WP 12 Thin Films

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- Introduction
- ALD technique
- AlN deposition
- NbN deposition
- Mulitilayer deposition
- Conclusions and future work

Introduction

RF cavities: **bulk niobium**

Functionnal coating
(inside the cavity)

Insulating AlN, t ≈ 10 nm

Superconducting NbN (Tc > 9K) t ≈ 80 nm

Insulating AlN, t ≈ 10 nm

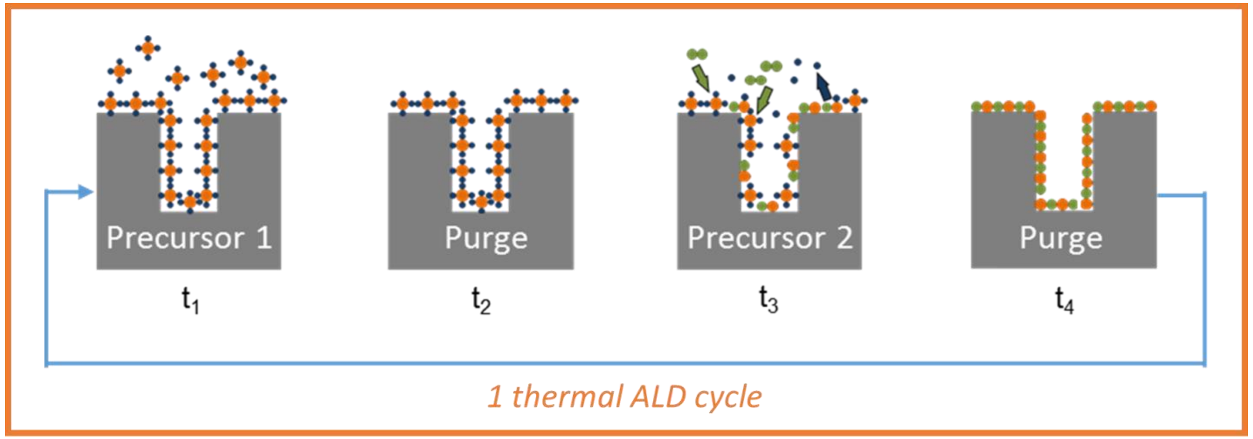
Bulk Nb, Tc = 9 K
RF cavity

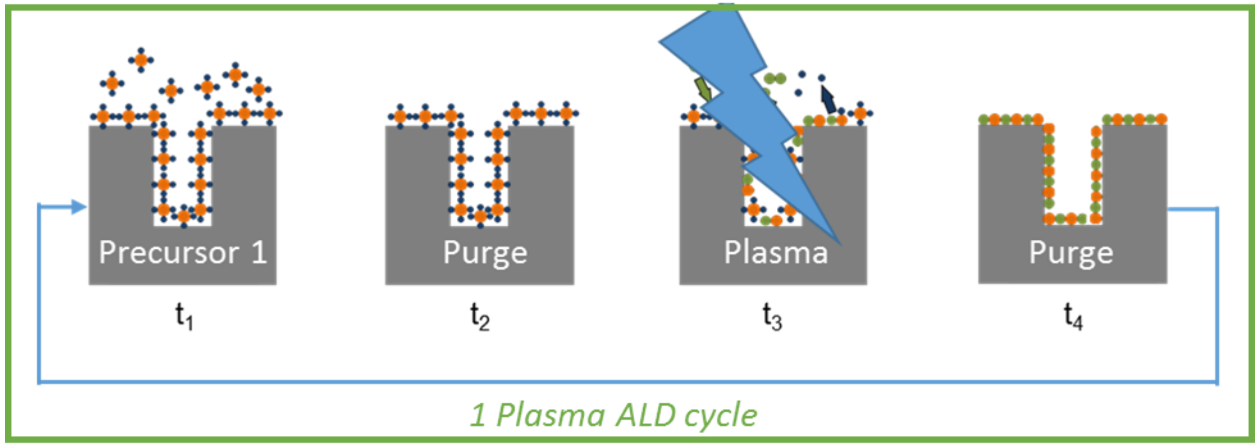
→ **Enhancement of the quality factor** and reduction of the electomagnetic loss

A. Gurevich, "Enhancement of RF breakdown field of SC by multilayer coating". Appl. Phys.Lett., 2006. ***88***

Challenge : conformal deposition on complex shape → **Atomic Layer Deposition**

ALD technique





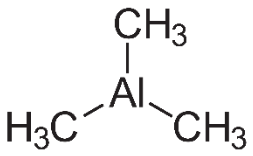
- Higher reactivity, lower deposition temperature than ALD
- Slightly less conformal than thermal ALD

- **Sequential** introduction of the precursors, surface **saturation** : **conformal process**
- Mono-atomic layers (ideally in the « **ALD window** »)

AlN deposition

- Thermal ALD issue :
  - NH3 reactive above 350°C
  - TMA decomposition above 375°C

- Most common settings in plasma ALD* :
  - Al source : TMA
  - N source : NH3 **plasma**
  - Temperature : 350°C



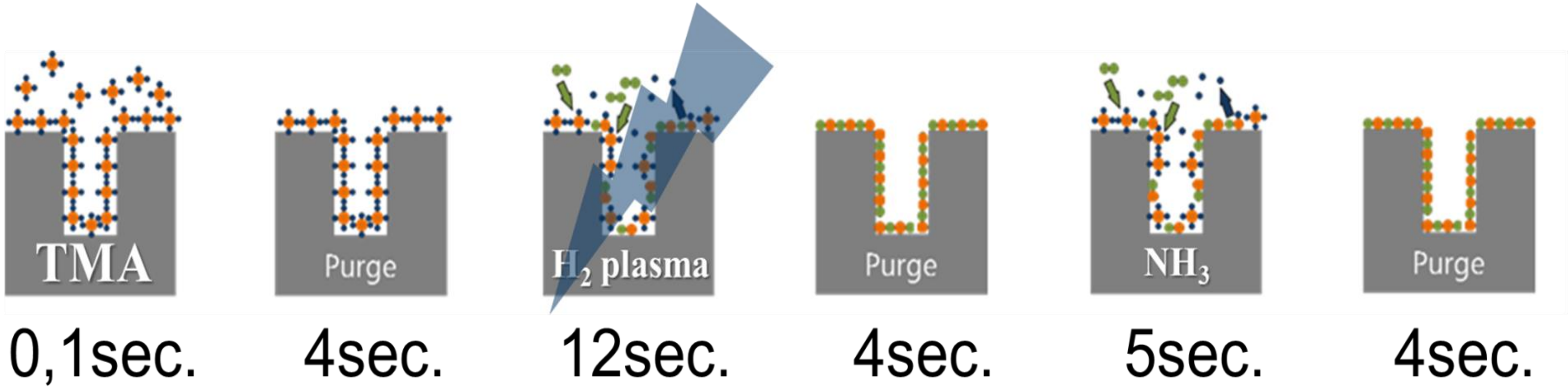
TMA

New deposition strategy : use of a hydrogen plasma for precursor reduction

*Banerjee et al., P hys. Status Solidi C **12**, No. 7, 1036–1042 (2015)*

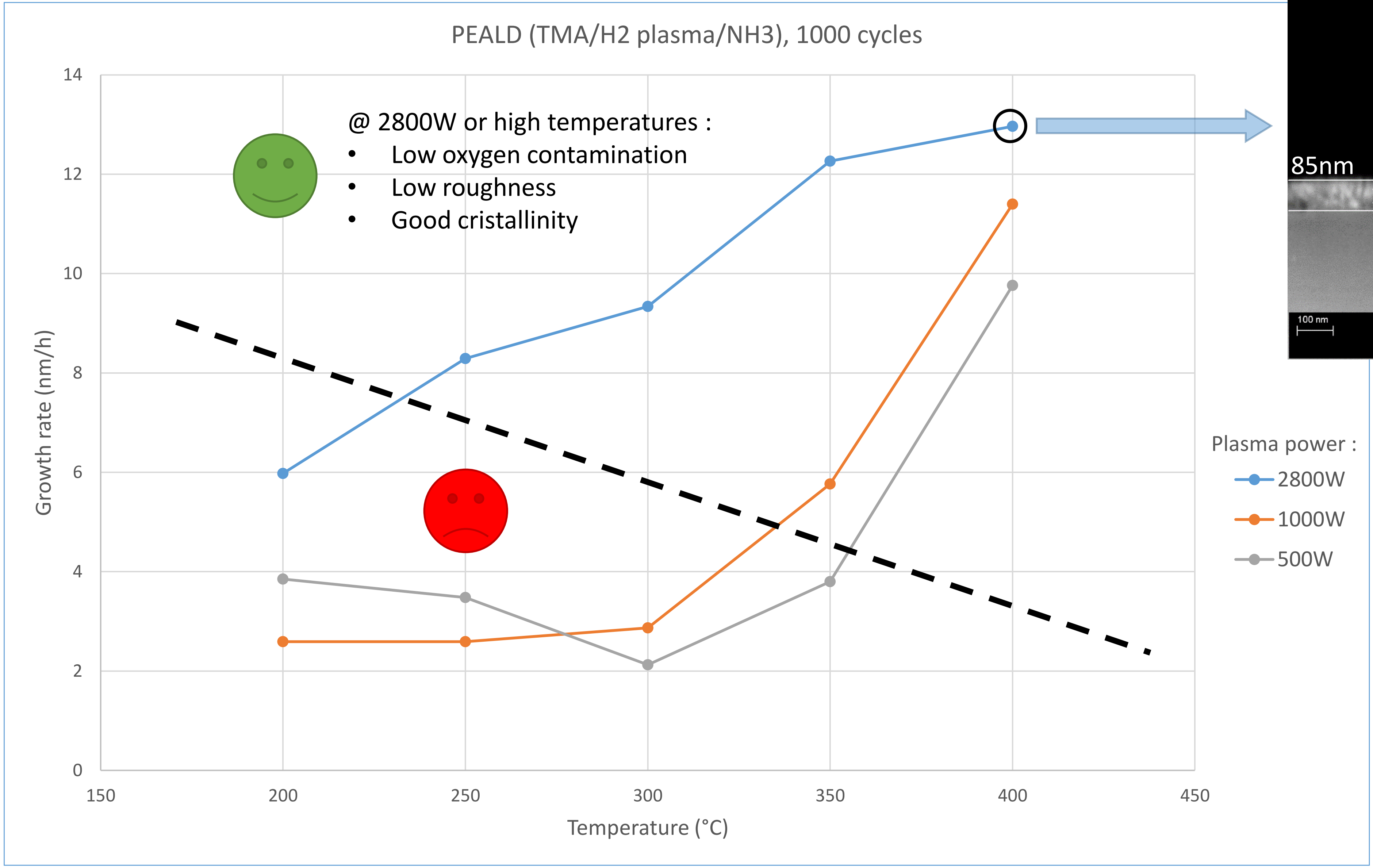
AlN deposition

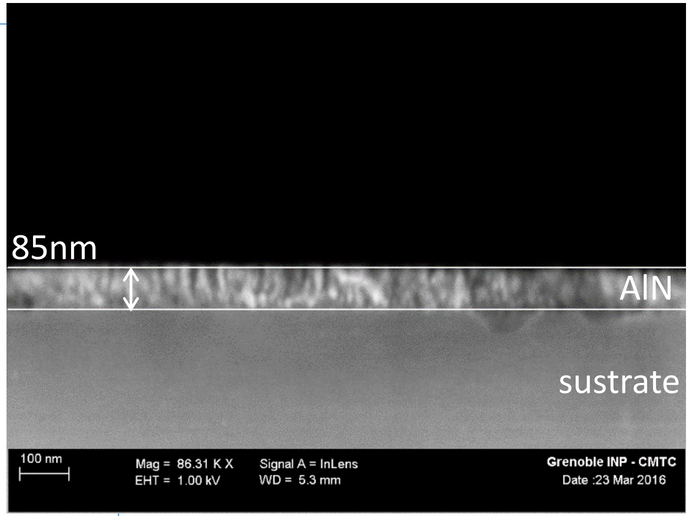
PEALD : plasma enhanced atomic layer deposition
= classic ALD with two more steps → 6 times cycles
→ **Supercycle**



- Constant parameters :
  - 1000 cycles
  - 8 hPa in pressure
  - Pulses time
- Studied parameters :
  - Temperature
  - Plasma power

AlN deposition



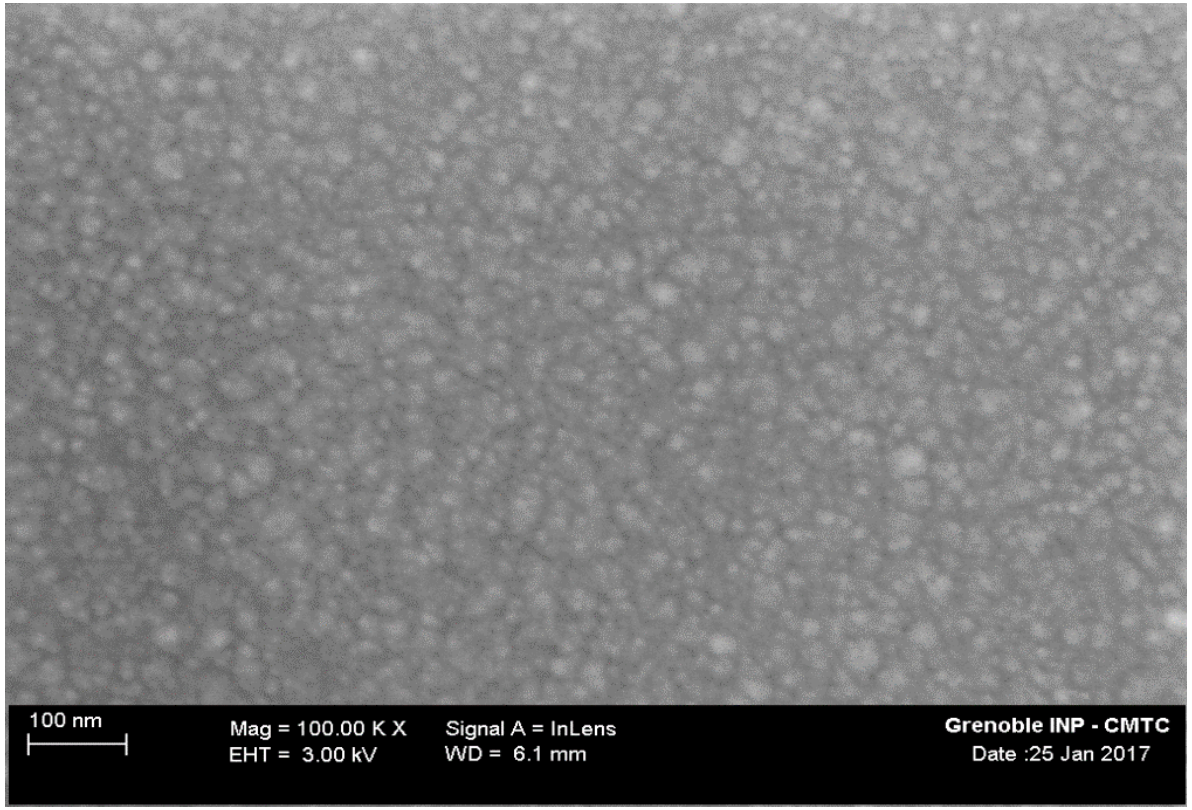


Good processing conditions

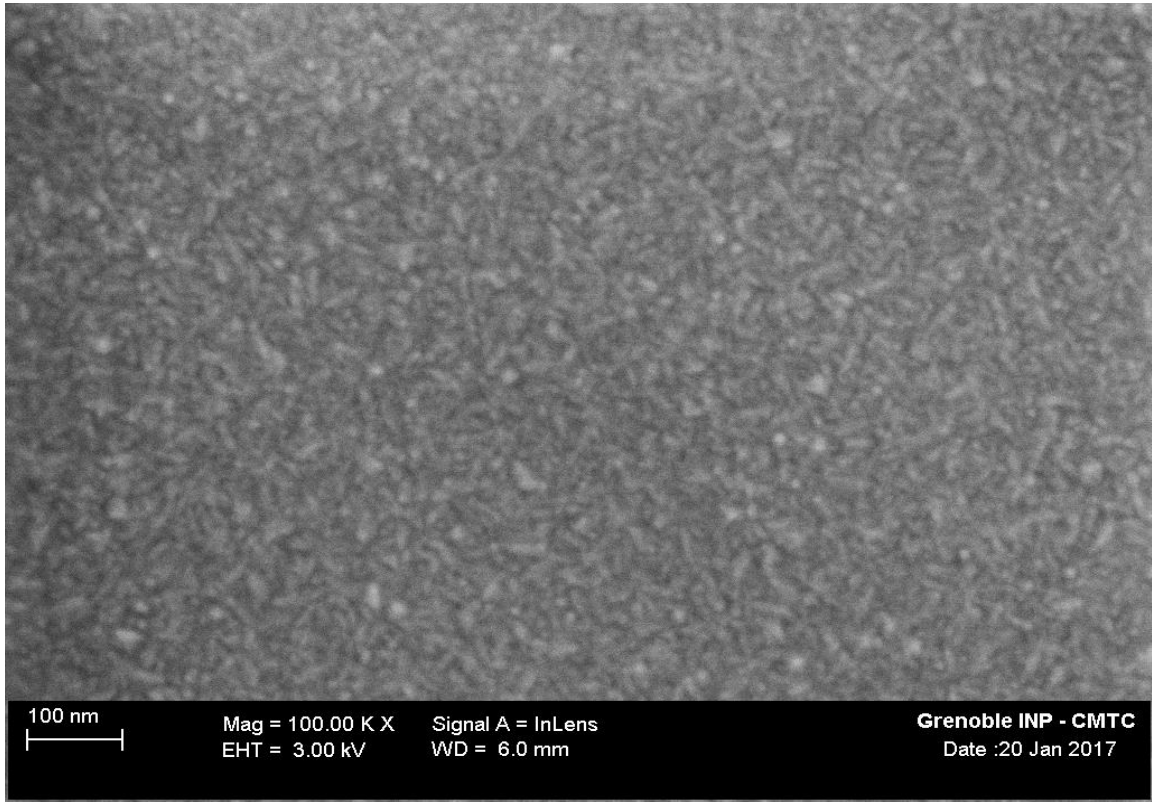
- Above 400°C @ 500W
- Above 350°C @ 1000W
- Above 250°C @ 2800W

AlN - SEM images

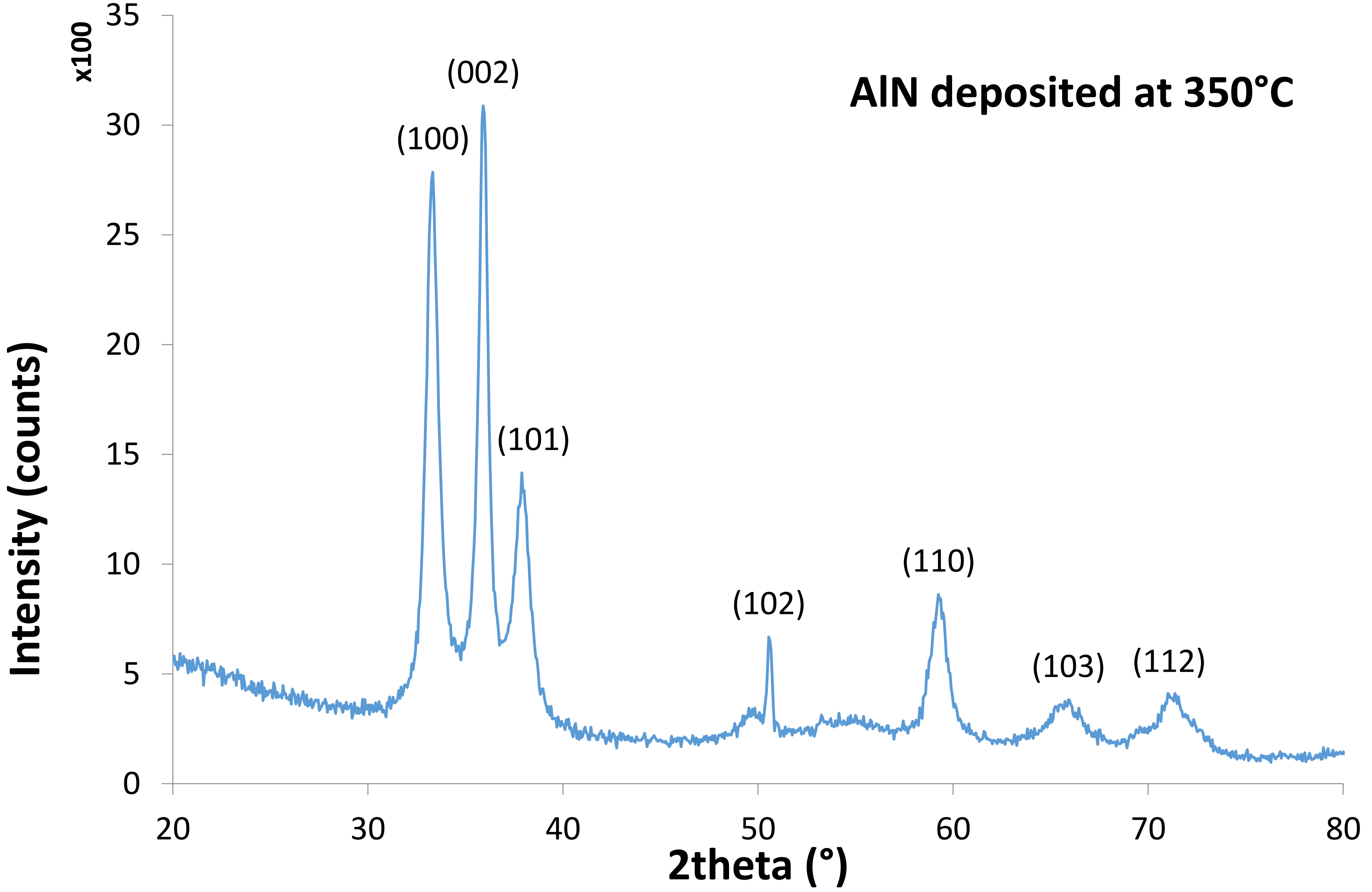
AlN deposited at 350 °C (90 nm thick)



AlN deposited at 400 °C (90 nm thick)

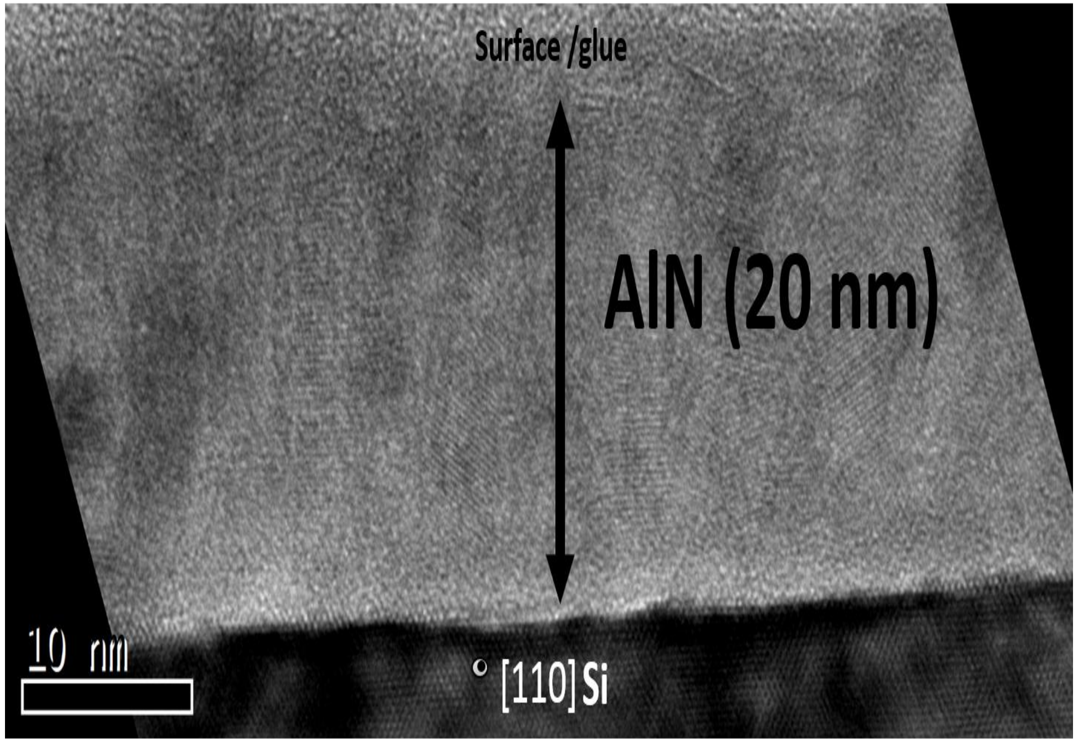


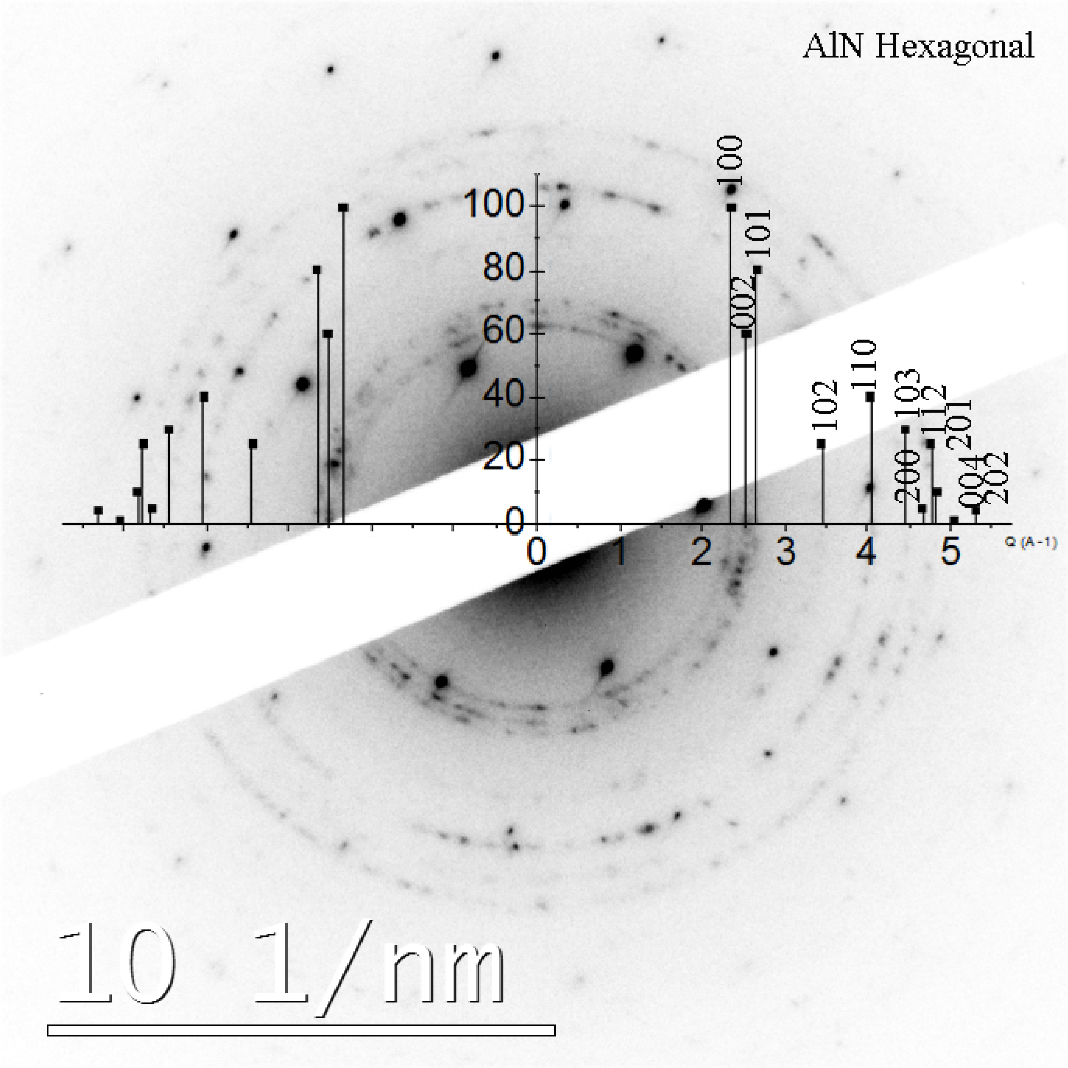
AlN - XRD


AlN deposited at 350°C
x100
Intensity (counts)
2theta (°)
35
30
25
20
15
10
5
0
20
30
40
50
60
70
80
(100)
(002)
(101)
(102)
(110)
(103)
(112)

AlN- TEM

TEM


Surface /glue
AlN (20 nm)
10 nm
[110] Si

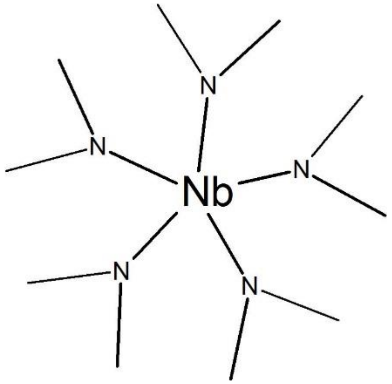

AlN Hexagonal
100
80
60
40
20
0
0
1
2
3
4
5
Q (A-1)
100
101
002
102
110
200
103
112
201
004
202
10 1/nm

Summary on AlN depositions

- **AlN has been successfully optimised with the following growth conditions:**

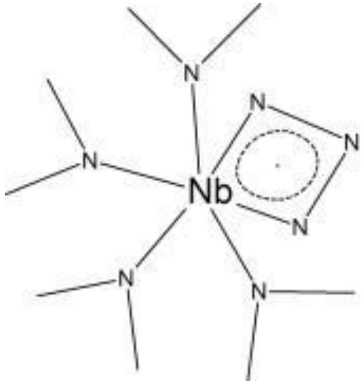
precursor TMA (20 °C)
growth temperature : 350 °C
plasma power: 2800 W

Niobium nitride deposition



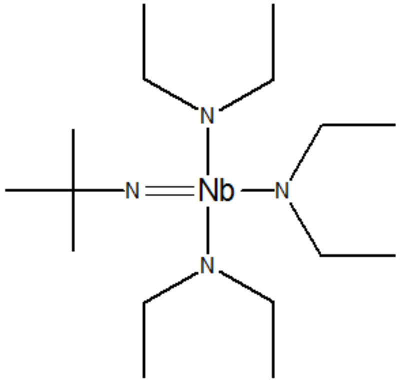
PDMAN

- Tested
- No transport



Modified PDMAN

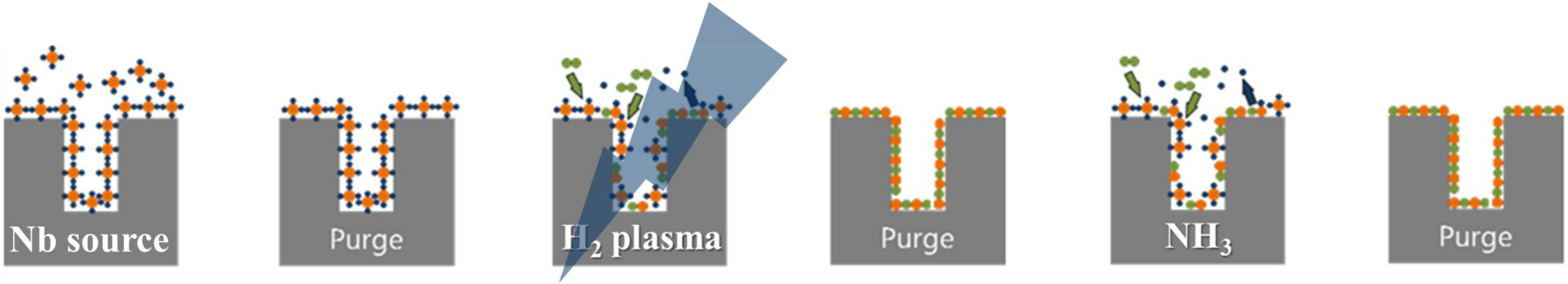
- Homemade
- Currently tested
- Transport @ 150°C



TBTDEN

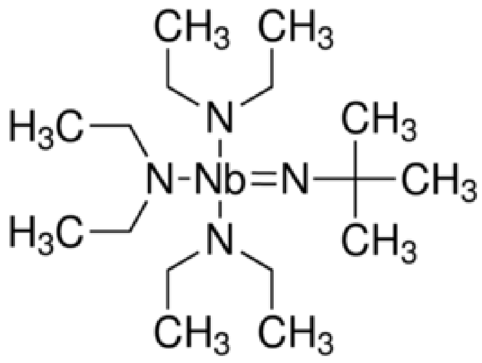
- Currently tested

General strategy : niobium reduction $\mathbf{Nb^{V}}$ (precursor) → $\mathbf{Nb^{III}}$ (NbN)



NbN thin films

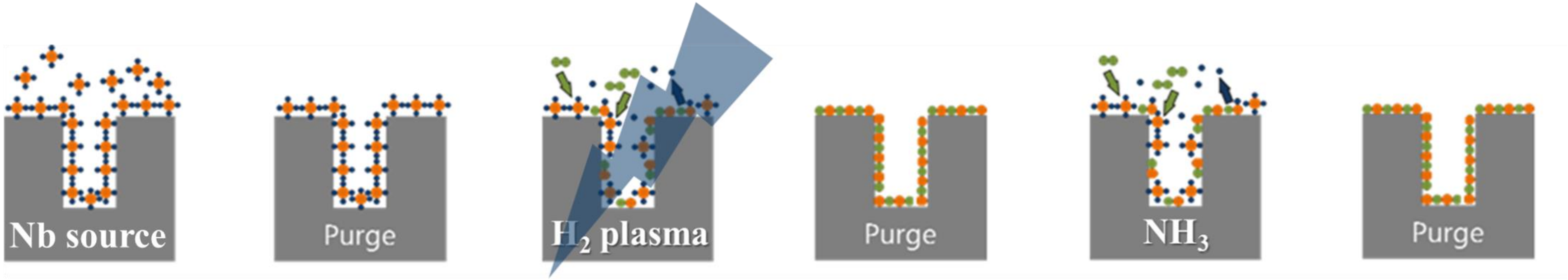
Metallic precursor Nb^{+V} : TBTDEN



General strategy : niobium reduction **Nb^{V}** (precursor) → **Nb^{III}** (NbN)

SuperCycle NbN

TBTDEN 1s / Purge 4s / H_2 plasma 12s / Purge 4s / NH_3 5s / Purge 4s

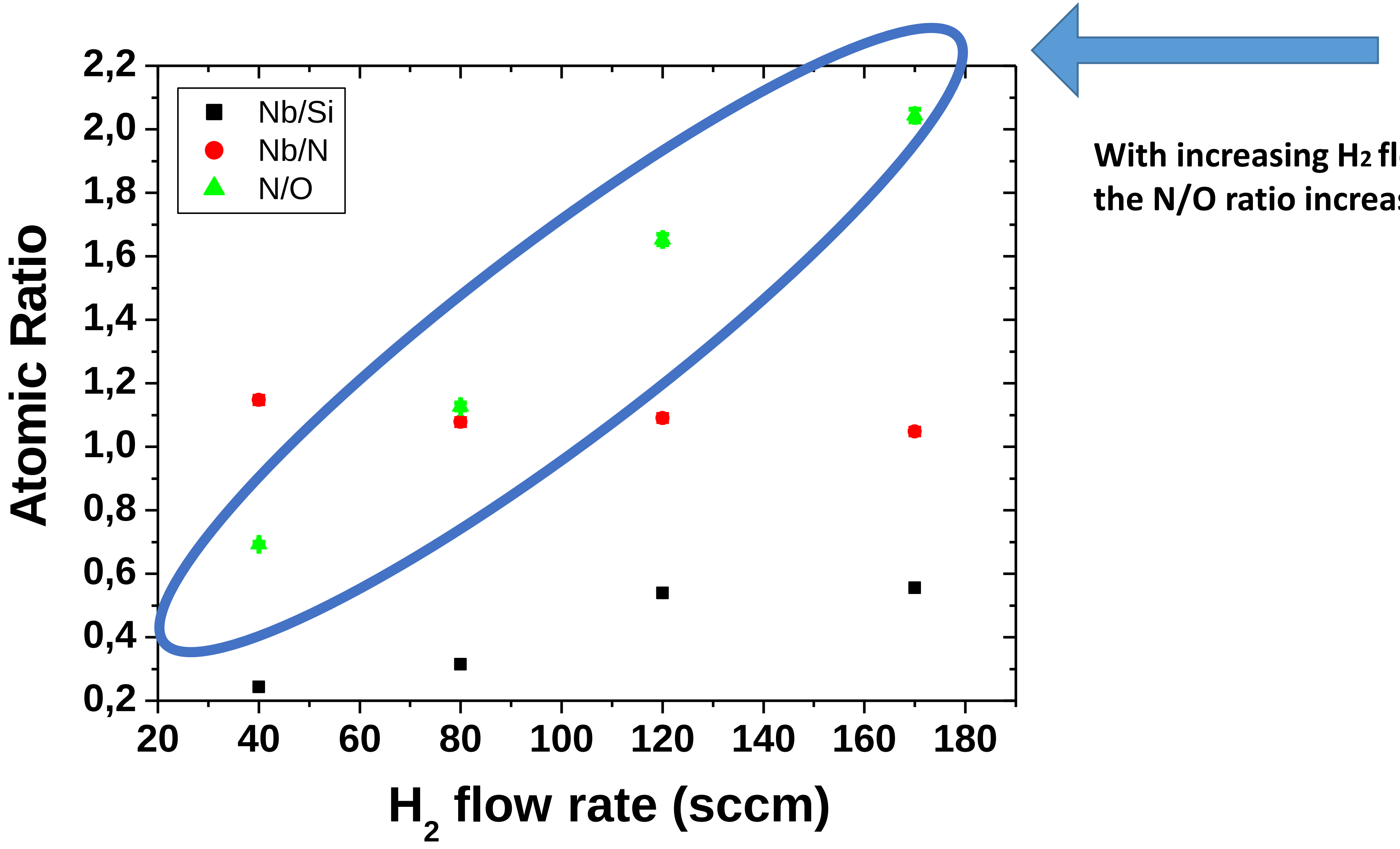


1s 4s 12s 4s 5s 4s

NbN thin films deposited as a function of H_2 flow rate

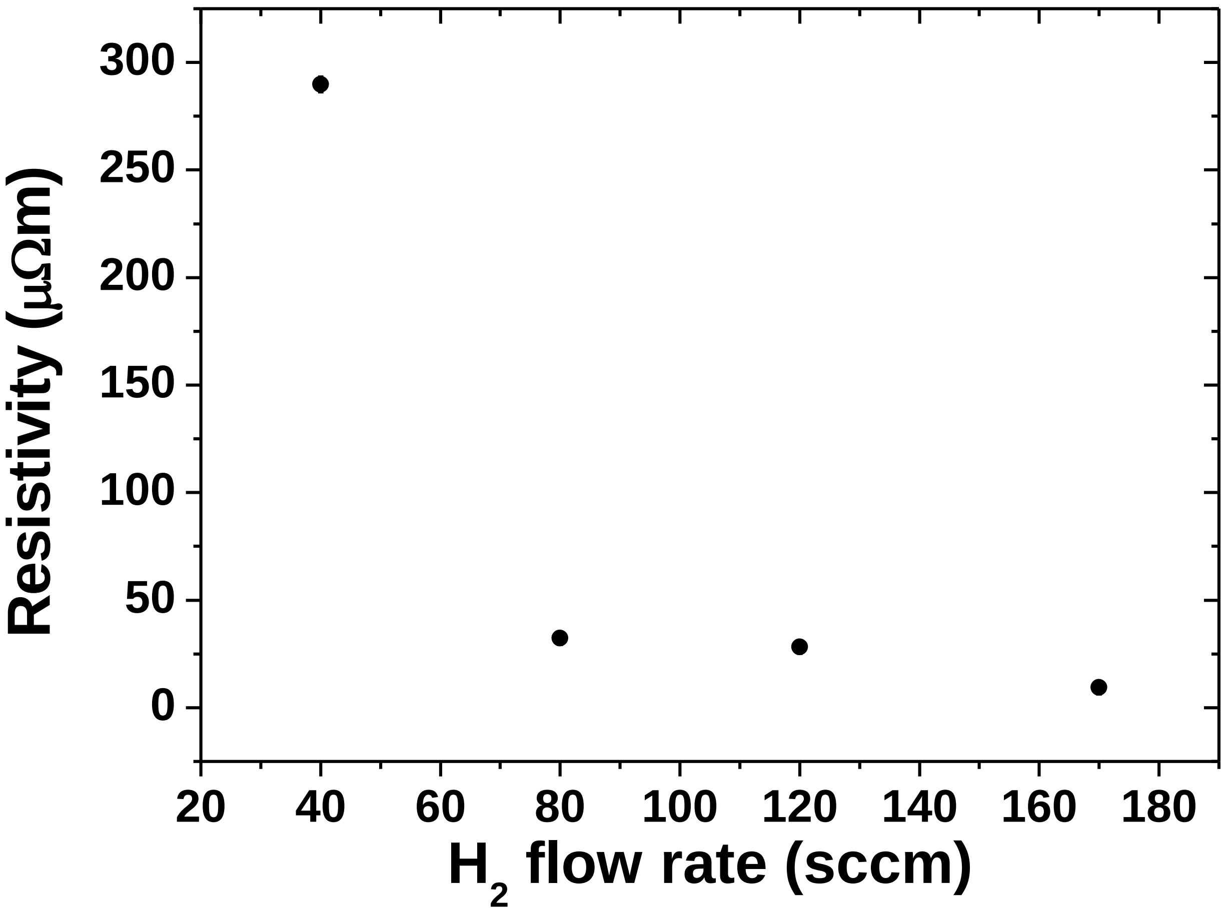
Sample No.	Growth conditions	Thickness
NbN7	350 °C; 2000 cycle; plasma:2.8 kW, 40sccm, 40hpa	60 nm
NbN8	350 °C; 2000 cycle; plasma:2.8 kW, 80 sccm, 40 hpa	60 nm
NbN9	350 °C; 2000 cycle; plasma:2.8 kW, 120 sccm, 40 hpa	60 nm
NbN10	350 °C; 2000 cycle; plasma:2.8 kW, 170 sccm, 40 hpa	60 nm

NbN- EDX



With increasing H_2 flow rate, the N/O ratio increases

NbN- Resistivity

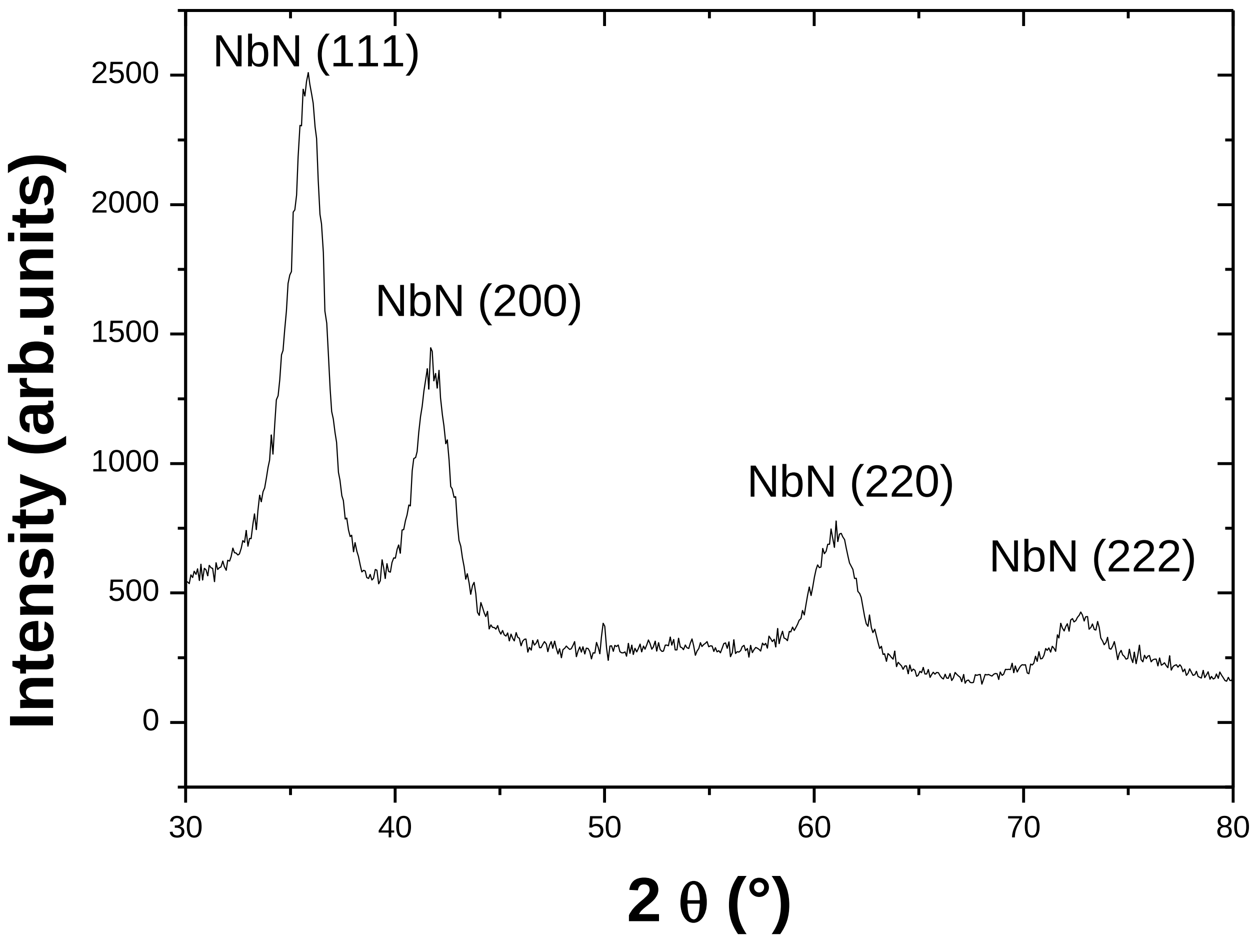


H_2 flow rate	Resistivity (μ Ω m)
40 sccm	289,75±3
80 sccm	32,3±0,3
120 sccm	28,20±0,3
170 sccm	9,38±0,1

Resistivities were measured by Van der Pauw measurements at room temperature

NbN - XRD

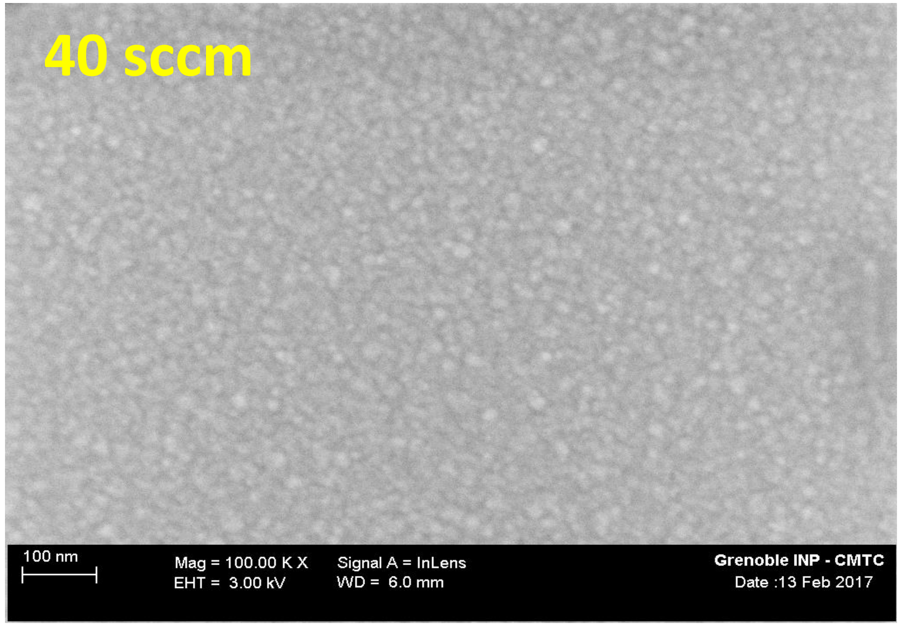
NbN Cubic δ phase

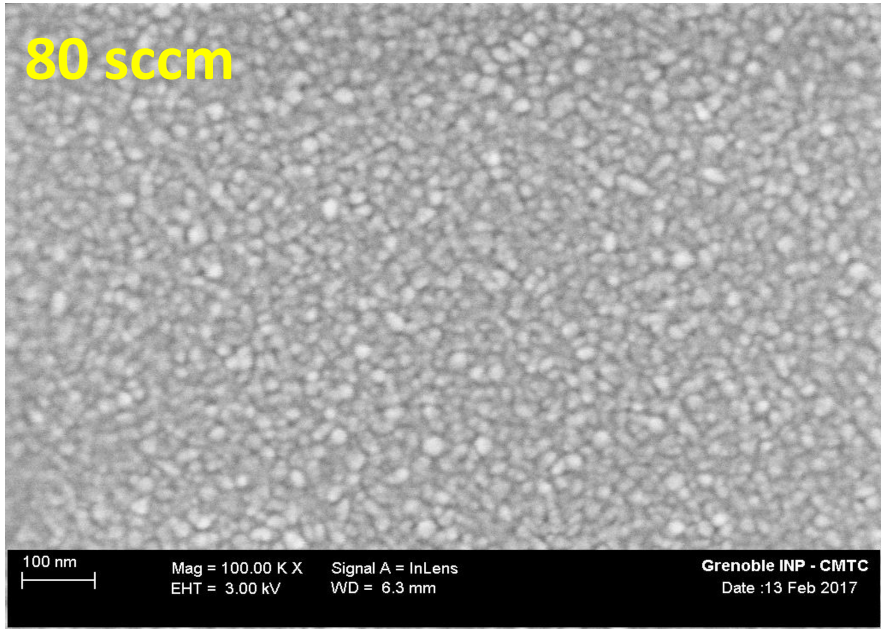


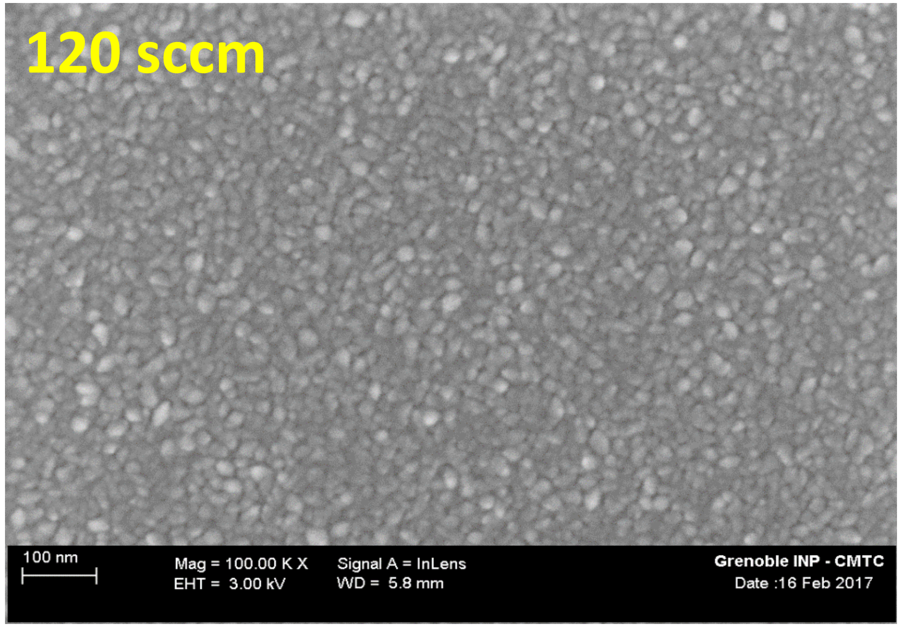
Calculated lattice parameter: a = 0.4335 nm

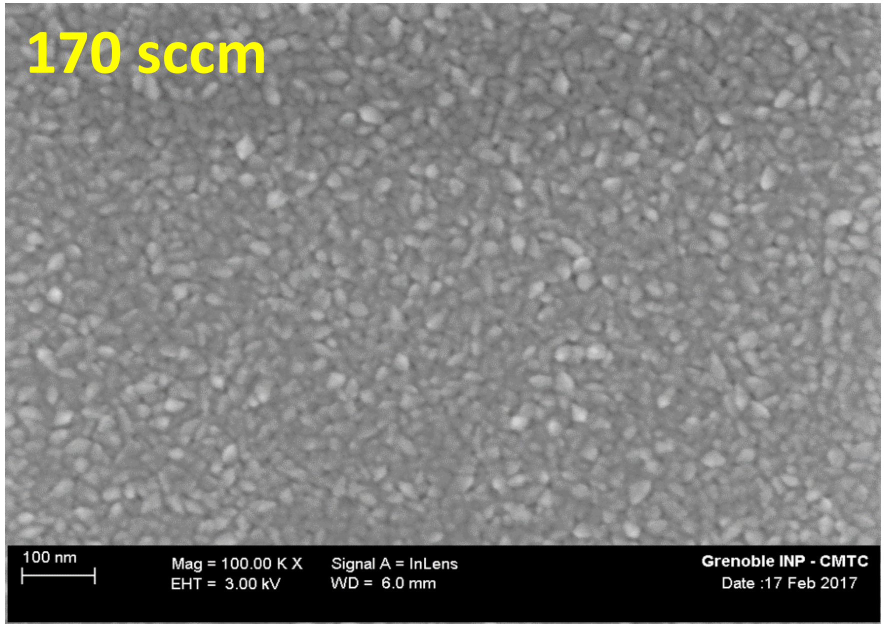
Reference: bulk NbN a= 0.439 nm

NbN - SEM


40 sccm
100 nm
Mag = 100.00 K X
EHT = 3.00 kV
Signal A = InLens
WD = 6.0 mm
Grenoble INP - CMTC
Date :13 Feb 2017


80 sccm
100 nm
Mag = 100.00 K X
EHT = 3.00 kV
Signal A = InLens
WD = 6.3 mm
Grenoble INP - CMTC
Date :13 Feb 2017


120 sccm
100 nm
Mag = 100.00 K X
EHT = 3.00 kV
Signal A = InLens
WD = 5.8 mm
Grenoble INP - CMTC
Date :16 Feb 2017


170 sccm
100 nm
Mag = 100.00 K X
EHT = 3.00 kV
Signal A = InLens
WD = 6.0 mm
Grenoble INP - CMTC
Date :17 Feb 2017

NbN - Characterization

T_C measurement : not superconducting yet

TEM of NbN is ongoing at LMGP

Summary on NbN

- The best NbN sample was deposited with percursor TBTDEN
  Substrate temperature 240 °C
  Plasma power 2000 W
  Growth rate 2.2 nm/h

- GIXRD result showed NbN cubic δ phase, no indication of niobium oxide crystallites

AlN/NbN multilayers

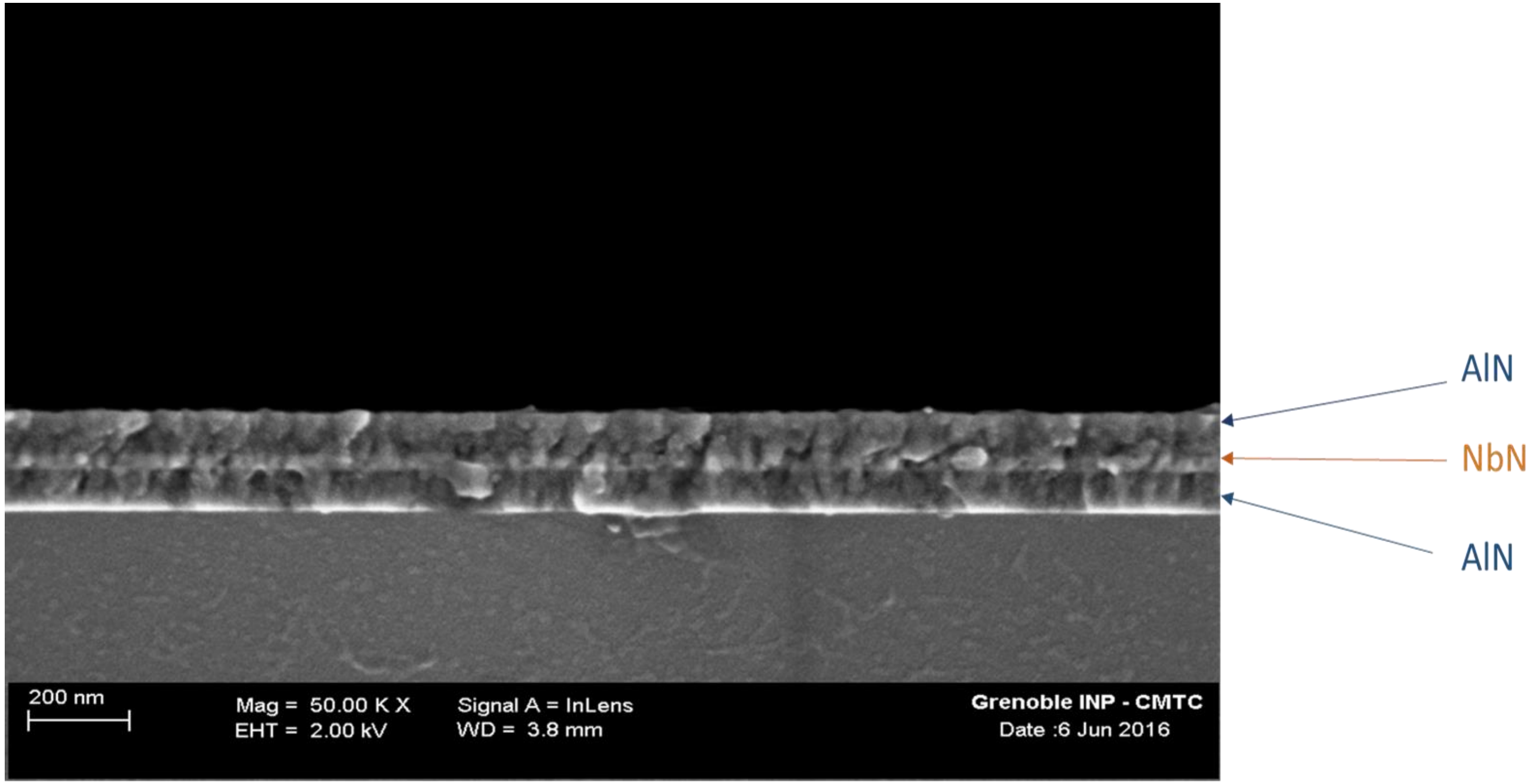
SuperCycle AlN à 350°C

TMA 0,1s / Purge 4s / H_2 plasma 12s / Purge 4s / NH_3 5s / Purge 4s

SuperCycle NbN à 240°C

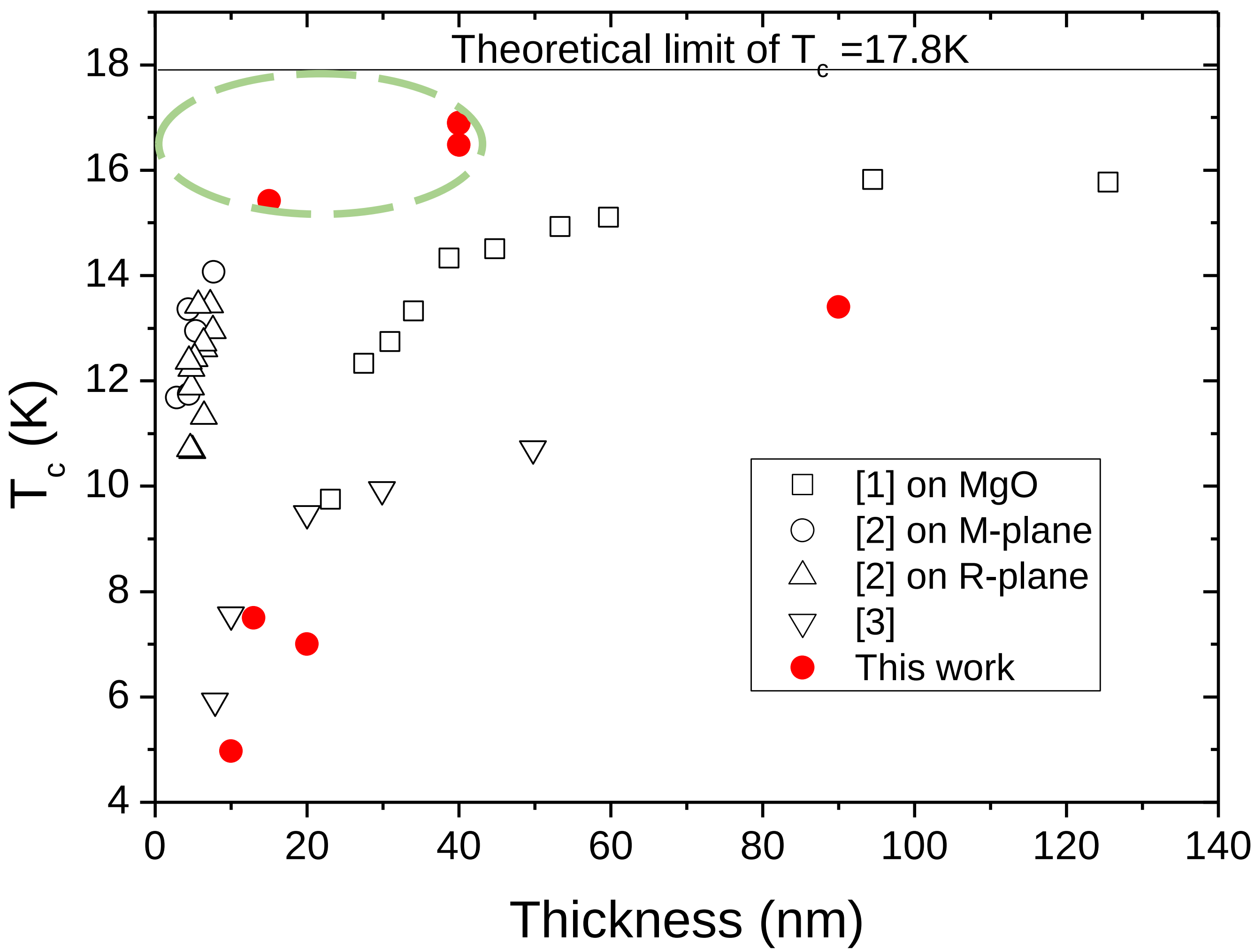
TBTDEN 1s / Purge 4s / H_2 plasma 12s / Purge 4s / NH_3 5s / Purge 4s

AlN/NbN multilayers



IB-NbN-12 260°C (NbN)

Superconducting properties



[1] Miki et al. Applied Physics Express 2 (2009) 075002
[2] Villegier et al. APL 2009
[3] Shiino et al. Supercond. Sci. Technol. 23 (2010) 045004

Highest T_c reported so far (17.06K) for NbN together with low resistivity value (50 µΩcm)

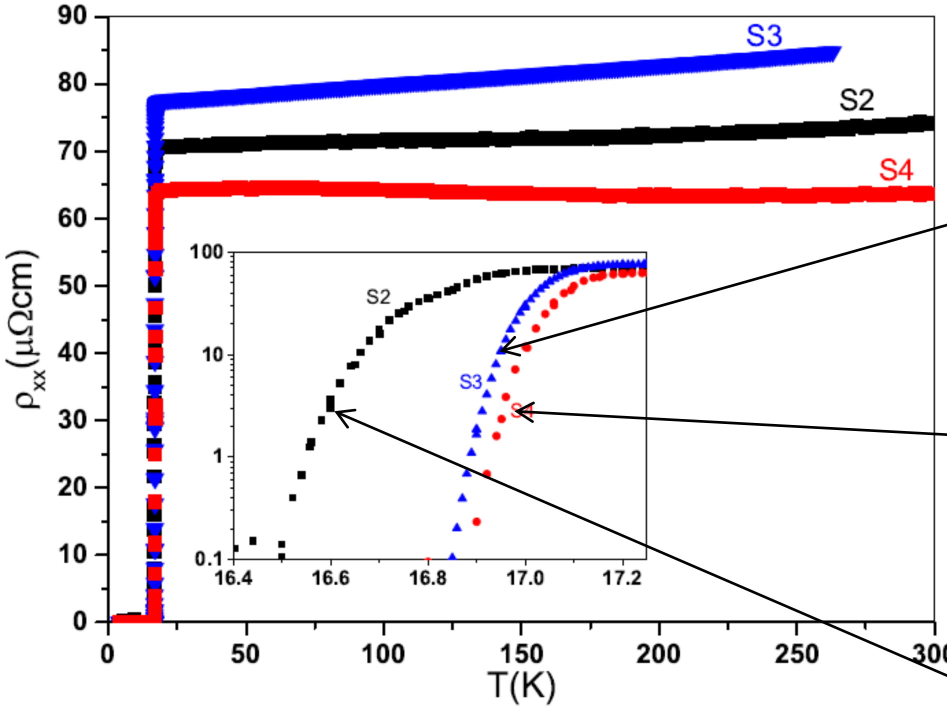
NbTiN/NbN multilayers by CVD

Crystalline quality vs electrical transport

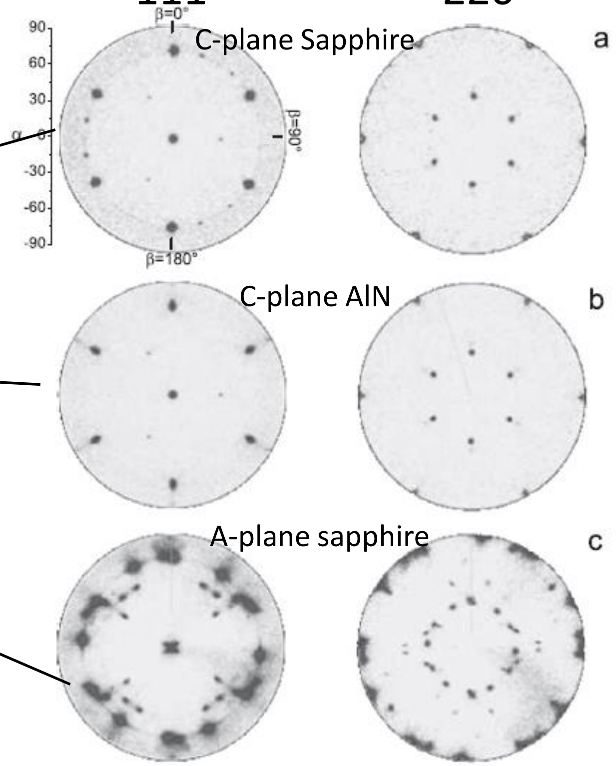
Electrical transport measurements performed down to 4 K in a PPMS.

Same growth conditions, different substrates

Temperature variation of resistance



Pole figures



CVD deposition of ML on HZB cavities



- Nb samples from the HZB cavities treated (BCP + heat treatment at Saclay) and sent to Grenoble
- Will be deposited with AlN/NbN layers by CVD and/or ALD
- Will be tested back at Saclay (small sample, magnetometry) and HZB (RF) mid-2017

Conclusions and Future work

- PEALD of AlN has been optimized
- PEALD of NbN has the expected cubic δ phase
- High quality NbN obtained by CVD (guideline for ALD)

Future work

- Development of the ALD process to get SC NbN
- AlN/NbN/AlN multilayers will be grown with various thickness combinations and will be characterized