RaDIATE Collaboration
Radiation Damage In Accelerator Target Environments

# Experimental Investigation of Irradiation Effects in Beryllium Beam Window after Exposure in the NuMI Beamline: Preliminary Results and Plans

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Rutherford Appleton Laboratory

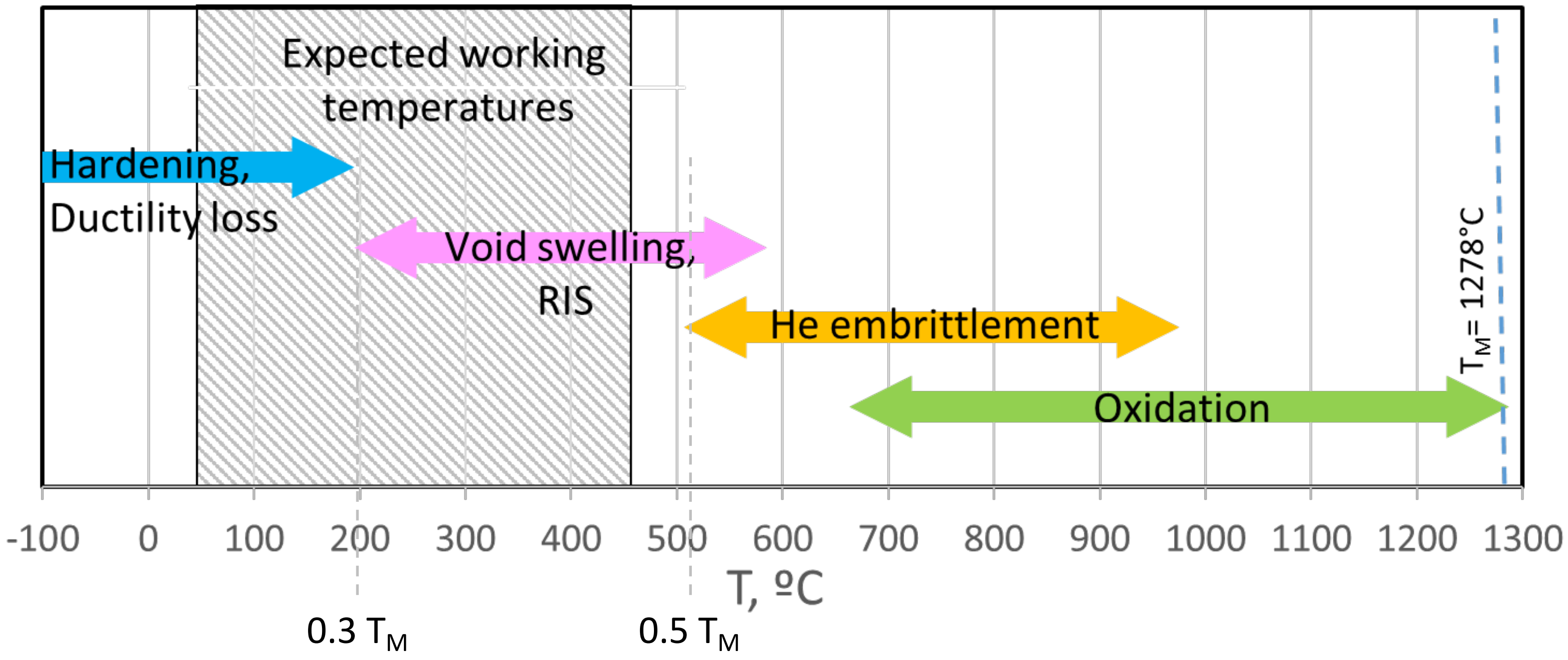
13th International Workshop on Spallation Materials Technology
3rd of November 2016

**Beryllium**

- is extensively used as a material of neutrino target parts, for example as beam windows;
- is a promising candidate for future high-power neutrino sources

**Expected working conditions for some parts of LBNF**

| Application | Operating conditions | | | | | Proton beam parameters |
|---|---|---|---|---|---|---|
| | Avg. T (°C) | Peak T (°C) | Total DPA | Gas production (appm/DPA) He | Gas production (appm/DPA) H | |
| Beam window (vacuum to air) | 200 | 300 | ~ 0.23/yr | >2000 | >2000 | 700 kW; 120 GeV; ~1 Hz; $\sigma_{rms}$ = 1.3 mm |
| Target | 375 | 450 | ~ 0.23/yr | >2000 | >2000 | 700 kW; 120 GeV; ~1 Hz; $\sigma_{rms}$ = 1.3 mm |




**Possible irradiation effects:**
- *embrittlement*
- *irradiation induced hardening*
- *swelling*
- *reduction of thermal conductivity*

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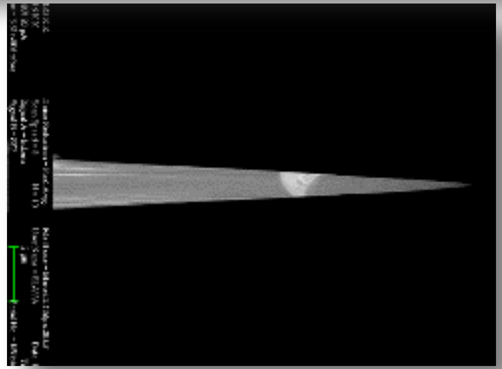
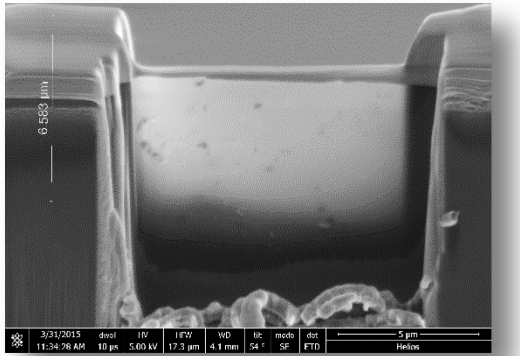
# Experimental investigation



- Investigation of the as-received and high-energy proton irradiated beryllium
- Ion irradiation experiments – first results

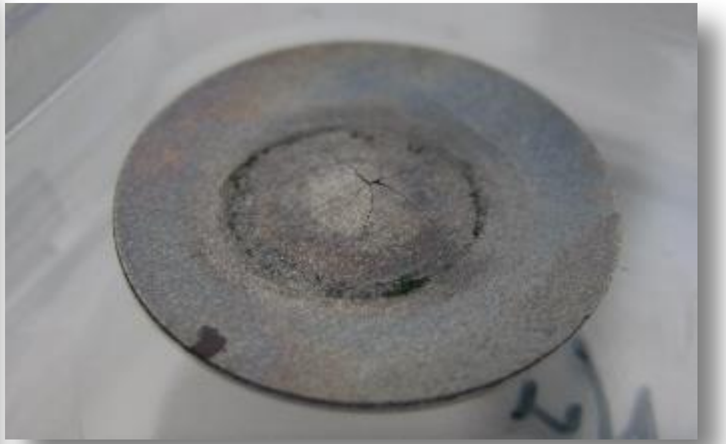



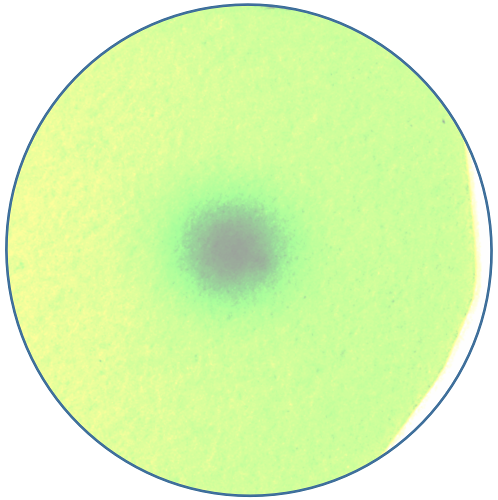
# NuMI beam window experiments

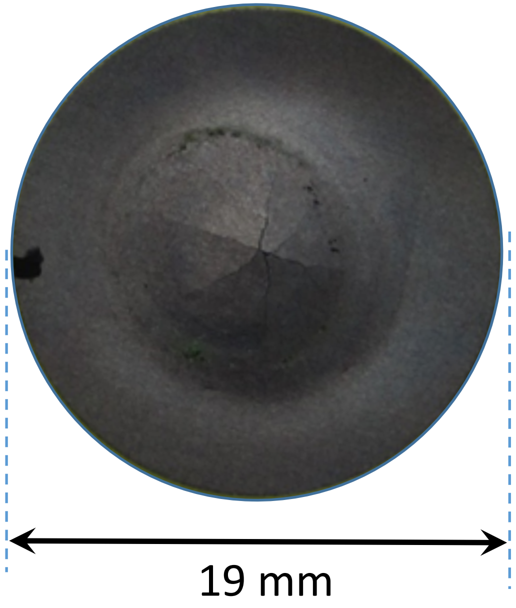
**300 kW NuMI beam window**

120GeV proton beam

- about $3\times10^{13}$ protons per pulse, 0.5 Hz
- $1.57\times10^{21}$ protons during its lifetime
- 1.1mm beam sigmas, X and Y
- T ≈ 50°C
- Up to 0.5 dpa

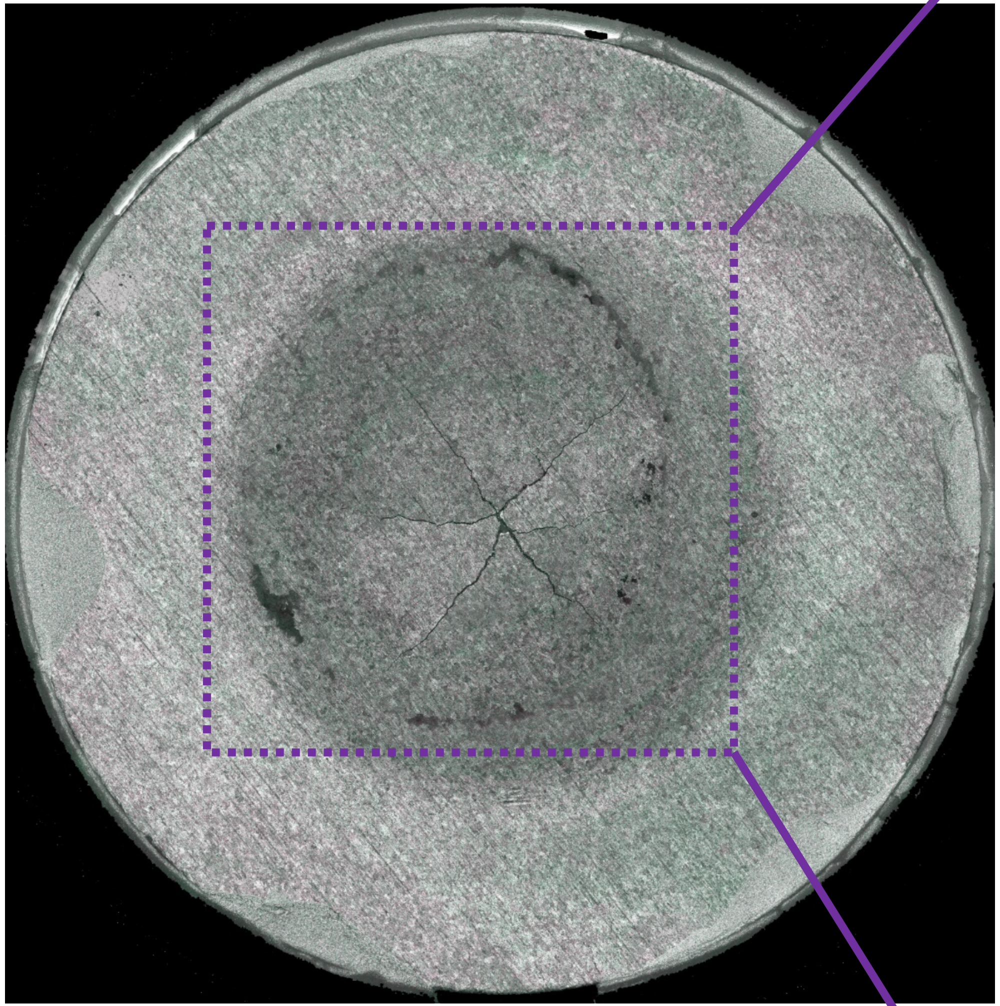


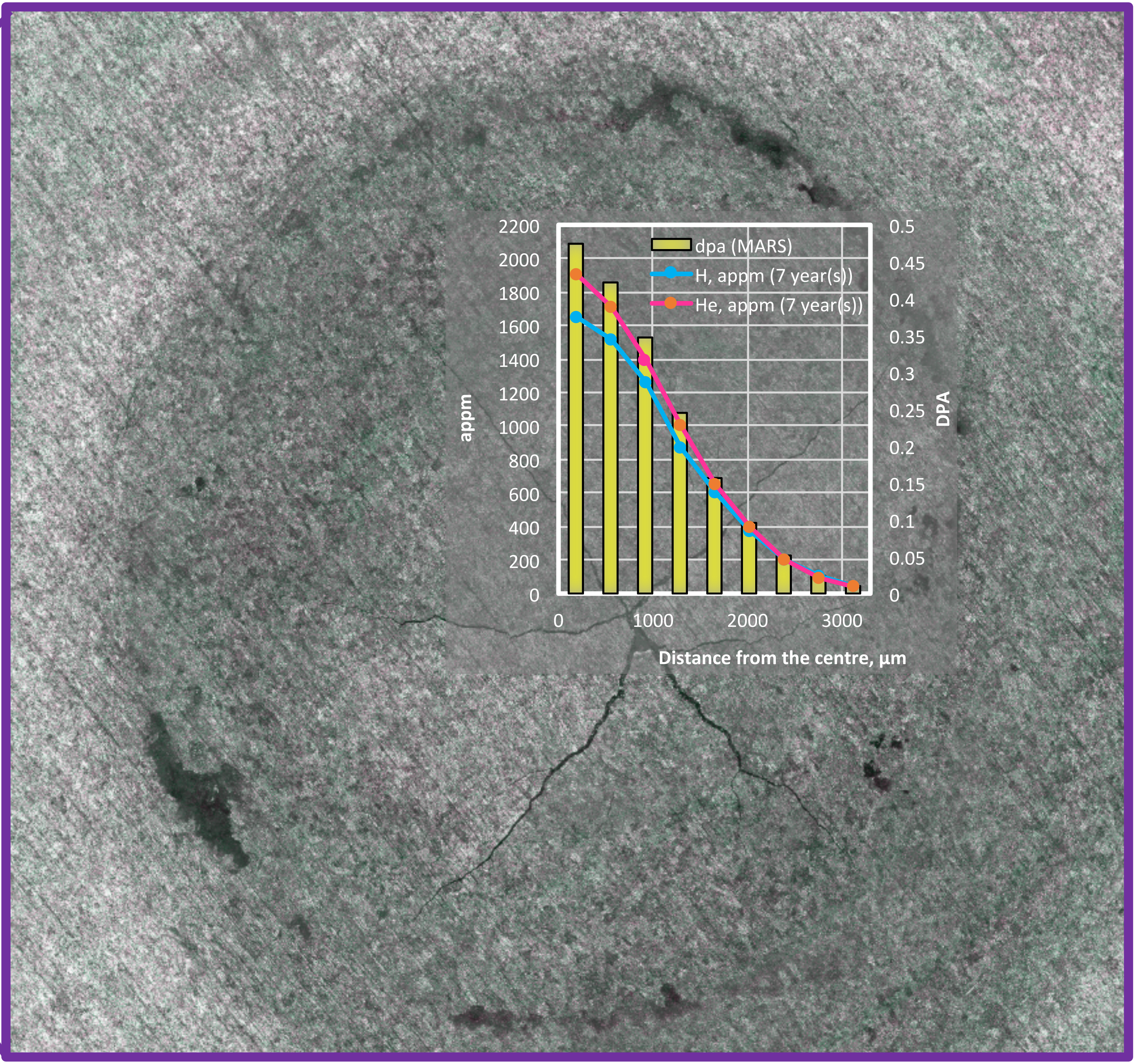





| | Approximate He production in beryllium, appm/dpa |
|---|---|
| SM-3 high-flux reactor (Russia) | 330 |
| BOR-60 reactor (Russia) | 280 |
| HFR, HIDOBE-01 irradiation campaign (Petten, Netherlands) | 160 |
| Beryllium reflectors in the ISIS neutron source (RAL, UK) | 220 (TS1) |
| | 110 (TS2) |
| DEMO fusion reactor | 600 |
| NuMI beam window (FNAL, USA) | 4000 |

# NuMI beam window: MARS data for dose and transmutation





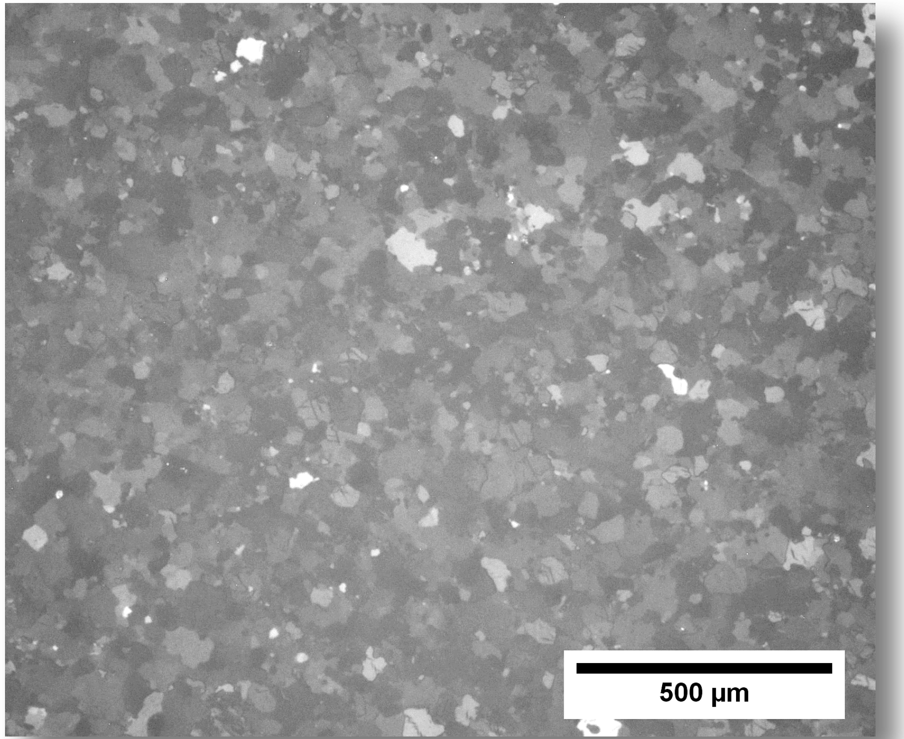

Considerable variation of “dpa” and transmutants production is likely to produce non-homogeneous changes across the surface of Be window.

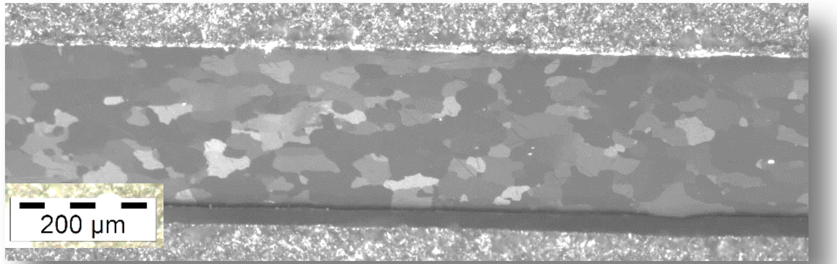
# Materials

## PF-60

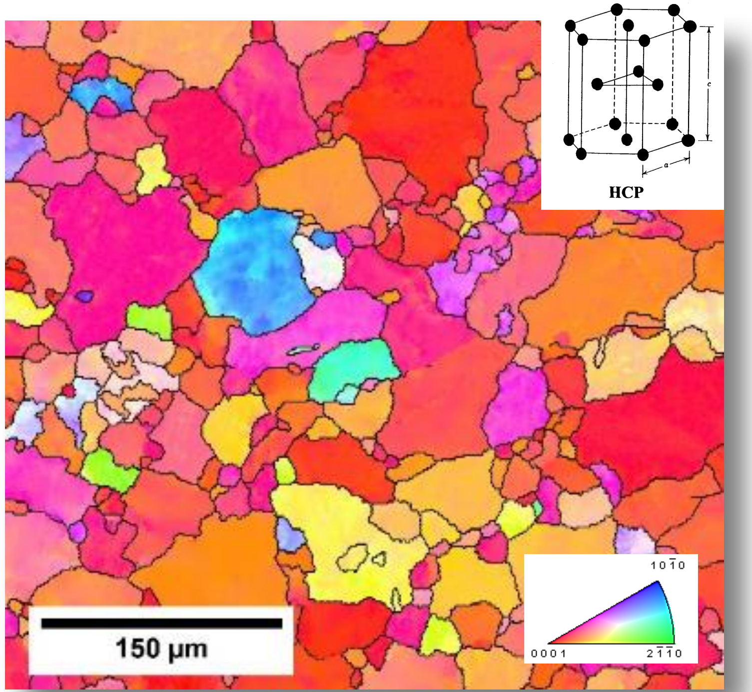
| | Max impurities, appm |
|---|---|
| Al | 170 |
| C | 450 |
| Fe | 130 |
| Mg | 810 |
| O | 2900 |
| Si | 130 |
| N | 195 |
| Be | balance |

Polarised-light optical microscopy images

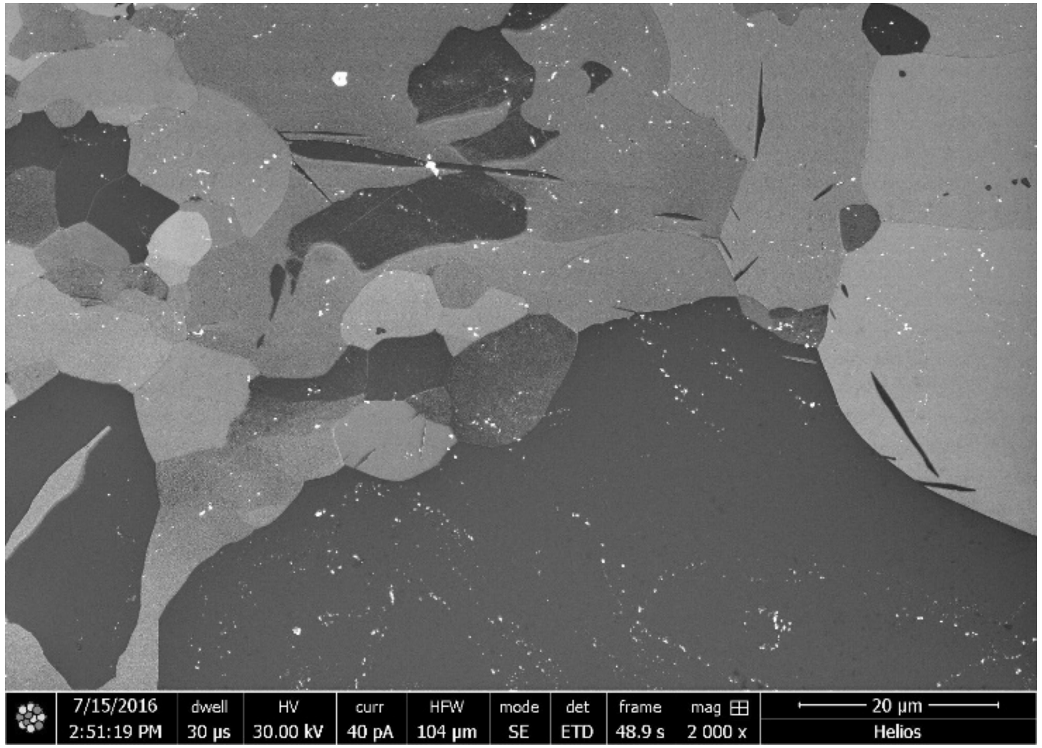



Surface normal-projected inverse pole figure orientation map, with high angle grain boundaries

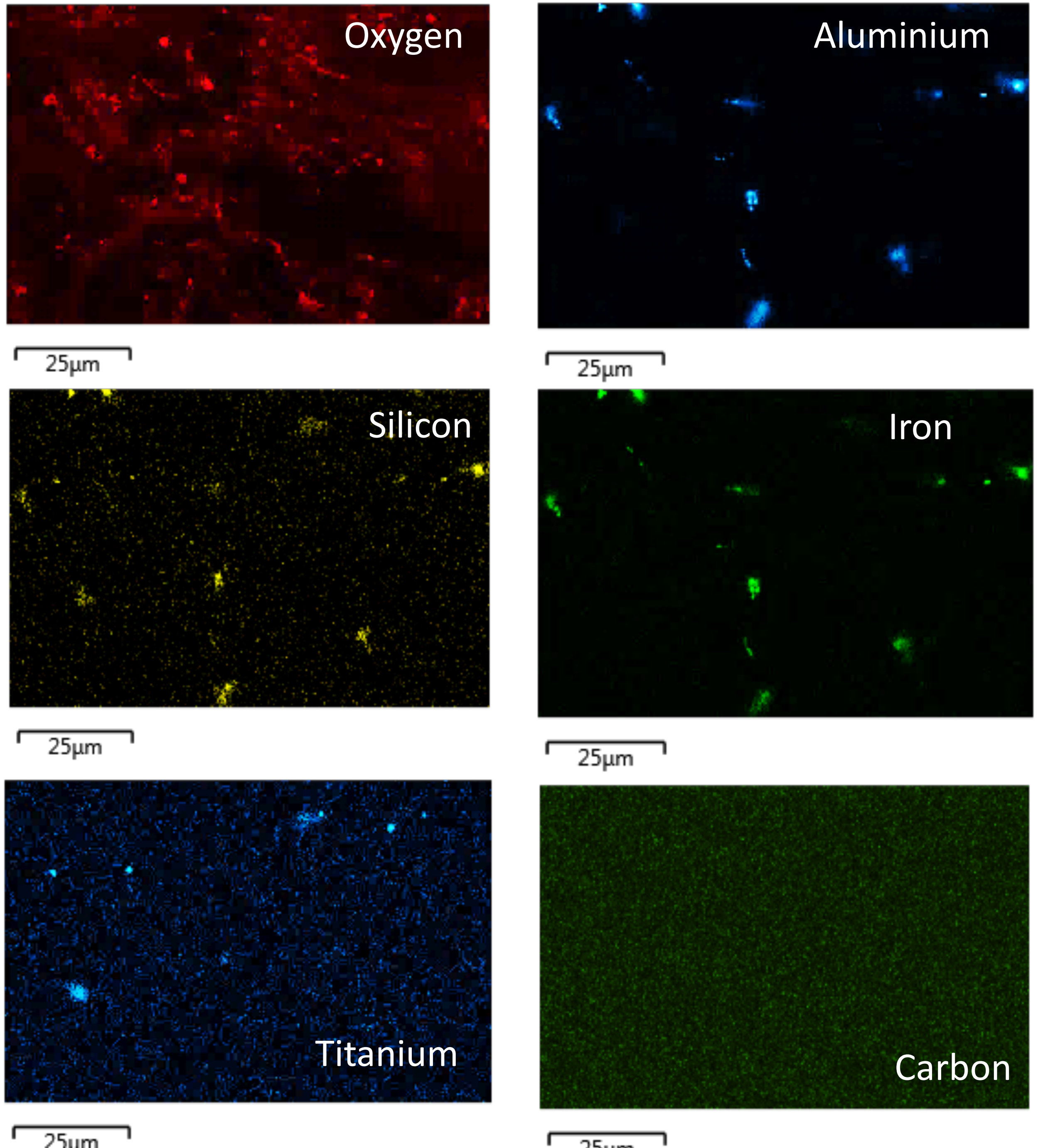



- Beryllium contains a lot of inpurities;
- the material has a strong (0001) texture

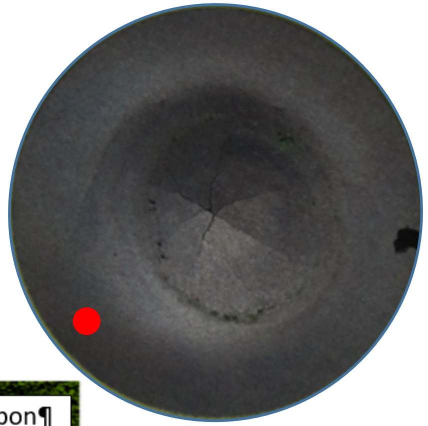



PF-60/cross rolled

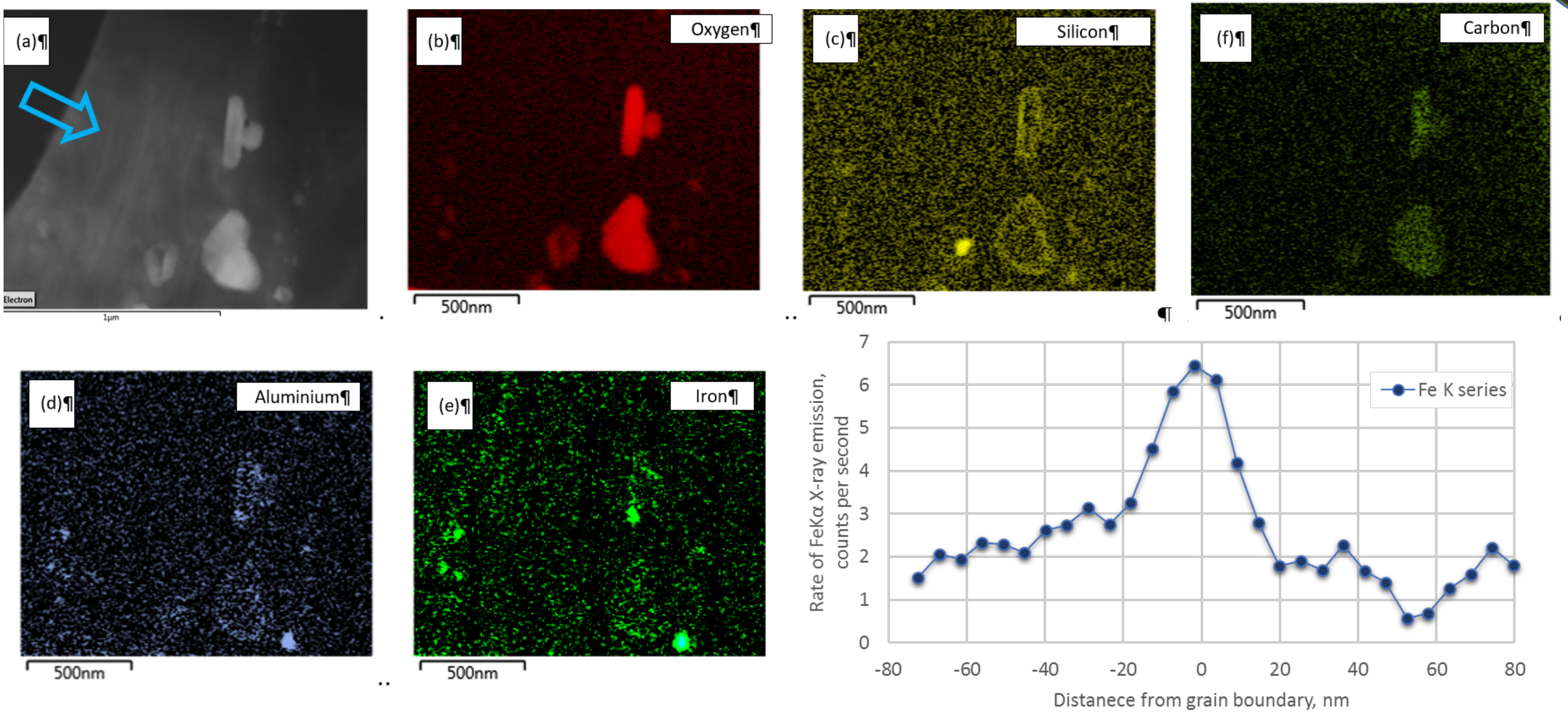
- Industrial beryllium is a non-homogeneous material.
- “White contrast particles” on SEM images (in the SE2 detector) are mainly inside grains



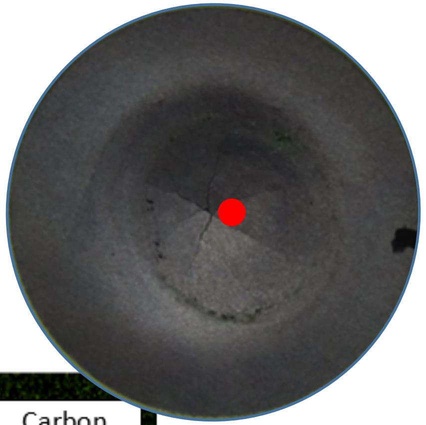
# STEM image of a near-grain boundary area in unirradiated PF-60 beryllium from the periphery of the NuMI beam window.

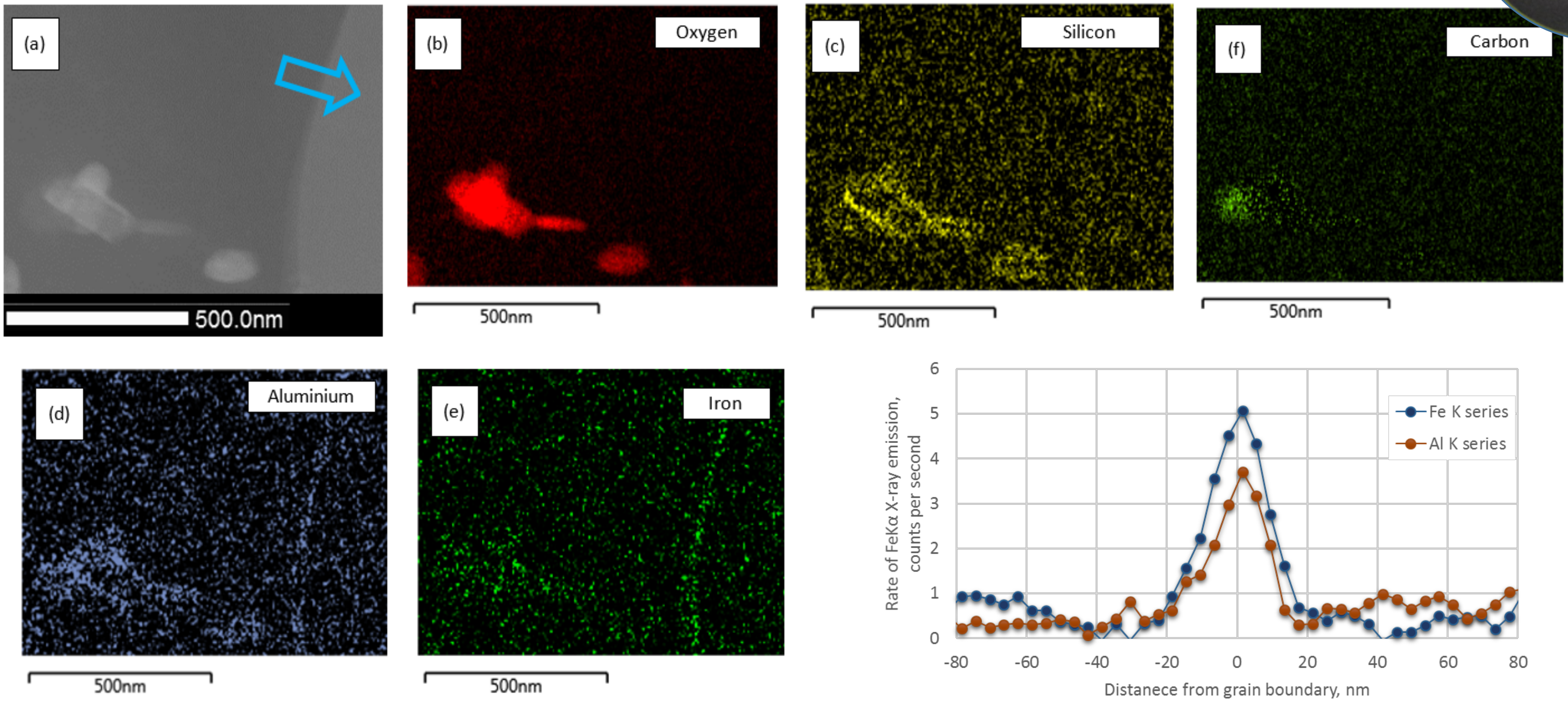


PF-60/cross rolled

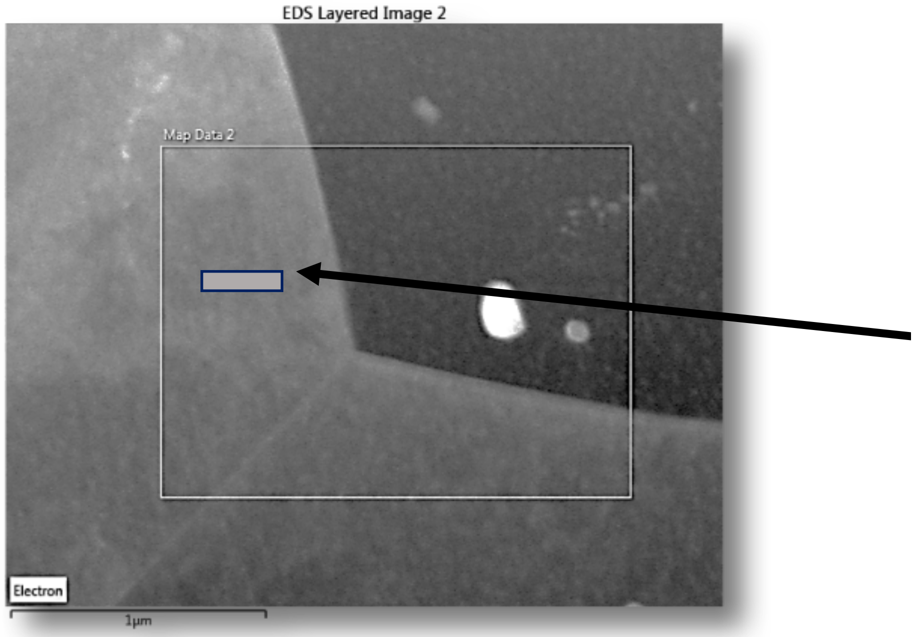


# STEM image of a near-grain boundary area i n radiated (0.45 dpa) PF-60 beryllium from the periphery of the NuMI beam window.

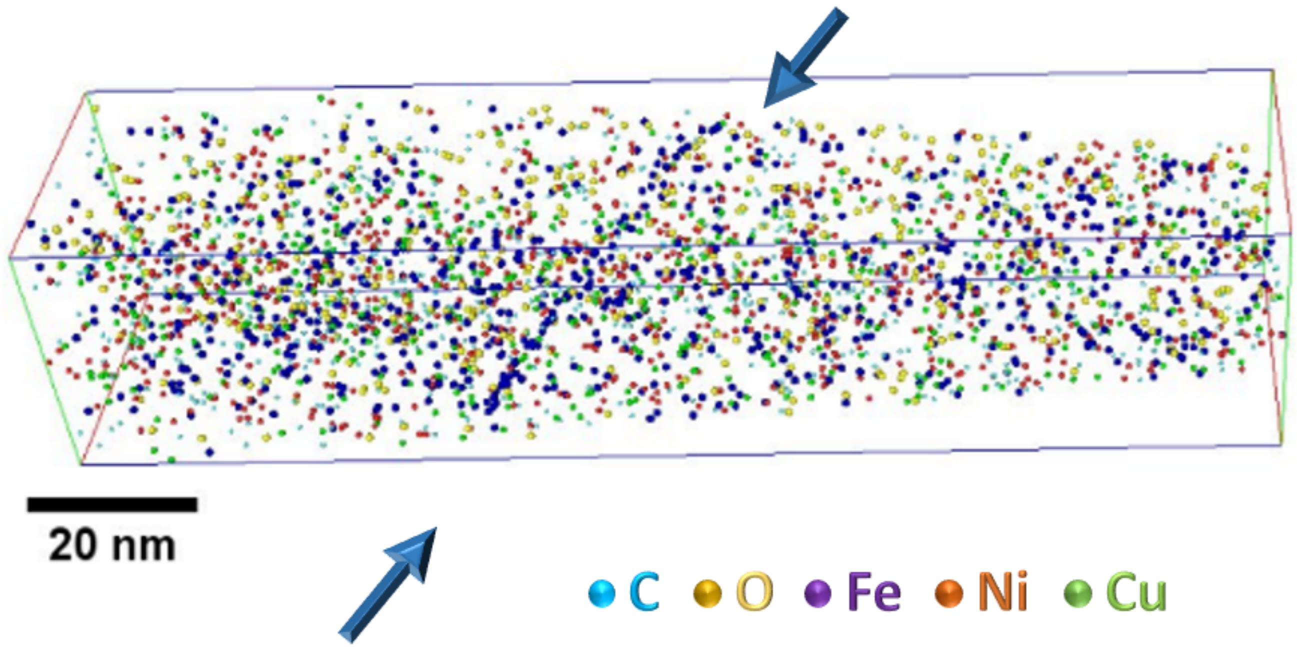




# APT of beryllium matrix in unirradiated PF-60 beryllium from the periphery of the NuMI beam window.

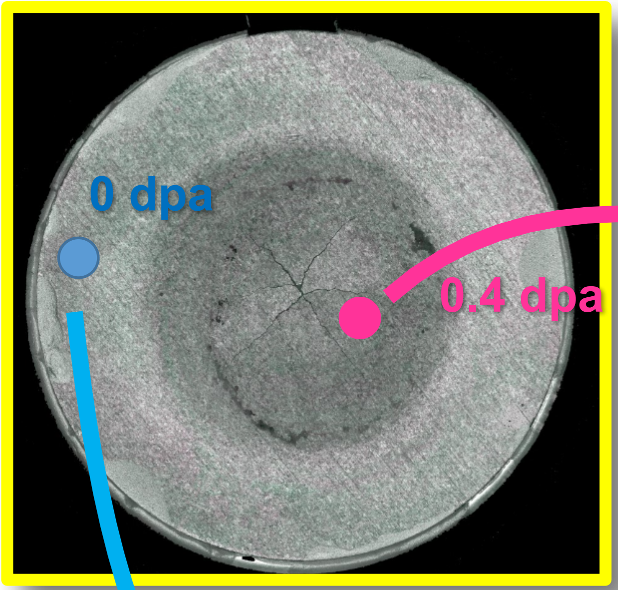


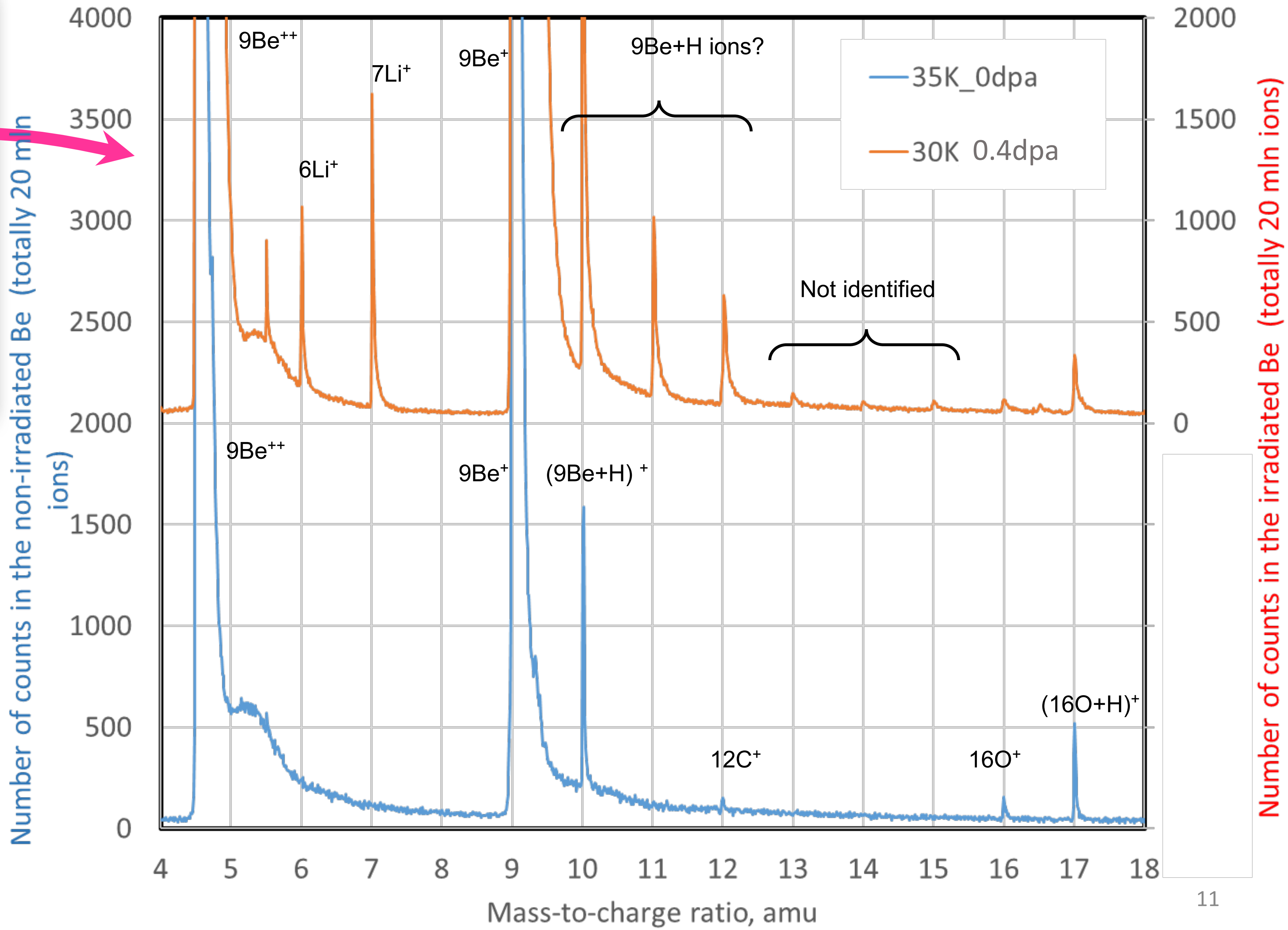

| PF-60 | Max impurities, appm | Impurities in Beryllium matrix (APT), appm |
|---|---|---|
| C | 450 | 15±10 |
| Fe | 130 | 55±10 |
| Ni | 31 | 25±10 |
| Cu | 14 | 15±10 |
| O | 2900 | 70±30 |
| Al | 170 | Not detected (ND) |
| Mg | 810 | ND |
| Si | 130 | ND |
| N | 195 | ND |




- All the detected impurities, except Fe, are randomly distributed in the beryllium matrix.
- a linear segregation of Fe atoms was detected, and is probably an atmosphere around a dislocation line

# APT of beryllium matrix in irradiated and non-irradiated beryllium

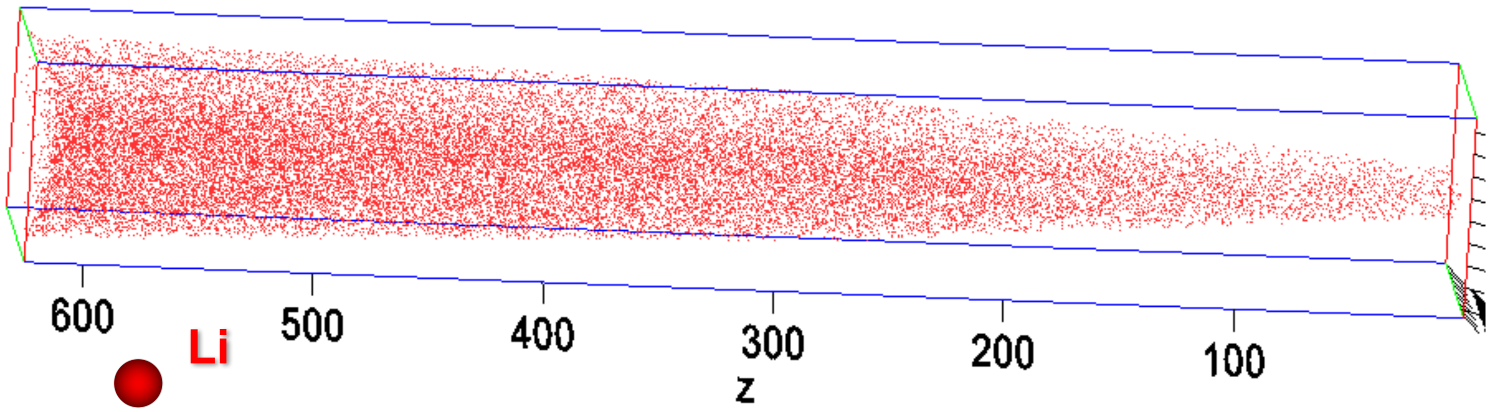



- New peaks on mass-spectrum – transmutation products

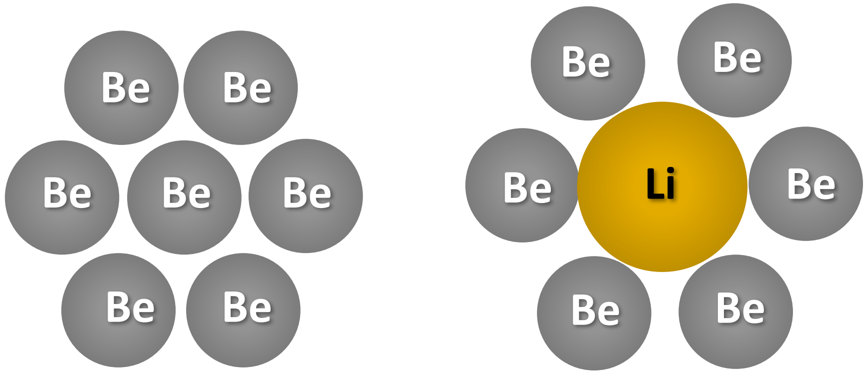
# APT of beryllium matrix in irradiated and non-irradiated beryllium (PF-60): chemical data

| | Max impurities, appm | Impurities in Beryllium matrix (APT), appm | Impurities in Beryllium matrix after p-irradiation 0.41 dpa (APT), appm |
|---|---|---|---|
| C | 450 | 15±10 | Cannot be measured |
| Fe | 130 | 50±10 | 50±10 |
| Ni | 31 | 25±10 | 22±10 |
| Cu | 14 | 15±10 | 15±10 |
| O | 2900 | 70±30 | 90±30 |
| Al | 170 | ND* | Lithium: 400±20 appm |
| Mg | 810 | ND | Others (no hydrogen): 170±40 appm |
| Si | 130 | ND | |
| N | 195 | ND | |




- Lithium becomes the major (solid) impurity in beryllium matrix (ca. 900 appm/dpa)
- Transmutant Li is homogeneously distributed
- Impurities (Fe, Ni, Cu, C, O) are still homogeneously distributed

# APT of beryllium matrix in irradiated beryllium: Lithium




**Atomic radius of Be = 112 pm**
**Atomic radius of Li = 167 pm (49% bigger)**

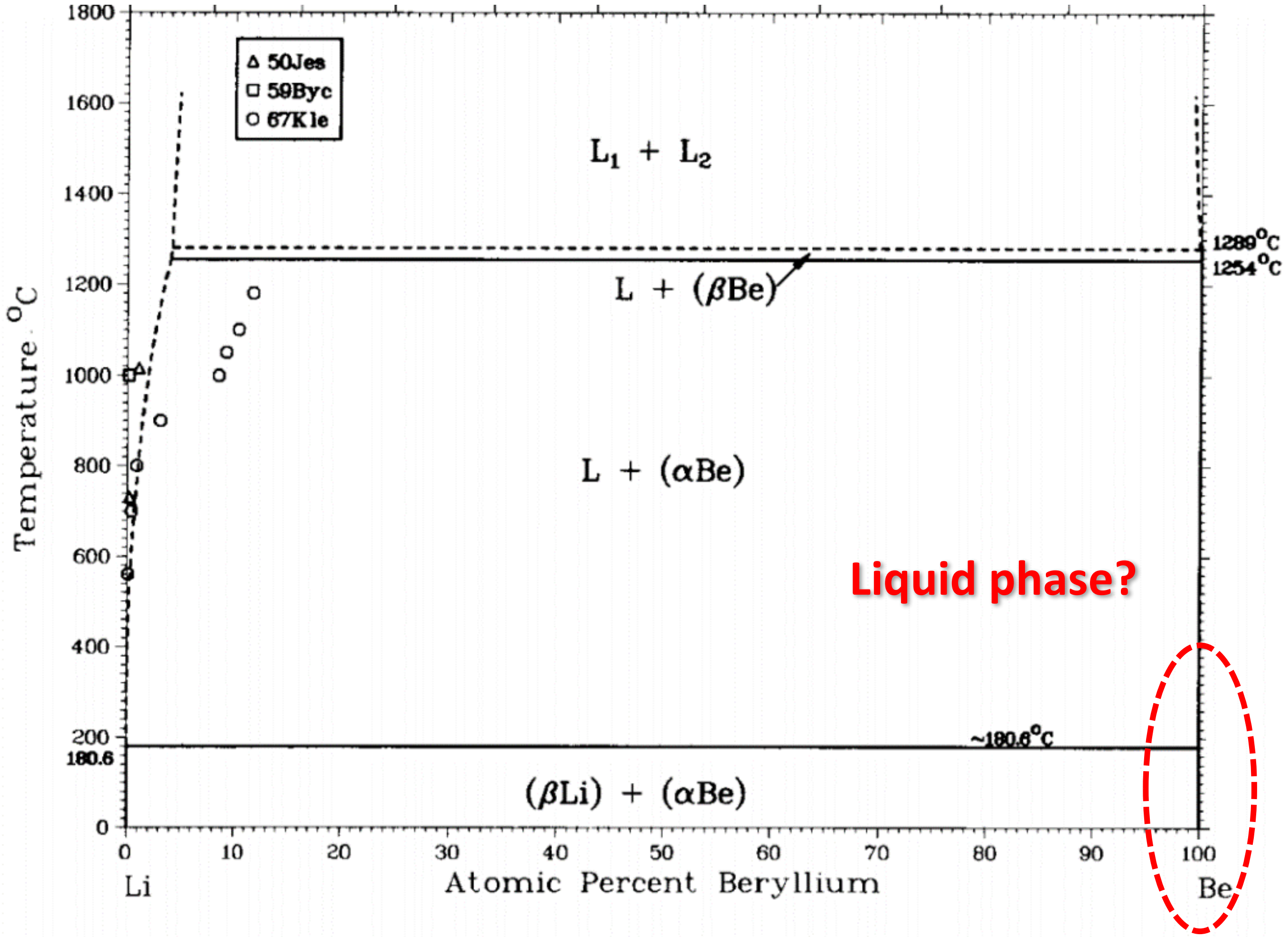
## Solid solution strengthening:

**$\Delta$ CRRS$_{BASAL}$ ≈2 MPa**
**$\Delta$ CRSS$_{PRISMATIC}$ ≈ 2.8 MPa** → **ΔYS ≈11 MPa**

Based on data of Aldinger in
Beryllium Science and Technology., 2012.

Based on Petch&Wright, Proc. R. Soc. Math. Phys. Eng. Sci. 370 (1980)

## Assessed Li-Be Phase Diagram with Selected Experimental Data

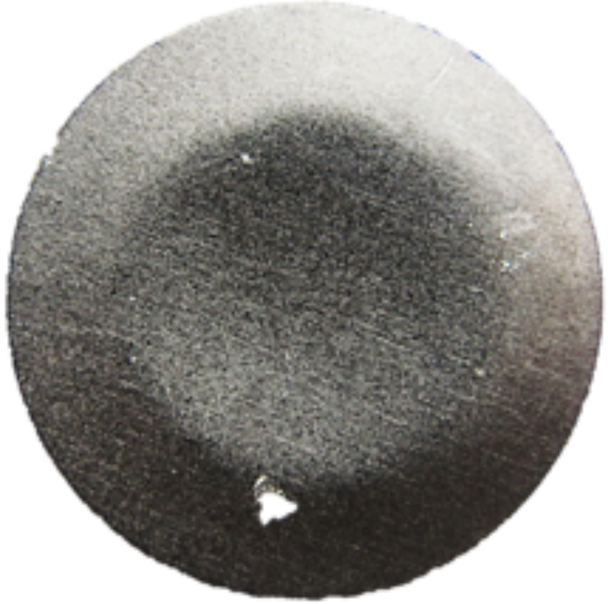


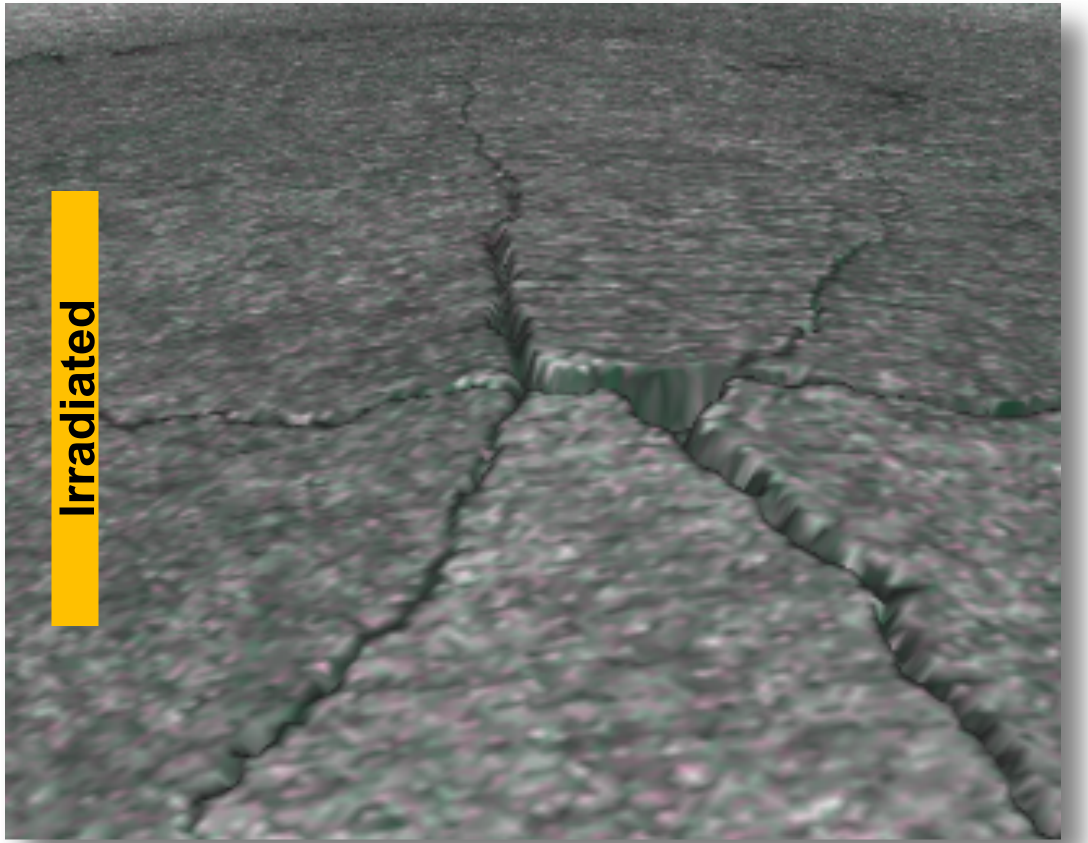

A.D. Pelton. 1985

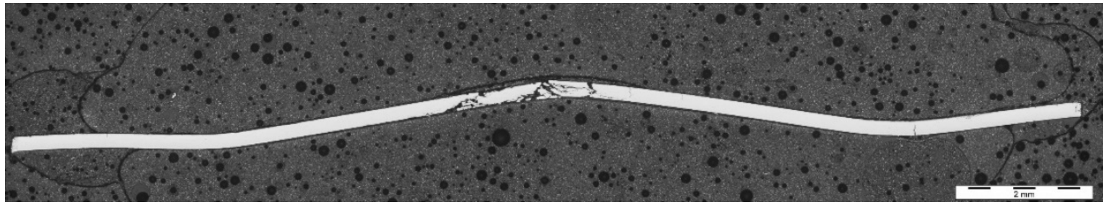
# NuMI beam window experiments

Irradiated

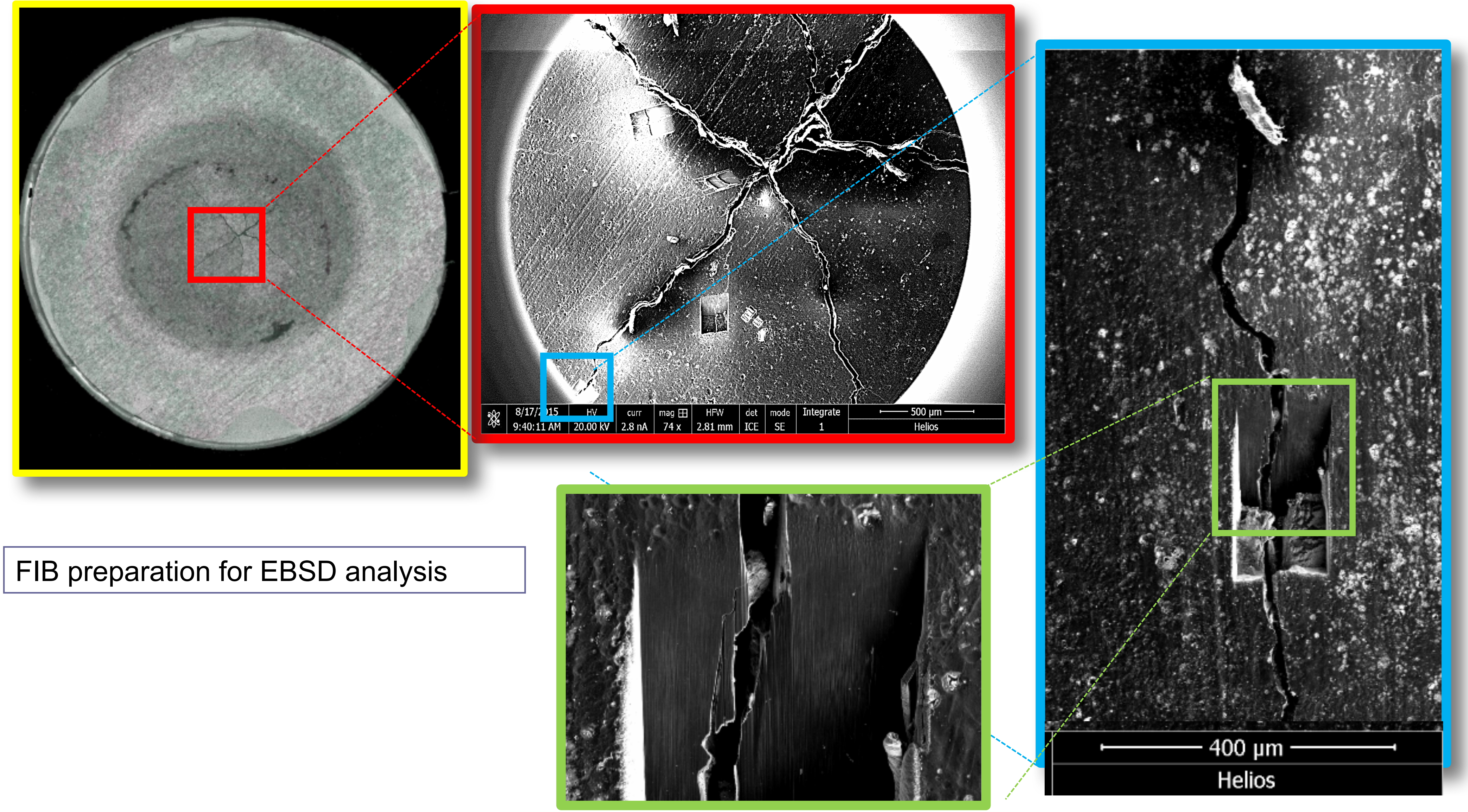
As-received



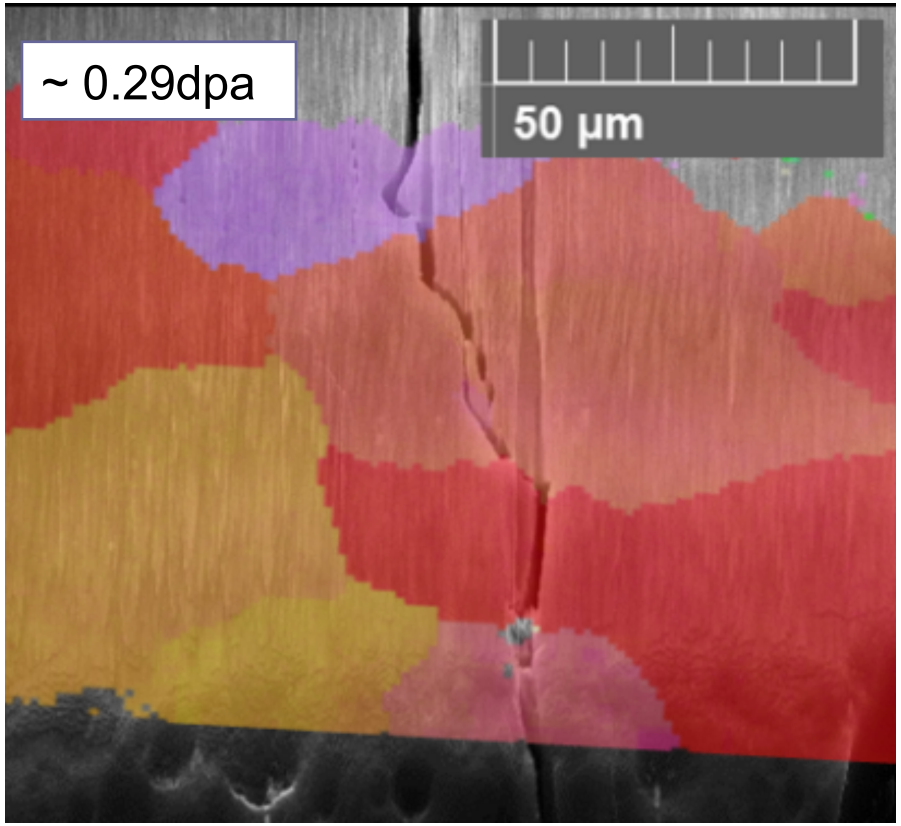


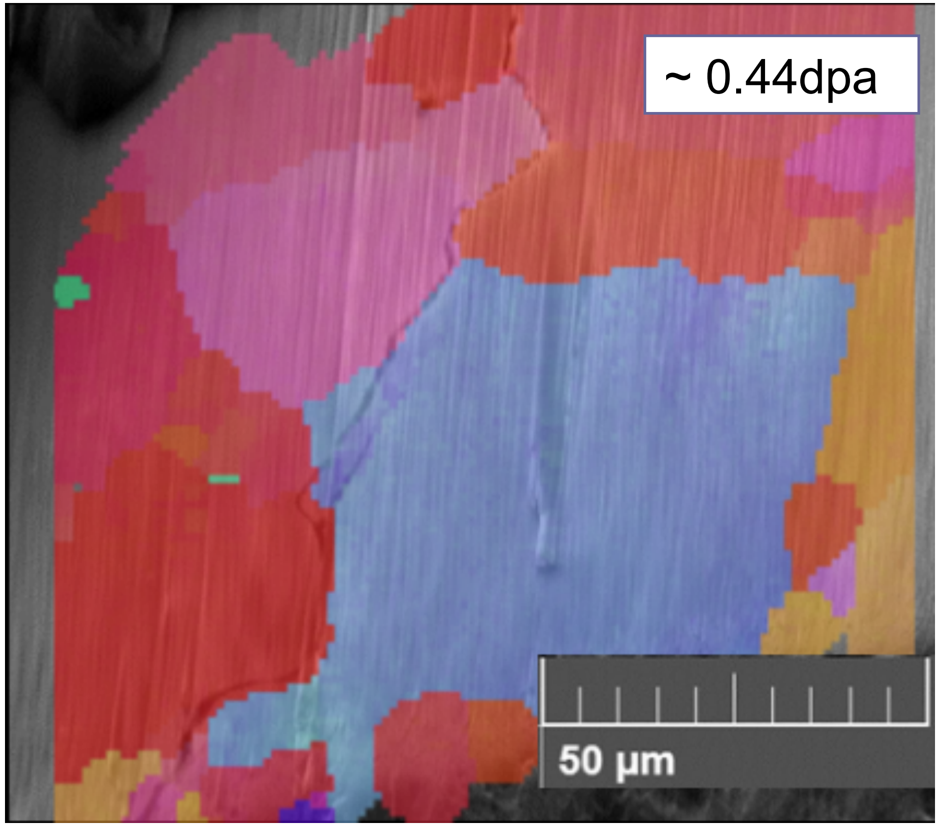



**Radiation induced degradation?**



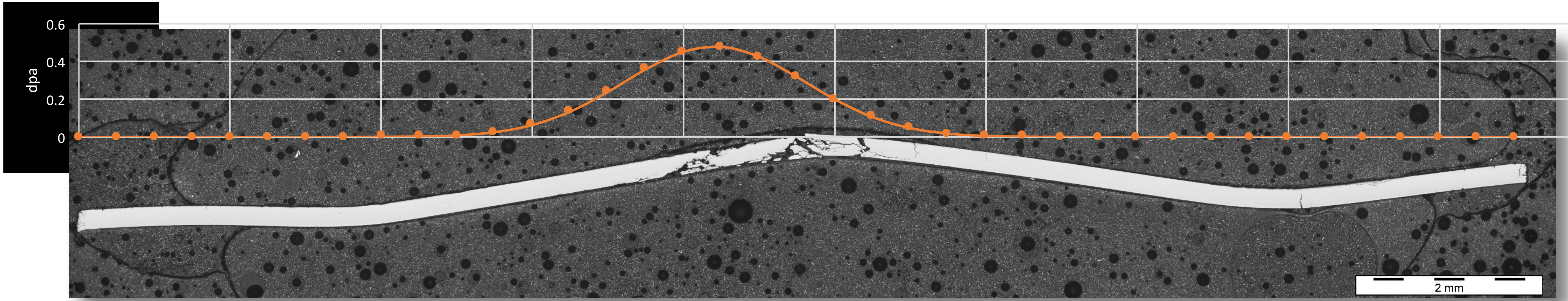

FIB preparation for EBSD analysis

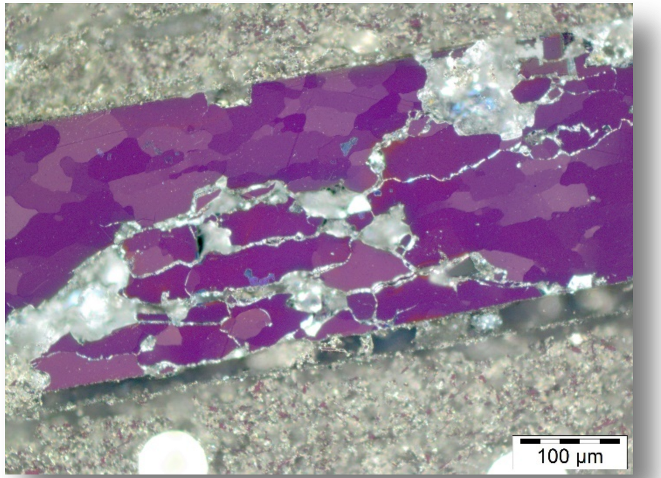


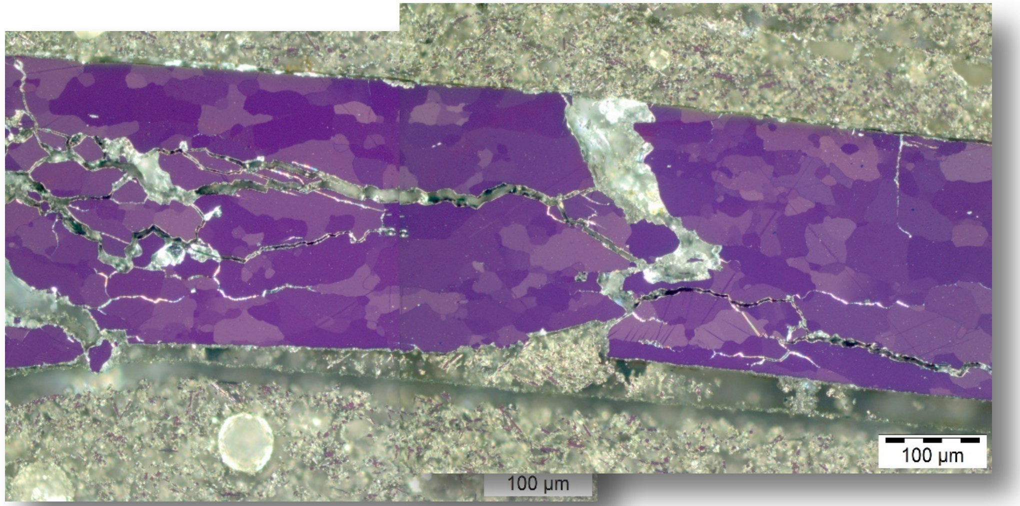



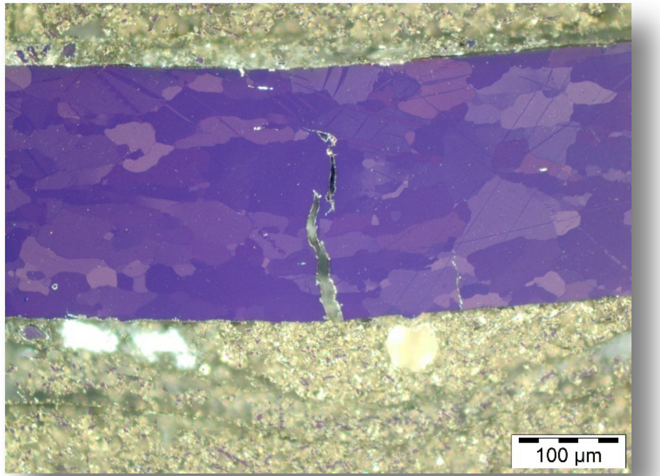

Transition form  transgranular fracture to grain boundary/mixed mode fracture

- Non-irradiated beryllium – mainly transgranular cleavage
- Grain-boundary fracture at low T irradiation may be caused by  strengthening of the matrix or "weakening" of GBs



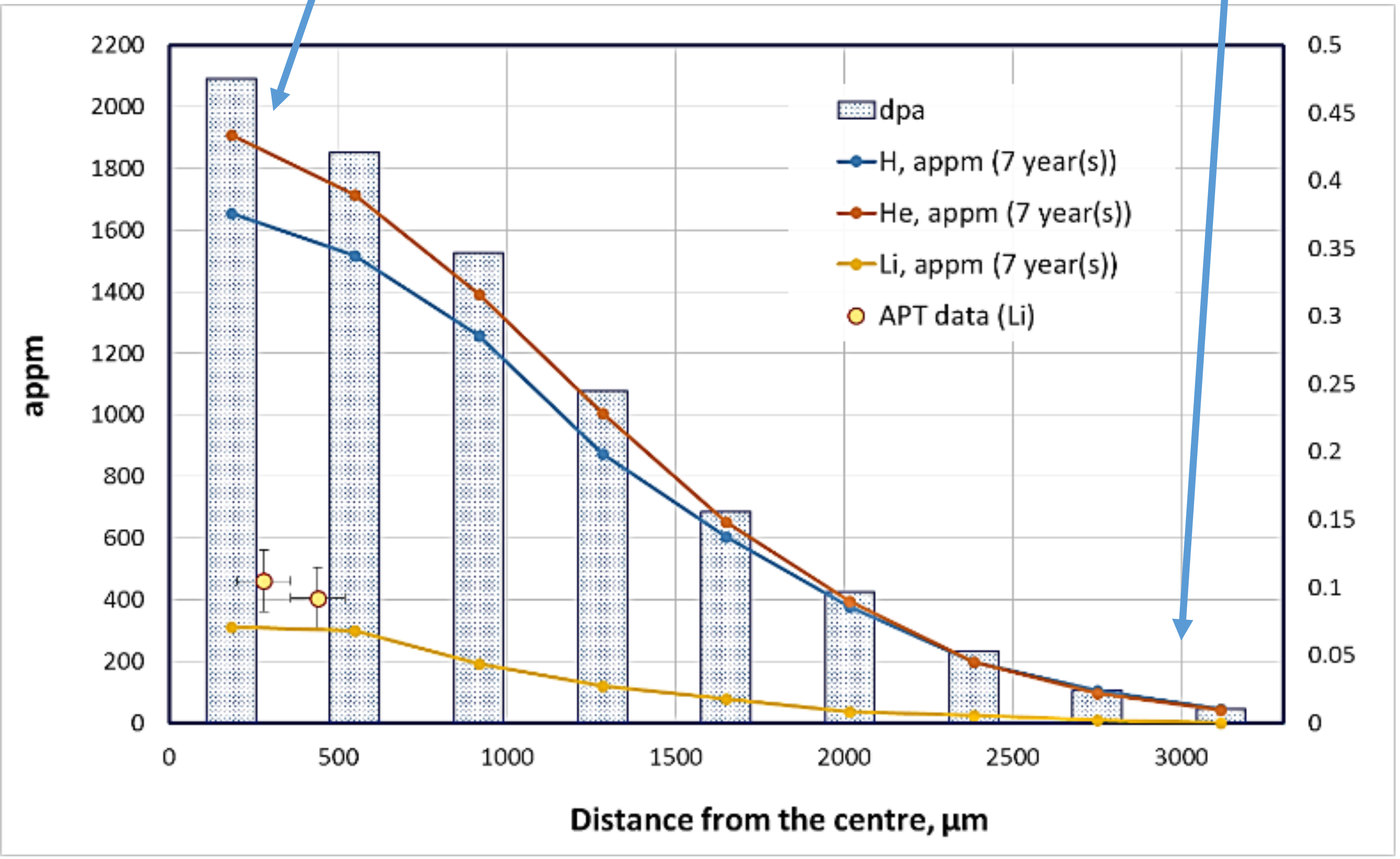



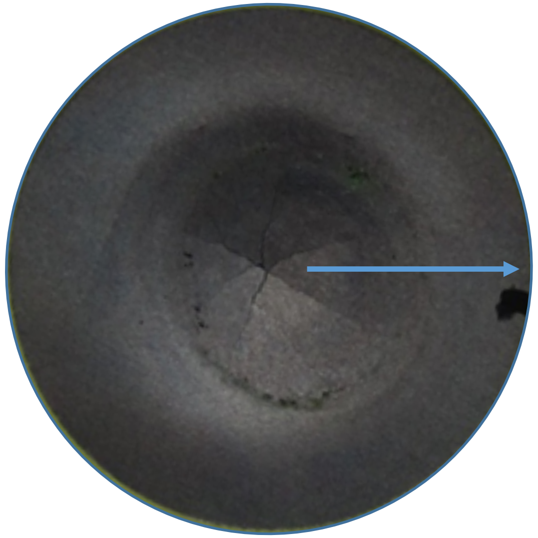




- More data on fracture behaviour will come

GB/mixed fracture?

Transgranular fracture



- Transmutant Li can be one of the reasons of properties degradation under proton irradiation
- **But detailed TEM and He/H transmutants distribution analysis is needed**
- **Micromechanical tests should be made**

## He implantation experiments: preliminary results

**Surrey Ion Beam Centre**, UK

**Ions**: He+
**Maximum beam energy**: 2 MeV => 7.5µm implantation depth (SRIM)
**Dose:** up to 1 dpa
**Temperature**: 50ºC and 200ºC

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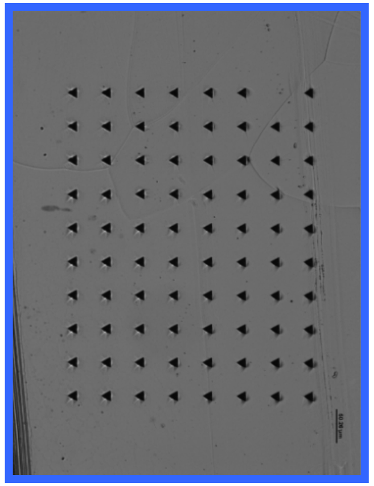
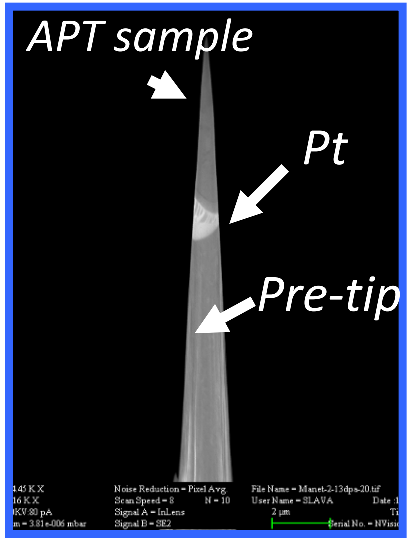
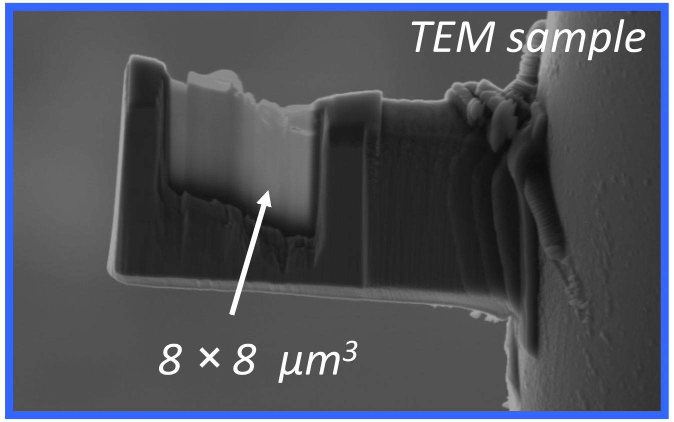
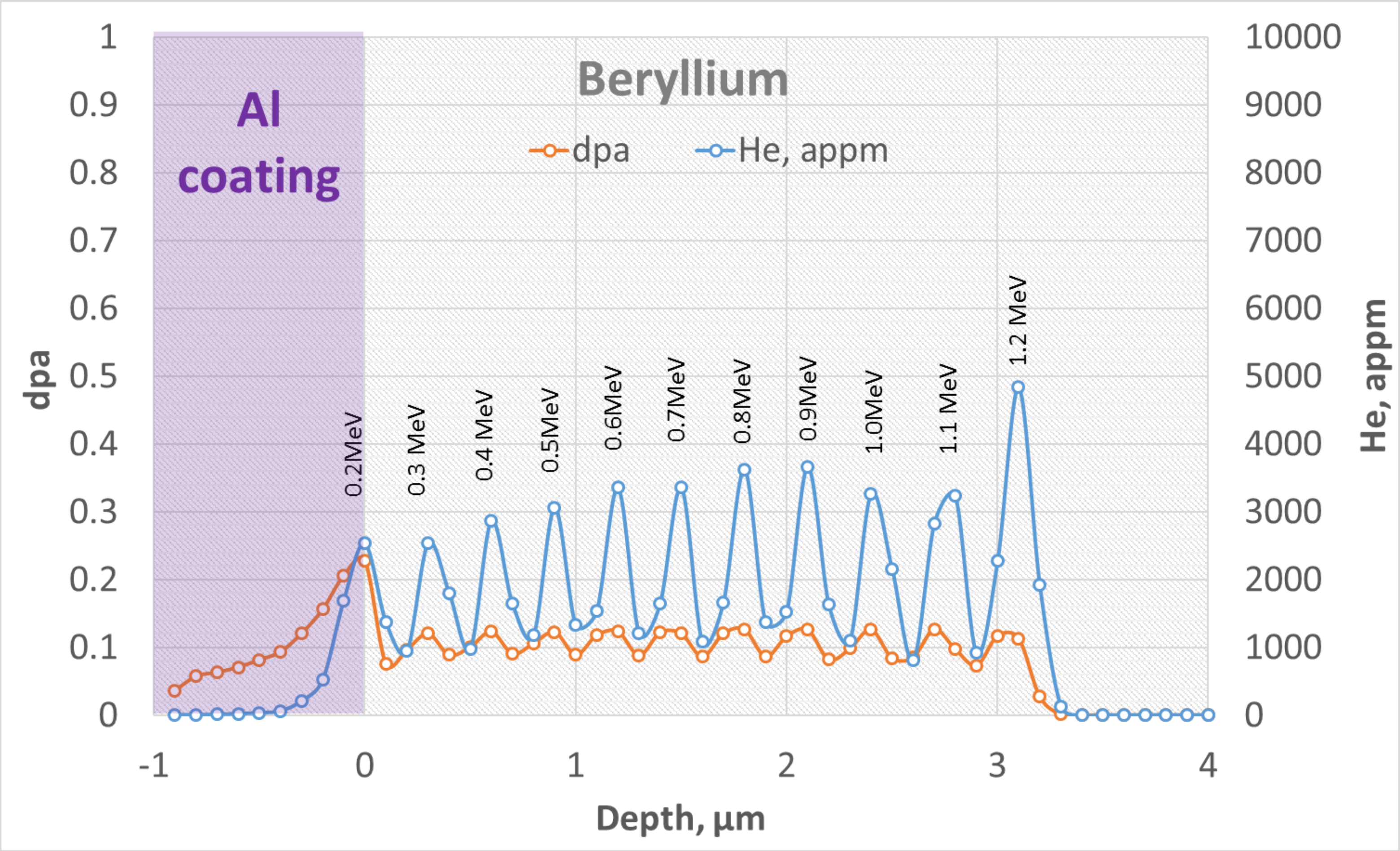
*Micromechanical tests*

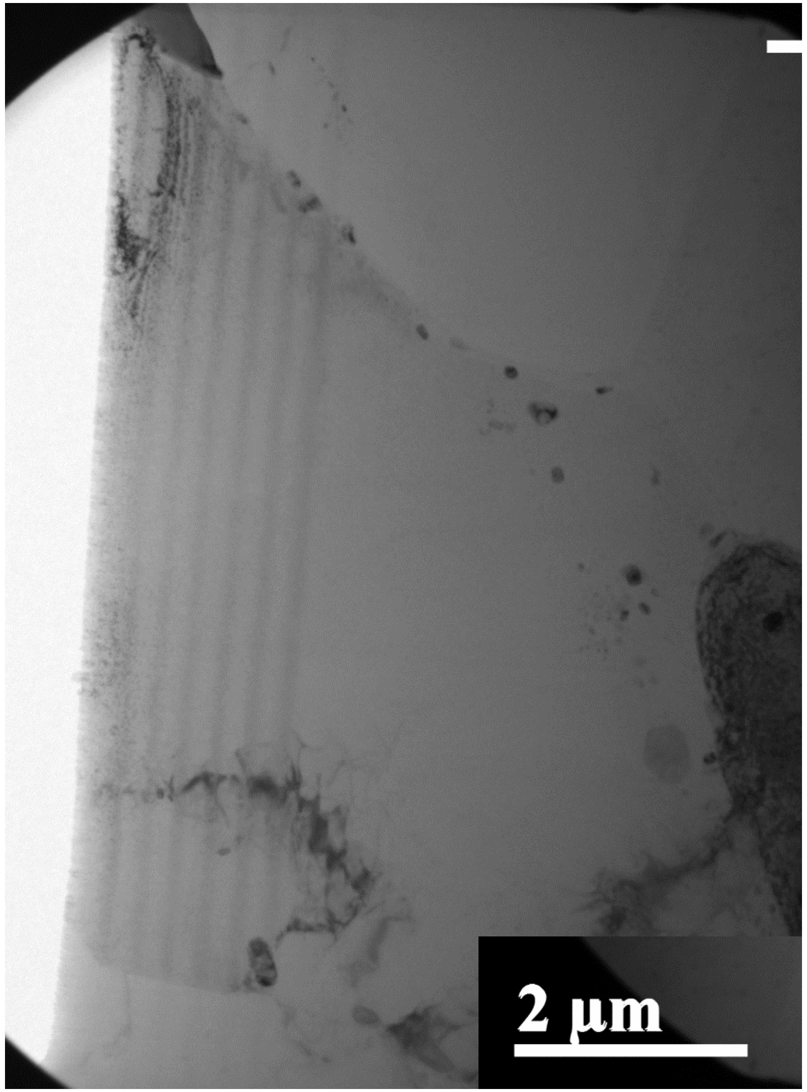




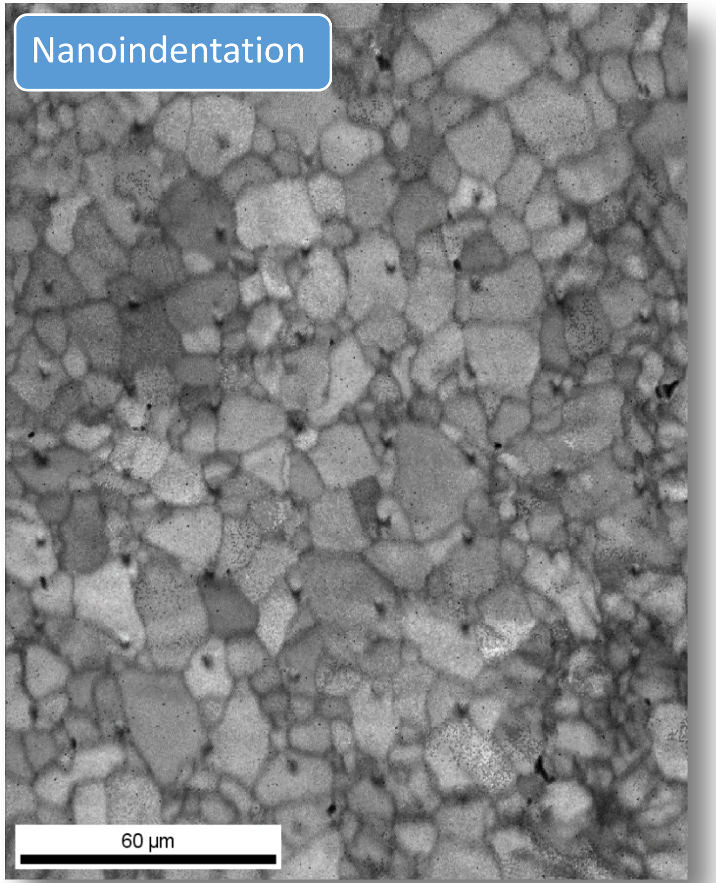



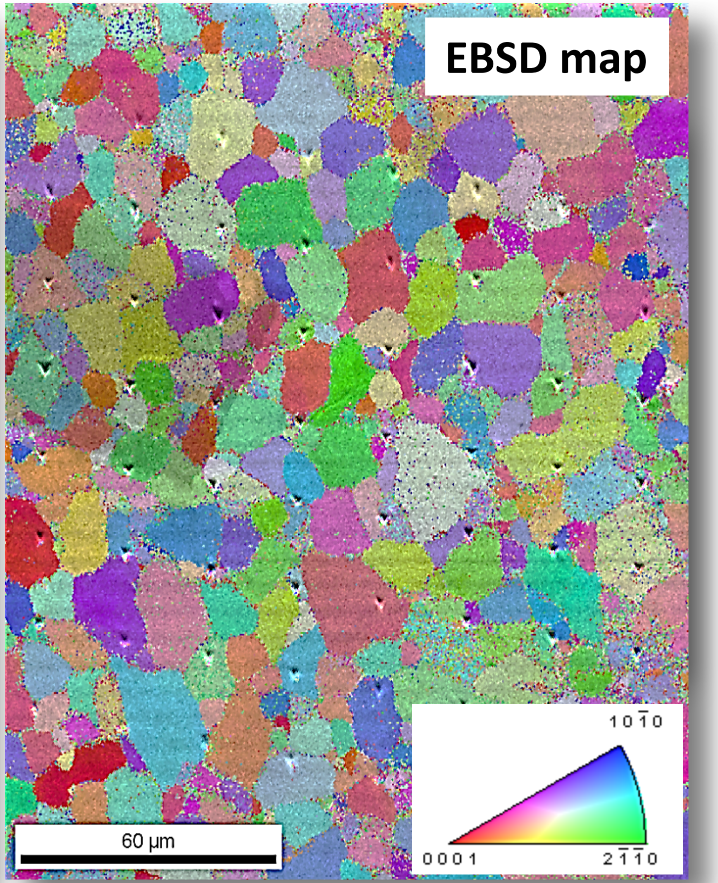
# He implantation in Be through Al degrader (1μm), high energy implantation

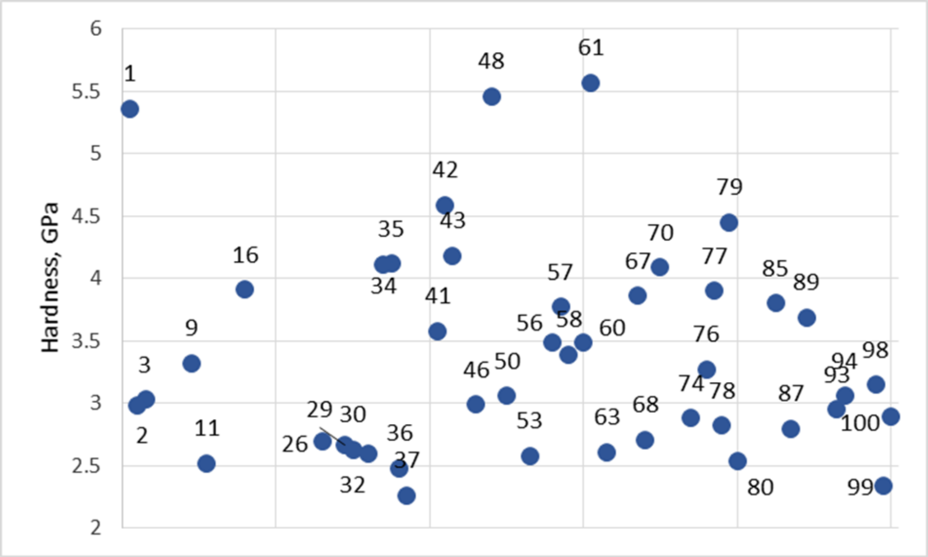


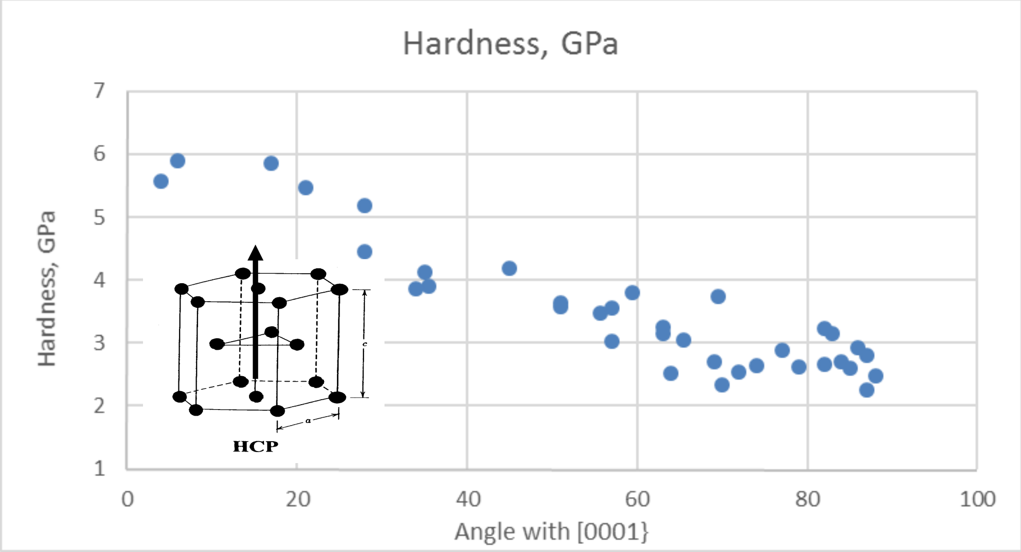




| Steps | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Energy, MeV | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 | 0.7 | 0.8 | 0.9 | 1 | 1.1 | 1.2 |
| He ions/cm$^2$, ×$10^{16}$ | 0.75 | 0.75 | 0.75 | 0.75 | 0.75 | 0.75 | 0.75 | 0.83 | 0.83 | 0.90 | 1.20 |

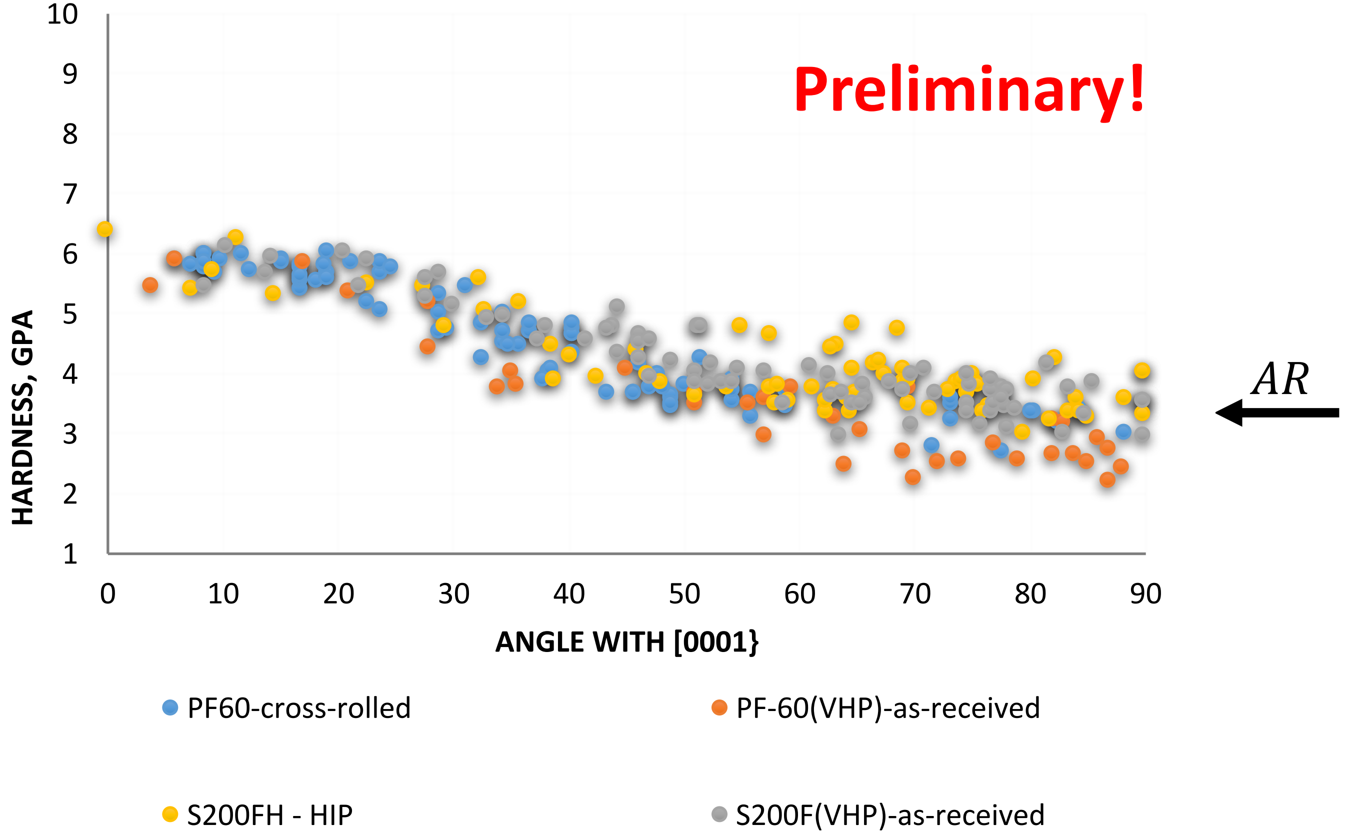


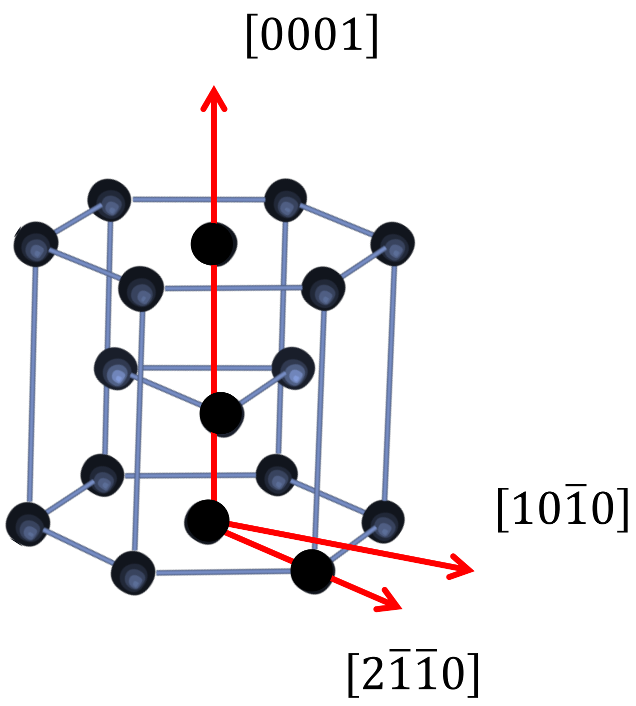



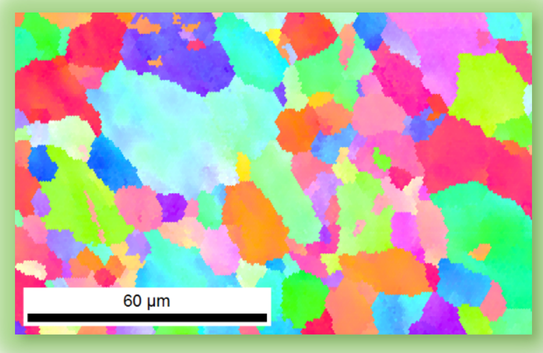




The scattering makes sense… but the anisotropy is impressive

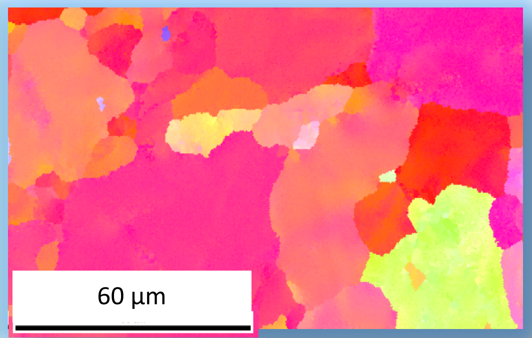


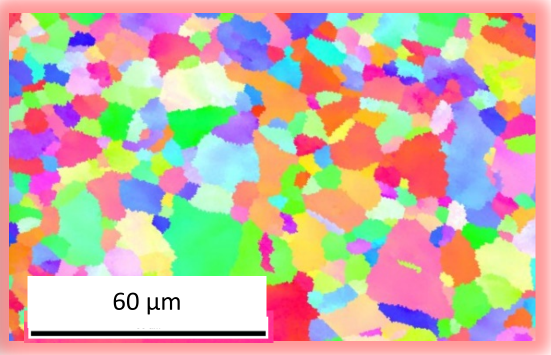

S65F, VHP



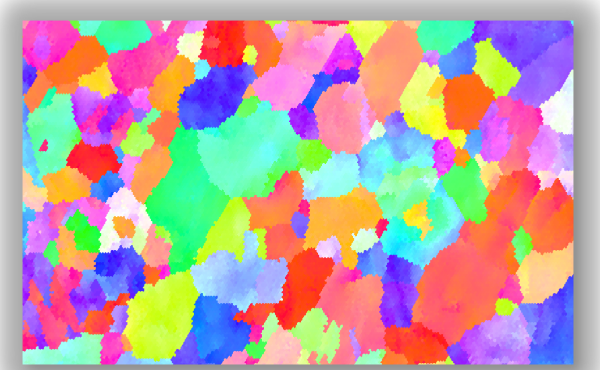

PF-60 (Cross-rolled)

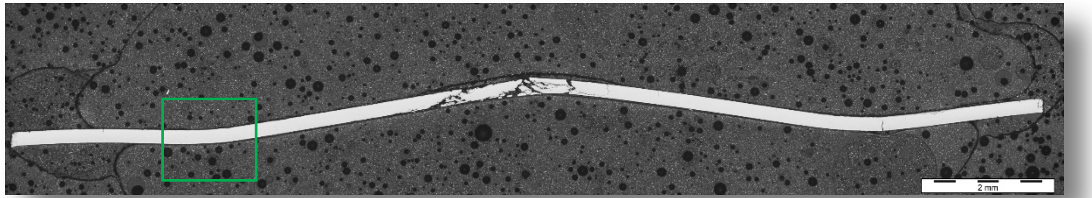


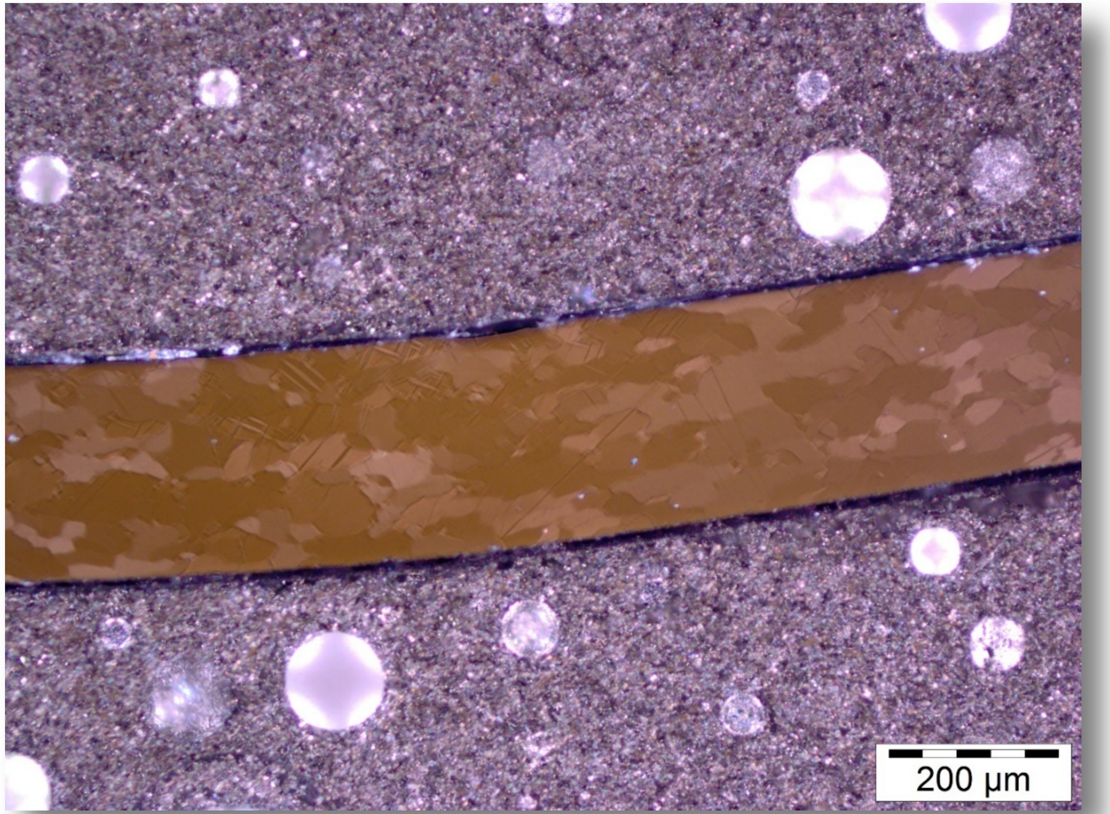

S200FH (HIP)

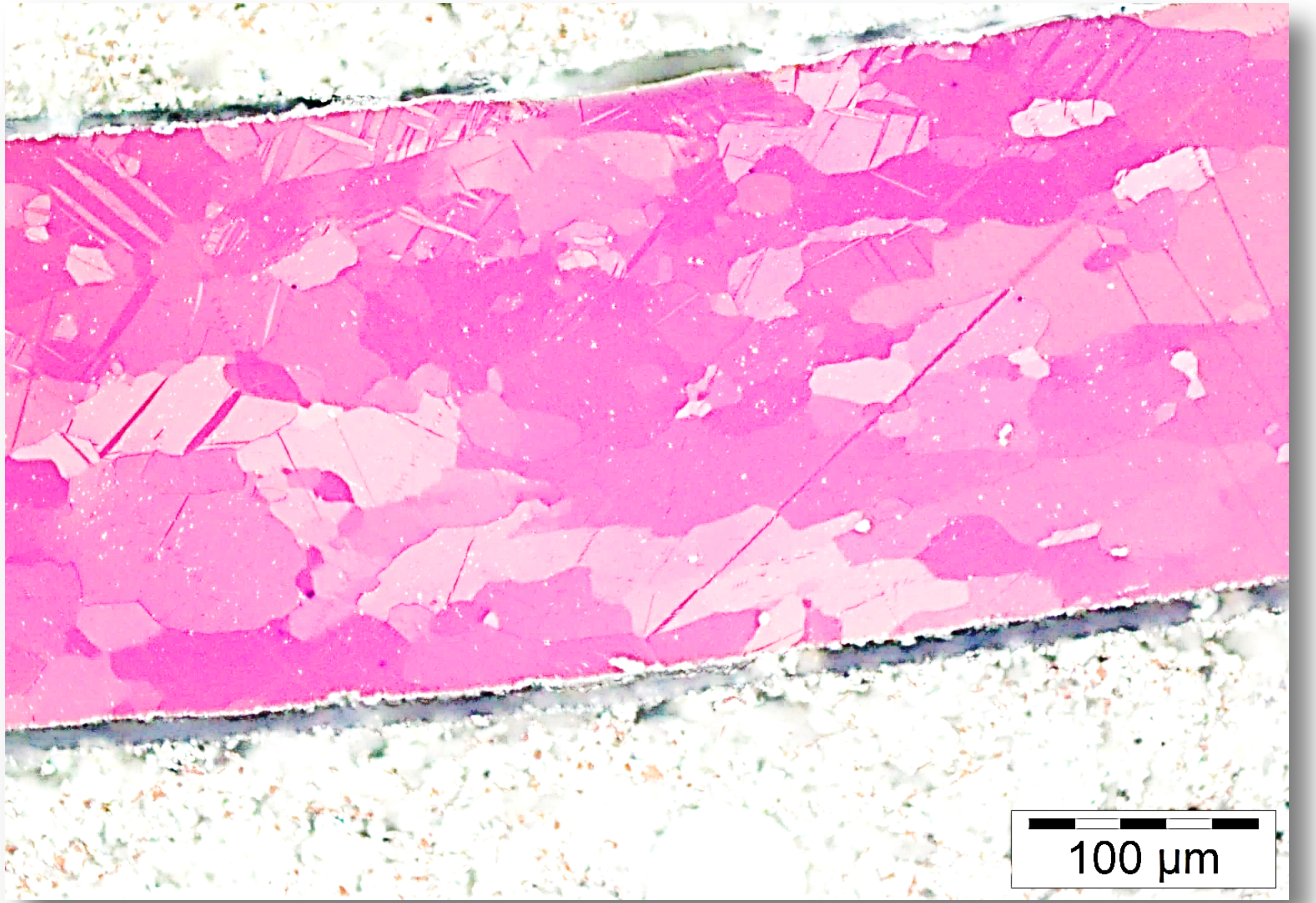



S200F (VHP)



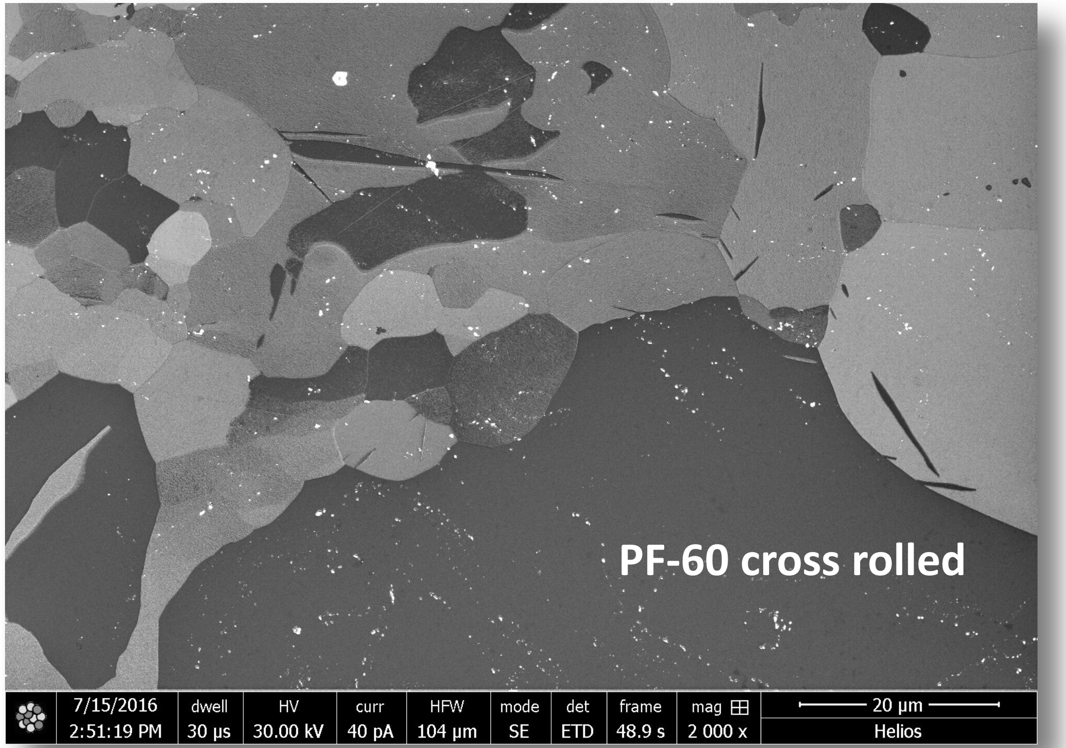


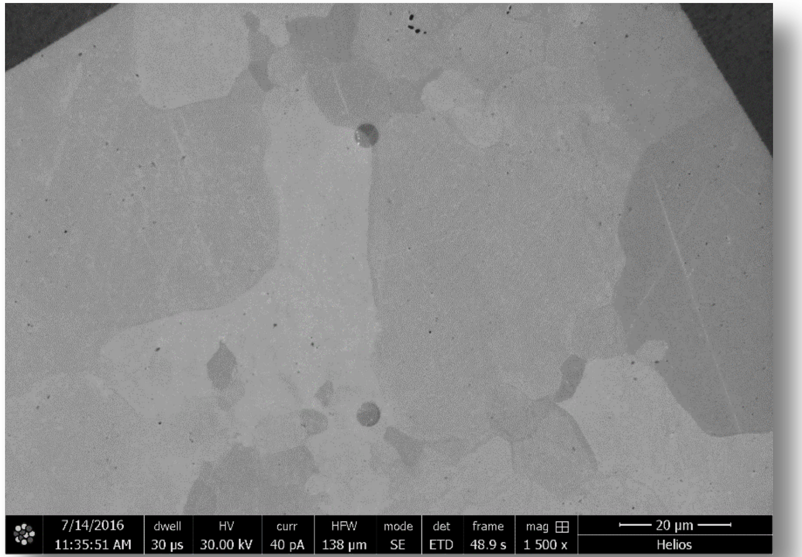



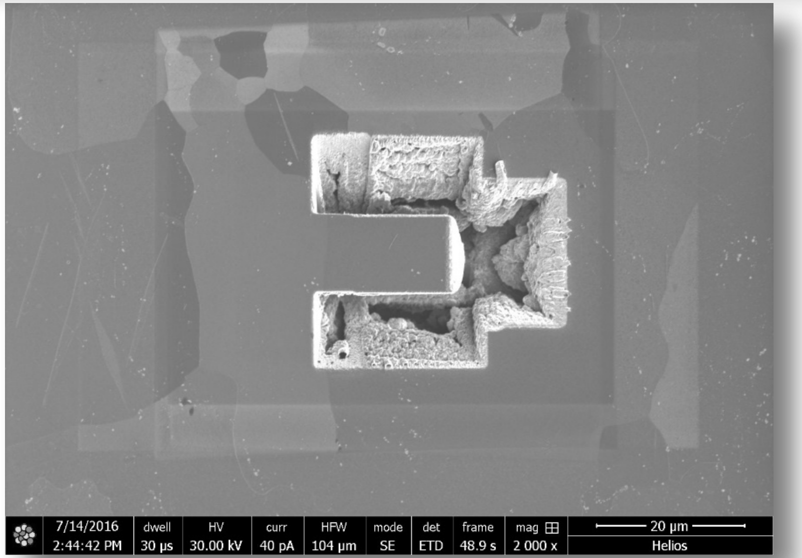



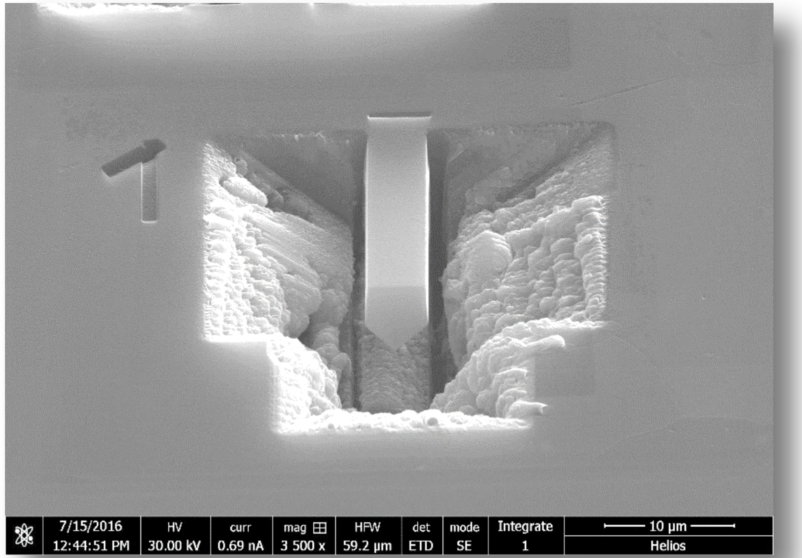

- Twinning is an important deformation mechanism under compression

# Micromechanical tests samples preparation

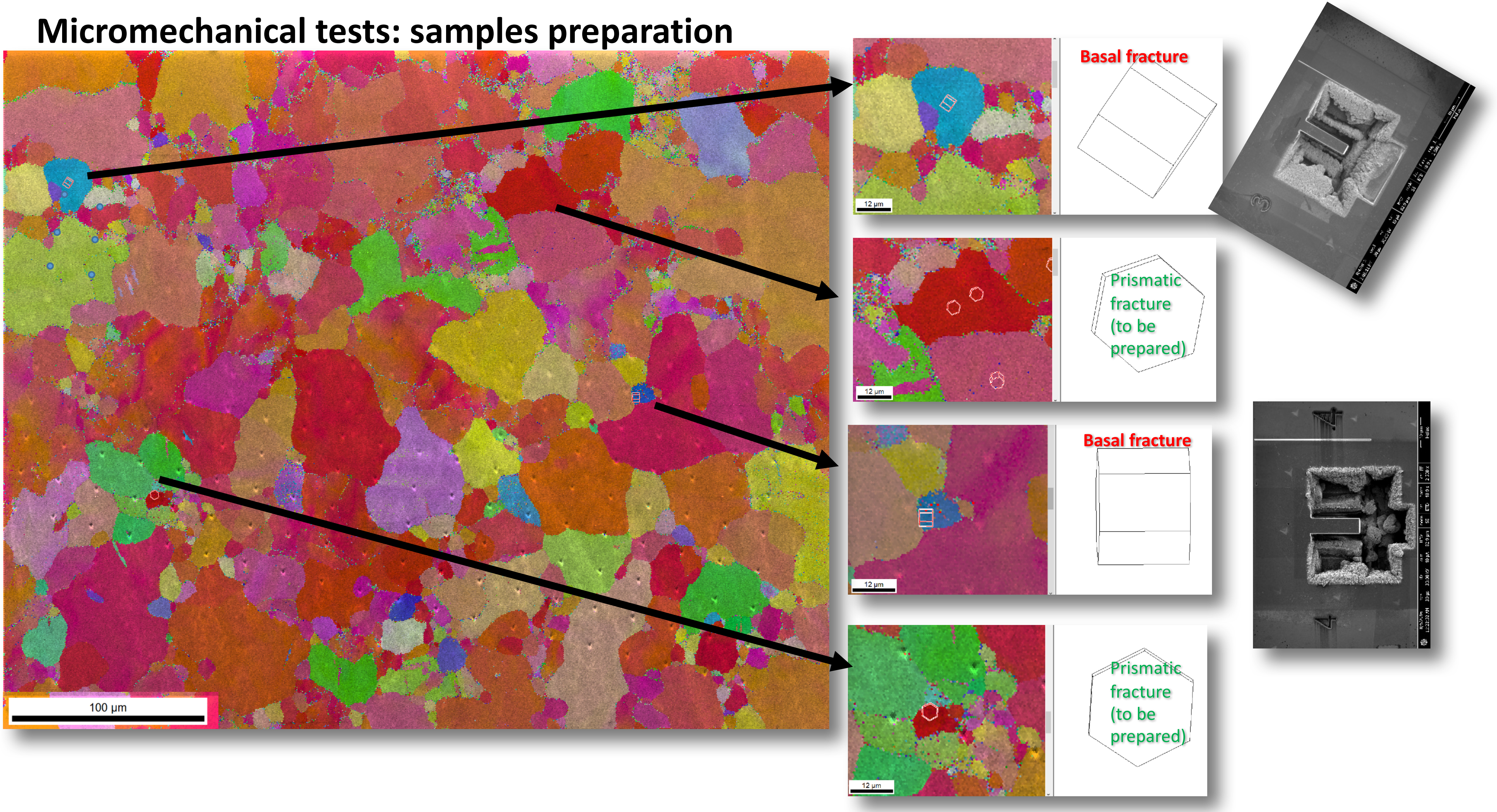


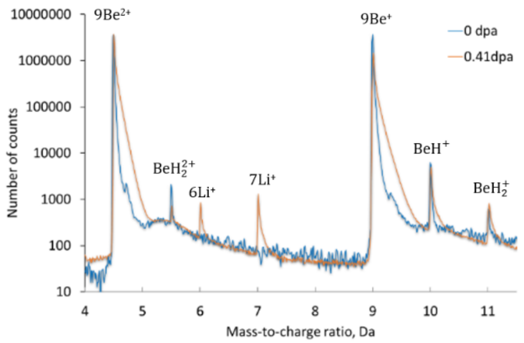



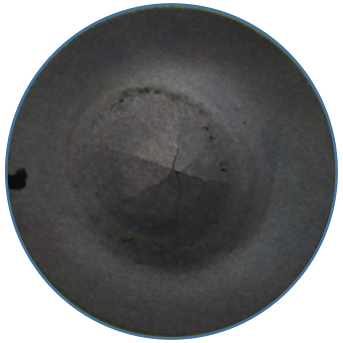
# Micromechanical tests: samples preparation

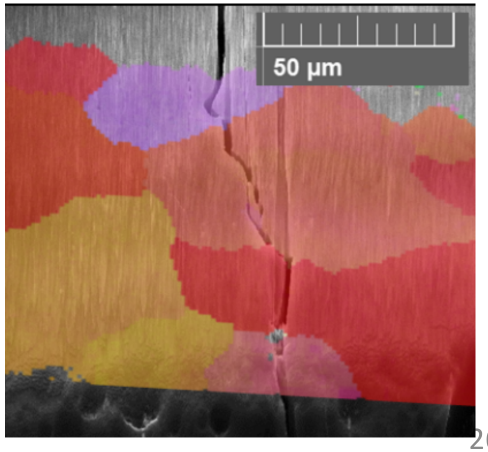


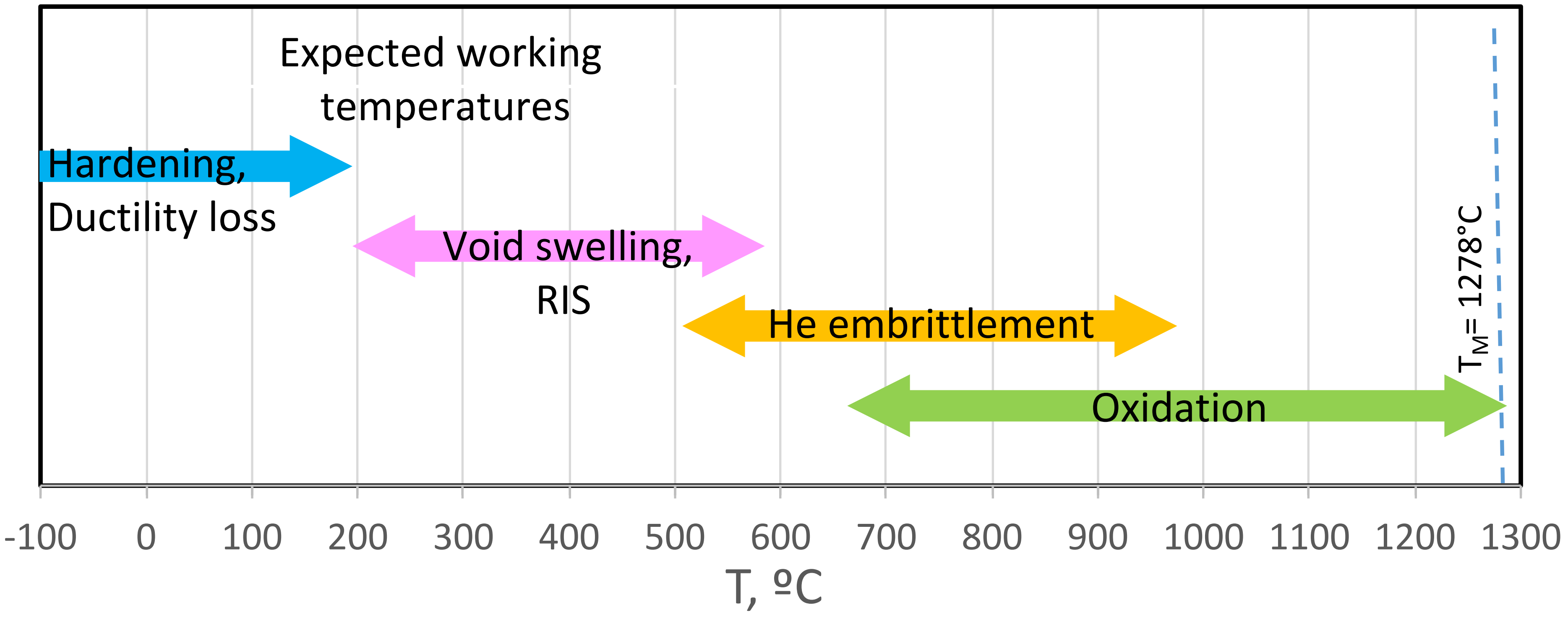
# Conclusions

- Beryllium is highly non-homogeneous at macroscale and highly anisotropic material.
- Industrial beryllium is an impure alloy.
- Impurities strongly create precipitates, segregate to BeO and dislocations
- Beryllium matrix (PF60) has some quantity of homogeneously distributed carbon, iron, nickel and cupper
- Proton irradiation cause Li production, which is homogeneously distributed (T=50°C)
- Signs of irradiator induced embrittlement and change in fracture mode (from intra- to inter-granular) under high energy proton irradiation
- He implantation to 0.1 dpa introduces significant hardening of berillium






Expected working temperatures
Hardening, Ductility loss
Void swelling, RIS
He embrittlement
Oxidation
T_M= 1278°C
-100 0 100 200 300 400 500 600 700 800 900 1000 1100 1200 1300
T, ºC