IRRADIATION EFFECTS ON MICROSTRUCTURAL EVOLUTION (PART 1): DEFECTS, SEGREGATION, PRECIPITATION

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Introduction

- SOTERIA Task 4.1 and D4.1 is focused on providing an understanding of irradiation effects proton- and neutron-irradiation that can contribute to the development of predictive models for IASCC.
- Research focused on IASCC tends to be treated as a subset of SCC: Stress – Susceptible Material – Reactive Environment (where irradiation contributes to both material susceptibility and the reactive environment)
- This first presentation focuses on **irradiation-induced defects, irradiation-induced solute redistribution, (transmutation effects) and irradiation-induced precipitation**, to assist in understanding how irradiation affects the material microstructure (and environment) for IASCC. (new SOTERIA results and selected historical)

Introduction

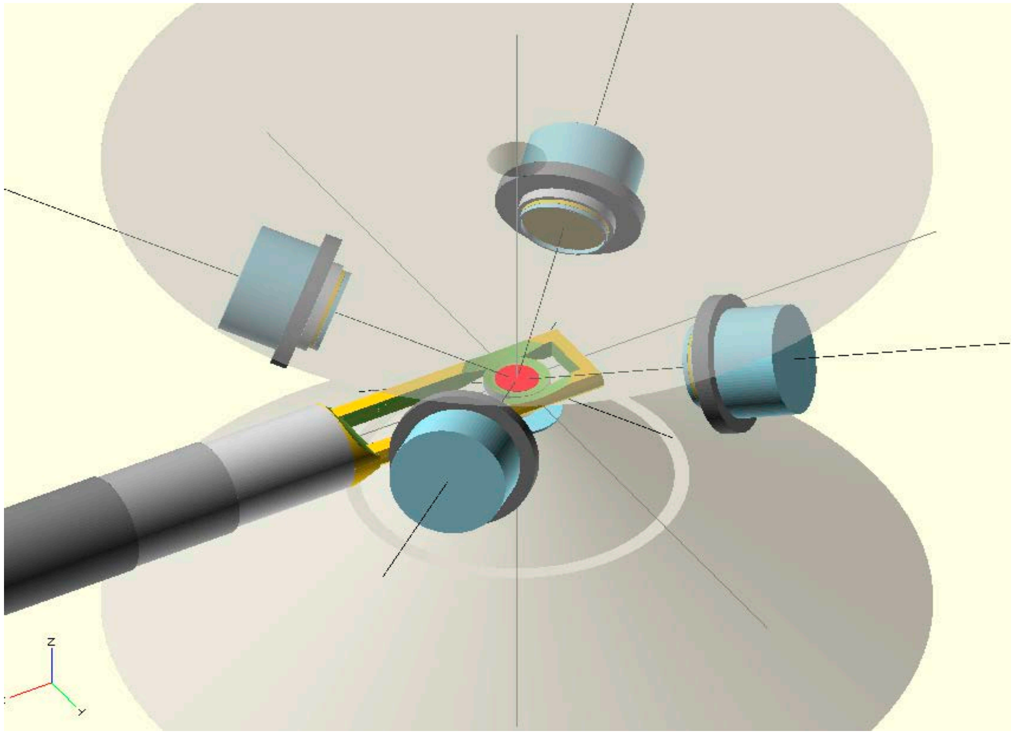
- Neutron irradiation: creation small defect clusters throughout the matrix (causing hardening), and solute segregation/redistribution/clustering, and in some cases precipitation.
- For fcc metals/alloys (austenitic stainless steels, Alloys X-750, 625):
  - "Black spot" damage; dislocation loops; stacking fault tetrahedra (Ni-base alloys)
  - Nanoscale cavity formation
  - Radiation-induced segregation/solute clustering
  - Radiation-induced precipitation
  - Transmutation effects (B, Ni)

All of these microstructural features can affect IASCC susceptibility

Characterisation

- **Analytical Electron Microscopy** (AEM) – encompassing conventional TEM/diffraction, STEM and STEM-EDX microanalysis – can provide **structural and nanoscale compositional information** concerning irradiation damage in metals
  - TEM: Conventional bright-field, centred dark-field (weak-beam); electron diffraction
  - STEM (imaging/HAADF): Complementary imaging
  - STEM-EDX microanalysis: *STEM-EDX Spectrum Imaging*; discrete "spot" analyses (semi-quantitative/quantitative with care)
  - STEM-EELS: Discrete analyses and Spectrum Imaging (qualitative)

Advanced Analytical Electron Microscopes



Multi Detectors and/or larger Solid Angles Improves Collection Efficiency

FEI Super X Geometry

4 @ 30mm² Cylindrical Detectors

$$\wedge_{total} \sim \copyright \wedge_{30} = 0.68 \text{ sR}$$

FEI Talos: ~0.8-0.9 sR: Provides improved X-ray collection (improved detectability/sensitivity)

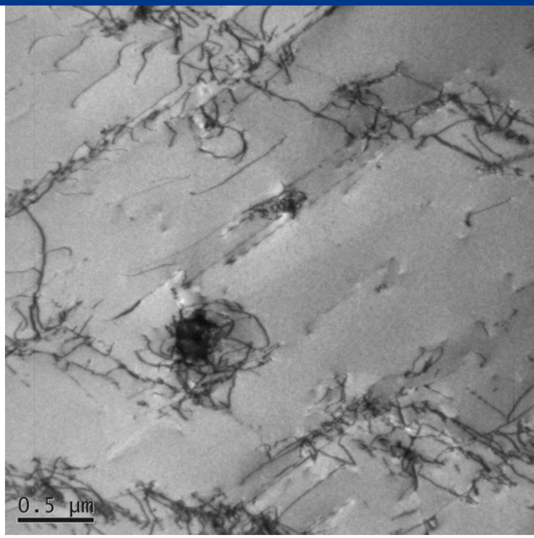
XEDS Solid Angle Calculator http://tpm.amc.anl.gov/NJZTools/XEDSSolidAngle.html

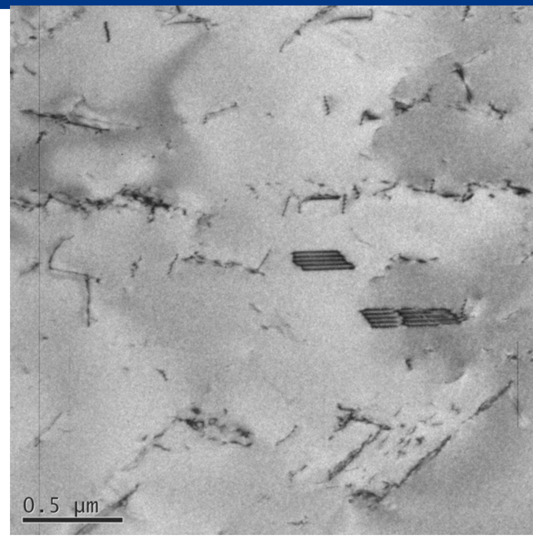
doi:10.1017/S1431927614000956.

Experimental: Characterisation

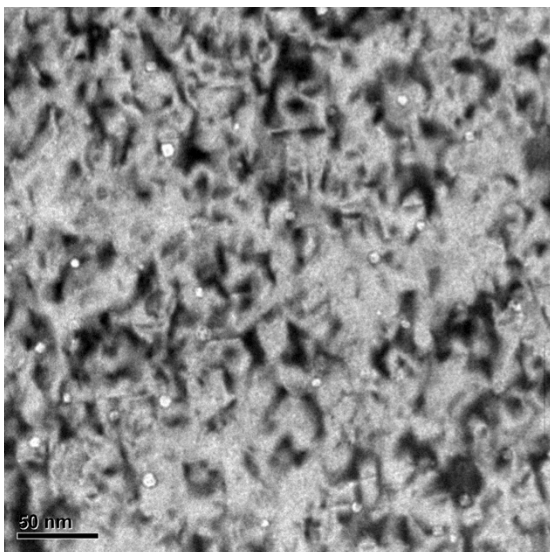
- TEM Specimen Preparation:
  - Neutron-irradiated: Twin-jet electropolish
  - Proton-irradiated: Mechanical dimpling to <~ 20 μm thickness + twin-jet electropolish
  - **Caution for FIB due to ion-irradiation defects that appear similar to neutron and proton irradiation-induced defects!**
- Characterisation: Conventional TEM and AEM (STEM-EDX) for analysis of irradiation-induced damage (defects, precipitation, segregation)

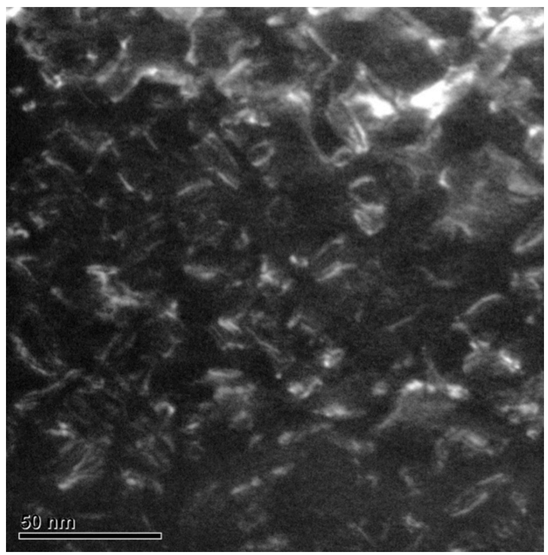
Type 316L austenitic stainless steel





Non-irradiated baseline:
- Annealed, some dislocations associated with processing/cooling

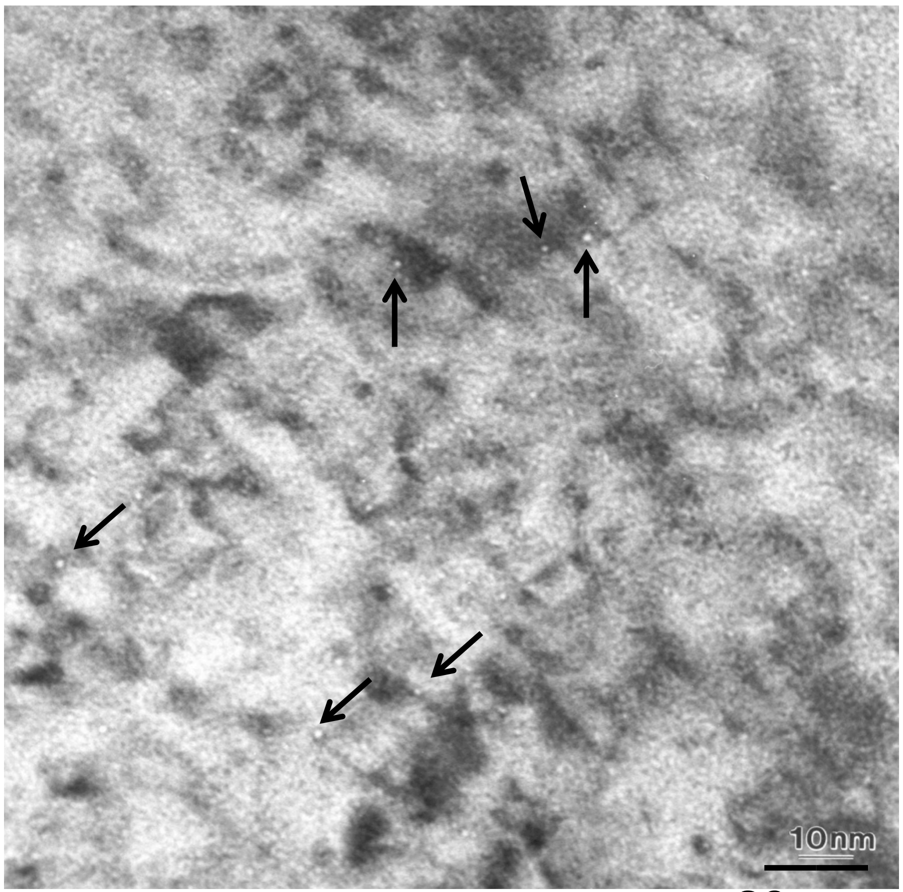




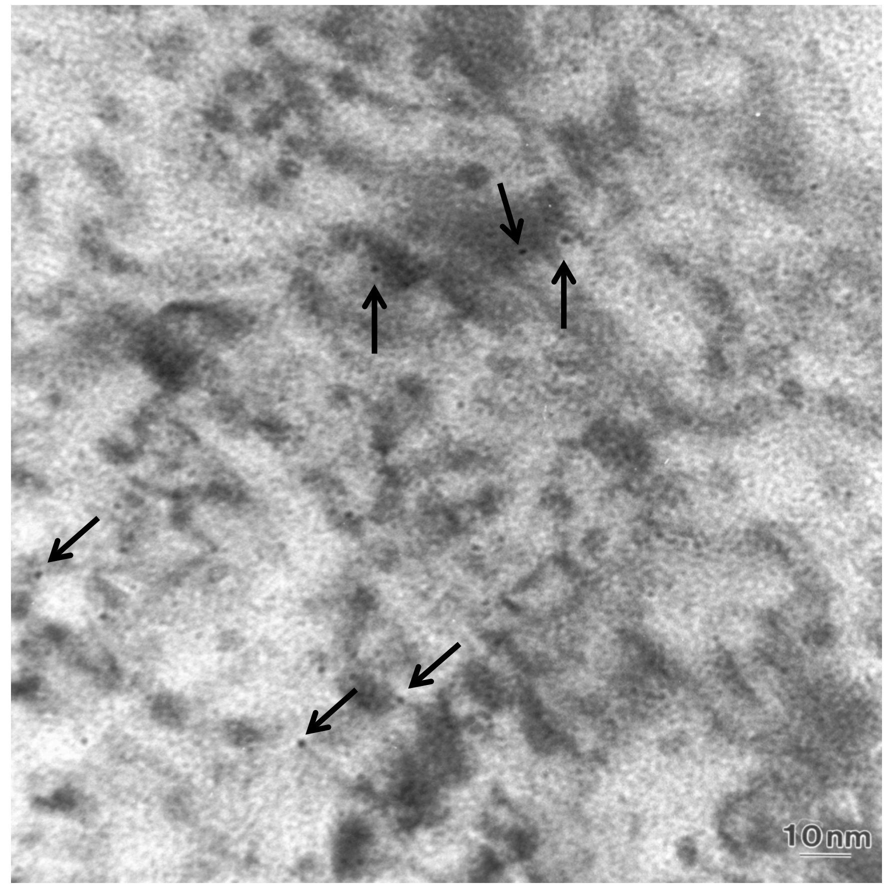
Proton-Irradiated (~5 dpa est.) at 350° C:
- Numerous very fine (~5- ~10 nm diameter) cavities
- Numerous dislocation loops (~5 to ~30 nm)

Neutron-irradiated CW 316 SS: ~35dpa

Irradiation-induced nanoscale cavities (~1-2 nm) in flux thimble tube at ~310° C (~6 X $10^{23}/m^3$)

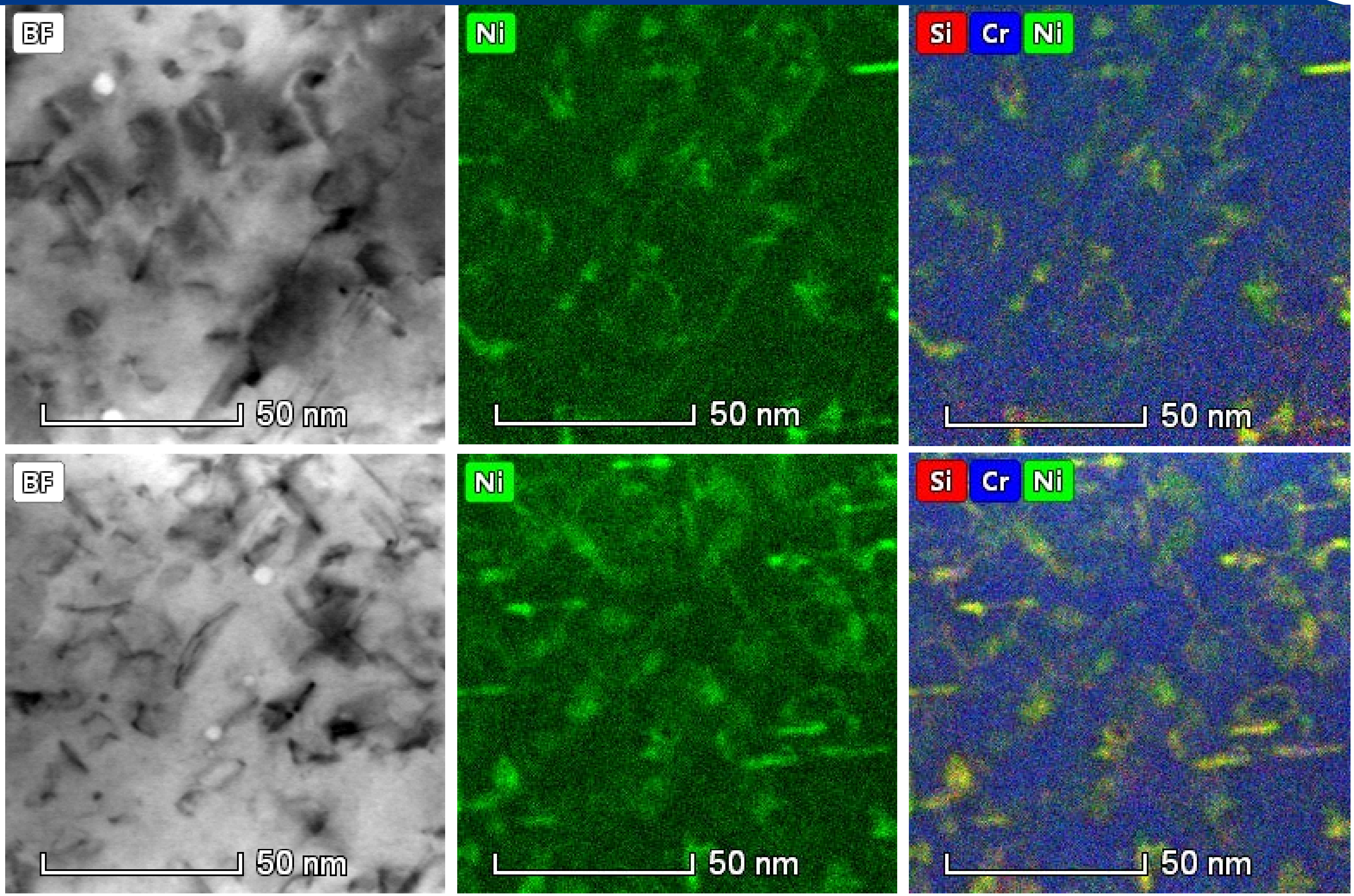


20 nm



Foster *et al.*, JNM (1995)

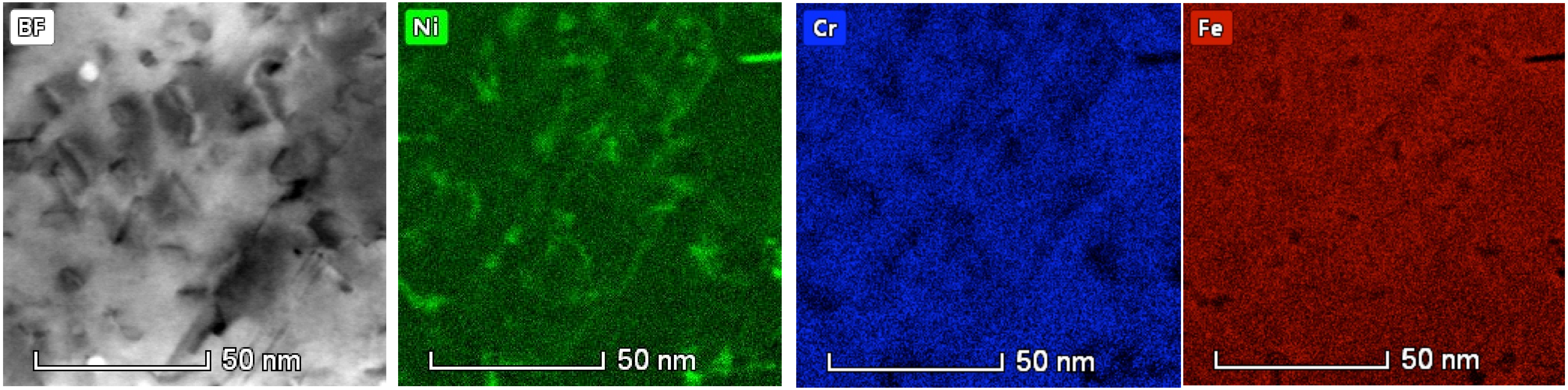
Proton-irradiation-induced segregation: 316L



~5 dpa

- Note significant Ni and Si segregation to loops, cavities, etc.
- Fe and Cr displaced

Proton Irradiation-induced segregation – 316L

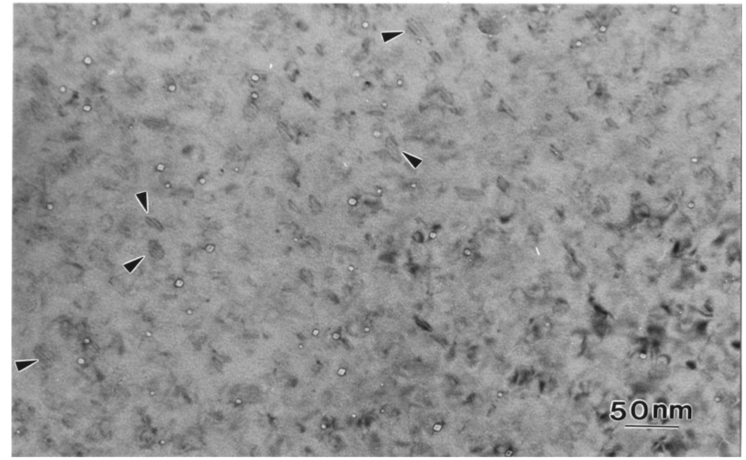


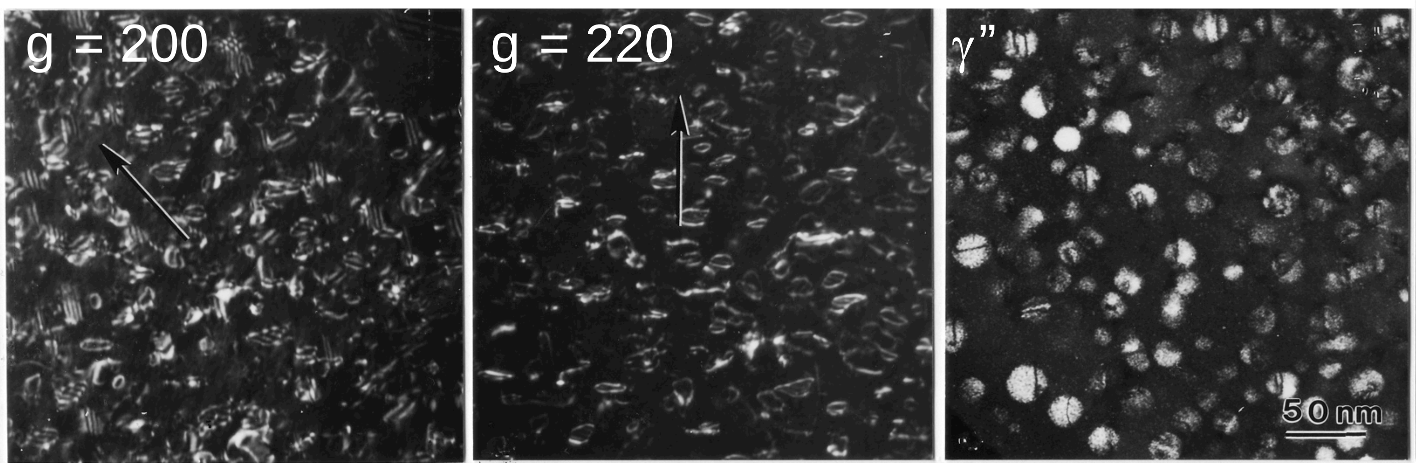
- Fe and Cr displaced from loops, dislocations and cavity/matrix interface
- Different from thermal “sensitization” (Cr-depletion to levels <~12 wt.% due to $M_{23}C_6$ precipitation in range ~550-~850° C)
  - Fe increased in Cr-depleted zone, Ni is constant
  - Irradiation-induced segregation: Ni and Si increase, Cr AND Fe levels are reduced

IASCC of Ni-base Alloy X-750 (γ')

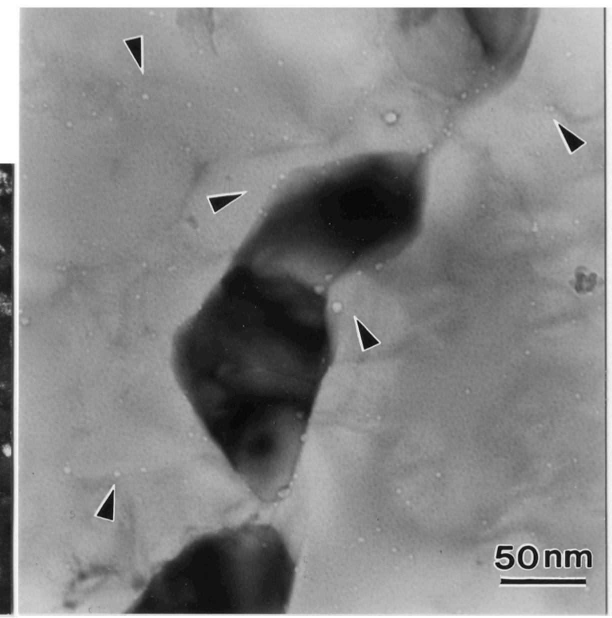
Neutron-Irradiated (~2 X10^{20} n/cm^2)

- Ultrafine (~5-10 nm) cavity formation
- Irradiation-induced loops and defects
- PIA (800°C) agglomerated He into bubbles/cavities (B effect!)
- IASCC related to He embrittlement





γ' HRDF

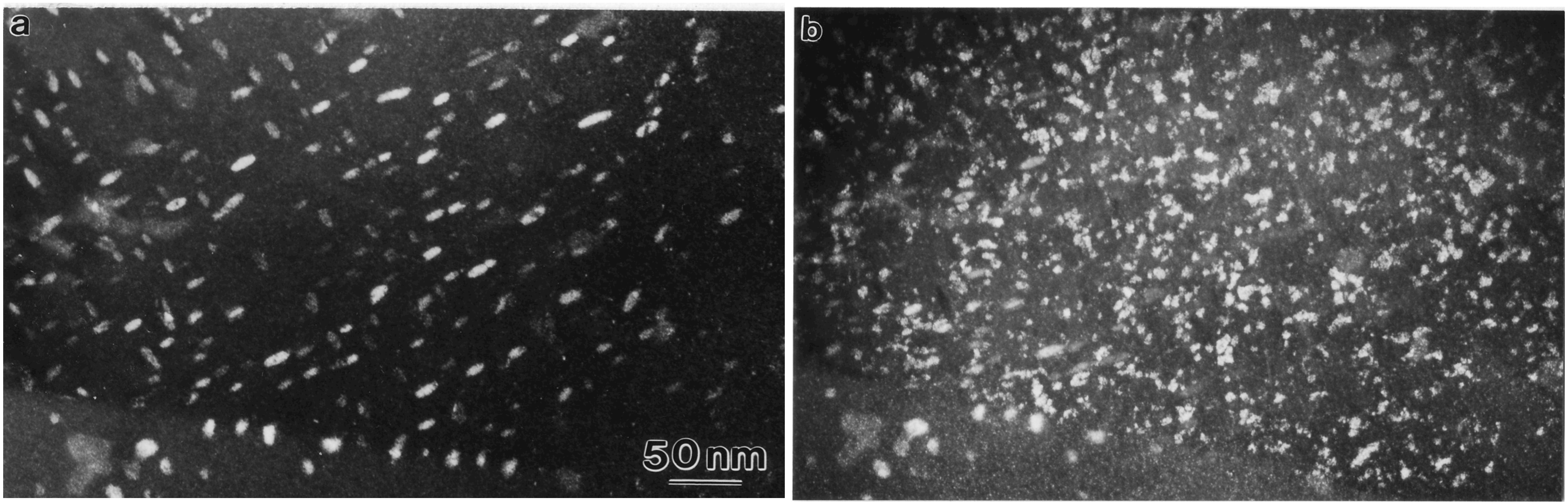


Bajaj and Burke, 1995

IASCC-resistant Direct-Aged Alloy 625 (γ'')

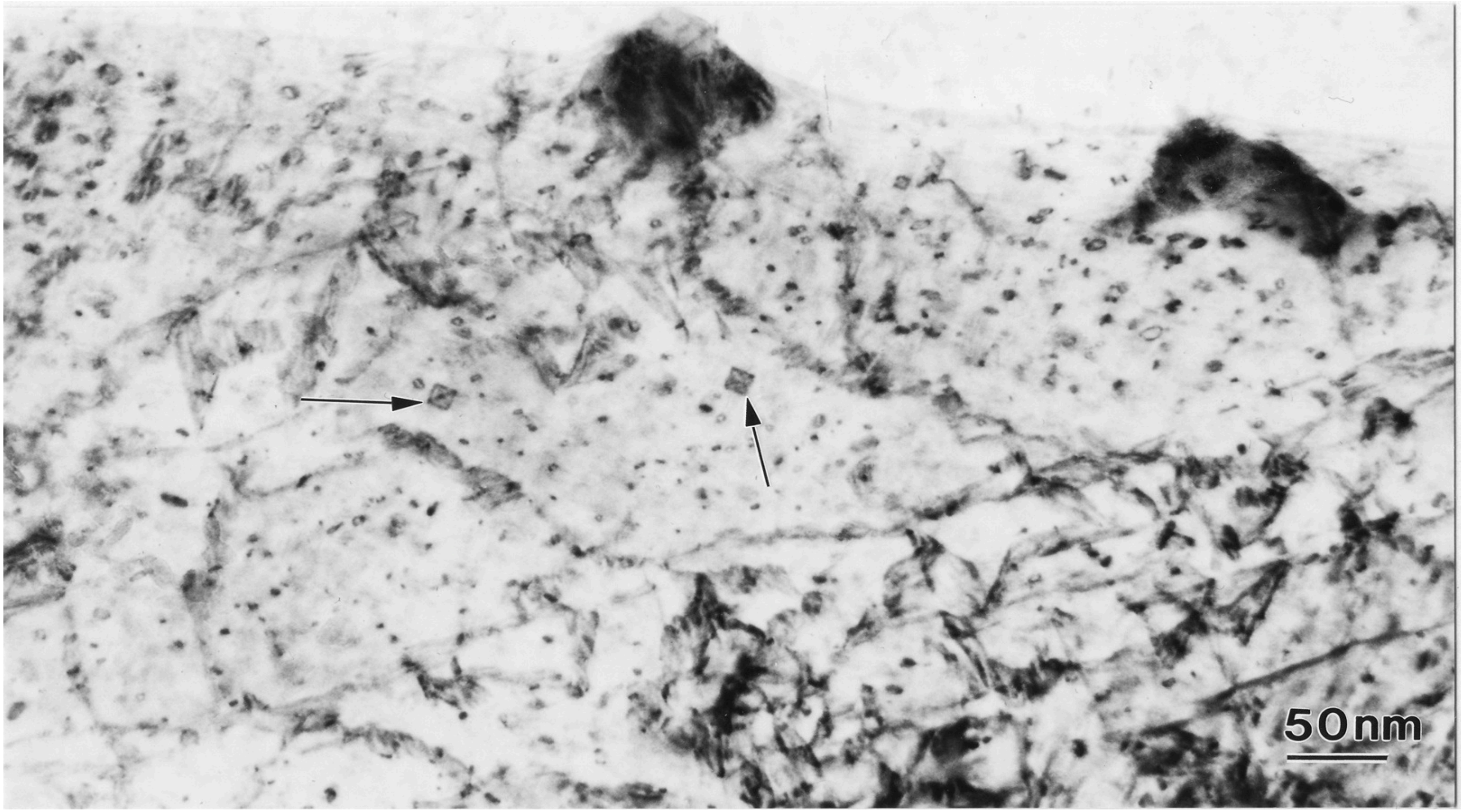
- Irradiation-induced "softening"
- Irradiation-induced "black spot" damage, SF tetrahedra
- Irradiation-induced precipitation of a Pt_2Mo-type phase
- Apparent dissolution of γ''
- Homogeneous deformation/slip

Bajaj et al., 1995
Burke and Bajaj, 1995, 1997, 1999



- ~ 2 X 10^{20} n/cm^2 at ~ 370° C
- [001] Centred Dark-Field (2 variants of γ□)

Assessing Irradiation Damage in DA-625: Microstructural Characterization



Irradiation damage in neutron-irradiated Alloy 625
~2 X 10^{21} n/cm^2, E>1 MeV at ~370° C

Burke and Bajaj, 1995

Discussion

Results of examinations of irradiated FCC alloys (SOTERIA Type 316L SS, CW316 SS Flux Thimble Tube, and Ni-base alloys HTH X-750 and DA-625)

- ❑Significant defect structures (loops, 'black spots', cavities, etc.) can form during neutron and proton irradiation.
- ❑Neutron irradiation can promote the formation of metastable phases/precipitates (Pt_2Mo-type Ni_2Cr) in some Ni-base alloys leading to softening.
- ❑Advanced AEM analyses have revealed that pronounced nanoscale segregation of Ni and Si can occur during proton-irradiation at 350° C in Type 316L SS
- ❑Segregation of Ni and Si to defects is not equivalent to "thermal sensitization" of austenitic stainless steels

Discussion

- Irradiation-induced damage will affect the yield and UTS of alloys (Wade Karlsen: deformation behaviour/structures)
- Irradiation-induced microstructural changes can affect IASCC susceptibility
  - B and Ni transmutation effects: He embrittlement?
  - Precipitation of metastable phases (possible "softening")
  - Irradiation-induced segregation can alter local electrochemical response of the alloy (hi Ni-Si/reduced Fe-Cr)
- Complex material/environment interaction leading to IASCC initiation

Summary

- Detailed characterisation of neutron-irradiated and proton-irradiated alloys have confirmed that:
  - Irradiation-induced defects (loops and “black spots”) as well as very fine cavities can readily form during neutron and proton irradiation (dependent on dose and T_{irrad})
  - Nanoscale irradiation-induced solute redistribution (segregation and clustering) can impact the alloy’s SCC susceptibility
- Need: Detailed analysis identifying the nanoscale “Precursor” reactions leading to IASCC