Detection of defects and deuterium in displacement-damaged tungsten by applying Rutherford backscattering spectroscopy and nuclear reaction analysis in channeling configuration

DeHydroC
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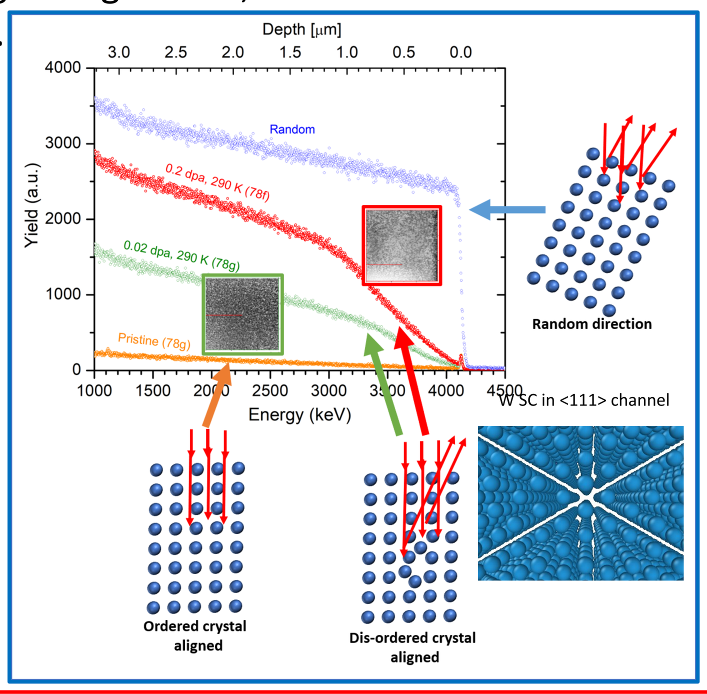
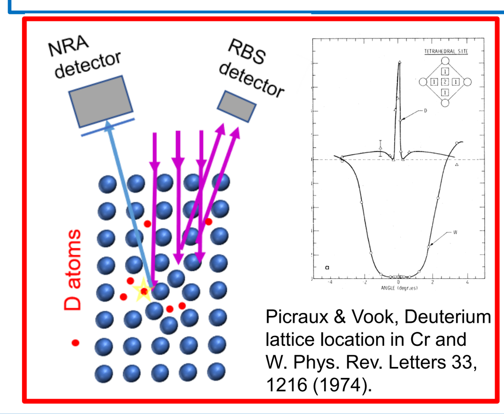
Introduction

In a future nuclear environment, 14 MeV neutrons from the D-T fusion reaction will create defects in the W lattice, affecting the physical properties of the material. Understanding the interaction of hydrogen with the host lattice is crucial for fusion research, since low hydrogen isotope (HI) retention is a stringent requirement for a fusion reactor. In this work, we show first results of the development of an advanced characterisation techniques to study lattice disorder and HI location. By combining different ion beam techniques in the channeling configuration, the influence of structural defects on HI retention and vice versa is studied.

To examine the generated defects created by high energy W ion irradiation in the W lattice, we utilized Rutherford backscattering spectrometry in channeling configuration (RBS-C), a well-established method for studying lattice disorder and defect evolution induced by irradiation. To quantify the disorder, the change in the ion yield of light ions backscattered along a specific crystallographic direction is measured [1].





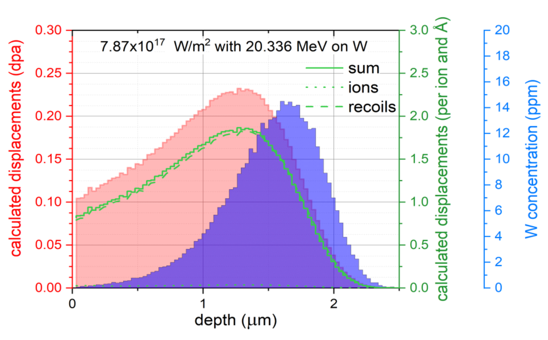
Picraux & Vook, Deuterium lattice location in Cr and W. Phys. Rev. Letters 33, 1216 (1974).

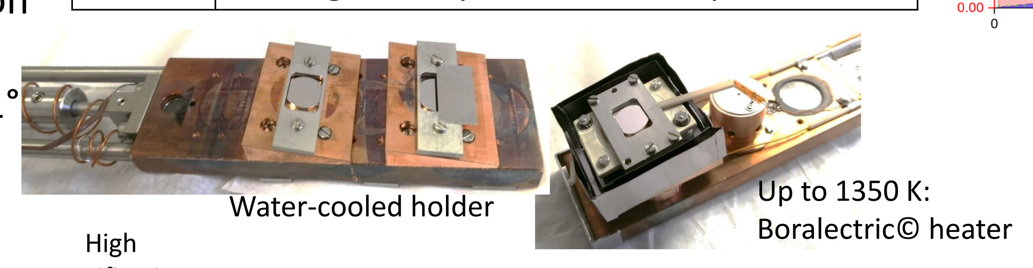
The use of nuclear reaction analysis (NRA-C) in conjunction with the RBS-C can provide information on the location of other species in the host material. In our case, we employed the ^{3}He nuclear reaction with deuterium to study the position of deuterium in the tungsten lattice.

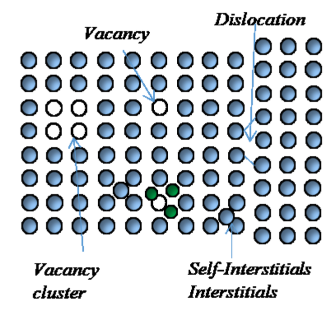
Sample production and irradiation

- W (111) crystals (Surface Preparation Laboratory B.V.) and W (100) crystals (MaTecK) were vibro-polished and annealed at 2350 K for 5 min in UHV.
- Defects were created by irradiation with 10.8 MeV W^{3+} ions. Sample was tilted by 7° and rotated by 11° to minimize channeling.

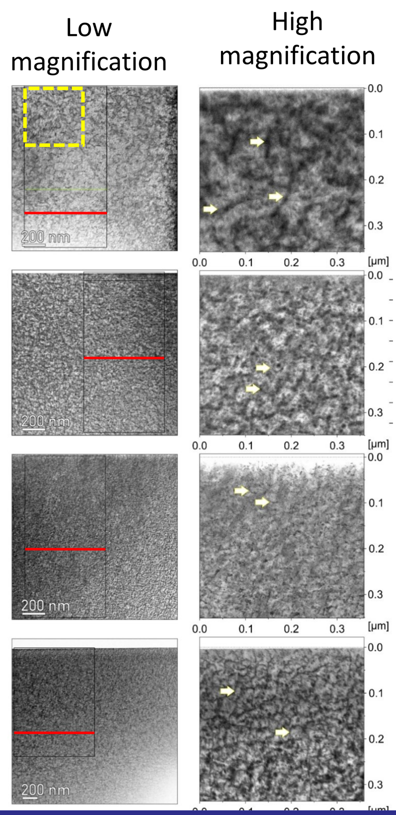
Sample	Predominant defect expected
78f	"heavily damaged standard": 0.2 dpa, 290K
78g	"single vacancies": 0.02 dpa, 290 K
78c	"small vacancy clusters": 0.02 dpa, 800 K
78b	"big vacancy clusters": 0.2 dpa, 800 K







TEM ANALYSIS



78f (0.2 dpa, 290 K) Depth ≈1.1 µm
Dense **network of dislocation lines (DL)** (~100 nm), which are already nicely visible at low-mag overview micrographs. DL directions coincide with 110 >111 > 11-2 > 111 planes.

78g (0.02 dpa, 290 K) Depth ≈ 0.7 µm
Only very **short dislocation lines** (~ 20-30 nm) in <111> , with prevailing U-shaped loops around "black dots" (size ~10 nm)

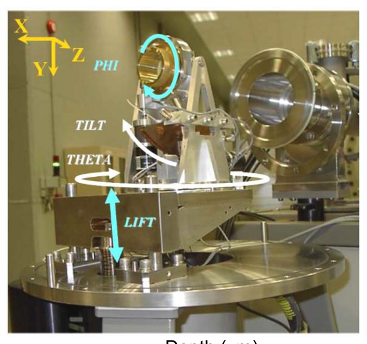
78c (0.02 dpa, 800 K) Depth ≈ 0.8 µm
Dislocations: mainly **dots and several isolated lines** (< 50 nm, in <111> direction). Dots are smaller, several nm.

78b (0.2 dpa, 800K) Depth ≈ 1.1 µm
Several **dislocation lines** and larger **black dots**. DL lines are forming a network in <111>, forming square "polygons" with ~30 nm edge.

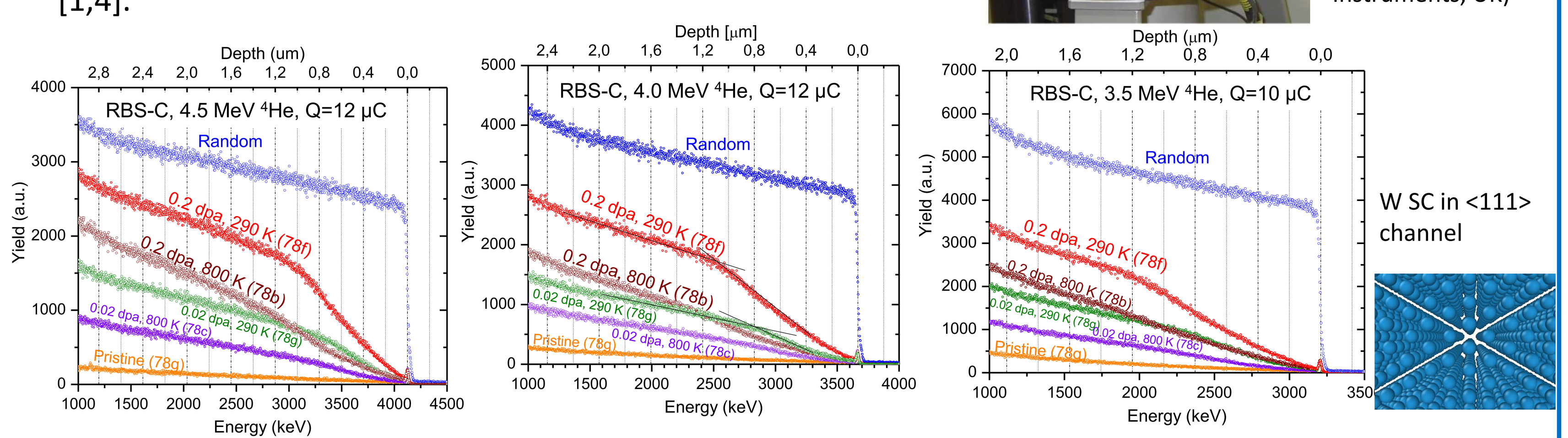
Results

Channeling Rutherford Backscattering Spectroscopy (RBS-C)

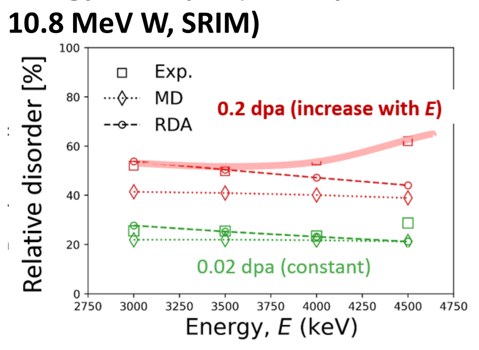
- Multi-energy RBS-C analysis along the <111> direction
- Four He^{++} beam energies of 4.5, 4.0, 3.5, and 3.0 MeV
- W (111) single crystals: W-irradiated.
- The response of the induced structural damage signal versus analysing energy gives important information about the extension of the defects (uncorrelated or extended defects) [1,4].

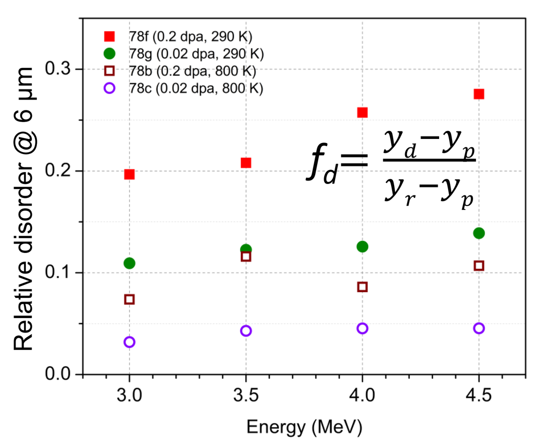


CMAM-UAM (Madrid, Spain): 5 MV Accelerator Cockcroft-Walton (High Voltage Engineering Europe) Goniometer (Panmure Instruments, UK)



Relative disorder, f_d as a function of energy at 0.6 µm (max. dpa with 10.8 MeV W, SRIM)

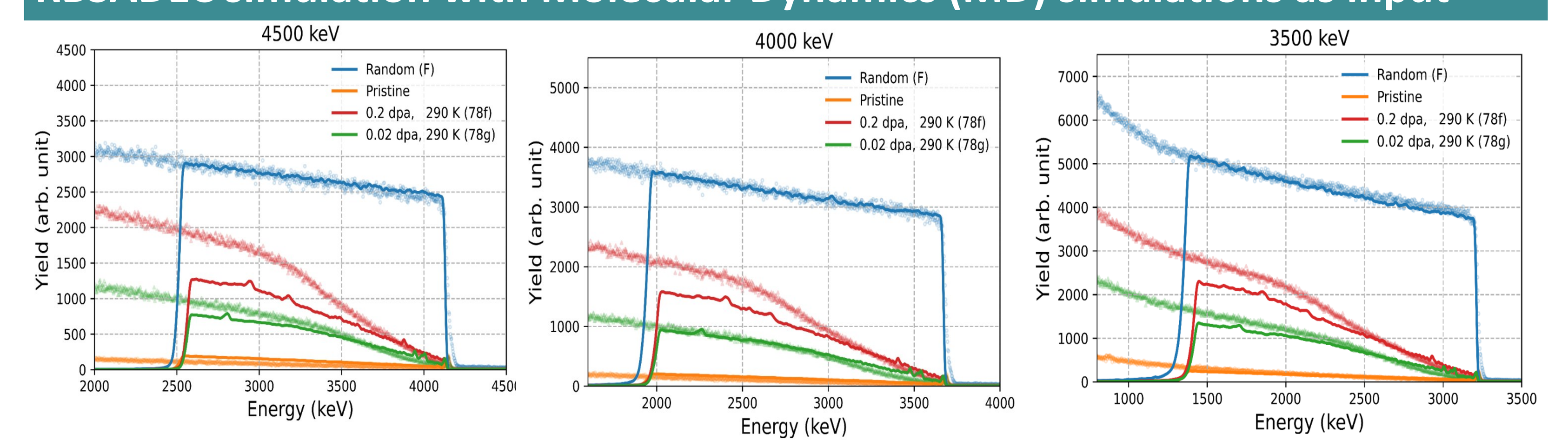




$$f_d = \frac{y_d - y_p}{y_r - y_p}$$

78f: positive slope –extended defects – dislocation lines (TEM)
78g: no slope –discontinuous defects – small dislocation loops (TEM)
78c: no slope –discontinuous defects – dots and isolated lines (TEM)
78b: no slope –discontinuous defects - (dislocation lines and black dots TEM)

RBSADEC simulation with Molecular Dynamics (MD) simulations as input



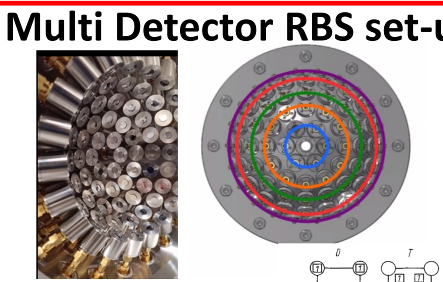
RBSADEC code [4] results (lines) compared with ^{4}He RBS/C spectra (circles). As input, realistic damage structures produced with overlapping molecular dynamic cascade simulations (MD) were used [Granberg et al. J. Nucl. Mater. 556 (2021) 153158].

For more details see [5]

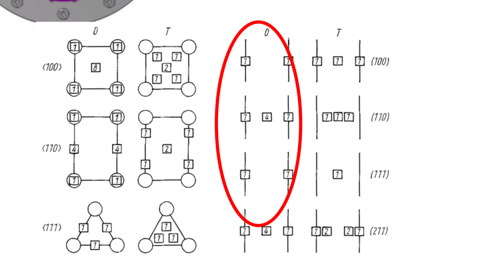
Channeling Nuclear Reaction Analysis (NRA-C)

- Simultaneous RBS-C and NRA-C measured at the Hedgehog setup for RBS channeling at Ion beam center at HZDR.
- Samples: W (100) single crystal: W-irradiated and exposed to 5 eV/D plasma at 370 K

Multi Detector RBS set-up @ HZDR



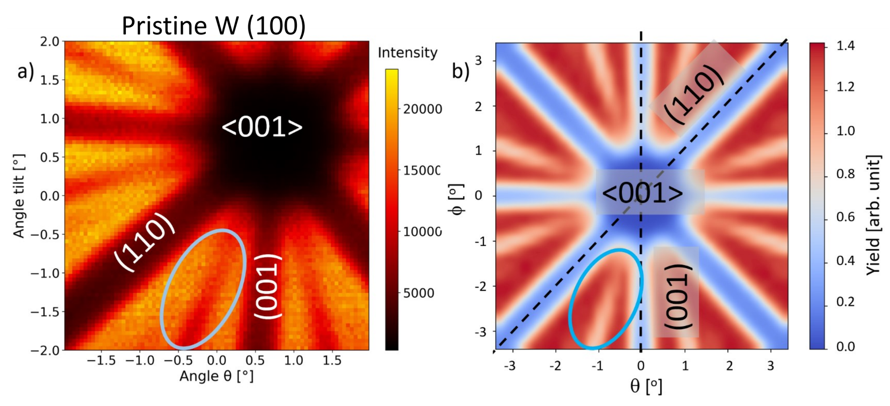
- W (100) single crystal should show a difference in the C-NRA signal when D is near the octahedral (OIS) or tetrahedral interstitial sites (TIS) [3].



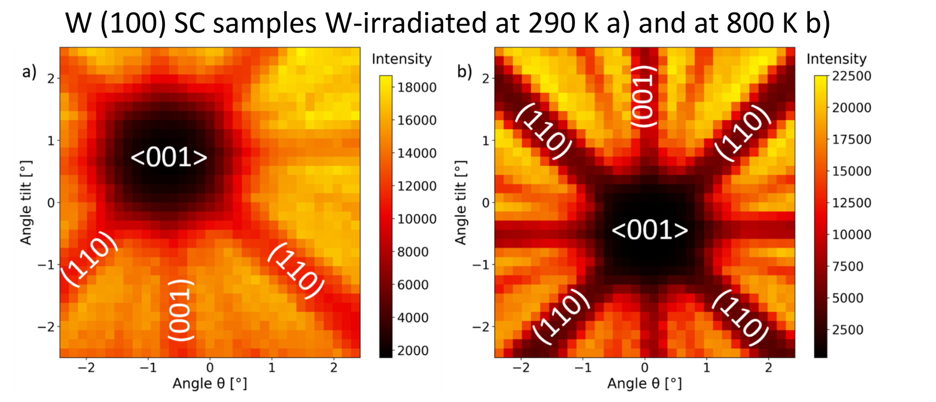
[3] Cabstanjen, Phys. Stat. Sol. (a) 59, 11–26 (1980).

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

Measured (a) and simulated (b) RBS-C 2D maps of the RBS yield.



W (100) SC samples W-irradiated at 290 K a) and at 800 K b)

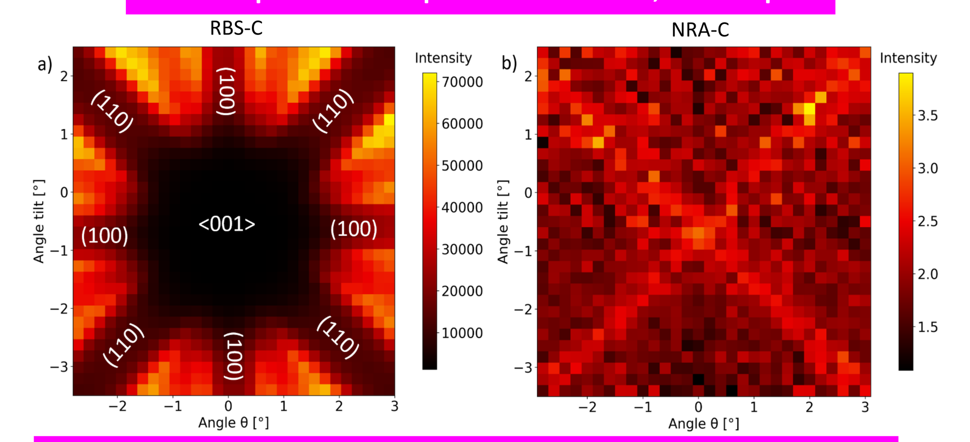


- Less pronounced planar channels for damaged sample

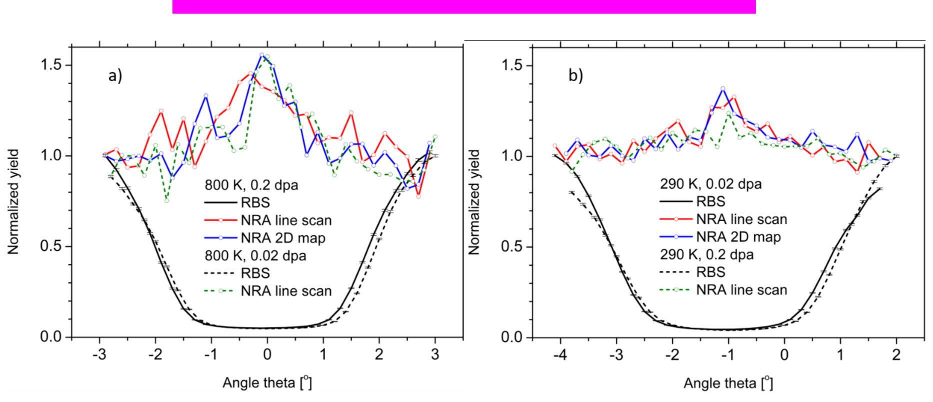
^{3}He 0.8 MeV – simultaneous RBS-C and NRA-C 2D maps

Nuclear reaction $D(^3He,p)^4He$ was used for this purpose

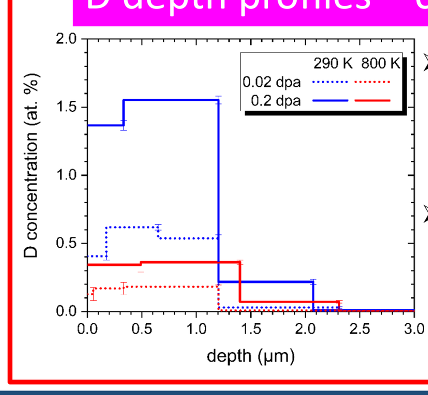
2D map for sample #2: 800 K, 0.2 dpa



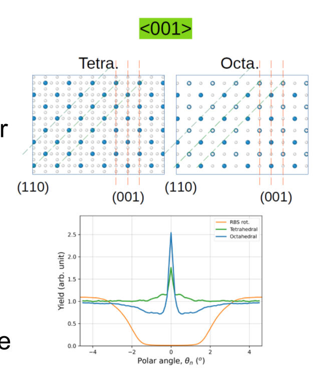
Line scans of RBS-C and NRA-C



D depth profiles – classical ^{3}He measurements



- The highest D concentration obtained for 290 K / 0.2 dpa sample. The D concentration decreases for lower damage dose and higher irradiation temperature of 800 K -> less defects are created at lower dose and at higher temperatures.
- PALS shows that for samples irradiated at 290 K mainly single vacancies and small vacancy clusters up to V4 are present, whereas for samples irradiated at 800 K large vacancy clusters are present with more than 25 vacancies.



Angular scans accros the <100> axial channel:

- 800 K damaged samples: NRA yield peaks in the centre of the channel, where the RBS is at its minimum.
- Room-temperature-irradiated samples: NRA yield peaks in the centre of the channel for the low dose. For the high dose sample, hardly any peak is observed.

For more details see [6]

Conclusions

Radiation-induced defect production in W 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). 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. For the first time for W, molecular dynamics (MD) simulations of overlapping cascades were used as input. With MD, a very good agreement between the simulated and experimental spectra was obtained for the sample prepared at a lower dose. 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 [5].

NRA-C using a ^{3}He probing beam was measured for displacement-damaged W where the created defects were before decorated with D in order to determine the position of D at defects. Maximum signal was obtained in the <100> axial channel and in the (110) planar channel. RBSADEC code was upgraded in order to model the NRA-C spectra [Jin et al. Phys. Rev. Mater. 2024]. RBSADEC simulations in combination with density functional theory are in progress to study the position of hydrogen in vacancies and vacancy clusters is in progress. A first comparison indicates that D is positioned close to tetrahedral sites.

References

[1] Feldman et al., Academic Press, San Diego, (1982), pp. 88–116.
[2] Hu et al., J. Nucl. Mater. 556, (2021) 153175.
[3] Cabstanjen, Phys. Stat. Sol. 59, (1980) 11–26.
[4] Zhang et al., Phys. Rev. E 94, (2016) 043319.
[5] Markelj et al., Acta Materialia 263 (2024) 119499.
[6] Markelj et al., Nucl. Mater. Energ. 39 (2024) 101630.

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