

# Progress with Nb/Cu film engineering with energetic condensation

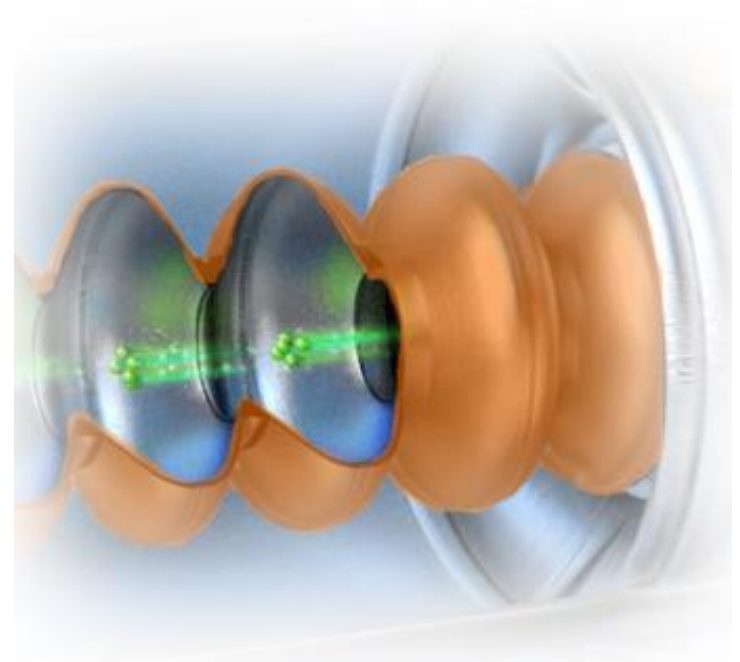
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O. Trofimova, J.K. Spradlin

L. Vega Cid, M. Arzeo, S. Aull - CERN

S. Keckert - HZB

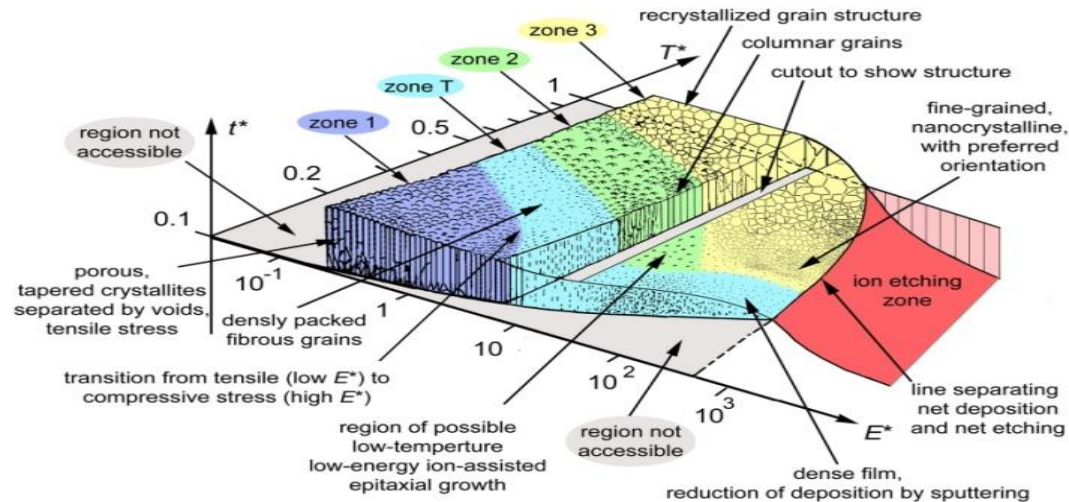


# OUTLINE

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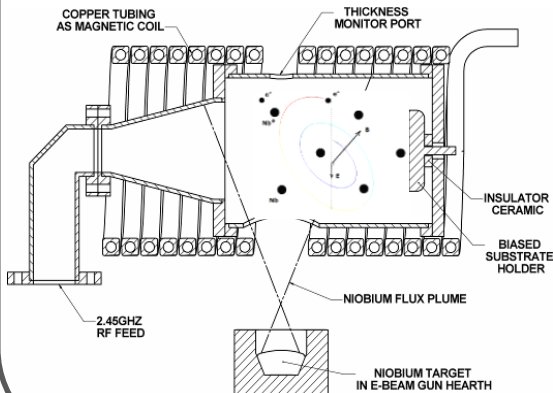
- Introduction
- ECR Nb/Cu film structure
- RF Behavior of ECR Nb/Cu measured by QPR
- Conclusion

# Energetic Condensation via ECR



Anders, André. "A structure zone diagram including plasma-based deposition and ion etching." *Thin Solid Films* 518.15 (2010): 4087-4090.

## Electron Cyclotron Resonance (ECR)



No working gas

**Singly charged ions**  
(64eV)

**produced in vacuum**

**Controllable**  
**deposition energy**  
**with Bias voltage**

Excellent bonding

No macro particles

## High Power Impulse Magnetron Sputtering (HiPIMS)

Requires working gas

**Multiply charged ions of Nb & Kr**

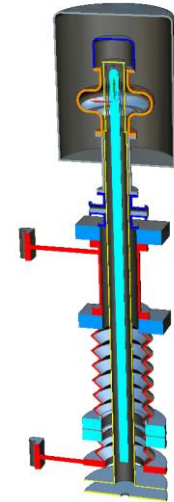
**Presence of neutrals**

**Controllable**  
**deposition energy**  
**with Bias voltage**

Excellent bonding

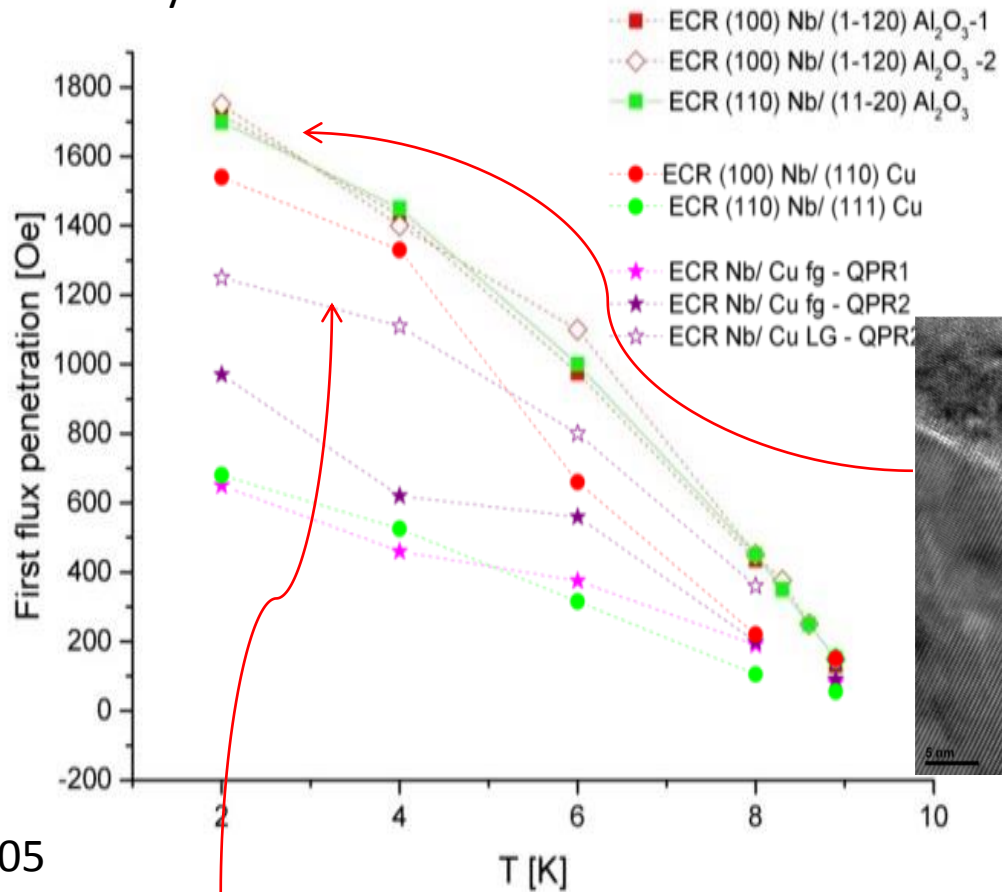
No macro particles

Ease of cavity configuration

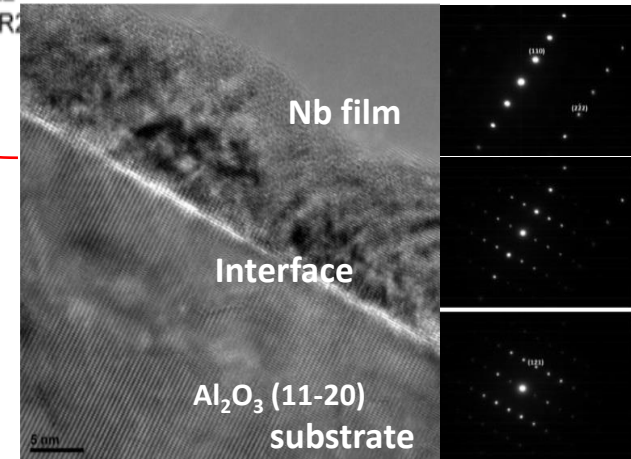


# ECR Nb film properties

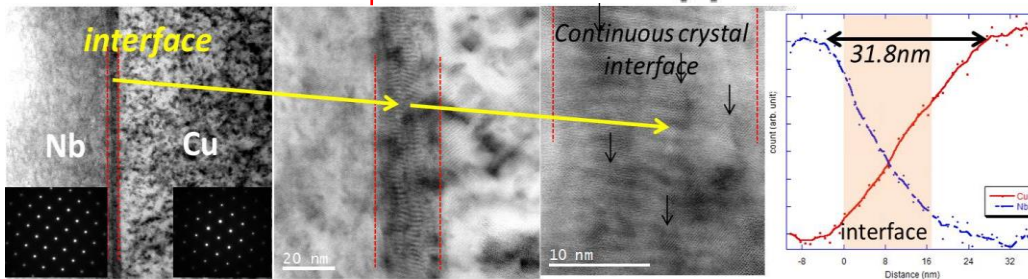
## DC SQUID Magnetometry



RRR 725



RRR 305



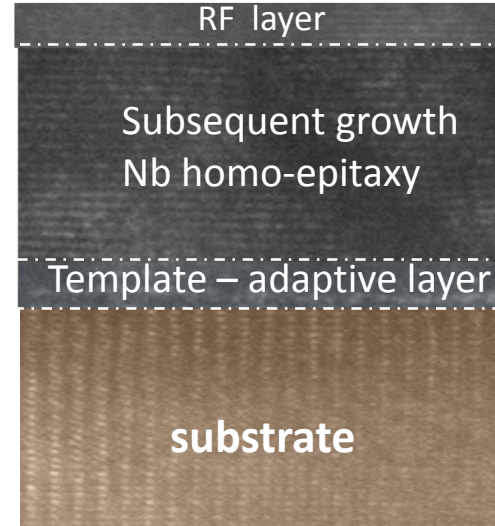
**Full control of interface for enhanced adhesion**

# Energetic Condensation via ECR

## Energetic Condensation

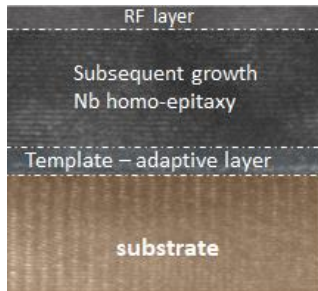
Sequential phases for film growth

- ❑ Interface
- ❑ Film nucleation
- ❑ Growth of appropriate template for subsequent deposition
- ❑ Deposition of final surface optimized for minimum defect density.

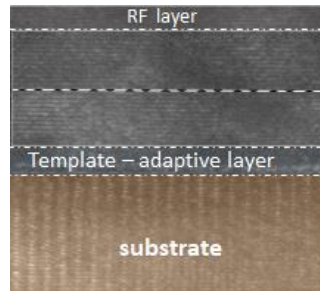


## Opportunity for film engineering

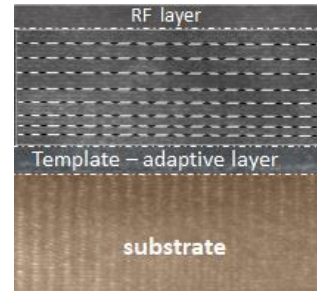
### Continuous



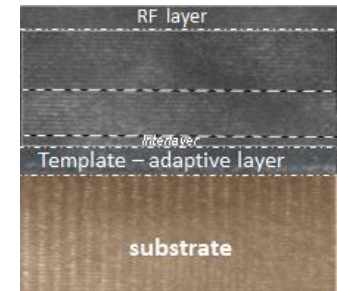
### Interrupted



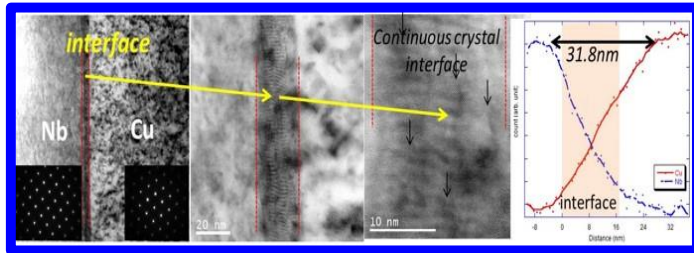
### Interrupted multiple steps



### Interlayer/Interrupted



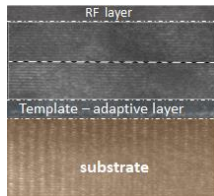
# Nb Film Structure Engineering



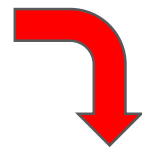
Nb/Cu



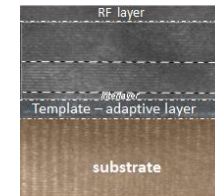
360 °C



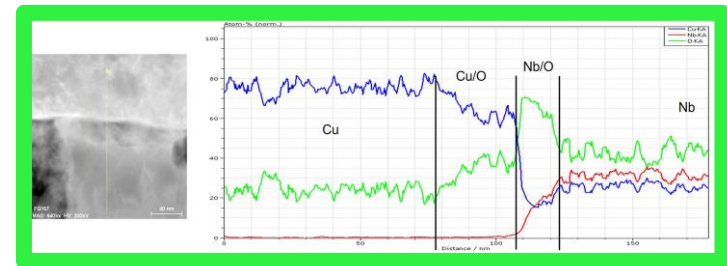
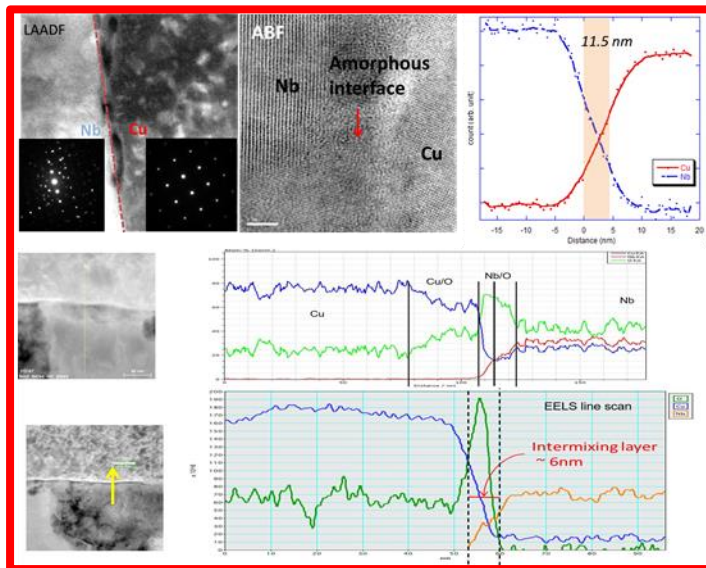
200 °C



Nb/CuO



Nucleation 184 eV  
Subsequent growth 64 eV



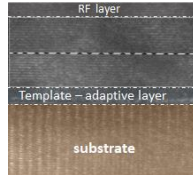
Nb/Nb<sub>2</sub>O<sub>5</sub>/Cu

# Nb film engineering - Hetero-epitaxy at 360°C

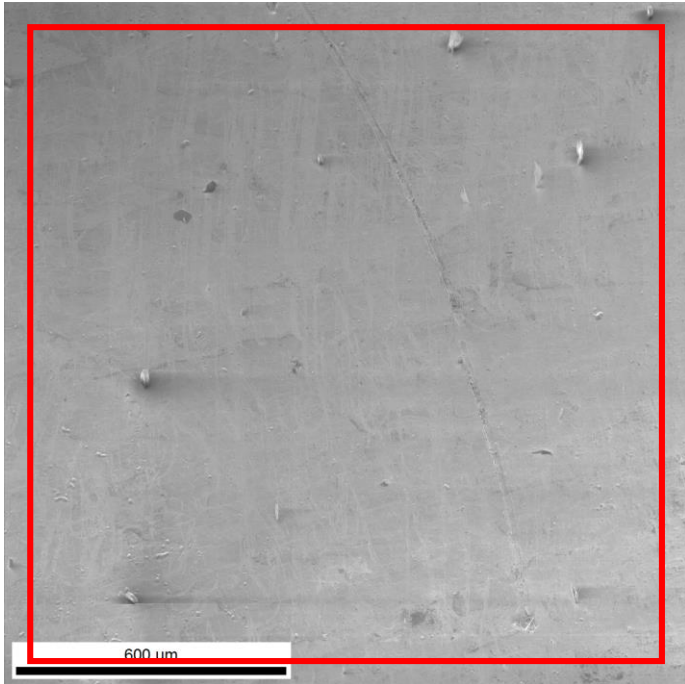
## Nucleation @ 184 eV + subsequent growth @ 64 eV in 2 steps



IPF Map+CI 0.1

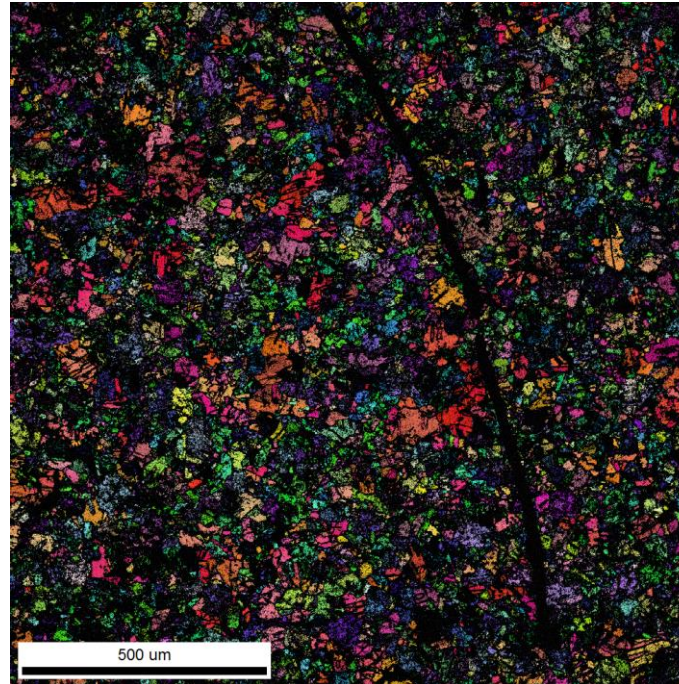


SE Image with EBSD Region Highlighted



1500 μm

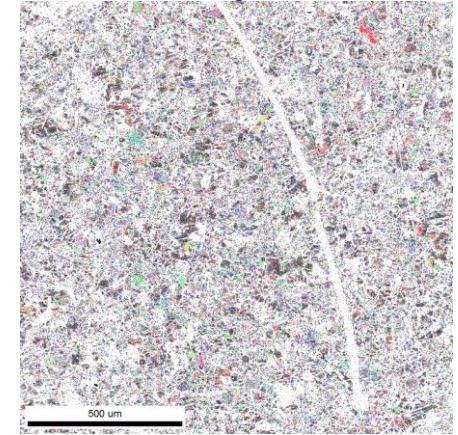
IPF w CI of 0.1



Gray Scale Map Type: Confidence Index  
0...0.971 (0.000971...0.971)

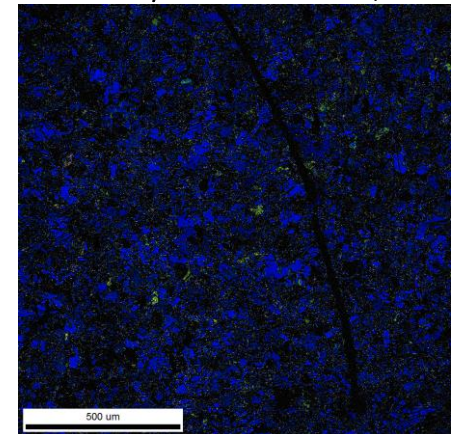


GROD Angle vs Axis



Gray Scale Map Type: Grain Reference Orientation Deviation - Angle  
0...18.3625 (-0.342974...1.23185)

Geometrically Nec Dislocations w/ CI of 0.1



Color Color Map Type: Geometrically Necessary Dislocations

| Min | Max     | Total Fraction | Median Fraction |
|-----|---------|----------------|-----------------|
| 0   | 738.879 | 0.369          | 0.369           |

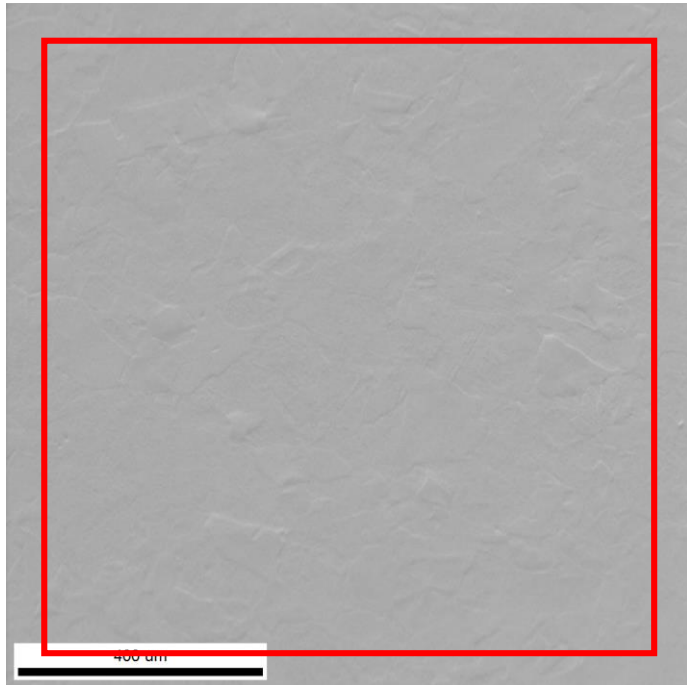
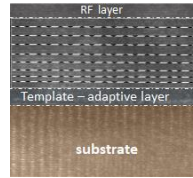


# Nb film engineering- Altered hetero-epitaxy at 360°C

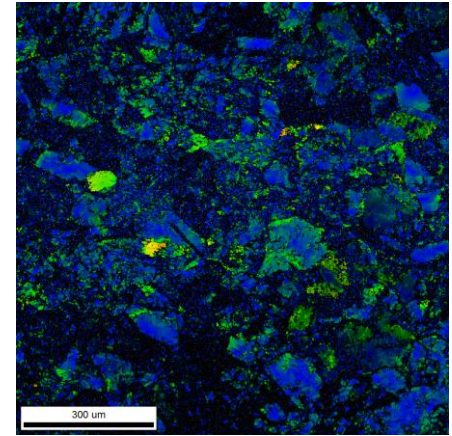
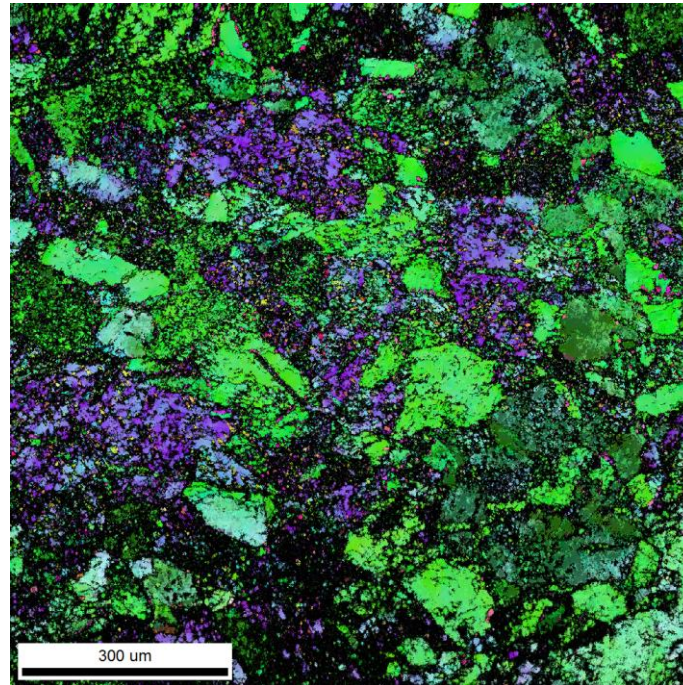
## Nucleation @ 184 eV + subsequent growth @ 64 eV in 10 steps



IPF Map+CI 0.1

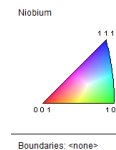
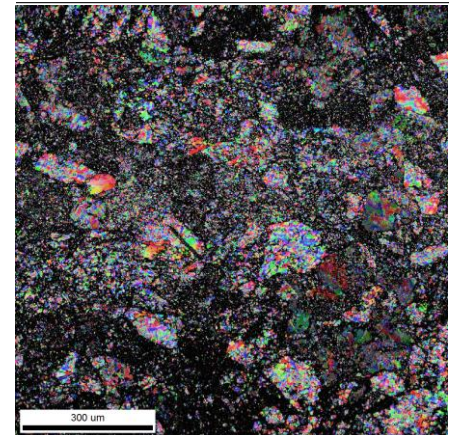


1.5 mm



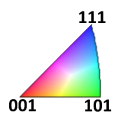
Color Coded Map Type: Grain Reference Orientation Deviation - Angle

|  | Min | Max     | Total Fraction | Partition Fraction |
|--|-----|---------|----------------|--------------------|
|  | 0   | 42.5711 | 0.750          | 0.750              |



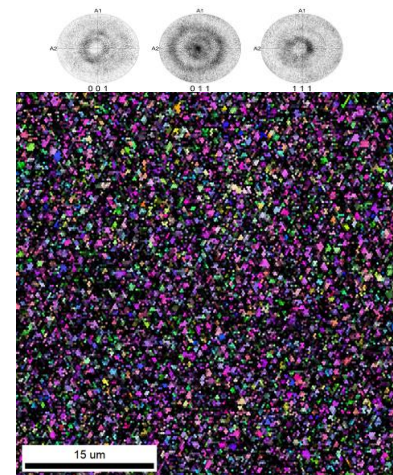
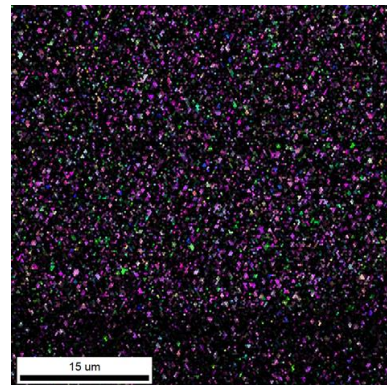
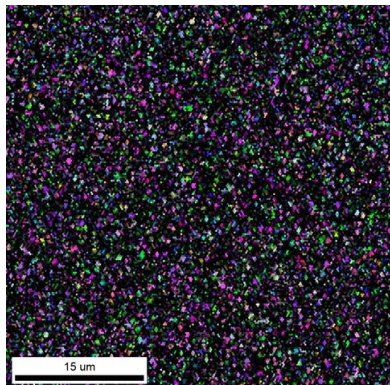
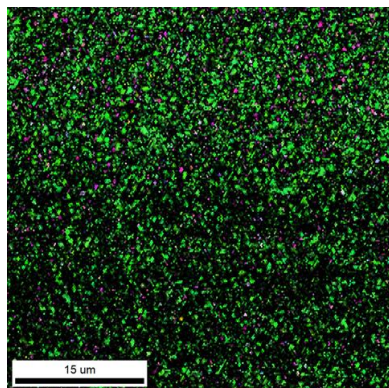


# Nucleation + 10 sequential coating : influence of energy

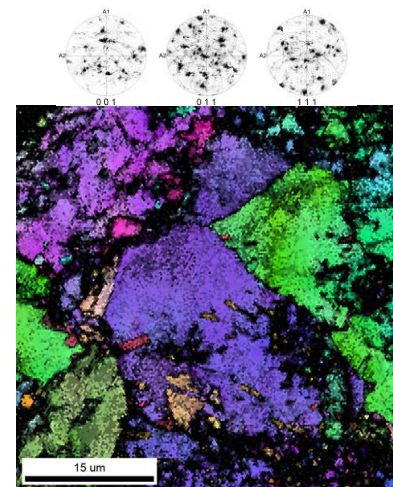
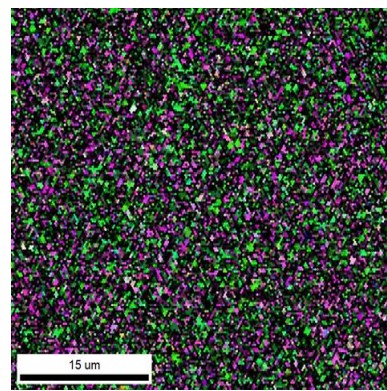
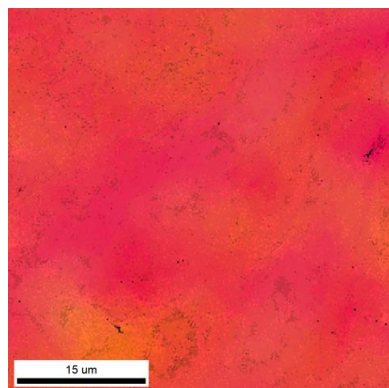
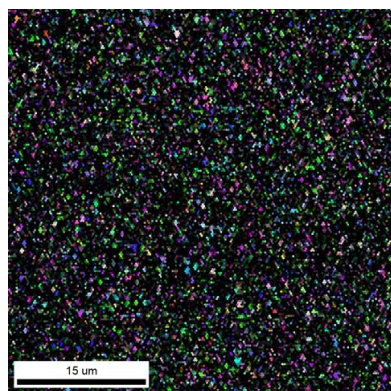


IPF Map+CI 0.1

94 eV



244 eV



Cu 100

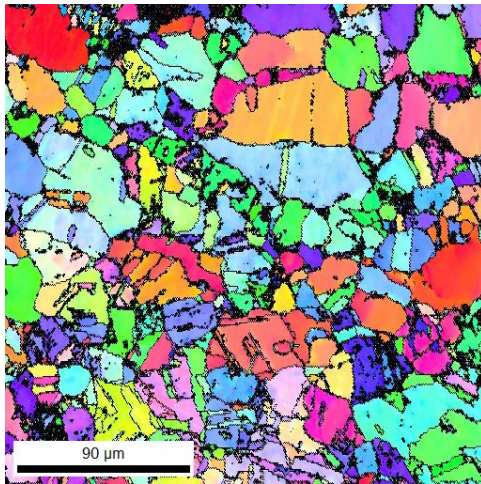
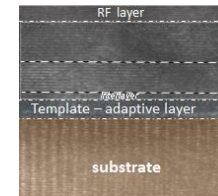
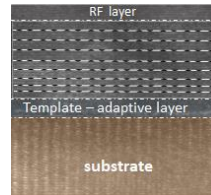
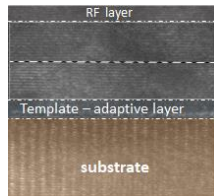
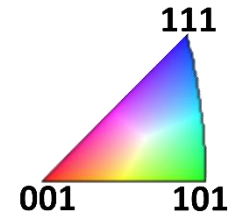
Cu 110

Cu 111

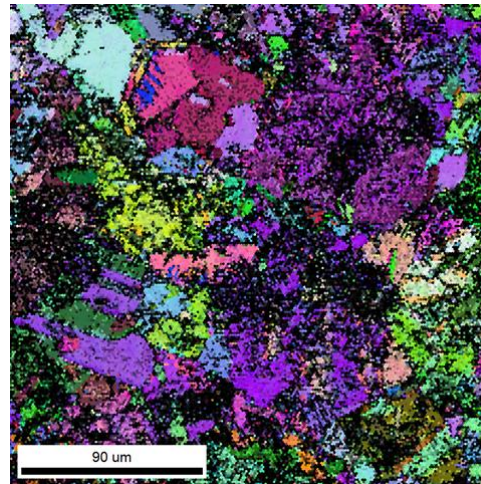
Cu fine grains

# Nb Film Engineering – Altered heteroepitaxy

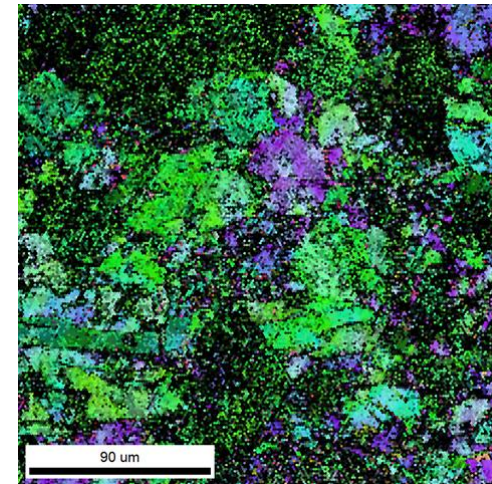
Nucleation at 184 eV , 360C



Hetero-epitaxial growth

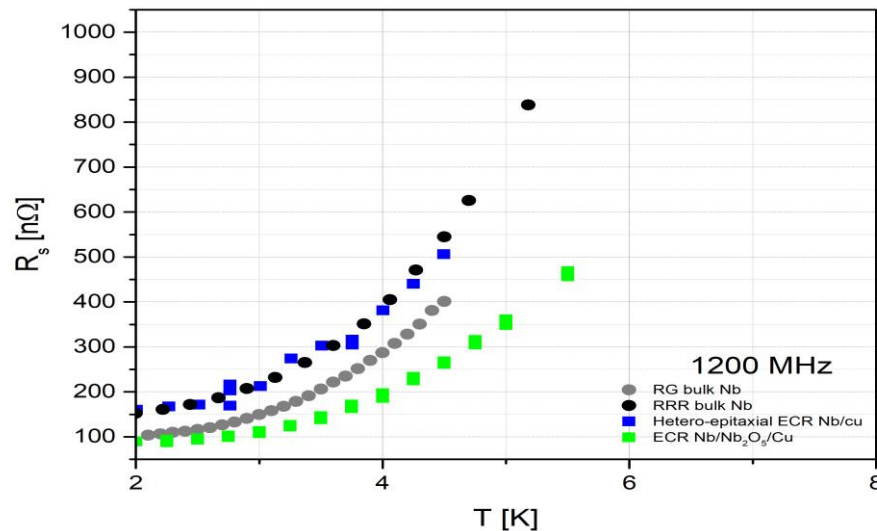
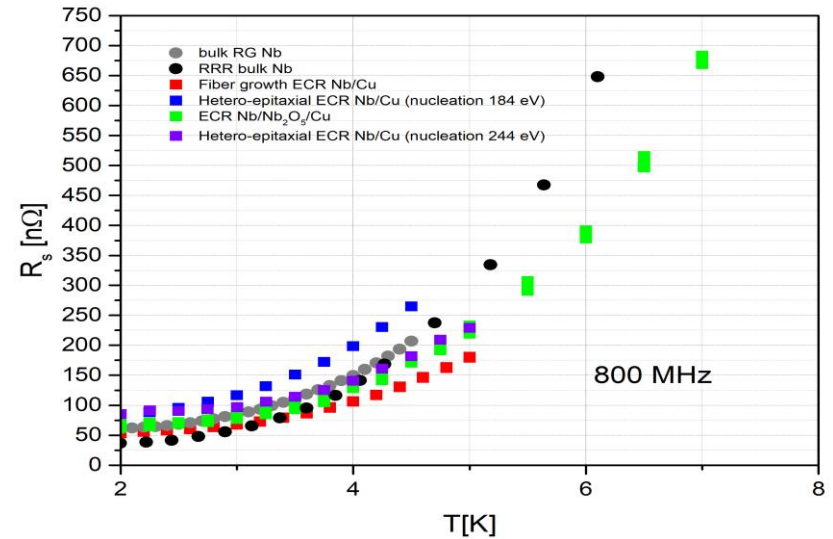
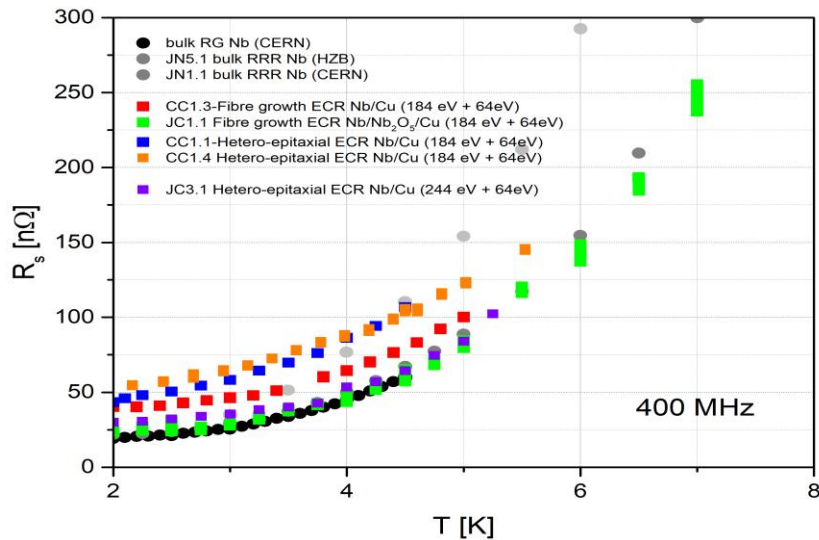


Hetero-epitaxial growth disrupted with subsequent re-nucleation?



Same large crystal size but preferentially (110)?

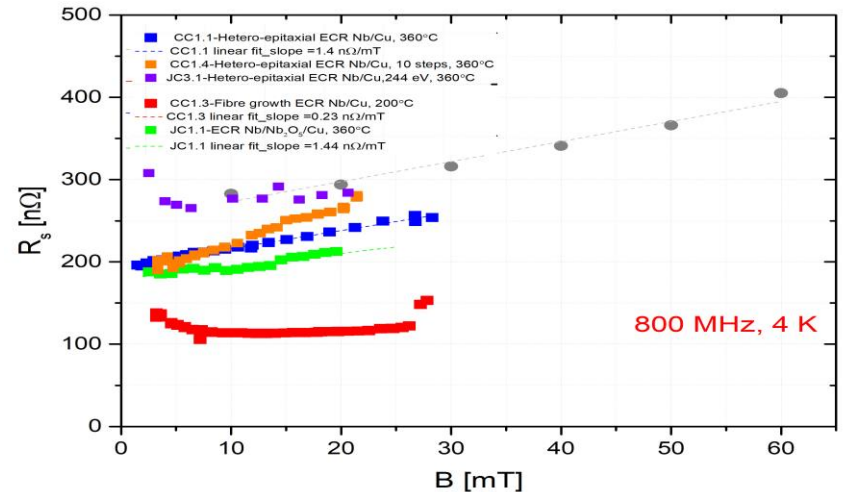
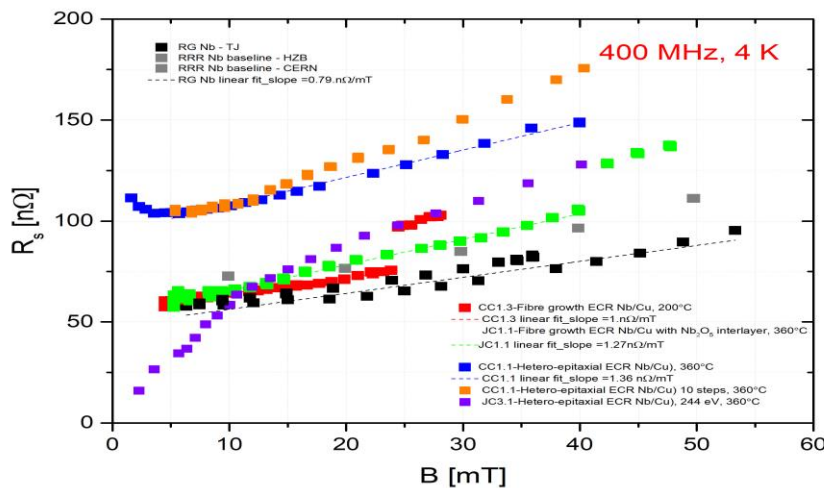
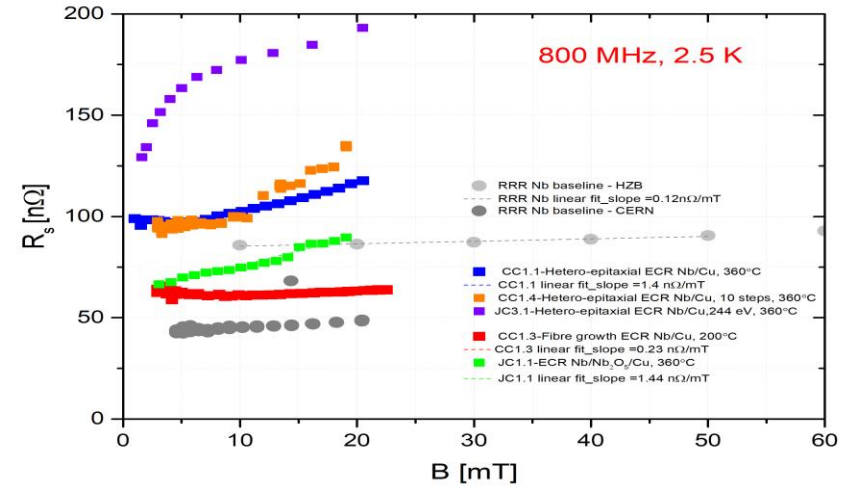
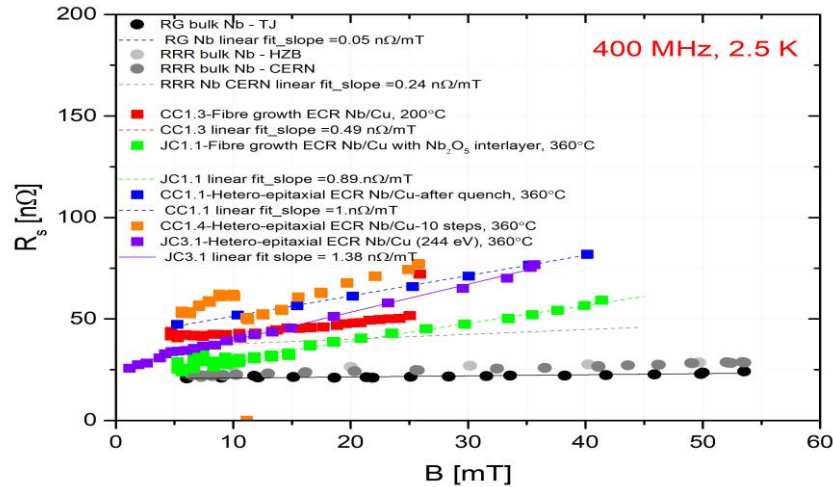
# ECR Nb films $R_s$ vs T



**Fiber growth ECR Nb/Cu films  
behave closer to bulk Nb in  
QPR measurements**

# ECR Nb/Cu Film RF Results on QPR samples

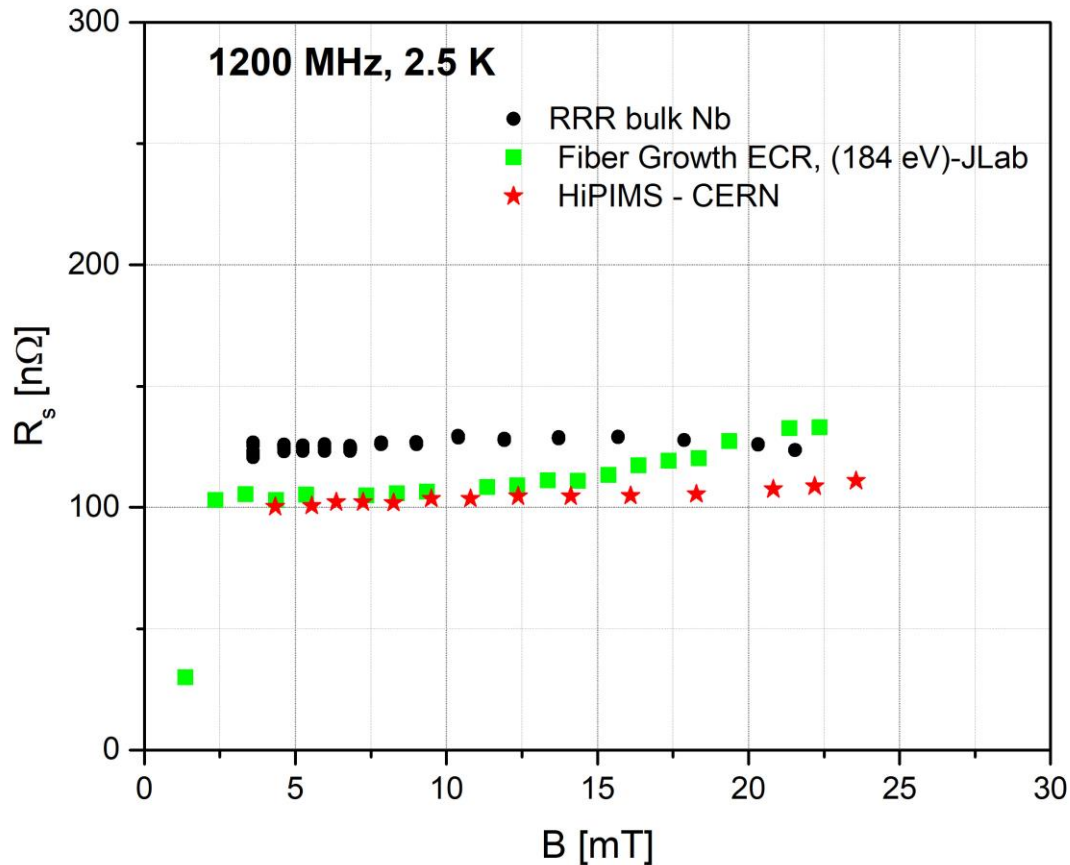
RF measurement at CERN: S. Aull, M. Arzeo, L. Vega Cid



**Some ECR films show mitigation of the Q-slope  
Insight on interface, coating temperature influence**

# ECR Nb/Cu Film RF Results on QPR samples

RF measurement at CERN: S. Aull, M. Arzeo, L. Vega Cid

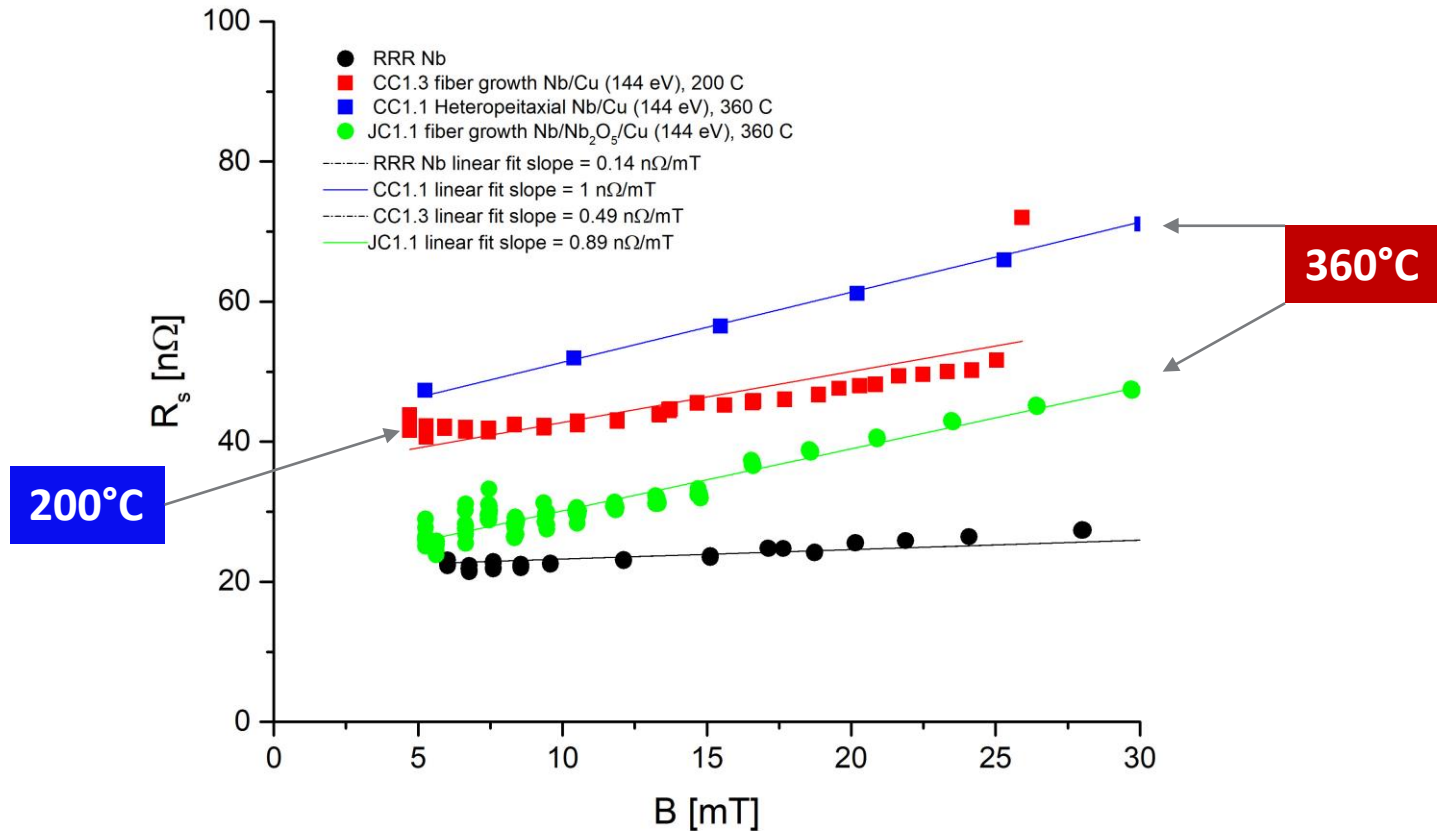


**Energetic Condensation Nb/Cu films show similar RF behavior compare to bulk Nb in QPR measurements**

# ECR Nb/Cu Film – Fiber Growth vs. Hetero-epitaxy

RF measurement at CERN: S. Aull, M. Arzeo, L. Vega Cid

400 MHz, 2.5 K



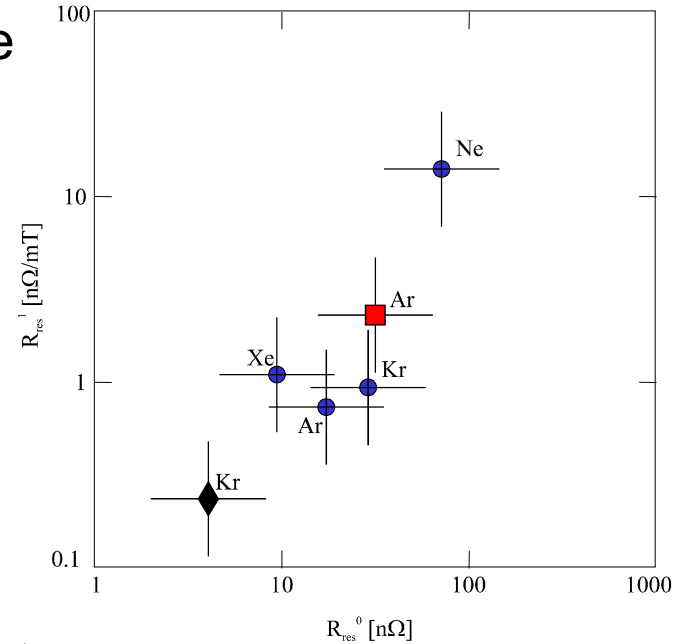
**Fiber growth ECR Nb/Cu show better mitigation trend of the Q-slope  
Insight on interface, coating temperature influence**

# CONCLUSION

- ❑ Several “knobs to turn” to tune Nb film structure and superconducting properties
- ❑ Mitigation of Q-slope for energetic condensation films

❑ **ECR fiber growth films thus far seem better performing than hetero-epitaxial films. Also observed for HiPIMS Nb/Cu (cf. F. Avino’s talk)**

**cf. CERN DC magnetron sputtering studies**



Physica C 351 (2001) 421-28

- ❑ Establish adequate process controls
- ❑ **Need better substrates and chemical processes**
- ❑ Need more RF measurements statistics