Monitoring Meeting:
Radiation Effects Modelling and Experimental Validation
San Sebastian , July 1st- July 2nd, 2010

Quantitative TEM investigation of neutron irradiated EUROFER97 from ARBOR 1

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Activity 8:	Experiments: development & user's facilities
Task:	TEM & SEM microstructural investigations of irradiated specimens
WP:	WP10-MAT-REMEV-08-02/KIT/BS
Reporting period:	December 2009 - June 2010
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Institution:	KIT

Objectives for Work Programme 2010

Analysis of the neutron irradiation induced evolution of microstructure in EUROFER97

- irradiation ***dose dependence*** of ***sizes*** and ***volume densities*** of radiation defects (e.g. dislocation loops, voids/bubbles, precipitates)
- neutron ***flux*** dependence of ***sizes*** and ***volume densities*** of radiation defects

→ **Correlation of changes in the microstructure to the changes in the mechanical properties (PIE)**

Verification of models for evolution of the radiation defects with dose

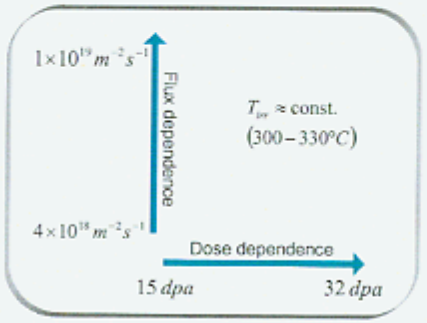
Introduction: Irradiation programs

- Different irradiation programs were performed to simulate neutron damage
- Initially the effect of the irradiation dose on defect sizes and densities is studied
- In the next step the influence of the neutron flux should be analyzed

ARBOR I (Bor 60)
T_{irr}: 330 °C
Dose: 32 dpa
Flux: 1.8×10^{19} m^{-2} s^{-1}

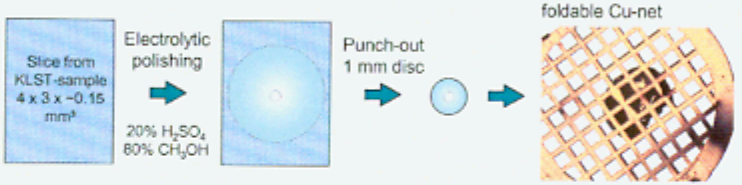
WTZ (Bor 60)
T_{irr}: 330 °C
Dose: 15 dpa
Flux: 1.8×10^{19} m^{-2} s^{-1}

HFR IIb - SPICE (HFR)
T_{irr}: 300 °C
Dose: 15 dpa
Flux: 4×10^{18} m^{-2}s^{-1}



Experimental

Preparation of irradiated TEM-samples in the hot cells of the KIT Fusion Materials Laboratory (FML)

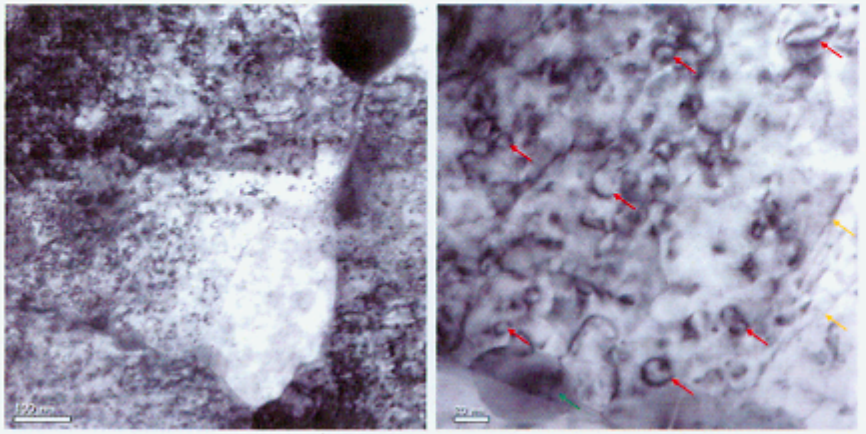


Tecnai G² F20 X-TWIN

- 200 kV, FEG
- 0.25 nm res.
- Tilt angles α, β max. 30°
- HAADF detector
- EDX
- Post-column GIF (res. ≤0.8 eV)
- EELS, EFTEM
- digital camera (2k x 2k pixel)

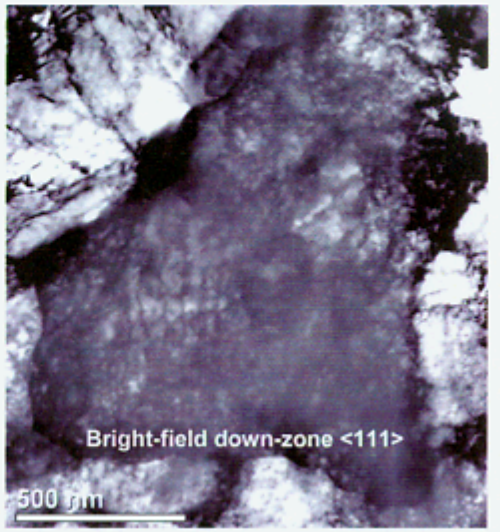
TEM investigation of EUROFER97, 32 dpa
Bright-field imaging

Irradiated microstructure:

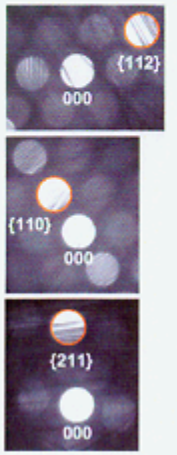


Complex dynamical diffraction contrast ➡ WBDF imaging for quantification

TEM investigation of EUROFER97, 32 dpa
Grain #1, ZA <111>



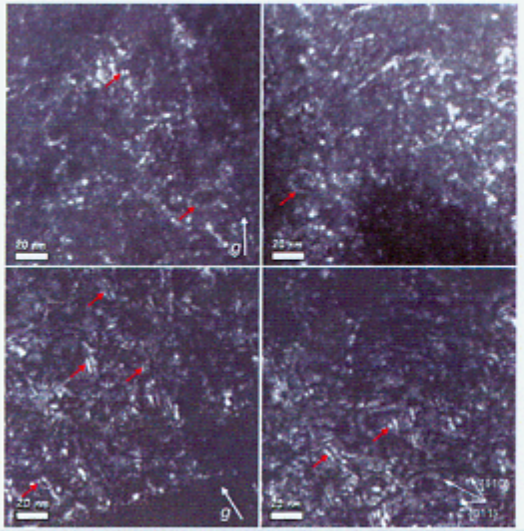
3 different diffraction conditions were used:



TEM investigation of EUROFER97, 32 dpa
WBDF imaging grain #1

g={211}
g(3.1g)

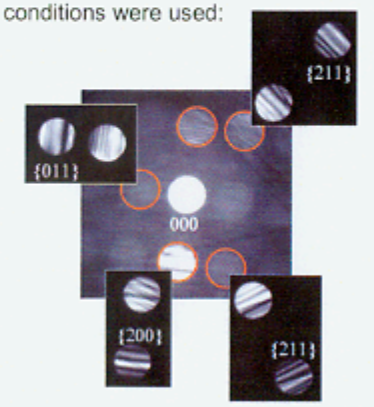
g={110}
g(7.1g)



- Dislocation loops with diameters of up to 20 nm
- Small defect clusters visible as white dots
- To some extent the defects are hard to identify
- Detrimental background contrast

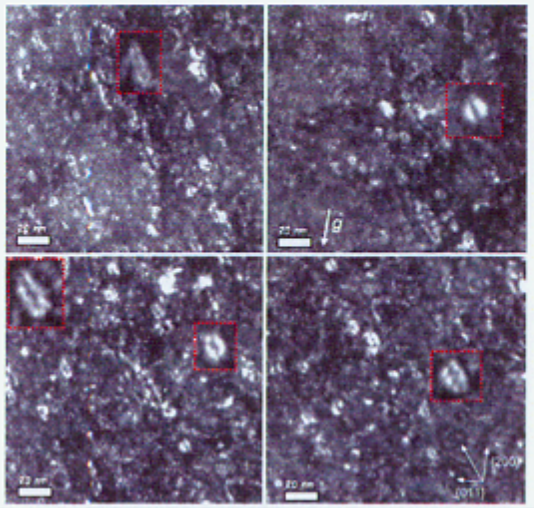
TEM investigation of EUROFER97, 32 dpa
Grain #2, ZA <011>

5 different diffraction conditions were used:



TEM investigation of EUROFER97, 32 dpa
WBDF imaging grain #2

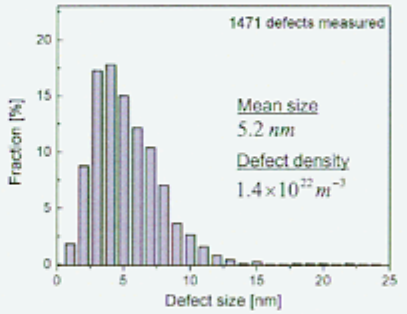
g={200}
g(4.1g)



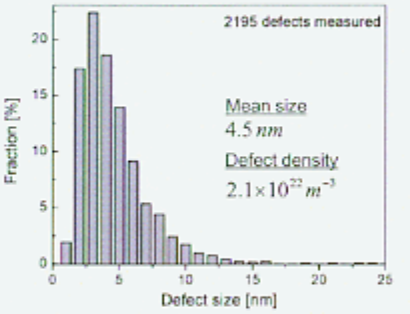
- Dislocation loops with diameters of up to 25 nm
- Loops appear edge on, with strong double arc contrast
- Most loops are oriented along {211} directions
- Less background contrast

TEM investigation of EUROFER97, 32 dpa
Quantitative results

Grain #1: g={110}, g(7.1g)



Grain #2: g={200}, g(4.1g)



- CBED → $t_{grain\ \#1}$ = 146 nm; $t_{grain\ \#2}$ = 106 nm
- Density of visible defects is about $2.1\times10^{22}\,m^{-3}$

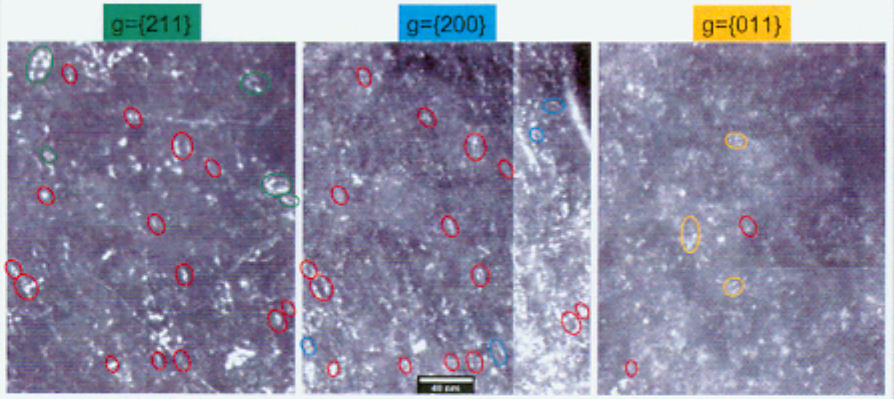
TEM investigation of EUROFER97, 32 dpa
Quantitative results - literature data

Material	Irr. source	Dose (dpa)	T_{irr} (°C)	Av. defect size (nm)	Defekt density (m^{-3})	Reference
EUROFER97	n (BOR60)	31.8	332	4.9	2.1×10^{22}	This work
EUROFER97	n (HFR)	16.3	300	(5-25)	4.0×10^{21}	[1]
F82H	n (HFR)	8.8	302	5.4	4.5×10^{22}	[2]
F82H	n (HFR)	9.2	310	6.9	2.8×10^{22}	[2]

- Except [1] no quantitative data is available for irradiated EUROFER97
  → Comparison with F82H at similar irradiation conditions
- Average size of defects in EUROFER97 seems to be low for 32 dpa
- Identification of larger loops was difficult → underestimation of size
- BUT: different composition, heat treatment, flux …

[1] M. Klimenkov et al., Submitted to the Journal of Nuclear Materials, Proceedings of the ICFRM-14, Sapporo (2009)
[2] R. Schäublin et al., Materials Research Society Symposium Proceedings, vol. 650 (2001), p. R181

TEM investigation of EUROFER97, 32 dpa
Burgers vector analysis

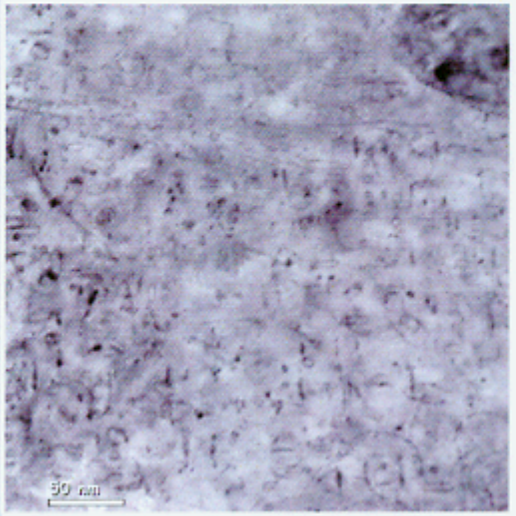


Most probable Burgers vectors:

b = <100>	b = ½ <111>
b = ½ <111>	b = ½ <111>

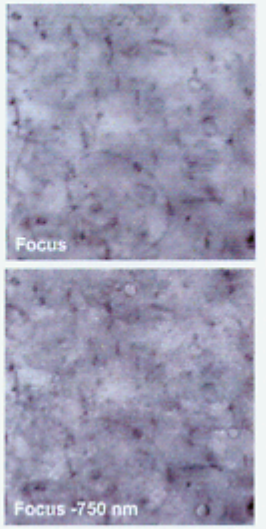
→ Majority of loops have Burgers vectors b = ½<111>

TEM investigation of EUROFER97, 15 dpa
Bright-field imaging



- Qualitative investigation of 15dpa EUROFER97 samples (WTZ) started
- Bright-field kinematical micrographs show high density of loops (diameters of up to 30 nm)

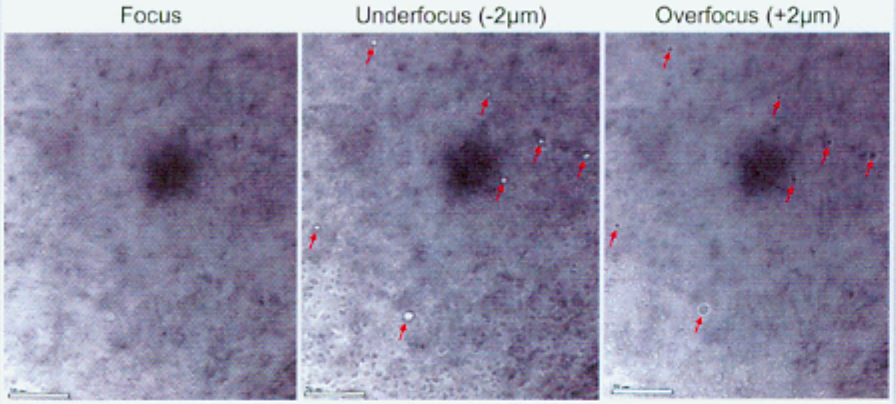
TEM investigation of EUROFER97, 15 dpa
Bright-field imaging



- Qualitative investigation of 15dpa EUROFER97 samples (WTZ) started
- Bright-field kinematical micrographs show high density of loops (diameters of up to 30 nm)
- With objective lens underfocused voids are visible

  → verify with through-focus series

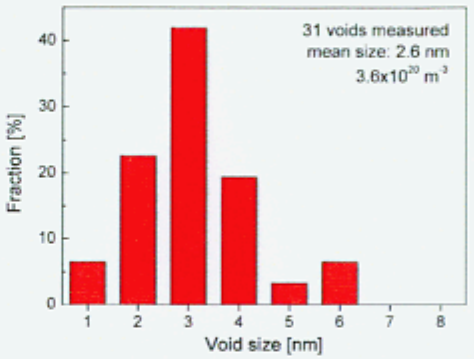
TEM investigation of EUROFER97, 15 dpa
Through-focus series

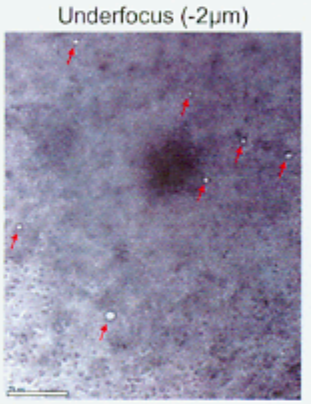


➡ Changing Fresnel contrast confirms presence of voids

➡ EELS measurement did not show any He

TEM investigation of EUROFER97, 15 dpa
Through-focus series





➡ Changing Fresnel contrast confirms presence of voids

➡ EELS measurement did not show any He

Main Conclusion of the Work Done

- More than 3600 defects were measured in 32 dpa sample (for the 2 size distributions)
- Maximum loop diameter is 25 nm, average value is 5 nm
- Density of visible defects is about 2.1 x 10^{22} m^{-3}
- Majority of loops have Burgers vector b = ½ <111>
- Voids found in 15 dpa sample

Status of the whole task (level of achievements)

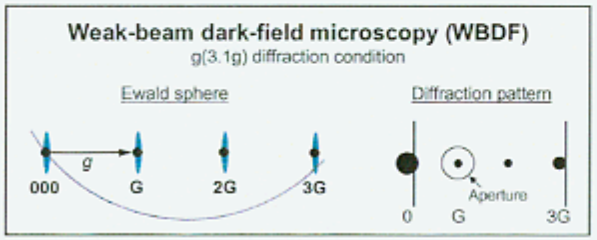
- Detailed quantitative investigation of high dose irradiated EUROFER97 from ARBOR 1
- Determination of size distributions and densities of dislocation loops
- Determination of the Burgers vectors of dislocation loops

Work planned for the next period

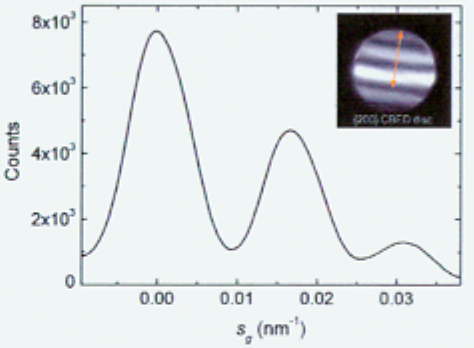
- Looking for voids in 32 dpa irradiated EUROFER97 (ARBOR1)
- Quantitative investigation of 15 dpa (WTZ) samples
- EFTEM analysis to look for α' precipitation
- Use of quantitative microstructure analysis to verify models for evolution of the radiation defects in RAFM

TEM investigation of EUROFER97
Procedure to obtain quantitative data

1. Choose grain and align major zone axis parallel to electron beam
2. Tilt sample to different weak-beam diffraction conditions
3. Record WBDF images of defects
4. Measure sample thickness
5. Analyze images with respect to defect sizes and densities
6. Use g·b=0 invisibility criterion to determine Burgers vectors



TEM investigation of EUROFER97
CBED thickness measurement



Grain #1, ZA <111>

- 2 measurements
- mean thickness: 146 nm

Grain #2, ZA <011>

- 3 measurements
- mean thickness: 106 nm

Theory: P. M. Kelly et al., Physica Status Solidi (a), vol. 31 (1975), p. 771
S. M. Allen, Philosophical Magazine A, vol. 43 (1981), p. 325

DM-script: V. Hou: Thickness by CBED, Digital Micrograph(tm) Script Database, http://www.felmi-zfe.tugraz.at/dm_scripts/