Task 2: Evaluation of the Causes & Mechanisms of IASCC in PWRs - Microstructural Examination of Austenitic SSs & Alloy 690 irradiated to 25 dpa

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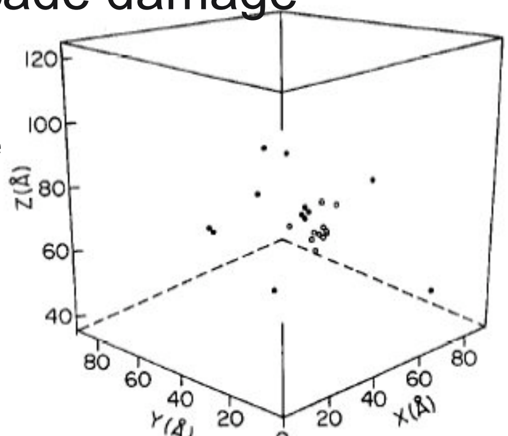
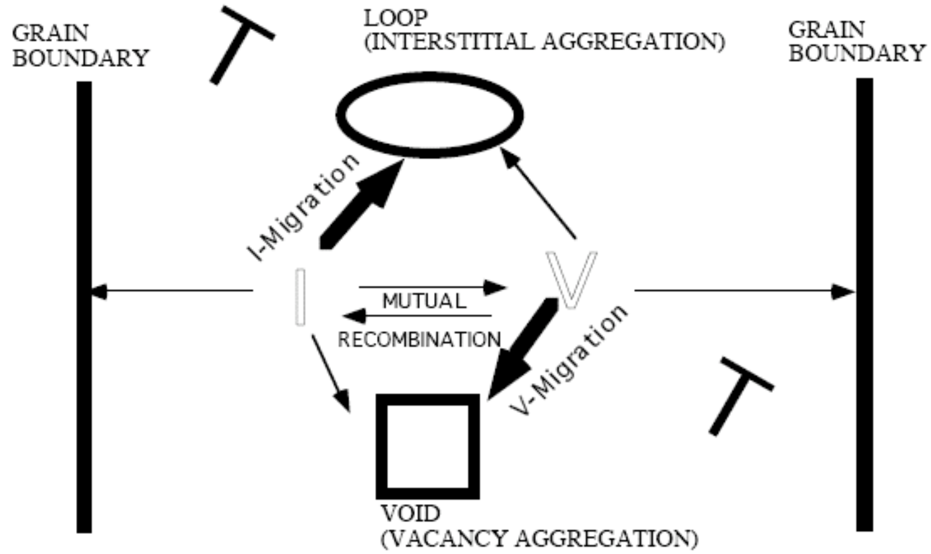
Background (1)

- Fate of point defects generated from cascade damage

Point defects at the end of a 5 keV cascade in Cu



T. Diaz de la Rubia et al., Phys, Rev. Lett., 1987

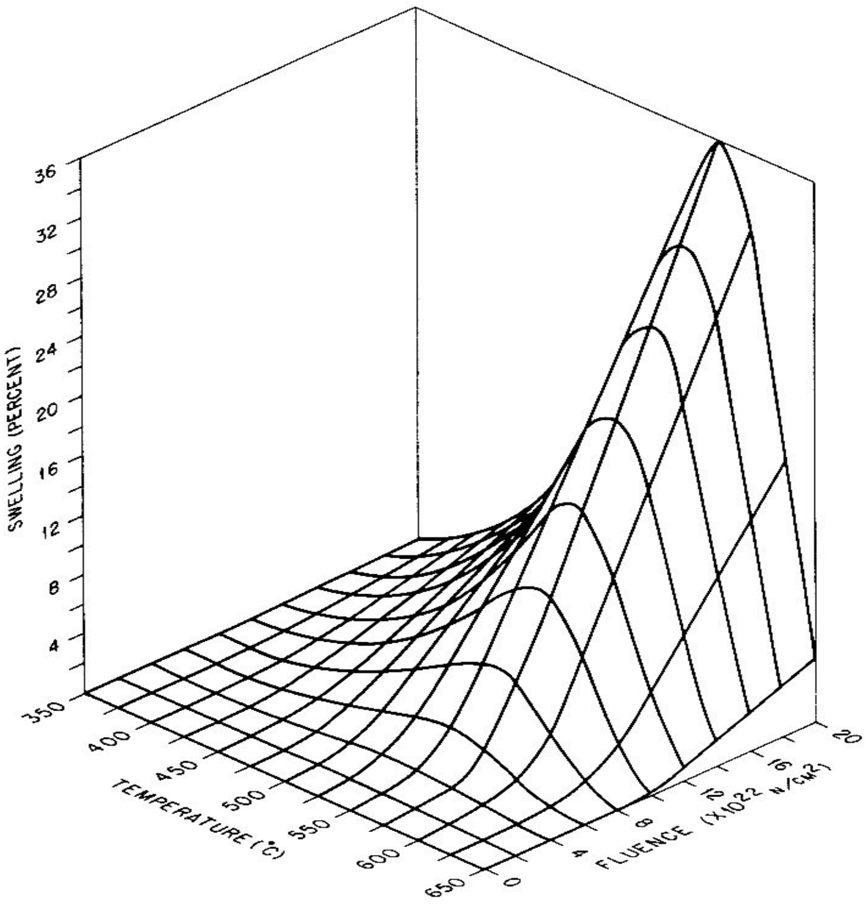


- Microstructurally visible features:
  - Dislocation loops
  - Voids
  - Precipitations

Irradiation microstructure evolution is a dynamic process controlled by irradiation condition, e.g. irradiation temperature and damage rate.

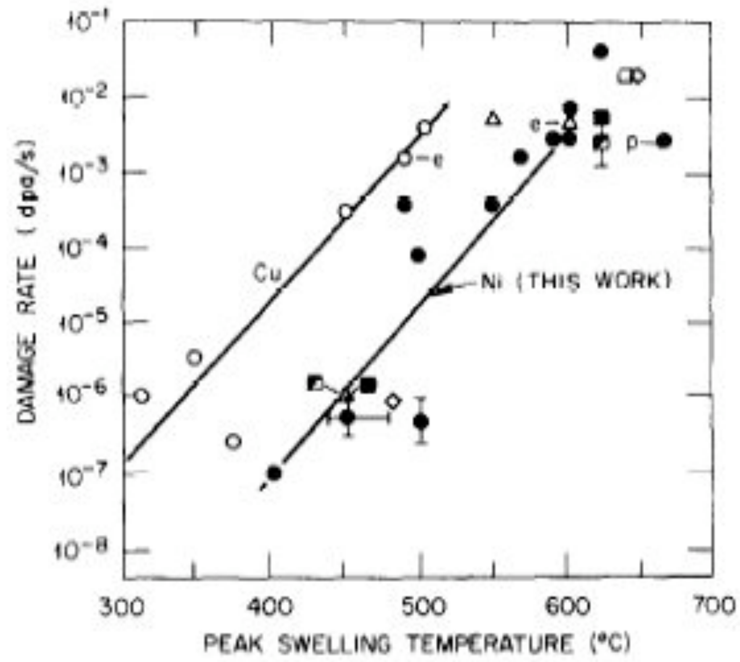
Background (2)

- Example – void swelling depends on temperature and dose rate.



Percent Swelling as Functions of Temperature and Fluence

Mansur. JNM, **216**(1994)97



Packan, JNM, **78**(1978)143

Background (3)

- Insufficient microstructure data on reactor internals for the irr. condition relevant to PWRs
  - Historically, high dose irr. microstructure studies mainly focus on fast breeder reactors or fusion system.
  - Extrapolating these data to PWR relevant conditions requires caution. (Chung, NUREG/CR-6897, 2006)

- PWR irr. condition
  - High EOL dose (life extension situations, it could be as high as ~100 dpa).
  - Moderate irradiation temperature (normally 295 ~ 340ºC, but could be as high as 370 ~ 400ºC with local γ heating).
  - Low damage rate (~ 10^{-7} dpa/s).
  - He/dpa ratio: 2.5 ~ 6 appm/dpa

Objectives: Under PWR relevant temperature & dose
- examine void swelling
- characterize irradiation microstructure

Experimental (1)

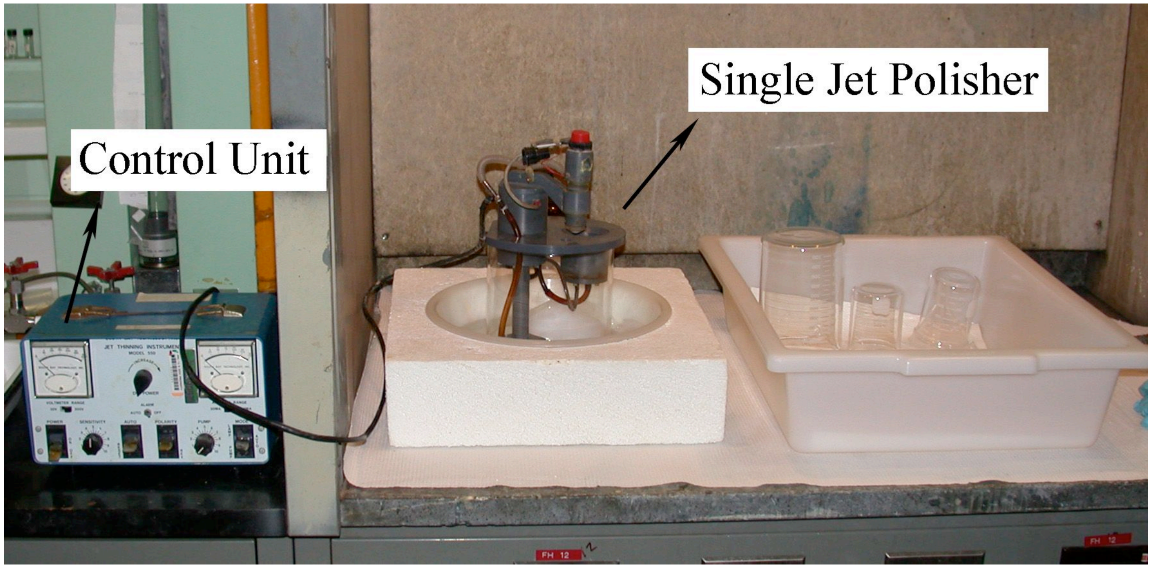
- 3-mm TEM disks irradiated in BOR-60 reactor

Mat Type	Heat ID	Ni	Si	P	S	Mn	C	N	Cr	Other Elements
304 CW	2333CW	8.50	0.65	0.031	0.029	1.38	0.035	0.068	18.3	Mo 0.37
304 SA	C12	8.23	0.47	0.018	0.002	1.00	0.060	0.070	18.43	B<0.001
316 LN SA	623	10.33	0.70	0.007	0.002	0.97	0.019	0.103	17.23	Mo 2.38, Cu 0.21
304A	C9	8.75	0.39	0.013	0.013	1.72	0.062	0.065	18.48	B<0.001, O 0.0101
HP 304L SA	945	9.03	0.03	<0.005	0.005	1.11	0.005	0.003	19.21	O 0.047, Mo<0.005
CF-8 Cast SS		8.08	1.07	0.021	0.014	0.64	0.063	0.062	20.64	Mo 0.31, δ 23.4%
CF-8 Cast SS		9.34	1.08	0.008	0.007	0.60	0.062	0.045	20.33	Mo 0.32, δ 13.5%
CF-3 Cast SS		9.40	0.92	0.012	0.005	0.57	0.009	0.052	19.49	Mo 0.35, δ 13.5%
690 Base	690	61.49	0.05	-	<0.01	0.15	0.030	-	29.24	Fe 9.02
690 GBE	GBE690	59.40	0.30	-	0.003	0.42	0.010	-	29.10	Fe 10.26
347 SA		10.81	0.29	0.023	0.014	1.56	0.030	0.021	18.06	Nb 0.60, Mo 0.29, Cu 0.09

- Irradiation condition
  - Dose ~ 24.5 dpa
  - Temperature ~ 325ºC
  - Dose rate ~ 9.4x10^{-7} dpa/s.
  - He/dpa ratio < 1 appm/dpa
- Sample radioactivity ~ 100mR/hr on contact; 0.7 mCi.

Experimental (2)

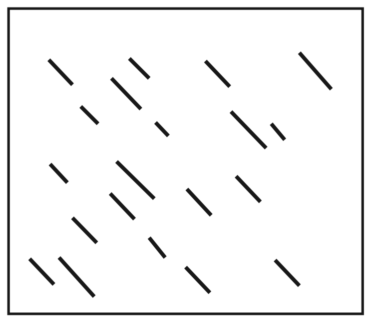
- Electrochemical method was used to prepare TEM foils



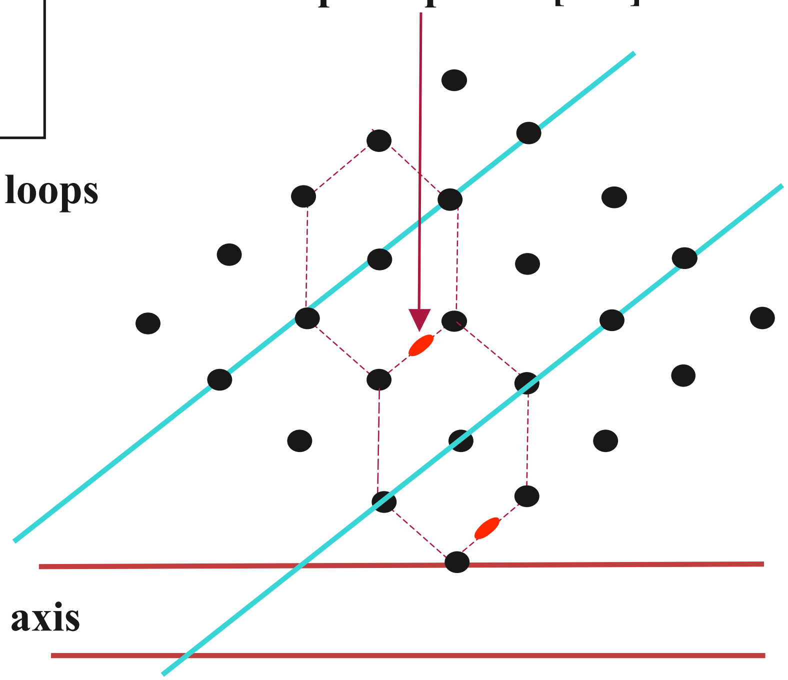
- Two-step sample preparation:
  - Uniform thinning from 250 µm to 100 µm.
  - Perforation.
- Polishing condition:
  - For SS: 6% $HClO_4$ solution @ -20^{o}C, and 70 mA.
  - For Alloy 690: 10% $HClO_4$ solution @ -40^{o}C, and 110 mA.

Experimental (3)

- Imaging faulted loops in irradiated materials using Rel-rod streak



1/4 of total faulted loops



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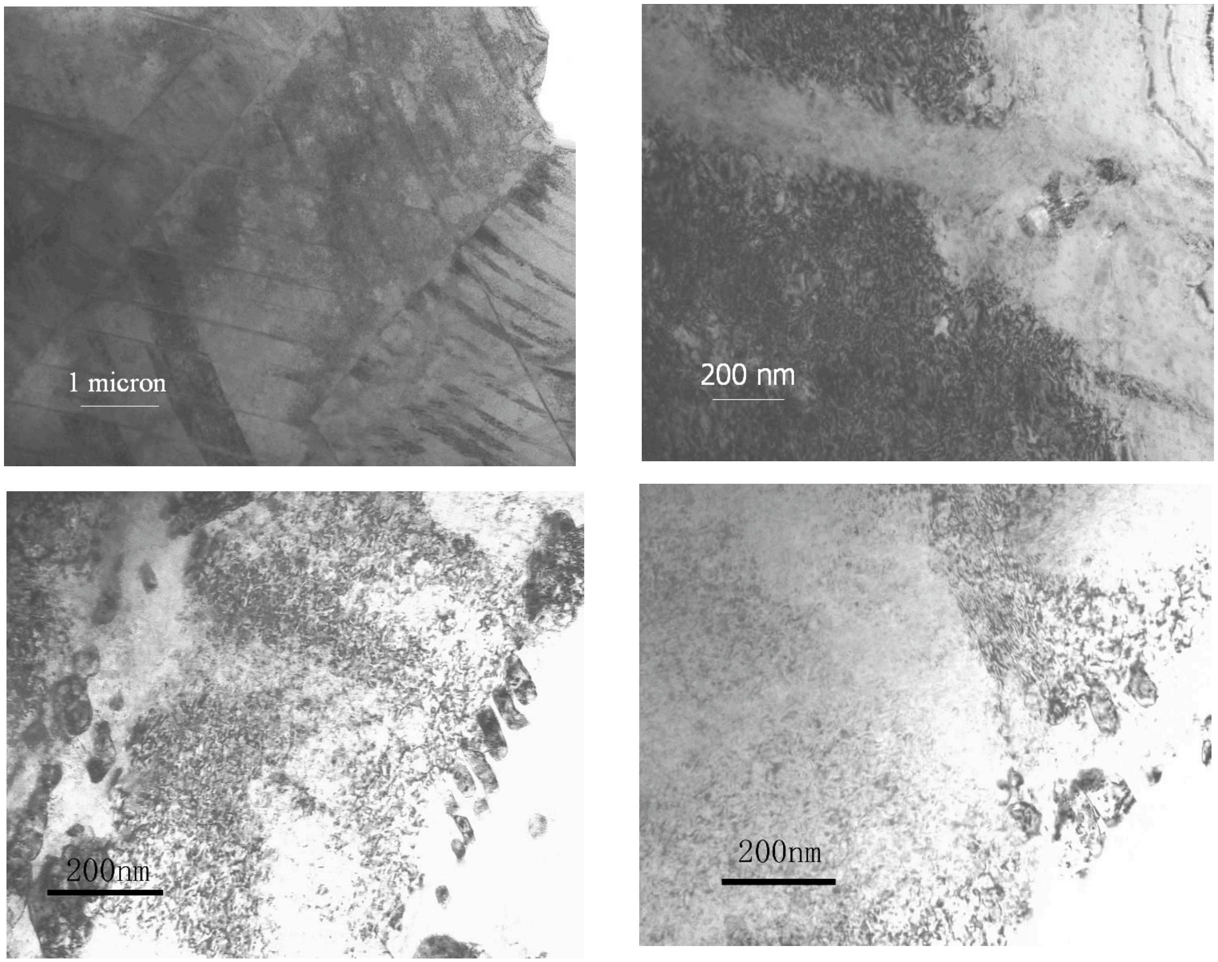
Austenitic SSs (1)

- Type 304 SS from ABB (Heat 2333), 35% cold-worked



Control sample

Irradiated to 20 dpa

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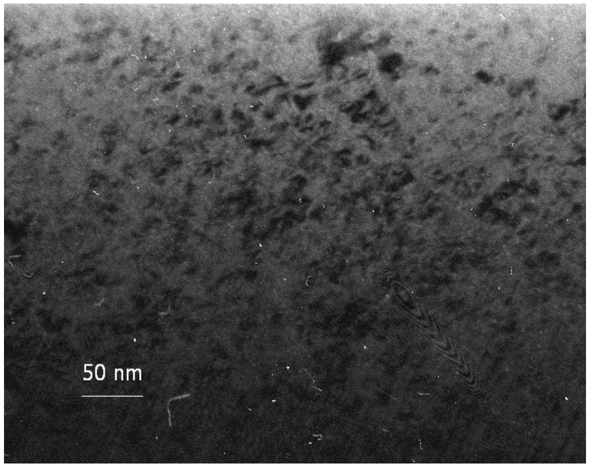
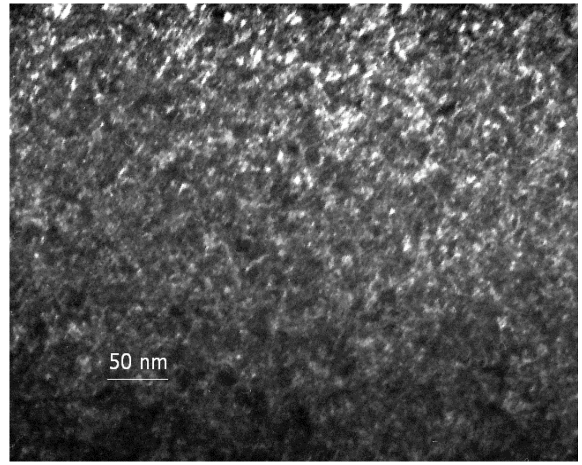
Austenitic SSs (2)

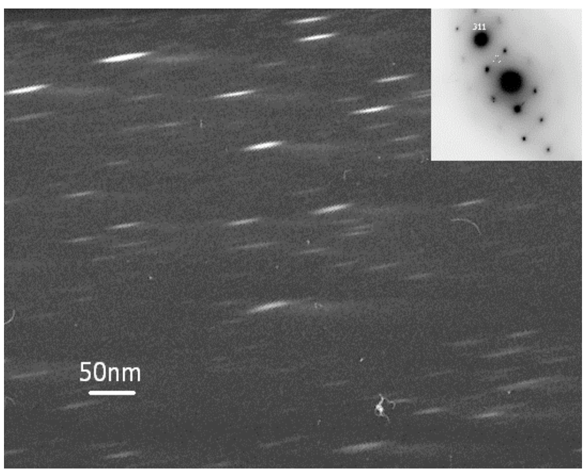
- Type 304 SS with low-S (Heat C12), SA



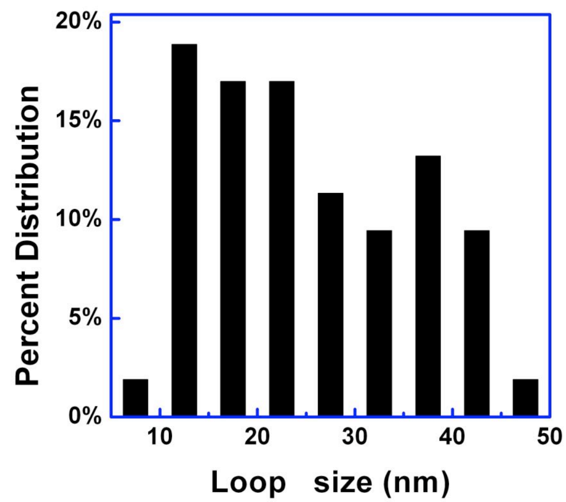
Bright Field image



Weak Beam Dark Field image

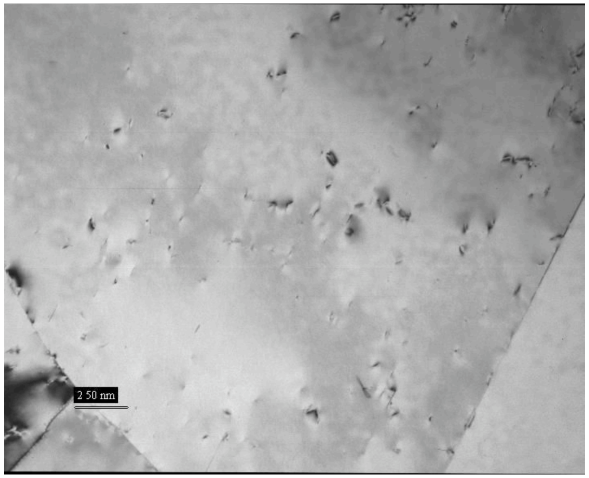


Rel-rods DF image of faulted loops

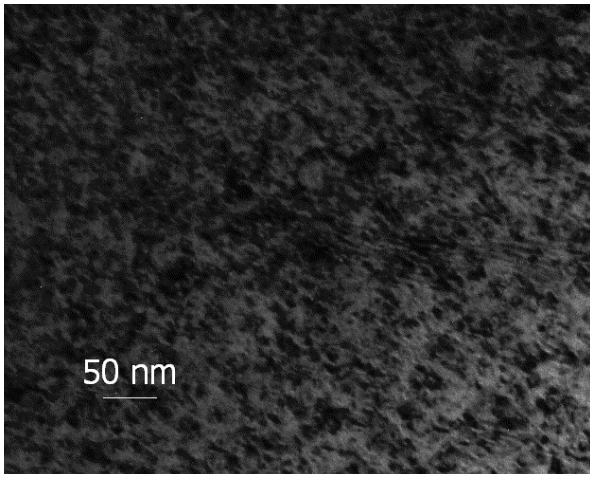


Austenitic SSs (3)

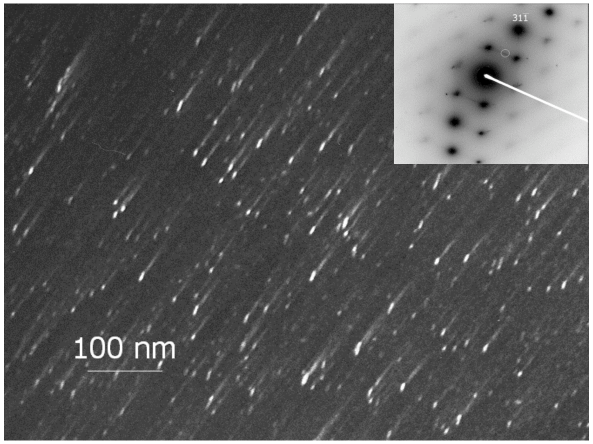
- Type 304 SS with high-S (Heat C9), SA



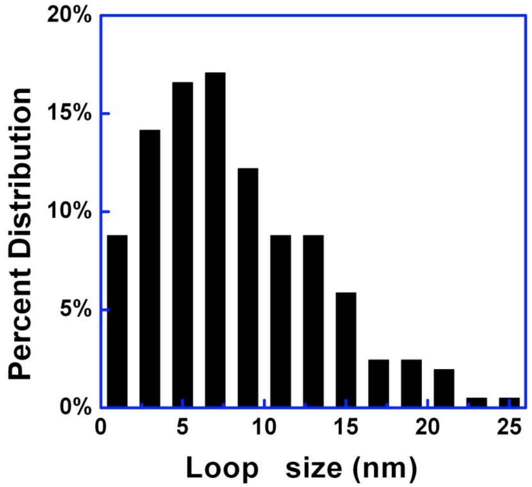
Bright Field image of control sample



TEM image of irradiated A2 sample

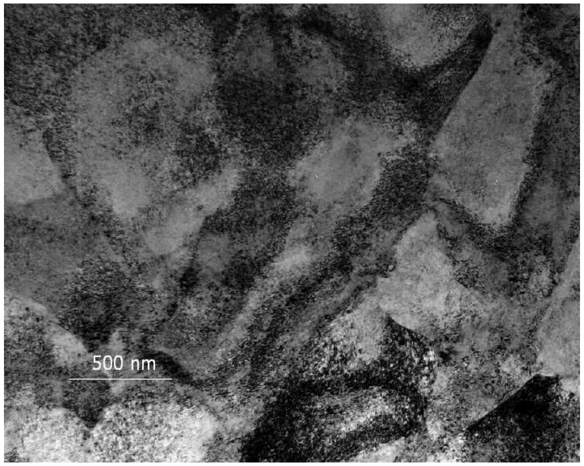


Rel-rods DF image of faulted loops

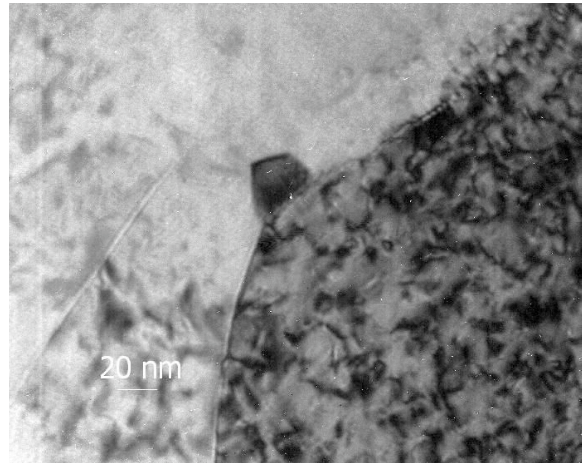


Austenitic SSs (4)

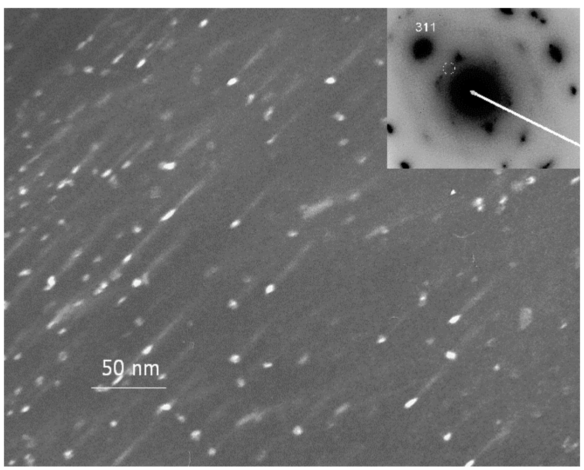
- Type 316 LN SS (Heat 623), SA



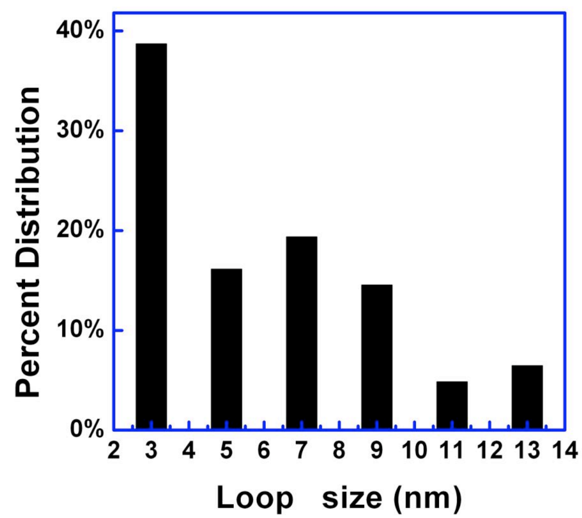
Bright Field image



Grain boundary

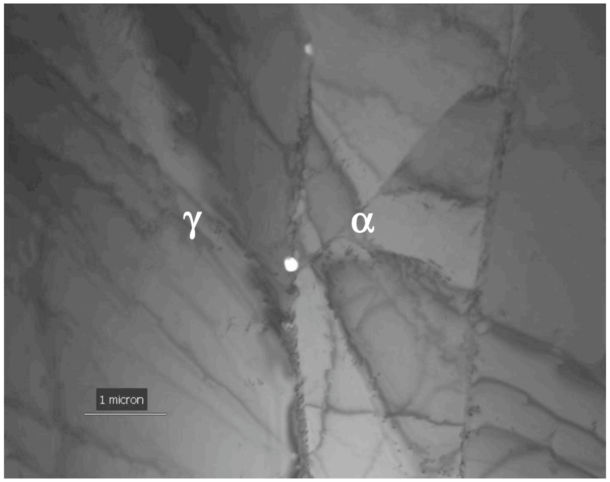


Rel-rods DF image of faulted loops

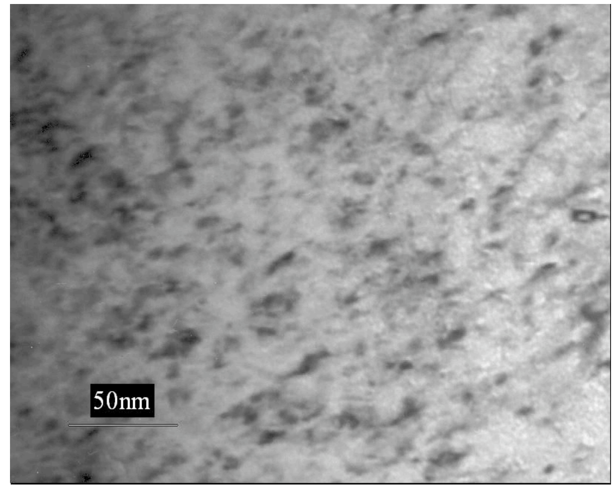


Duplex SSs (1)

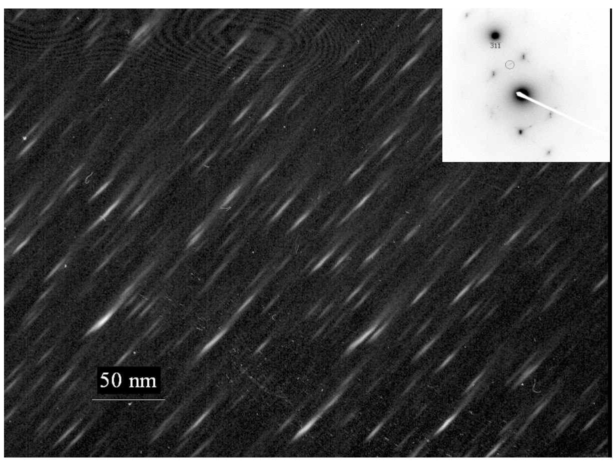
- CF-8 Cast SS (Heat 68), high-C and 23% ferrite.



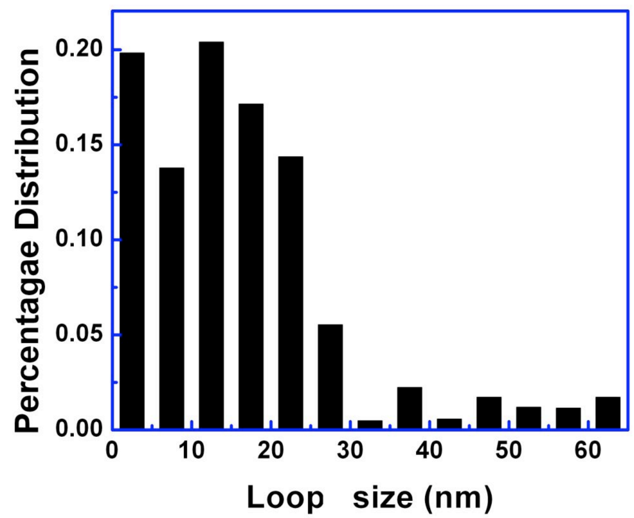
TEM image of control sample



BF image of austenite in irradiated sample

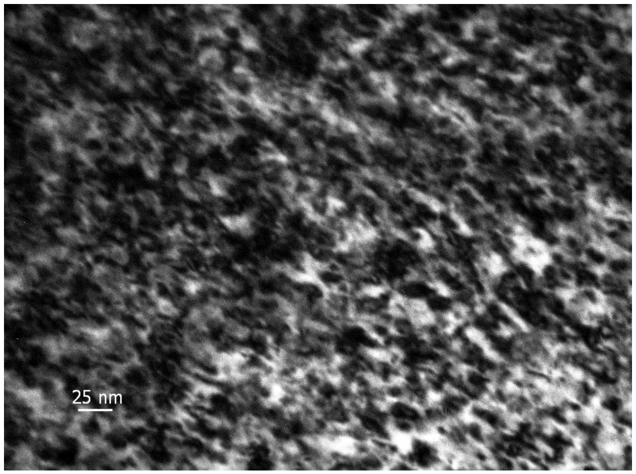


Rel-rods DF image of faulted loops

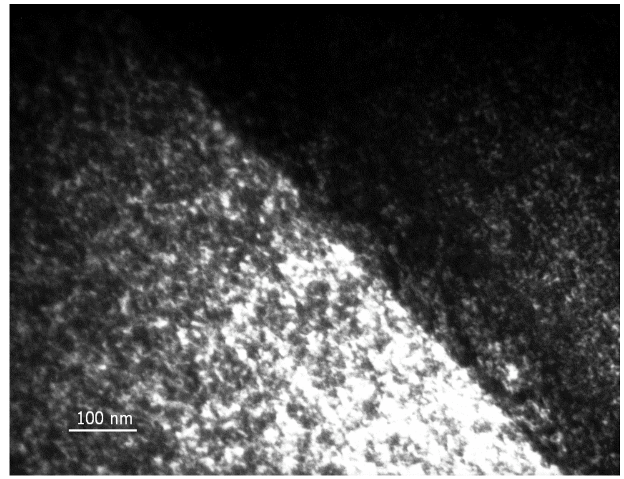


Duplex SSs (2)

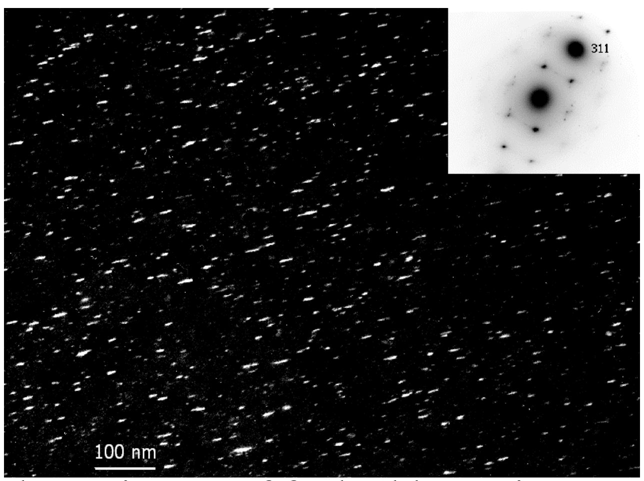
- CF-3 Cast SS (Heat 52), low-C and 13% ferrite.



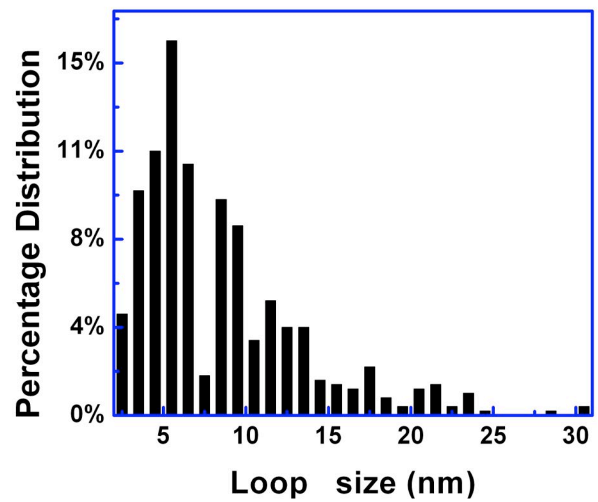
BF image of austenitic phase



BF image of grain boundary of austenite and ferrite

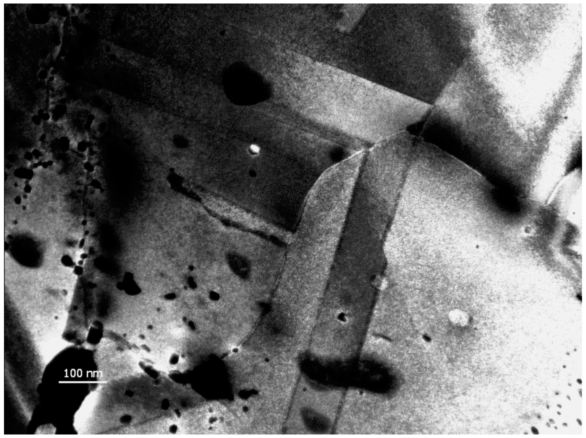


Rel-rods DF image of faulted loops in austenite

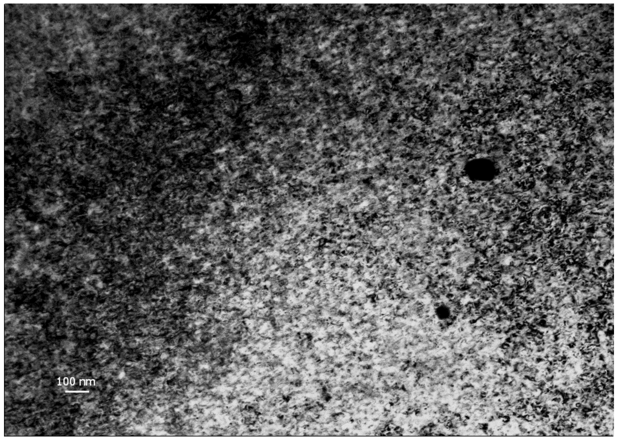


Alloy 690 (1)

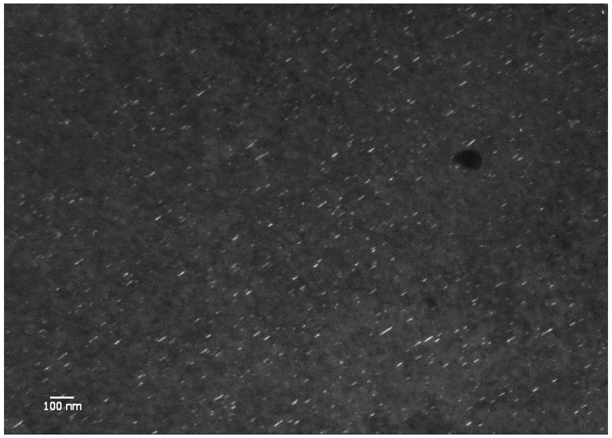
- Alloy 690 (Heat 690 Base)



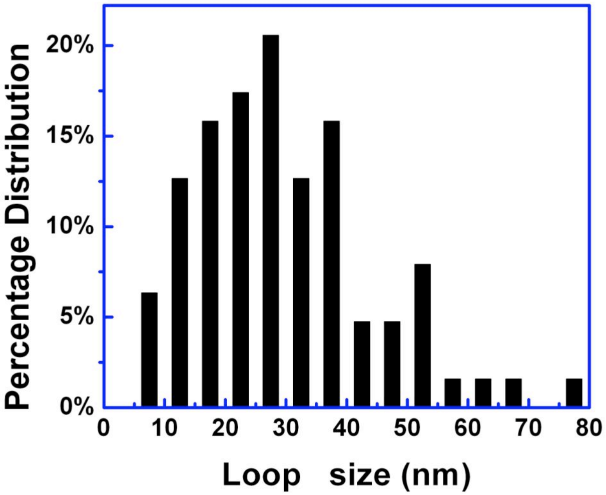
Voids in BF image with g=200



BF image of dislocation structures



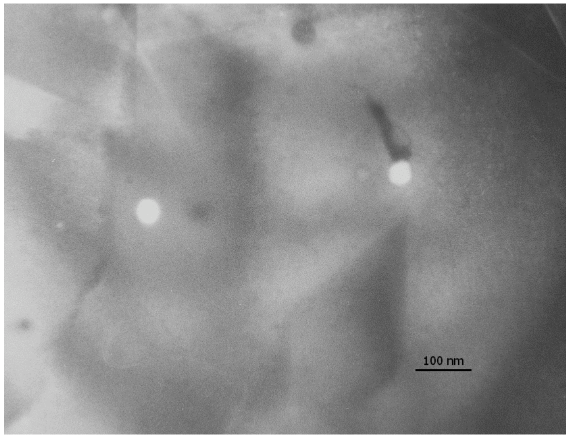
Rel-rods DF image of faulted loops



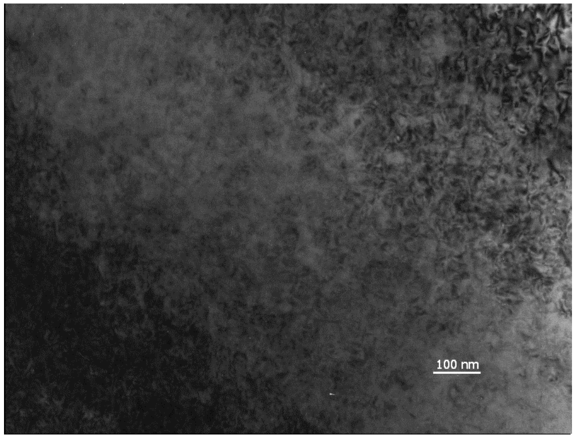
(d)

Alloy 690 (2)

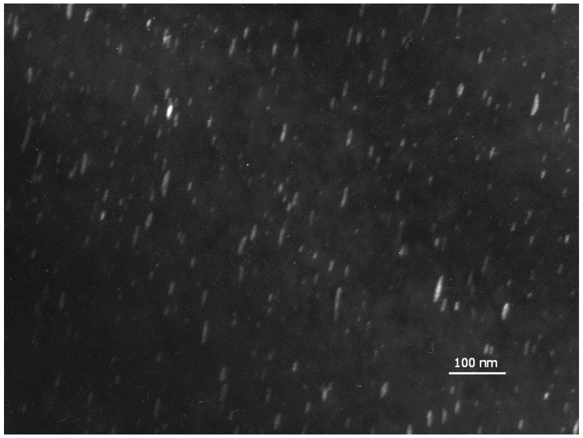
- GBE Alloy 690 (Heat 690 GBE)



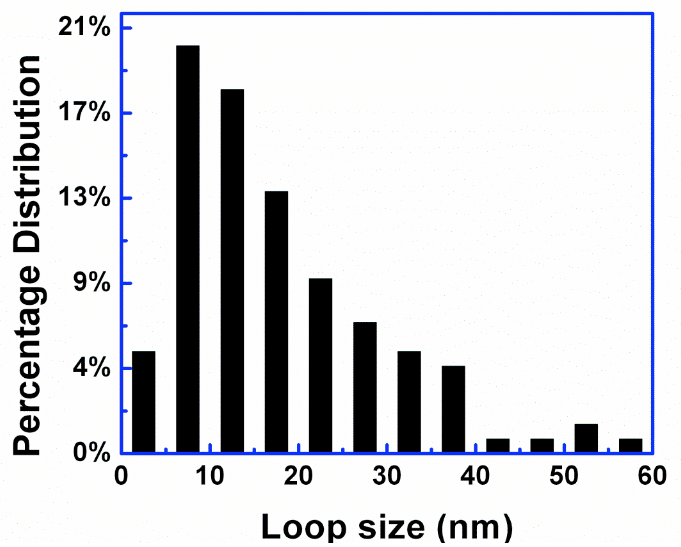
Voids in BF image with g=200



BF image of dislocation structures



Rel-rods DF image of faulted loops



Summary

Average size and density of Frank loops in the irradiated Austenitic steels

Material Type	Mat. Code	Average Size (nm)	Dislocation Density (m^{-3})	Voids
316 CW	B2	Not measured	Not measured	No
304 SA	A3	25.5	3.0×10^{22}	No
316 LN SA	B3	5.8	1.68×10^{22}	No
304 SA	A2	8.1	4.6×10^{22}	No
HP 304L SA	A8	12.2	1.2×10^{22}	No
347 Stainless steel	D1	9.39	2.0×10^{22}	No

Average size and density of Frank loops in γ phase in irradiated cast steels

Material Type	Mat. Code	Average Size (nm)	Dislocation Density (m^{-3})	Voids
CF-8 Cast SS	C4	16.25	1.74×10^{22}	No
CF-8 Cast SS	C2	7.7	3.14×10^{22}	No
CF-3 Cast SS	C1	8.48	2.8×10^{22}	No

Quantitative characterization of Frank loops in Nickel Alloys

Material Type	Mat. Code	Loop Mean Size (nm)	Loop density (m^{-3})	Voids
Alloy 690 base	E2	28.9	1.13×10^{22}	Observed
Alloy 690 GBE	E1	17	1.37×10^{22}	Observed

Conclusions

- No voids were observed in irradiated SSs, while some voids were found in the base and GBE treated Alloy 690.

- The density & size distribution of dislocation loops in SSs & Alloy 690 are consistent with reported results relevant to PWR in literatures.

- Radiation induced fine precipitates were not observed in high density in any examined materials.

- Future work
  - Exam the void swelling at higher dose.
  - Establish the dose dependence of dislocation loop characteristics.
  - Exam the deformation microstructure in irradiated material if possible.

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