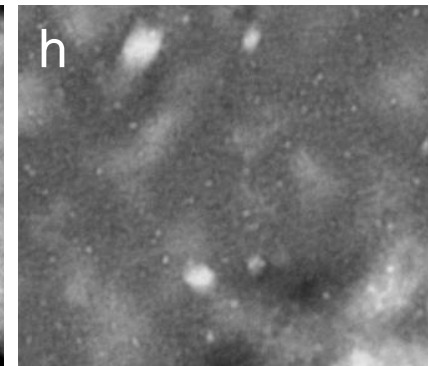
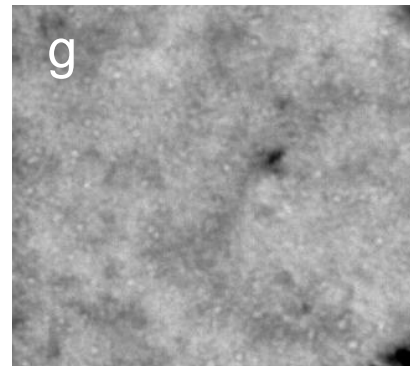
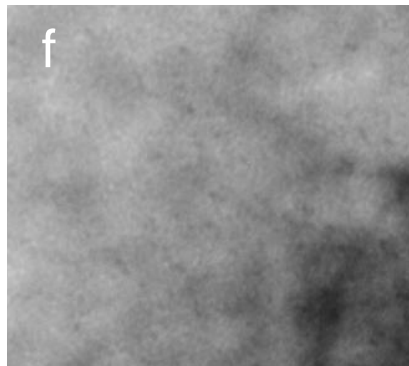
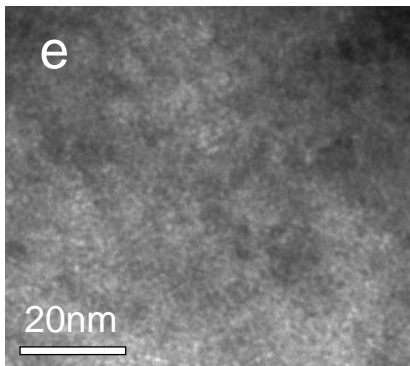
Micro-hardness of Optimax-A before and after 600°C/2h annealing

Micro-hardness and Microstructure (Optimax-A)

Microstructure of Optimax-A before annealing



Defects

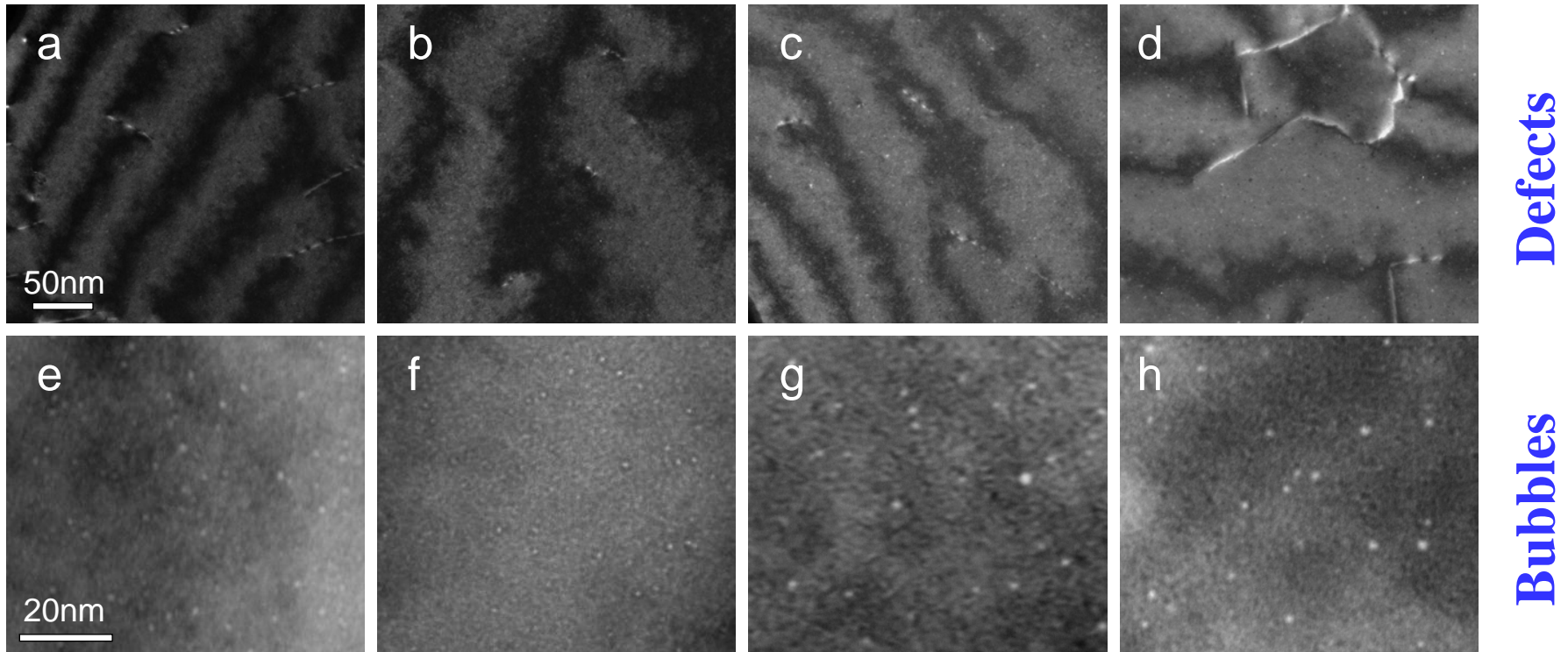


Bubbles

4.6dpa/280appm He 6.3dpa/430appm He 8.96dpa/720appm He 10.5dpa/910appm He

Micro-hardness and Microstructure (Optimax-A)

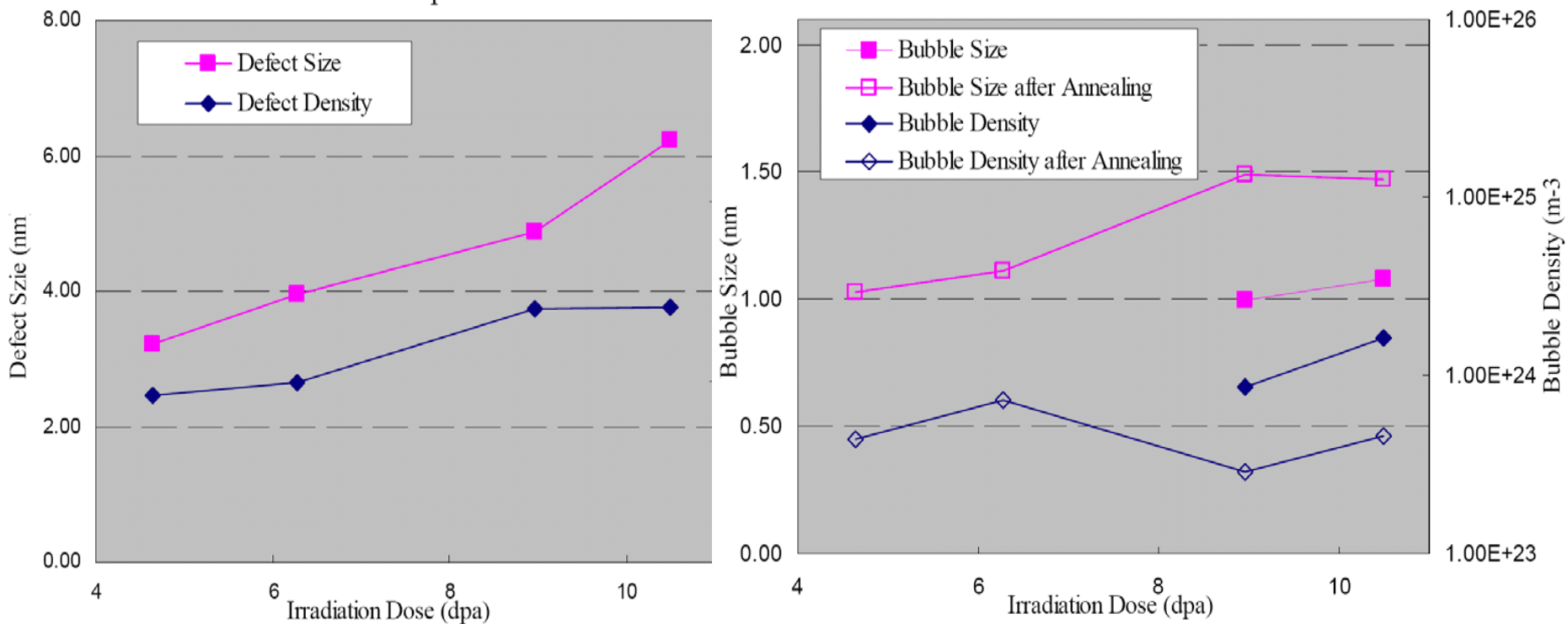
Microstructure of Optimax-A after 600°C/2h annealing



4.6dpa/280appm He 6.3dpa/430appm He 8.96dpa/720appm He 10.5dpa/910appm He

Micro-hardness and Microstructure (Optimax-A)

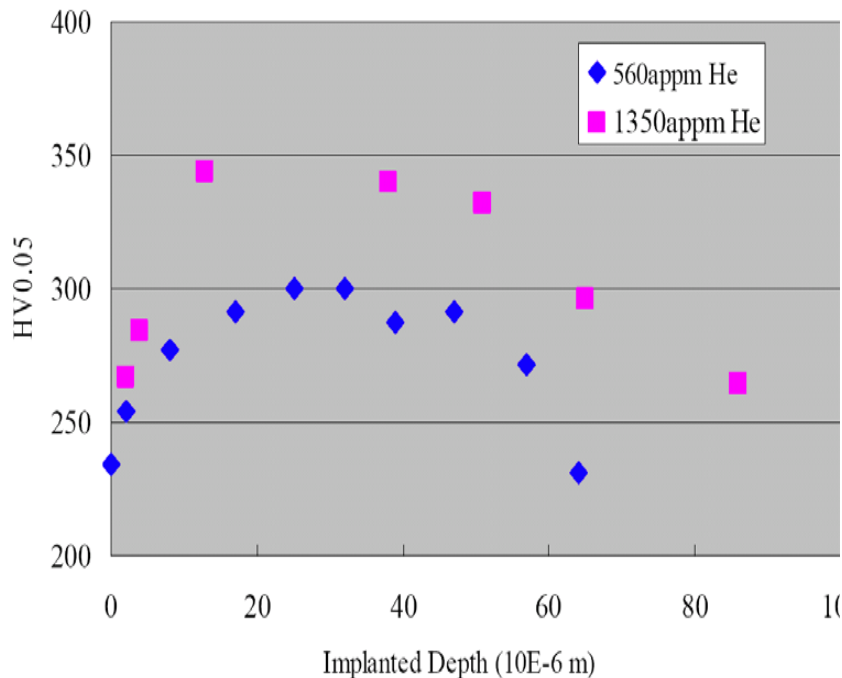
Quantitative analysis of defects and bubbles in Optimax-A after STIP irradiation



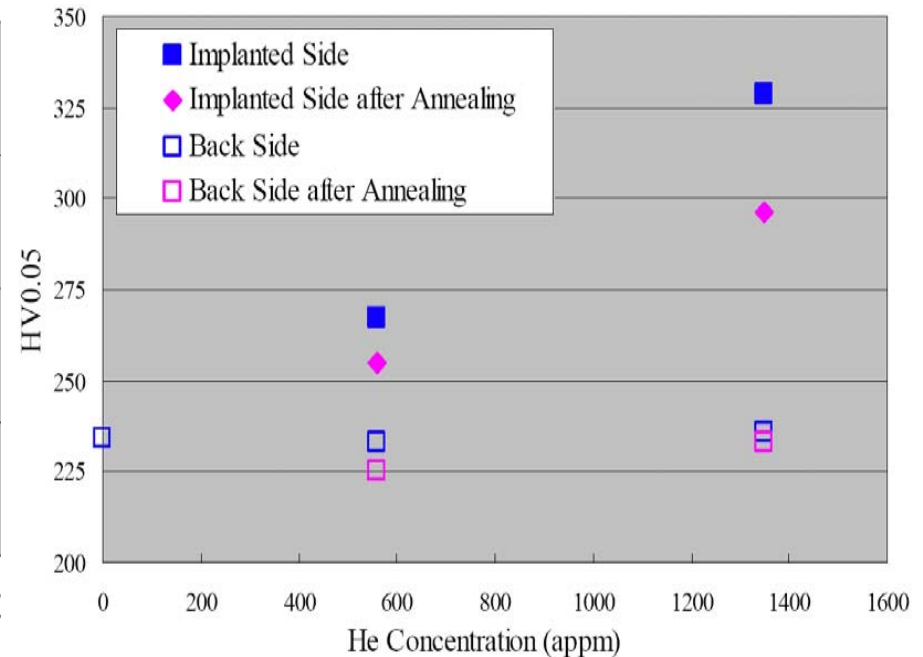
Size and density of defects (left) and bubbles (right) of Optimax-A

Micro-hardness and Microstructure (F82H)

Micro-hardness of F82H after Helium implanted



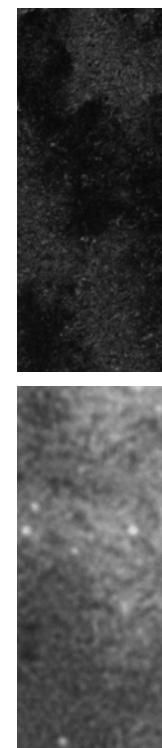
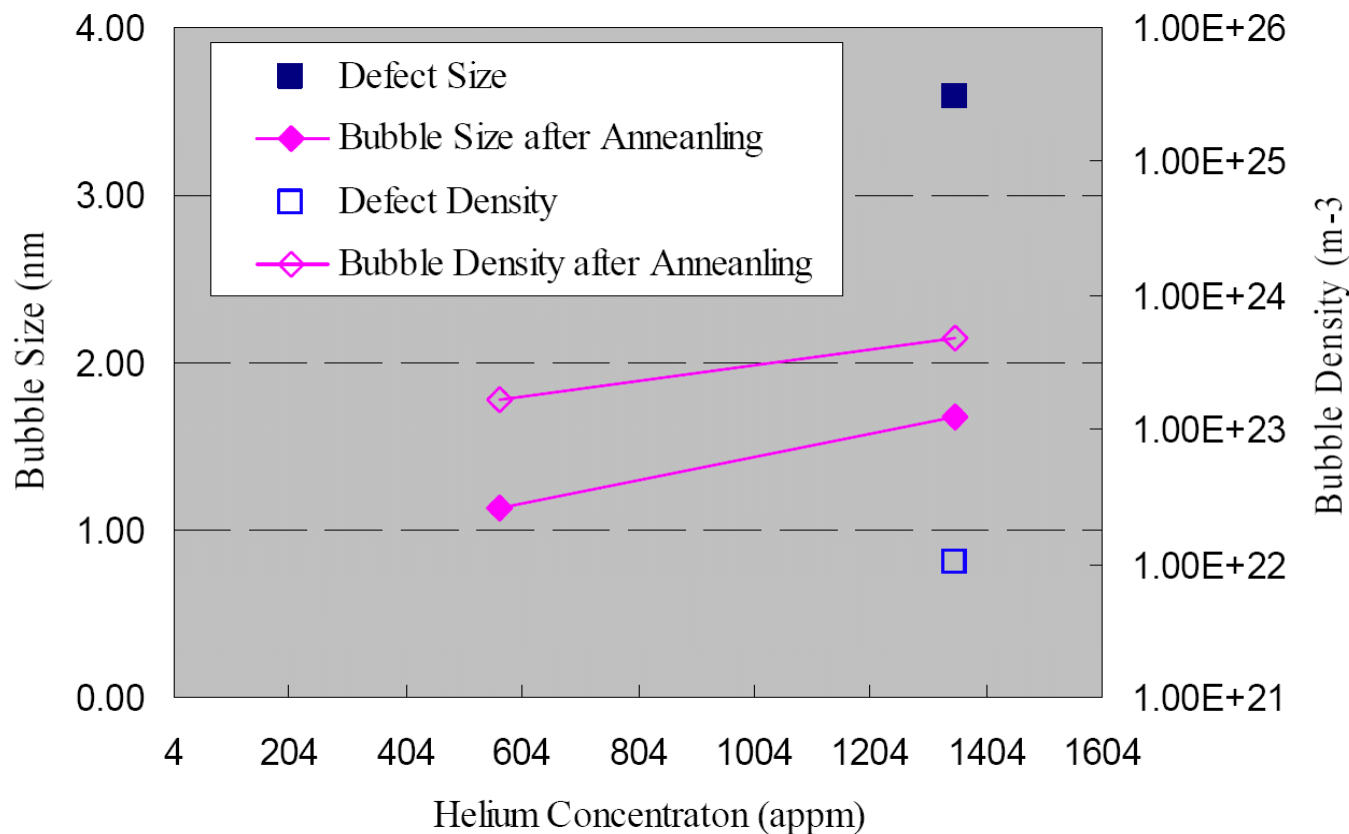
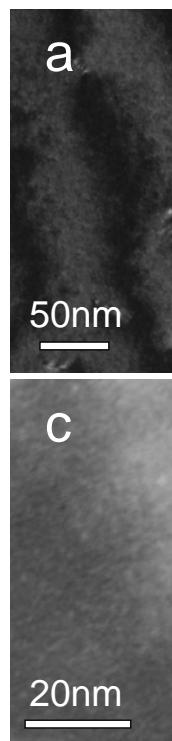
Micro-hardness of F82H after Helium implanted with implanted depth



Micro-hardness of F82H after Helium implanted with He concentration

Micro-hardness and Microstructure (F82H)

Microstructure of F82H after Helium implanted



Defects

Bubbles

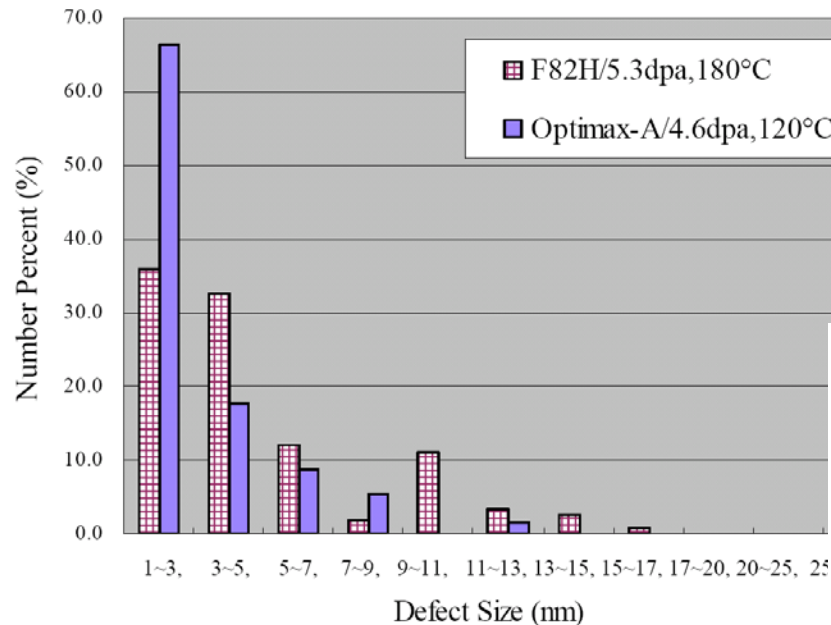
560appm

600°C/2h annealing 600°C/2h annealing
le at R.T.

Discussion

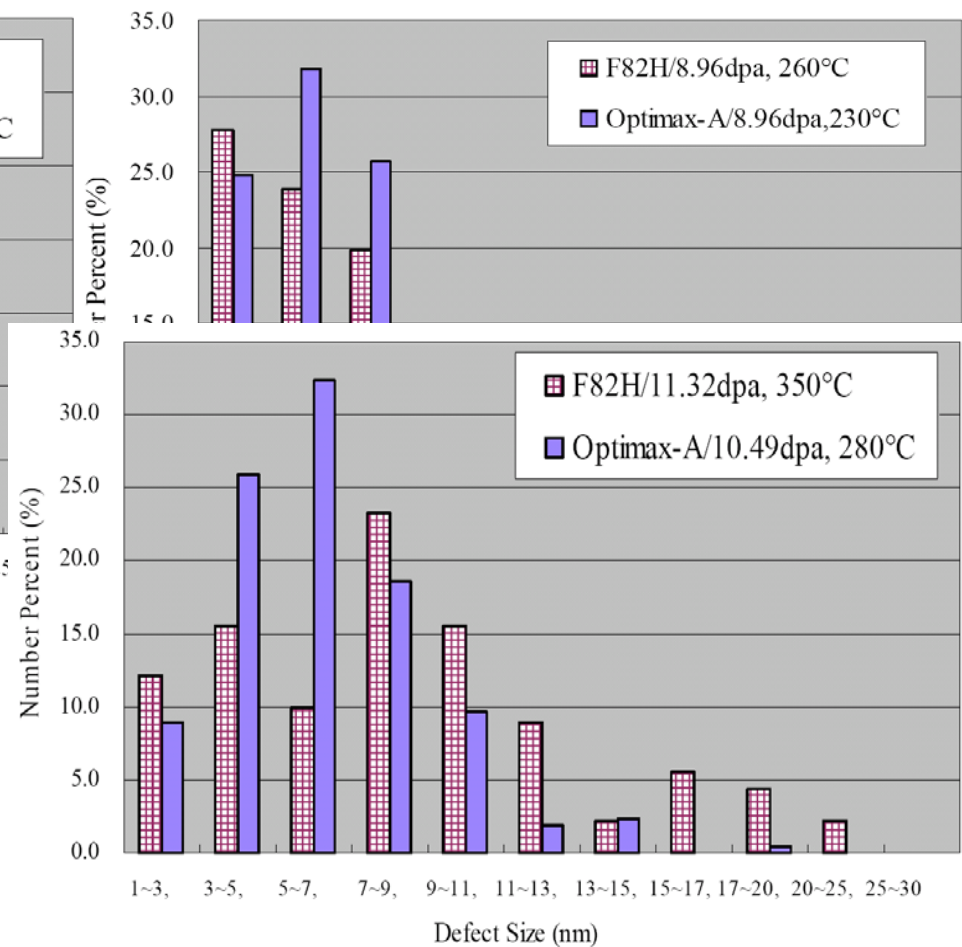
Discussion

Defects with irradiation temperature



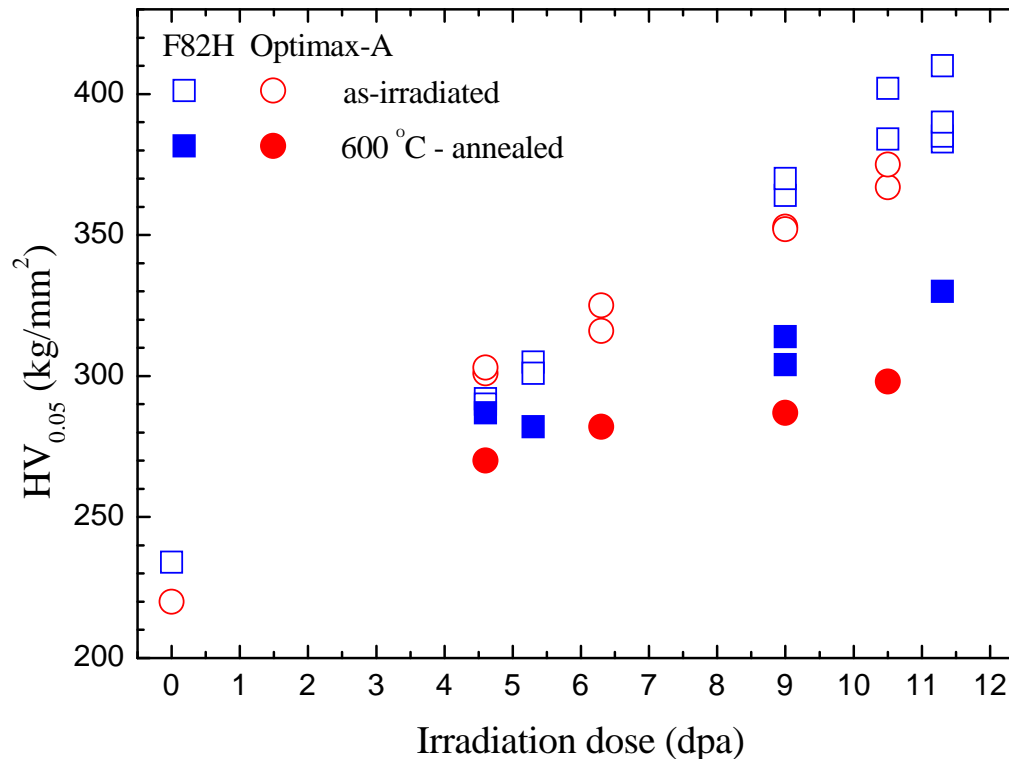
Defects after STIP irradiation

Mean size of F82H is larger than that of Optimax-A. Probable reason is the irradiation temperature of F82H was higher than that of Optimax-A.



Discussion

Defects with irradiation dose



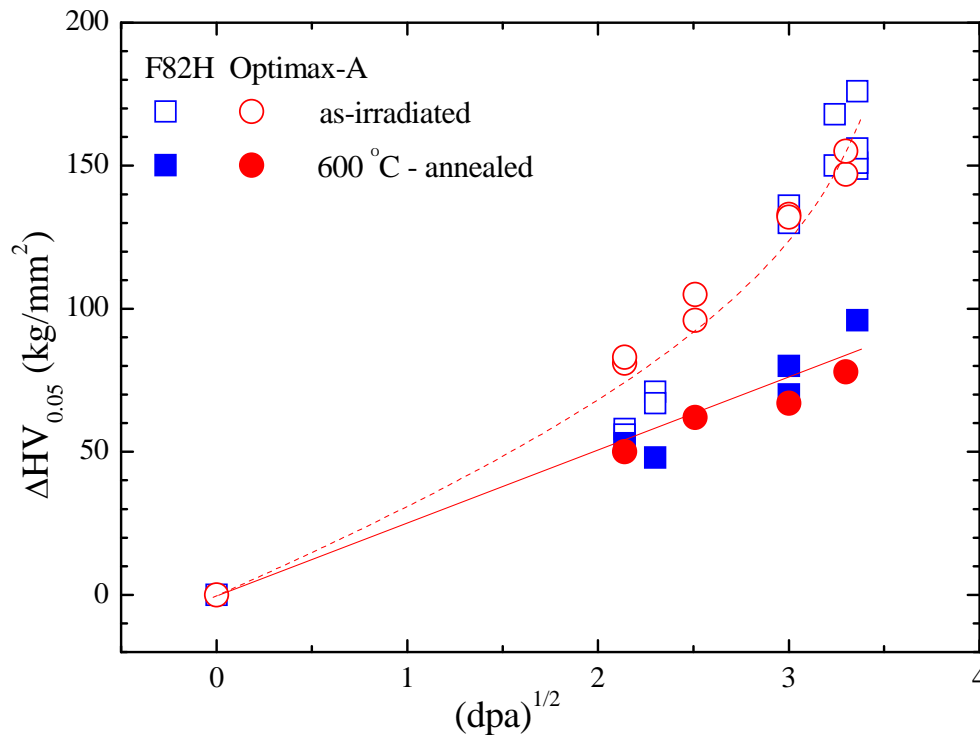
Microhardness increased with irradiation dose before and after annealing.

Good linearly relationship before annealing.

Micro-hardness of F82H and Optimax-A after STIP irradiation

Discussion

Helium effects on hardening



Micro-hardness of F82H and Optimax-A after STIP irradiation

$$\Delta\sigma = 2.7 \Delta H_v$$

$$\Delta\sigma = 3.06 \Delta \tau_s$$

$$\Delta \tau_s = \alpha G b (N d)^{1/2}$$

$$\alpha = \sim 0.1$$

G: Shear modulus, 80 Gpa
b: Burgers vectors, 2.49 Å

Barrier strength (α) of helium bubbles with sizes 1~1.5 nm is about 0.1.

Conclusions

Conclusions

The present results of FM steels irradiated in STIP show that:

- 1) There is a good linear correlation between micro-hardness and irradiation dose (4.6~11.3 dpa) before annealing.
- 2) Hardening in the as-irradiated specimens can be attributed to the combined contribution of defect clusters and bubbles. 600° C/2h annealing can mostly anneal out the irradiation induced defects.
- 3) Although the small helium bubbles are weak obstacles, significant hardening can still be produced because of their very high density, $10^{23} \sim 10^{24} \text{ m}^{-3}$. Preliminary evaluations indicate that the barrier strength of helium bubbles with sizes 1 ~1.5 nm is about 0.1.

Thank you for attention!