Joint Research Centre (JRC)

Preparation and characterization of thin film nuclear targets by vacuum evaporation

D. Sapundjiev, G.Sibbens, A.Moens, K.Luyckx, M.Peeters, R.Eykens, Y.Aregbe

IRMM - Institute for Reference Materials and Measurements

Geel - Belgium

http://irmm.jrc.ec.europa.eu/

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Outline

- Targets preparation at IRMM
- Physical Vapour Deposition lab (PVD)
- Preparation of thin film nuclear targets by PVD
- Thin film monitoring and characterization
  - *Acoustic impedance thickness monitor*
  - *Post deposition characterization*
- Film thickness results comparison
- Other measurement techniques
- Ongoing and future research
- Conclusions

Target preparation at IRMM

Target preparation lab – established as a support of the neutron physics unit for production and characterization of targets for neutron physical experiments.

Fabrication methods:

- mechanical shaping - larger targets and backings (cutting, rolling, punching, pressing, etc)
- electrodeposition
- physical vapour deposition (PVD)

After long standstill - fully operational as of May 2009

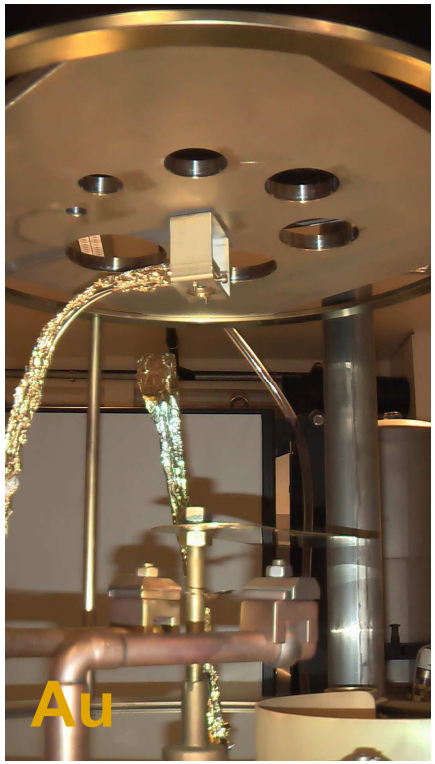
The following thin films can be fabricated by PVD:

- LiF, ^{6}LiF films
- ^{10}B deposits
- gold layers
- actinide targets $^{235}UF_4$ (controlled zone)

LiF and Au vapour deposition set-up

Construction and features:

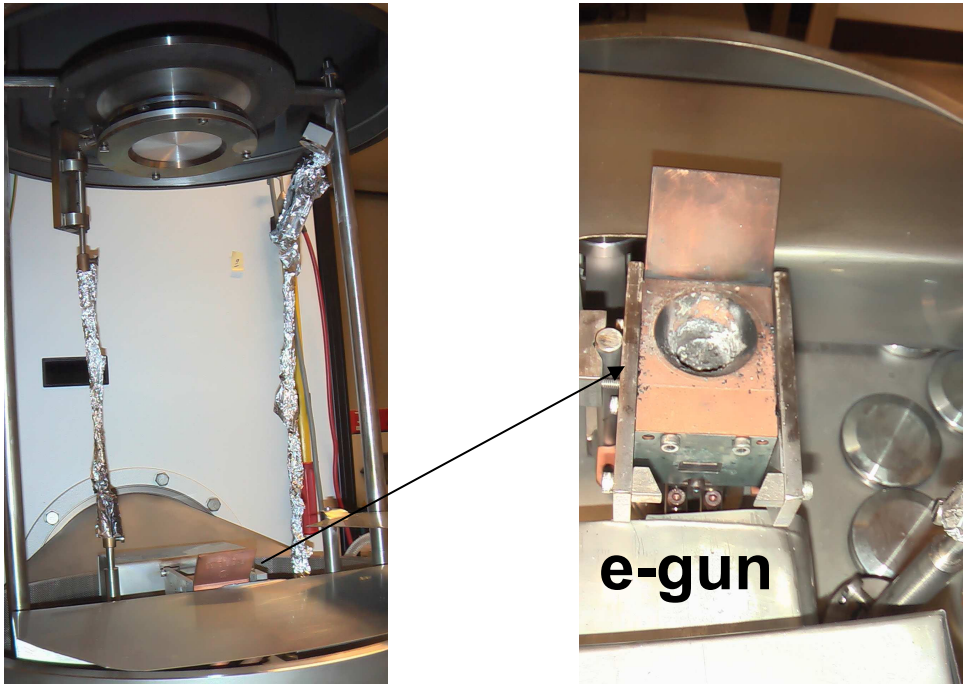
- resistance heating (Ta or other refractory metal) crucible
- substrate holder and carousel for substrate rotation
- typical evaporation rates: 0.3 – 3 Å /sec



^{10}B vapour deposition set-up

Construction and features:

- bent e-beam heating - for evaporation of highly reactive elements/materials
- target rotation, specially designed target holders/masks to reduce edge effects



Substrate and backings:

- in-house fabricated polyimide (PI) foils; other substrates (mechanical shaping)
- supplied by the ordering party
- or commercially available materials

Deposition procedure and parameters:

- substrate installation
- evacuation - base pressure p ~ $5x10^{-6}$ Torr
- evaporation - deposition rate 0.3 - 3 Å/sec
- cooling down and venting with inert gas (Ar)

Two main tasks:

1. to ***monitor*** the film growth during deposition
2. to determine the properties of the produced film - composition, area and uniformity

Requirements to the film characterization methods:

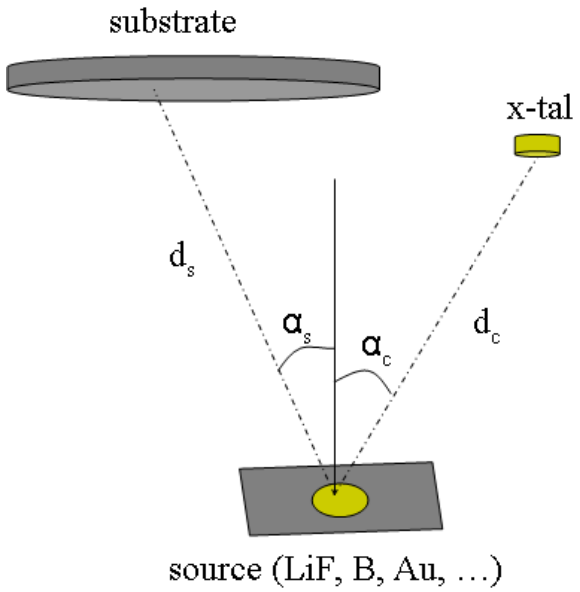
- non destructive
- preserve target composition and stability
- produce results with sufficient accuracy
- additional specifications (from the requesting party)

Acoustic impedance thickness monitor

To control the deposition process - measures – frequency and derives deposition rate.

- simple and sensitive
- indirect measurement!

Not suitable for film thickness characterization!



Input parameters:

- acoustic impedance
- density
- *tooling factor*

Post deposition characterization

Universal method is not available;

Depending on the physical properties of the films:

- spectrophotometry - for optically transparent materials
- α - particles energy loss (thickness smaller than the range)

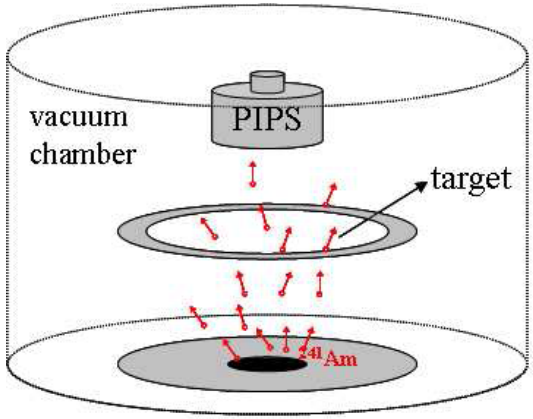
Alternative methods (under testing):

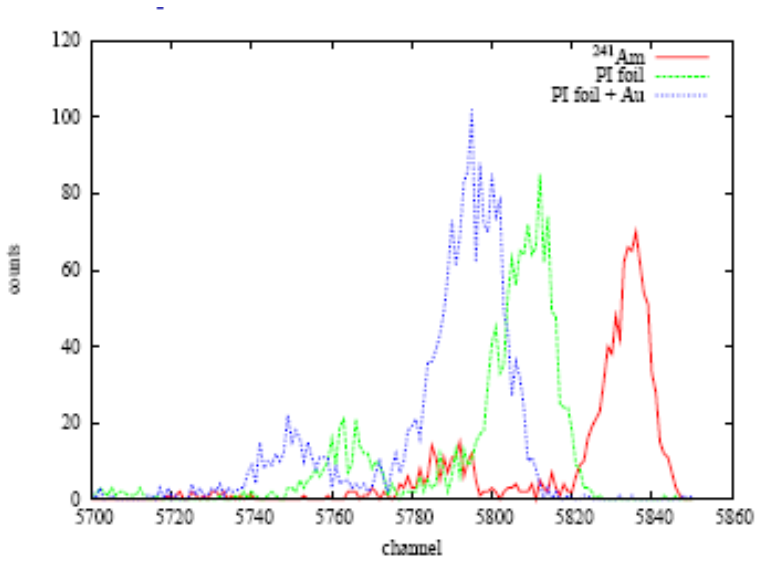
- **A**tomic **F**orce **M**icroscopy (AFM)
- **ellipsometry** - measures the modulation of polarized light on reflection/refraction: high accuracy; does not require calibration; suitable for optical films;
- **m-line spectrometry** - optically transparent films
- **profile meters** - similar to AFM, lower accuracy

Measure the energy loss on passing through substrate, and thin layers

- direct measurement!
- information about uniformity – by scanning the sample
- only for ``thin'' films and substrates
- requires stable measurement system and careful spectrum analysis

Experimental set-up and spectra:





Film thickness results comparison

Film thickness measurements by various methods available in the lab:

sample	thickness monitor	photospectrometry	α-spectroscopy
LiF on Al	50 ± 2%	11.1 ± 2% (or 66 ± 2%)	n.a.
PI foil	n.a.	32.1 ± 2%	31.3 ± 1%
Au on PI foil	50 ± 2%	n.a.	54.8 ± 1%

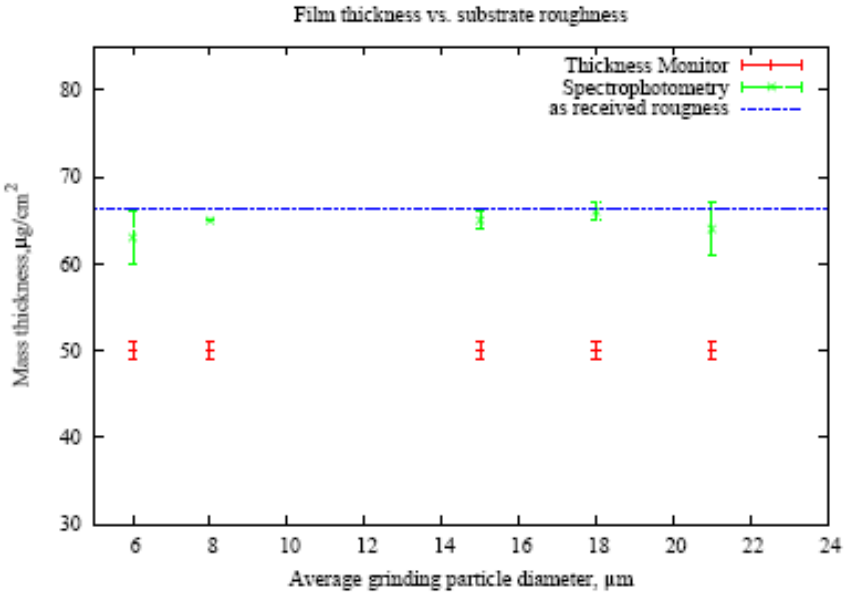
Discrepancy - additional independent measurement and/or (re-)calibration of the available equipments?

Influence of substrate roughness

Objective: investigate the effect(s) of the surface conditions on the thickness measurement

Experimental procedure:

- *Al* round samples of thickness 0.25 mm and diameter 22 mm
- polished to roughness: *6, 8.4, 15.3, 18.3, 21.8* µm
- 50.1 µg/cm² LiF deposit



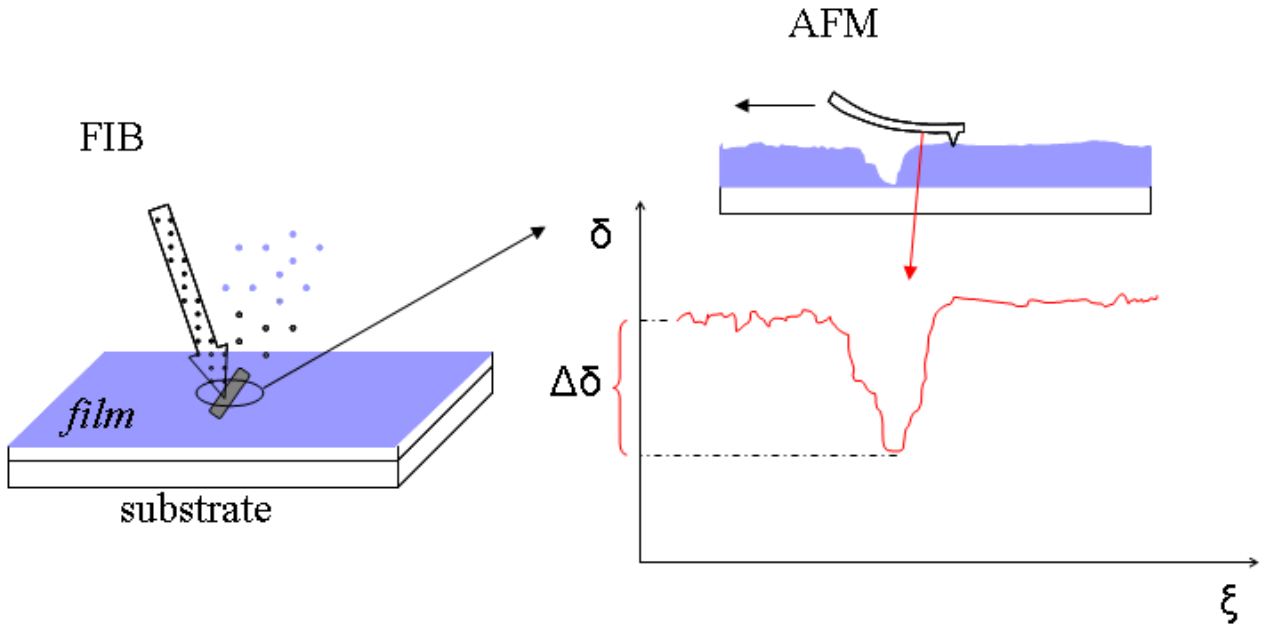
Conclusion:
no observable effect on the thickness value

Film thickness measurement by AFM 1

To calibrate the available equipment (thickness monitors and spectrophotometry)

Procedure:

- using focused ion beam (FIB) to sputter narrow (microns) channel into the thin film;
- scan across the channel and determine the depth (thickness) of the layer



Advantages:

- high precision ~Å
- for optical and non optical films, dielectric and conductors
- suitable for calibration!

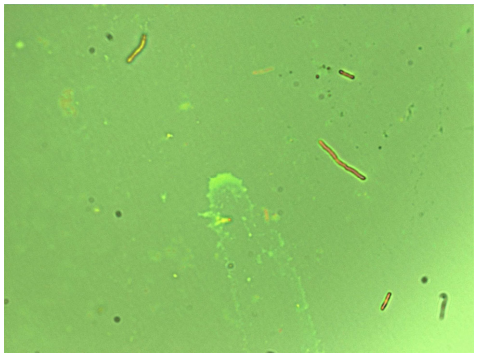
Disadvantage:

- destructive, results in implanted species of the FIB ions;
- probes only a small area of the sample (few μm^2)
- difficult to determine when the deposit is removed (leave the substrate intact)

Thin film measurements:

- α - particles spectroscopy thickness measurement set-up
- evaluate the applicability of ellipsometry and *m-line* thickness measurement (in collaboration with universities)
- deposition of pure lithium metallic targets

Substrate fabrication:
polyimide foils preparation and characterization to improve homogeneity and thermal expansion mismatch with frame



PI foil light micrograph

Preparation of Li metallic targets

Difficulties

- reactive with oxygen and nitrogen
- highly flammable
- post deposition treatment, storage, and transportation: crucial!!!

Experimental details:

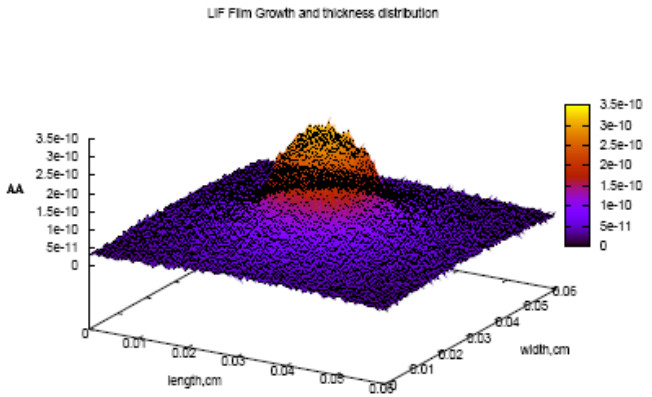
- deposition set-up - LiF evaporator, or:
- separate deposition set-up placed in a glovebox
- post-deposition reduction?
- stability measurements XRD to determine the time evolution of Li_2O

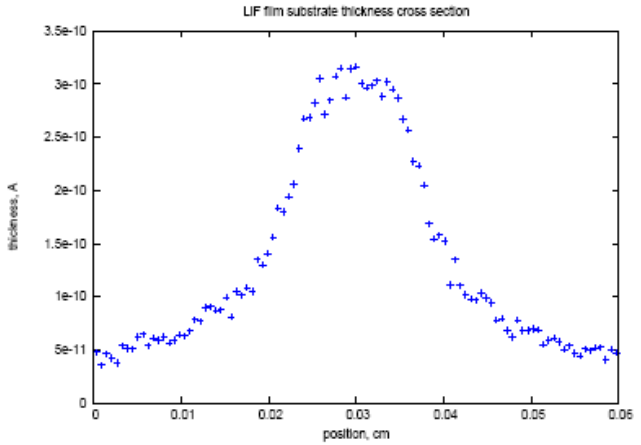
Objective: to calculate the dimensional characteristics of the deposited films in dependence of the experimental conditions

Input data and parameters

Model based on Hertz-Knudsen evaporation rate equation:

- physical properties of the material-density, molecular state, vapour pressure, mean free path;
- experimental - geometrical position, temperature, pressure, source and substrate dimension, others;





Conclusions

The target preparation group at IRMM has resumed the production of thin nuclear targets by PVD:

- The evaporation procedure has been optimized in terms of improving the accuracy of the film growth monitoring
- The available methods for post- deposition thickness measurements and calculation have been revised and compared
- Discrepancies in the obtained result point the necessity of recalibration
- Alternative techniques for thin film thickness measurement are necessary and currently being studied;
- Design and construction of additional thin film measurement devices (α - particle spectroscopy, m-line spectroscopy, others)
- Purchase of new equipment (spectrophotometer, etc.)