Enabling research project, EuroFusion, Materials (CfP-FSD-AWP21-ENR-02): Exhaust and plasma-wall interaction (Material-related aspects)

Detection of defects and hydrogen by ion beam analysis in channelling mode for fusion – DeHydroC

Project code: CfP-FSD-AWP21-ENR-02-JSI-01

Report on research activities in 2021

Sabina Markelj

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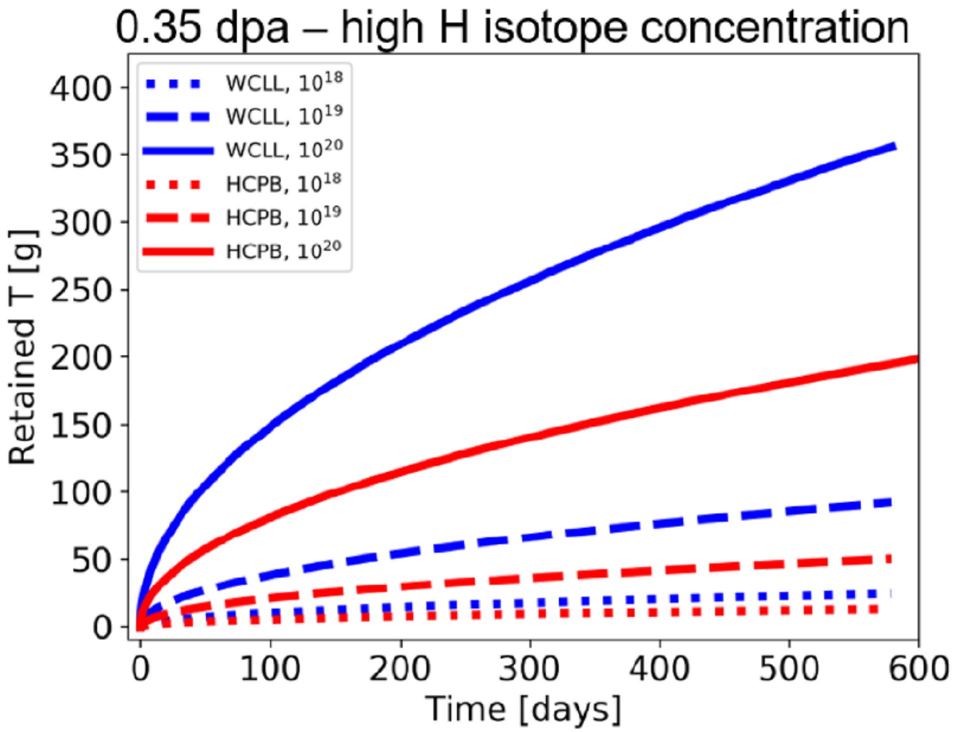
cea

University of Helsinki

Team members

Benificiary	Names	Expertise	Contact	PM - 1.year	PM - 2.year	PM- 3.year	Mission days/y
JSI	Sabina Markelj	HI interaction, sample irradiation, ion beam analysis (IBA)	sabina.markelj@ijs.si	6	6	8	5,8,5
	Esther Punzón Quijorna	Channelling, IBA (Pos-doc)	esther.punzon-quijorna@ijs.si	6	10	10	5,5,5
	Mitja Kelemen	Construction, IBA, channelling (PhD, pos-doc)	mitja.kelemen@ijs.si	7	8	7	8,5,5
	Matjaž Vencelj	Detectors	matjaz.vencelj@ijs.si	2	2	2	/
	Primož Pelicon	IBA, construction, channelling	Primoz.Pelicon@ijs.si	2	2	2	/
	Janez Zavasnik	TEM/SEM	janez.zavasnik@ijs.si	2	2	2	/
	Andreja Sestan	TEM/SEM, sample preparation (PhD, pos-doc)	andreja.sestan@ijs.si	2	2	2	/
IPP	Thomas Schwarz-Selinger	Sample irradiation, IBA, HI interaction, TDS	Thomas.Schwarz-Selinger@ipp.mpg.de	3	3	3	5,5,5
	Wolfgang Jacob	HI interaction, TDS	Wolfgang.Jacob@ipp.mpg.de	0	2	2	/
UHEL	Flyura Djurabekova	Multiscale modelling, RBSADEC development	flyura.djurabekova@helsinki.fi	2	3	3	0,5,5
	Xin Jin	Code development, MD, RBSADEC (PhD)	ville.jantunen@helsinki.fi	12	12	12	0,0,5
	Ilja Makkonen	DFT for RBSADEC (pos-doc)	Ilja.makkonen@helsinki.fi	0	3	3	/
	Tommy Ahlgren	IBA, HI interaction, MRE modelling	tommy.ahlgren@helsinki.fi	0	3	3	/
	Kenichiro Mizohata	IBA, RBS-channelling	kenichiro.mizohata@helsinki.fi	2	0	0	/
	Filip Tuomisto	PAS	filip.tuomisto@helsinki.fi	2	3	2	/
CEA	Christian Grisolia	MRE modelling	Christian.GRISOLIA@cea.fr	0	2	2	/
	Etienne Hodille	MRE modelling, MD	Etienne.HODILLE@cea.fr	2	3	3	0,5,5

Tritium retention in DEMO



Arredondo et al. *Nuclear Materials and Energy 28 (2021) 101039.*

- Tritium retention dominated by trapping at defects created by neutron irradiation

First modelling of tritium permeation and retention in W for DEMO first wall

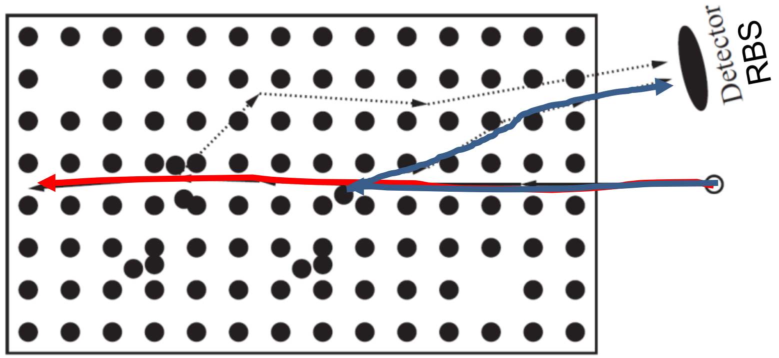
⇒ **Significant T retention**
⇒ **No steady state permeation flux within blanket lifetime**

- Calculation based on experimental studies by Markelj et al. NF (2019), Pecovnik et al. NF (2020)
- Applicable for DEMO? – need for microscopic understanding

- Which are the defect types responsible for tritium retention?

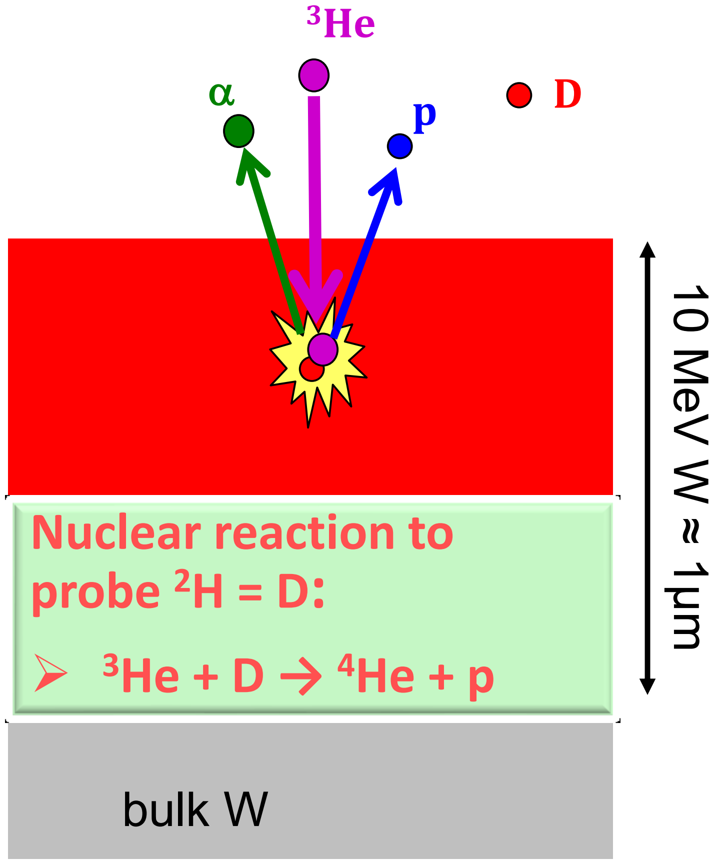
RBS channeling

Channeling Rutherford Backscattering Spectroscopy (C-RBS) is a well known method to measure disorder in materials due to ion irradiation – derive information of point defect distribution



Identify defects in displacement damaged tungsten by C-RBS with help microstructure analysis methods by:
- Electron Microscopy (SEM, TEM, STEM, …..)
- Positron Spectroscopy (PALS, DB-PAS)

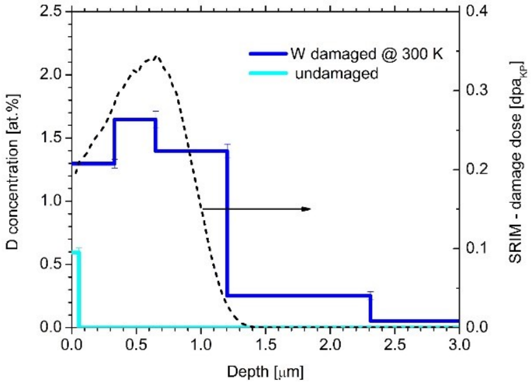
D retention analysis by nuclear reaction analysis (NRA)



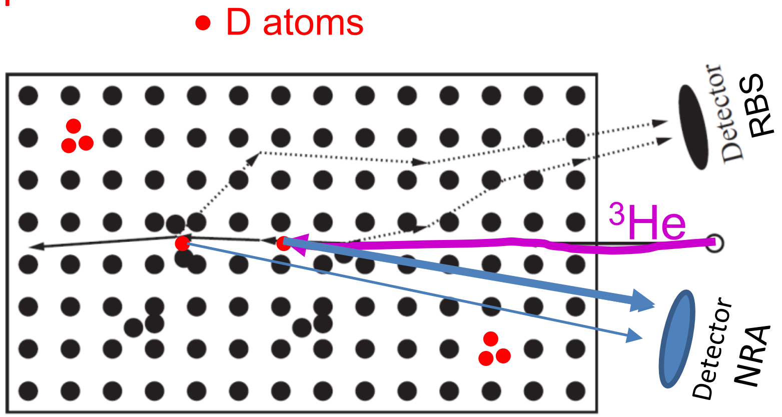
Deuterium depth profiles - Analyzing protons from nuclear reaction D(^{3}He,p)^{4}He at different ^{3}He energies from 700 keV up to 4.3 MeV

Hydrogen isotopes are trapped in defects created by i.e. high energy ion irradiation

➢ D retention is our way to determine defect concentration



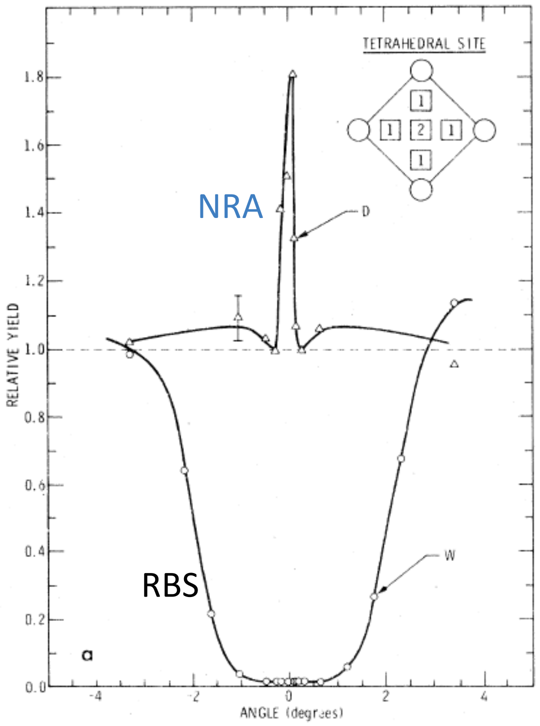
➢ Make NRA in channelling mode – obtain information where D is trapped



NRA channeling

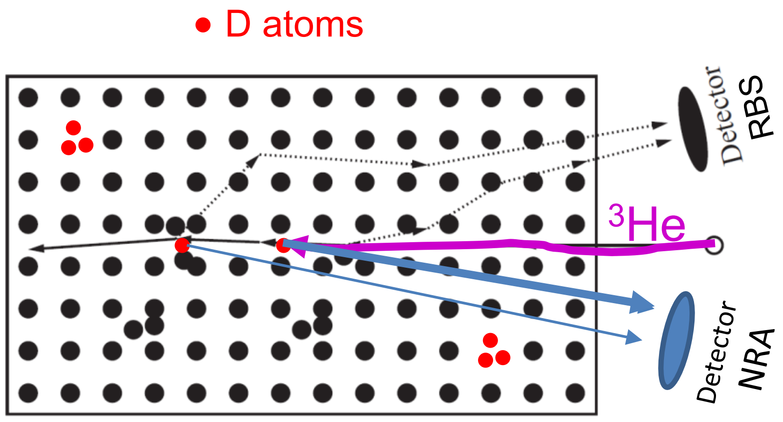
Method to determine hydrogen interstitial sites in metals in 70’s/80’s [Fukai, The metal-hydrogen systems, Springer book 2005]

- Examples of detection of hydrogen in metals by NRA-channeling



Angular scans through the <100> axis on W

[Picraux 1974] Picraux, S. T. & Vook, F. L. Deuterium lattice location in Cr and W. Phys. Rev. Letters 33, 1216 (1974).



Detection of D via nuclear reaction analysis (NRA) $D(^3He, p)^4He$

But:

- ➢ Only qualitative measure: point defects
- ➢ 30 keV D ions created vacancies by themselves, even possible to determined position of solute atoms, signal should be dominated by trapped D!

Goal of the project

Development and application of a **ion channelling method** at the tandem accelerator laboratory in Ljubljana to characterize produced defects and D retention simultaneously. As an upgrade to existing channelling procedures (**RBS channeling = C-RBS**), we want to combine it with our absolute quantitative deuterium detection method (NRA) and perform it in the channelling mode, so-called **Channelling-NRA.**

- Create samples with one dominating defect type and decorate them with D
- Use binary collision approximation (BCA) code RBSADEC recently developed at the UHEL [Zhang et al. Phys. Rev. E 94, 043319 (2016)] and further develop the modelling algorithm to identify defects from RBS-channeling and D position from NRA-channeling spectra
- The code is able to generate the C-RBS spectra from an arbitrary read-in atomistic structure, containing realistic defects produced in molecular dynamics (MD) simulations – information by TEM and PAS analysis.
- **The main goal of this project is to develop an experimental setup and analysis procedure that will enable detection of individual types of defects and the amount of hydrogen trapped in the defects.**

DeHydroC project: 1. June 2021 – 31. May 2024

Work Package structure:

- **WP1 - The In-Situ Ion Beam Analysis in Channelling mode (INSIBA-C)**
  - T1.1 Incorporation of the goniometer in the INSIBA experimental station – JSI.
  - T1.2 Detection system for ion beam methods - JSI.
- **WP 2 - Sample production and defect characterization**
  - Task 2.1 Production of samples with dominant defects in the material – JSI and MPG.
  - Task 2.2 Characterization of defects – University Helsinki (UHEL), JSI, MPG.
  - Task 2.3 Simulation and interpretation of C-RBS spectra - UHEL, CEA, JSI.
  - Task 2.4 In-situ C-RBS and sample heating – JSI, MPG, UHEL
- **WP 3 - Deuterium retention studies**
  - 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.
  - Task 3.3 – Modelling of deuterium position in lattice/defect and identification of D position - UHEL, CEA, JSI.
- **WP 4 - Management, Dissemination, Communication and Exploitation.**
  - Task 4.1 Management.
  - Task 4.2 Dissemination, communication and exploitation:

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  - **Task 4.1 Management.**
  - **Task 4.2 Dissemination, communication and exploitation:**

DeHydroC project: 1. June 2021 – 31. May 2024

		Month 6	Month 12	Month 18	Month 24	Month 30	Month 36	
WP1	Task 1.1		M1					D1
	Task 1.2			M2				
WP2	Task 2.1	M3						
	Task 2.2			M4				
	Task 2.3				M5			D2
	Task 2.4				M6			D3
WP3	Task 3.1				M7			D4
	Task 3.2					M8		
	Task 3.3					M9		D5
WP4	Task 4.1							
	Task 4.2							

Activities in 2021

- Task 1.1 Incorporation of the goniometer in the INSIBA experimental station – JSI.
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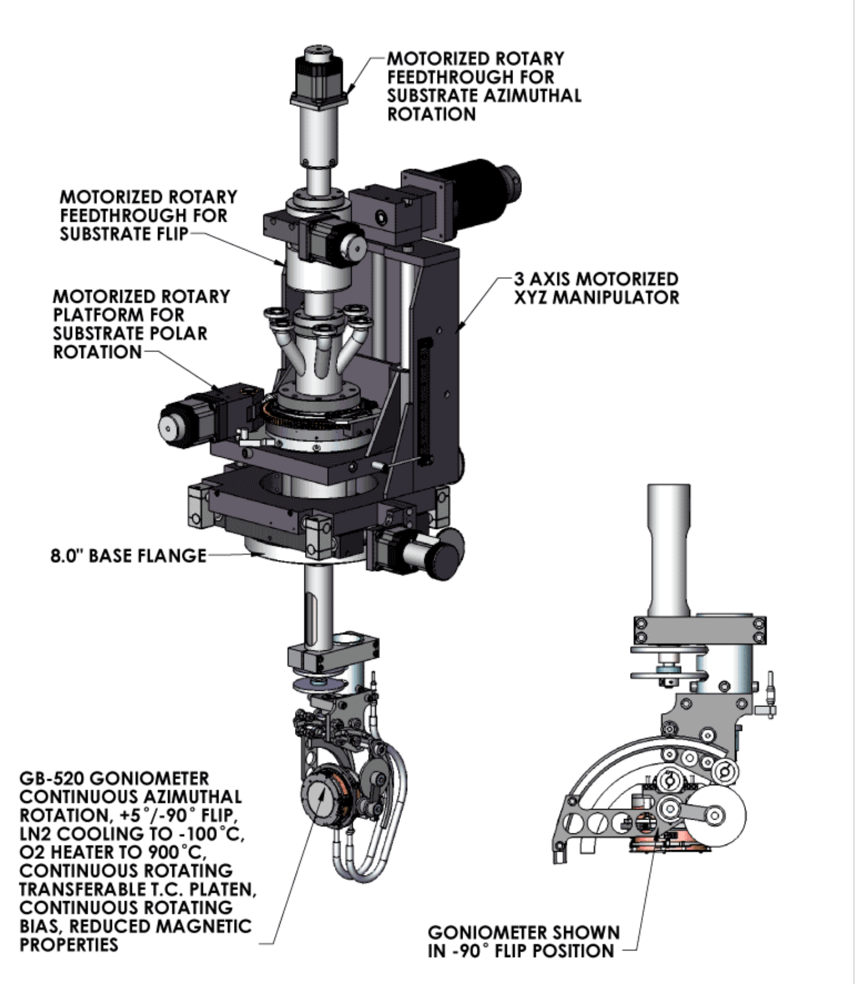
  - D1.4: First simulations of C-RBS spectra.

6-axis goniometer

- ✓ JSI – 6-axis goniometer specified, public call, final order at National Electrostatic Corp. (NEC) 08/2021, Delivery time ca. 8 months



Resolution in rotation: 0.01 degrees

Sample platen heating to 1200^{o}C continuous

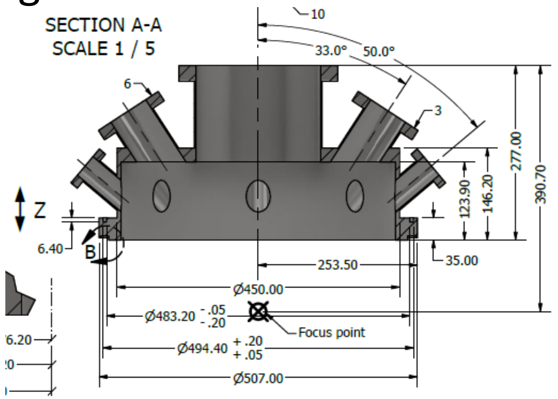
Liquid nitrogen cooling

Possible sample bias to 500 V

Sample size 4 cm

Data acquisition system for 5 detectors with computer and program for spectra collection

Additional: Load-lock chamber with magnetic arm



New cover for the vacuum chamber – ordered

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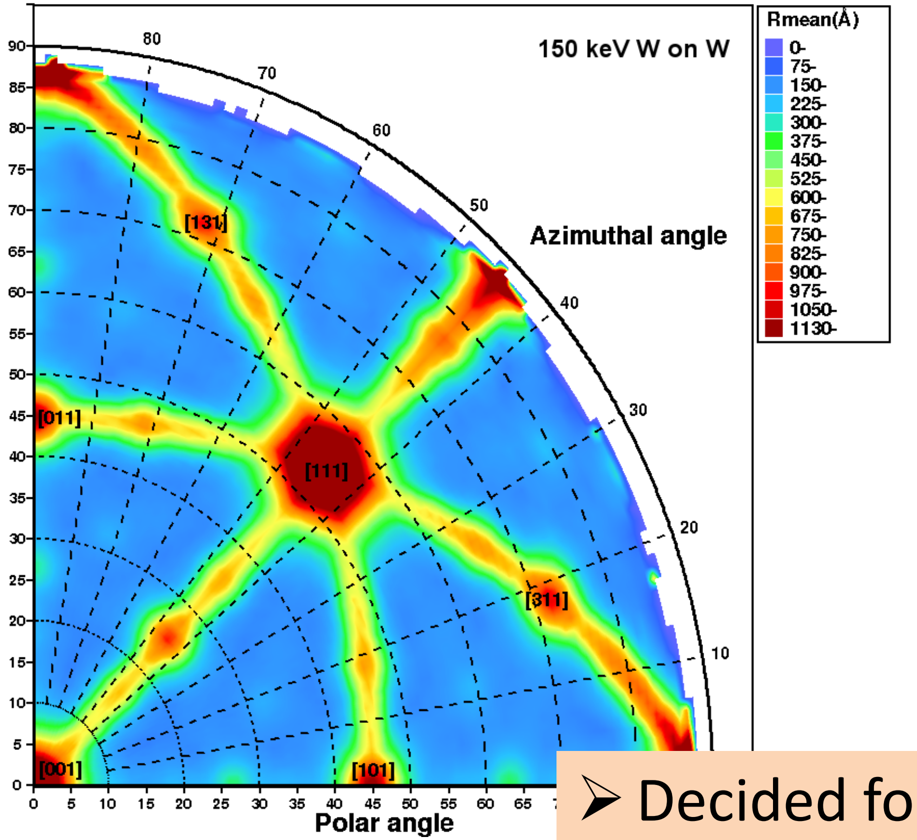
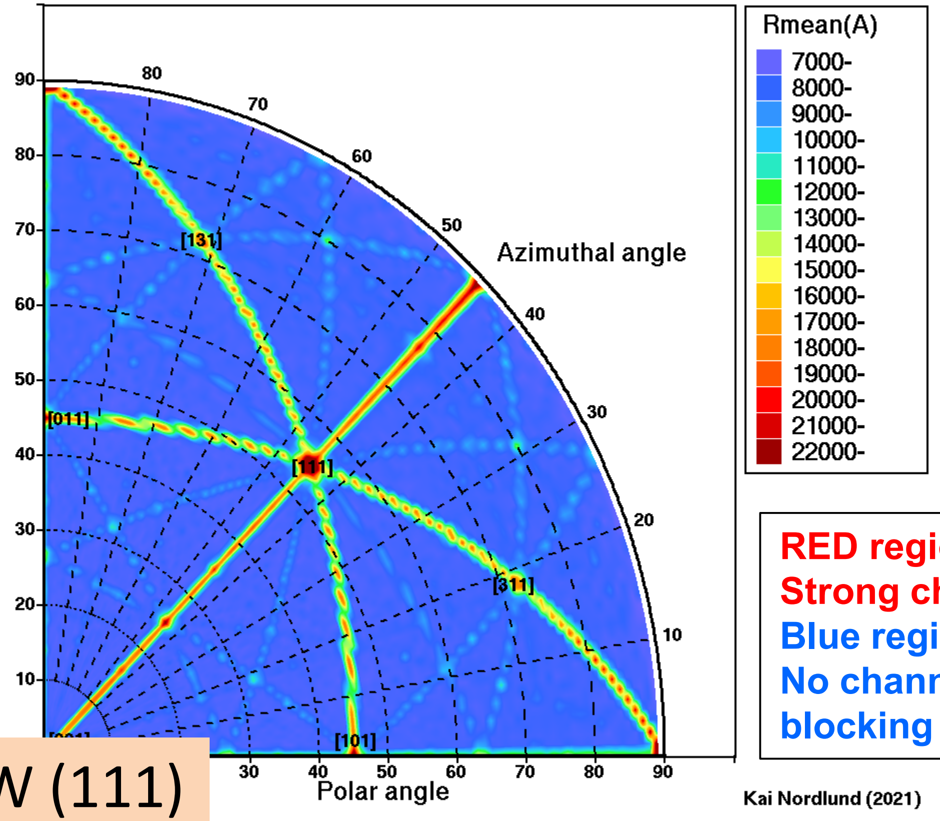
- **Choice of single crystal orientation?**
- To examine channelling systematically, VTT team developed a Molecular Dynamics-recoil interaction approximation approach (MD-RIA) to do simulations of ion movement in all different crystal directions
[K. Nordlund, F. Djurabekova, and G. Hobler. Phys. Rev. B, 94:214109, 2016]

Sample results of channeling maps: BCC



Mean ion range of 10 MeV W ions on W by angle



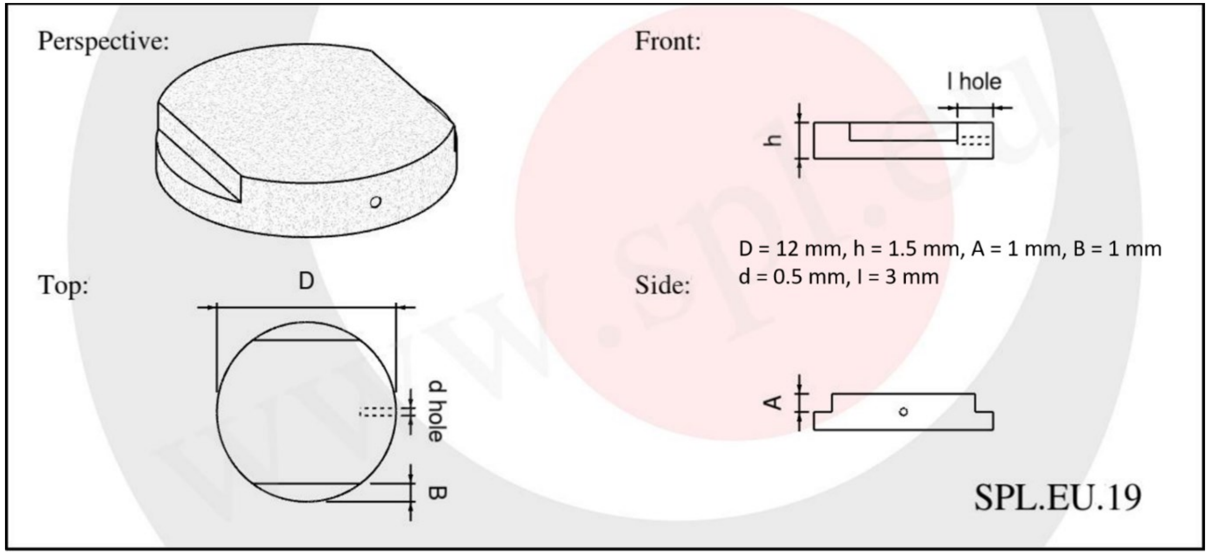
Kai Nordlund (2021)

- Decided for W (111)

RED regions: Strong channeling
Blue regions: No channeling or blocking

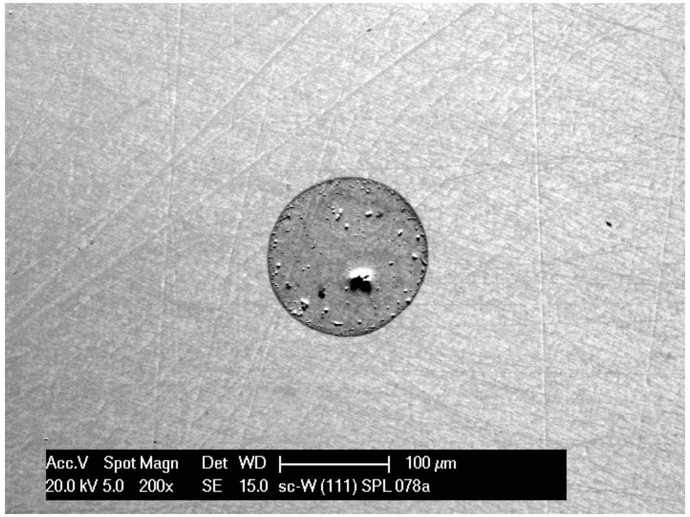
Single Crystal W (111) - Sample geometry

- Ask for quotations at MaTec, Princeton Scientific Corp., SPL
- W single crystal (111) – purchase of 9 samples at Surface Preparation Laboartory (SPL), Netherlands
- Sample design for easy handling, mounting, transfer
- Thermocouple hole

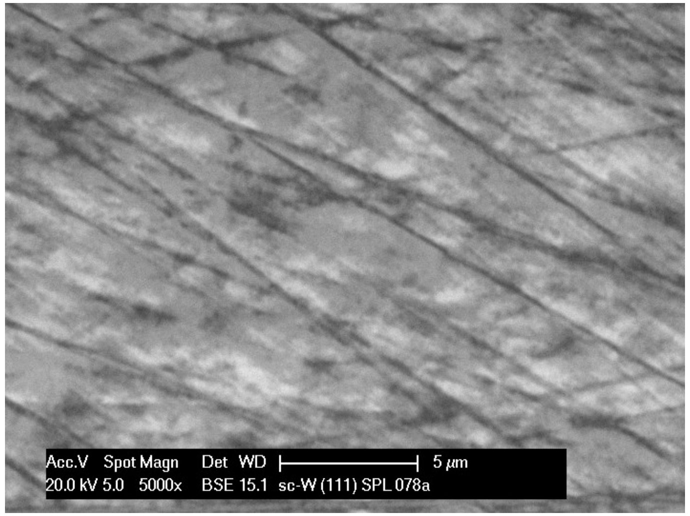


As delivered samples

- Mirror polished surfaces
- But near surface distortions
- Action – additional material removal at IPP – electropolishing?



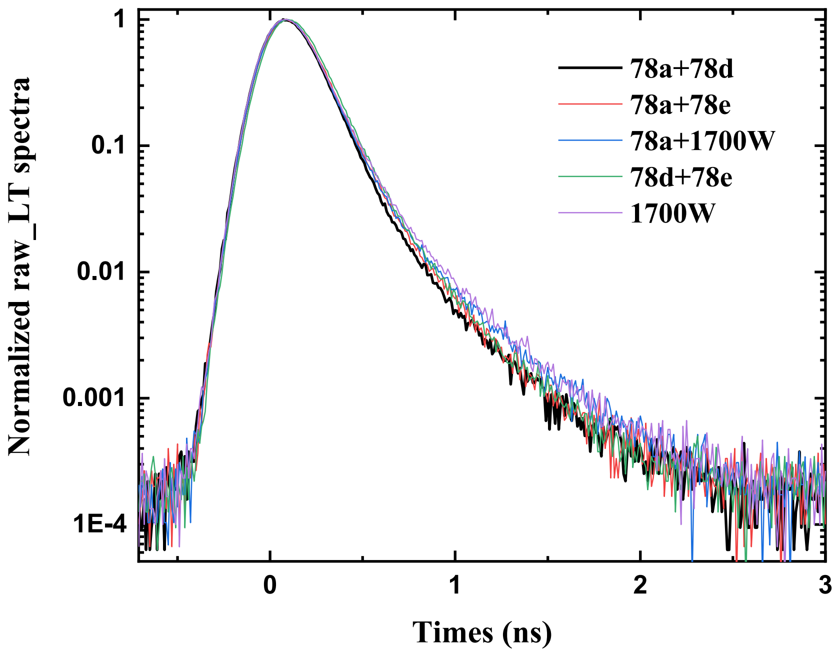
SEM: scratches + mosaic indicating distorted zone



Positron anihilation lifetime spectroscopy

- 3 samples sent to Helsinki – check for the sample quality
- Best results with electropolishing and annealing, but electropolishing was not reproducible

	ID	Definition
1)	78a	Electropolished + 2325 K
2)	78d	Only 2325 K
3)	78e	As is



Samples	Poitron lifetime (ps)
1700W+1700W	118
78a+78d	108
78a+78e	116
78d+78e	118
1700W+78a	110

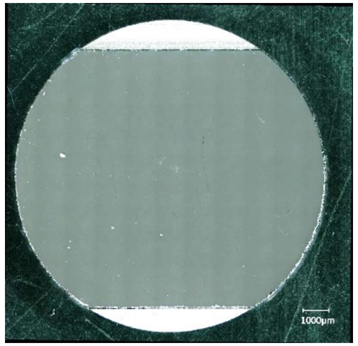
Chemo mechanical ‚Vibro‘ polishing

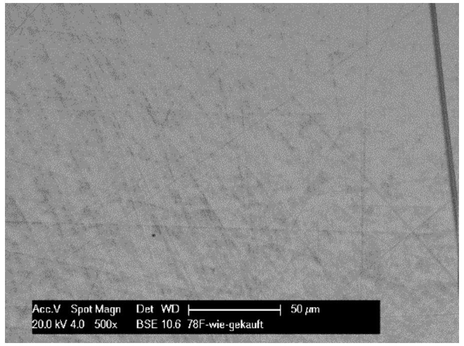
- Reproducible results of surface by ‘vibro’ polishing

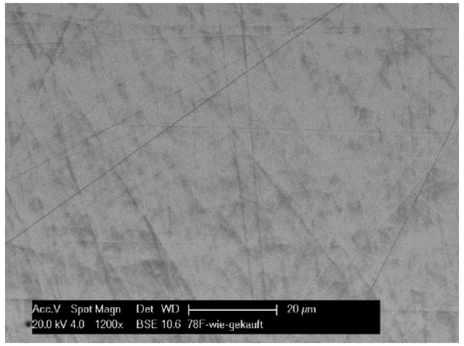
optical microscopy

SEM

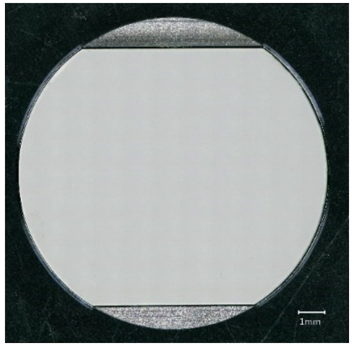
As is

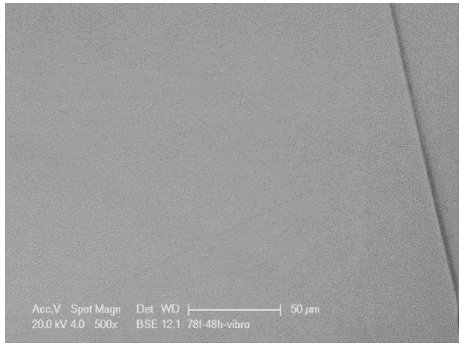


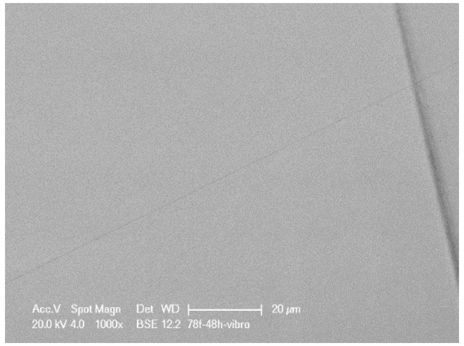




48 h
vibro







Initial plan for damaging – creation of single dominating defects in sample

DeHydroC goal, to make samples with one dominating defect type - different options

Light ions (200 keV D ions)

- Isolated cascades: vacancies?
- Effect of D?

Heavy ion W (10.8 MeV W ions)

- Denser cascades

Damage level

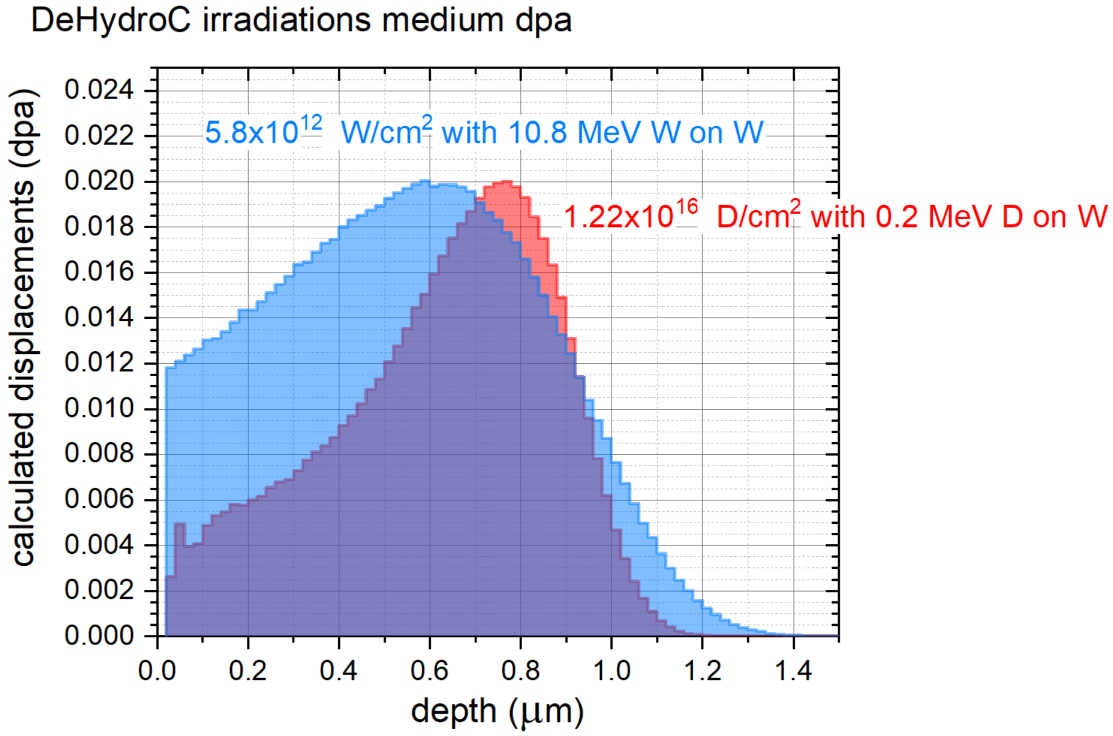
- ‘High’, to see something in C-RBS
- ‘Low’, to learn something from PAS

Temperatures

+ room temperature

+ 800

7 degrees rotated and tilted



D:tms-Daten\SRIM\ab2016W in W10.8MeVWaufWEd90eVEb0eV\ SRIM to dpa_DeHydroC.opj(Graph6)
17.09.2021 14:55:49

Samples prepared

Decided to go with only 10.8 MeV W ion irradiation, according to recent publications [Hu et al. JNM 556 (2021) 153175]

- **Mainly single vacancies at room temperature**
- **Larger vacancy clusters at 773 K**

Journal of Nuclear Materials 556 (2021) 153175

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

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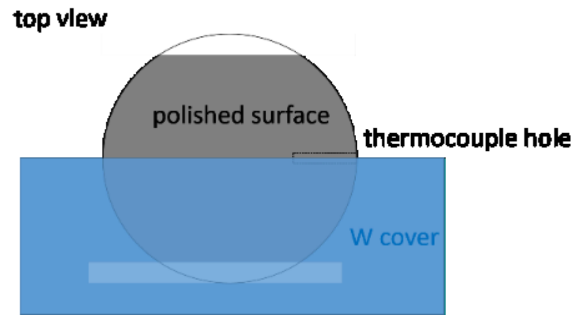
Effect of purity on the vacancy defects induced in self–irradiated tungsten: A combination of PAS and TEM

Z. Hu[a], P. Desgardin[a], C. Genevois[a], J. Joseph[a], B. Décamps[b], R. Schäublin[c], M-F. Barthe[a,*]

[a] CEMHTI, CNRS, UPR3079, University of Orléans, F-45071 Orléans, France
[b] IJCLab/CNRS, Paris - Saclay University, France
[c] Laboratory of Metal Physics and Technology, Department of Materials, ETH Zurich, Switzerland

✓ For RBS channeling in Madrid + TEM characterization

	ID	Definition
1)	78f	'Heavily damaged standard': 0.2dpa, 290K
2)	78b	'Big vacancy clusters': 0.2 dpa, 800 K
3)	78g	'Single vacancies': 0.02 dpa, 290 K, half covered
4)	78c	'Small vacancy clusters': 0.02 dpa, 800 K



✓ For PAS/PALS analysis Helsinki

	ID	Definition
1)	78h	'Single vacancies': 0.02 dpa, 290 K
2)	78 x	undamaged
3)	…	
4)	…	

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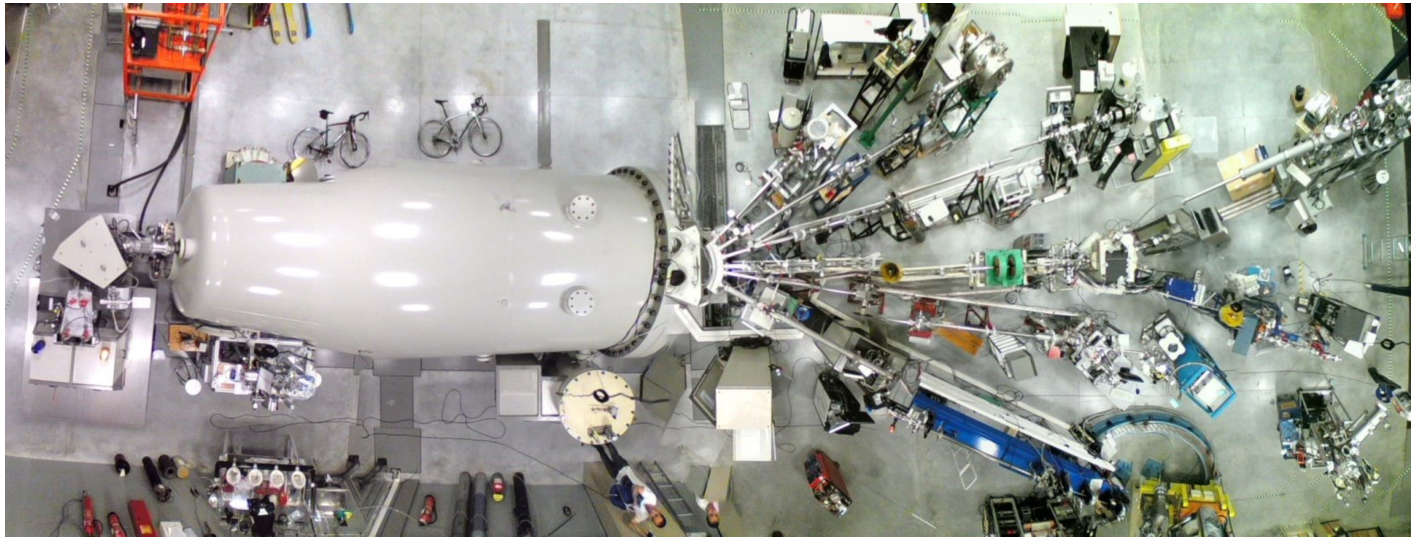
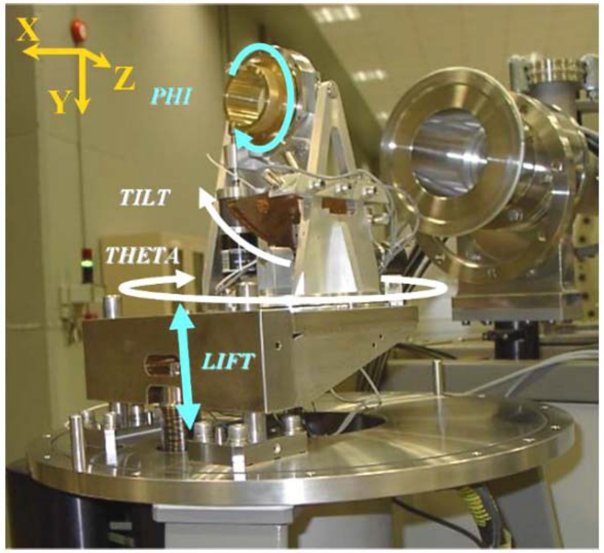
Analysis of irradiated samples by C-RBS in Madrid

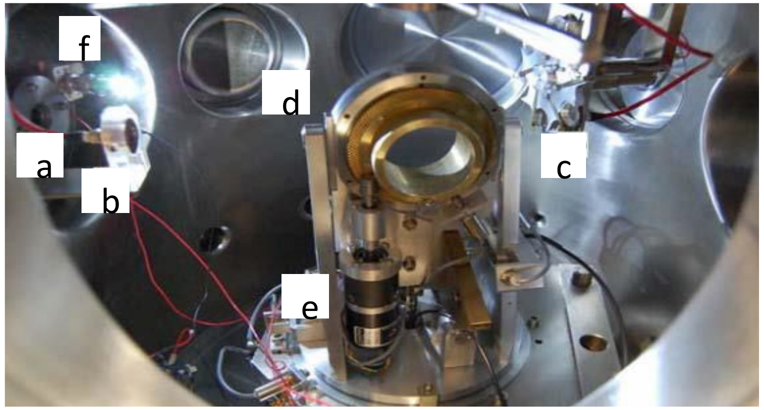
- JSI team visited *CMAM (Centre for Micro Analysis of Materials), UAM (Madrid.) for C-RBS measurements in September*



5MV Accelerator Cockcroft-Walton (High Voltage Europe)

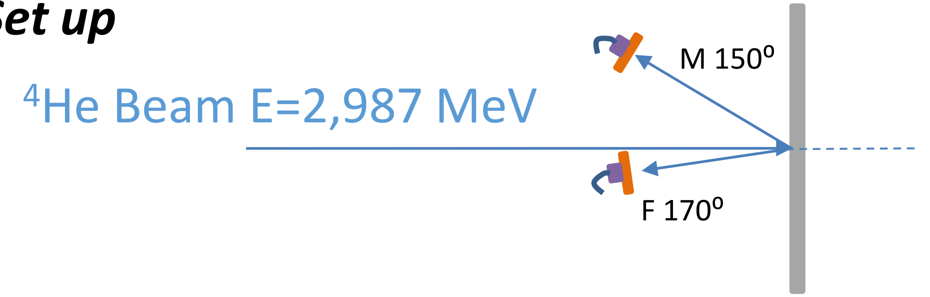


3 angles goniometer
(no heating)
(Panmure Instruments, UK)



a: beam entrance
b: Si F detector
c: Si M detector
d: Ge detector (gamma)
e: goniometer
f: light

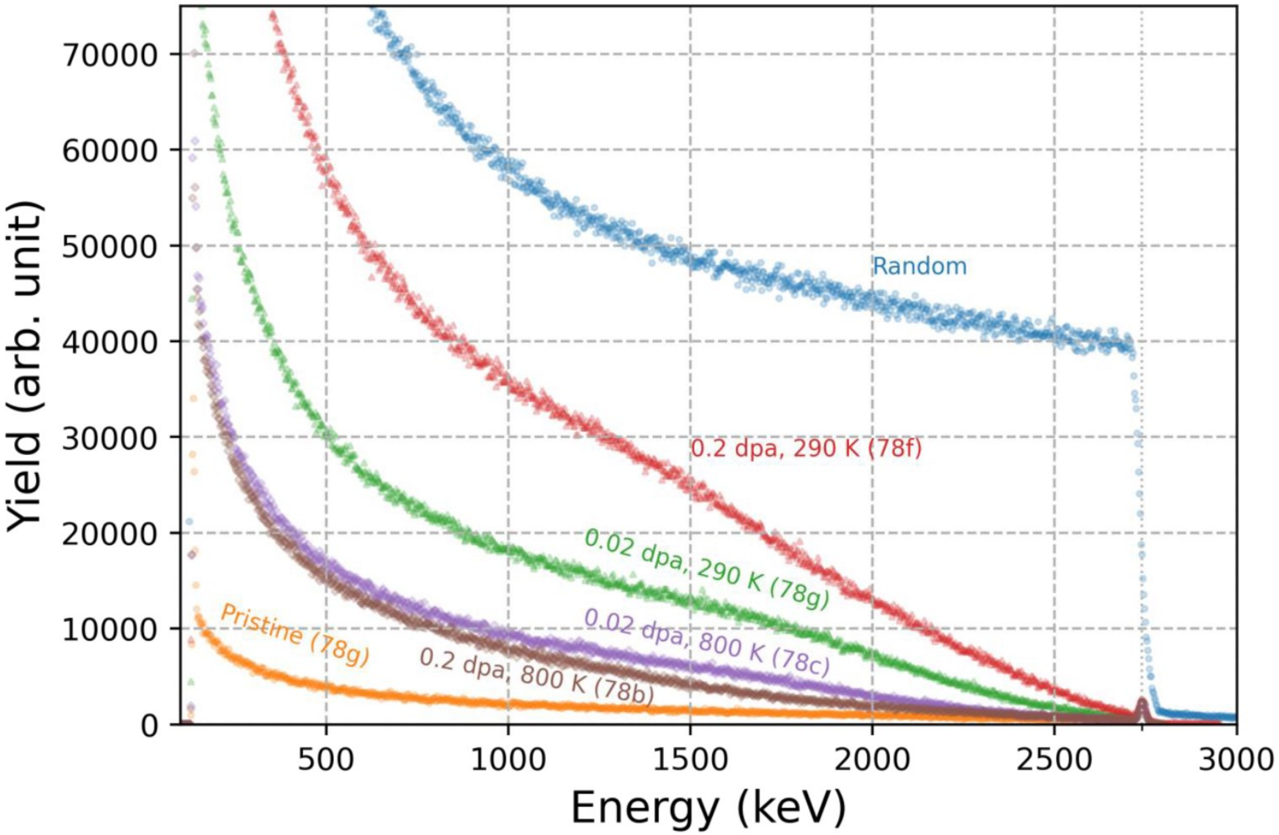
Set up



Analysis of irradiated samples by C-RBS – measurements

10.8 MeV W on W

Sample	Maximum dpa	Irradiation temperature (K)	Expectation of defects (DeHydroC meeting 07/10/2021)
78g	0.02	290	Single vacancies
78f	0.20	290	Heavily damaged
78c	0.02	800	Small vacancy clusters
78b	0.20	800	Big vacancy clusters



Random = Σ100 spectra (5 μC)
78f heavily damaged standard: 0,2dpa 290 K
78g single vacancies: 0,02 dpa 290 K

78c small vacancy clusters: 0,02 dps 800 K
78b big vacancy clusters: 0,2 dpa 800 K

78g pristine

$$Relative\ Dechanneling\ (1050) = \frac{\chi_i - \chi_{pristine}}{\chi_{random} - \chi_{pristine}}$$

RD (78f) = 0,088
RD (78g) = 0,040
RD (78c) = 0,014
RD (78b) = 0,013

- ✓ Starting material (pristine sample) has a very good crystallinity
- ✓ We have observed differences between the irradiation damage treatments

Analysis of C-RBS spectra by RBSADEC simulation using randomly displaced atoms (RDA)

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10.8 MeV W on W

Sample	Maximum dpa	Irradiation temperature (K)	Expectation of defects (DeHydroC meeting 07/10/2021)
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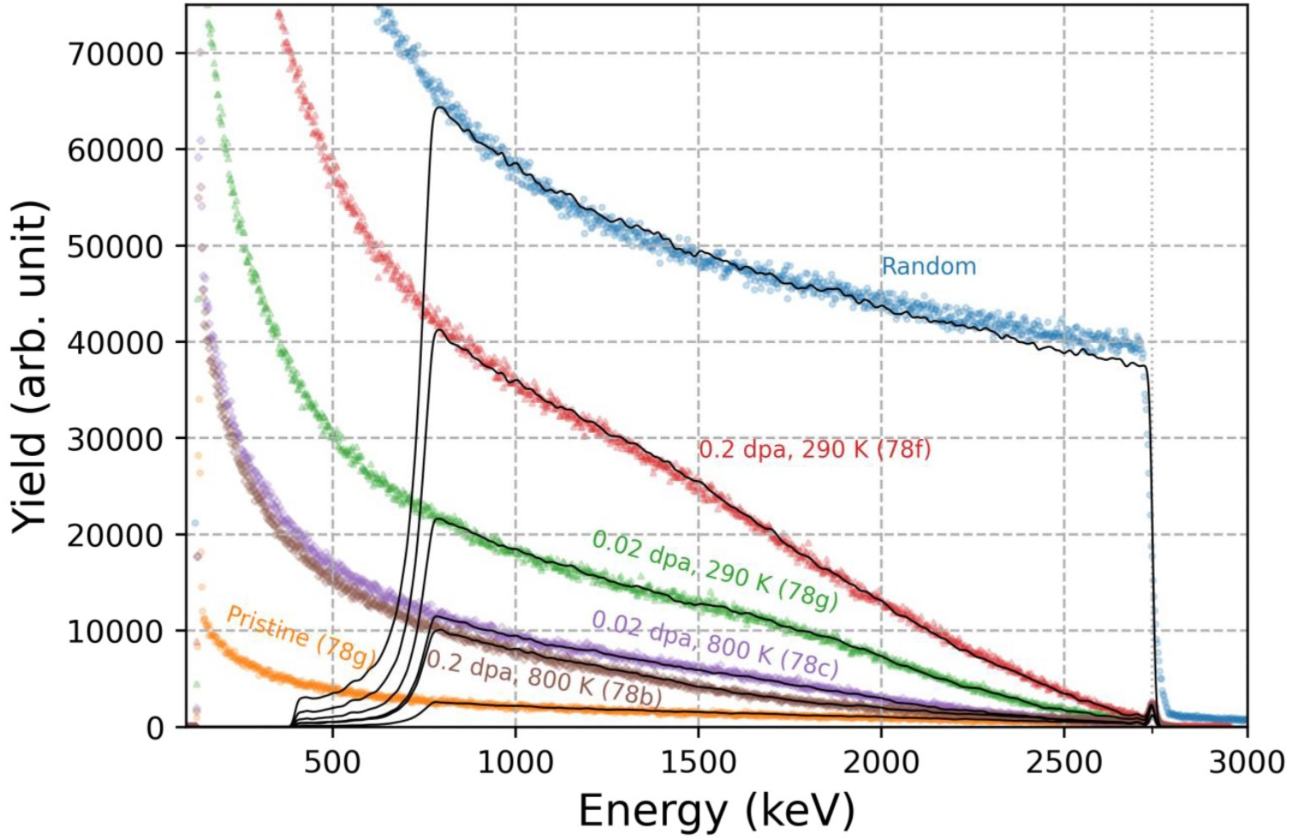


➢ **RBSADEC-Simulation:**

- 2.987 MeV He ions on <111>-W
- Detector: 170°
- Temperature: 290 K
- Detector resolution: 16.5 keV (FWHM)
- Depth: 1.5 µm

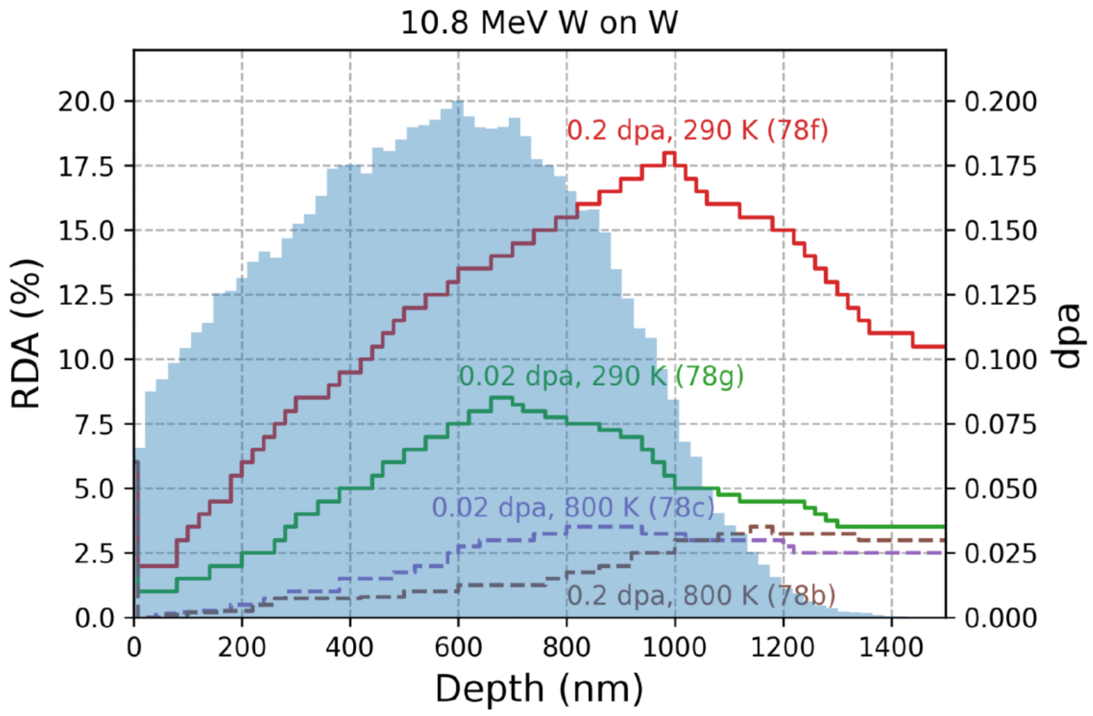
- Random: a sample with 100 % randomly displaced atoms (RDA)
- Aligned-pristine: a perfect pristine sample (relatively high ion beam divergence)
- Aligned-damaged: samples with RDAs

Analysis of C-RBS spectra by RBSADEC simulation using randomly displaced atoms (RDA)

UNIVERSITY OF HELSINKI

➢ dpa profile

- SRIM - Full damage cascades
- 7° inclined
- Threshold displacement energy: 90 eV
- Maximum dpa: 0.2 at 600 nm



➢ RDA profiles

1) Surface disorder: 6 RDA in first 8 nm

2) Maximum RDAs in bulk and corresponding depth

Samples	Max. RDA (%)	Depth (nm)
78g (0.02 dpa, 290 K)	8.5	680
78f (0.20 dpa, 290 K)	18	990
78c (0.02 dpa, 800 K)	3.5	870
78b (0.20 dpa, 800 K)	3.5	1160

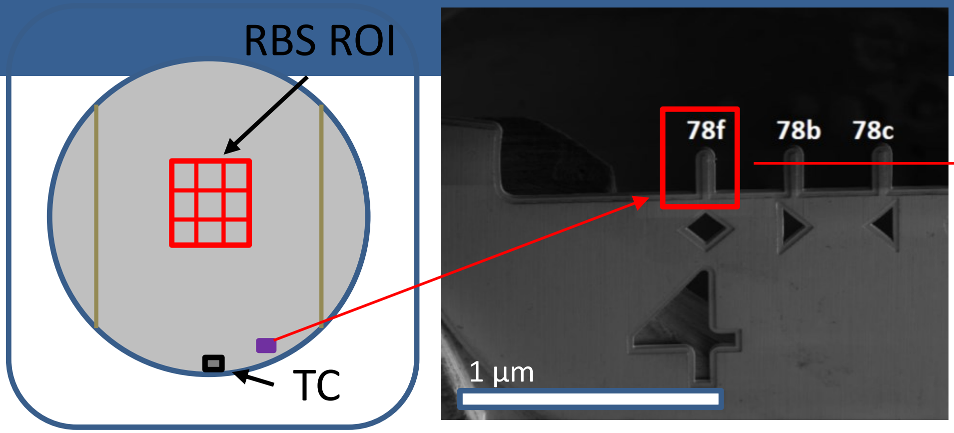
(not 10 times) (higher) (same) (higher)

3) RDAs at deep regions (dechanneling, ~dislocations)

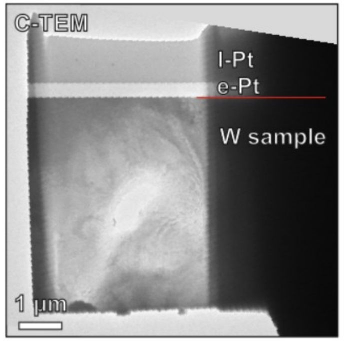
4) Temperature effect:
- Decrease of RDA (annealing)
- Close RDAs for 78g,c & b after 1200 nm (dechanneling, a common defect at both temperatures)

7

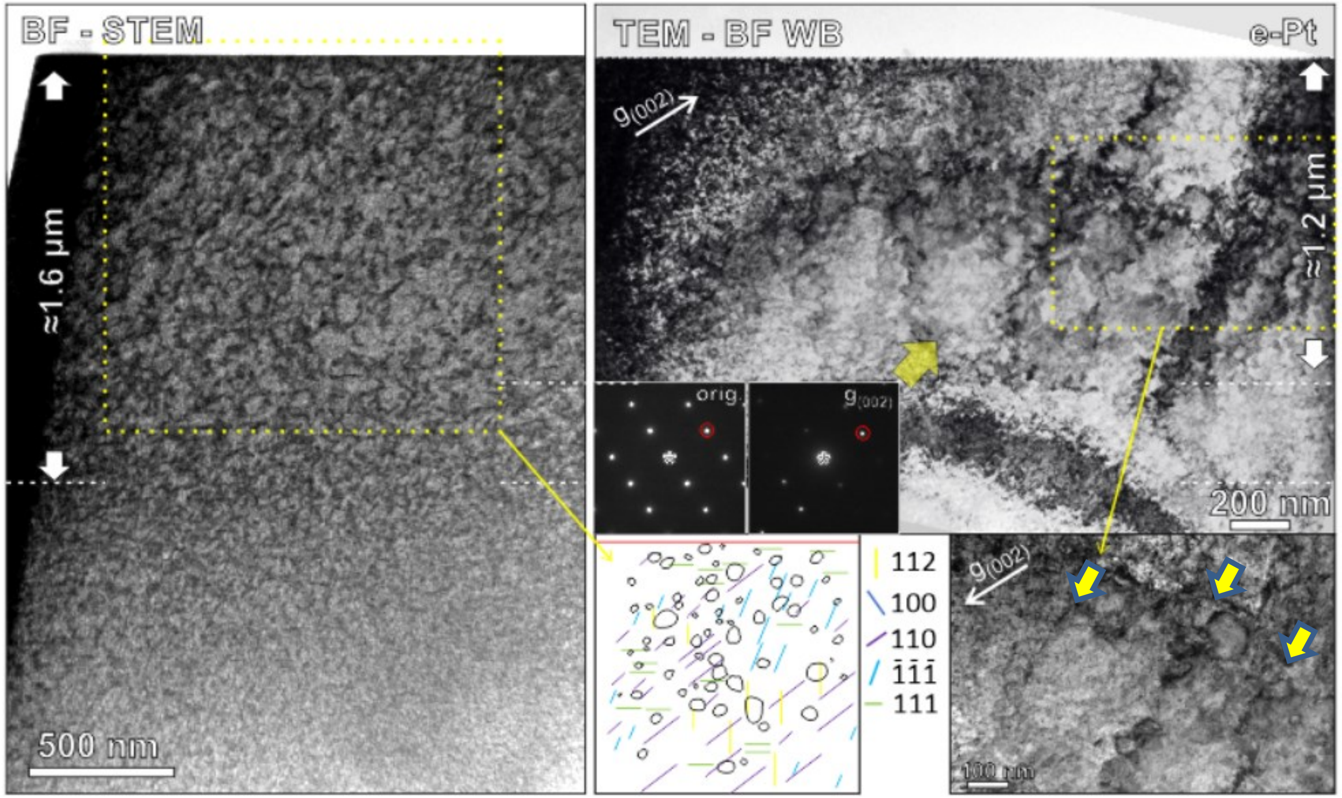
TEM analysis of SC W (111) samples after RBS



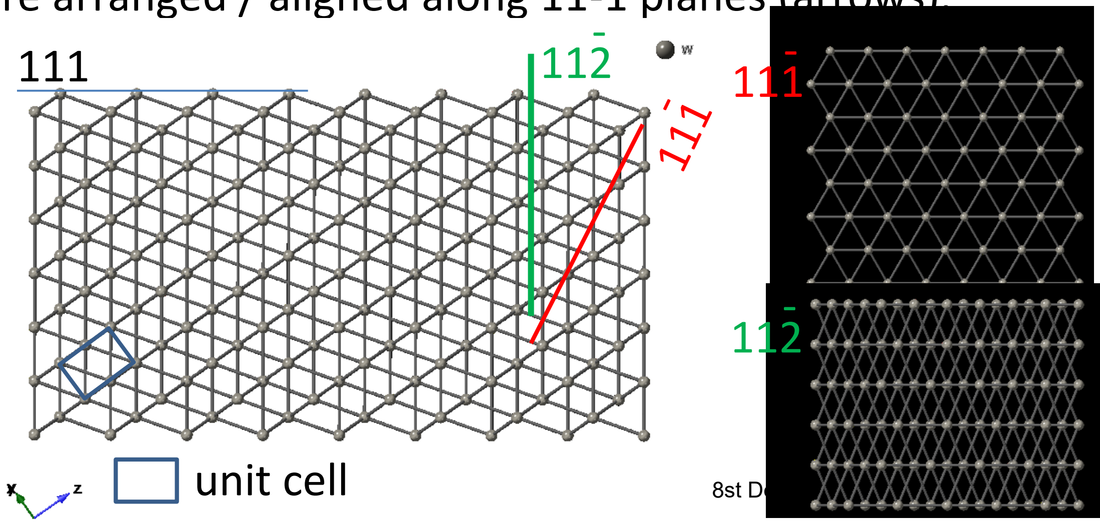
1. TEM sample prep by site-specific FIB



2. TEM lamella orientation and analysis (=> what we are looking at)

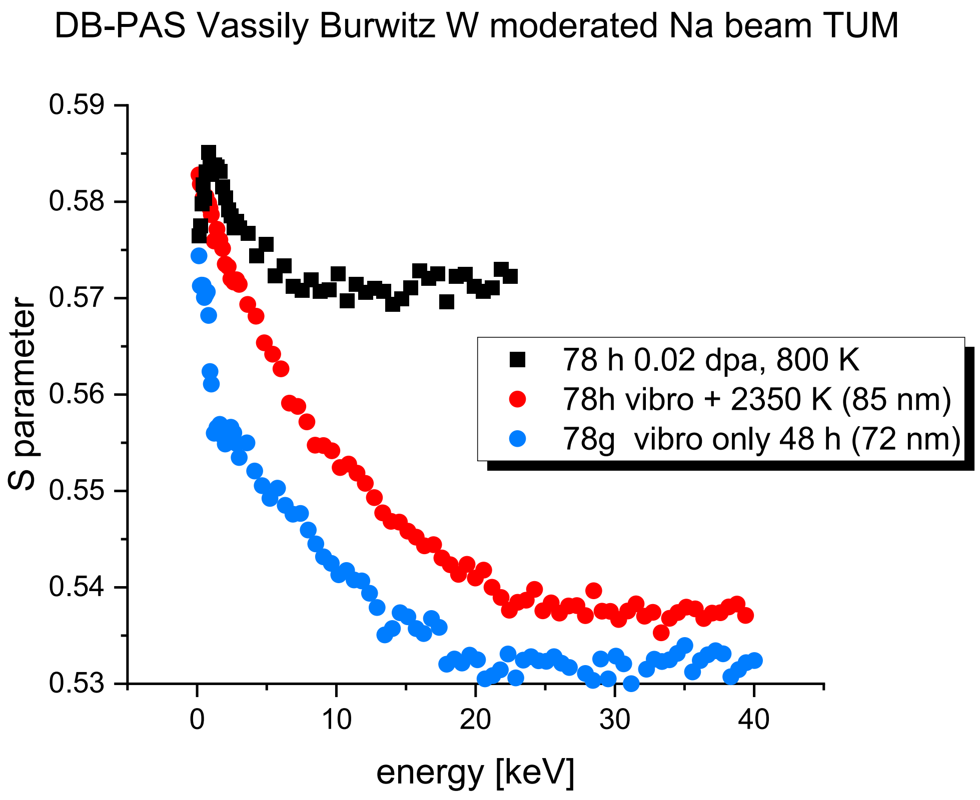


Bright-field (BF) STEM shows heavy defects in the first 1.6 mum below surface. Defects are mainly line dislocations following all major crystal planes, and dislocation loops (circles). As it appears, the DL loops are arranged / aligned along 11-1 planes (arrows).



8st D

DB PAS at TUM



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78g-h_vibro_only-annealed_0.02dpaPAS-raw.opj
Graph3
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Positron annihilation lifetime spectroscopy (low energy) not available in Helsinki till spring – technical problems

- Analysis by Doppler broadening PAS at Technical University Munich
  - Defects clearly visible
- Interpretation ongoing

Conclusions

Project is proceeding as planned

- ✓ **D1.1 (Task 1.1) Purchase of goniometer with cooling and heating capabilities accomplished.**
- ✓ **D1.2 (Task 2.1): First batch of samples prepared.**
- **D1.3 (Task 2.2):**
  - ✓ **First successful C-RBS measurements in Madrid**
  - ✓ **Microstructure analysis of samples by TEM – ongoing, PAS - ongoing**
- ✓ **D1.4 (Task 3.2): Successful simulations of C-RBS spectra by RBSADEC code using randomly displaced atoms.**
- **WP 4 management - four zoom meeting organized with the team members, details and presentations available on Indico: KickoffMeeting - April, Status meeting in June, October, November**

No changes required for 2022

Thank you for your attention