Detection of DEfects and HYDROgen by ion beam analysis in Channeling mode for fusion – DeHydroC

Project code: ENR-MAT-01-JSI

Reporting meeting - activities 2023
6. February 2024

Sabina Markelj (PI) on behalf of the team

Team members

Benificiary	Names	Expertise	Contact
JSI	Sabina Markelj	HI interaction, sample irradiation, ion beam analysis (IBA)	sabina.markelj@ijs.si
	Esther Punzón Quijorna	Channelling, IBA (Post-doc)	esther.punzon-quijorna@ijs.si
	Mitja Kelemen	Construction, IBA, channelling (PhD, Post-doc)	mitja.kelemen@ijs.si
	Matjaž Vencelj	Detectors	matjaz.vencelj@ijs.si
	Primož Pelicon	IBA, construction, channelling	Primoz.Pelicon@ijs.si
	Janez Zavašnik	TEM/SEM	janez.zavasnik@ijs.si
	Andreja Šestan	TEM/SEM, sample preparation (PhD, Post-doc)	andreja.sestan@ijs.si
MPG	Thomas Schwarz-Selinger	Sample irradiation, IBA, HI interaction, TDS	Thomas.Schwarz-Selinger@ipp.mpg.de
	Wolfgang Jacob	HI interaction, TDS	Wolfgang.Jacob@ipp.mpg.de
UHEL	Flyura Djurabekova	Multiscale modelling, RBSADEC development	flyura.djurabekova@helsinki.fi
	Xin Jin	Code development, MD, RBSADEC (PhD)	xin.jin@helsinki.fi
	Ilja Makkonen	DFT for RBSADEC (Post-doc)	Ilja.makkonen@helsinki.fi
	Tommy Ahlgren	IBA, HI interaction, MRE modelling	tommy.ahlgren@helsinki.fi
	Kenichiro Mizohata	IBA, RBS-channelling	kenichiro.mizohata@helsinki.fi
	Filip Tuomisto	PAS	filip.tuomisto@helsinki.fi
CEA	Christian Grisolia	MRE modelling	Christian.GRISOLIA@cea.fr
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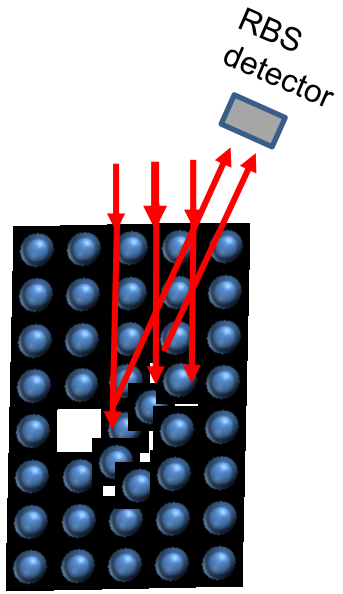
Methodology

Channeling Rutherford Backscattering Spectroscopy (RBS - C)



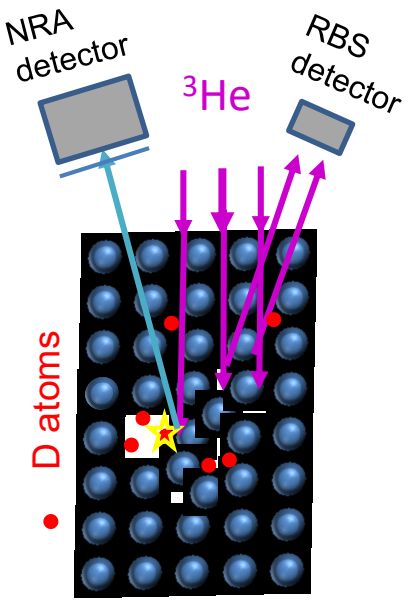
- Disorder in materials due to irradiation (depth resolved)

Defect type?

- Sensitive mainly to dislocation structure (loops, lines)

Nuclear Reaction Analysis (NRA - C)

$D(^3He,p)^4He$



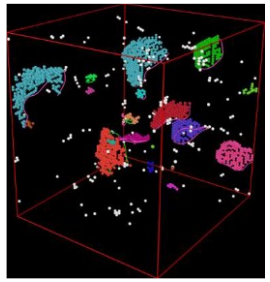
- Ion beam techniques
- Non-destructive!
- In situ!

- Deuterium populates defects in materials – analysis depth resolved

Defect type?

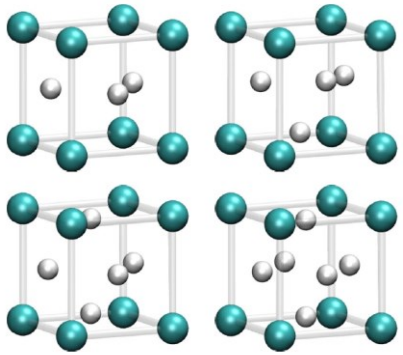
- D traps mainly in open volume defects (vacancies, vacancy clusters)

Molecular Dynamics (MD)



- Collision cascades $(\sim 10-100\ \mathrm{nm})$
- High radiation dose

Density Functional Theory (DFT)



Heinola et al. PR B (2010)

- Hydrogen atom positions around vacancy/vacancy cluster

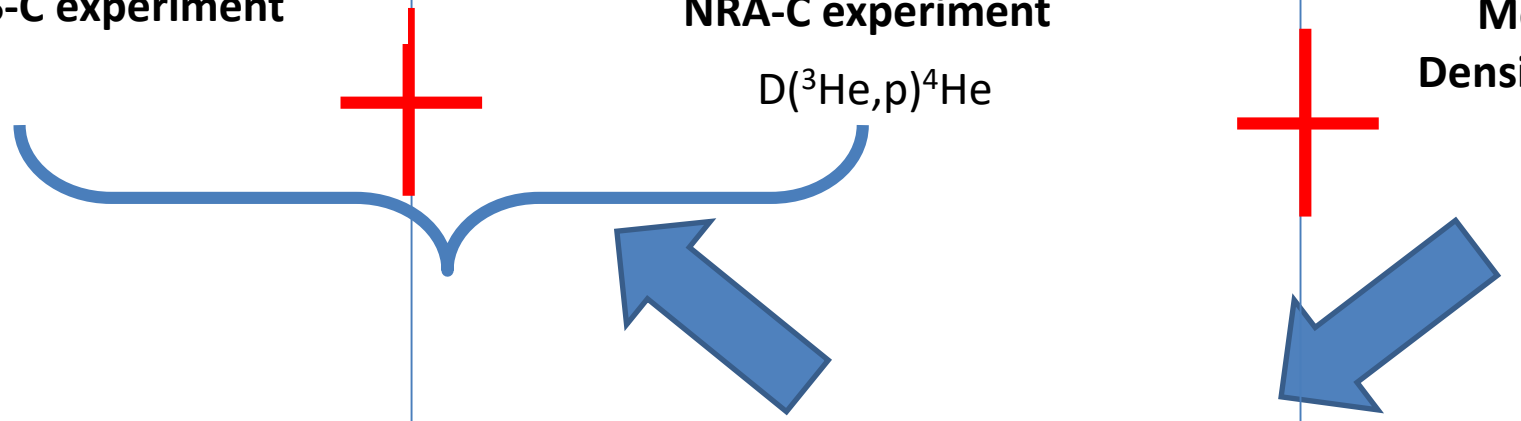
Methodology

RBS-C experiment

NRA-C experiment

$D(^3He,p)^4He$

Molecular Dynamics (MD)
Density Functional Theory (DFT)



RBS-C / NRA-C simulations
RBSADEC code

[S. Zhang *et al.*, *Phys. Rev. E*, 94 (2016) 043319]

UNIVERSITY OF HELSINKI

Combination of MD, DFT
RBS-C, NRA-C experiments and simulations

Defect types and deuterium locations!

Tasks and objectives in 2023

- Task 1.1 Incorporation of the goniometer in the INSIBA experimental station – JSI.
- Task 1.2 Detection system for ion beam methods
  - **O3.1 (Task 1.1)) C-RBS spectra obtained with new channeling set-up. (D1)**
- Task 2.1 Production of samples with dominant defects in the material – MPG and JSI.
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- Task 3.2 Development of C-NRA method - JSI, UHEL, MPG.
  - **O3.4 (Task 3.1 and Task 3.2) Detection of deuterium by C-NRA method. (M8)**
- Task 3.3 – Modelling of deuterium position in lattice/defect and identification of D position - UHEL, CEA, JSI.
  - **O3.5 (Task 3.3) Incorporation of C-NRA in RBSADEC. (M9)**

Tasks and objectives in 2023


On schedule


Delayed

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Management

WP 4 management - four zoom meetings organized with the team members, details and presentations available on Indico, links to indico sites on Wiki page https://wiki.euro-fusion.org/wiki/Project_No4

- March 2023 – three-day meeting in Ljubljana, Slovenia
- June 2023
- September 2023
- December 2023

Publications:

- Jin et al., Effect of lattice voids on Rutherford backscattering dechanneling in tungsten, J. Phys. D: Appl. Phys. 56 (2023) 065303 (13pp) https://doi.org/10.1088/1361-6463/acad12 ID: 33582
- Markelj et al., Unveiling the radiation-induced defect production and damage evolution in tungsten using multi-energy Rutherford backscattering spectroscopy in channeling configuration, Acta Materialia 263 (2024) 119499, https://doi.org/10.1016/j.actamat.2023.119499 ID: 35374

Under review:

- Jin et al. Analysis of lattice location of deuterium in tungsten and its application for predicting deuterium trapping conditions, Submitted to PRM (Rejected at Acta Materialia) ID: 36157
- Markelj et al., First study of the location of deuterium in displacement damaged tungsten by nuclear reaction analysis in channeling configuration, Submitted to NME as ICFRM proceedings ID: 33906

In preparation:

- Zavašnik et al. Microstructural analysis of tungsten single crystals samples irradiated by W ions: the effect of dose and temperature on damage evolution

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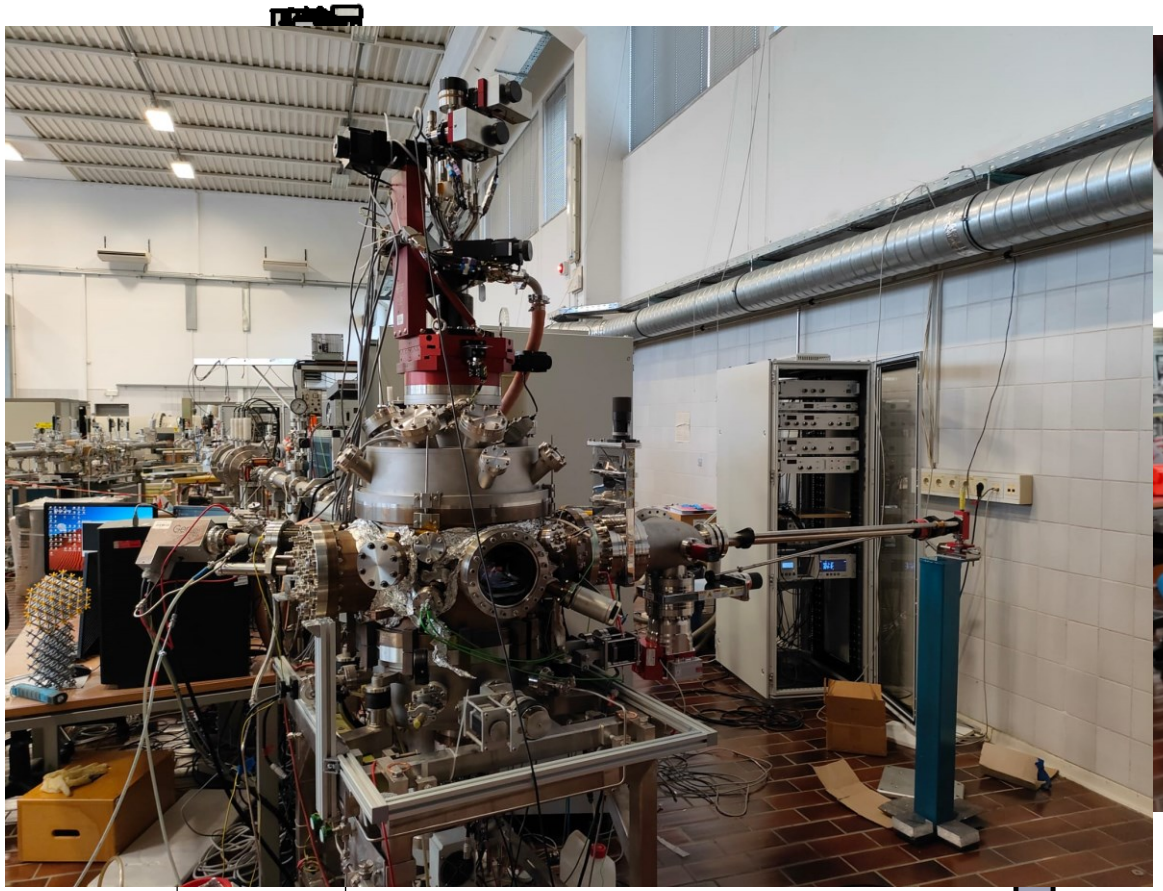
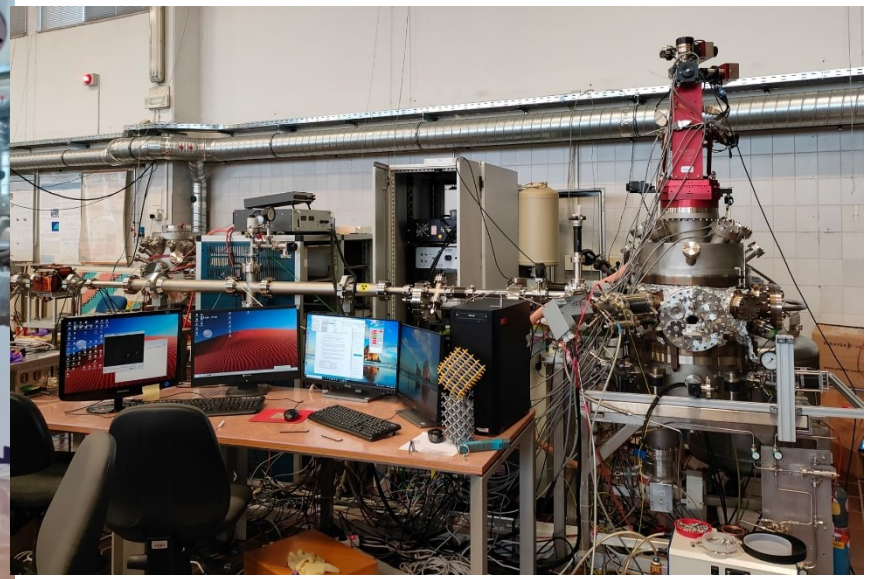
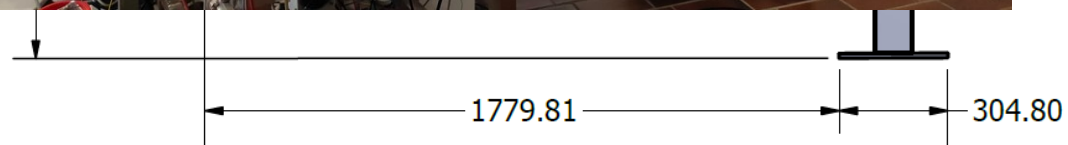
6-axis goniometer

- ✓ JSI – 6-axis goniometer specified, public call, final order at National Electrostatic Corp. (NEC) 08/2021. Delivery scheduled for June 2022, delivery of last missing parts on 22nd January 2024!







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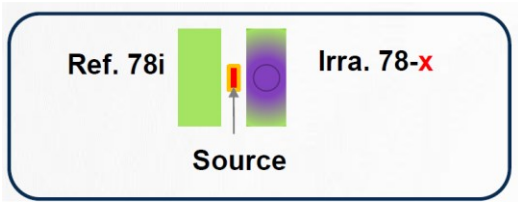
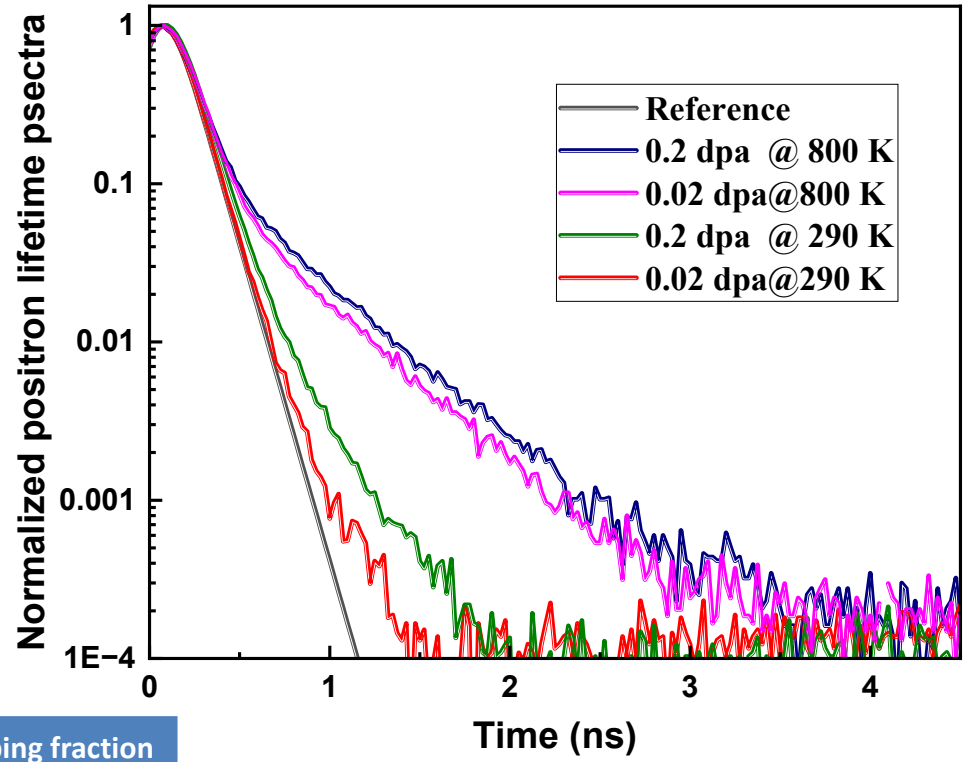
Positron annihilation lifetime spectroscopy

Samples	Dose (dpa)	Temperature
78d	0.02	290 K
78a	0.2	290 K
78h	0.02	800 K
78e	0.2	800 K
78i	0	Reference





Lifetimes for irradiated samples

The average positron lifetime in the non-irradiated sample has been determined as 108 ps;

- **Excellent crystalline quality**

Sample irradiation conditions	τ_{ave} (ps)	τ_1 (ps)	τ_2 (ps)	I_2 (%)	Trapping fraction (%)
2325K_ref.	108.4	108	--	--	--
0.02dpa_290K	116.4	110 (2)	224 (30)	5.2 (2.5)	7
0.2dpa_290K	135.2	114 (1)	265 (8)	14.2 (1.3)	17
0.02dpa_800K	168.9	102 (1)	429 (4)	20.4 (0.4)	19
0.2dpa_800K	192.6	106 (1)	453 (3)	24.9 (0.3)	24

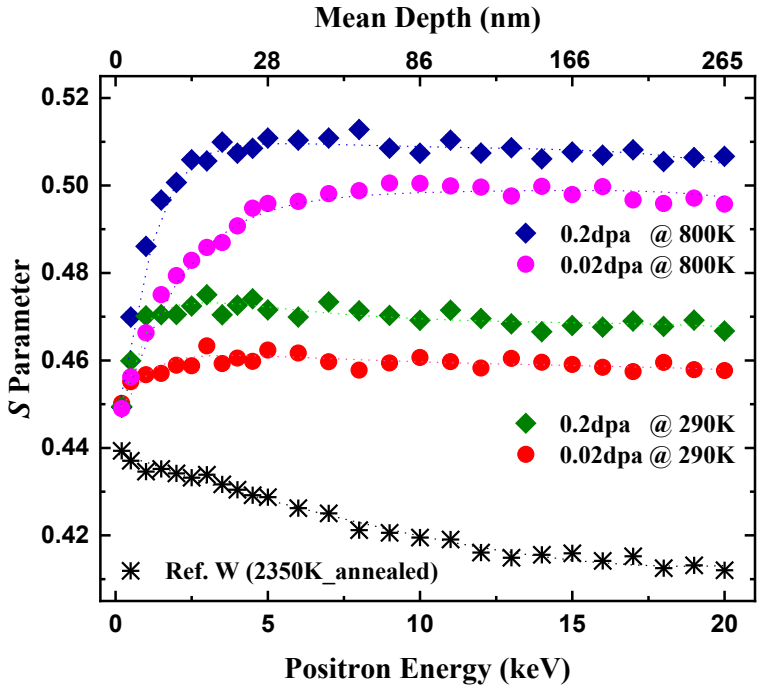
V>1,
V_2-V_4

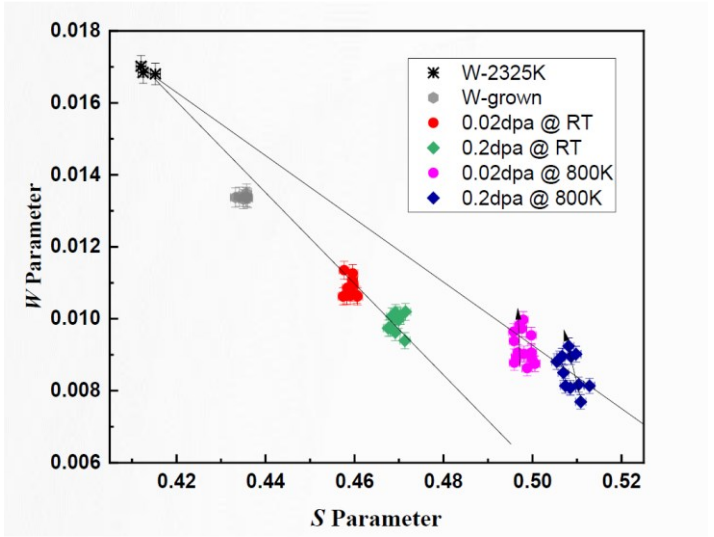
> V_{25}

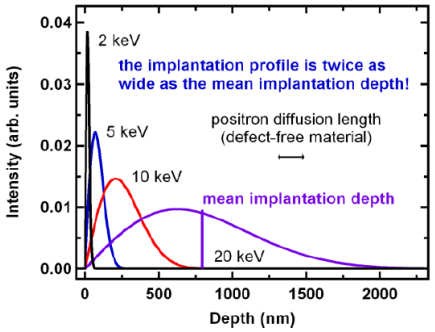
Single vacancy 160-200 ps
[Yang 2022, Heikinheimo 2019]

Positron annihilation - Doppler broadening

- The DBS measurements of positron annihilation radiation were performed on the same single crystal samples with a variable monoenergetic slow positron beam at the Institute of High Energy Physics, Chinese Academy of Science, China (IHEP).
- Positron implantation energies varied from 0.2 to 20 keV, corresponding to a mean penetration depth up to 0.27 μm (maximum information depth ~ 0.5 μm). The line-shape parameters, S and W, are integrated from the Doppler spectrum.







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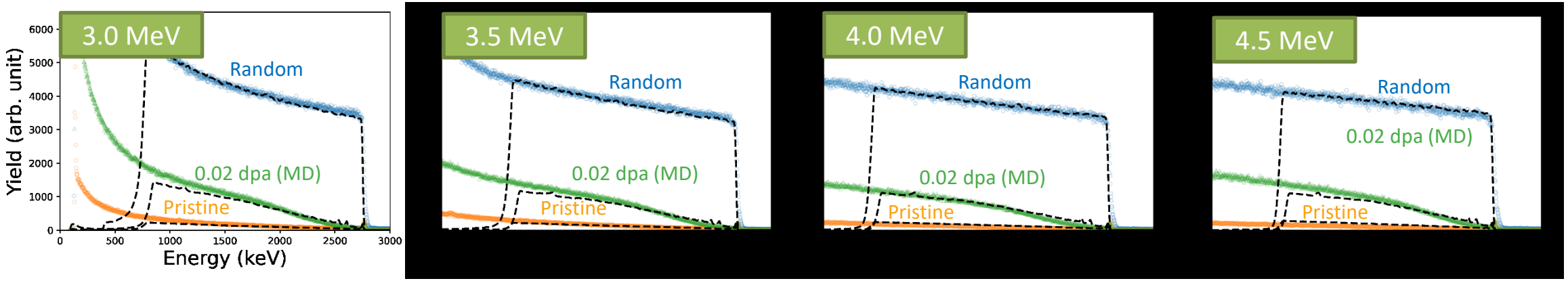
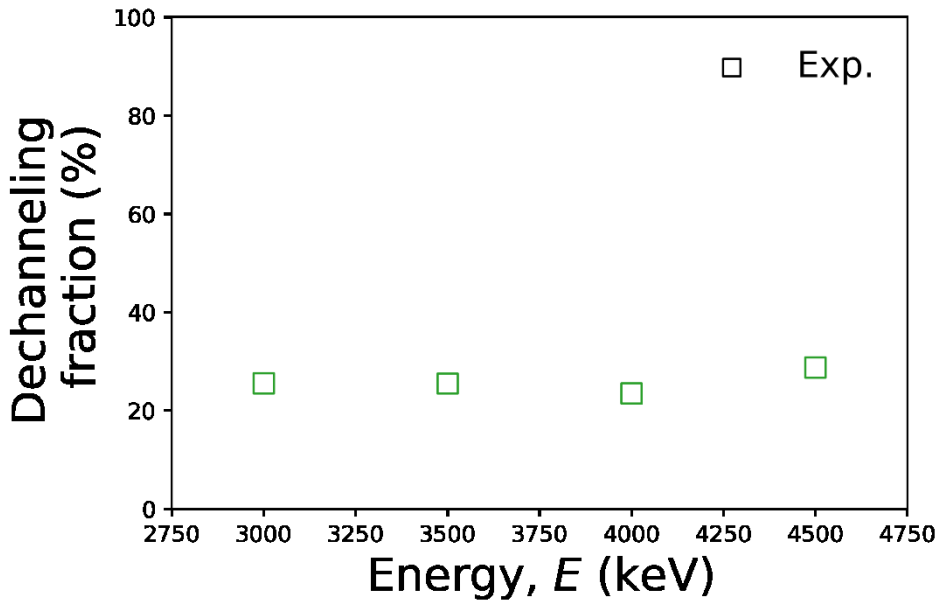
Multi-energy RBS-C (0.02 dpa, 290 K)



Step A. creation of radiation defects – Molecular dynamics simulation
Collision cascade
Primary knock-on atom: 10 keV
Size of MD cells: ~20nm
Overlapping of cascades: high damage dose (evolution of defects)
[F. Granberg et al., J. Nucl. Mater. 556 (2021) 153158]

Step B. Merge of MD cells – input for RBSADEC
Stack 70 MD cells along the [111] direction (the direction of depth)
The depth distribution of cascade number in the MD cells
Merge to one big cell (1.5 µm)

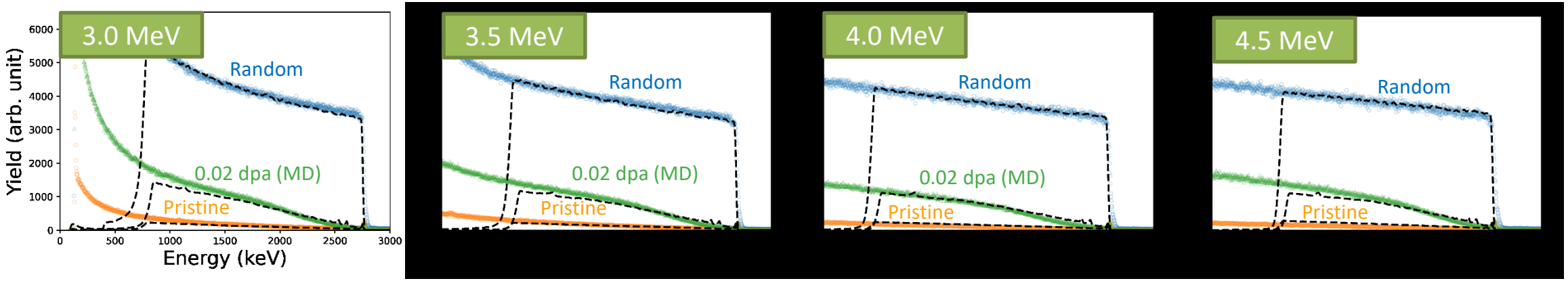


(Dechanneling calculated at $1.2 - 1.3\ \mu m$)

❑ **Dechanneling (energy)**

- **Experiments: no obvious trend**

Multi-energy RBS-C (0.02 dpa, 290 K)



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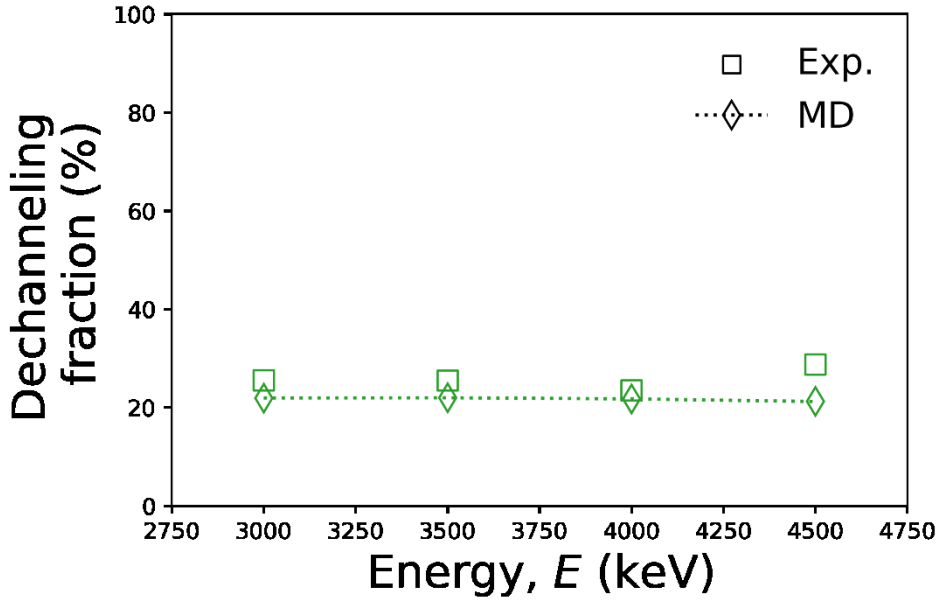

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- **Simulations (MD): constant**

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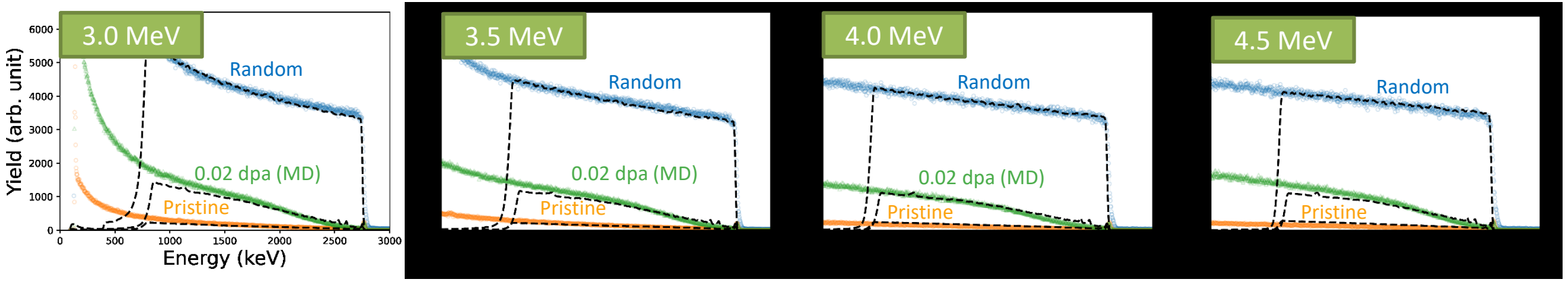

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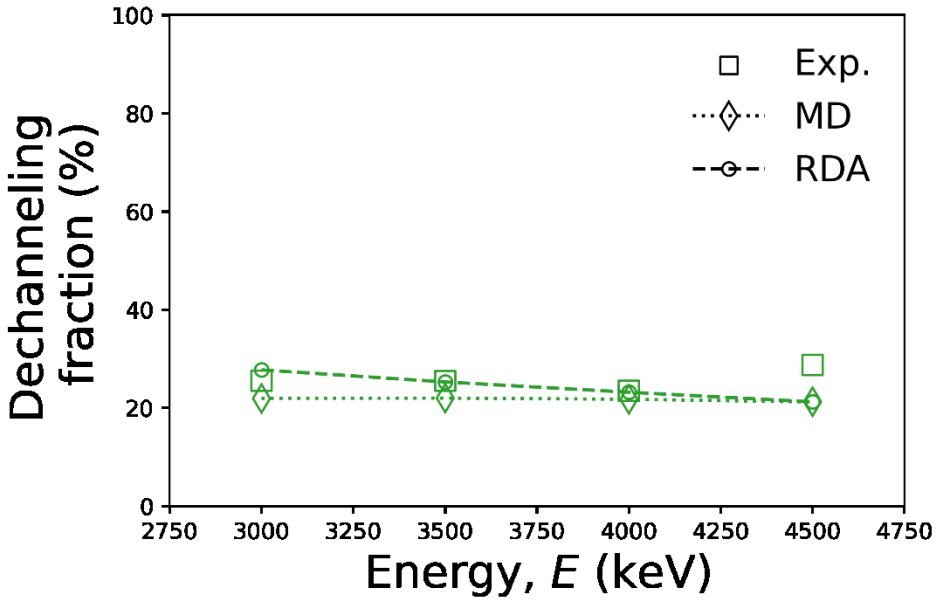


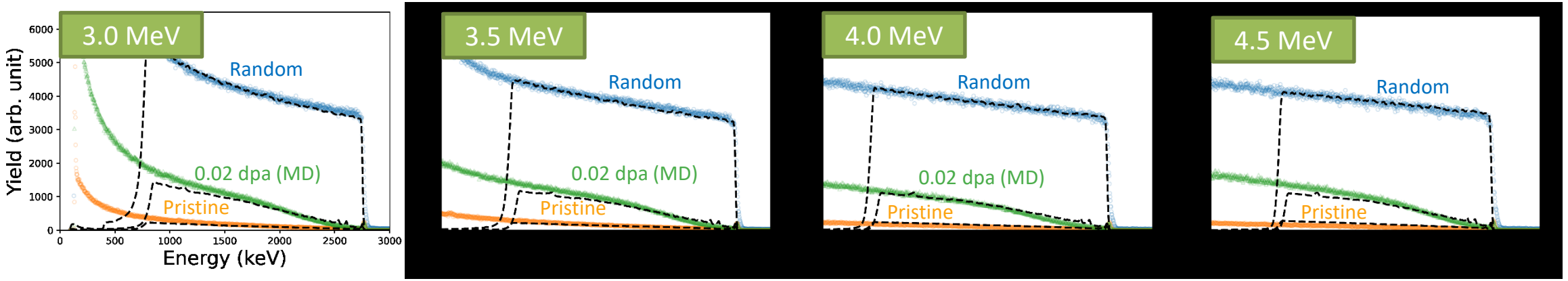
(Dechanneling calculated at $1.2 - 1.3\ \mu m$)

❑ **Dechanneling (energy)**

- **Experiments: no obvious trend**
- **Simulations (MD): constant**
- Simulations (RDA): negative slope

Defect nature: dislocation loops

Multi-energy RBS-C (0.02 dpa, 290 K)



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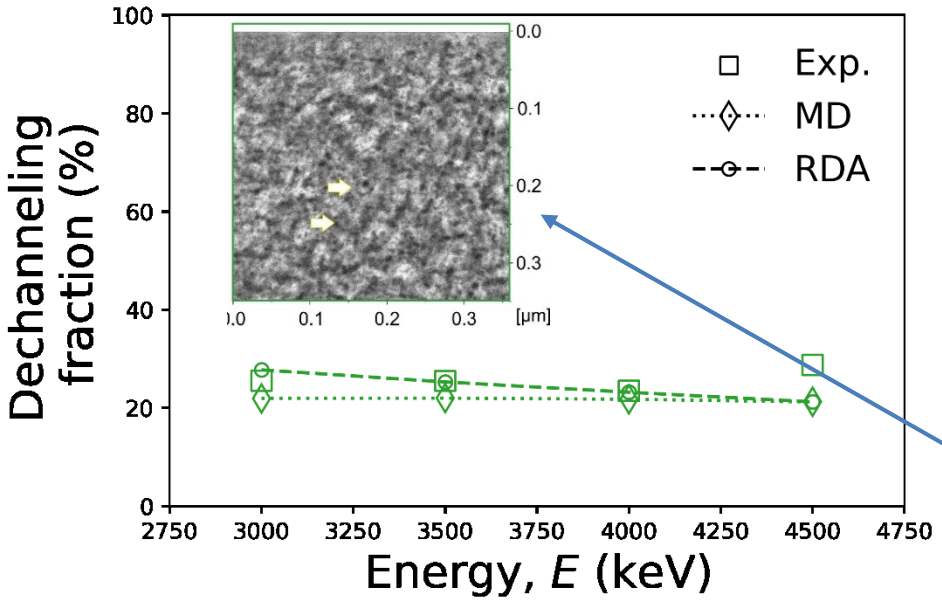


(Dechanneling calculated at $1.2 - 1.3\ \mu m$)

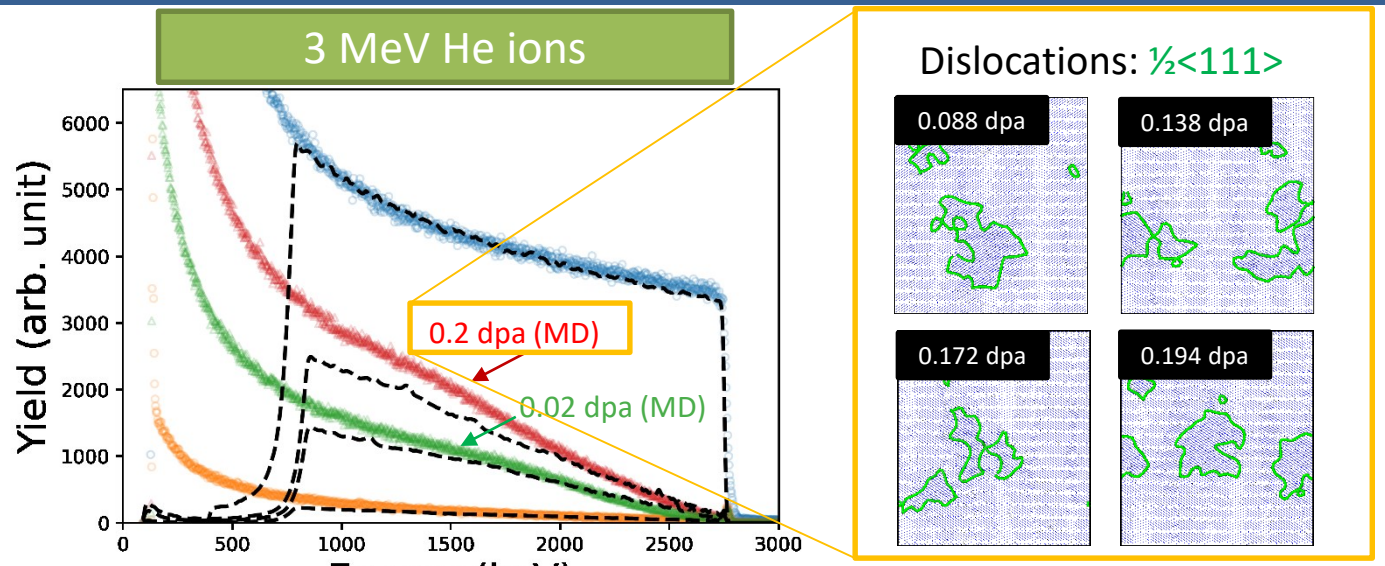
❑ **Dechanneling (energy)**

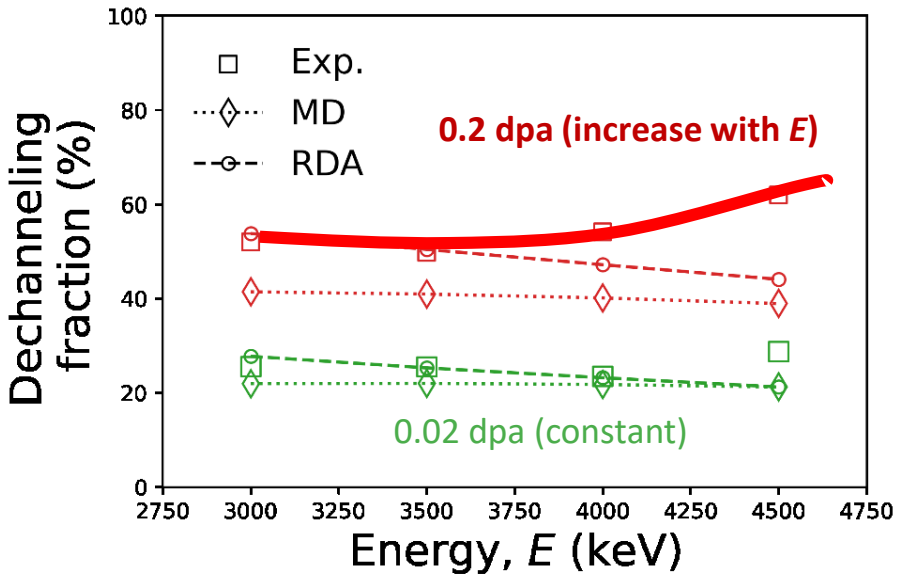
- **Experiments: no obvious trend**
- **Simulations (MD): constant**
- Simulations (RDA): negative slope

Defect nature: dislocation loops

- **STEM results:**
  U-shaped dislocation loops around "black dots" (size ~ 10 nm).

Multi-energy RBS-C (0.2 dpa, 290 K)



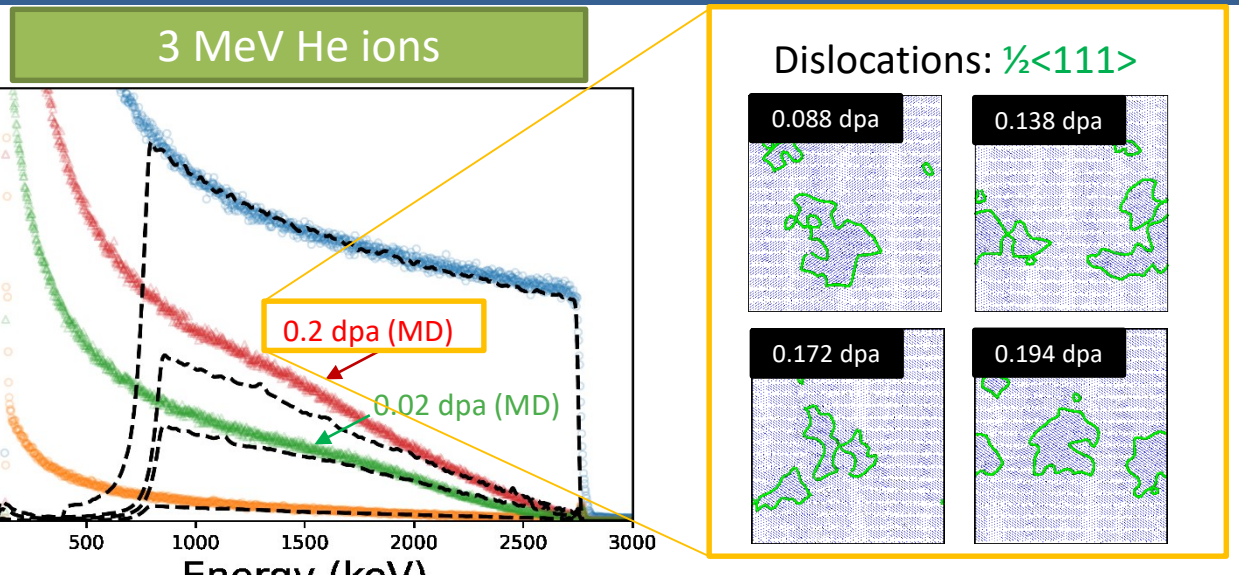


- **RBS/c experiments (high damage, 0.2 dpa)**
- Higher yield than that of 0.02 dpa
- **RBS/c simulations (MD cells)**
- **Dechanneling as a function of *E***

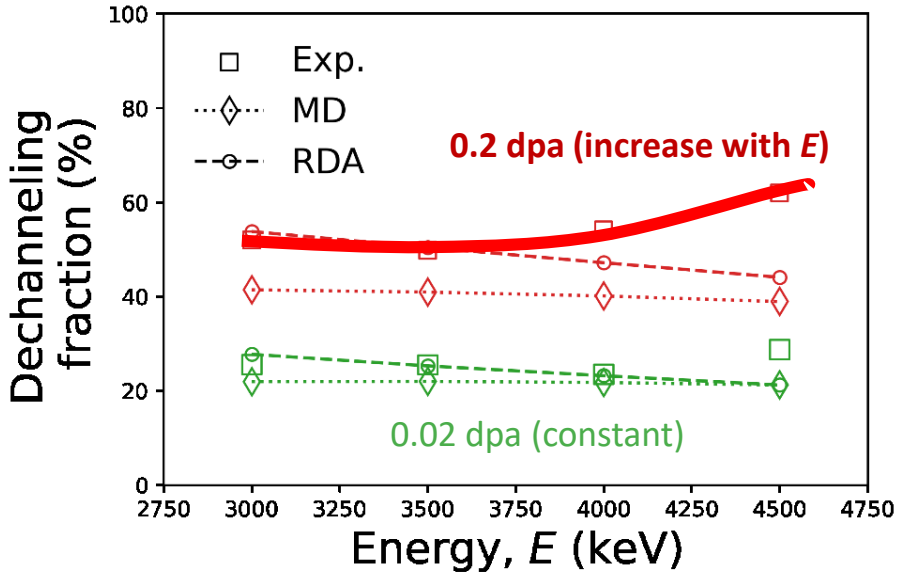
	MD	Exp.	RDA
Dechan.	Constant	Increases with E	Decrease
Defect nature	Dislocation loops		

(S. Markelj, et al., Acta Mater., 263 (2024) 119499)

Multi-energy RBS-C (0.2 dpa, 290 K)

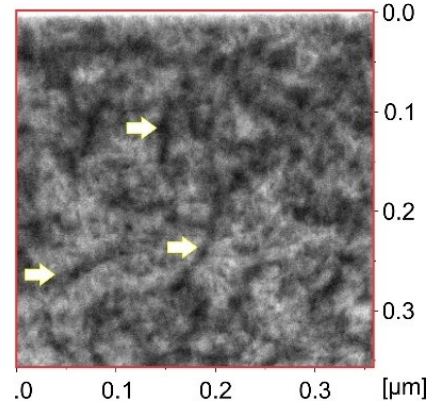


Lacking agreement between experiment and simulation due to limited size of MD cells – work in progress



- **RBS/c experiments (high damage, 0.2 dpa)**
  - Higher yield than that of 0.02 dpa
- **RBS/c simulations (MD cells)**
  - **Dechanneling as a function of *E***

	MD	Exp.	RDA
Dechan.	Constant	Increases with E	Decrease
Defect nature	Dislocation loops	Dislocation lines	
Strain field	$1/r^3$	$1/r$	



- **STEM results:** Dense networks of dislocation lines (length > 100 nm).
- No lines in MD cells: limited size of cells

(S. Markelj, et al., Acta Mater., 263 (2024) 119499)

Publication summarizing the work on tungsten-irradiated W (111) at RT

Acta Materialia 263 (2024) 119499

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Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Unveiling the radiation-induced defect production and damage evolution in tungsten using multi-energy Rutherford backscattering spectroscopy in channeling configuration

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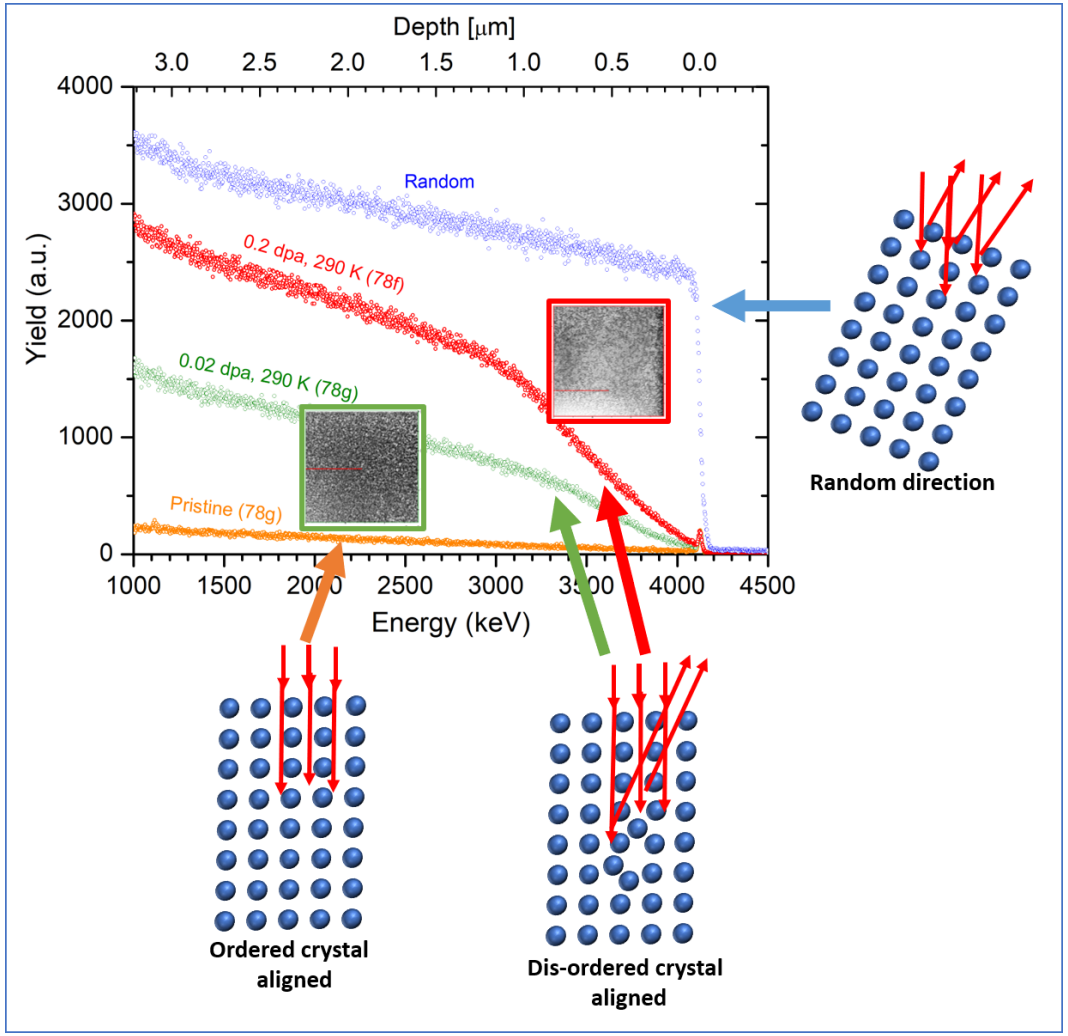
ARTICLE INFO

Keywords:
Tungsten
Defects
C-RBS
Displacement damage
Molecular dynamic simulations
TEM

ABSTRACT

Radiation-induced defect production in tungsten was studied by a combination of experimental and simulation methods. The analysis of structural defects was performed using multi-energy Rutherford backscattering spectroscopy in channeling configuration (multi-energy C-RBS). To create different microstructures, (111) tungsten (W) single crystals were irradiated with W ions at two different doses (0.02 and 0.2 dpa) at 290 K. Detailed transmission electron microscopy (TEM) analysis of the samples revealed the presence of dislocation lines and loops of different sizes. The RBSADEC code was used to simulate the measured C-RBS spectra, recorded with four different He beam energies along the ⟨111⟩ direction. For the first time for tungsten, molecular dynamics (MD) simulations of overlapping cascades were used as input. The well-known method of randomly displaced atoms (RDA) was applied for comparison. RDA does not provide a satisfactory understanding of the nature of the induced defect structure. With MD, a very good agreement between the simulated and experimental spectra was obtained for the sample prepared at a lower dose, despite the fact that the absolute defect densities are two orders of magnitude higher than those found with TEM. A discrepancy is observed for the high-dose-irradiated sample, which is ascribed to the presence of extended defects such as dislocation lines, which are clearly observed by TEM, but cannot be formed in finite size MD cells. RBSADEC with MD cells as input can describe correctly the response of the RBS signal with analysing beam energy while RDA as input gives the wrong trend.



Tasks and objectives in 2023

- Task 1.1 Incorporation of the goniometer in the INSIBA experimental station – JSI.
- Task 1.2 Detection system for ion beam methods
  - **O3.1 (Task 1.1)) C-RBS spectra obtained with new channeling set-up. (D1)**
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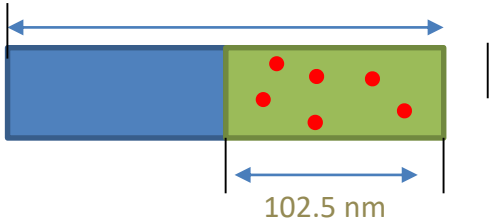
- Task 2.3 Simulation and interpretation of C-RBS spectra - UHEL, CEA, JSI.
  - **O3.2 (Task 2.2, 2.3, 3.1) Defect identification by C-RBS and correlation to TEM and PAS measurements – report. (D2)**
- Task 2.4 In-situ C-RBS and sample heating – JSI
  - **O3.3 (Task 2.4) In-situ sample heating in INSIBA-C. (M6)**
- Task 3.1 Characterization of defects by D retention studies and MRE modelling - JSI, MPG, CEA, UHEL.
- Task 3.2 Development of C-NRA method - JSI, UHEL, MPG.
  - **O3.4 (Task 3.1 and Task 3.2) Detection of deuterium by C-NRA method. (M8)**
- Task 3.3 – Modelling of deuterium position in lattice/defect and identification of D position - UHEL, CEA, JSI.
  - **O3.5 (Task 3.3) Incorporation of C-NRA in RBSADEC. (M9)**

Incorporation of NRA-C into RBSADEC code

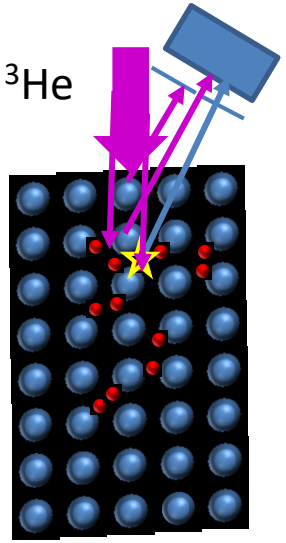
❑ Incorporation of NRA-C into RBSADEC

- RBS-C: a pristine W target
- NRA-C:

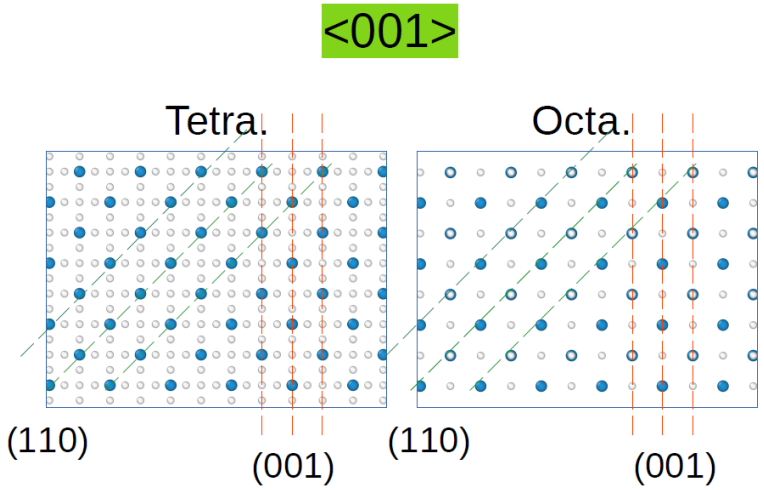


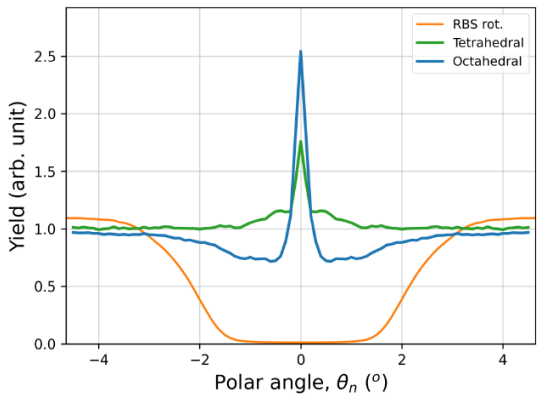
- 0.1 % of D at tetrahedral sites

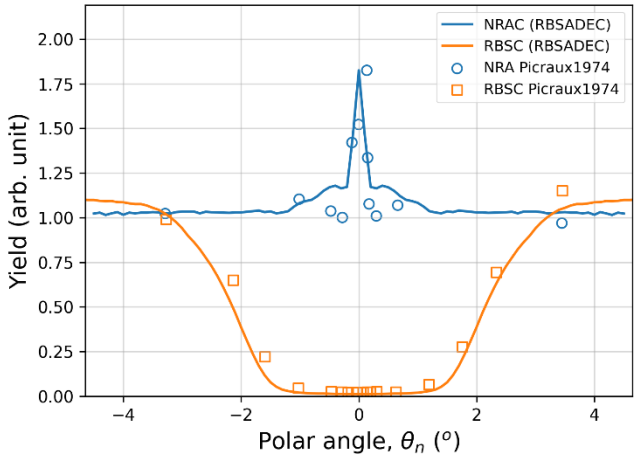
750 keV ^{3}He



Angular scan







S. Picraux, Phys. Rev. Lett., 33, 1974)

3×10^{15} cm^{-2} 30 keV D on W

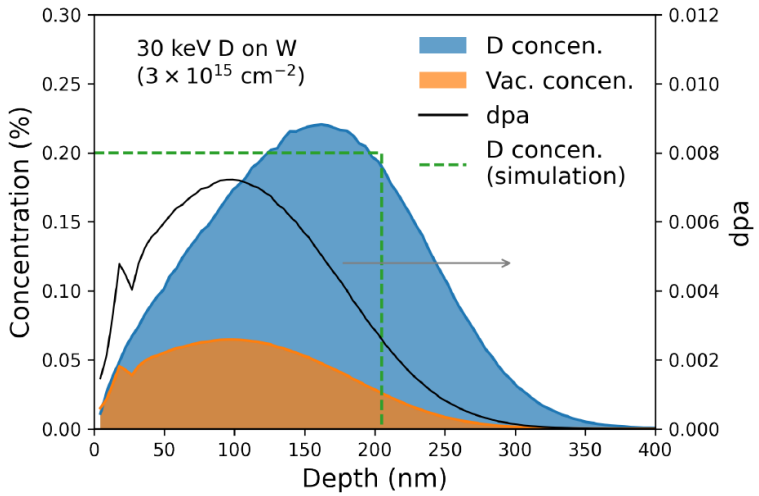
Difference between experiments and simulations: not exactly on tetrahedral sites? – Vacancies, some position close to tetrahedral sites, etc.

- Effect of damage dose: D sites change
- ➢ Improving the fit: adjust D locations, D location according to DFT calculations

Development of C-NRA simulation and detection of D by RBSADEC code

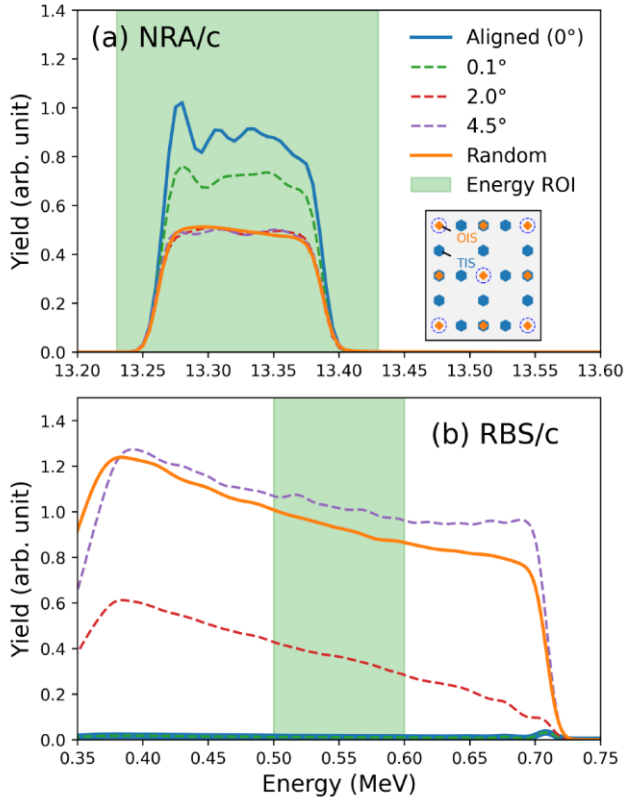
- SRIM calculation of vacancy distribution for 30 keV D ions in W for Picraux experiment

(S. Picraux, Phys. Rev. Lett., 33, 1974)

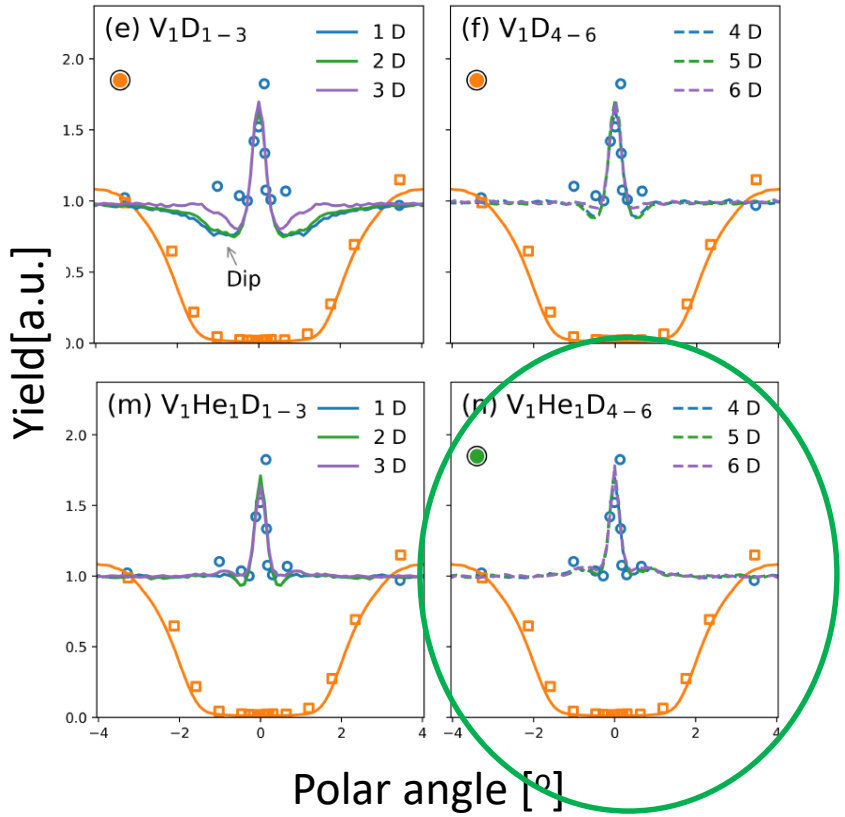


- Creation of vacancies
- Multiple hydrogen occupancy in a vacancy [Heinola et al. PR B (2010), Fernandez et al. Acta Materialia (2015)]
- Hydrogen occupancy for exp. conditions calculated by MHIMS

- **Calculation the NRA and RBS yield**



- **Best agreement obtained for He-field vacancy with 5 H atoms**



[Jin et al. submitted to PRM.]

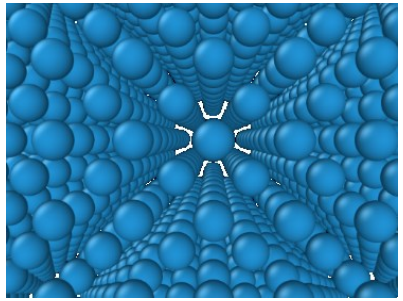
Sample preparation - Tungsten single crystals

- Samples: NRA-C tungsten single crystals (100)
- Irradiation: 10.8 MeV W ions
- **Two damage doses:**

Low ($5.8\times10^{16} m^{-2}$) & High($5.8\times10^{17} m^{-2}$)

SRIM-KP

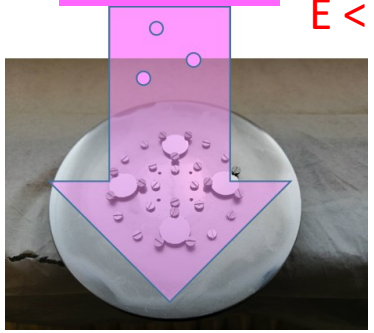
0.02 dpa **0.2 dpa**



W (100)

D plasma

T = 370 K,
E < 5 eV/D



Two temperatures:
290 K & 800 K

Single vacancies @ low dose

Vacancy clusters Diff. sizes

'gentle' loading = 'decoration' of defects
ion flux: 6×10^{19} D/(m^2s)
ion fluence: $1\cdot10^{25}$ D/m^2 (48 h)

- W (100) single crystal should show a difference in the C-NRA signal when D is near the octahedral (OIS) or tetrahedral interstitial sites (TIS) [Cabstanjen, H.-D. Phys. Stat. Sol. (a) 59, 11–26 (1980)].

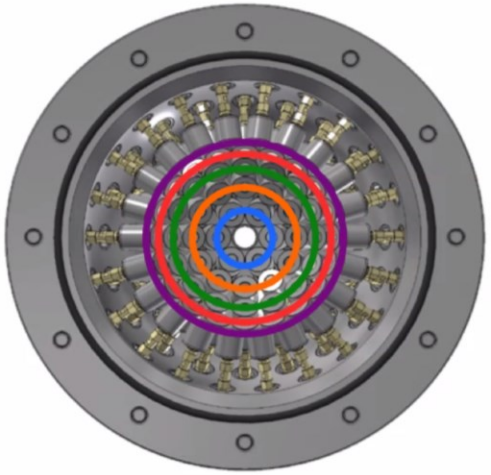
Based on Hu et al. JNM 556 (2022) 153175 – open volume type defects

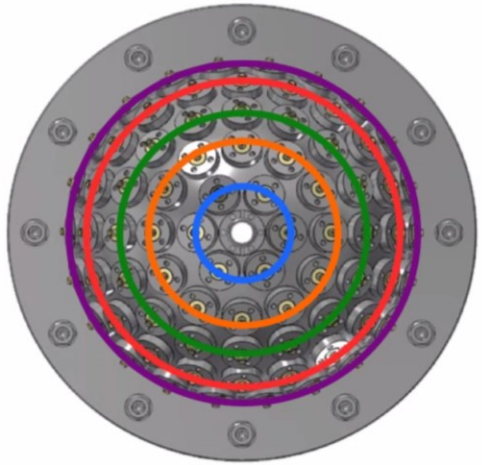
Sample	Irradiation conditions	Predominant defect expected
78g / #1	0.02 dpa, 290 K	single vacancies
78f / #5	0.2 dpa, 290K	heavily damaged standard
78c / #3	0.02 dpa, 800 K	small vacancy clusters
78b / #2	0.2 dpa, 800 K	big vacancy clusters

Channeling measurements at HZDR – The Hedgehog system

Multi Detector RBS Setup

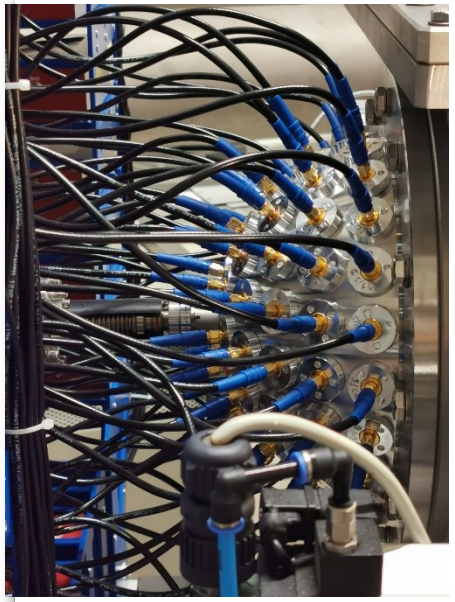
- Sensitive samples / insulating samples
- Higher sensitivity for trace elements
- Lateral mapping
- Channeling scans and maps
- Multiple backscattering angles to reduce the ambiguity of RBS

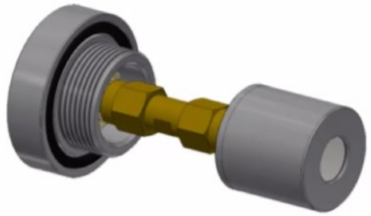




6x	165° =	53.8 msr
12x	150° =	107.6 msr
16x	135° =	143.5 msr
20x	120° =	179.3 msr
22x	105° =	197.3 msr

Ω = 681.4 msr
9.2% of half sphere





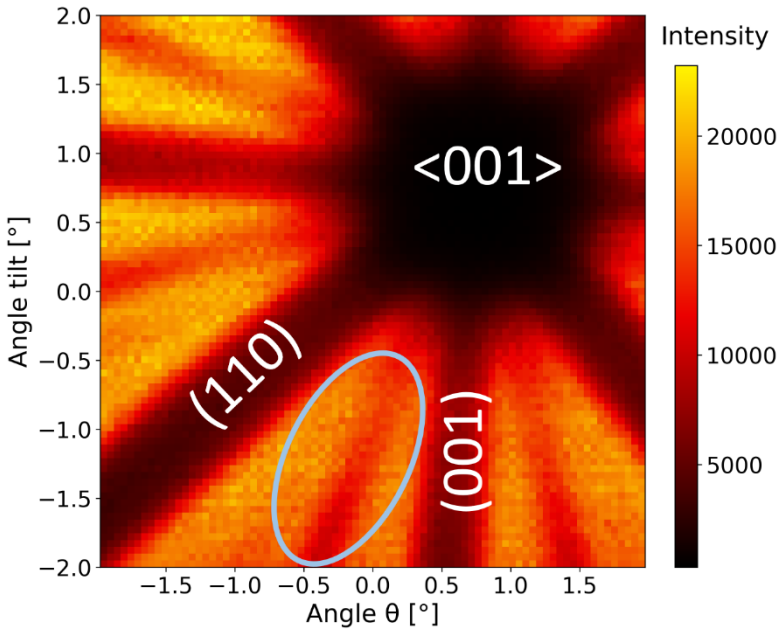
- 200 PIPS **detectors fabricated @IBC**

HZDR

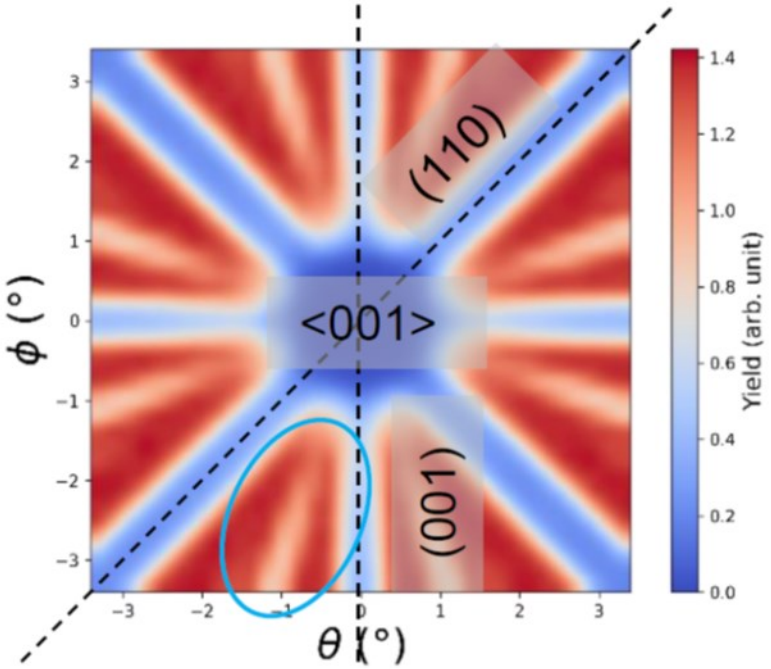
RBS-C measurements (2D maps)

^{4}He 2.5 MeV RBS-C 2D maps / search for the channel

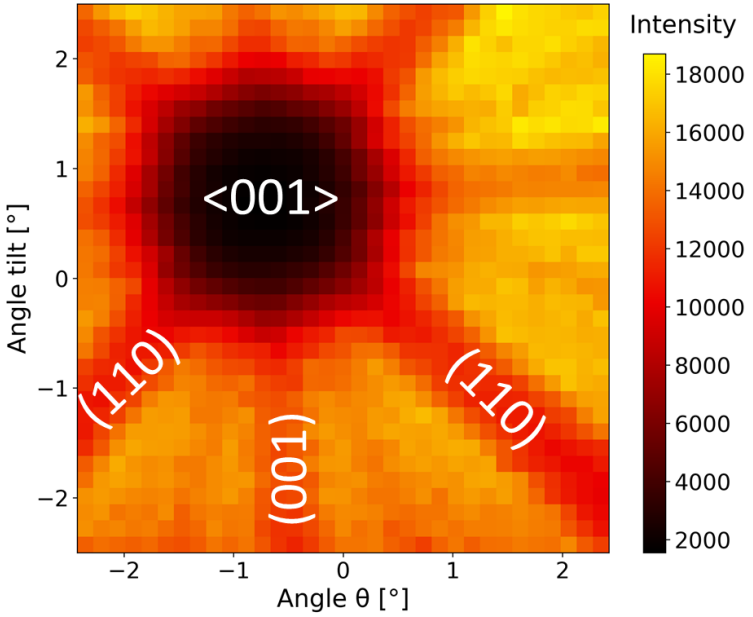
#4 - SC W (100) - Reference



Simulation – identification of channels



#5: 290 K, 0.2 dpa

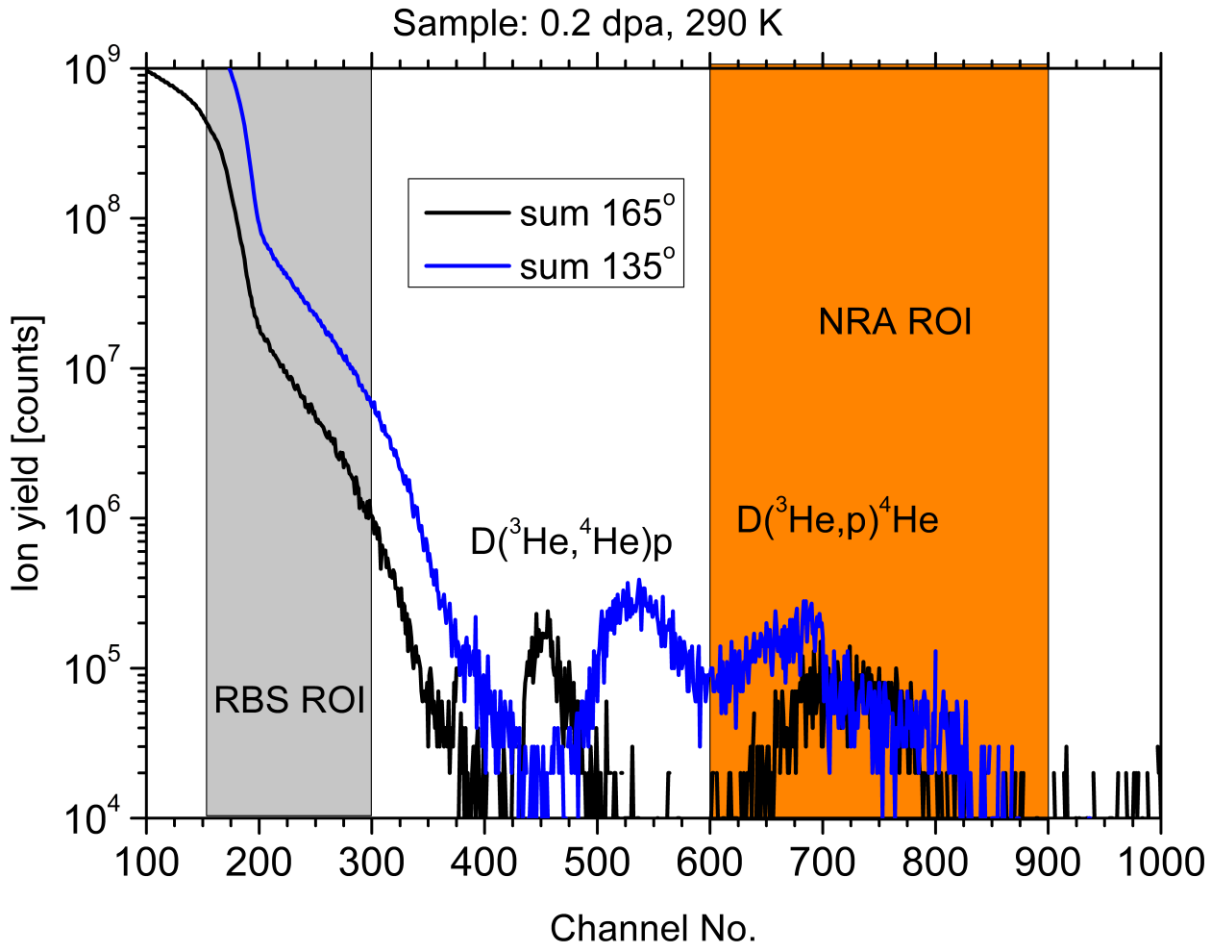


Channels not well visible anymore for high irradiation dose

RBS-C was performed at the Hedgehog setup for RBS channeling at Ion beam center at HZDR.

NRA-C and RBS-C: DeHydroC first measurements

^{3}He 0.8 MeV – simultaneous RBS-C and NRA-C 2D maps

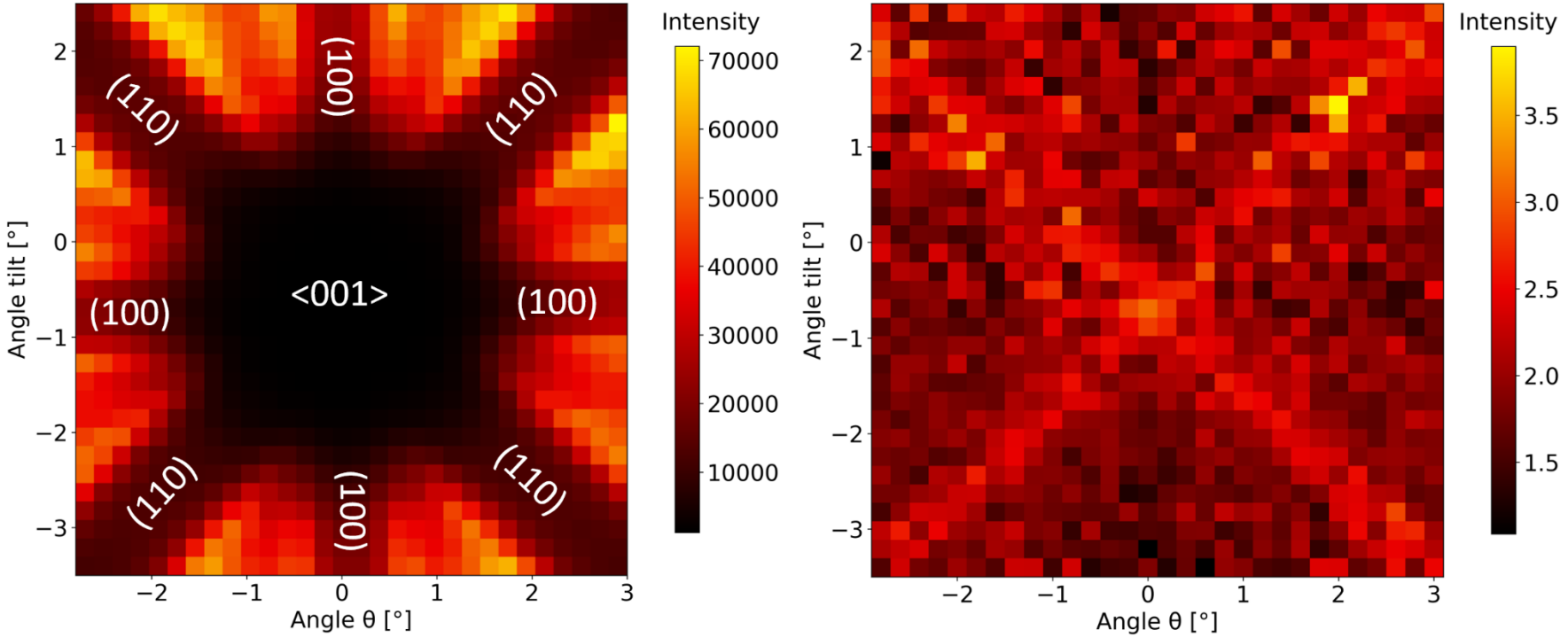


RBS-C was performed at the Hedgehog setup for RBS channeling at Ion Beam Center at HZDR.

NRA-C and RBS-C: DeHydroC first measurements

^{3}He 0.8 MeV – simultaneous RBS-C and NRA-C 2D maps

2D map for sample #2: 800 K, 0.2 dpa



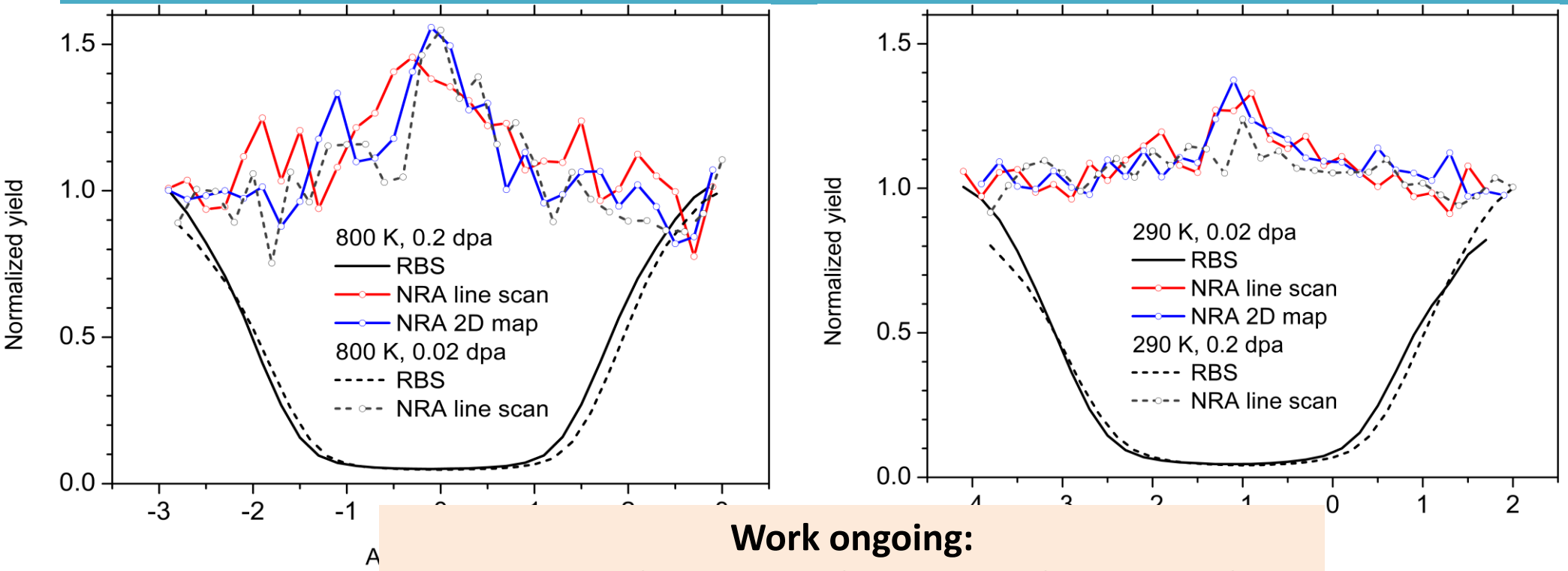
RBS-C

NRA-C

RBS-C was performed at the Hedgehog setup for RBS channeling at Ion Beam Center at HZDR.

NRA-C and RBS-C: angular scans

^{3}He 0.8 MeV – simultaneous RBS-C and NRA-C 2D maps

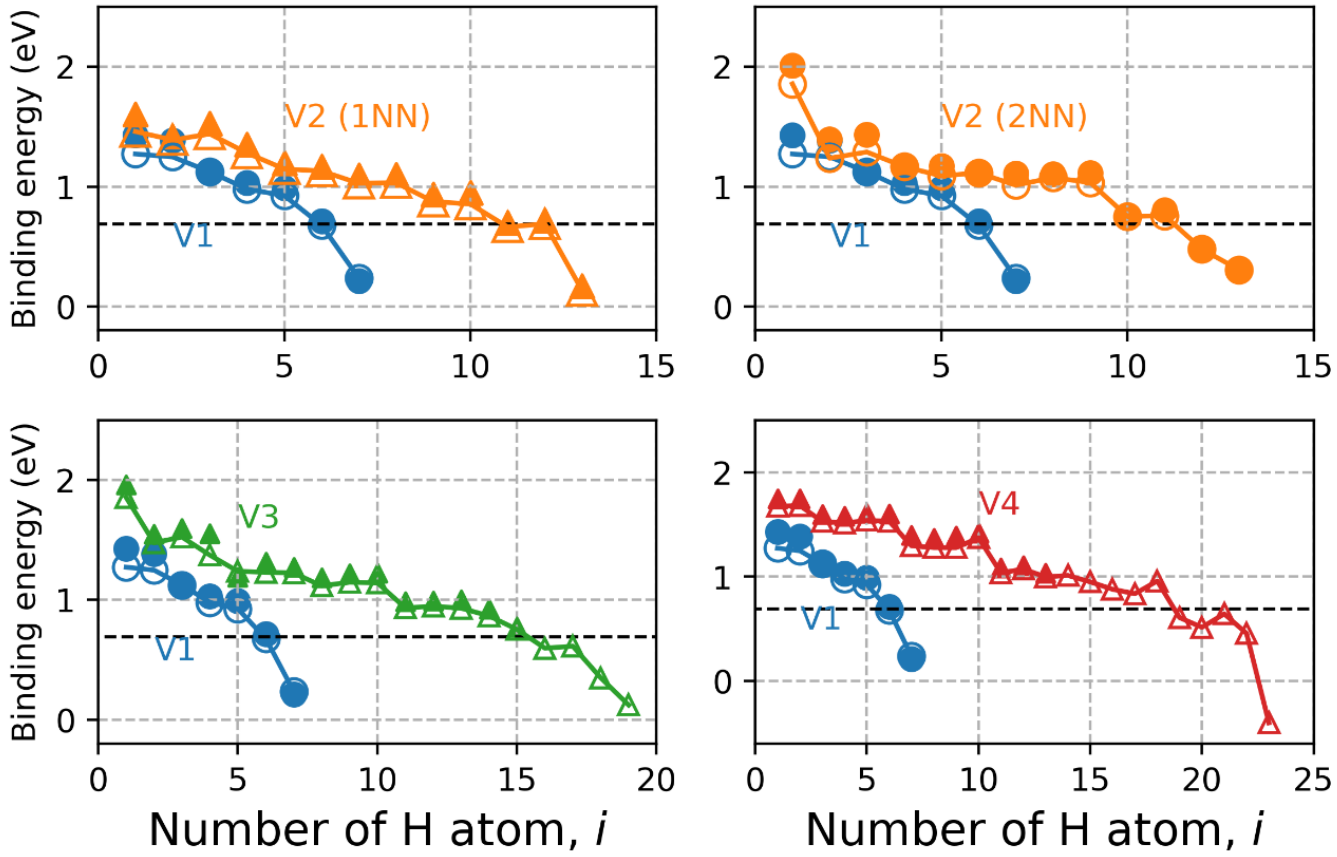


Work ongoing:
NRA-C simulations, combination with DFT - multi occupancy of H in vacancy and vacancy clusters

- The NRA signal peal r NRA signal compared to the 0.02 dpa/290
- Speculation: vacancy clusters are larger in size and the deuterium location in these clusters has a broader distribution, further away from the centre of the cluster.

DFT calculations of vacany clusters

- Binding energy of H to V_nH_{i-1}



(empty and filled markers: energy without and with ZPE)

- DFT calculations

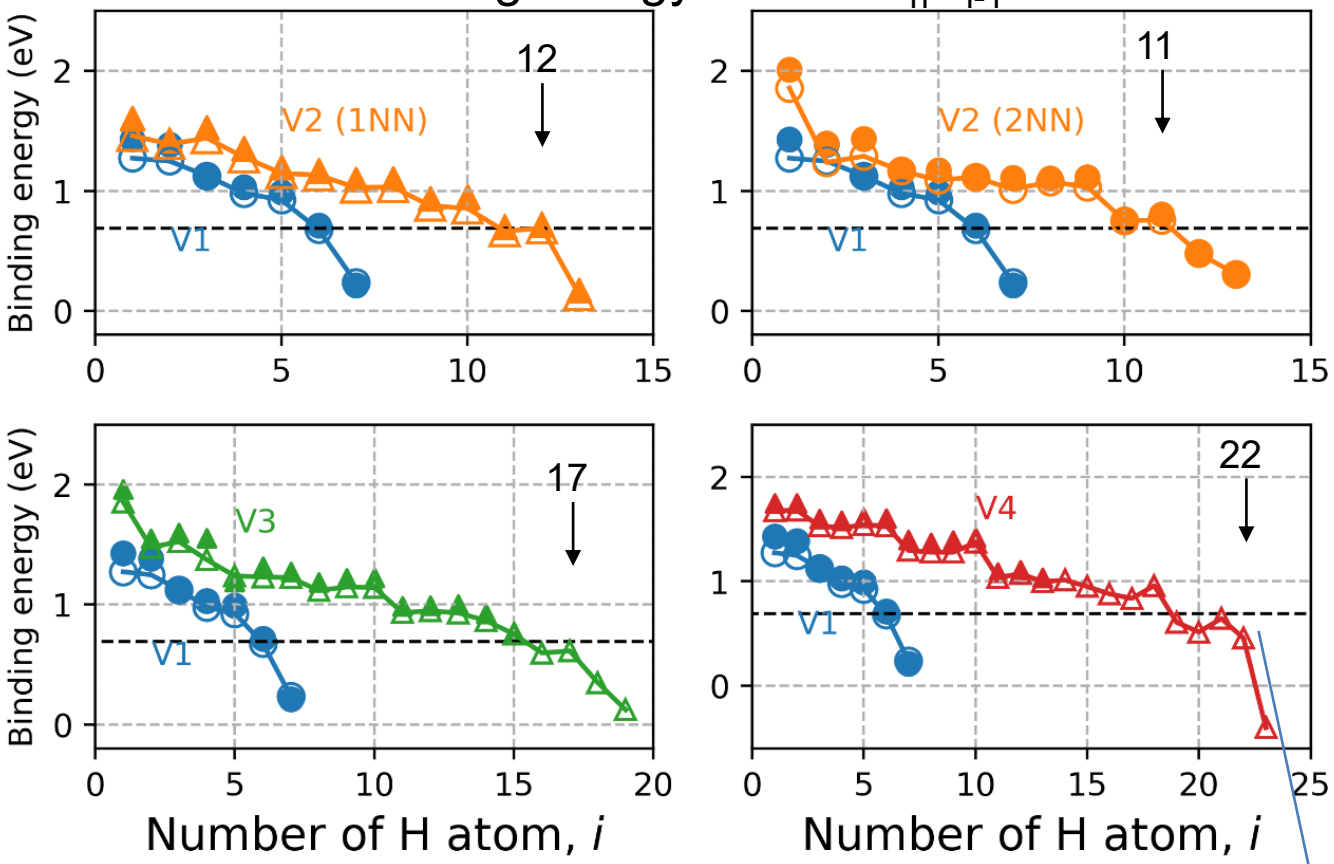
  - VASP
  - 4 * 4 * 4 supercell
  - Cut-off energy: 500 eV
  - Position and volume relaxation

- Estimation of the maximum number of H atoms attaching to the vacancy surface:
- Threshold binding energy of 0.69 eV (black dashed line)

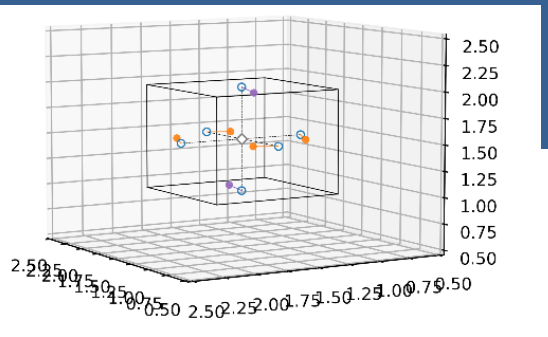
(from N. Fernandez, et al., Acta Mater., 94, 2015)

DFT calculations of vacany clusters

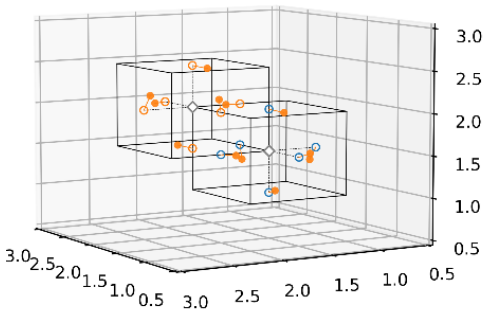
- Binding energy of H to V_nH_{i-1}



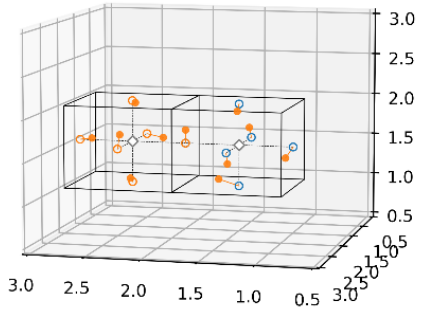
(empty and filled markers: energy without and with ZPE)



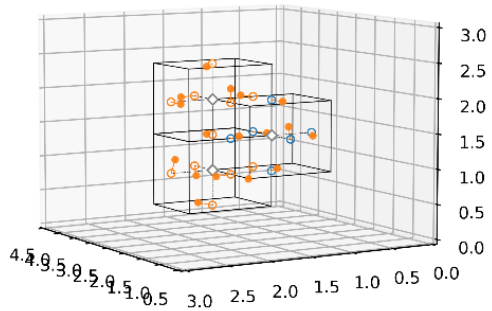
V1: 6 faces



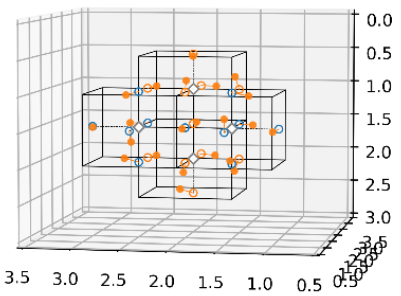
V2 (1NN): 12 faces



V2 (2NN): 11 faces



V3: 17 faces



V4: 22 faces

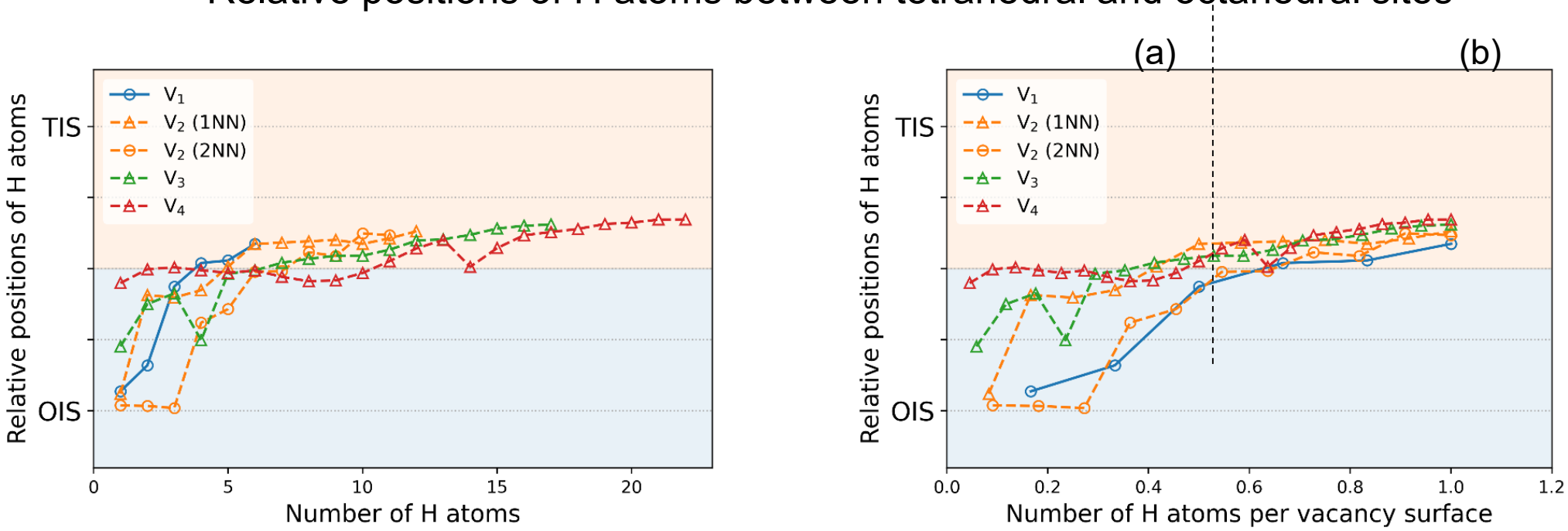
➢ Estimation of the maximum number of H atoms attaching to the vacancy surface: threshold binding energy of 0.69 eV (black dashed line)

(from N. Fernandez, et al., Acta Mater., 94, 2015)

➢ Close relation to the number of vacancy faces

DFT calculations

- Relative positions of H atoms between tetrahedral and octahedral sites



Change of H positions with the number of H atoms per vacancy surface:

(a): < half filled: clear different positions in different vacancy clusters

- Due to different types of vacancy surfaces

(b): > half filled: converge and get similar behavior.

Attendance at the conferences in 2023

PFMC conference:

- E. Punzon-Quijorna et al. "Multi-Energy Rutherford Backscattering Spectroscopy in Channeling configuration for the analysis of defects in tungsten" (poster)
- X. Jin et al. Study of the lattice location of deuterium implanted into tungsten using simulations of nuclear reaction analysis in channeling mode“(poster)

IBA conference:

- S Markelj et al., Analysis of deuterium and defects in tungsten by Rutherford backscattering spectroscopy and nuclear reaction analysis in channeling configuration“ (poster)
- X. Jin et al., Deuterium trapping conditions and potential location sites in tungsten by combination of nuclear reaction analysis in channeling mode with first principle calculations" (poster)
- F. Djurabekova et al., Simulation of Rurgerford Backscattering spectrometry in channeling mode from arbitrary atomistic structures (Invited)

ICFRM conference:

- X. Jin et al., Analysis of radiation effects in tungsten by comparing molecular dynamics simulations to experiments of RBS-Channeling" (contributed talk)
- S Markelj et al.,"Detection of defects and hydrogen by ion beam analysis in channeling mode for fusion - DeHydroC" (poster)

MINES:

- Markelj et al., Analysis of deuterium and defects in tungsten by Rutherford backscattering spectroscopy and nuclear reaction analysis in channeling configuration (Invited talk)

Attendance at the conferences in 2024

PSI conference May 2024:

- Markelj et al. Detection of defects and deuterium in displacement-damaged tungsten by applying Rutherford backscattering spectroscopy and nuclear reaction analysis in channeling configuration
- Hodille et al., Macroscopic modelling of D trapping in self-damaged tungsten with vacancy clusters using atomistic scale modelling data

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On schedule


Delayed

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  - **O3.4 (Task 3.1 and Task 3.2) Detection of deuterium by C-NRA method. (M8)**
- Task 3.3 – Modelling of deuterium position in lattice/defect and identification of D position - UHEL, CEA, JSI.
  - **O3.5 (Task 3.3) Incorporation of C-NRA in RBSADEC. (M9)**

Deliverables in 2024

- Study of C-RBS during sample annealing.
- Paper on microstructure analysis and correlation with HI retention.
- Detection of deuterium in lattice defects and comparison to modelling.

Thank you for your attention

Multi energy C-RBS – experiment

- ^{4}He ions along <111> channel
- Multiple energies of He ions (3 MeV to 4.5 MeV) – **Multi energy RBS-C**

➢ RBS-C spectra @ 4.5 MeV

Irradiation at 290 K:

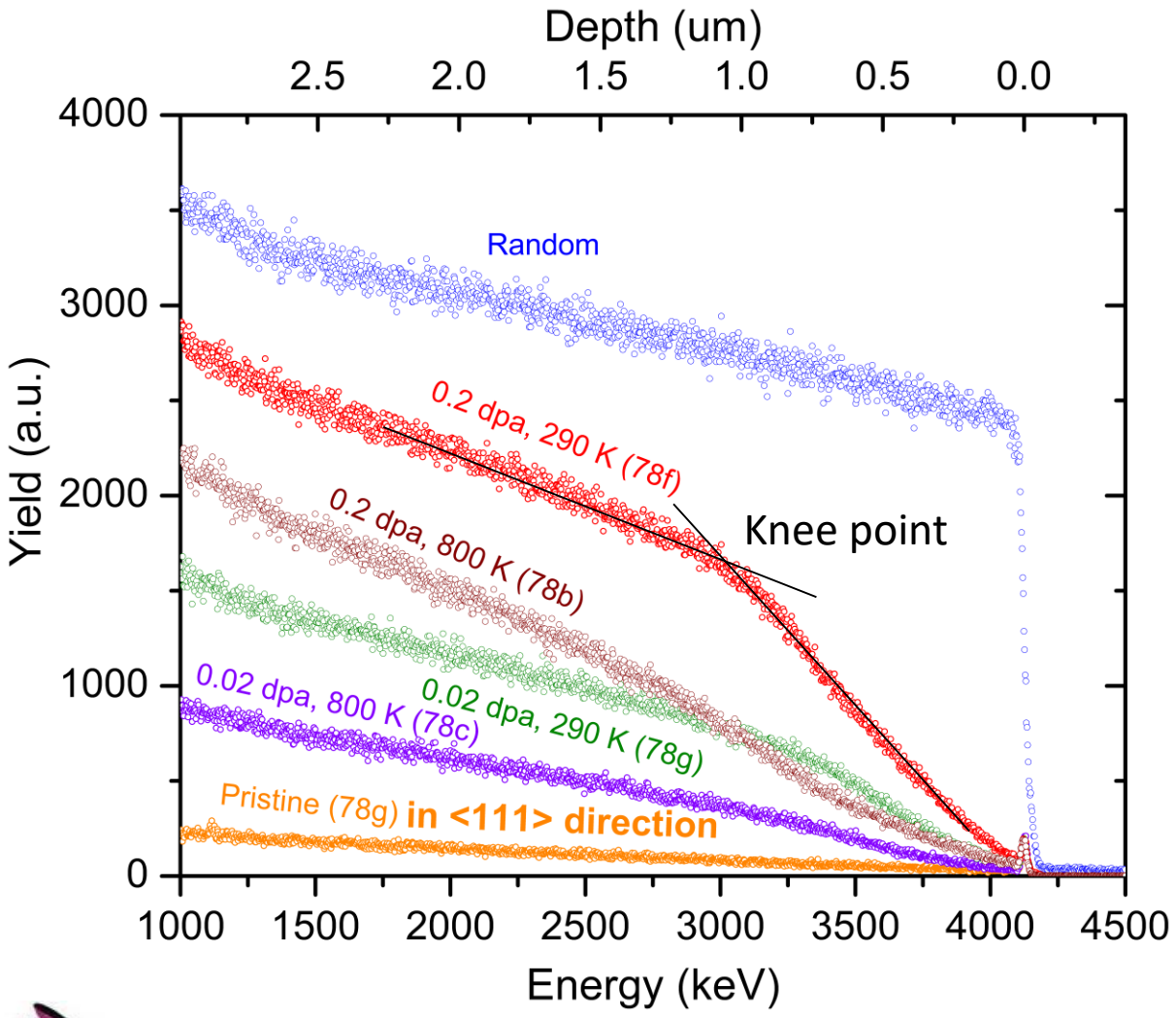
- 78f : 0.2dpa, 290 K (heavily damaged standard)
- 78g : 0.02 dpa, 290 K (single vacancies)

Irradiation at 800 K:

- 78c : 0.02 dpa 800 K (small vacancy clusters)
- 78b : 0.2 dpa 800 K (big vacancy clusters)

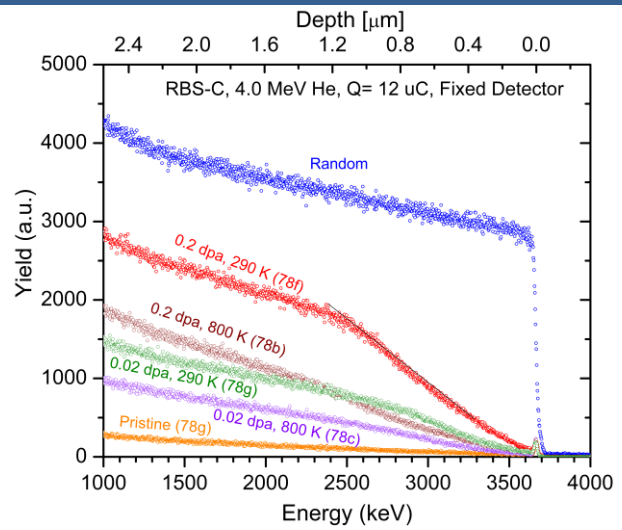
- **Clear differences between the irradiation damage treatments**
- **Knee point indicates damage depth**

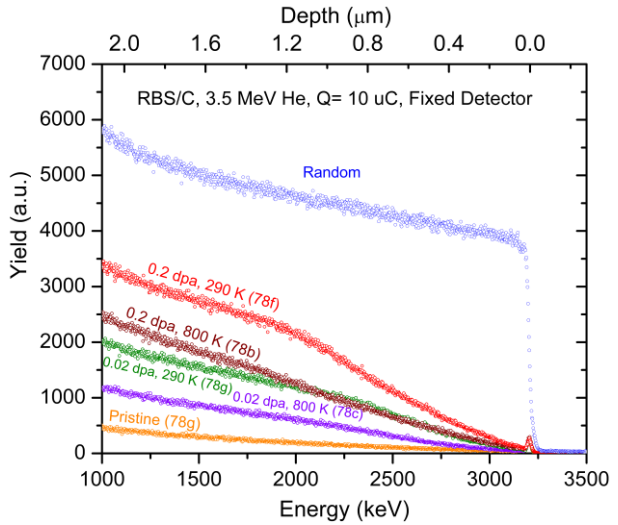
^{4}He beam E=4.5 MeV

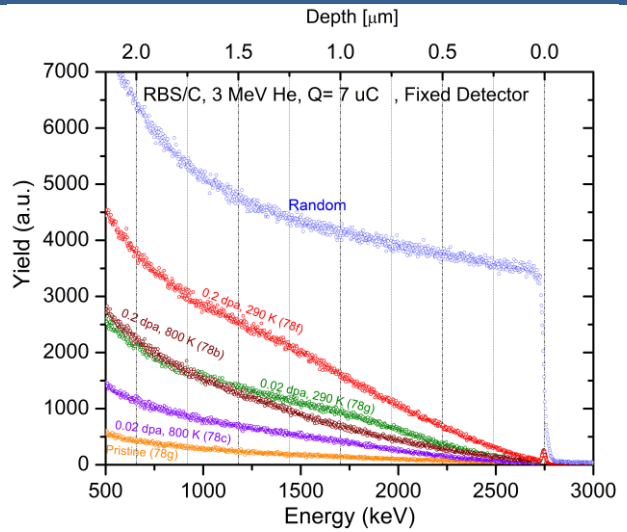


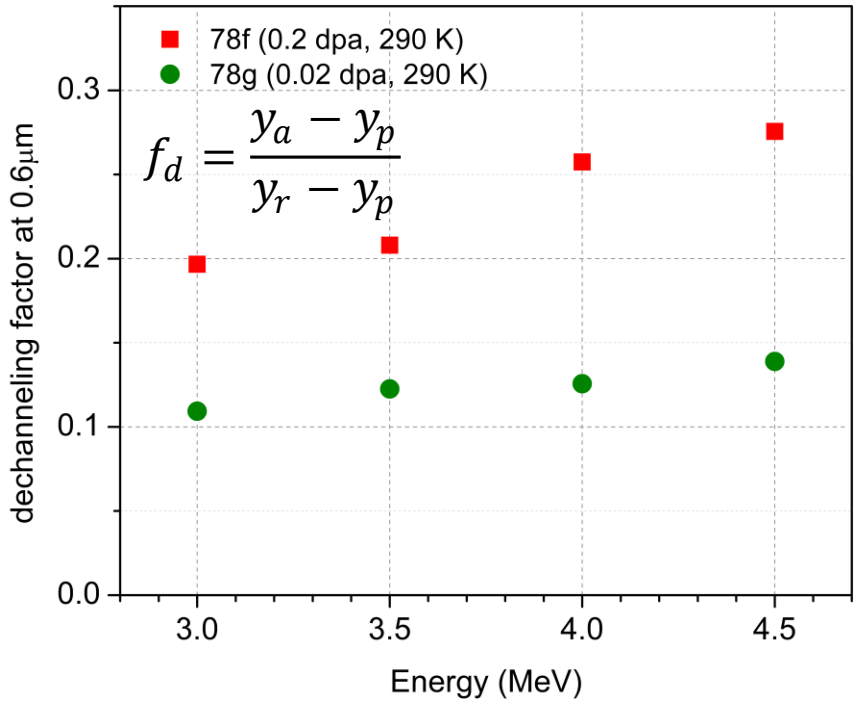
Measurements were preformed at CMAM, Madrid

Multi energy RBS-C









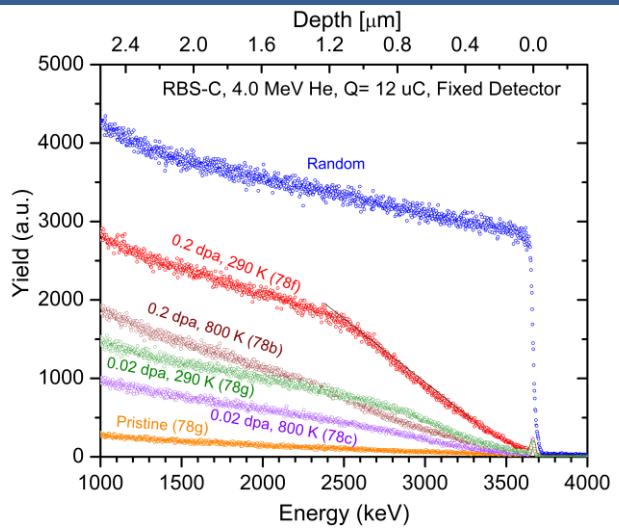
Dechanneling factor as a function of energy at 0.6 um

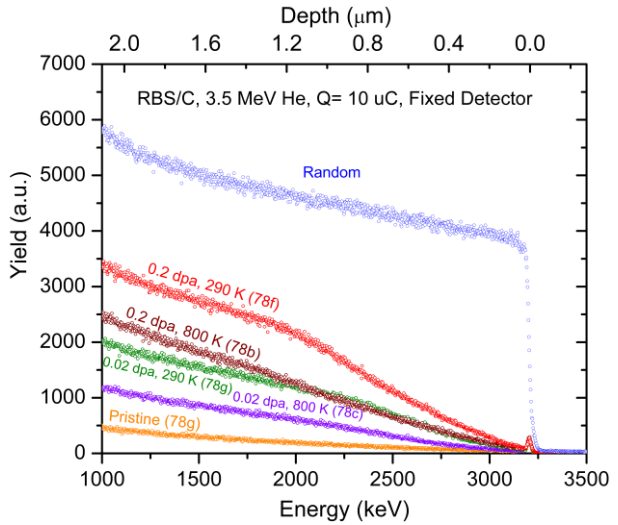
78g (0.02 dpa, 290 K): no slope – discontinuous defects – small dislocation loops (TEM)

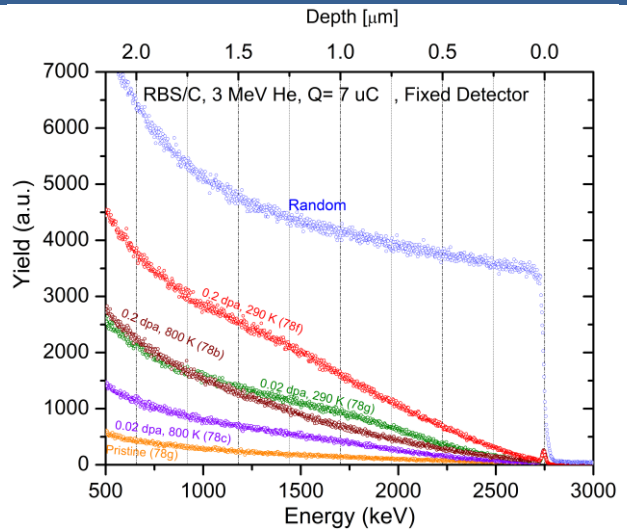
78f (0.2 dpa, 290 K): positive slope –extended defects – dislocation lines (TEM)

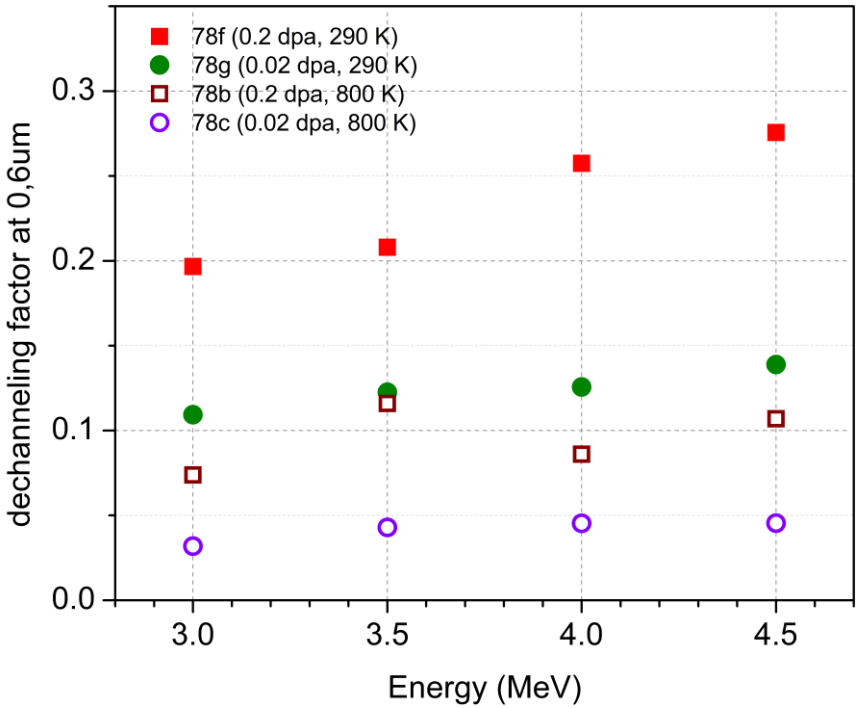
- Following: *FELDMAN, MATERIALS ANALYSIS BY ION CHANNELING. Submicron Crystallography, 1982, Academic press*

Multi energy RBS-C









Dechanneling factor as a function of energy at 0.6 um

78g (0.02 dpa, 290 K): no slope – discontinuous defects – small dislocation loops (TEM)

78f (0.2 dpa, 290 K): positive slope –extended defects – dislocation lines (TEM)

78c (0.02 dpa, 800 K): no slope – discontinuous defects – dots and isolated lines (TEM)

78b (0.2 dpa, 800 K): no slope – discontinuous defects – (dislocation lines and black dots (TEM)

- Following: *FELDMAN, MATERIALS ANALYSIS BY ION CHANNELING. Submicron Crystallography, 1982, Academic press*