INFN-LNL-254(2019)

2018

Method for preparation Yttrium cyclotron targets for Zirconium-89 production

Hanna Skliarova, Sara Cisternino

INFN-LNL

2/28/2018

Contents

1 PROTOCOL FOR PREPARATION OF NATURAL YTTRIUM THICK FILMS DEPOSITED ONTO NIOBIUM BACKING BY MAGNETRON SPUTTERING.

1.1 Substrates (cyclotron target backing) preparation.

Niobium discs Ø24mm×0.5mm (Figure 1) are cut starting from 15cm x 15cm x 0.5mm Goodfellow® 99.9% Nb sheet (Figure 2) by particular mechanical cutting technique (Figure 3).

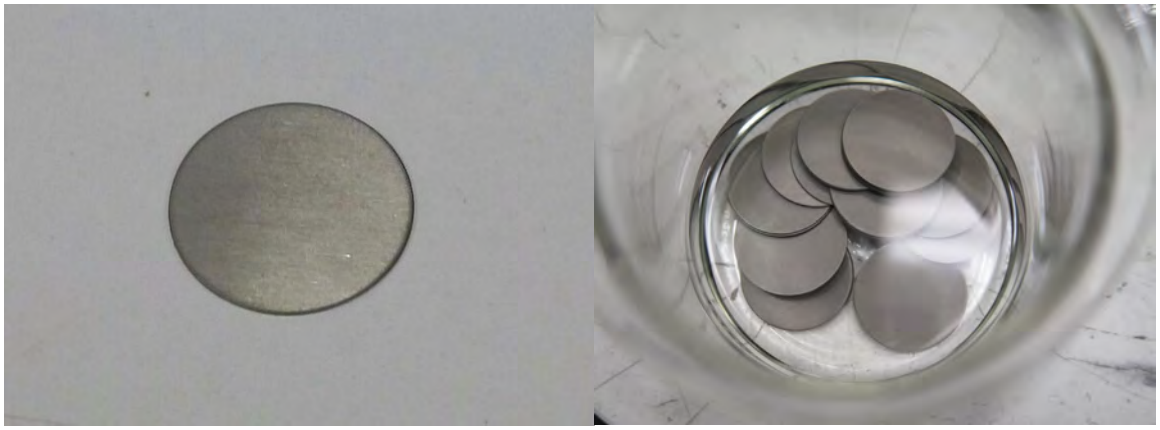

Figure 1. Nb discs Ø24mm×0.5mm

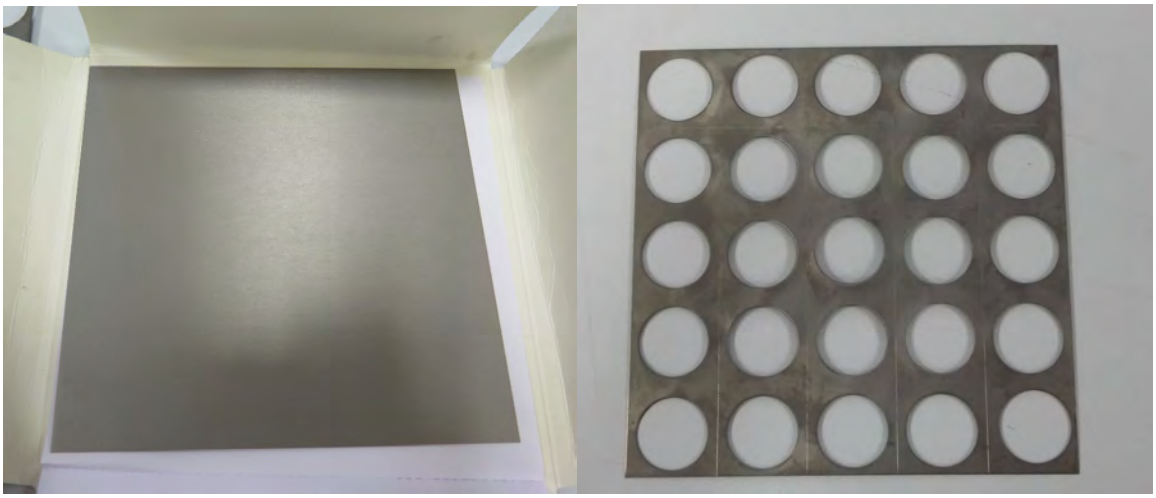

Figure 2. Nb 15cm x 15cm x 0.5mm sheet before and after cutting

Nb substrate cleaning procedure include:

a. Ultrasonic bath cleaning with Rodaclean® soap 20 min at 40˚C
b. Ultrasonic bath cleaning with deionized water 20 min at 40˚C

c. Rinsing with ethanol (storage in ethanol in plastic box)
d. Mechanical cleaning with ethanol and AlfaWipe®, drying with nitrogen gas immediately before positioning onto substrate-holder

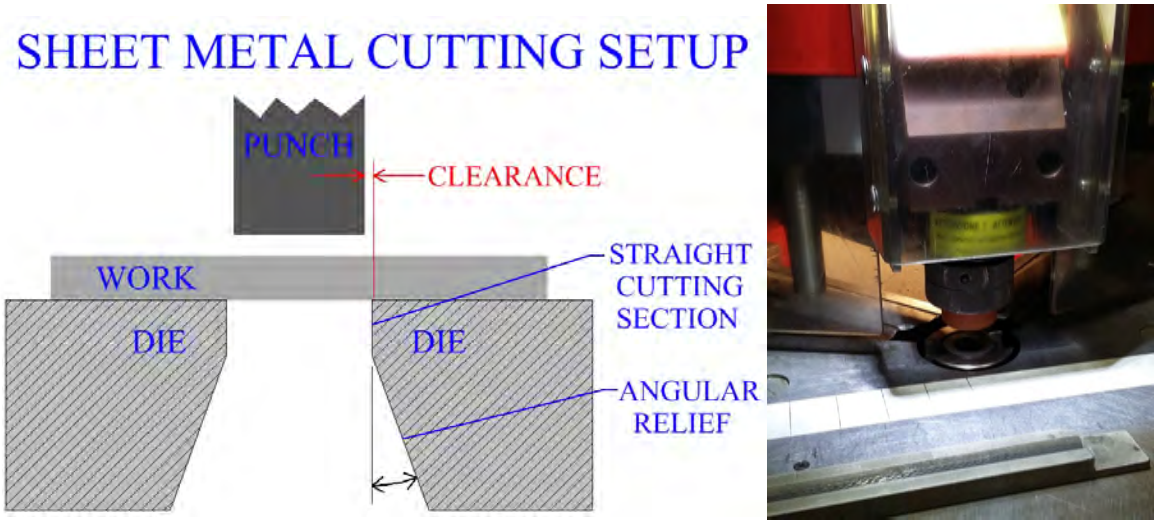

Figure 3. Metal sheet cutting setup

1.2 Sputtering chamber opening.

The Y deposition process is realized in the working chamber N4 in the 4-chamber sputtering system (Figure 4).

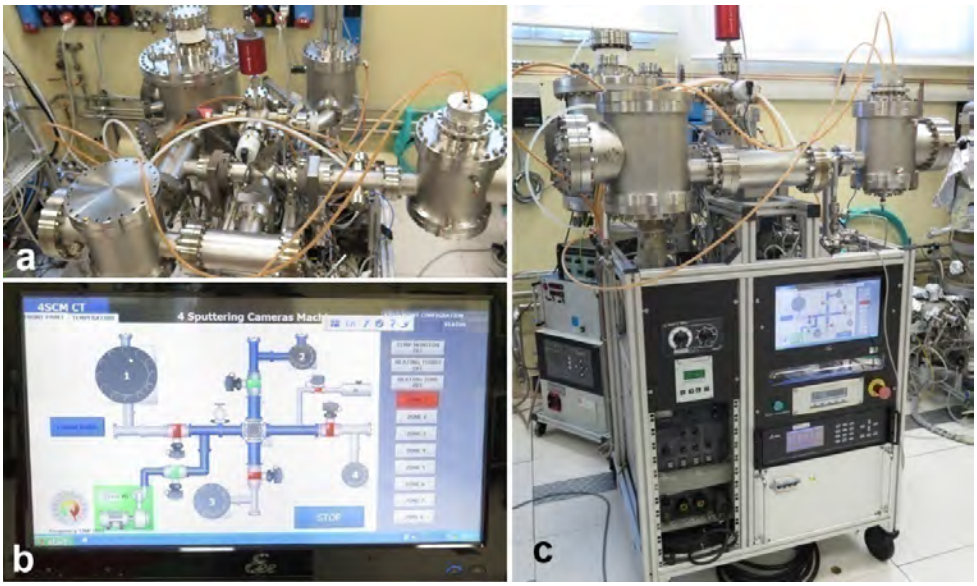

Figure 4. Complete 4-chamber sputtering system: a) top view, b) PLC control system, c) full system.

The chamber opening is realized with the aspiration positioned around the flange of substrate-holder, aspiration system turned on (Figure 6), with the use

of PPE (in ideal case paper filter mask, transparent protective glasses, nitrile gloves). The aspiration system is equipped with HEPA filter closed in home-made plastic box (Figure 5) to minimize the problems of yttrium flammability (flammability 2/4) and yttrium oxide toxicity (toxicity 2/3). The three chains of the crane are connected to the substrate-holder (Figure 7). Then the upper flange of the chamber – CF DN150 of the substrate holder should be unscrewed. It is normally closed with Viton® o-ring.

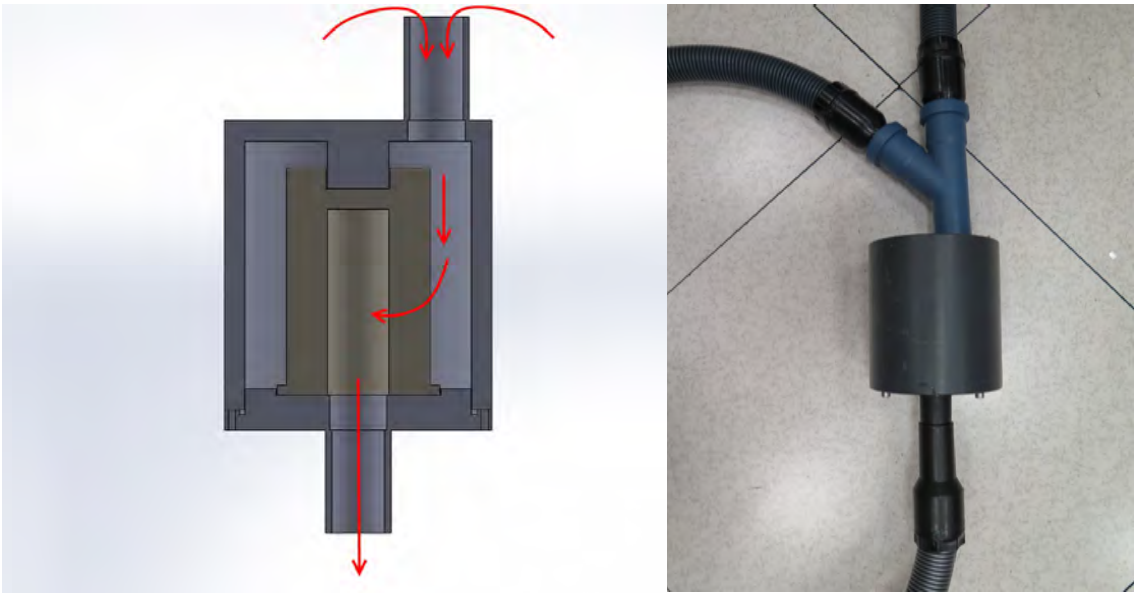

Figure 5. Box for the HEPA filter and aspiration direction

Figure 6. Upgrade of the system with aspiration ring and HEPA filter

The substrate-holder ground should be detached.

Venting is done in the next order:

a. Closing gate of turbo-pump
b. Stop pumping
c. Inserting Ar with the flow meter at 99sccm (max) up to 10 mbar
d. Closing Ar flow
e. Venting chamber with nitrogen (standard)
f. At 1000mbar (atm) the substrate-holder can be lifted 1 cm by manipulator with aspiration in the gap for some minutes.

The substrate-holder is returned into standard position in order to detach the crane chains. Then the substrate-holder can be manipulated manually to take out deposited samples or position new substrates (PPE, attention).

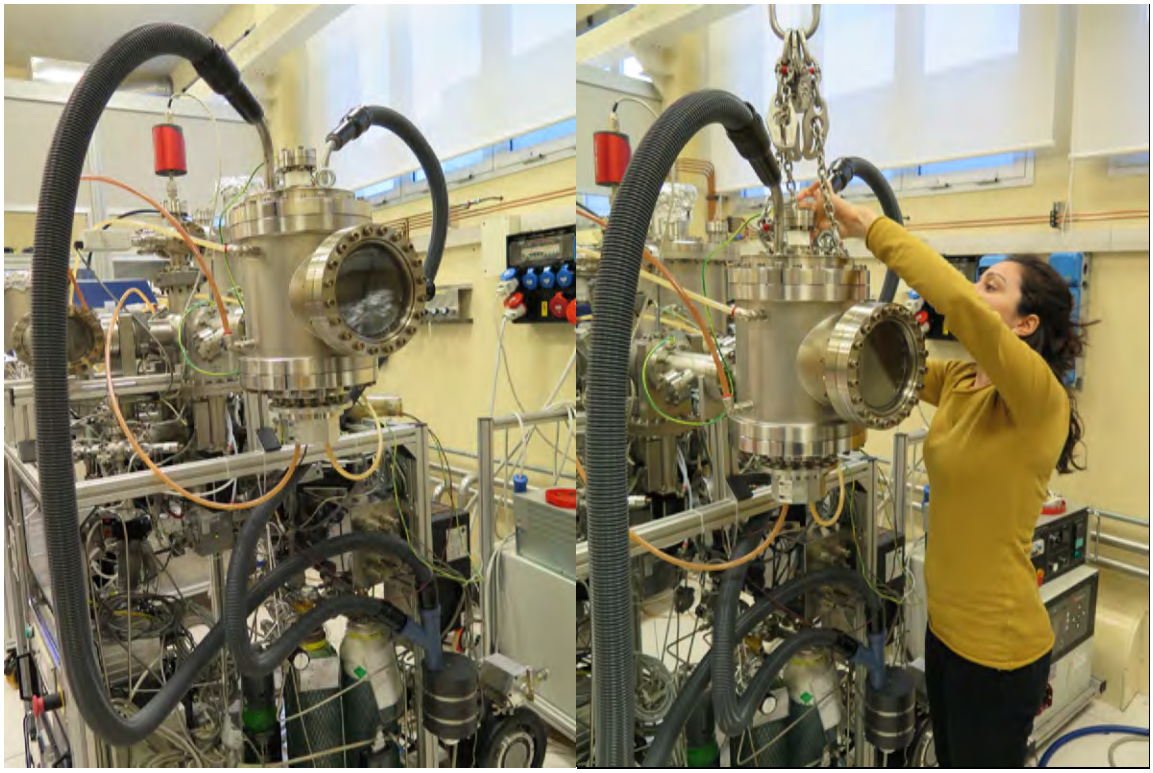

Figure 7. Placing aspiration ring and connecting chains to SH

1.3 Y sputtering target substitution

The lower flange of the chamber N4 – CF DN150 of the magnetron is unscrewed carefully with the use of PPE (possible sparks attributed by friction). The work is done in two for the safety reason. The magnetron is dismounted and placed onto clean table, free of flammable materials, covered with Al foil.

Figure 8. Detaching used Y sputtering target from magnetron

First the outer shield and then target centring shield are dismounted with attention (Figure 8). The eroded Y target is vacuumed (Figure 9) and stored in Chemical lab in the storage for flammable solids, the same place where new Y sputtering targets are located. When the study is completed the eroded targets should be treated according to the rules of environmental management.

Figure 9. Eroded Y sputtering target packaging

With a particular attention the rests of Silver-paint are cleaned by acetone (Figure 10). Metallic dust should be aspirated with the aspiration system (pay attention to sparks!) through the same box with HEPA filter.

Figure 10. Cleaning magnetron

The new Y sputtering targets (Ø50.8mm×5mm, 90g) are stored in original hermetic plastic bags in coloured plastic boxes in the flammable chemicals storage in Chemical lab. The plastic bag should be opened directly before use. The new target of Y is positioned on the magnetron with the use of Silver-paint for increased thermal contact (Figure 11) and blocked with centring shield (cleaned). Then external magnetron shield is attached. Both shields are present in 2 equal units in order to guaranty cleaning of the couple of shields before each new deposition.

Figure 11. New Y sputtering target positioning

The magnetron is placed back to the lower CF DN150 flange of the sputtering chamber.

1.4 Positioning substrates on the substrate-holder.

Figure 12. Niobium substrates preparation

Niobium substrates are additionally cleaned with ethanol and AlfaWipe® (Figure 12) and dried with nitrogen before use. Each substrate is weighed (Figure 12) and marked by scratch with roman numbers (I-VII) on the back side. 7 substrates are placed in their positions on the substrate holder (Figure 13), fixed with the mask (cleaned before from rests of yttrium).

When the substrate-holder with substrates is ready, the sputtering chamber cleaning by aspiration system is realized (with attention and use of proper PPE) and then the substrate-holder is paced and tight on the upper CF DN150 flange of the sputtering chamber with Viton® o-ring.

Figure 13. Mask and substrate-holder for 7 substrates

1.5 Vacuum

The vacuum chamber N4 is installed in 4-chamber vacuum system in Sputtering laboratory. Each chamber is separated from the central zone by

pneumatic gates (Figure 4). The pumping is realized from the central zone by the general pumping system, including Varian Tri Scroll 210l/min primary pump and Pfeiffer 360 l/mim turbomolecular pump (with Pfeiffer DCU display and operating unit). All the system is controlled with PLC-based LabVIEW® program.

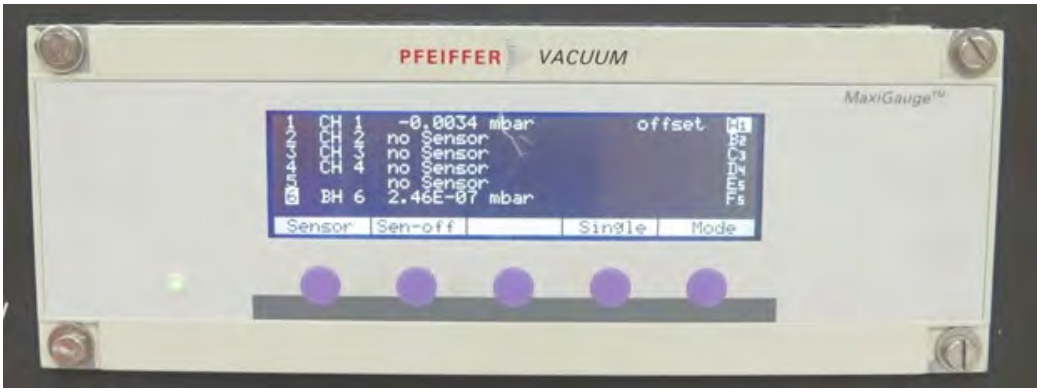

Figure 14. MaxiGauge™ pressure control box.

The algorithm of pumping chamber is:

a. The chamber N4, the central cross and pumping unit are in atmosphere
b. Opening gate from the chamber N4 to central cross
c. Opening gate from central cross to pumping unit
d. Start pumping

The base vacuum pressure is controlled with full-range Bayard-Alpert (BA) vacuum-meter. The vacuum-meter is connected to Pfeiffer MaxiGauge™ control box (Figure 14).

The deposition process is realized at vacuum base level of **2 x 10^{-6}mbar**.

1.6 Deposition

The deposition is realized after the turbomolecular pump is switched into **stand-by regime** (50% frequency) with PLC. The Both the Chamber N4 and magnetron should be connected to water cooling circuit. (Control the absence of errors and level of water in the chiller!). The water must circulate to cool down magnetron and chamber. The substrate-holder must be grounded as on the Figure 7.

The MKS multi gas mass-flow controllers powered by MKS 647C four-channel power supply/readout system (Figure 15) is used for gas flow control during

the deposition process. The pressure during MS deposition is controlled with capacitance vacuum-meter, since it is not sensitive to plasma as BA. The read-out is realized with MKS 647C.

Figure 15. MKS multi gas controller 647C

The argon pressure during deposition chosen for minimization of the intrinsic stress in film is **1.36 x 10^{-2} mbar**, corresponding to the 19 sccm gas flow. In the MKS 647C the CH1 is dedicated and calibrated for argon.

The magnetron is connected to the Advanced Energy MDX 1.5K magnetron drive - DC power supply (Figure 16). The process is realized with the control in power, fixed at **400W**. Magnetron plasma during Y deposition at described parameters is shown onto Figure 16. The total time of sputtering with one Ø5cm x 5mm sputtering target at such conditions is **8.5 hour** (with last 15 min remained to assure no impurities from silver-paint and copper base).

Figure 16. MDX 1.5K power supply and plasma

After the deposition the magnetron powering is switched off.

The argon flow is closed immediately after deposition is finished.

The turbomolecular pump is returned to the normal pumping regime with the PLC and the system is pumped until next morning.

The water flow for magnetron and chamber cooling can be closed the next morning after deposition.
No forced cooling of the substrate-holder is realized; thus the chamber can be opened to air just the **next day**, when it has reached the room temperature. Otherwise the SH can be modified adding a thermocouple for temperature control.
Following chamber opening is realized according to Paragraph 1.2 of the current protocol.

1.7 Targets analysis and packaging

After the sputtering chamber N4 is opened according to the Paragraph 1.2 of the current protocol, the substrate holder is dismounted and placed on the working table covered with aluminium foil and liberated from flammable materials.

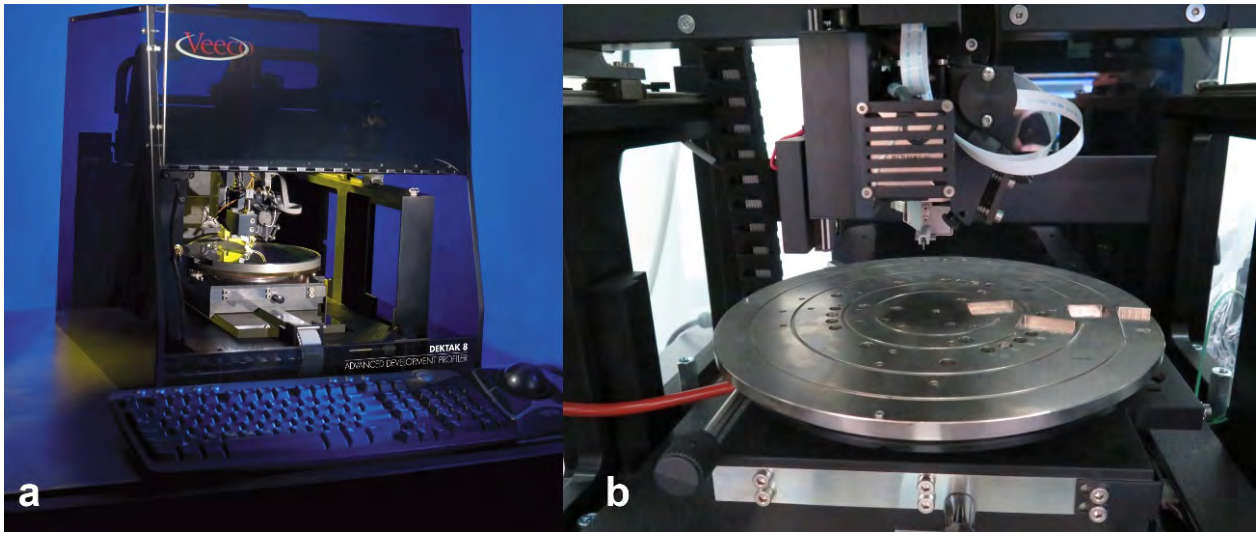

Figure 17. a) Dektak 8 profiler of Veeco; b) internal view of the system.

The mask (Figure 13) is dismounted and the targets are ready for the analysis. Each target is weighed with the balance and the profile is measured with the Dektak 8 contact profiler (Figure 17) once along the axis of the disc, covering the area of the deposit as shown on the Figure 18.

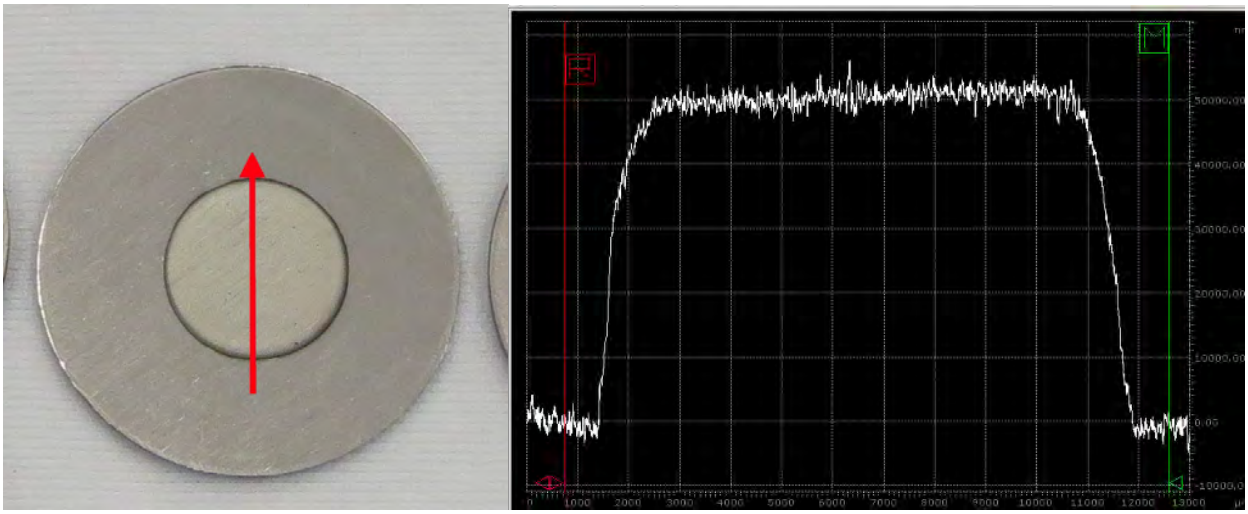

Figure 18. Target profile analysis

The parameters for Y target profile measuring with Dektak 8 are:

- length 13000um
- duration 60 sec
- stylus force 5 mg
- standard scan type
- meas range 262000nm
- profile hills

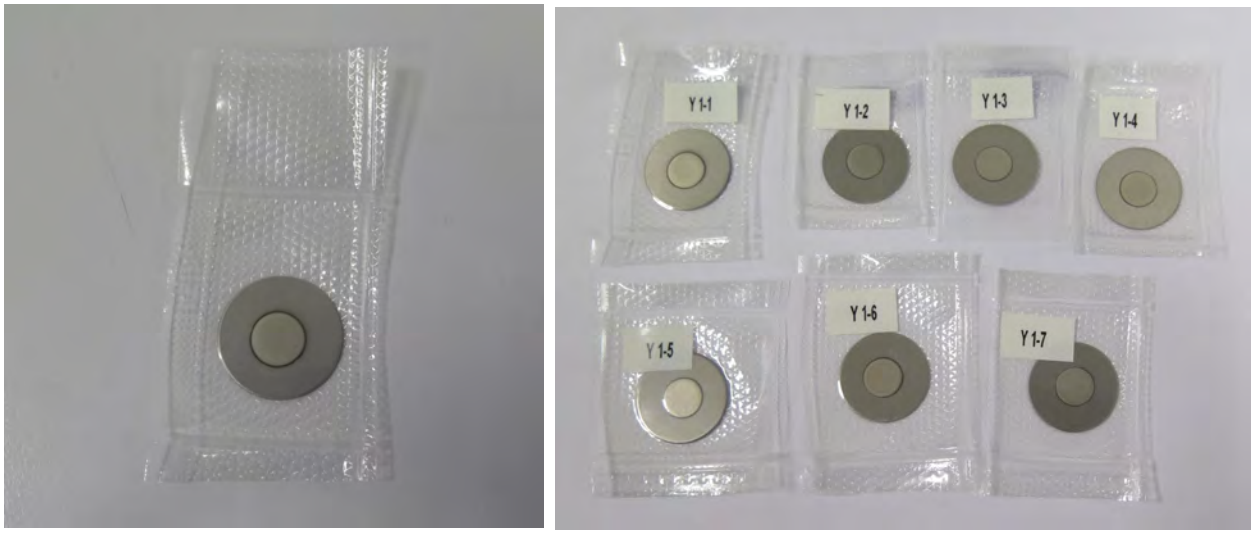

Figure 19. Ready target packaging and labelling

All the target analysis is realized in minimal time (up to 30 min after chamber opening for the last sample of the batch) in order to minimize the exposure of the sample to air. Immediately after profile measuring each target is packaged

in vacuum bag after air pumping as shown on Figure 9. The targets packaged with the proper labelling are shown on the Figure 19.

1.8 Chamber cleaning

After each process during the change of substrates and magnetron sputtering target the chamber should be cleaned with the vacuum-cleaner using dedicated filter (Figure 5).

Y deposited onto the magnetron shields (Figure 8) and substrate holder mask (Figure 13) should be chemically etched after each deposition, since they are becoming a source of metallic dust, that can become the reason of deposition blocking.

After the number of Y deposition processes also complete substrate holder, the shield of the sputtering chamber (Figure 20), the shield of the viewport, two CF DN 250 flanges are dismounted and should be chemically etched.

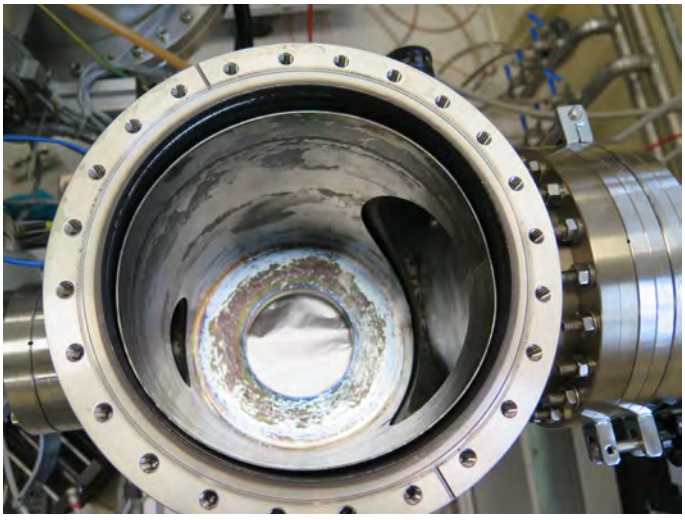

Figure 20. Chamber shield

Chemical etching of stainless steel parts is realized following the next receipt:

a. Ultrasonic bath with Rodaclean® soap at 40˚C for 30 min
b. Ultrasonic bath with water at 40˚C for 30 min
c. HCl 15-20% solution for 2 min at r.t.
d. Water rinsing
e. Ethanol rinsing
f. Nitrogen drying

1.9 Waste treatment

The Y traces in HEPA filters (Figure 21) after number of experiments can be treated similar to the shields and flanges by dissolution in HCl solution.

The solutions after the etching of the shields, flanges, etc. should be treated as standard acid wastes, since the Y^{3+}, contained in the solutions, has low toxicity.

The Y eroded sputtering targets also should be treated according to the rules of environmental management.

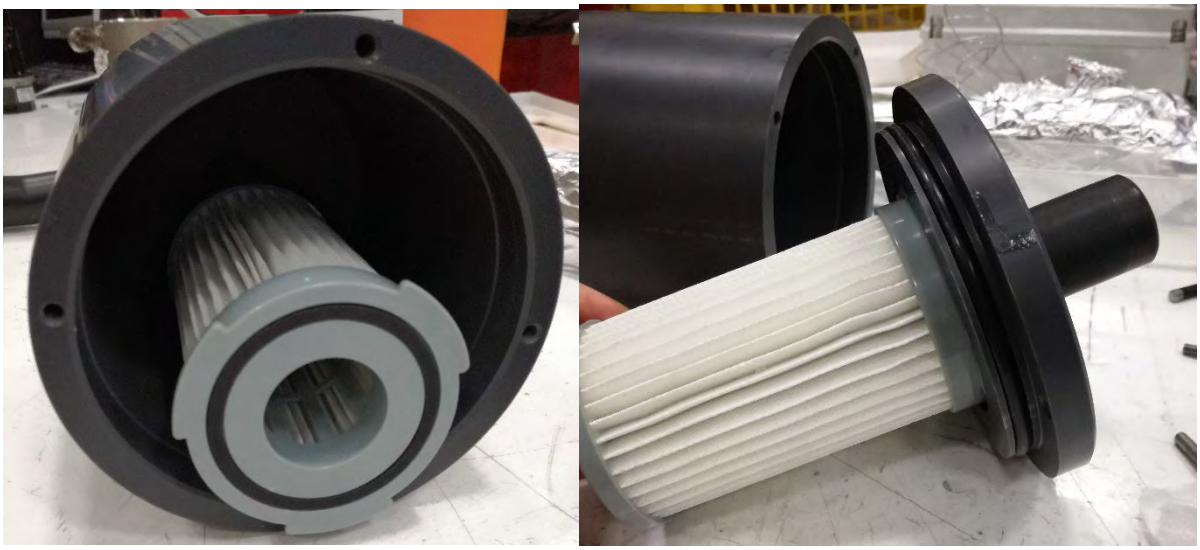

Figure 21. HEPA filter

2 SUPPORTING DOCUMENTS

2.1 Experiment parameters table

The next table of parameters should be fulfilled for each experiment in order to provide the repeatability of the process:

Experiment number	
Data of deposition	
Mask cleaning (yes/no)	*yes*
Shields cleaning (yes/no)	*yes*
New Y sputtering target (yes/no)	*yes*
Sputtering target-substrate distance, cm	*7*
Base vacuum pressure, mbar	*2×10^{-6}*
Ar flow, sccm	*19*
Sputtering pressure, mbar	*1.36×10^{-2}*
Magnetron PS power, W (fixed)	*400*
Magnetron PS current, A (range start..finish process)	
Magnetron PS voltage, V (range start..finish process)	
Deposition time, h	*8.5*
Deposition rate, um/h	

The fixed values are also presented in the table.

2.2 Targets analysis table

The next tables for each sample should be fulfilled in order to provide the quality control:

Sample number	**Weight**			**Y thickness, um**
	Nb substrate, g	After deposition, g	Y deposit, mg	
1-1				
1-2				
1-3				
1-4				
1-5				
1-6				
1-7				

Sample number	**Y mass, mg**	**Y thickness (calculated), mg/cm^2**	**Y thickness (measured with profiler), um**
1-1			
1-2			
1-3			
1-4			
1-5			
1-6			
1-7			

2.3 Personal Protective Equipment (PPE) list

1. Transparent glasses for mechanical protection: lens code 2C-1.2 XXX F (T) 3, frame code XXX EN166 2 F (T)
2. Disposable nitrile gloves
3. Disposable polyethylene gloves
4. Protective lab coat in cotton (antistatic)
5. Single-use filter face mask FFP1