

13th IFAST WP9 meeting



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Bundesministerium
für Bildung
und Forschung

LOT, Institut für Werkstofftechnik, Universität Siegen

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Tasks at Uni Siegen

1. Deposition studies:

- **NbTiN** in CC800: DCMS and HiPIMS
- **Nb₃Sn** in BoxCoater: RFMS, “test” the material, SIS structures
- **MgB₂** in PVD/SEY chamber: co-sputtering with RFMS
on metal (Cu, Nb...) as well as insulating (AlN, TiO₂, Al₂O₃) substrates

2. Substrate preparation: mechanical and electropolishing of Cu

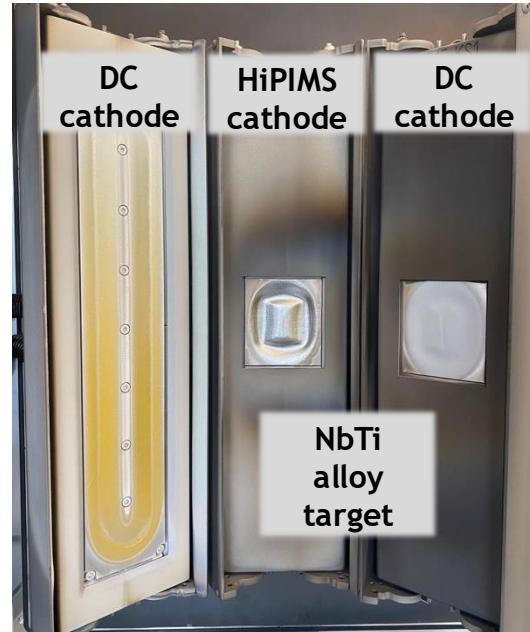
3. Sample characterization: film morphology, microstructure +

PAS experiments (HZDR Germany, Sebastian Klug, Oskar Liedke) +

SC and RF properties (INFN-LNL Italy, Dorothea, Davide, Giovanni; IEE Slovakia, Eugen)

Experimental setup

Commercial coating system CC800/9



- Target size: 100 x 88 mm²
- Target: NbTi alloy **80:20 wt%** (99,95%, Robeko)
 $\sim\text{Nb}_{0.67}\text{Ti}_{0.33}$ target composition
- Bake-out time: 6 h at 290 °C
- Base pressure: $\sim 6.0 \times 10^{-7}$ mbar
- MF plasma etching for substrate plasma cleaning

Substrates



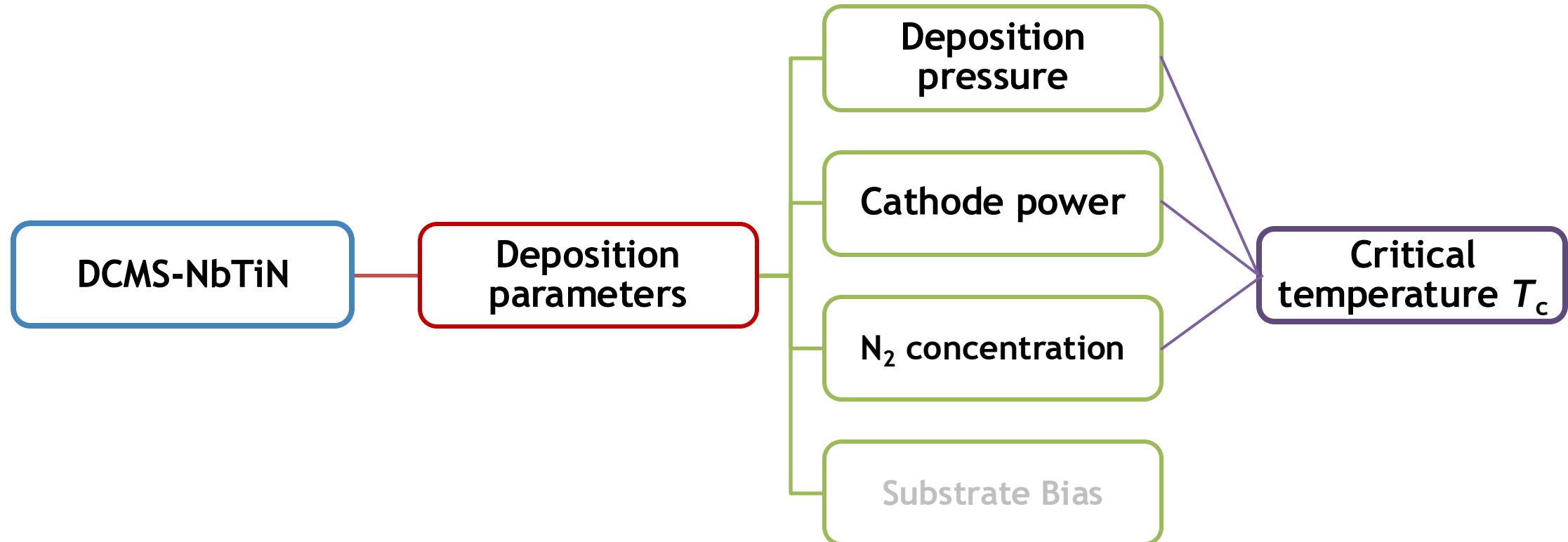
- p-doped Si<100>, 15 x 15 mm²
- Polycrystalline OFHC Cu samples, 1 mm thick, 25 x 25 mm²
- Sample treatment of Cu substrates: mechanical polishing + electropolishing in a solution of o-H₃PO₄ (85 %) and n-butanol (C₄H₉OH), 3:2 ratio

Characterization methods:

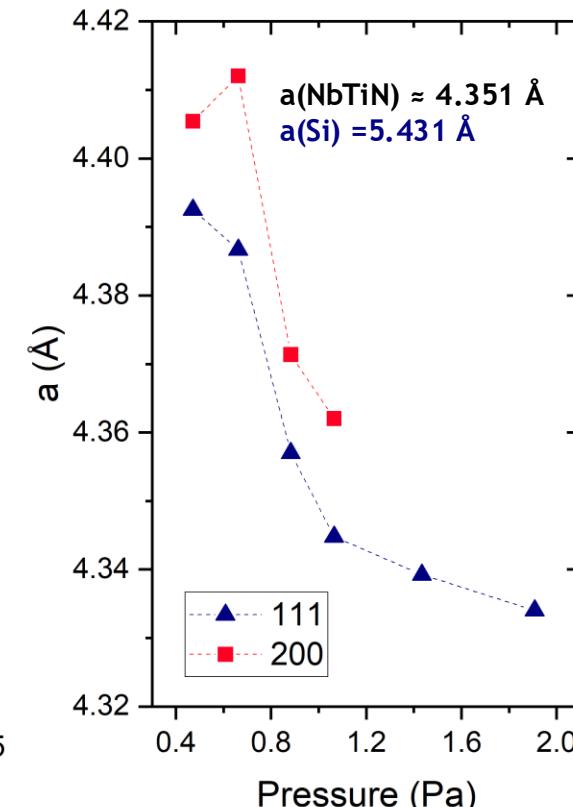
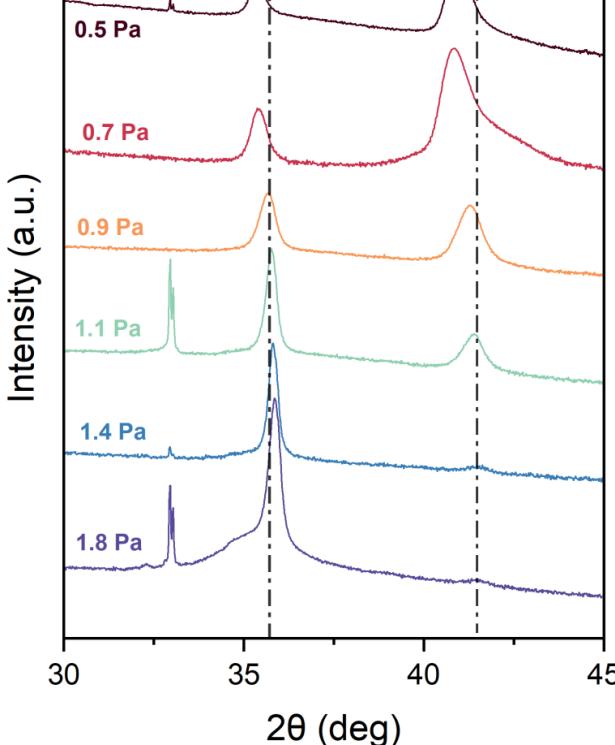
- SEM, EDX, XRD, AFM
- T_c (coil-induction) measurement station^[1] on Si samples

[1] D. Fonnesu et al., in *Proc. SRF'21*, East Lansing, MI, USA, Jun.-Jul. 2021, pp. 105-108
doi.org/10.18429/JACoW-SRF2021-SUPFDV018

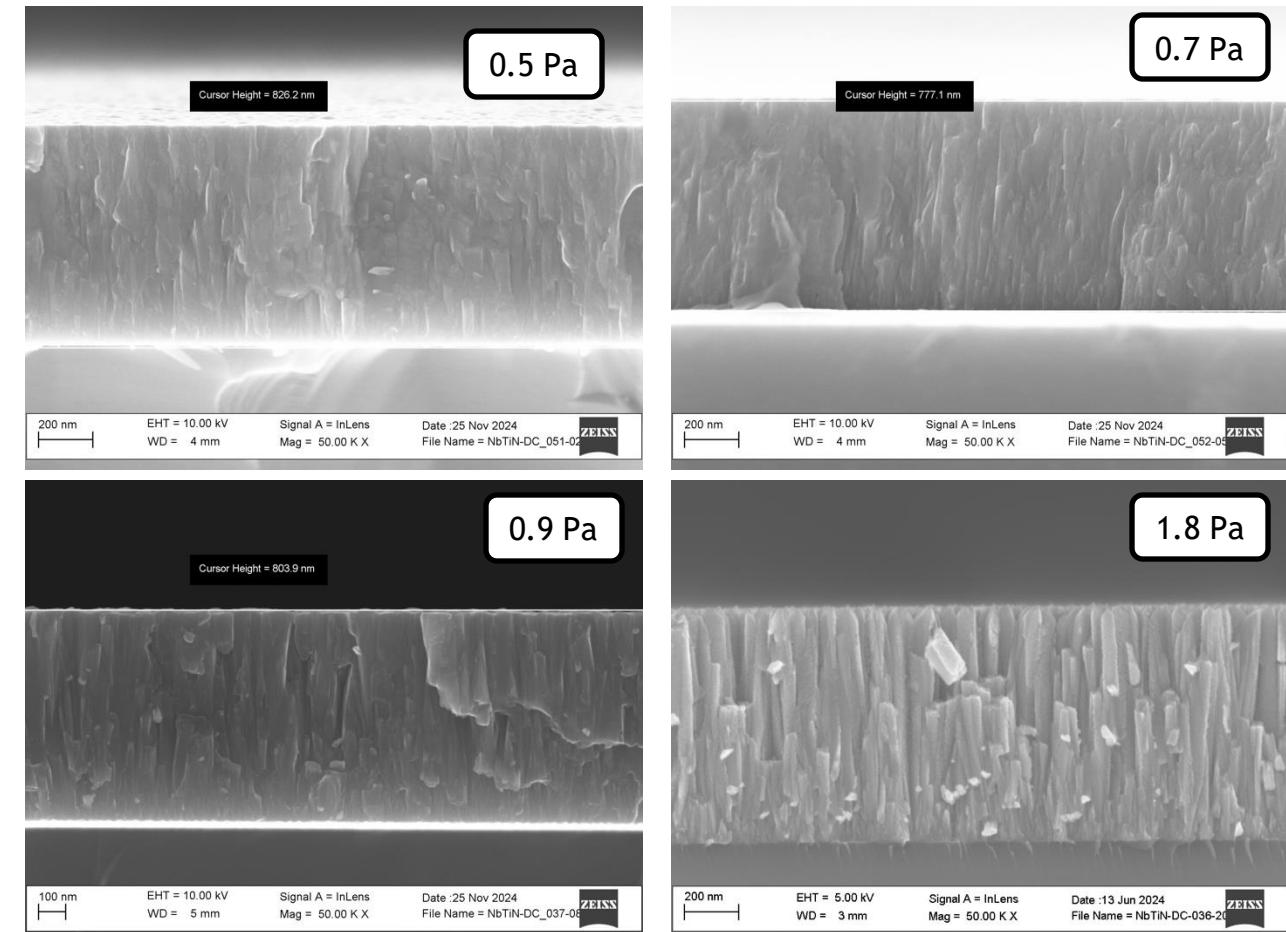
Experimental plan



Effect of deposition pressure

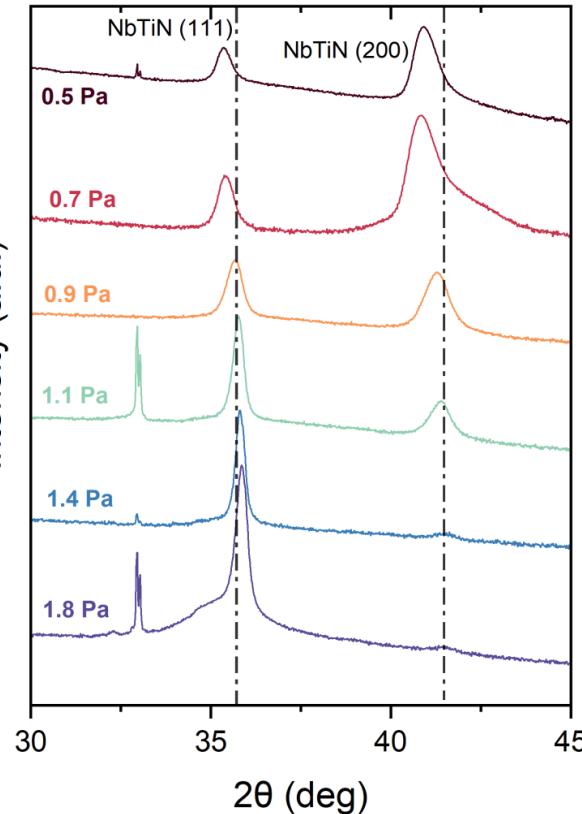


Deposition conditions:
 $P_{\text{target}} = 400 \text{ W}$, $N_2 = 9\%$, Bias = -50V

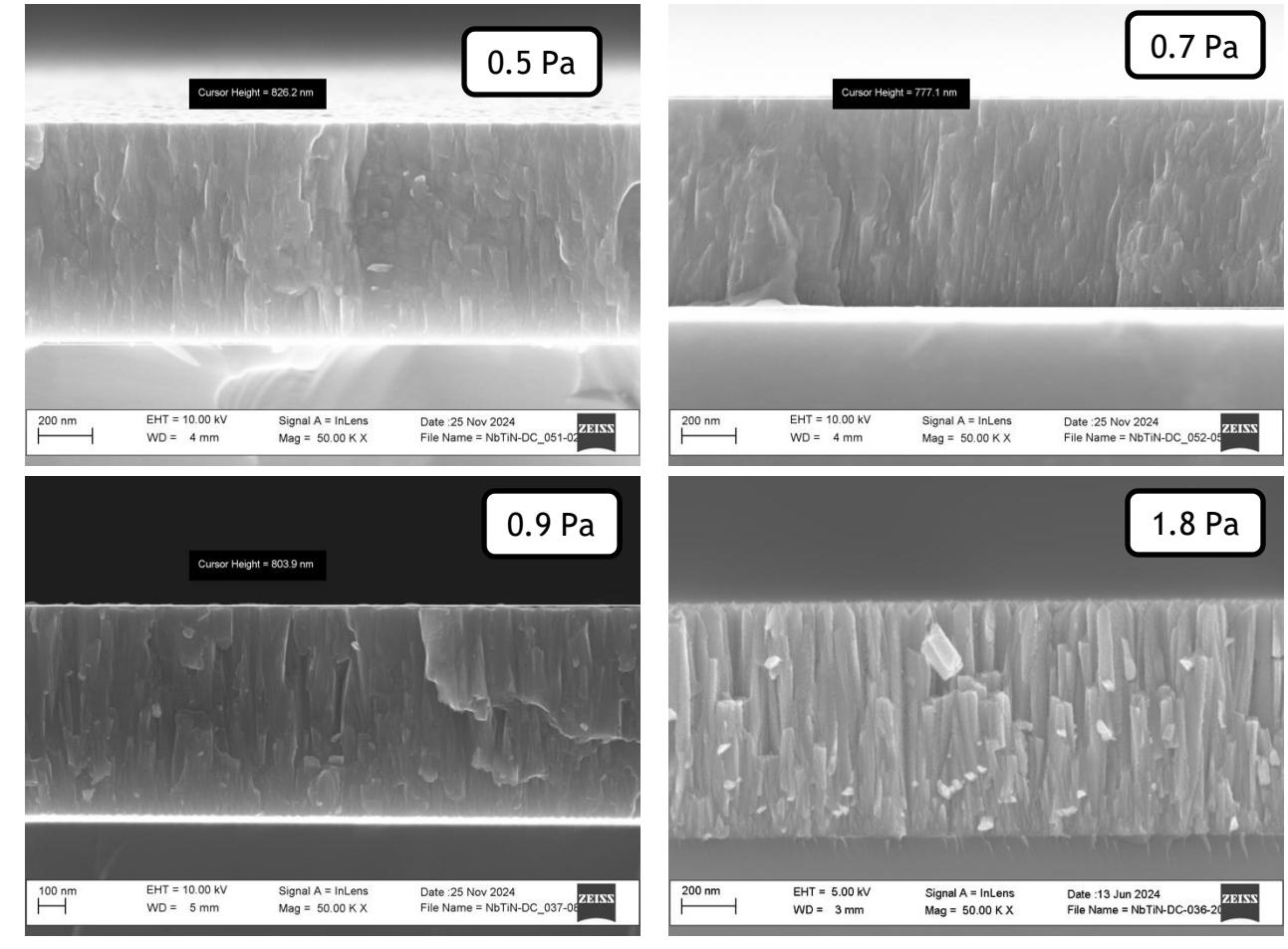
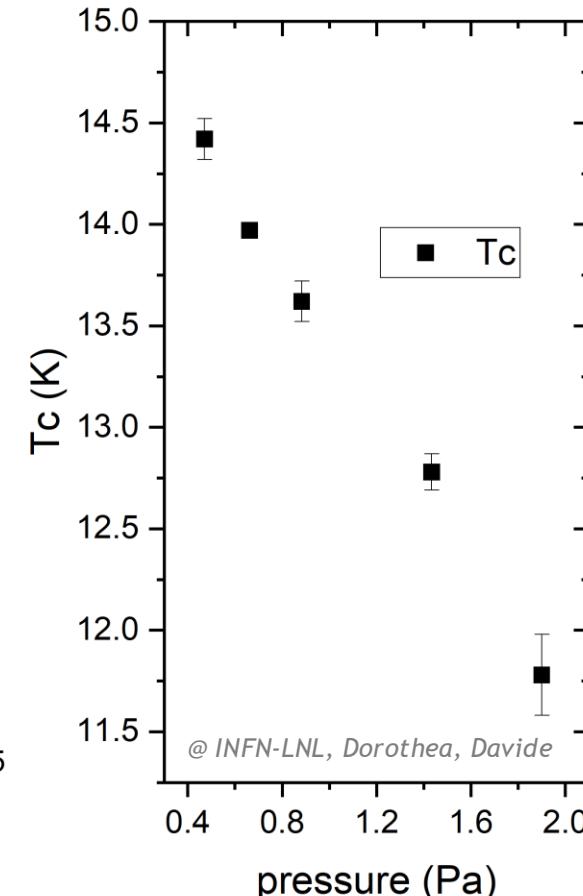


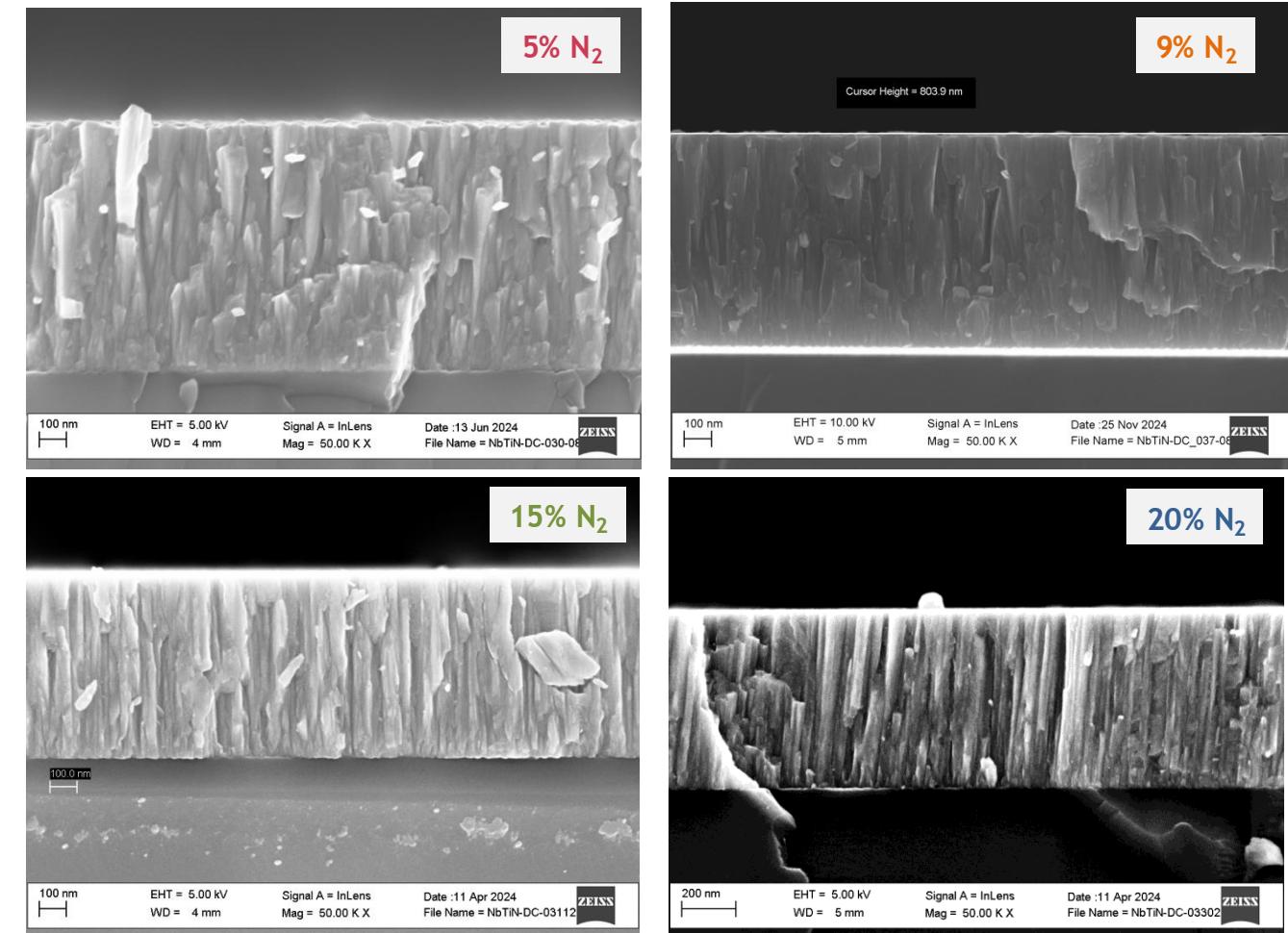
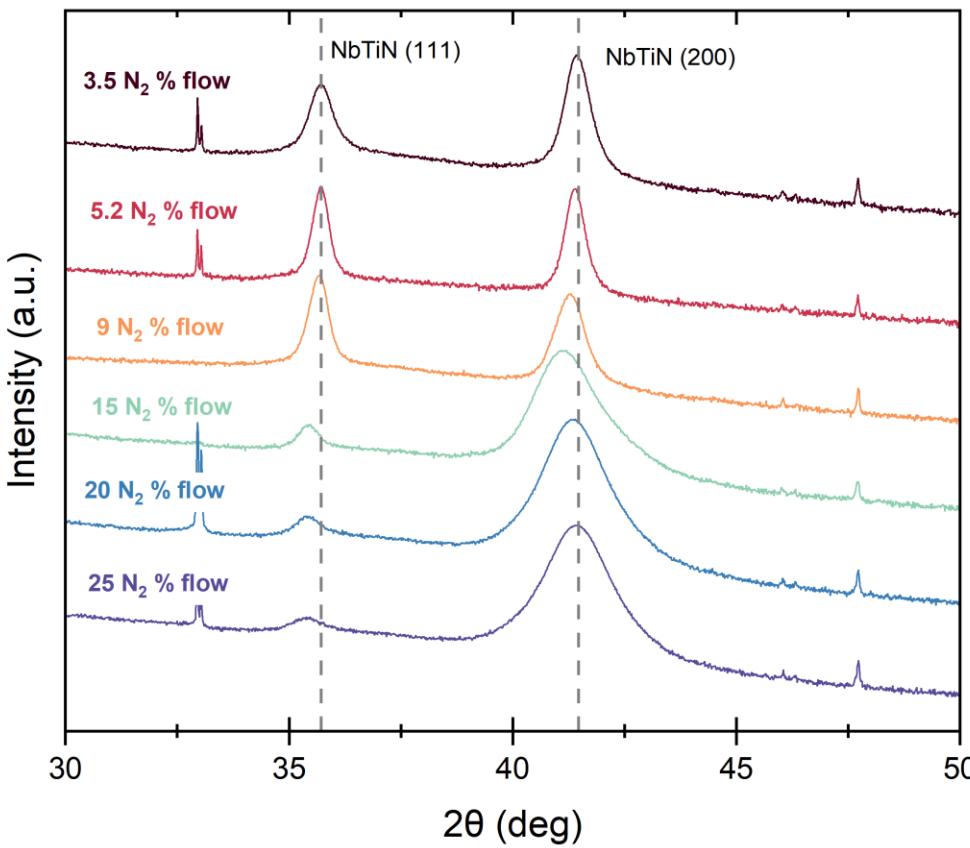
Effect of deposition pressure

Intensity (a.u.)



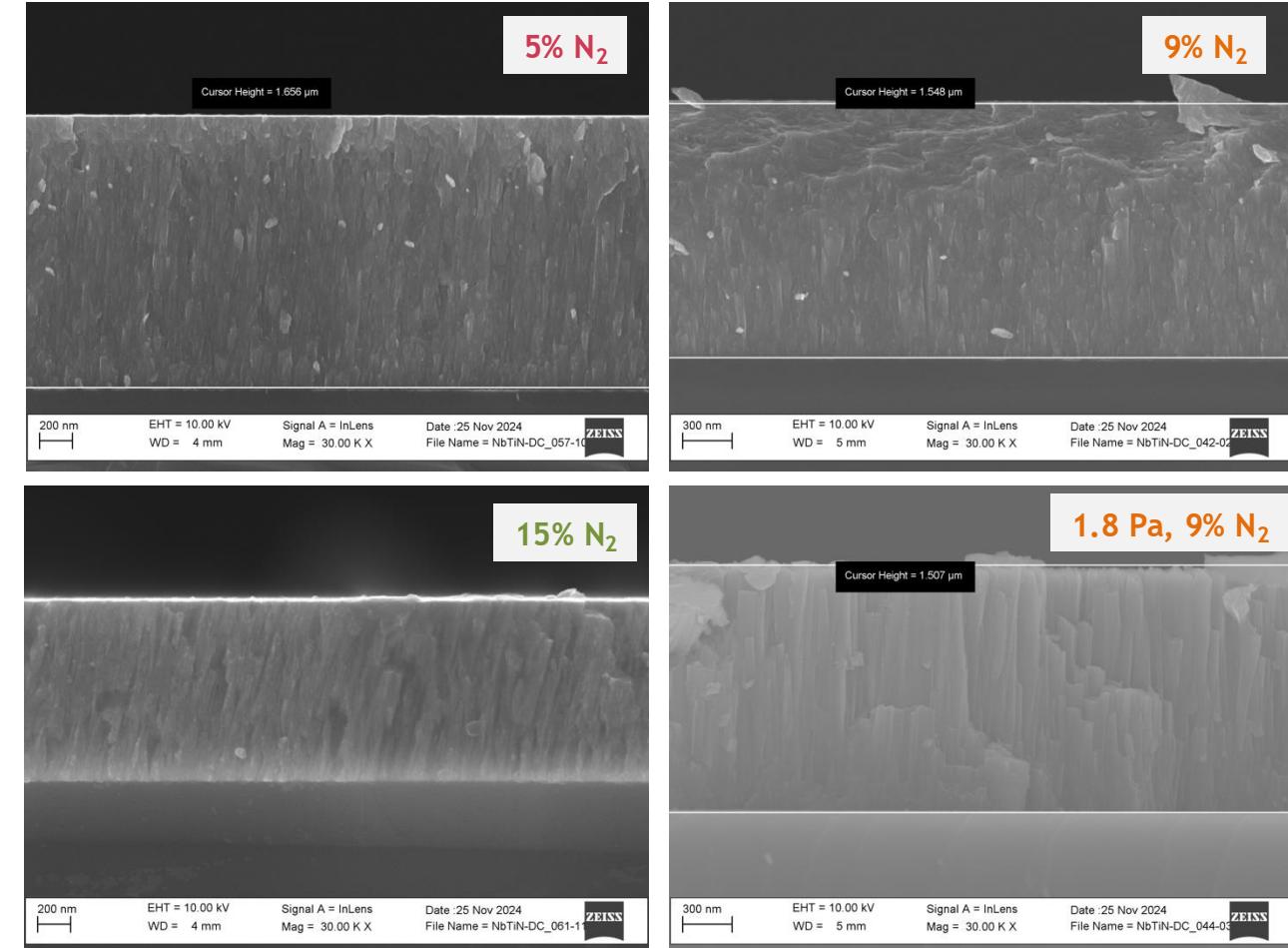
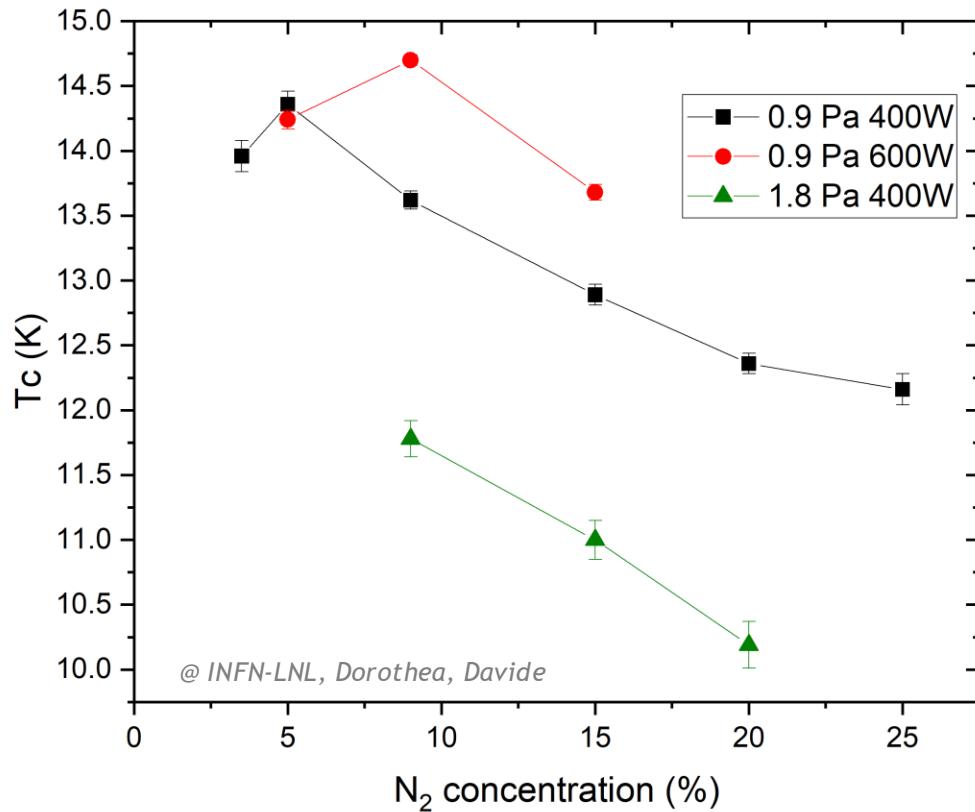
Deposition conditions:
 $P_{\text{target}} = 400 \text{ W}$, $\text{N}_2 = 9\%$, Bias = -50V

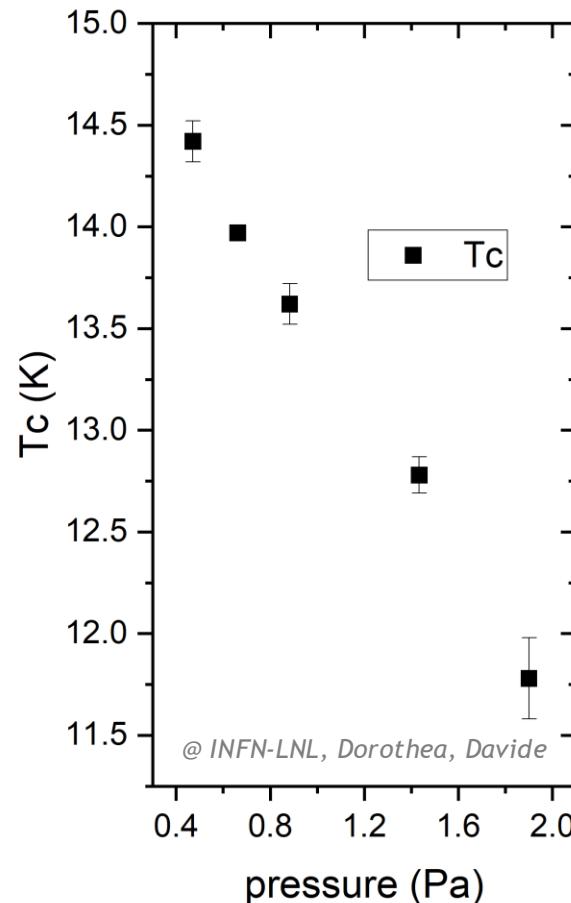
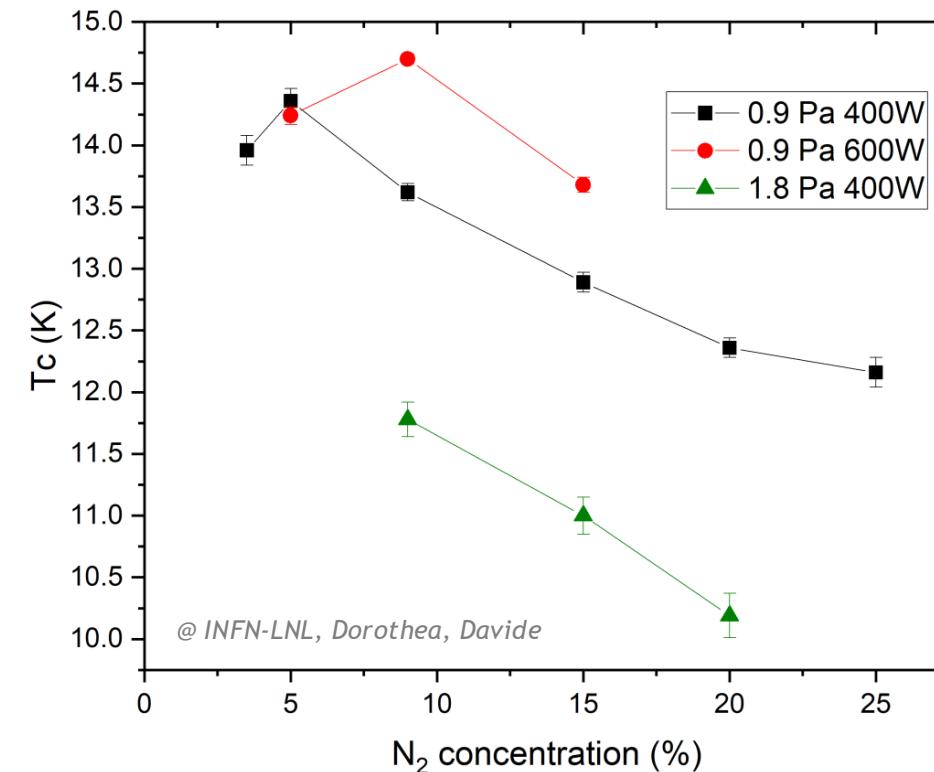


Effect of N₂ concentrationDeposition conditions: P = 400 W, p_{dep} = 0.9 Pa, Bias = -50V

Effect of N₂ concentration and cathode power

Deposition conditions: P = 600 W, p_{dep} = 0.9 Pa, Bias = -50V

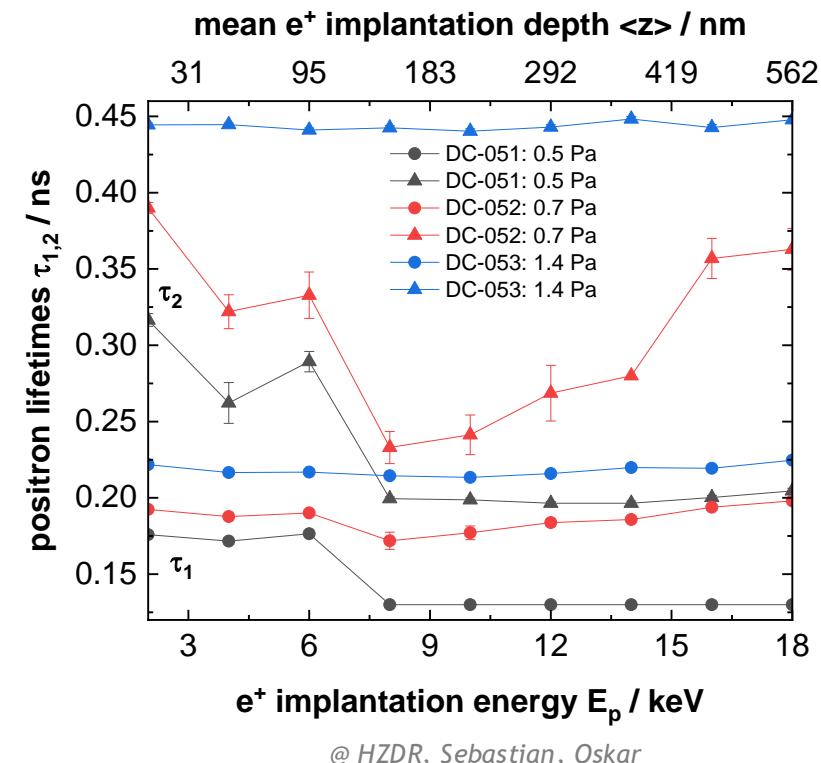
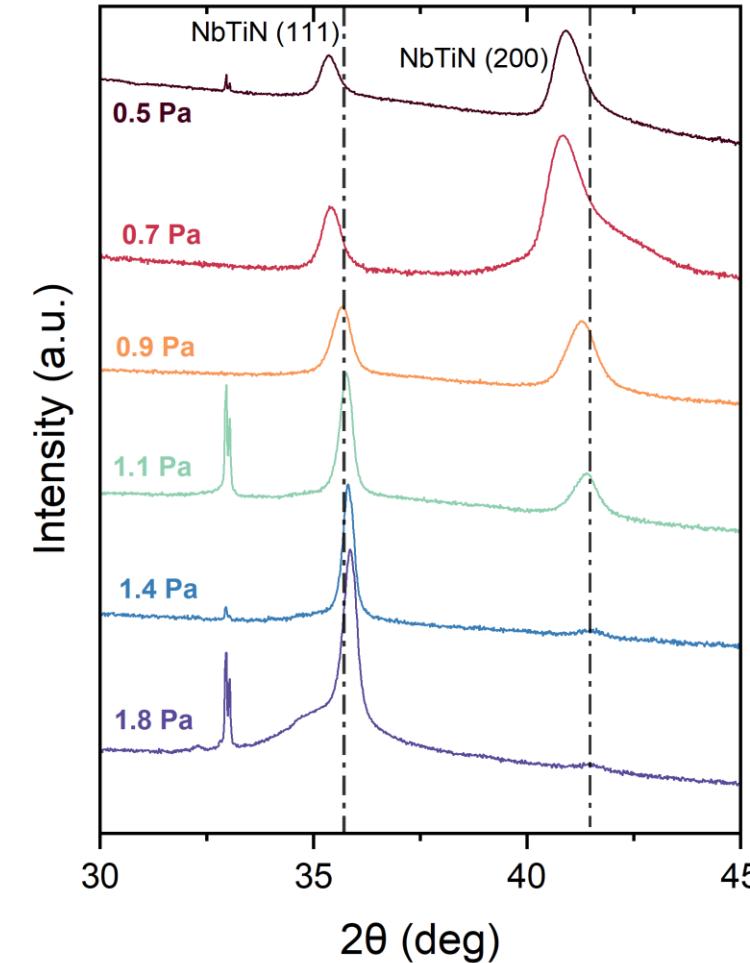
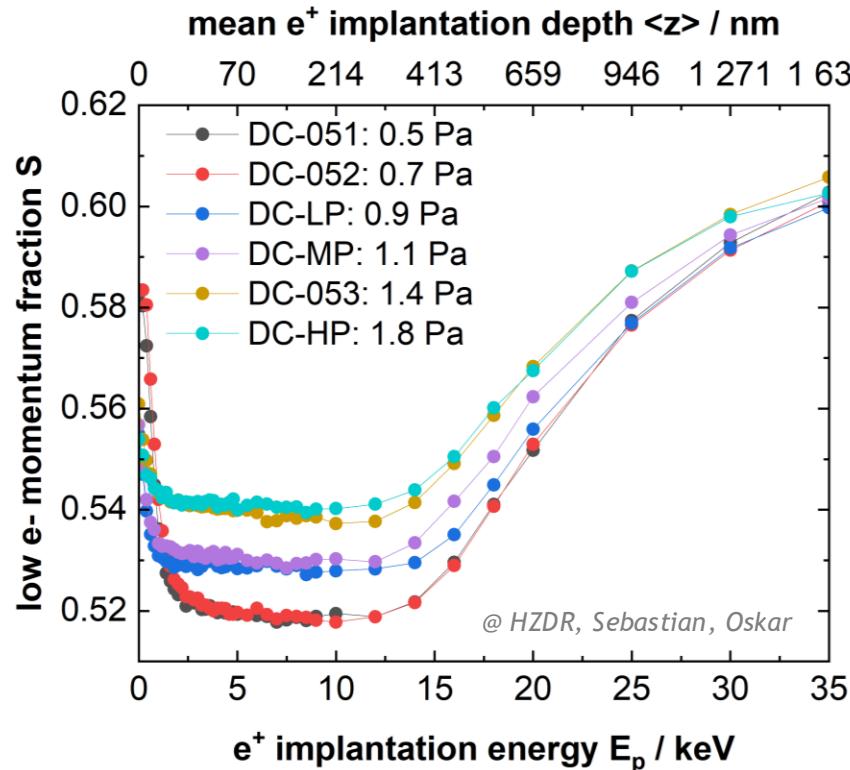




High T_c - where?

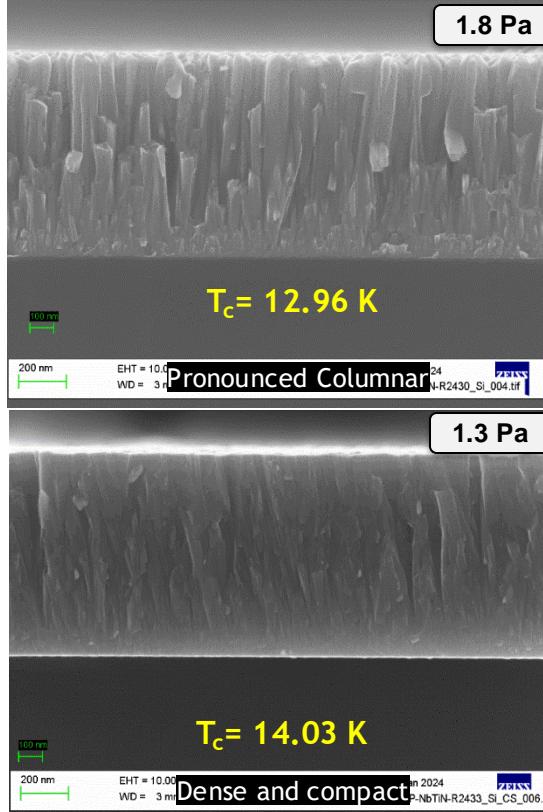
- **Low deposition pressure:** 0.5-0.7 Pa
- **High cathode power:** 600W (6.8 W/cm^2)
- **Low N_2 flow:** <9%, related to the cathode power
- **Deposition temperature:** old “ 250°C ” → real $>500^\circ\text{C}$
- **Target composition:** use NbTi alloy **70:30 wt%** instead of **80:20 wt%**

PAS experiments: deposition pressure



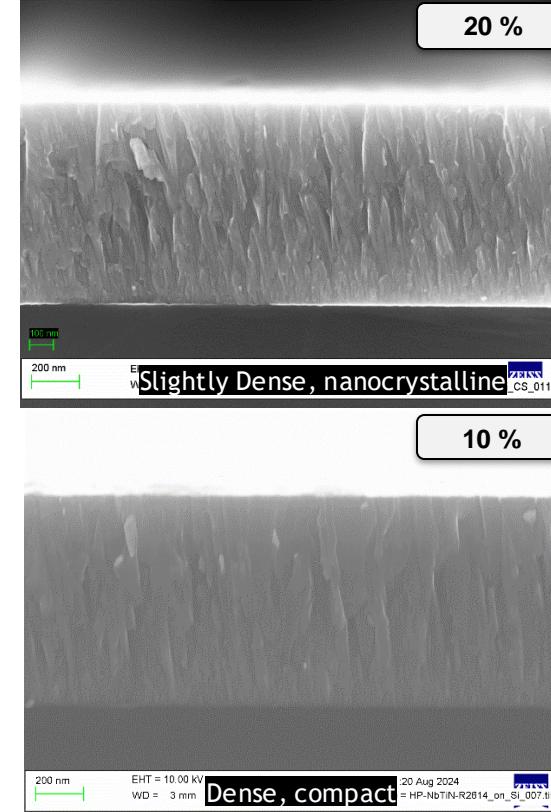
Results: SEM Micrographs (HiPIMS-NbTiN)

Power = 400 W, N₂% = 10, S.B = 50 V,
DCyc = 20%, S.T = 150 °C



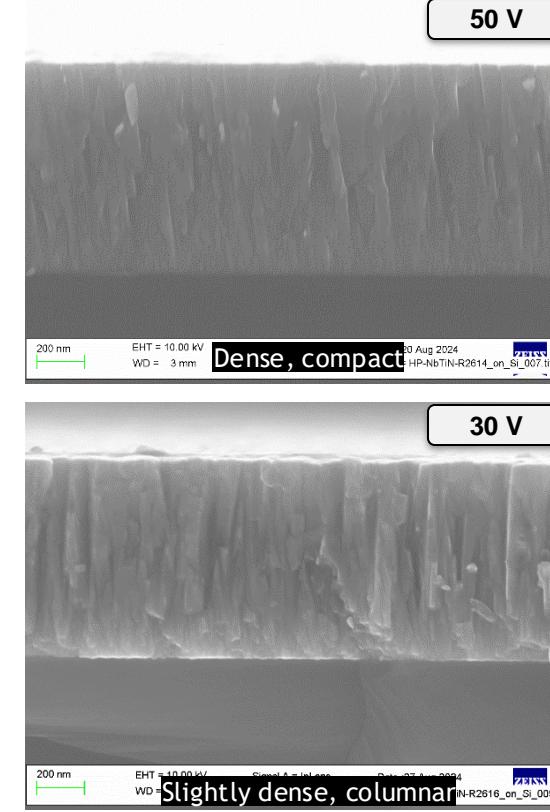
Deposition pressure study

Power = 400 W, D.P = 1.4 Pa, N₂% = 10,
S.B = 50 V, S.T = 150 °C



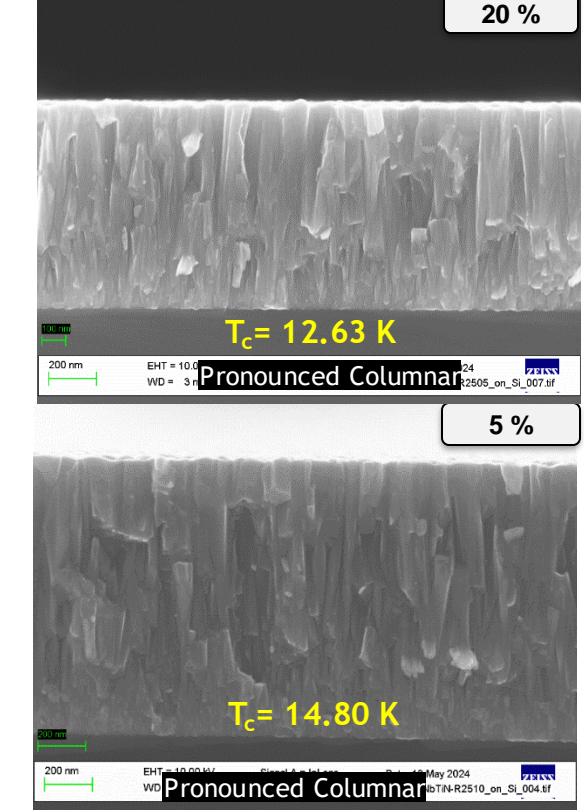
Duty cycle study

Power = 400 W, D.P = 1.3 Pa, N₂% = 10,
DCyc = 10%, S.T = 150 °C



Substrate bias study

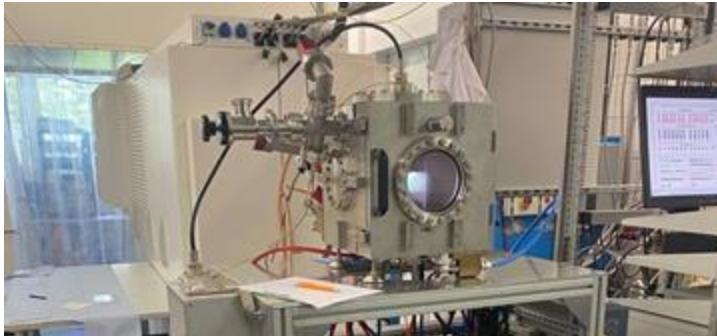
Power = 400 W, D.P = 1.3 Pa, S.B = 50 V
DCyc = 20%, S.T = 150 °C



Nitrogen flow study

Nb₃Sn RF sputtering

Nb₃Sn in BoxCoater



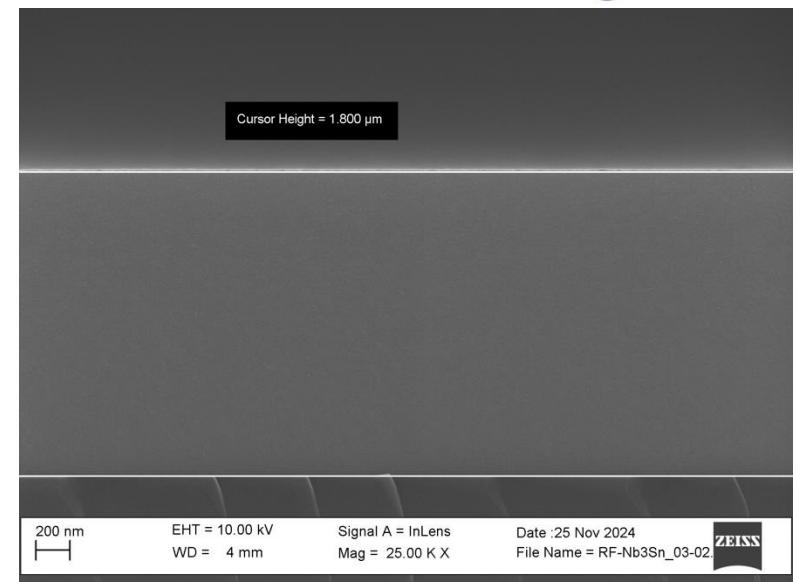
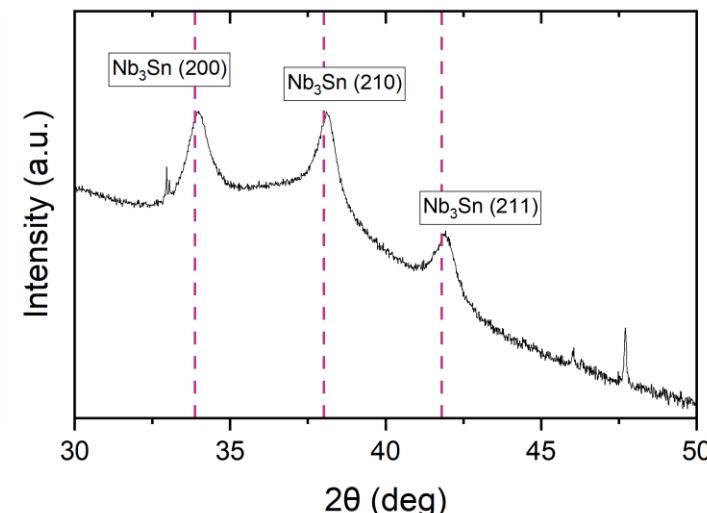
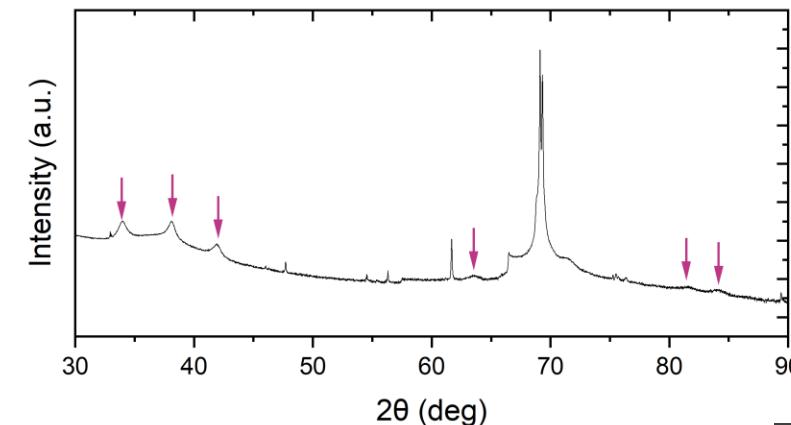
Test depositions for multilayer systems
Later process transfer to CC800 - HiPIMS

Parameters:

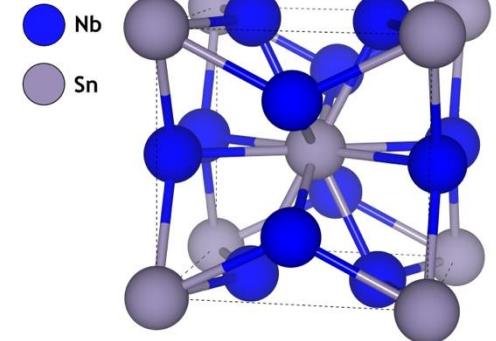
$T_{\text{dep}} = 600^\circ \text{C}$

P(cathode) = 100 W

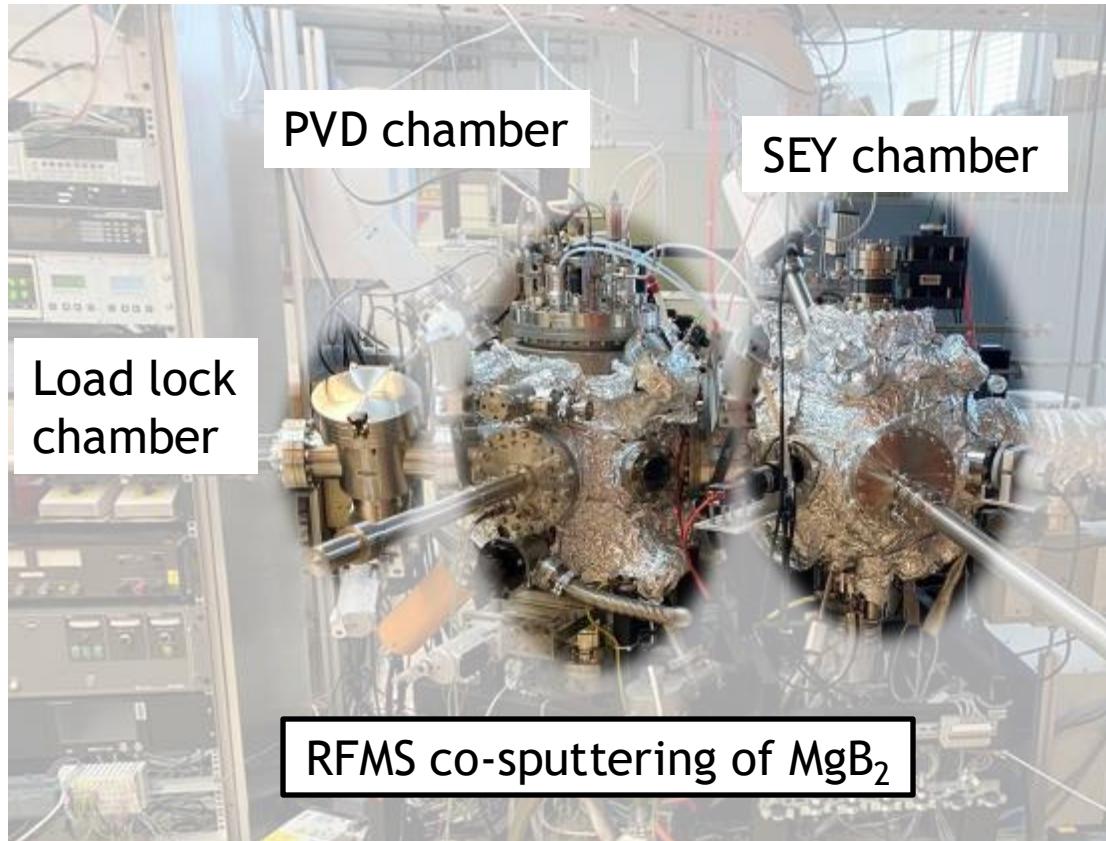
$p_{\text{dep}} = 1 \text{ Pa}$ (**Ar**) → must be **Kr**



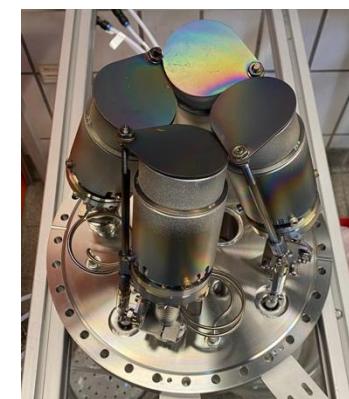
P m-3n



MgB₂ in PVD/SEY chamber



- Four 2" RF magnetrons (confocal)
- Targets **Mg** and **B**: variation of cathode powers
- Substrate temperature: set to 300°C
- Ar pressure: 0.5 - 1 Pa



4 cathodes
arrangement

Plans and outlook

- Optimization of **DC-NbTiN** deposition: change the target composition to **Nb:Ti = 70:30 wt%**!
- Results of T_c for **HiPIMS-NbTiN**: limited by the target composition as well?
- **SIMS** for the influence of N_2 flow concentration: DC- and HiPIMS-NbTiN
- Deposition of multilayer (SS or SIS) structures with
DC-NbTiN (“best” conditions) / DC-AlN/ **HiPIMS-Nb** or **bulk Nb**
- Start of **MgB₂** deposition in PVD/SEY deposition chamber by RF co-sputtering: **promising?**
- Deposition of **Nb₃Sn** in BoxCoater: test for the multilayer structures
- QPR samples for RF test and surface resistance

THANK YOU FOR YOUR ATTENTION!



