Examining microstructures and mechanical properties of neutron and ion irradiated T91 and HT9 alloy

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THE UNIVERSITY OF TENNESSEE KNOXVILLE

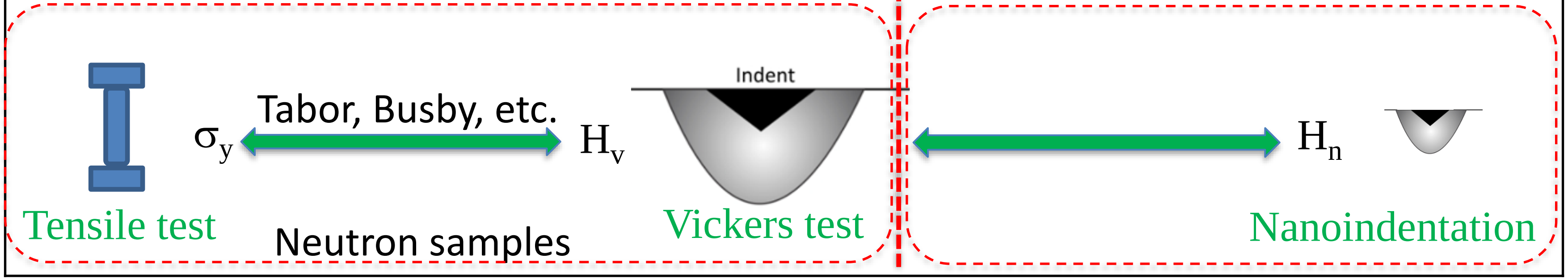
Simulating Neutrons with Accelerated Particles

Nuclear Science User Facilities

1 Introduction: Ion irradiation as a surrogate for reactor irradiation

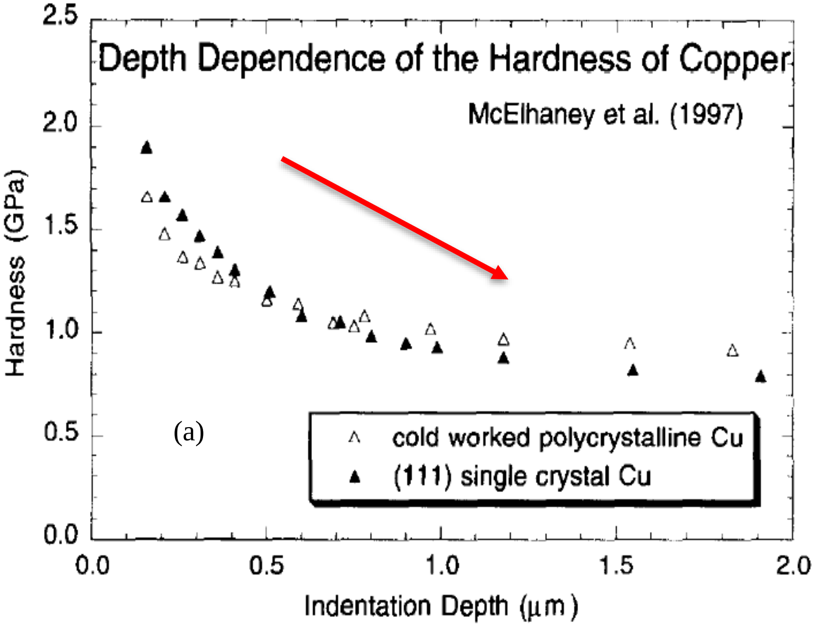
→A successful simulation requires high similarities in both neutron-modified microstructures and neutron-induced **bulk properties** with those caused by ion irradiation.

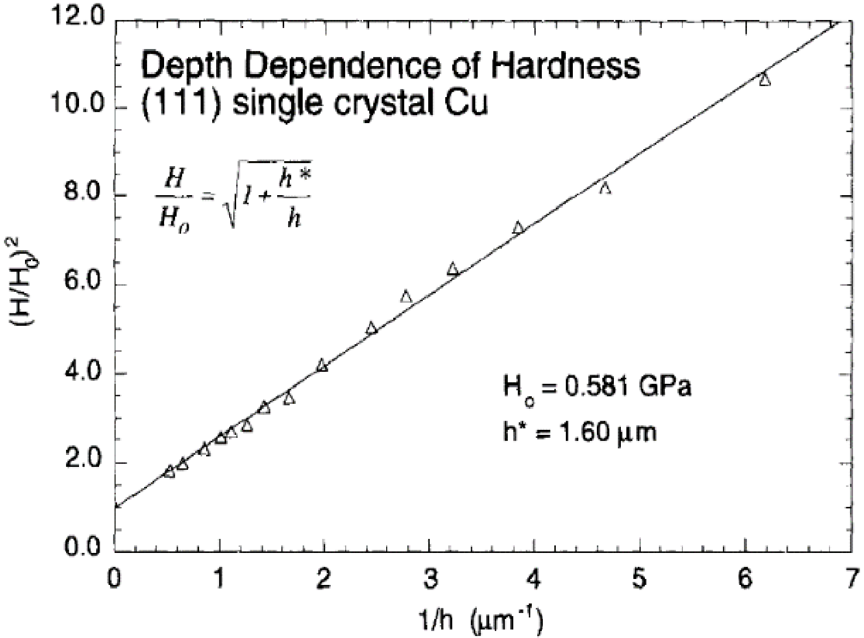
Neutron irradiation		**Ion irradiation**	
Pros	Cons	Pros	Cons
→Uniform damage layer →bulk tensile testing is feasible →closer to real reactor environment	→inevitable radioactivity →high cost →long time needed to achieve target dose	→little or no radioactivity →fast and easy access to experimental facility →economic	→limited volume for mechanical testing →much higher damage rate than in the reactors



1 Introduction: Nanoindentation background

- indentation size effects[1]





→Nix-Gao fitting was used to extract bulk equivalent hardness (H_0).

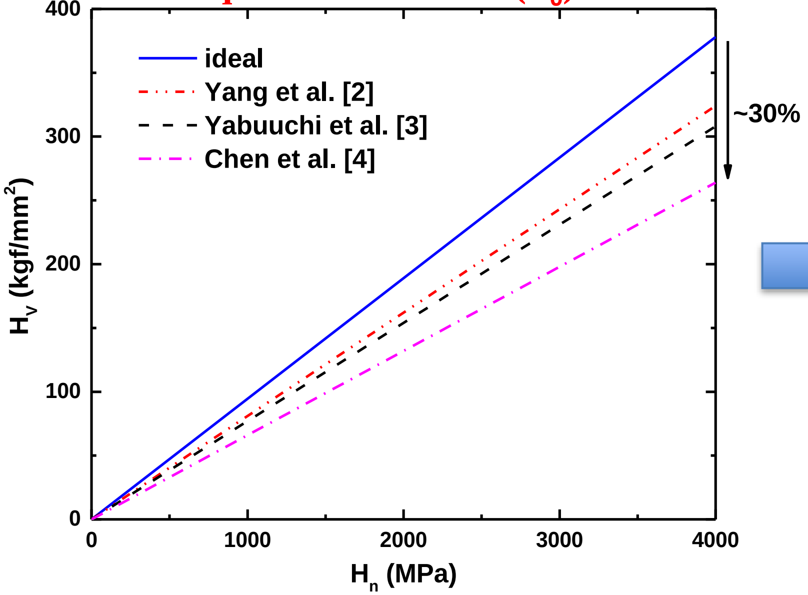
$$\frac{H}{H_0} = \sqrt{1+\frac{h^*}{h}}$$

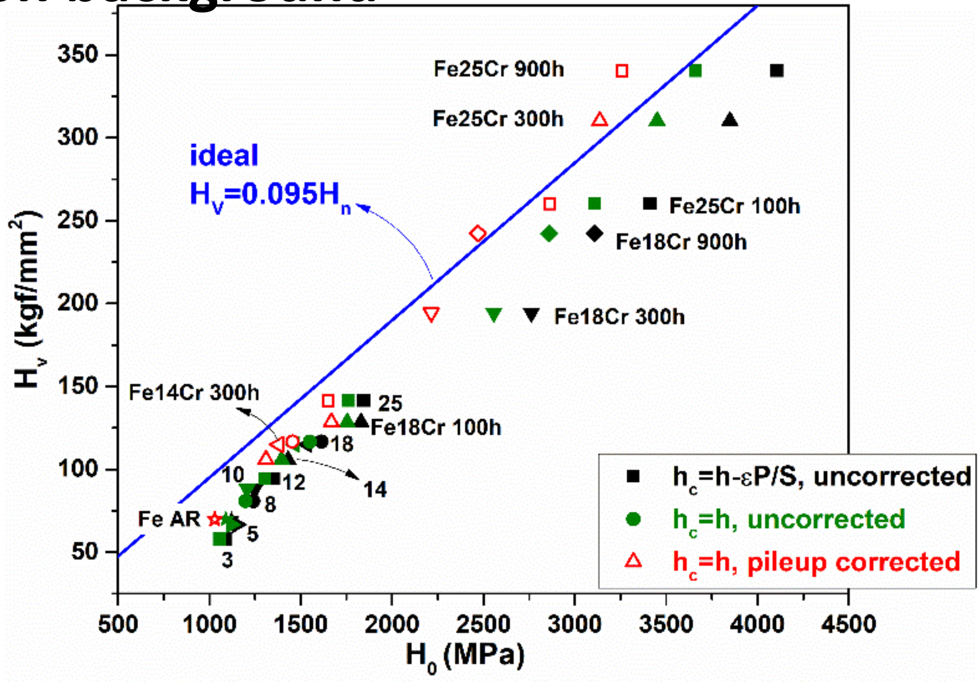
$$H^2 = {H_0}^2 + \frac{h^*}{h}{H_0}^2$$

[1]K. W. McElhaney et al. JMR. 1998
[2]Yang et al. JNM.2020
[3]Yabuuchi et al. JNM.2014
[4]Chen et al. Acta. Mater. 2020

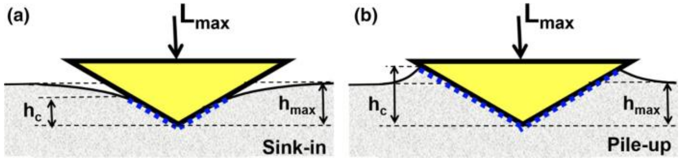
1 Introduction: Nanoindentation background

- **Vickers hardness (Hv) vs. Nanoindentation bulk equivalent hardness(H_0)**





- **Pileup/sink-in effects**



→**Nix-Gao model [5]** combined with **pileup corrections** provide a quantitatively accurate way to predict the bulk Vickers hardness in nano scale test volumes [6].

→**$Hv=0.0945H_0$** is used to covert the nanoindentation bulk equivalent hardness into Vickers hardness

[2]Yang et al. JNM.2020
[3]Yabuuchi et al. JNM.2014
[4]Chen et al. Acta. Mater. 2020

[5] William D.Nix et al. J. Mech. Phys. Solid, Vol. 46, No. 3, pp. 41 I-425. 1998
[6] P. Zhu et al. Toward accurate evaluation of bulk hardness from nanoindentation testing at low indent depth. Material & Design 213 (2022), 110317

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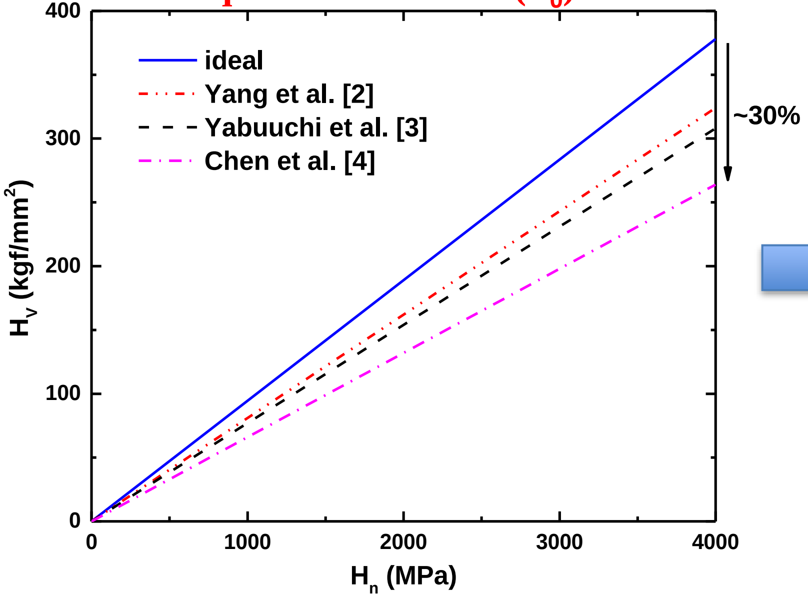

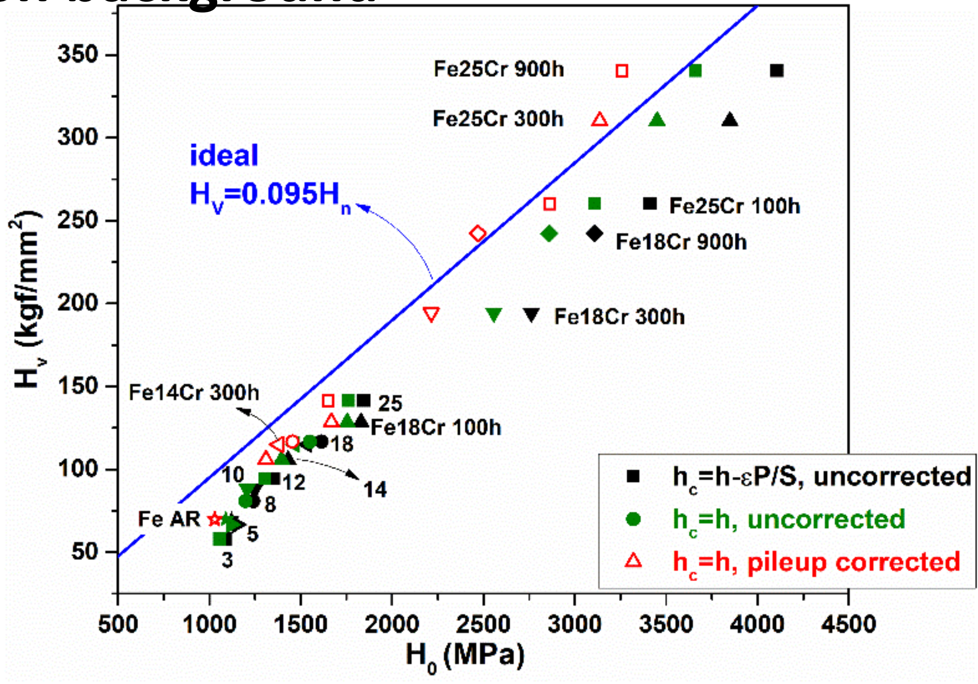

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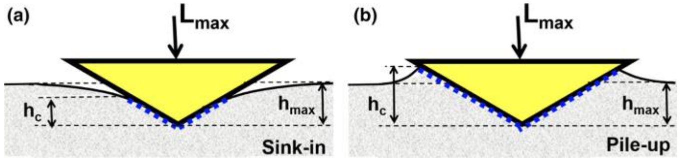

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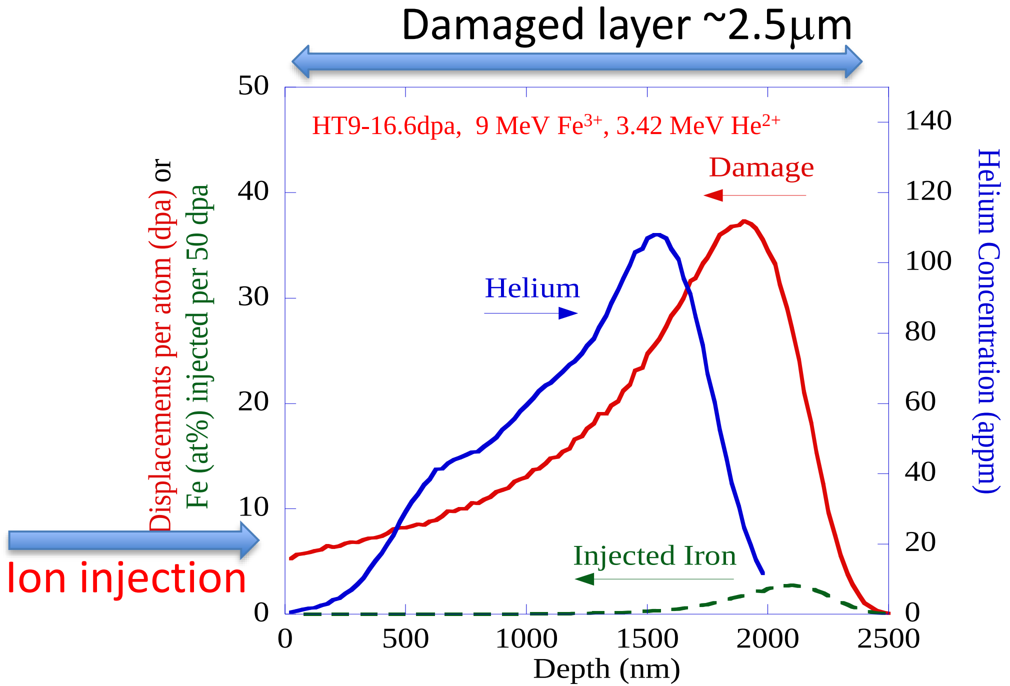
2 Sample information

9 MeV Fe^{3+}, 3.42 MeV He^{2+}

Dual ion	Irradiation T(°C)	dpa	He appm	He/dpa
T91	445	16.6	72	4.3
	520	16.6	72	4.3
HT9	445	72	309	4.3
	520	16.6	72	4.3

BOR-60

Neutron	Irradiation T(°C)	dpa
T91	369	16
	453	19
HT9	450	32
	453	19


Damaged layer ~2.5μm

Ion injection

- → The total irradiation depth for 9MeV irradiation is about 2.5 um.
- → The shallow damage layers inhibit the bulk mechanical testing methods such as tensile and Vickers hardness tests.

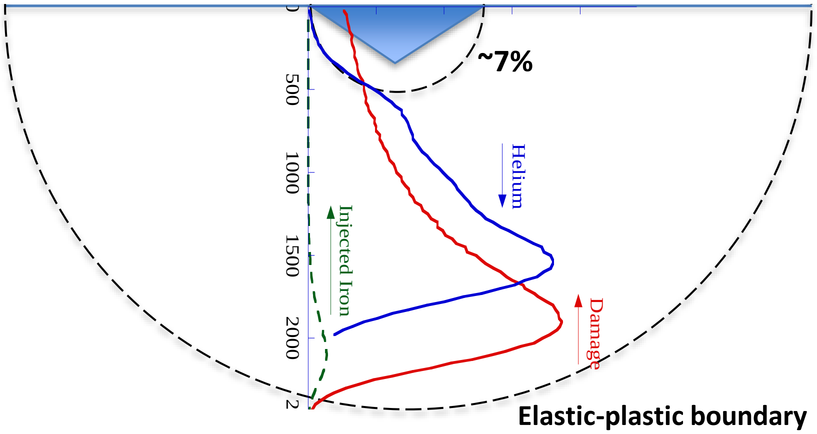
3 Results: Nanoindentation testing on dual-ion irradiated samples

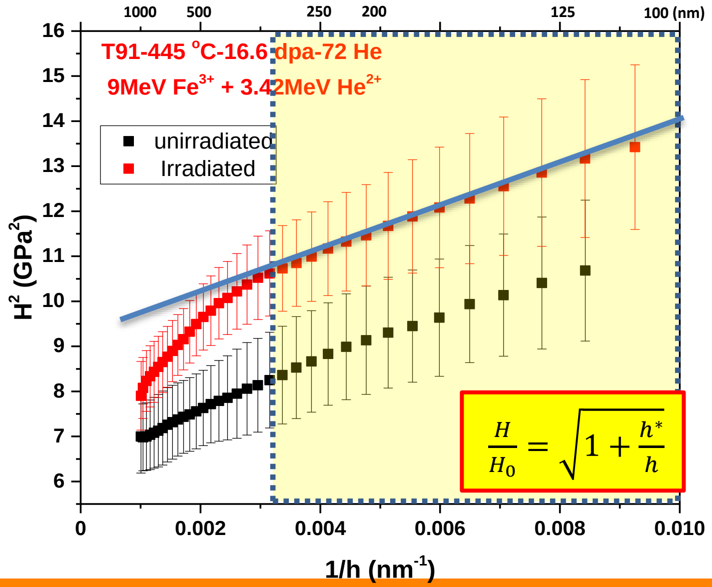
→the plastic zone generally expands 5-10 times the indentation depth.

→only data from first several hundreds of depth is available to avoid substrate effect.

→H^2 vs. 1/h curve for typical ion irradiated sample shows inflection around 300 nm, which infers the effect of un-irradiated substrate layer.

Strain extension under indenter





$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}}$$

L.E. Samuels & T.O. Mulhearn, J. Mech. Phys. Sol. 5 (1957) 125

3

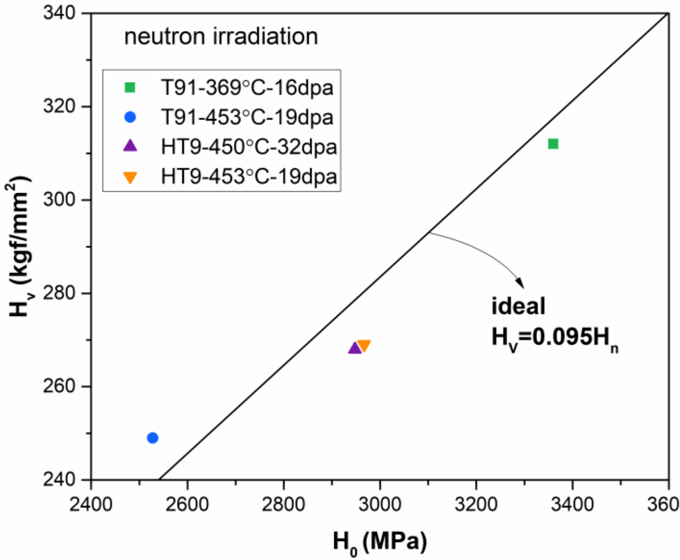
Results: Nanoindentation and Vickers on T91 and HT9

→The test protocol developed on thermally aged FeCr alloys was used to extract nanoindentation bulk equivalent hardness. Since the pileup measurement on neutron samples is not available, the same amount of pileup effect was estimated with the ion irradiated samples.

Dual ion irradiation:

Sample	temperature	dose	H_0 (pileup corrected)	pileup correction	Hv=94.5H_0	Measured Hv
	℃	dpa	(GPa)		(kgf/mm^2)	(kgf/mm^2)
T91	445	16.6	3.02	8%	286	-
		control	2.55	12%	241	247
	520	16.6	2.34	10%	221	
		control	2.34	9%	221	235
HT9	445	72	3.61	5%	341	-
		control	2.93	7%	277	-
	520	16.6	2.74	6%	259	-
		control	-	-	-	-

Neutron irradiation:



→Good agreement is achieved between nanoindentation and Vickers testing results after applying the nanoindentation test protocol on neutron irradiated samples.

3 Results: Temp. shift to account for dose rate

→A temperature shift is needed to account for the dose rate difference between ion and neutron irradiation.

→The temperature shift seems to result in both a qualitative and quantitative match of the **dislocation loops**, and seems to work well in the **mechanical properties** of neutron and dual ion irradiated samples.

$$T_2 - T_1 = \frac{kT_1^2/(E_v^m + E_v^f)\ln(G_2/G_1)}{1 - kT_1/(E_v^m + E_v^f)\ln(G_2/G_1)}$$

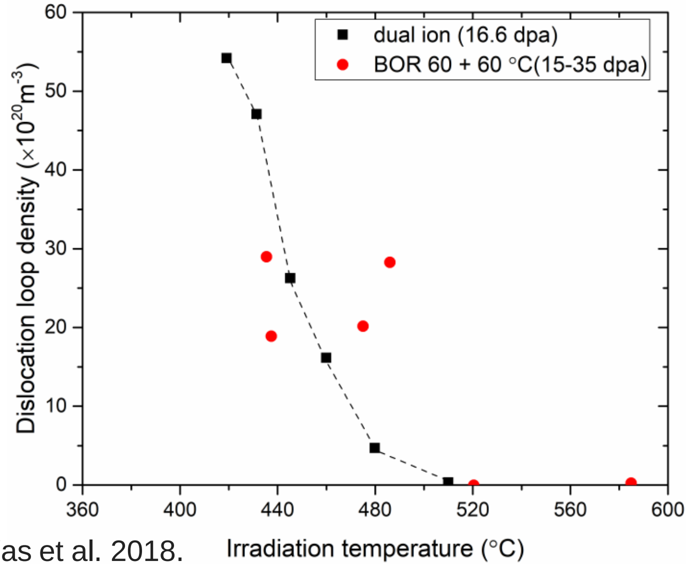
T_1, T_2 : irradiation temperature.

G_1 , G_2 : irradiation dose rate.

E_v^m : vacancy migration energy.

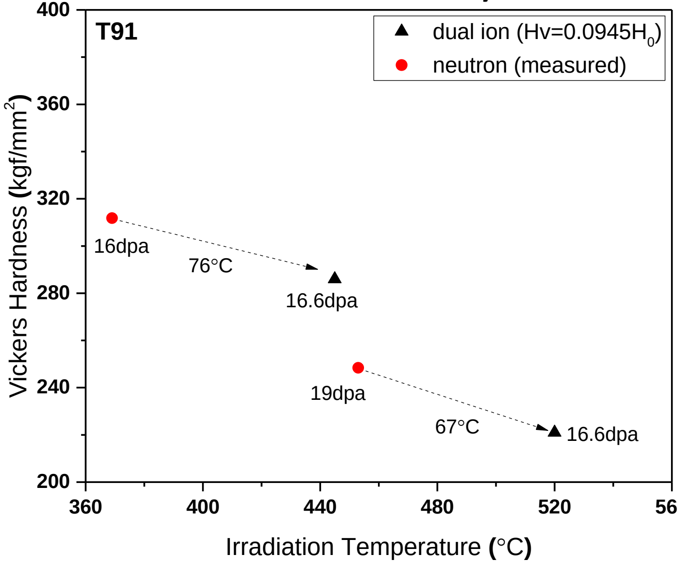
E_v^f :vacancy formation energy.

Literature study



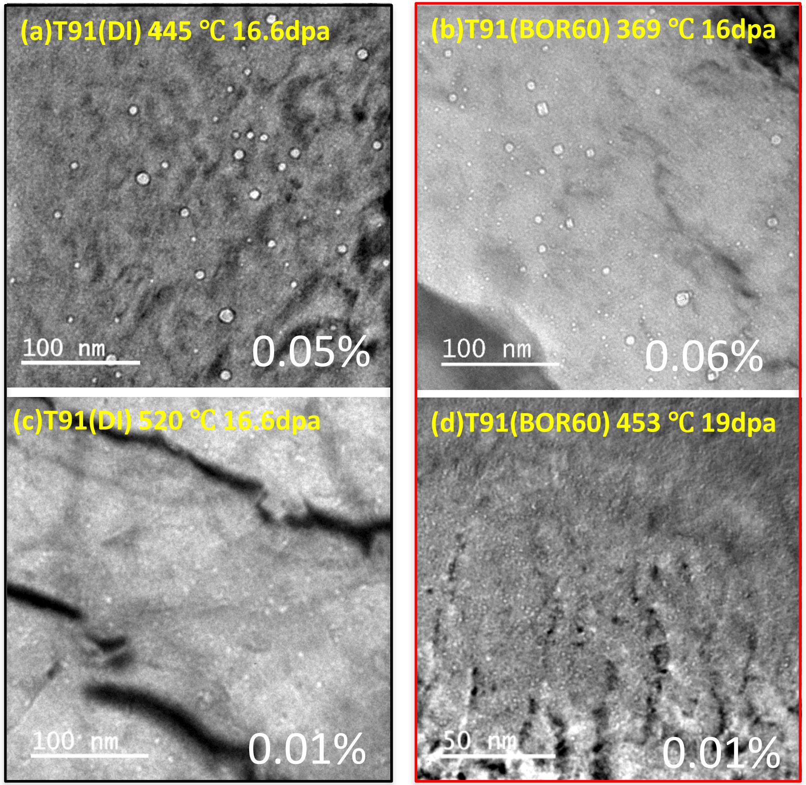
G. Was et al. 2018.

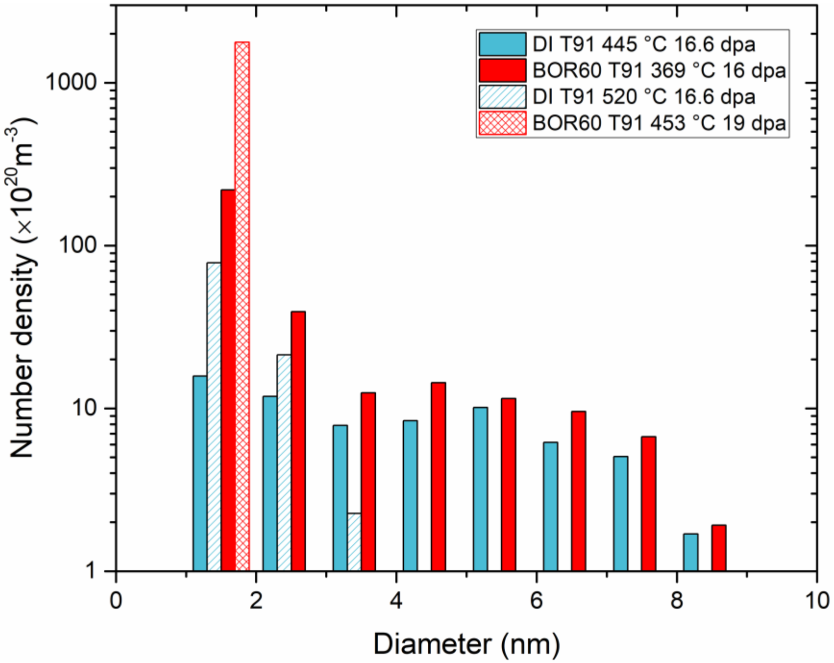
This study



3 Results: Cavities in irradiated T91

Dual ion Neutron



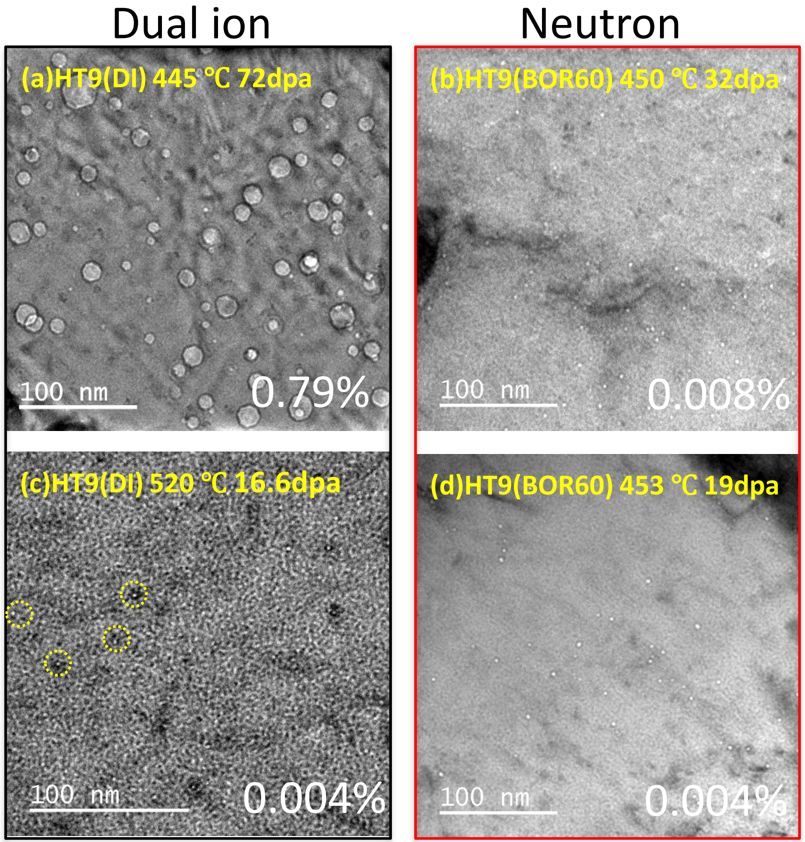


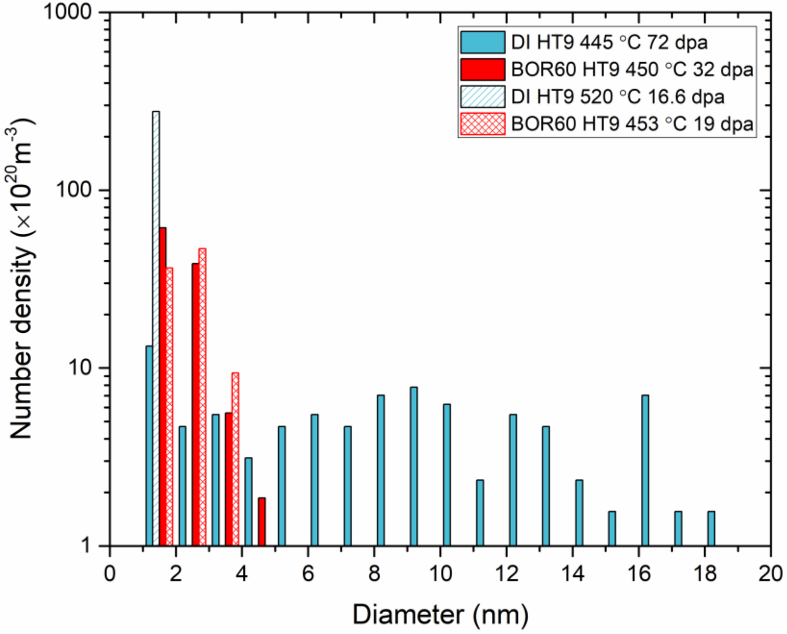
→Lower temperature generated a wider range of cavity size for both dual-ion and neutron irradiation.

→The void swelling matches well for dual ion and neutron samples with a ~70 ℃ temperature shift.

3

Results: Cavities in irradiated HT9



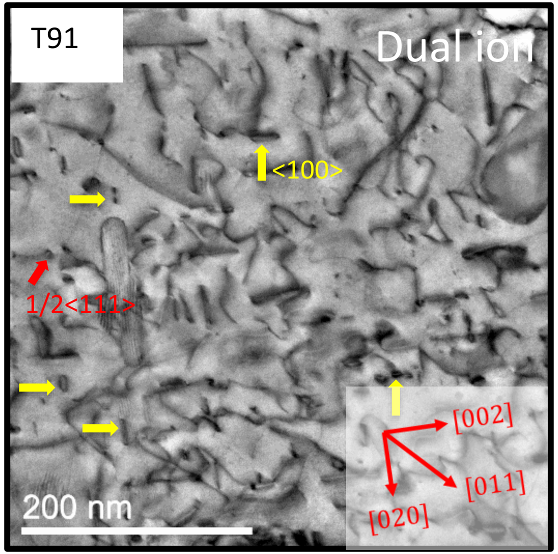


→No clear bimodal distribution was observed. At higher temperatures, the cavities tended to be limited to small sizes.

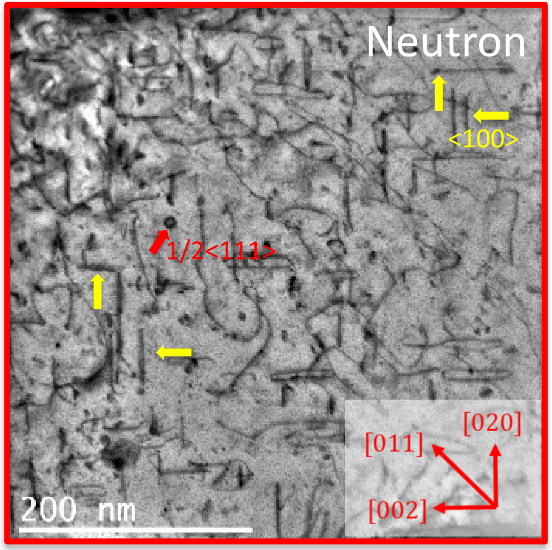
→The void swelling matches well for dual ion at 520 °C and neutron samples at 453°C.

3 Results: Dislocation loops in irradiated T91

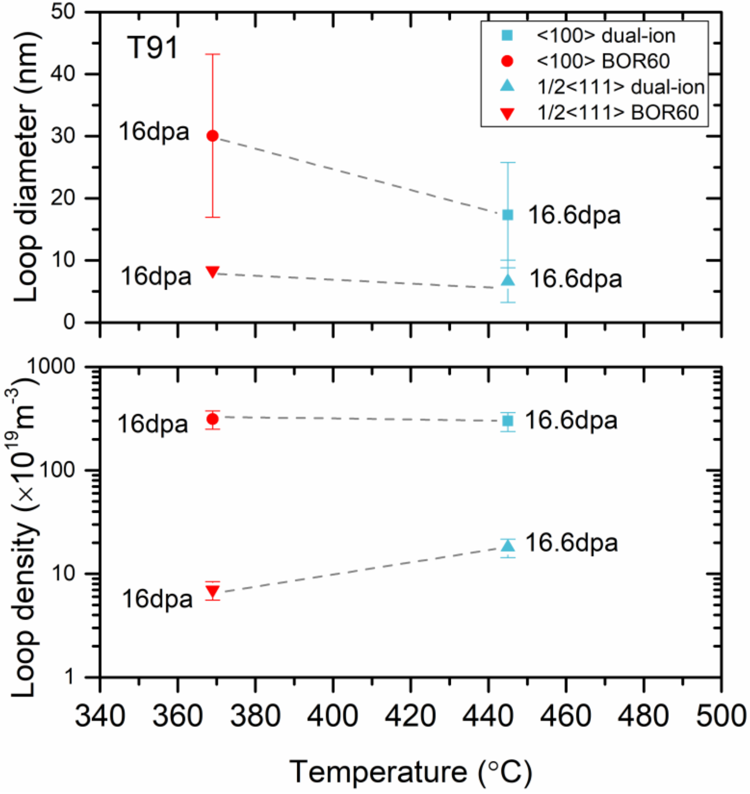
T91(DI) 445 ℃ 16.6dpa



T91(BOR60) 369 ℃ 16dpa



Loops were dominant by <100> type



→Shifting the temperature of BOR 60 irradiated T91 about ~70 ℃ results in a quantitative match of dislocation loops (both <100> and ½<111> loops) with dual ion irradiated T91 sample. The slight discrepancy may come from the non homogenous distribution of the dislocation loops.

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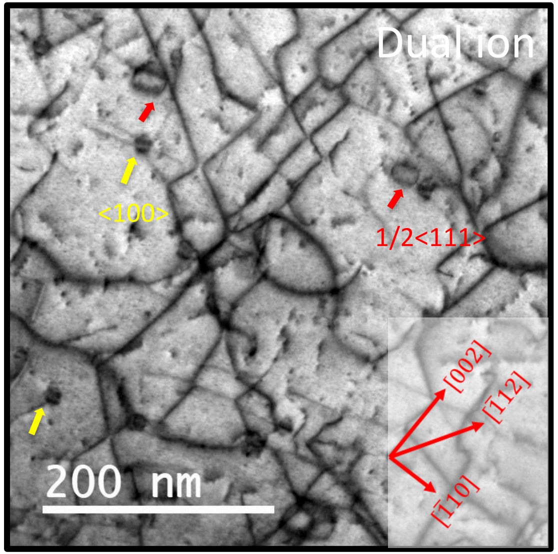
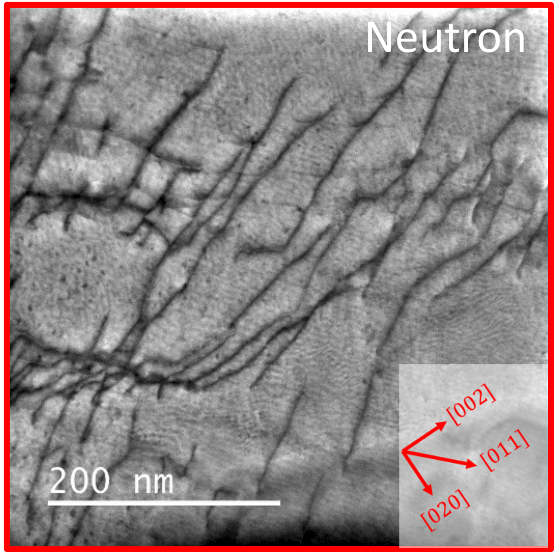
Results: Dislocation loops in irradiated HT9

HT9(DI) 520 °C 16.6dpa



HT9(BOR60) 453 °C 19dpa

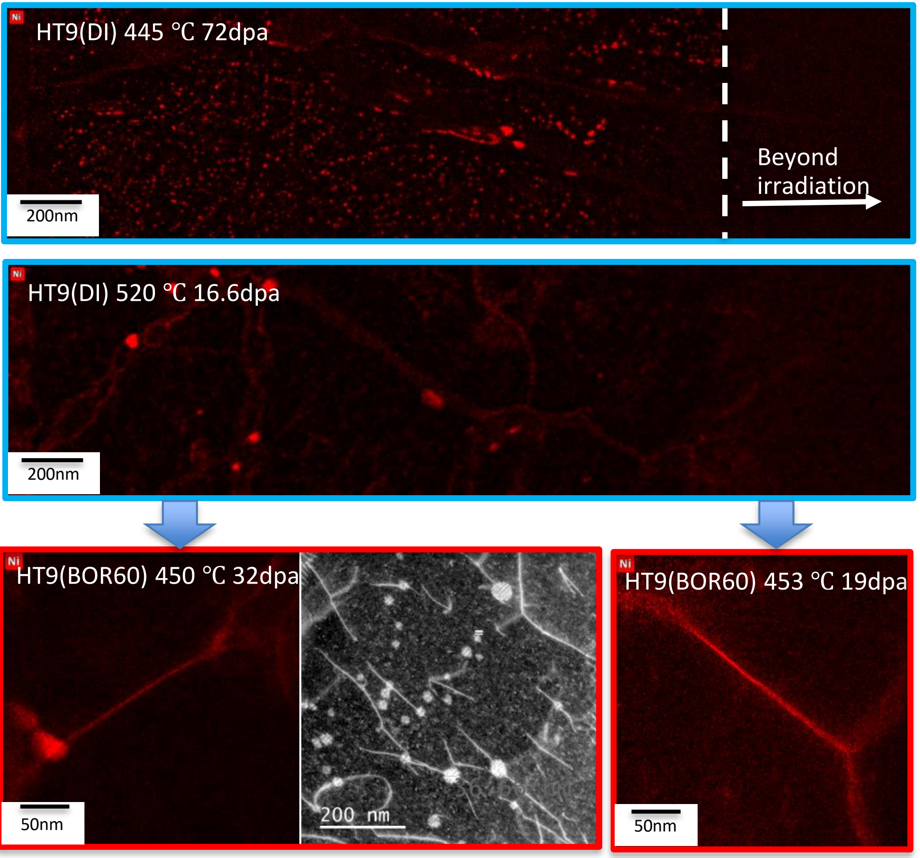


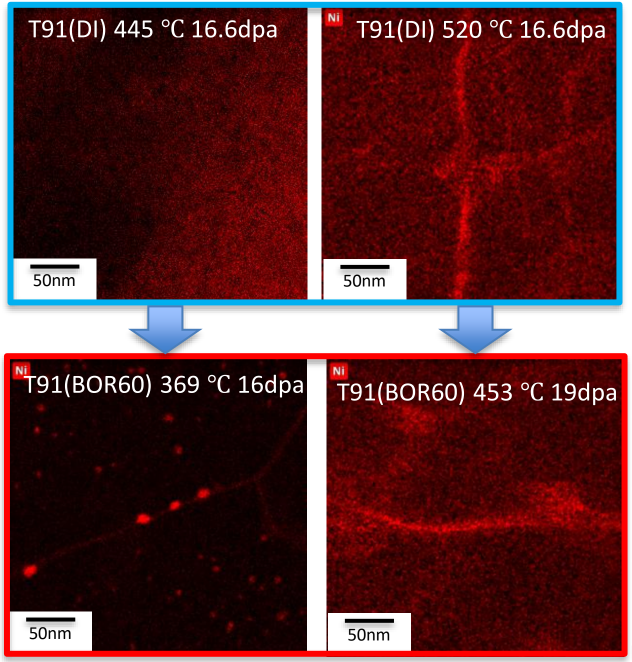
→However, dislocation loops were only observed in dual ion irradiated samples for HT9, in a very low density.

Material	Temperature	Dose	<100> loop		1/2<111> loop		Dislocation line
	°C	dpa	Average diameter (nm)	Number density ($\times 10^{21} m^{-3}$)	Average diameter (nm)	Density ($\times 10^{21} m^{-3}$)	Density ($\times 10^{14} m^{-2}$)
DI-HT9	445	72	34.3 ± 13.1	1.6 ± 0.3	-	-	4.4 ± 0.9
	520	16.6	10.4 ± 2.4	0.5 ± 0.1	31.0 ± 11.3	0.4 ± 0.1	4.2 ± 1.1

3 Results: Precipitates & Radiation Induced Segregation

→EDS mapping shows that Ni-rich clusters were only observed in the ion irradiated region.
→The Ni-rich clusters didn't match well between dual ion and neutron irradiation with a ~70 °C temperature shift.



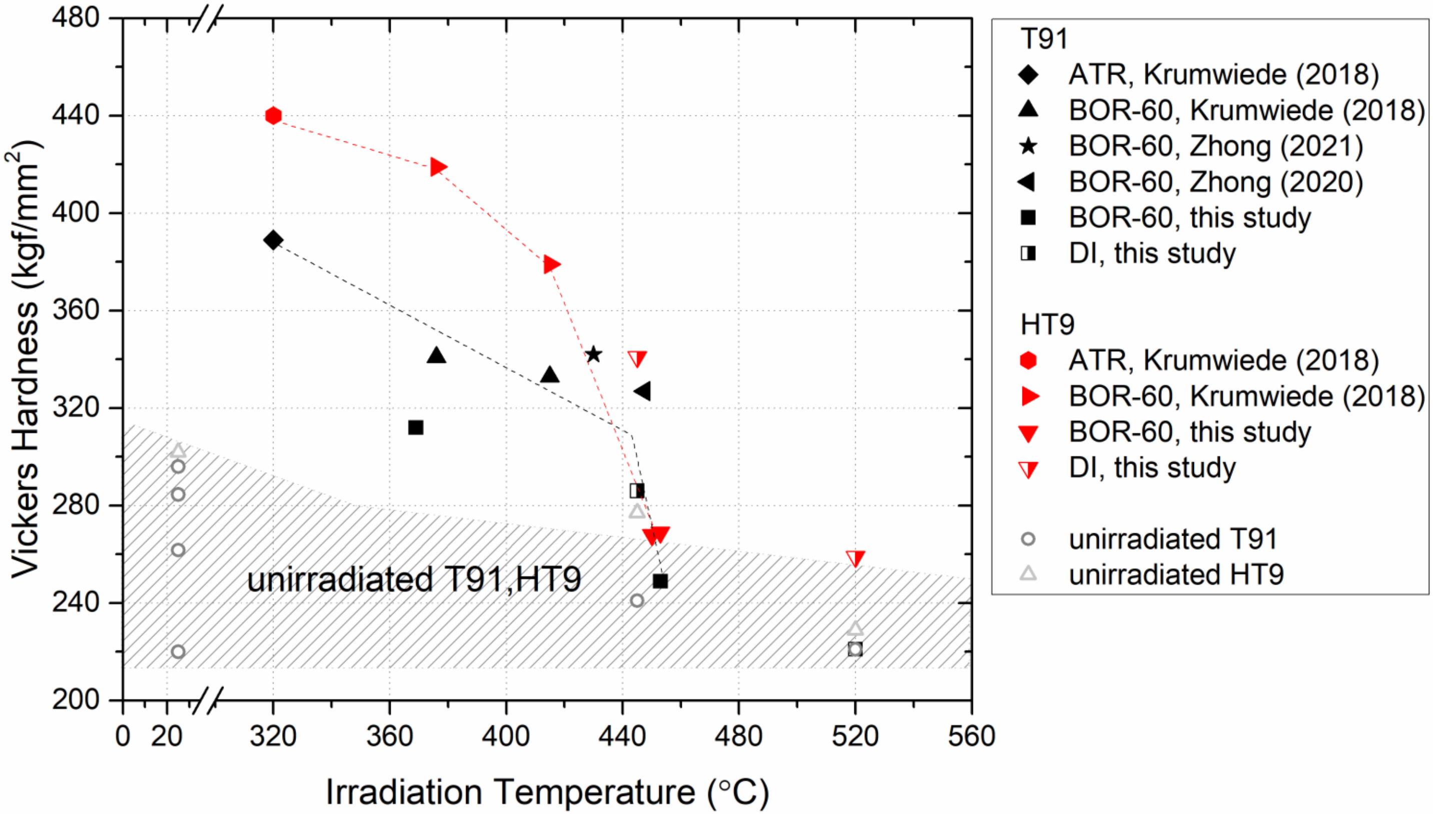


Sample-°C-dpa	Ppts
DI HT9 445 72	√
BOR60 HT9 450 32	√
DI HT9 520 16.6	N.A.*
BOR60 HT9 453 19	√ [1]
DI T91 445 16.6	N.A.
BOR60 T91 369 16	√
DI T91 520 16.6	N.A.
BOR60 T91 453 19	N.A.

*only counted Ni-clusters in the matrix

[1] C. Zheng et al. (2020) 151845

4 Discussion: Mechanical properties



→A rapid decrease of hardness occurred in 420-460℃ region, however the irradiation induced hardening is minor at higher temperatures. This means the comparable results in the previous slide at this relatively high temperature region only come from the insignificant irradiation hardening.

4

Discussion: Strengthening model

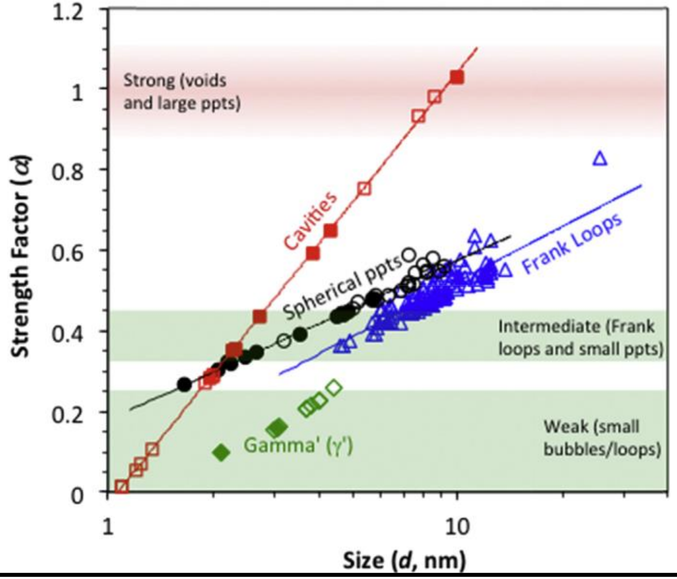
Dispersed barrier hardening(DBH): $\Delta\sigma = \alpha M \mu b\sqrt{Nd}$

$\Delta\sigma$----the change in yield strength,
M ----Taylor factor (3.06 for equiaxed BCC polycrystalline metals)
α----obstacle strength factor
b ----Burgers vector (a/2<111> in current study where a=0.248 nm)
d ----defect diameter
N---- is the defect number density.

Calculate each of the strengthening contribution

Hardening contribution in this study:

- Pure Fe: σ_{Fe}
- Solute solution: σ_{ss}
- Cavities (diameter<2nm and >2nm): σ_c
- Carbides: $\sigma_{carbides}$
- Ni/Si-clusters (large diameter): σ_{Ni}
- Dislocation loops: σ_{loop}
- Dislocation lines: σ_{line}
- Grain boundary: σ_{GB}



sample	Temp.	Dose	Disl. loops		Ni-rich	Carbides	Cavities	
	°C	dpa	<100>	1/2<111>	ppts.		d<2nm	d>2nm
DI-T91	445	16.6	0.50	0.33	-	0.74	0.21	0.72
	520	16.6	-	-	-	0.76	0.17	0.40
DI-HT9	445	72	0.64	-	0.48	0.91	0.13	1
	520	16.6	0.40	0.58		0.99	0.13	
N-T91	369	16	0.63	0.36	0.38	0.91	0.12	0.65
	453	19	-	-	-	0.74	-	-
N-HT9	450	32	-	-	0.67	0.83	0.19	0.43
	453	19	-	-	0.69	1	0.22	0.42

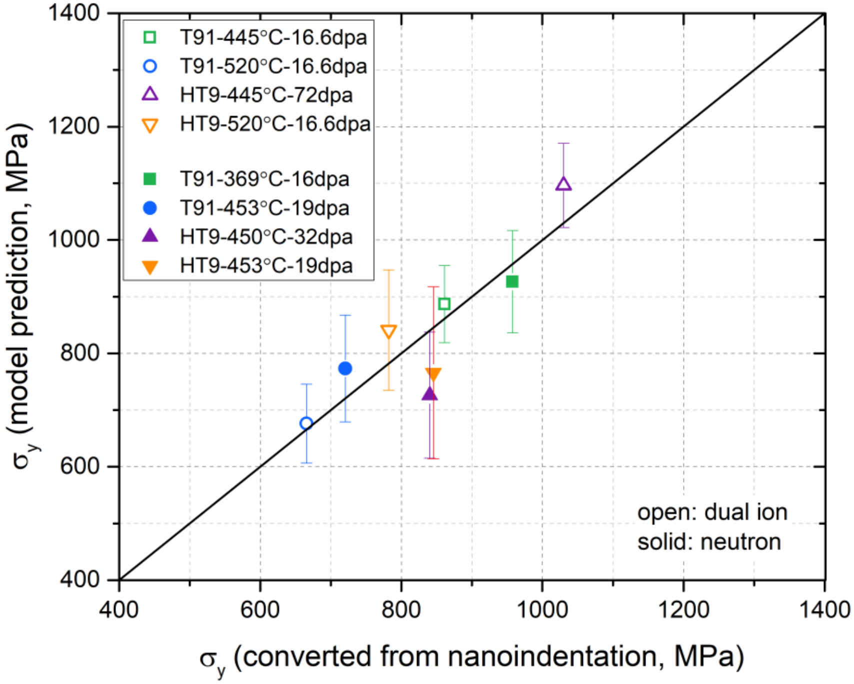
L. Tan, 728 J.T. Busby / Journal of Nuclear Materials 465 (2015) 724e730

4 Discussion: Microstructure vs. mechanical properties

A proper superposition method is needed!

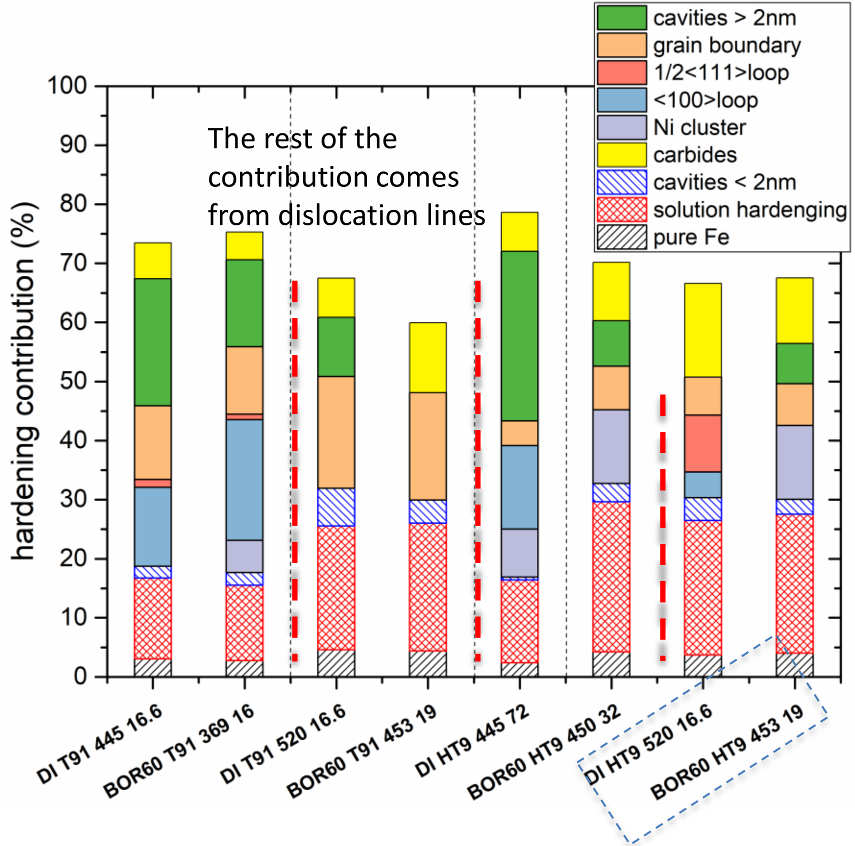
Obstacle with similar strengthening: **root-sum-square superposition**
Obstacle with different strengthening: **linear superposition**

$$\sigma_y = \sqrt{\sigma_{Fe}^2 + \sigma_{ss}^2 + \sigma_{c<2}^2} + \sqrt{\sigma_{\text{carbides}}^2 + \sigma_{line}^2 + \sigma_{Ni}^2 + \sigma_{<100>}^2 + \sigma_{1/2<111>}^2 + \sigma_{c>2}^2 + \sigma_{gb}^2}$$



→ The good quantitative agreement between the nanoindentation-tested mechanical properties and microstructure-predicted bulk properties indicates that nanoindentation can accurately predict bulk mechanical strength (within ~10% accuracy) in nano scale volumes.

4 Discussion: Microstructure vs. mechanical properties



The rest of the contribution comes from dislocation lines

- → This comparison provides another perspective of the problem of using a simplistic 70 ℃ temperature shift for the neutron vs. ion irradiation conditions.
- → The corresponding neutron and ion irradiation pairs of hardening data did not share the similar percentage of the individual strengthening contribution, which is a reflection of their inconsistent microstructures especially for HT9 alloys.

5

Conclusions

Ion irradiation as a surrogate of neutron irradiation

(1) Nanoindentation with pileup correction is important and effective for obtaining quantitatively accurate bulk equivalent hardness values for ion and neutron irradiated materials.

(2) The temperature shift theory works generally well for the cavities and dislocation loops observed in T91 following ion and neutron irradiations, but it is not suitable for the match of precipitates. Especially for the HT9 material, both dislocation loop and precipitate evolution didn’t follow the calculated temperature shift.

(3) The comparable nanoindentation results from dual-ion and neutron irradiated T91 and HT9 at elevated temperatures (>450 ℃) may be largely due to the minimal irradiation hardening at elevated temperatures. The discrepancy in the microstructures indicates that more complex modeling should be used to link ion vs neutron irradiation microstructures and hardening.

Acknowledgements

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