UNIVERSITÀ DEGLI STUDI DI PADOVA
SCIENCE FACULTY

MATERIAL SCIENCE DEGREE

INFN – LABORATORI NAZIONALI DI LEGNARO

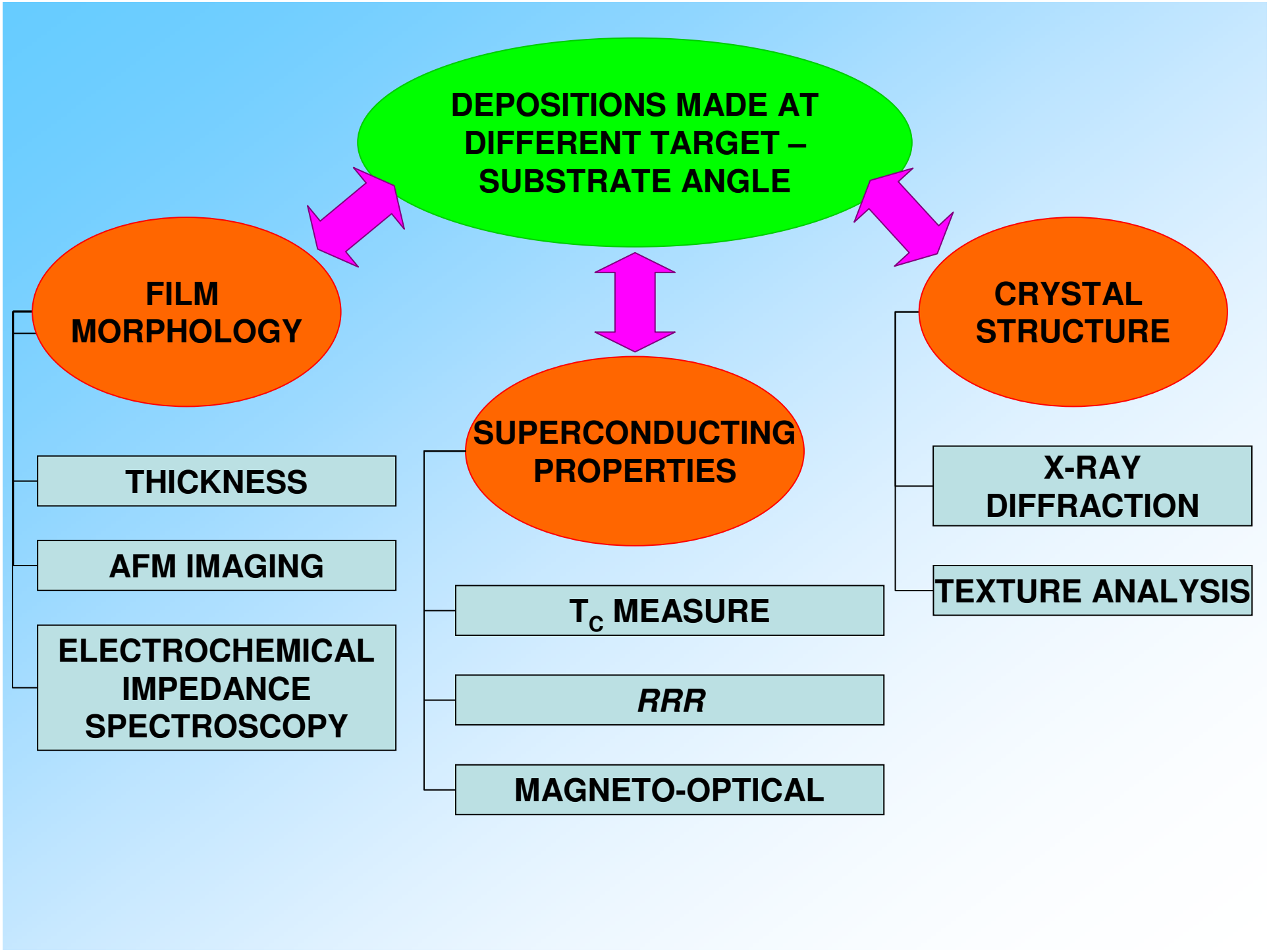
DIEGO TONINI

MORPHOLOGY OF NIOBIUM FILMS SPUTTERED AT DIFFERENT TARGET – SUBSTRATE ANGLE

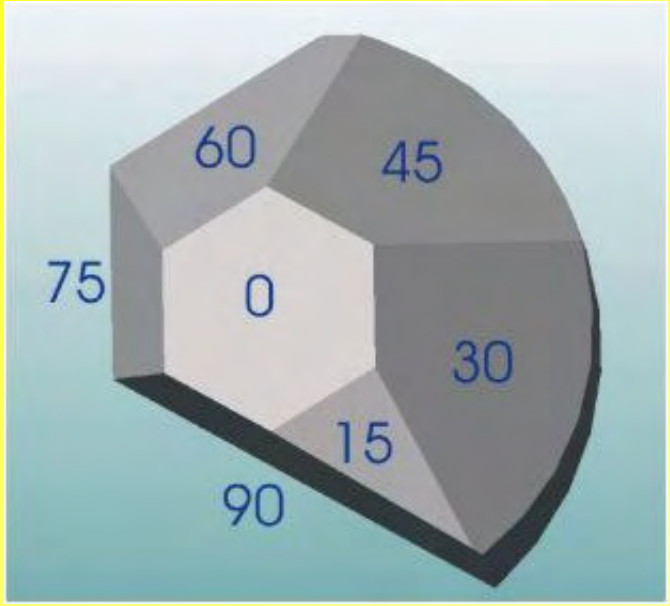
2 QUESTIONS…

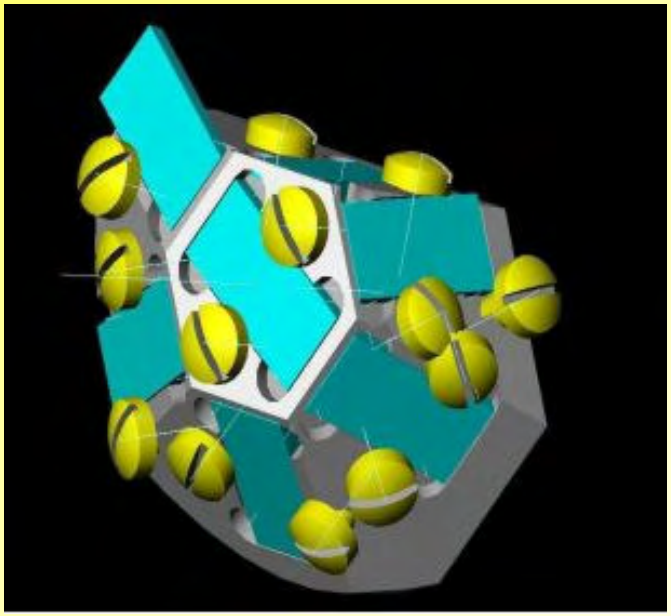
- ***What is the effect of target – substrate angle on the film properties?***

- ***How the film morphology varies changing target – substrate angle?***


DEPOSITIONS MADE AT DIFFERENT TARGET – SUBSTRATE ANGLE
FILM MORPHOLOGY
THICKNESS
AFM IMAGING
ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY
SUPERCONDUCTING PROPERTIES
T_C MEASURE
RRR
MAGNETO-OPTICAL
CRYSTAL STRUCTURE
X-RAY DIFFRACTION
TEXTURE ANALYSIS

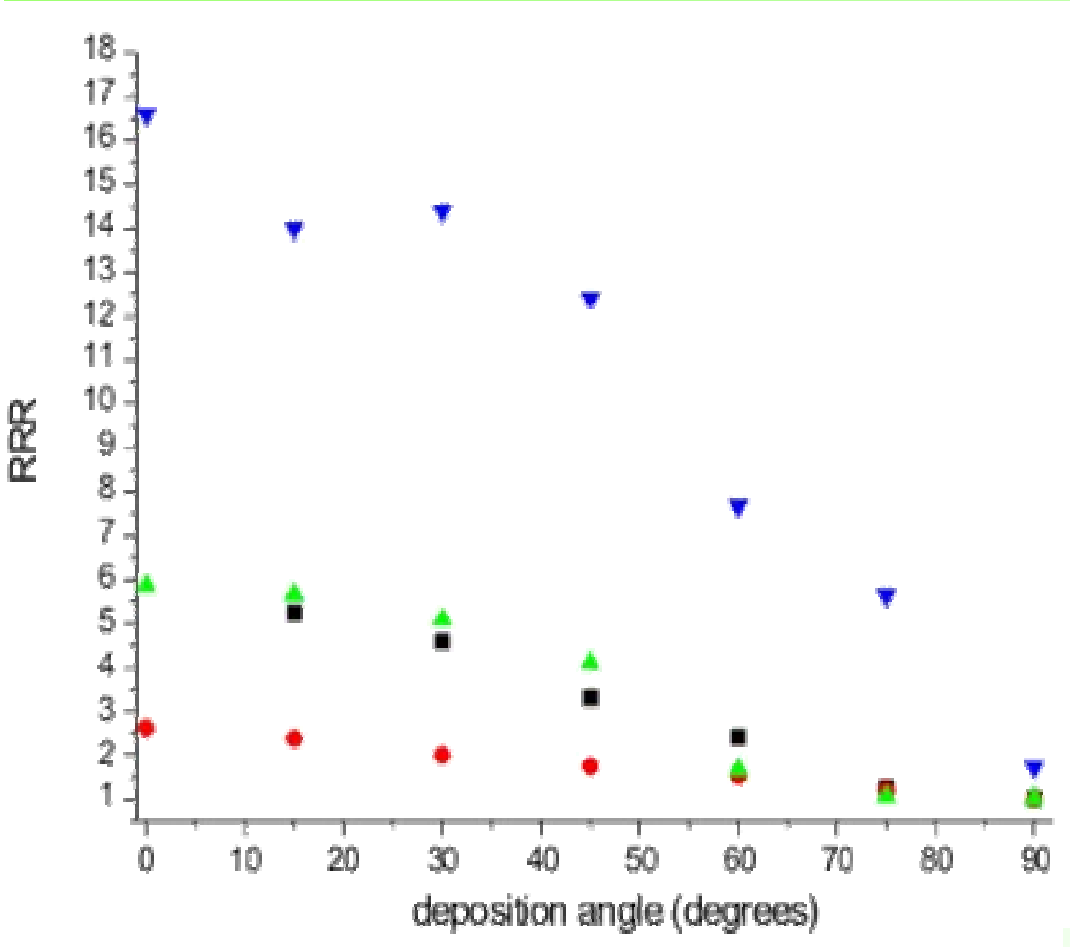
MULTI-ANGLE SAMPLE HOLDER





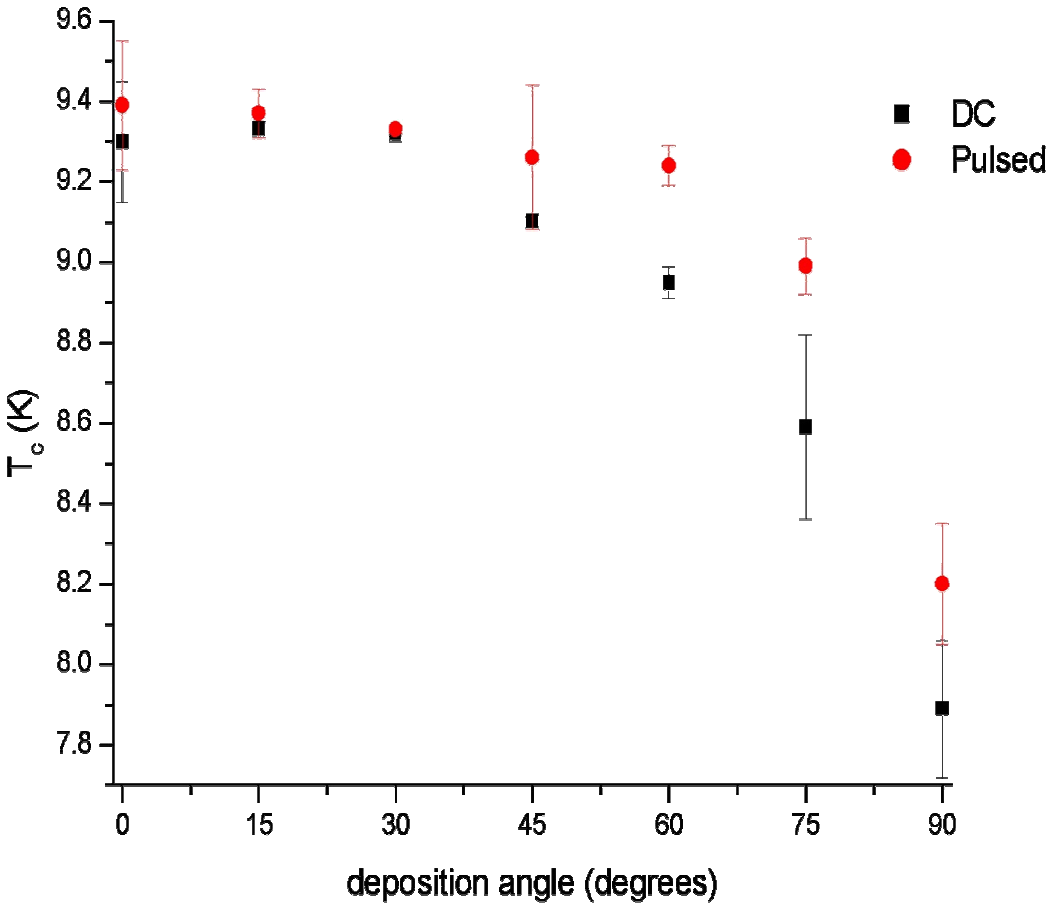
- **7 substrates coated in the same run**
- **Almost identical process conditions for each sample**

DC MAGNETRON SPUTTERING



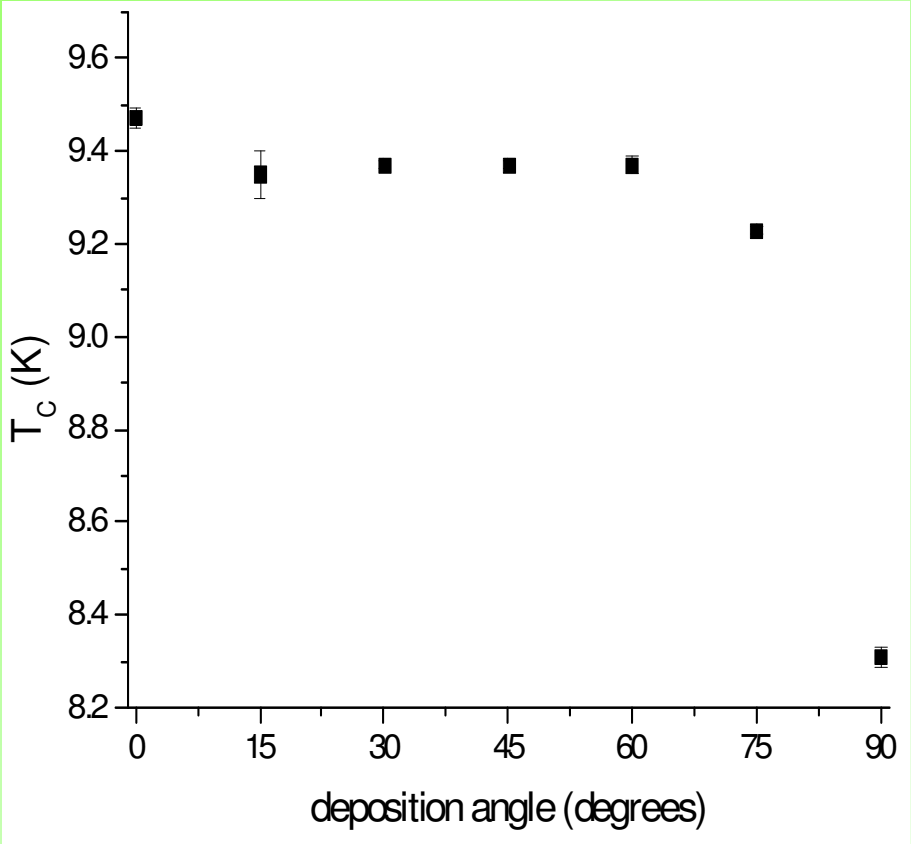
- **LOWER T_C AND *RRR* *INCREASING TARGET – SUBSTRATE ANGLE***

PULSED MAGNETRON SPUTTERING



- **BETTER PROPERTIES FOR PULSED CURRENT DEPOSITION**
- **THERE IS STILL THE SAME ANGLE DEPENDENCE OF T_C AND *RRR***

DC MAGNETRON SPUTTERING WITH SUBSTRATE HEATING

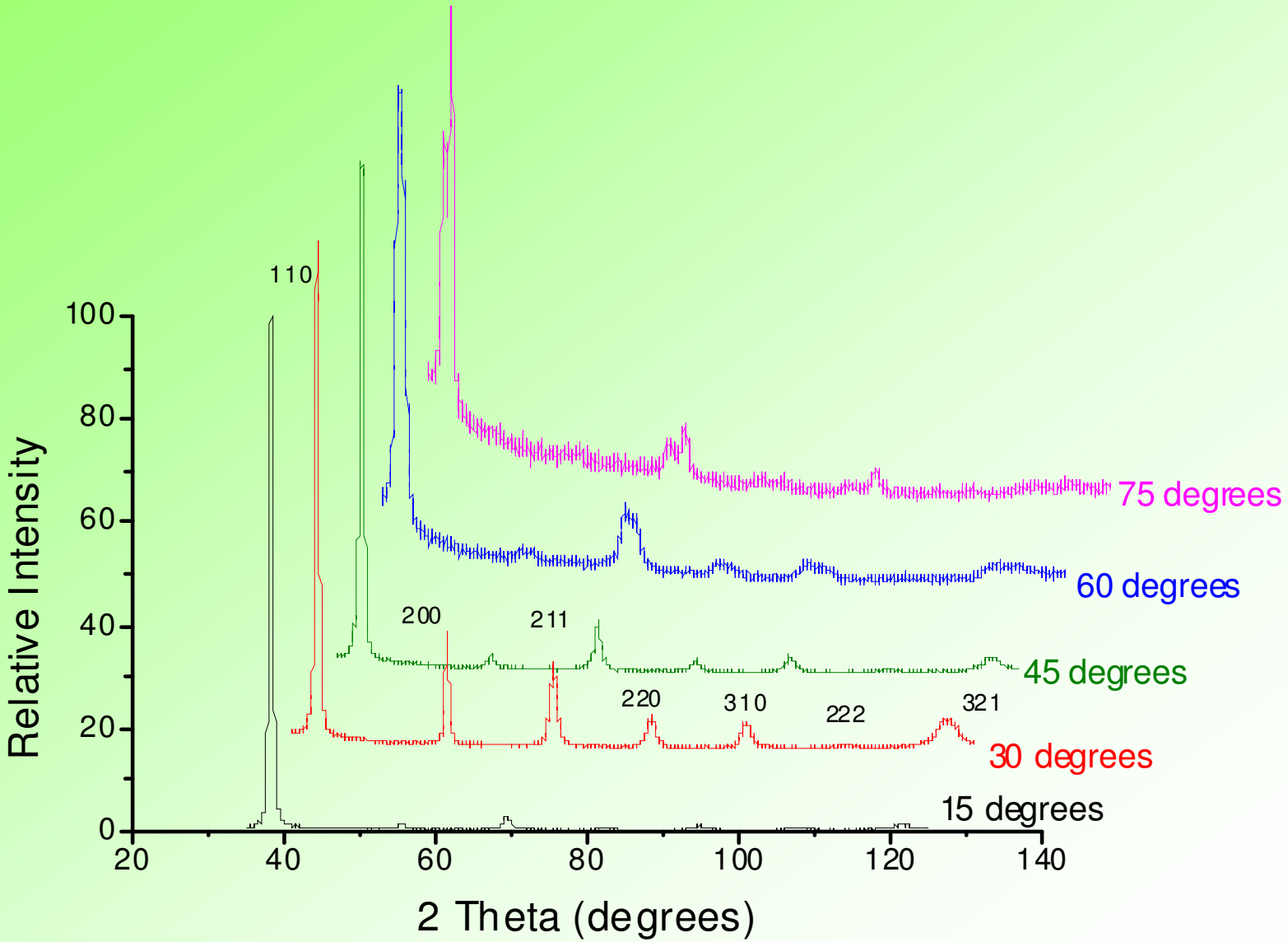


Substrate temperature kept at 600 ℃ during process

1. Better properties

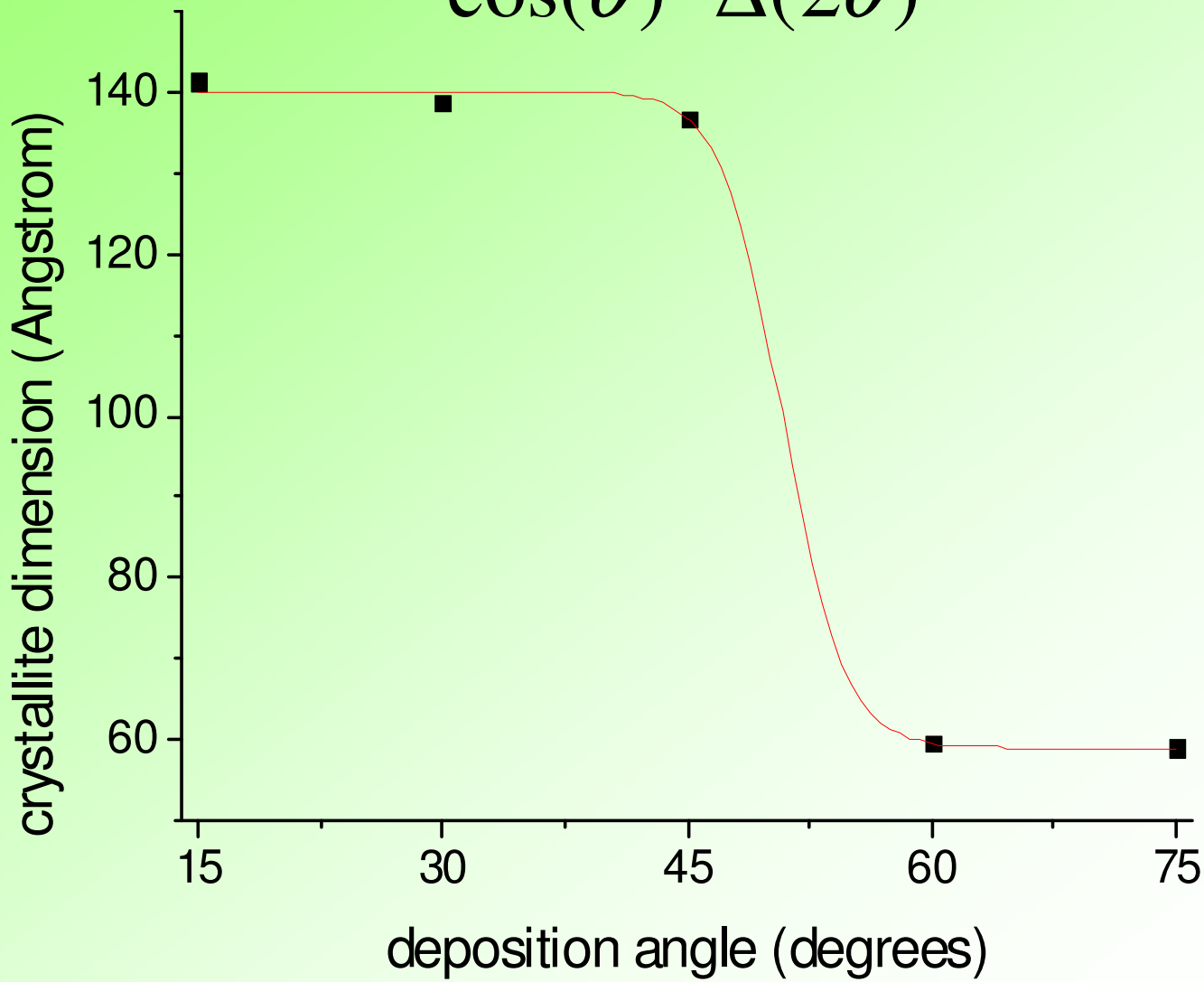
2. Less angle sensitivity

XRD SPECTRA OF FILM DEPOSITED AT DIFFERENT TARGET – SUBSTRATE ANGLE



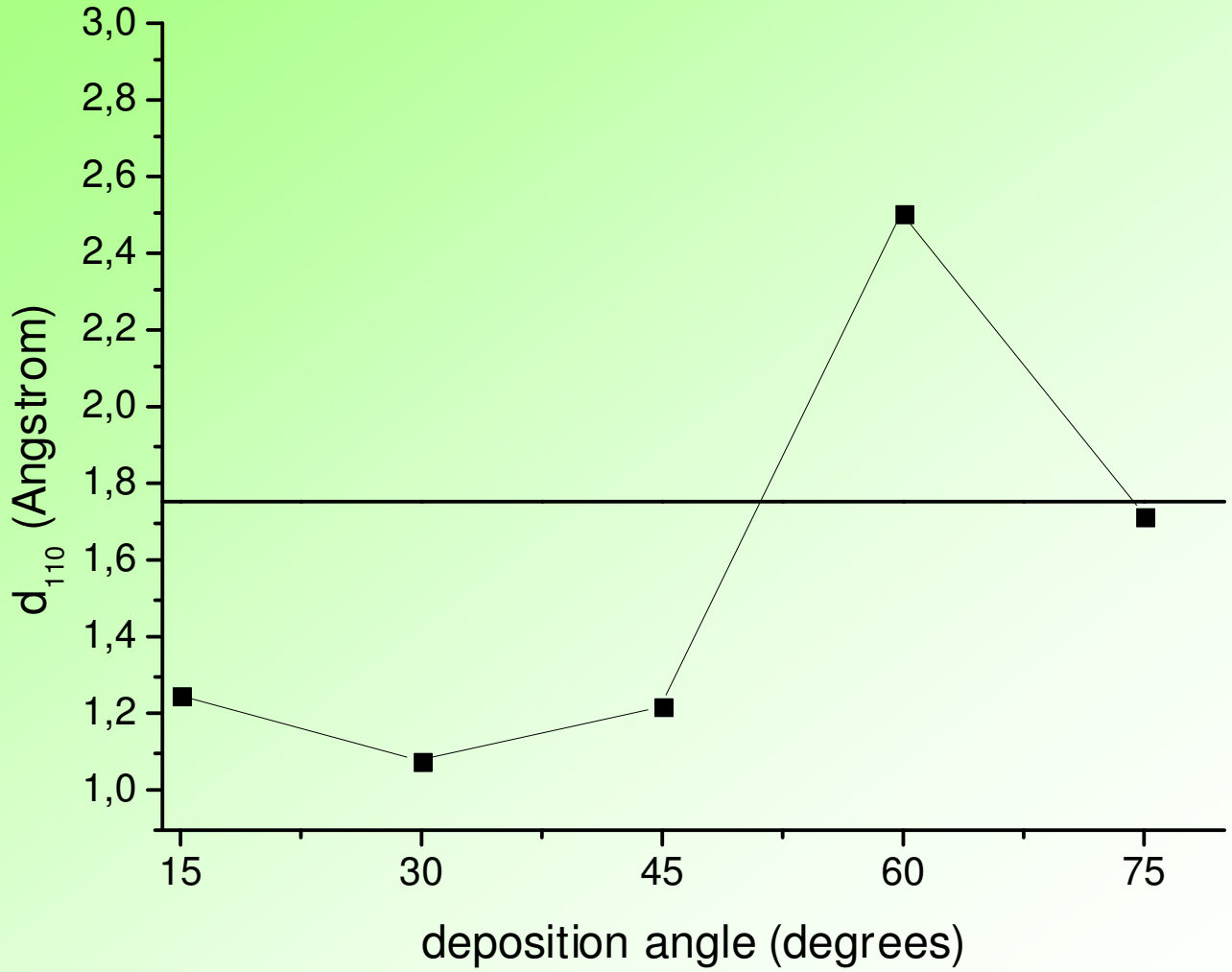
ESTIMATED GRAIN DIMENSION

$$D = \frac{0.9\lambda}{\cos(\theta) \cdot \Delta(2\theta)}$$

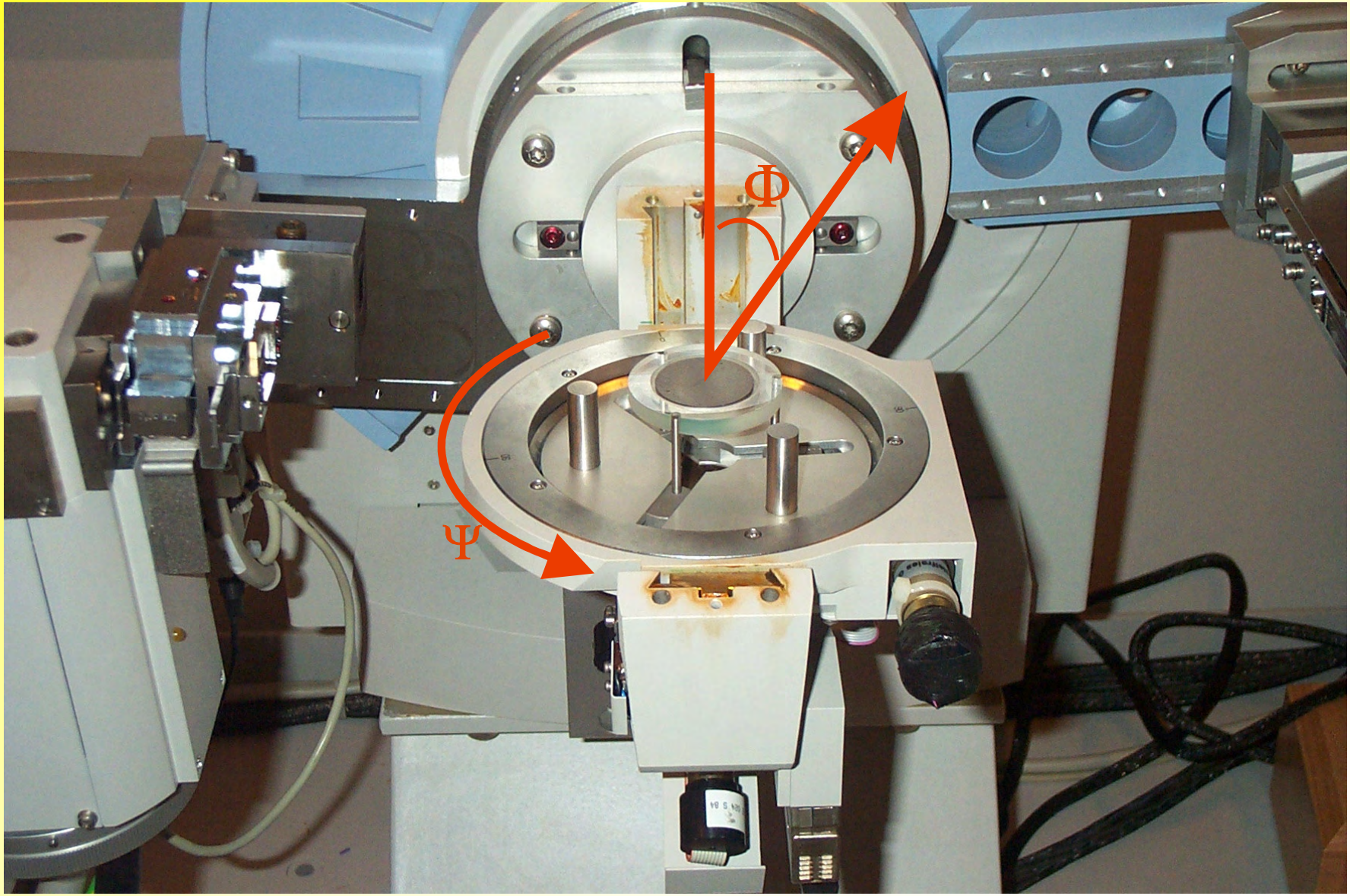


d_{110} OF CRYSTAL PLANES

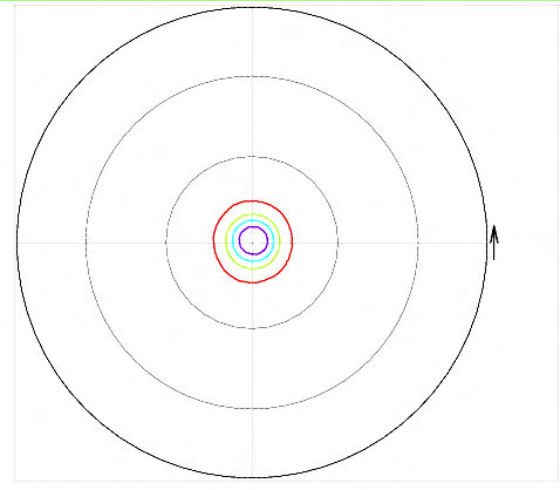
$$2d_{(hkl)}\sin\theta = n\lambda$$



TEXTURE ANALYSIS



DC MAGNETRON SPUTTERING



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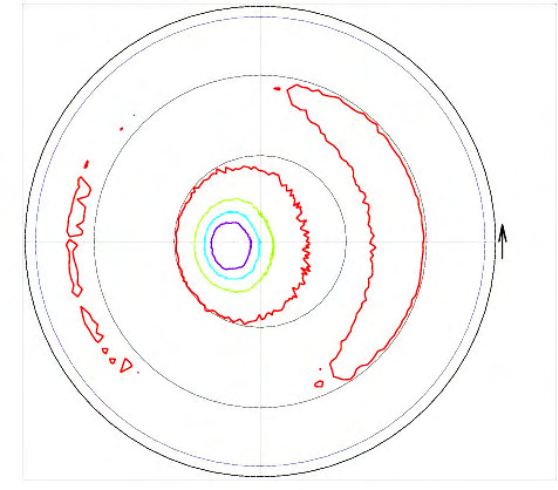

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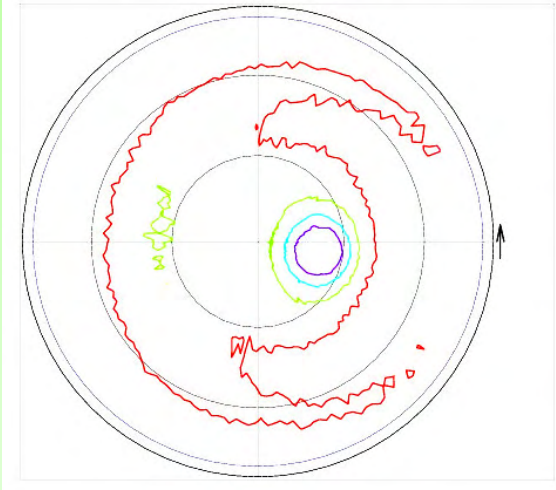

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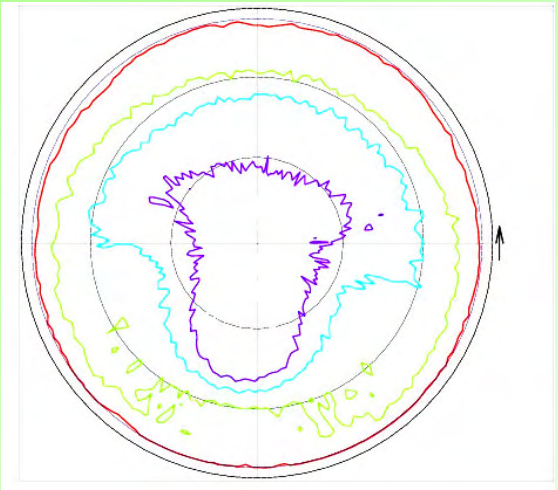

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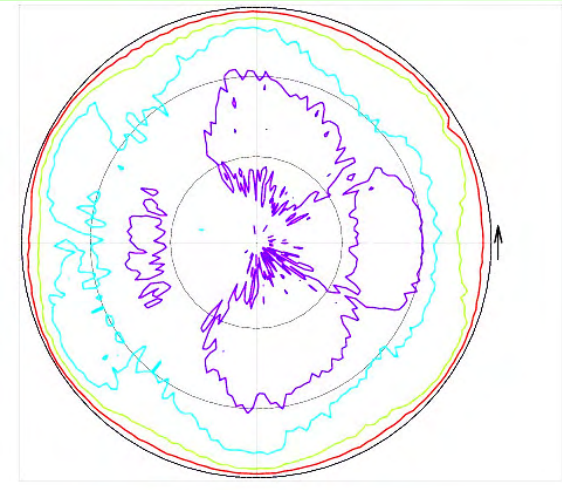

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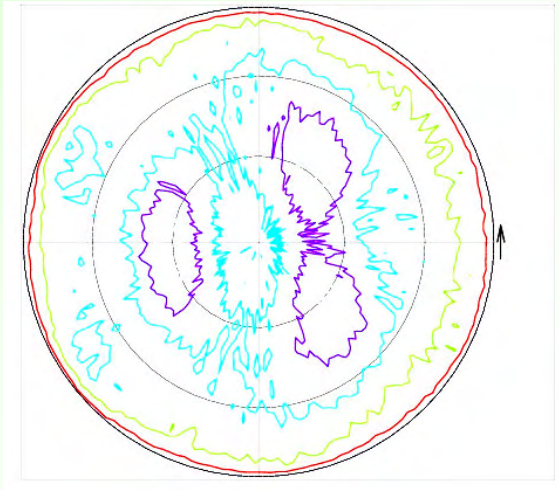

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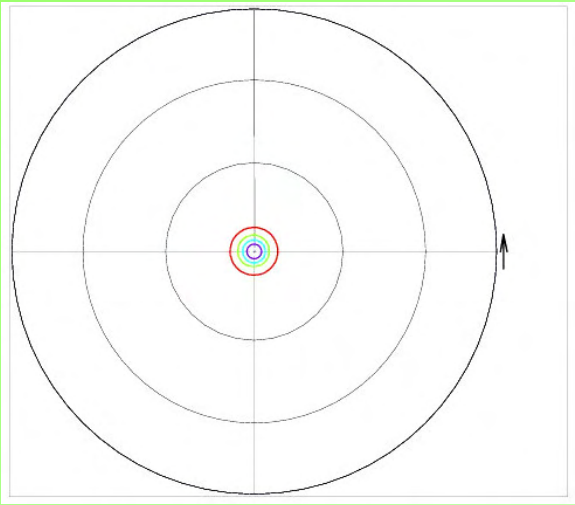
PULSED MAGNETRON SPUTTERING



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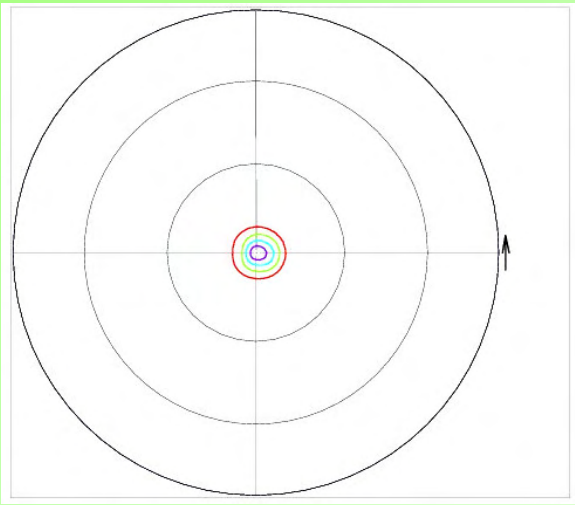

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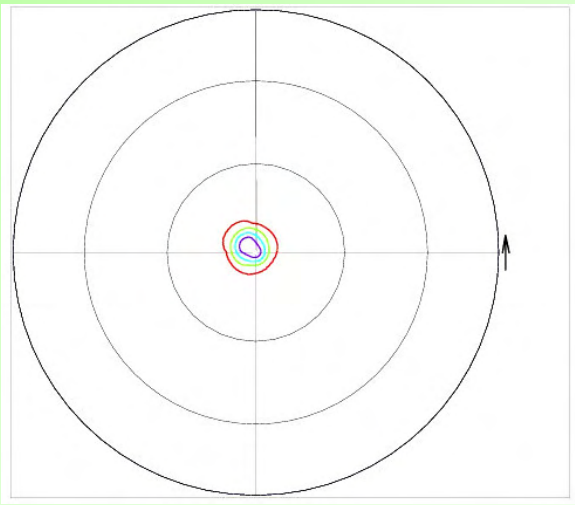

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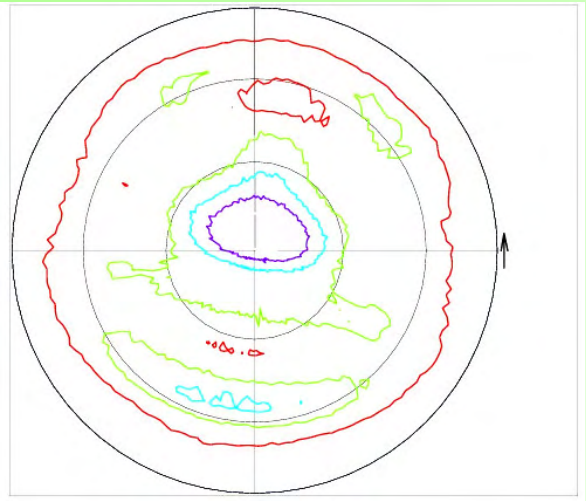

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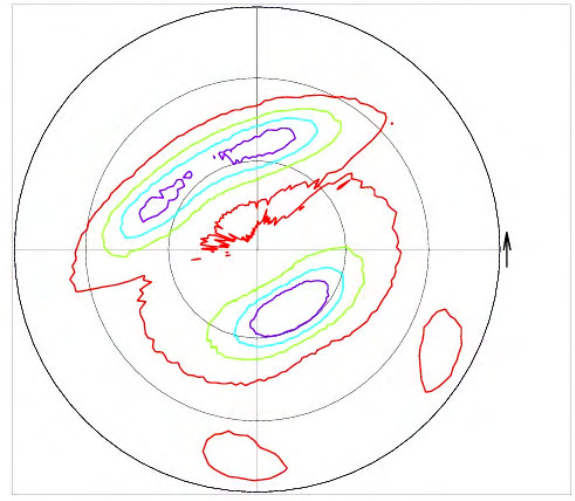

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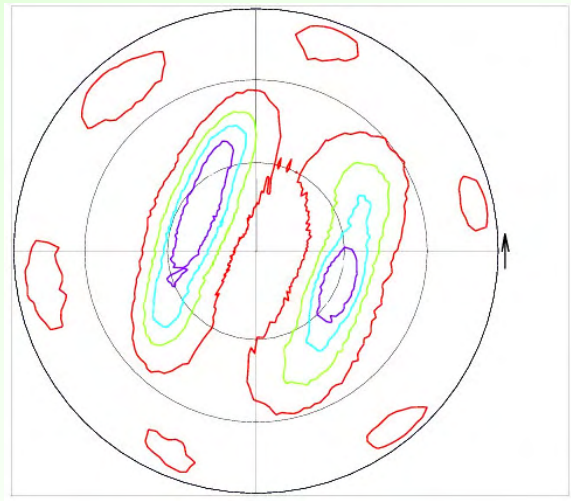

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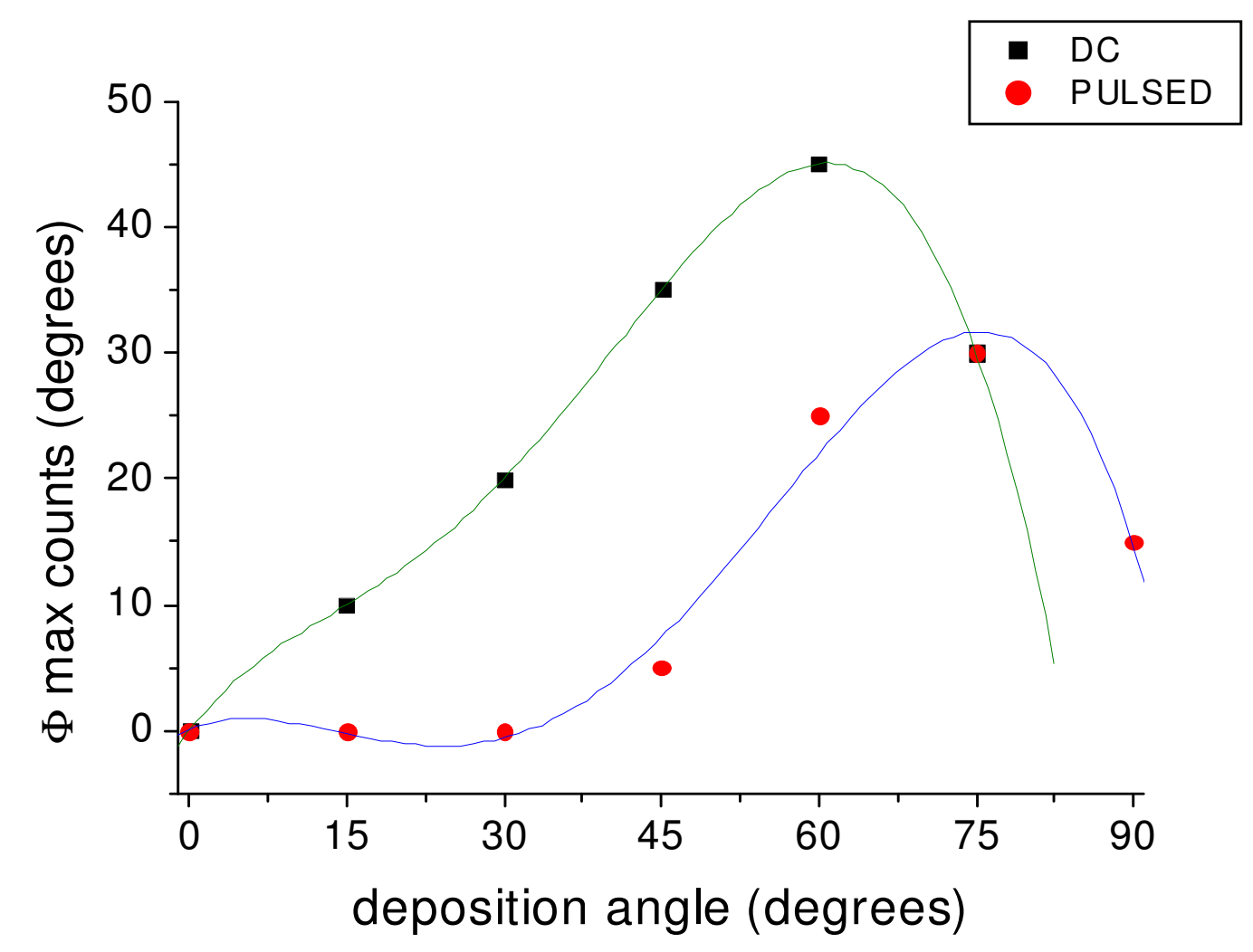
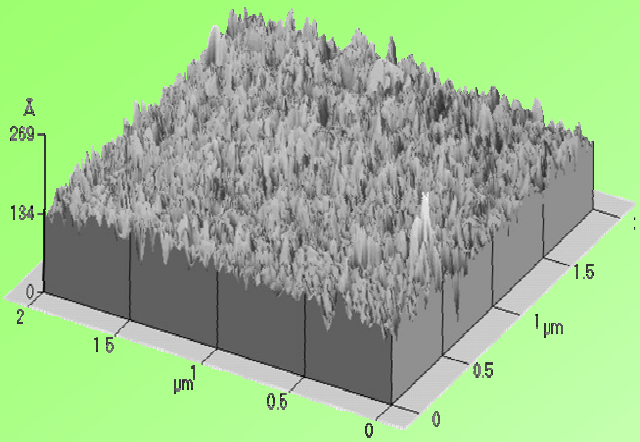
TEXTURE ANALYSIS

DC VS PULSED MAGNETRON SPUTTERING



ATOMIC FORCE MICROSCOPY
TOPOGRAPHIC IMAGES



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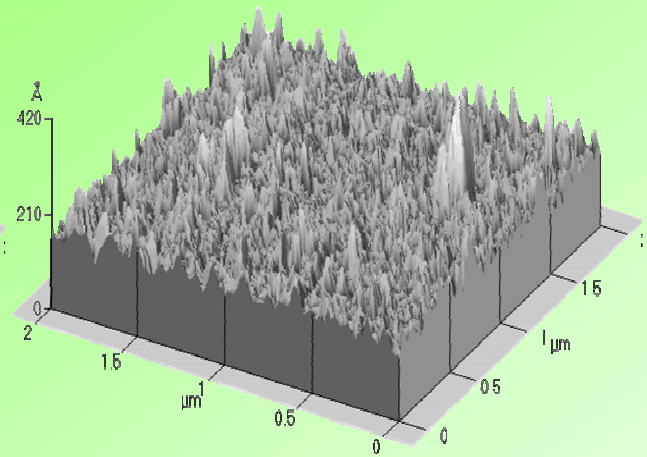

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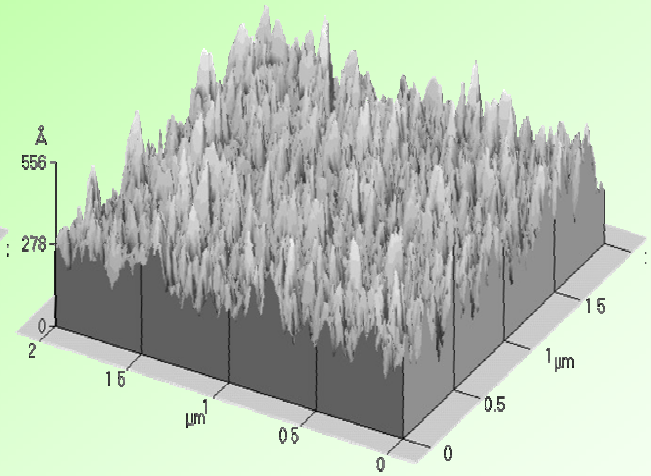

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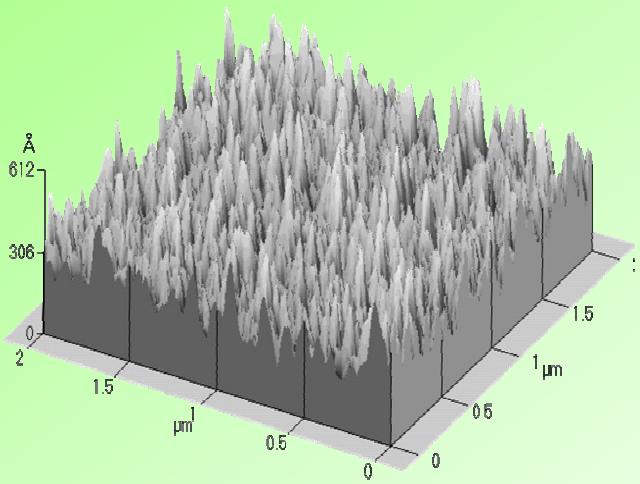

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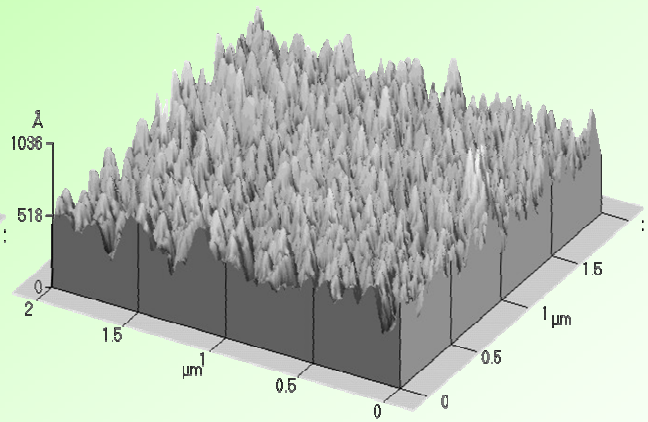

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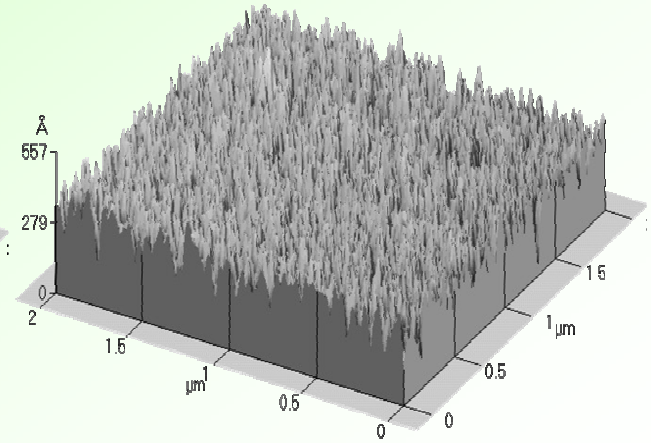
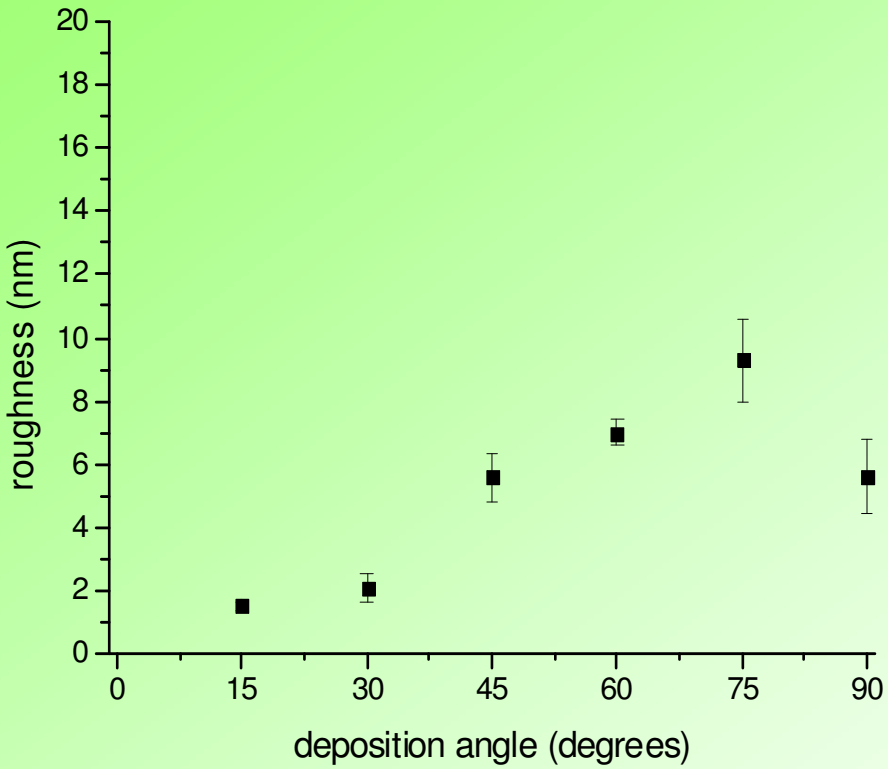
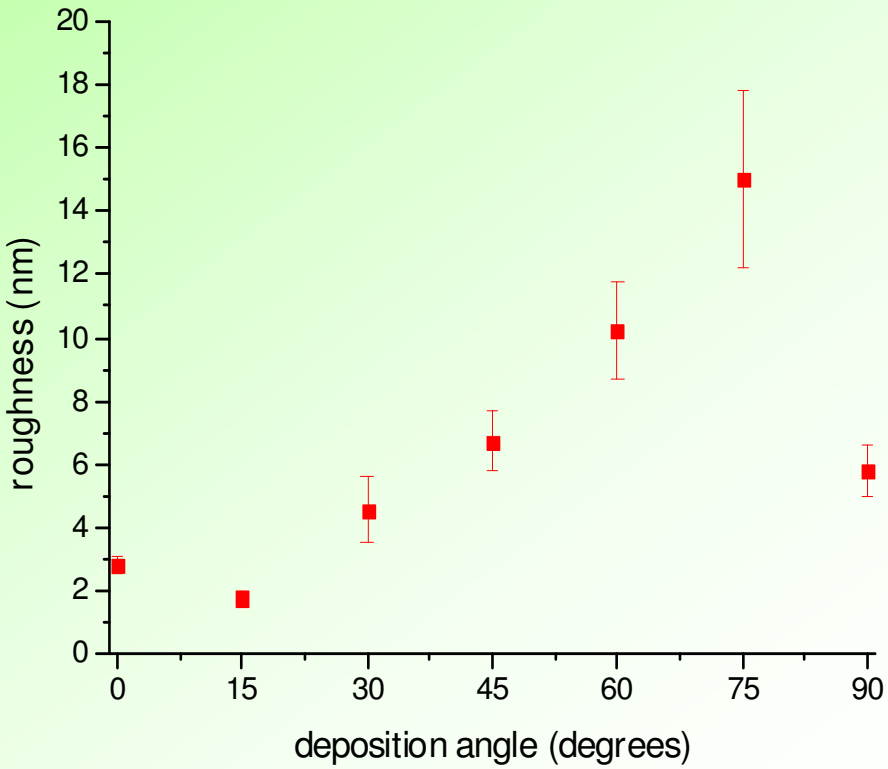


90 gradi

ATOMIC FORCE MICROSCOPY
DC MAGNETRON SPUTTERING

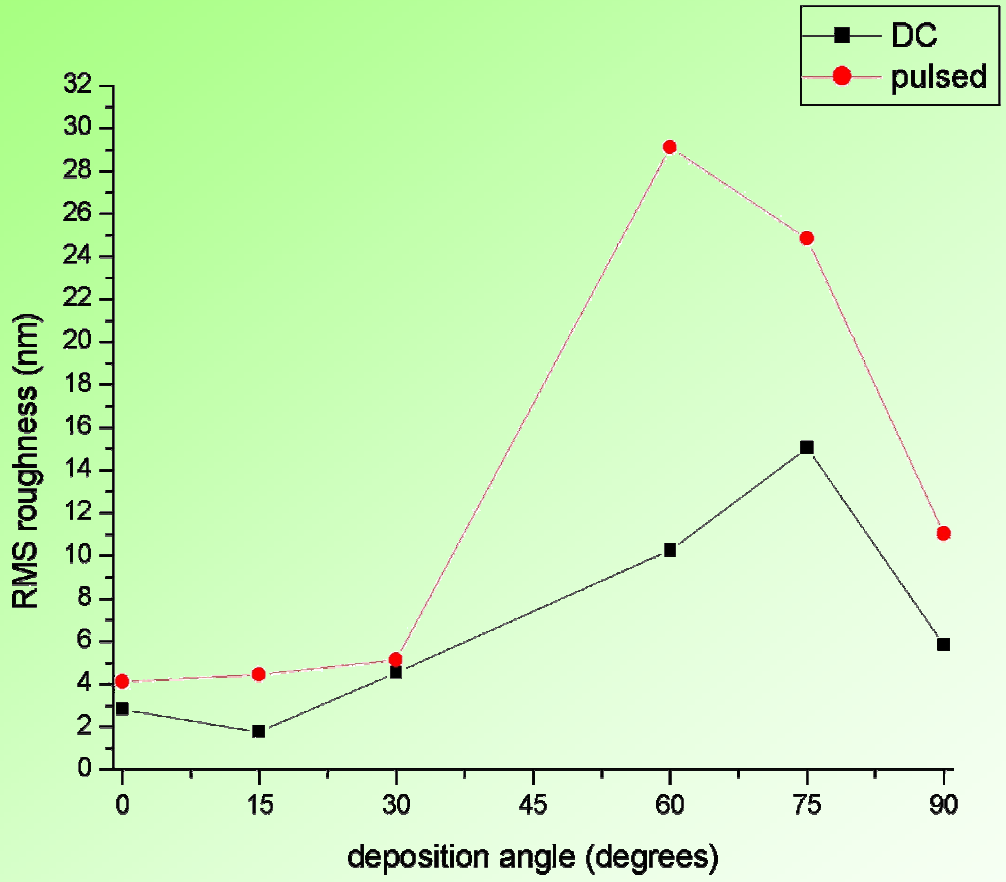


Variable thickness



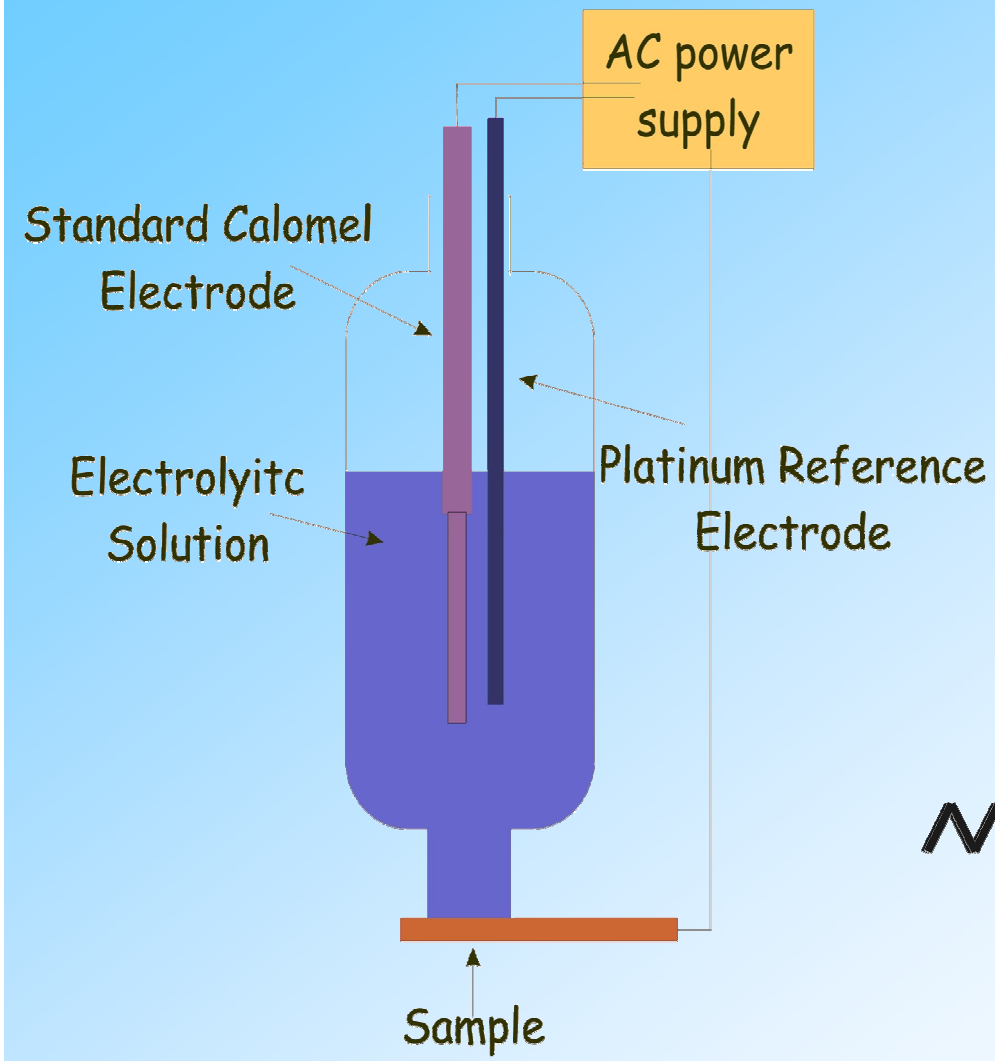
Uniform thickness

ATOMIC FORCE MICROSCOPY
PULSED MAGNETRON SPUTTERING



I campioni depositati in corrente pulsata sono sistematicamente più rugosi

ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

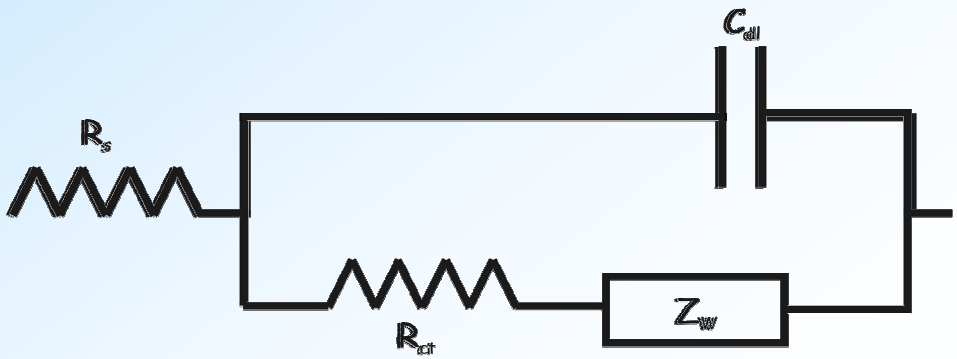


$$V(t) = V_0 \cos(\omega t)$$

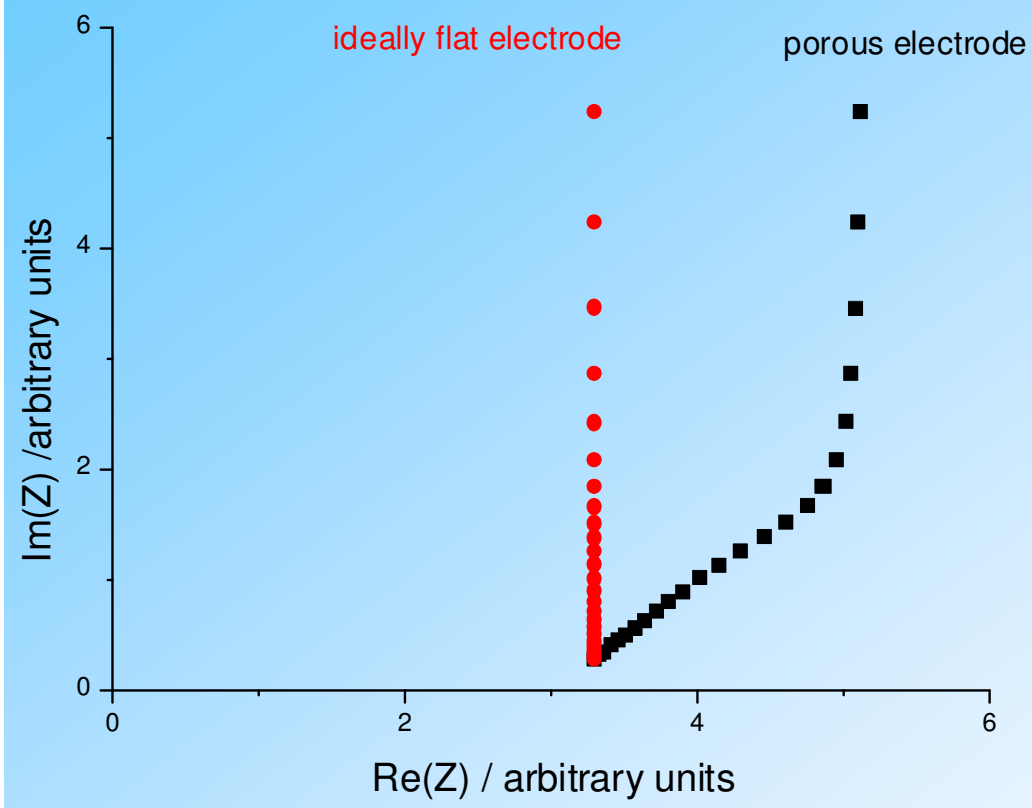
$$I(t) = I_0 \cos(\omega t - \varphi)$$

$$Z = \frac{V(t)}{I(t)} = Z_0 \frac{\cos(\omega t)}{\cos(\omega t - \varphi)}$$

$$Z = Z_0 e^{j\varphi}$$



ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY NYQUIST PLOT



FLAT ELECTRODE:

Real part of impedance represents R_s at any frequency

Immaginary part does not vary with frequency

NYQUIST PLOT IS A VERTICAL LINE

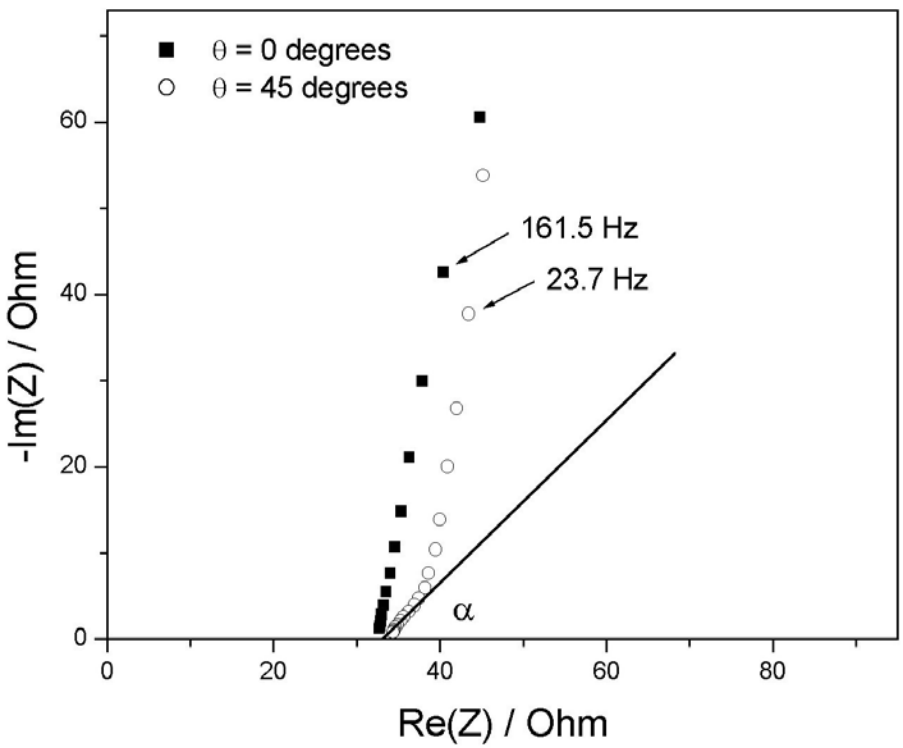
POROUS/ROUGH ELETRODE:

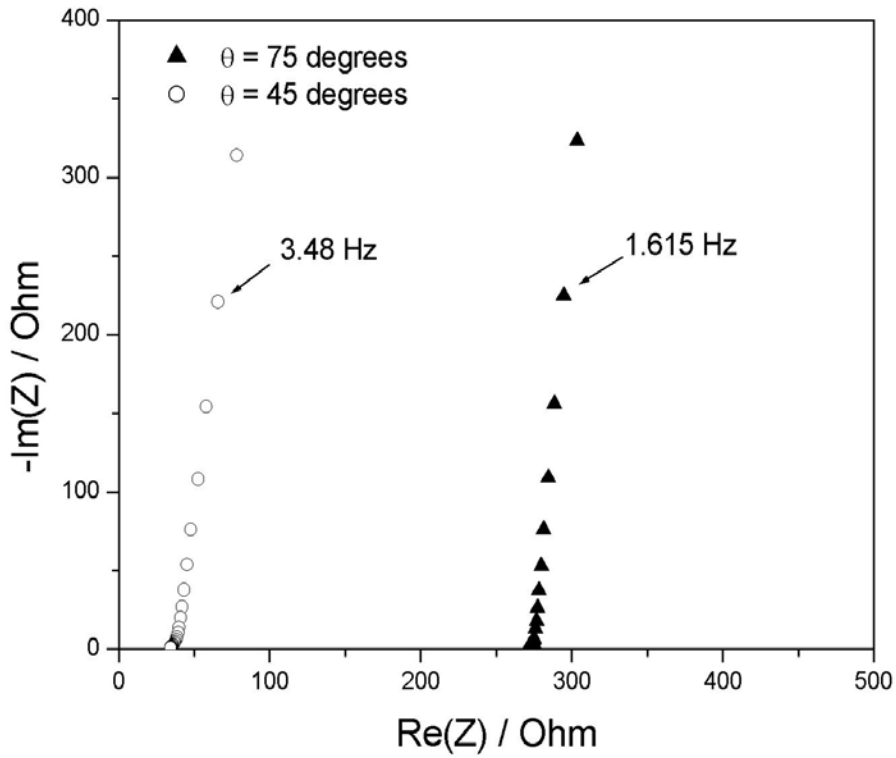
The apparent capacity C_{dl} depends on frequency because the penetration length of electric field inside the pores raises when decreasing frequency.

NYQUIST PLOT IS A 45° INCLINED LINE

ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY DC MAGNETRON SPUTTERING

VARIABLE FILM THICKNESS



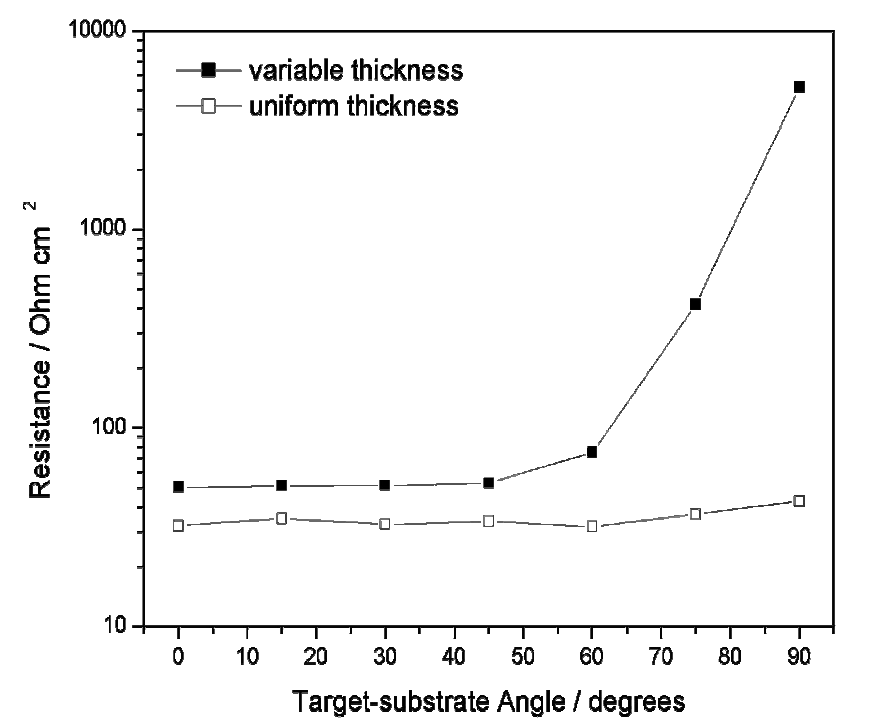


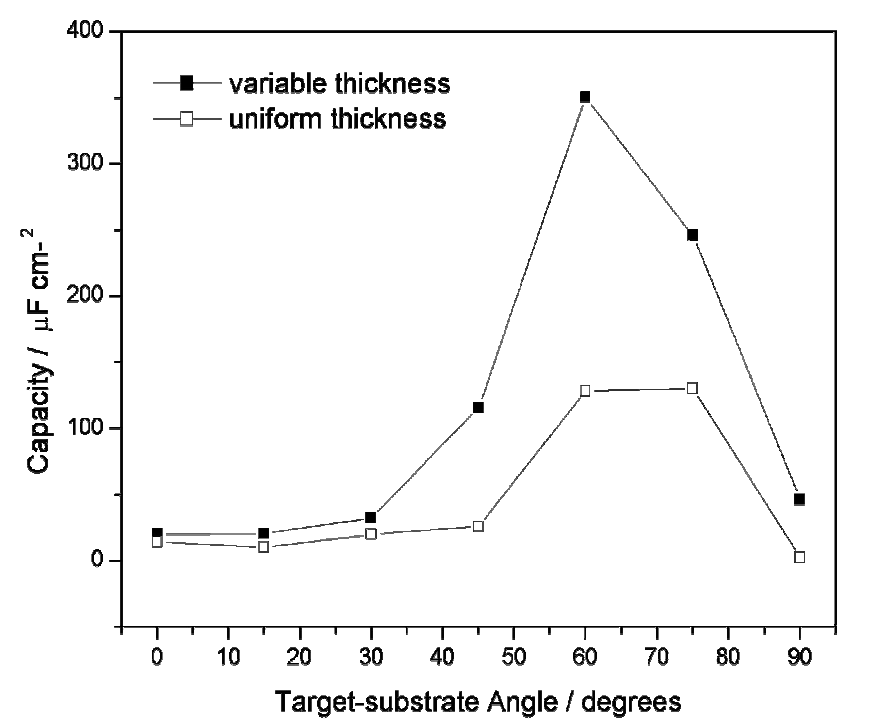
Up to 45° there is porous electrode behaviour

Film deposited at higher angles are less thick so resistence is greaer

ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY DC MAGNETRON SPUTTERING

COMPARISON BETWEEN FILM WITH DIFFERENT THICKNESS AND FILM WITH CONSTANT THICKNESS



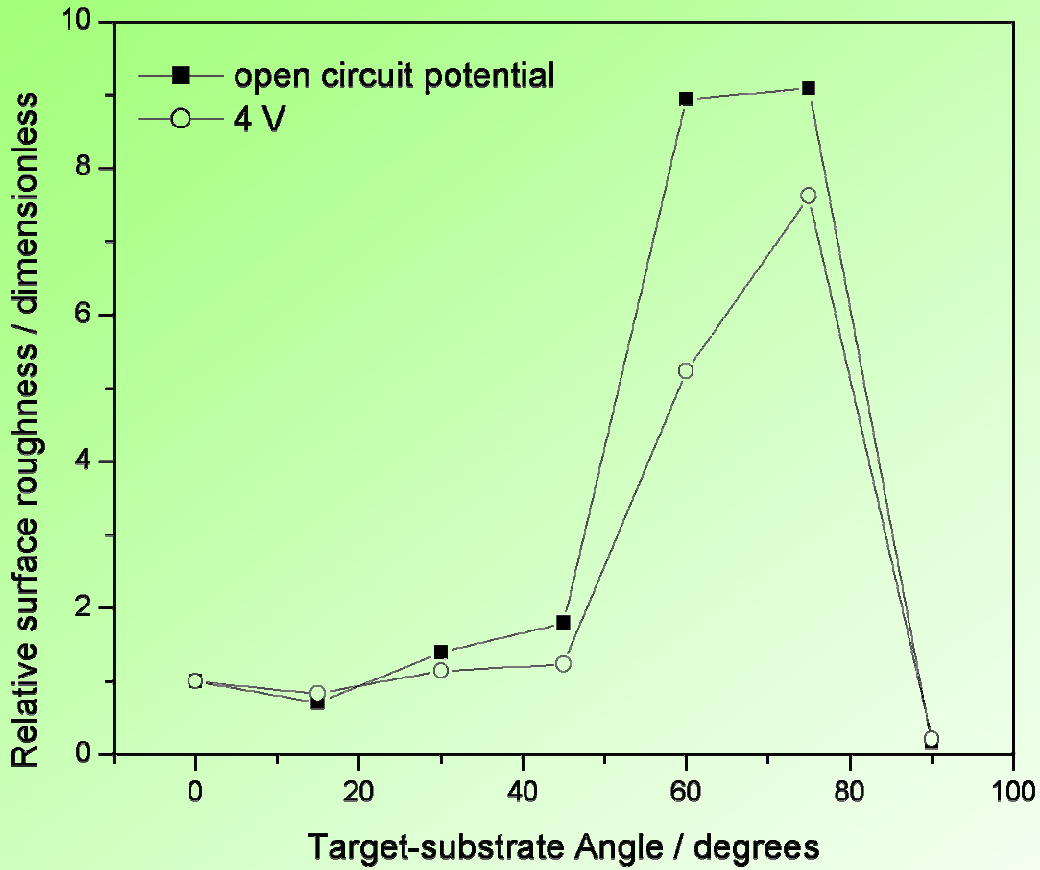


Films with the same thickness have the same resistance

There is a maximum in capacity at 60° target – substrate angle

ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY DC MAGNETRON SPUTTERING

Films with the same thickness – measures in passivaton condition



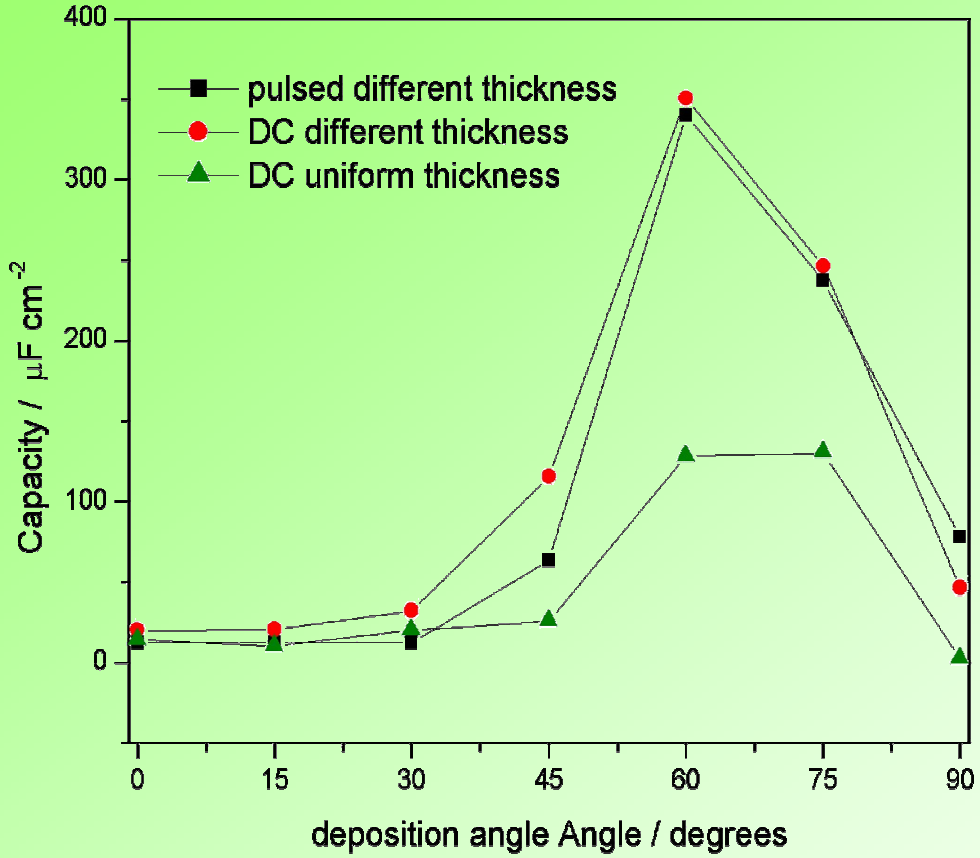
Imposed potential to create a passivation layer on the film

Oxide tichkness depends only on applied potential

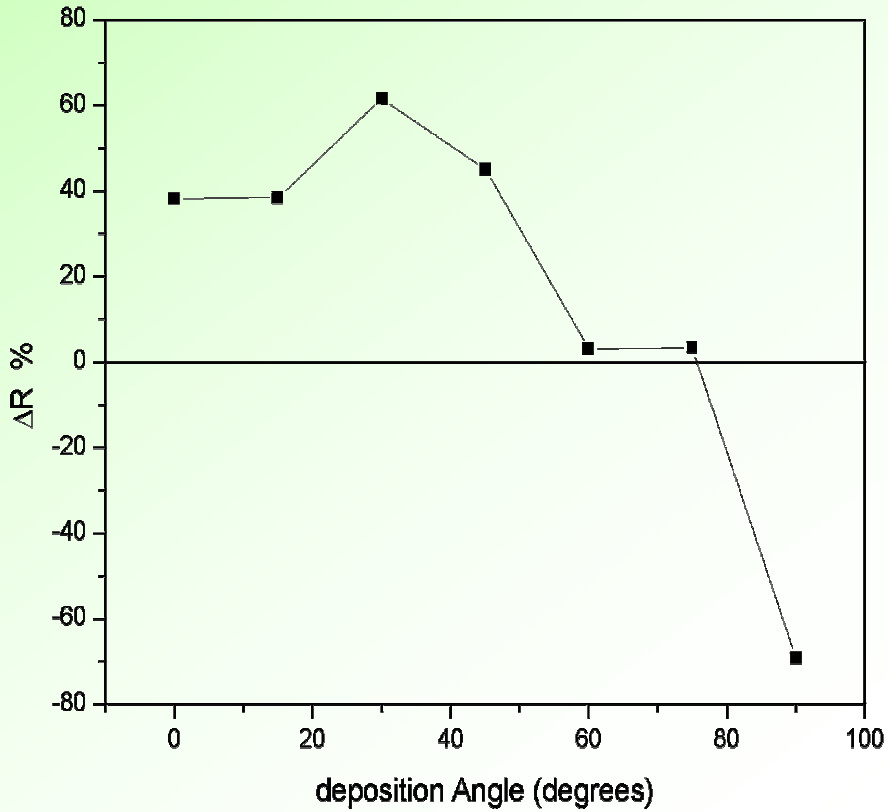
Oxide growth follows niobium film morphology

Capacity shows the same angle dependence of non oxidized films

ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY PULSED MAGNETRON SPUTTERING

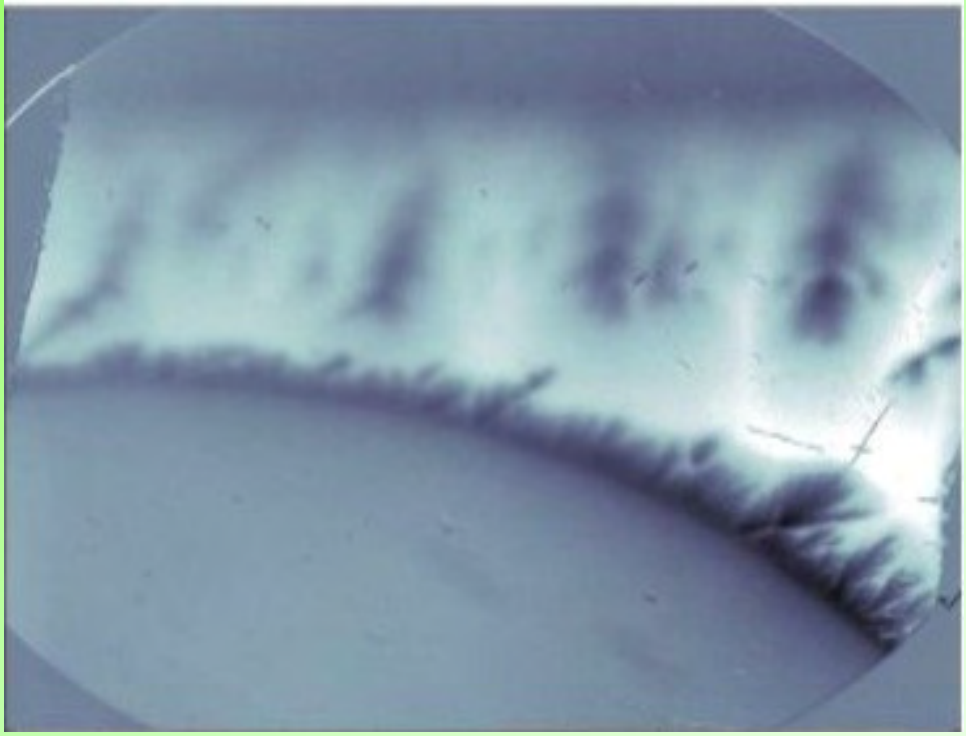


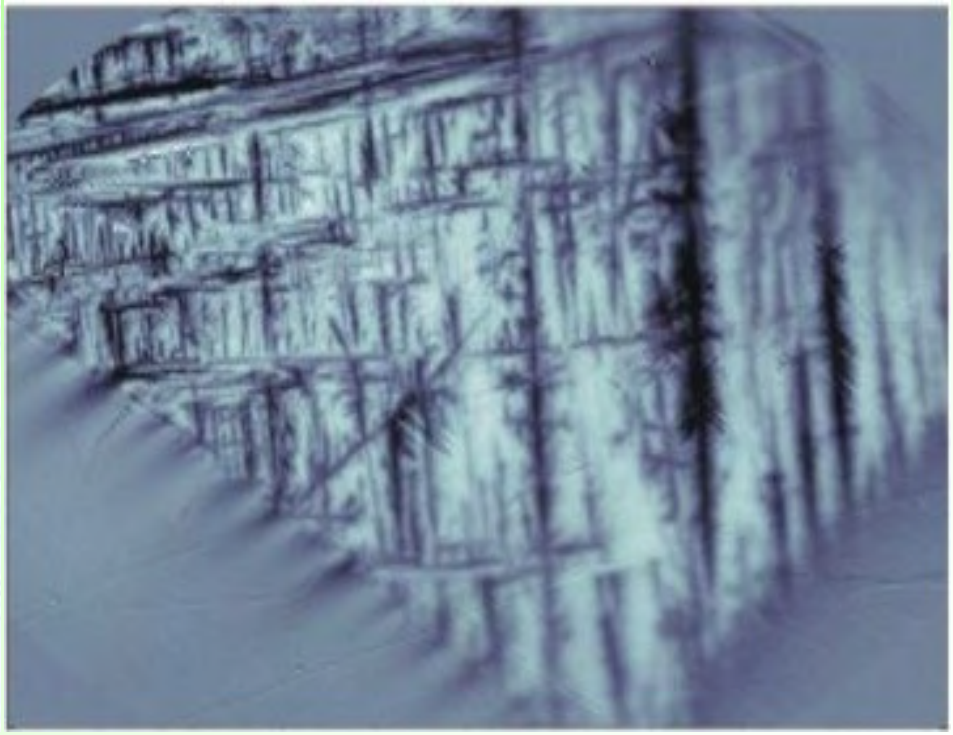
$$\Delta R = \frac{C_{ac}^{cont} - C_{ac}^{pulsed}}{C_{ac}^{pulsed}} \times 100$$



MAGNETO-OPTICAL ANALYSIS

OFHC copper substrate (not electropolished)





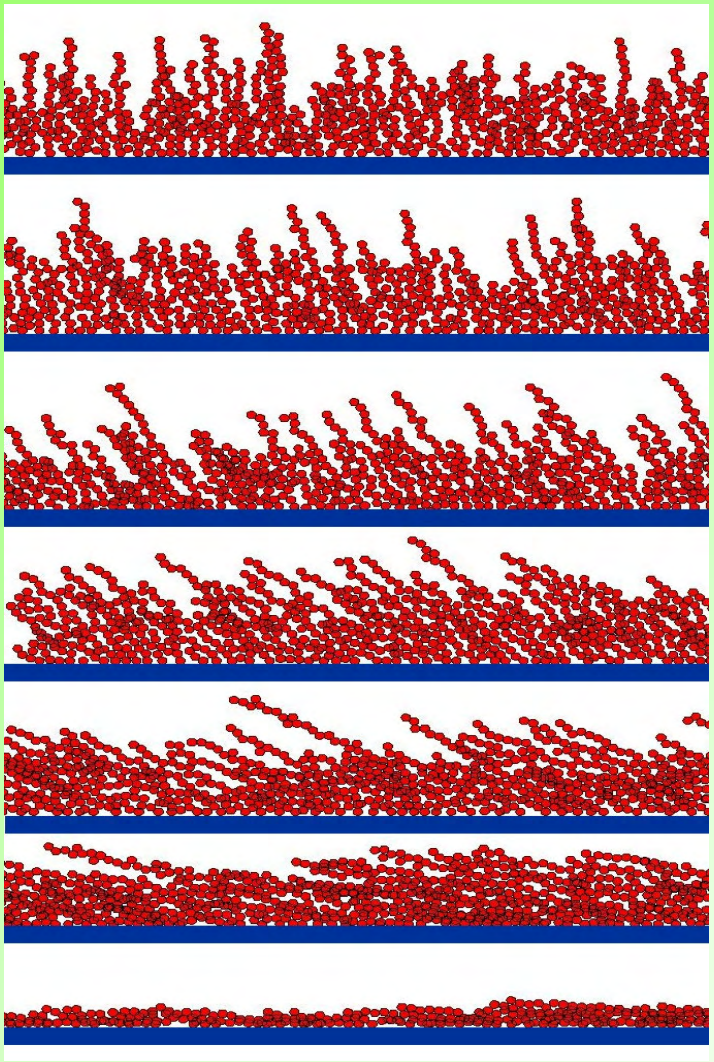
Target – substrate angle: 0°

Good connectivity respect to vortex penetration

Target – substrate angle: 45°

Vortex penetrate along substrate discontinuity

SIMULATION OF THIN FILM GROWTH



0°

15°

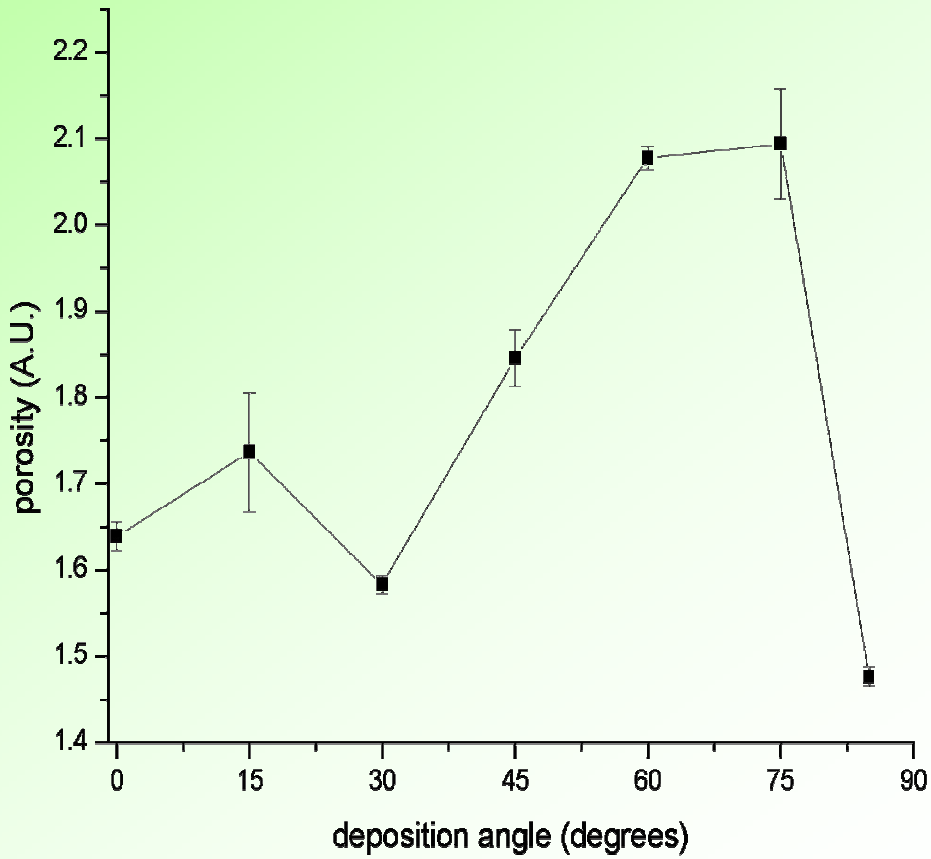
30°

45°

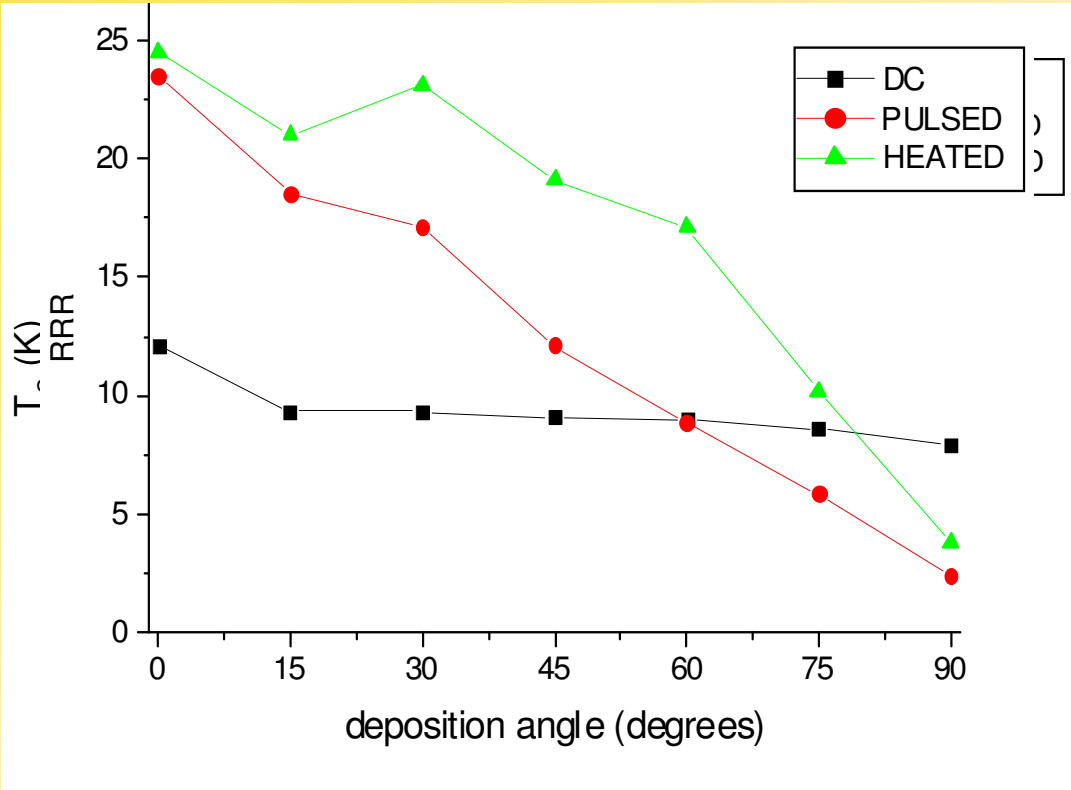
60°

75°

85°



CONCLUSIONS
s.c. and transport properties



Properties depend on target – substrate angle

Pulsed magnetron sputtering and heating of the substrate reduce the angle dependence

CONCLUSIONS
Crystal structure

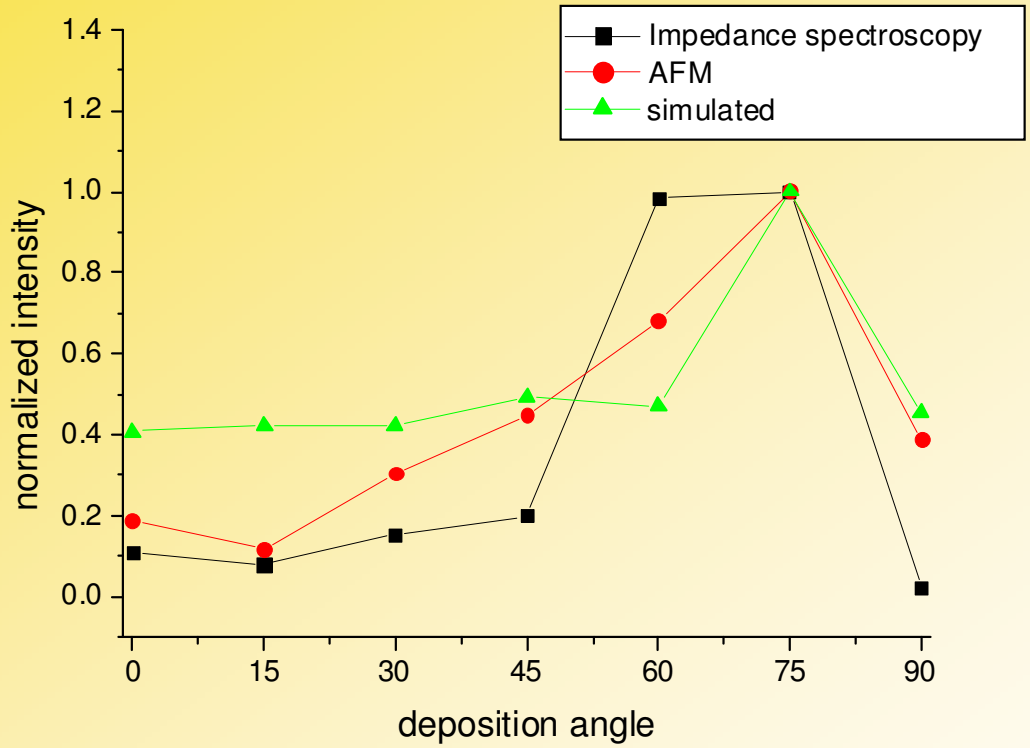
Films deposited at higher angles tend towards amorphization

Lattice parameter has a maximum at 60° target – subtrate angle

(110) crystal planes of the growing film orient along niobium atoms arrival direction

This effect is reduced using pulsed magnetron deposition

CONCLUSIONS
morphology



There is a maxmum in roughness between 60° and 75° target – substrate angle

This is not a thickness effect but is due to deposition dynamics

Fine.