Ni target development for TULIP project

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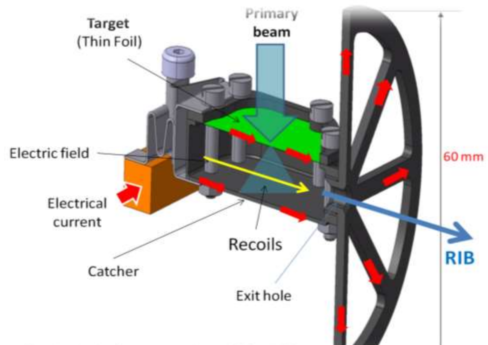
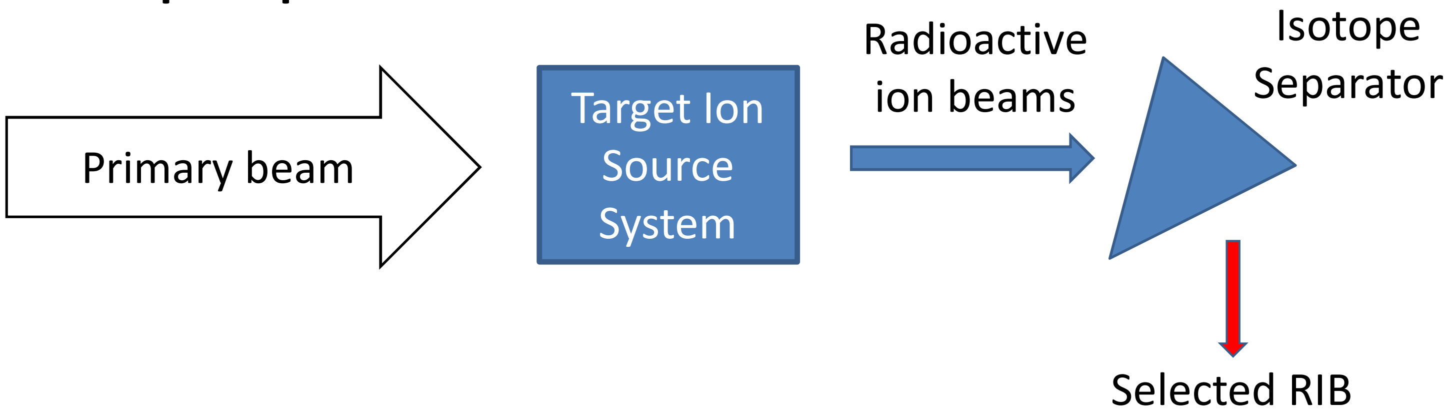


Fig 1: Vertical cross section of the TISS

ANR CNRS TULIP GANIL SPIRAL2 IPN

Production of radioactive ion beams at GANIL/SPIRAL1

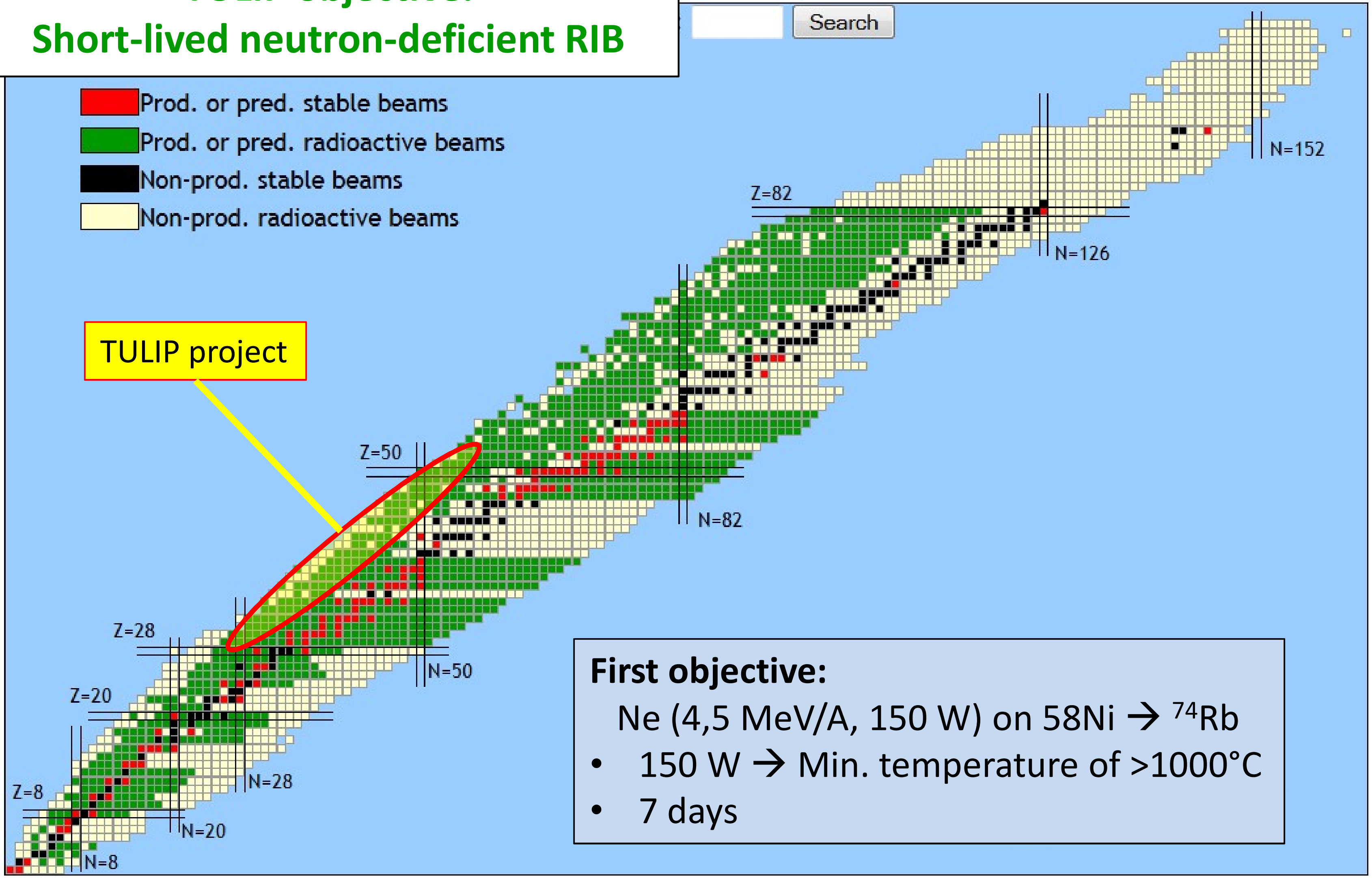
Method: Isotope Separator On Line



Primary beams: from C to U, Energy up to 95 MeV/A

- Thick target, from C to Nb
- « Thin » targets: submitted to safety autorisation for M > Nb

TULIP objective:
Short-lived neutron-deficient RIB

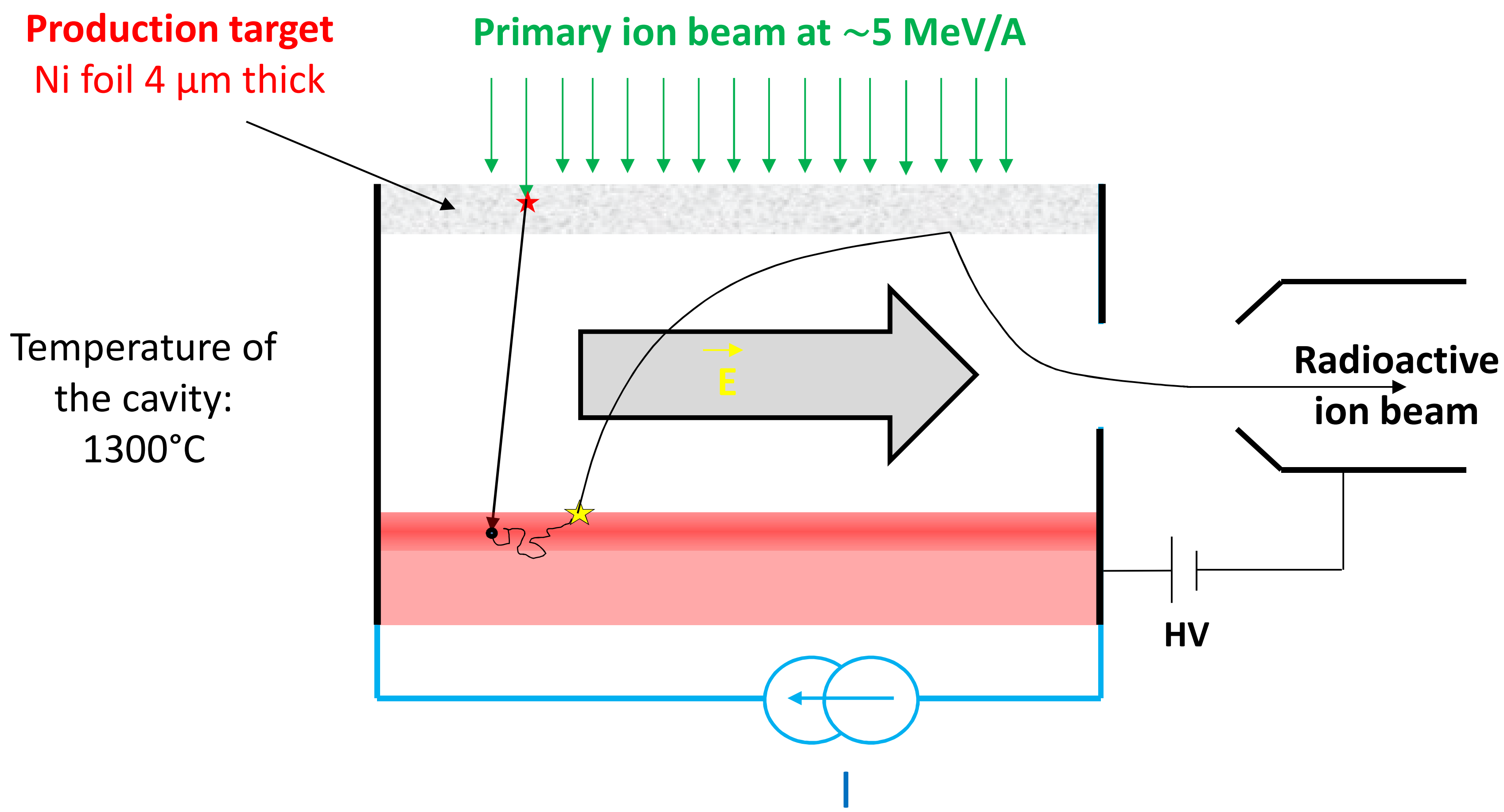


First objective:

Ne (4,5 MeV/A, 150 W) on 58Ni → ^{74}Rb

- 150 W → Min. temperature of >1000°C
- 7 days

Principle

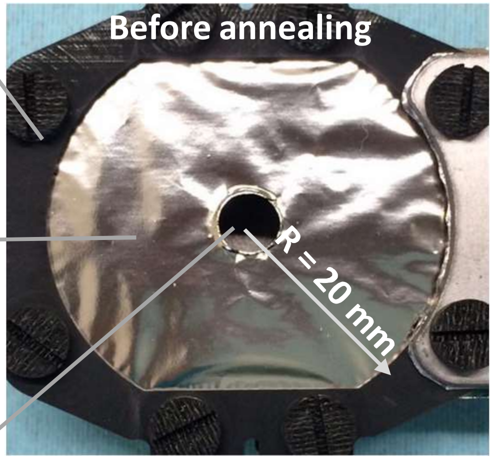


Production target
Ni foil 4 µm thick
Primary ion beam at ~5 MeV/A
Temperature of the cavity: 1300°C
E
Radioactive ion beam
HV
I

Thermal tests with TULIP V2:
4 µm thick pure Ni target

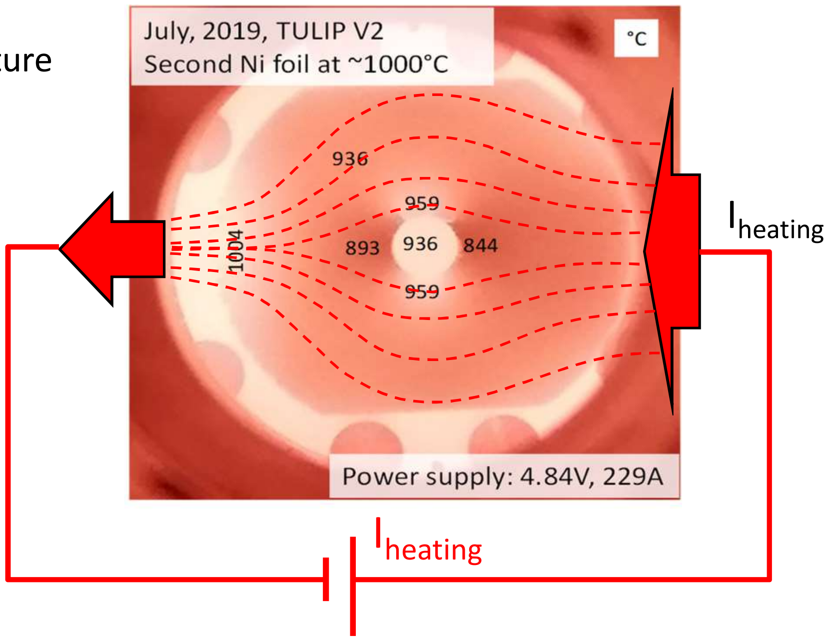
Graphite cavity

Ni target



6 mm hole for catcher temperature measurement

Annealing: 3 hours at up to 1350°C

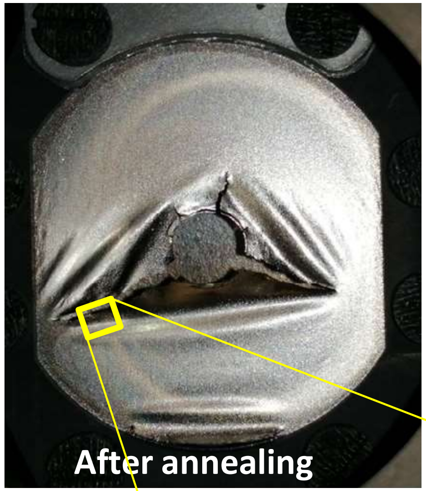


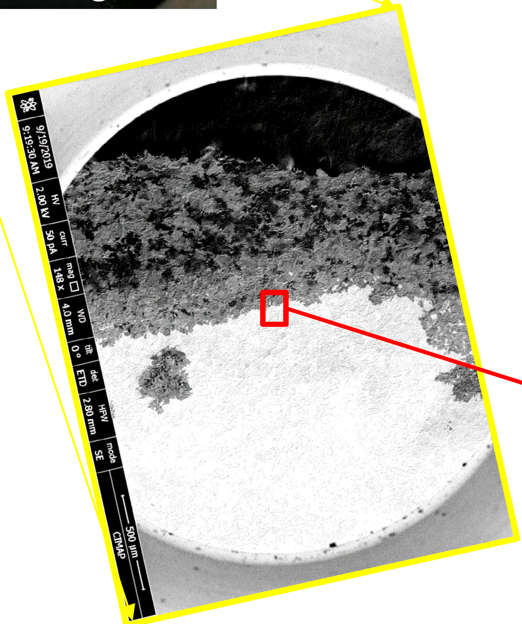
Shrinking and tearing of the foil

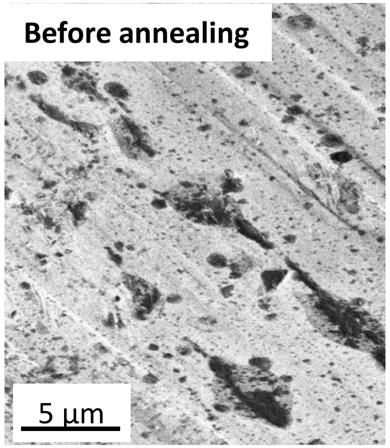
Why?

Ni foil supplier: Goodfellow, réf LS 482946

SEM (Scanning Electron Microscope) analysis of the foil damage

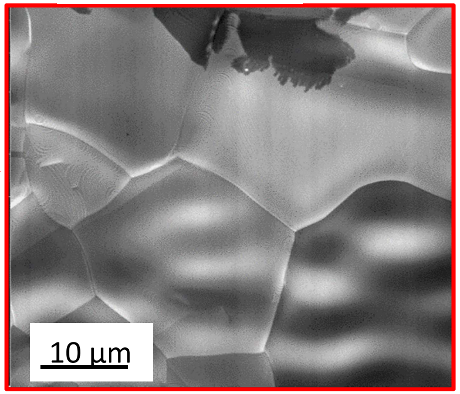






Traces of rolling

After annealing



~ 3h at T ~1350°C

Crystallisation

Crystallization, shrinking, fusion, tearing

Thermal tests

Behaviour of the target foil at high temperature (1350°C max.): 3 targets tested

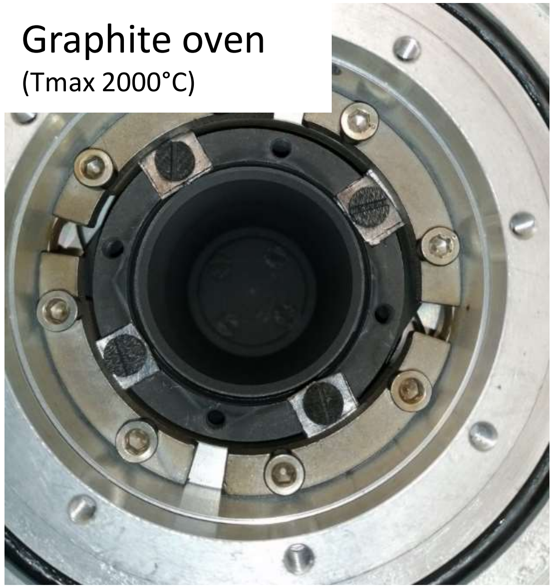
- One with TULIP V1: temperature difference of 450°C* at the surface of the target. Lifetime : ~3 hours at ~**1310°C**
- Two with TULIP V2: temperature difference of 153°C* . Lifetime : ~3 hours at **1350°C** and ~8 hours at ~ **1300°C** at maximum

- Temperature difference reduced to 109°C* on TULIP V3 and target mounted with more slack ➔ beginning of shrinking after 2,5h x **1040°C**

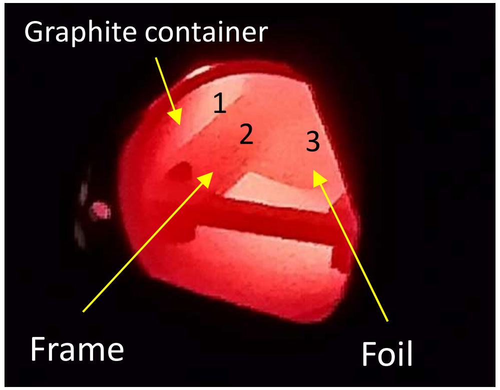
** According to ANSYS simulations*

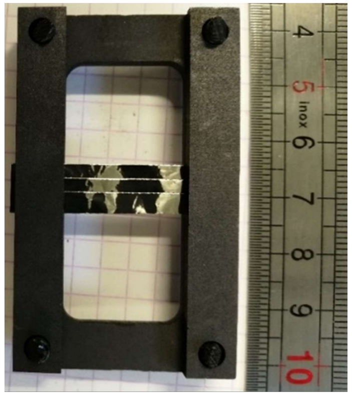
Systematic annealing of pure Ni (4 µm) foil in a graphite oven

Graphite oven
(Tmax 2000°C)



Frame and sample in the oven



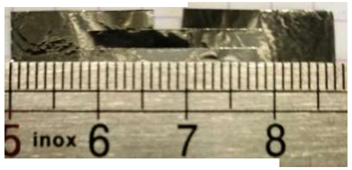


Samples for microscopic analysis

1

5.2.2020, 2^{h}x800°C

2

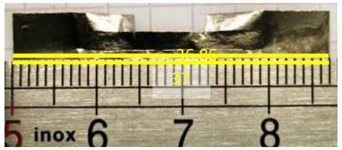


6.2.2020, 2^{h}x800°C + 2^{h}x837°C

3

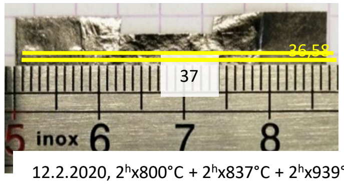
7.2.2020, 2^{h}x800°C + 2^{h}x837°C + 2^{h}x939°C

4



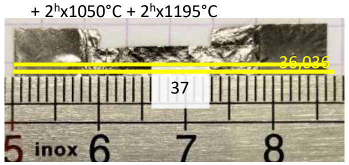
11.2.2020, 2^{h}x800°C + 2^{h}x837°C + 2^{h}x939°C + 2^{h}x1050°C

5



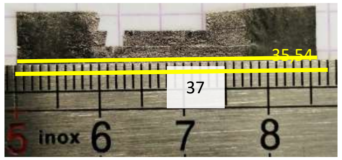
12.2.2020, 2^{h}x800°C + 2^{h}x837°C + 2^{h}x939°C + 2^{h}x1050°C + 2^{h}x1195°C

6



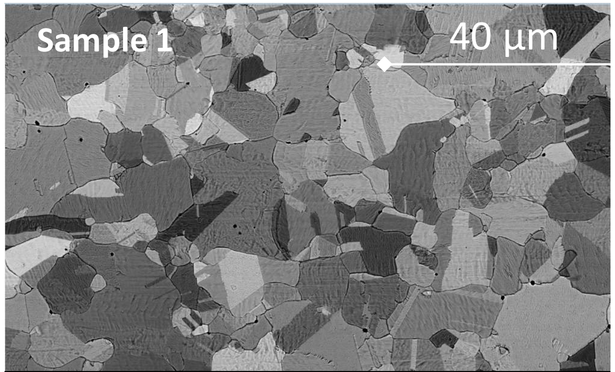
13.2.2020, 2^{h}x800°C + 2^{h}x837°C + 2^{h}x939°C + 2^{h}x1050°C + 2^{h}x1195°C + 2^{h}x1285°C

7

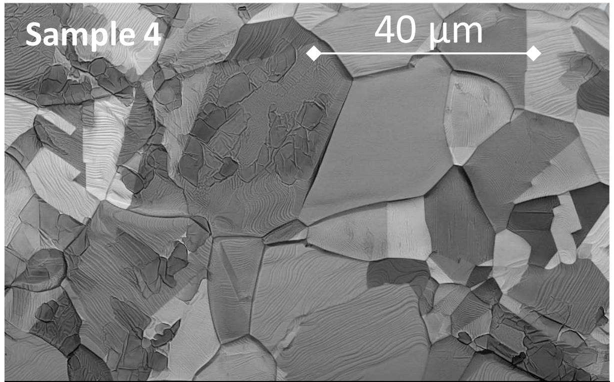


14.2.2020, 2^{h}x800°C + 2^{h}x837°C + 2^{h}x939°C + 2^{h}x1050°C + 2^{h}x1195°C + 2^{h}x1285°C + 15^{h}x1293°C

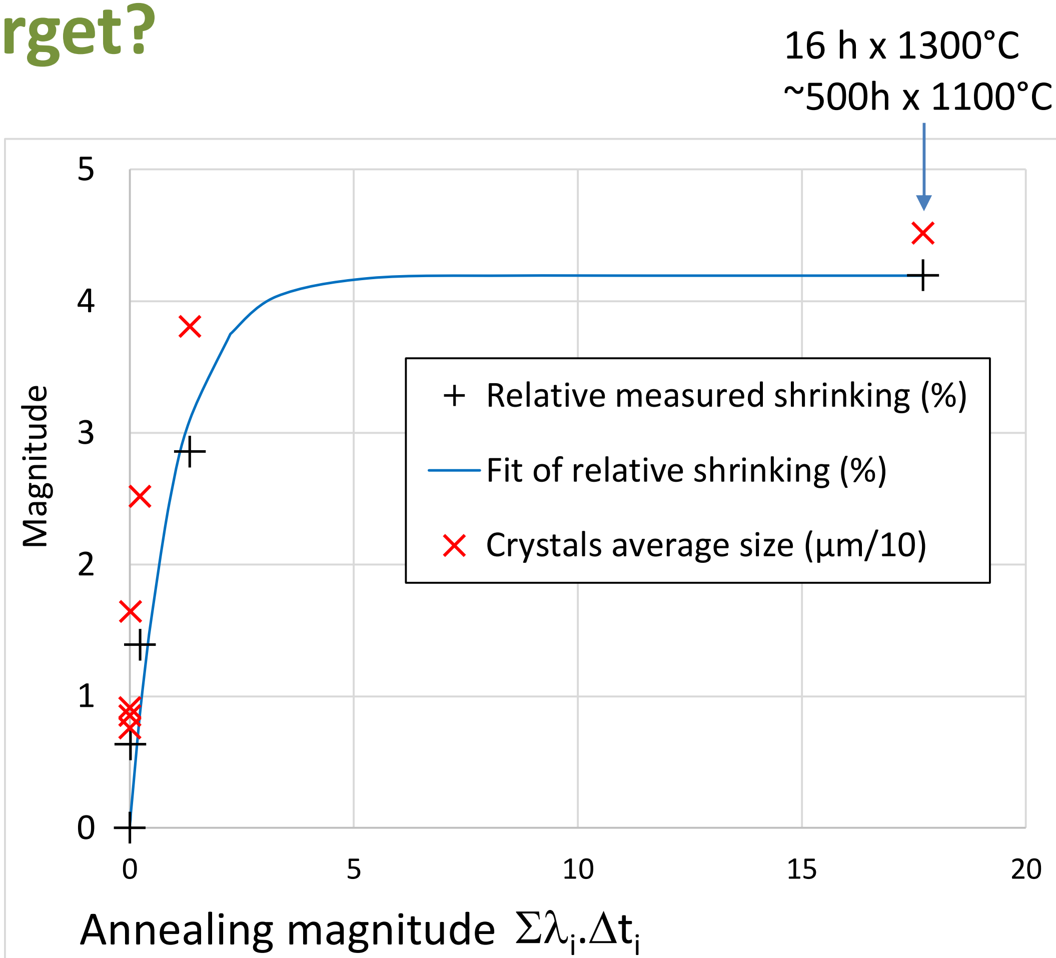
What changes in the target?



After the first annealing cycle



After the fourth annealing cycle



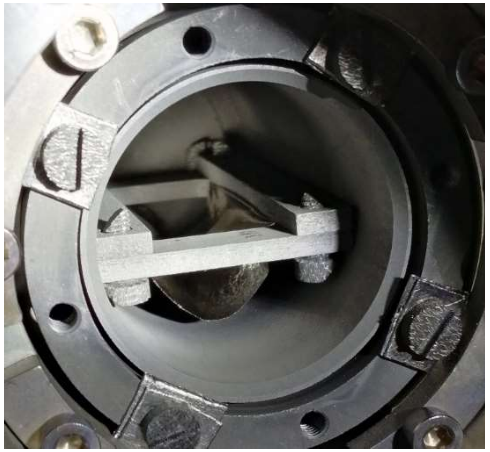
takes into account:

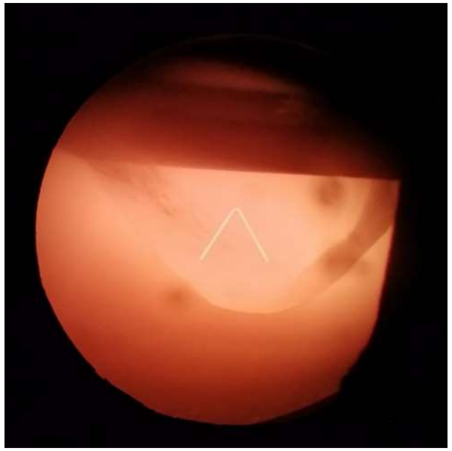
- the mobility of atoms,
- the temperature,
- the annealing time.

➔ **Possible saturation**

New test to verify the saturation existence

After 31h07 at 1110°C





Result

- Can be handled after annealing but more fragile
- Evaporation rate of 4,7%
- Relative shrinking higher than 10%: no saturation!

Pure Ni target used at 1300°C, and even at 1110°C must be abandonned

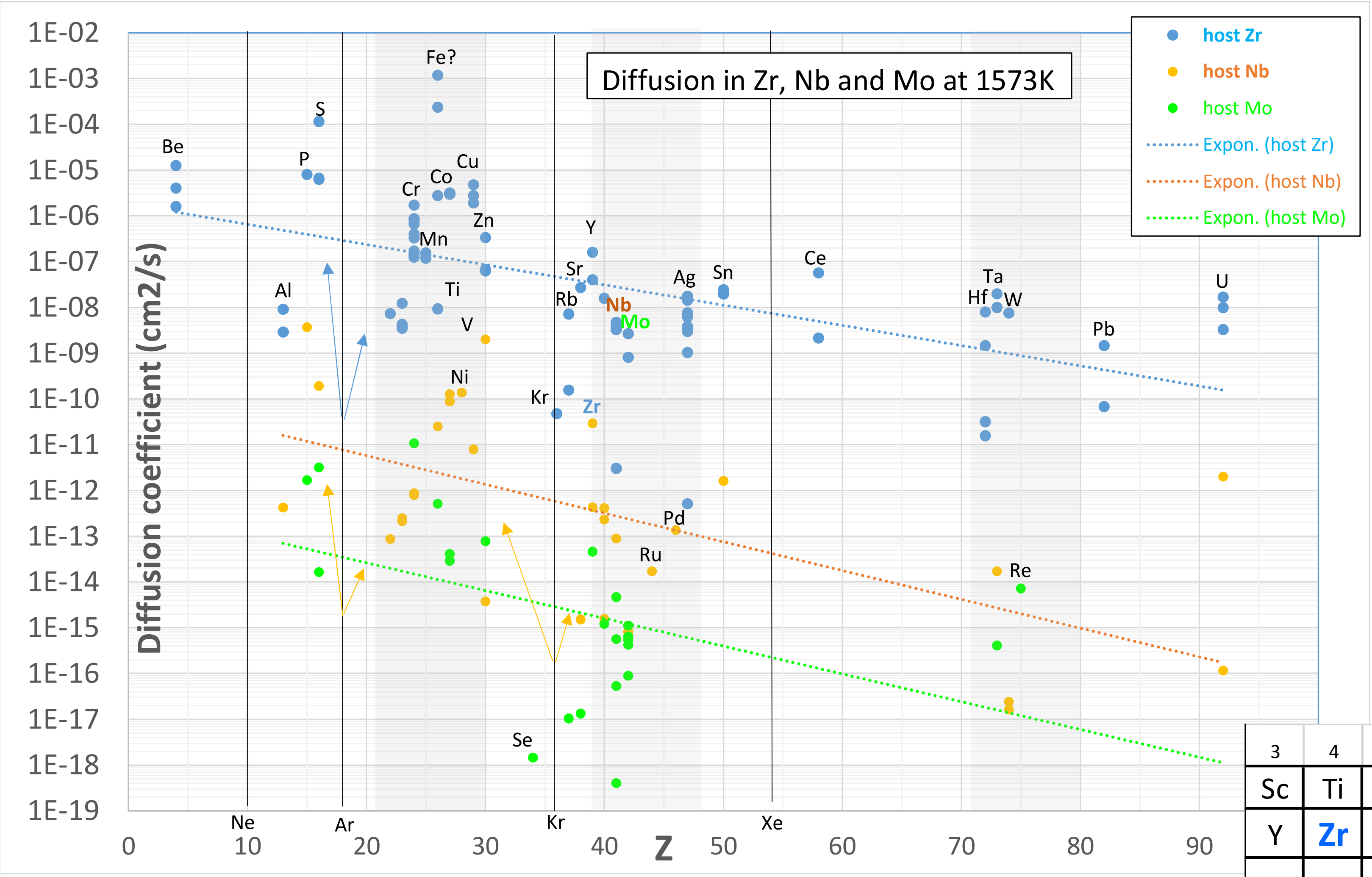
TULIP

How to stabilize the target material?
or
How to limit the mobility of the atoms?

By adding another element?

Which one?

Mobility of atoms in a material <-> Diff. Coefficient

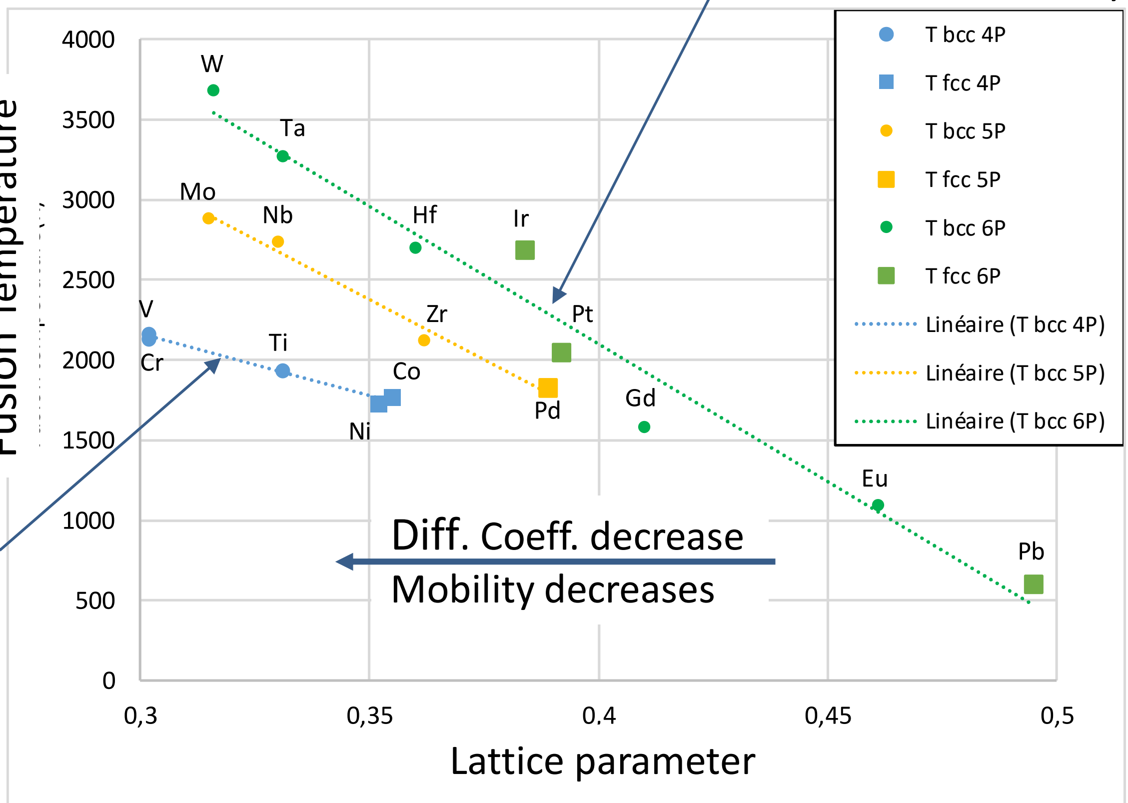


3	4	5	6	7
Sc	Ti	V	Cr	
Y	Zr	Nb	Mo	Tc
		Ta	W	Re

Handbook of self-diffusion and impurity diffusion in pure metals » de G. Neumann et de G.J. Beyer et al., NIM B 204 (2003) 225-234

Lattice parameter and mobility

Elements of the sixth period:
Out of SPIRAL1 safety authorization



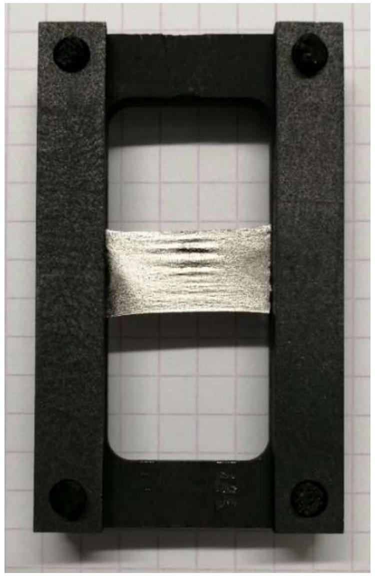
Elements of the fourth period:
Too close to Ni

➔ **Mo could be an adequat material.**

(Binary phase diagrams of 40 potential Ni-X alloys have also been considered)

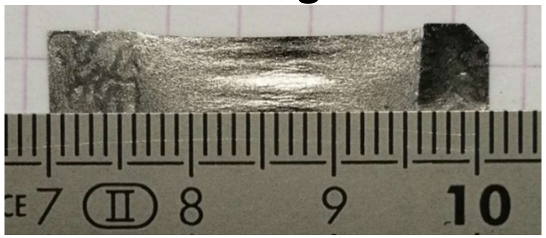
First test with a sample of Mo (0,09 µm) - Ni (4µm) -Mo (0,09 µm)

Mo deposited on Ni by evaporation



Before annealing

After annealing



29.5 h at 1250°C

Relative shrinking lower than 1% (10% for pure Ni at 1110°C x 31h)
Relative mass evaporation of 0.8% (> 4,2% for pure Ni at 1290°C x 15h)

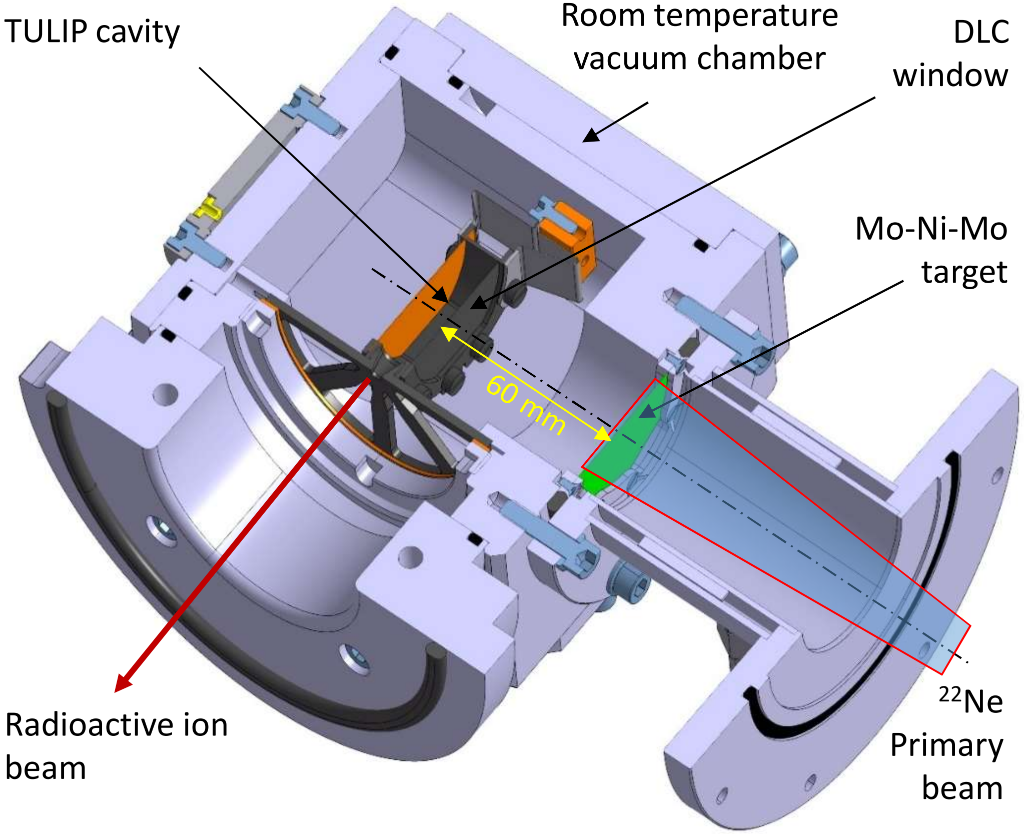
➔ Encouraging result, but will it be sufficient for 7 days?

➔ Next test with 0,3 µm of Mo on each face, and at lower temperature

How to reduce the target temperature?

By placing the target 60 mm upstream from the cavity,

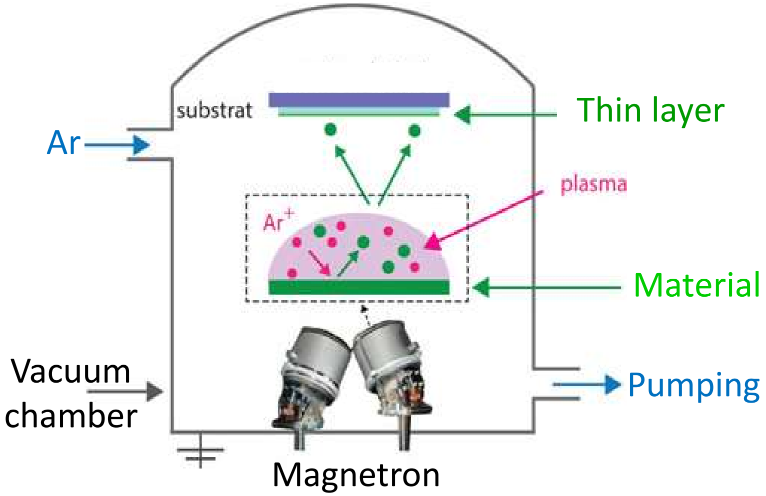
- T_{target} = 370°C without beam
- T_{target} ~ 1100°C with 30W of beam deposited in the target



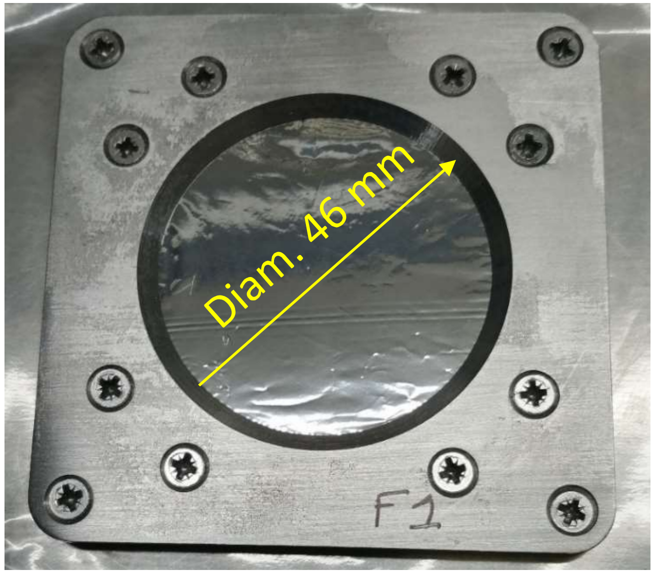
Realization of a Mo (0,3 µm) - Ni (4µm) -Mo (0,3 µm) sandwich target

Mo deposited on Ni by cathodic sputtering

Cathodic sputtering



Done by Kerdry company
https://kerdry.com



Mo thickness measured on each face after deposition: 0,3 µm +/- 2%

Thermal test of Mo (0,3 µm) – Ni (4,2 µm) – Mo (0,3 µm) target

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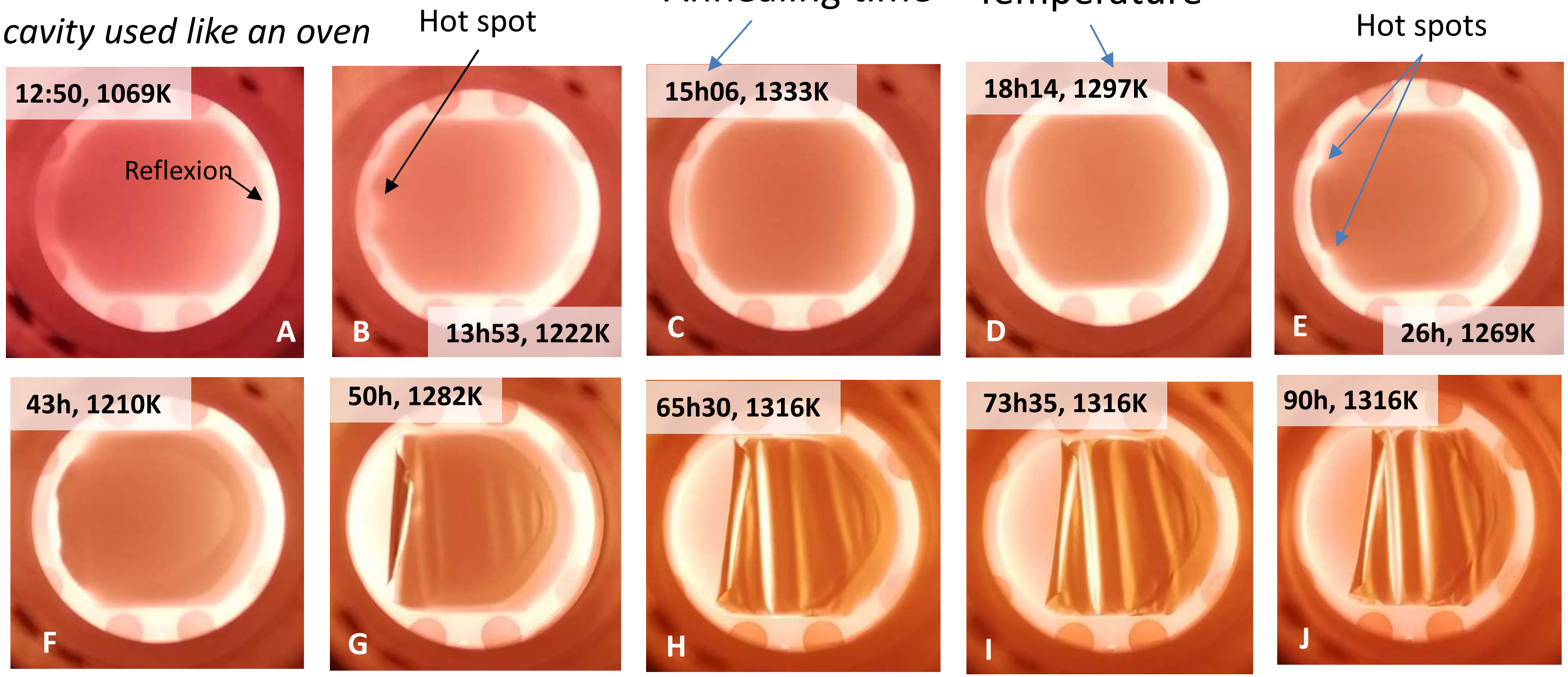
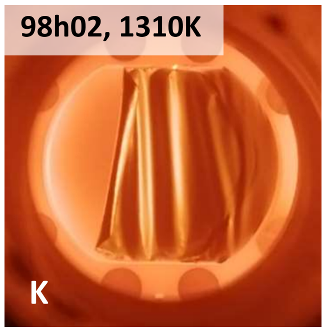
Installed on the TULIP cavity used like an oven





After 98 hours of annealing at an average temperature of ~1000°C

- Foil still flexible, easy to handle
- Relative shrinking close to 0% (>10% for pure Ni after 31h07 at 1110°C)
- Relative mass evaporation of 10% (> 4,7% for pure Ni after 31h07 at 1110°C)

➔ **Acceptable for the first on-line test**

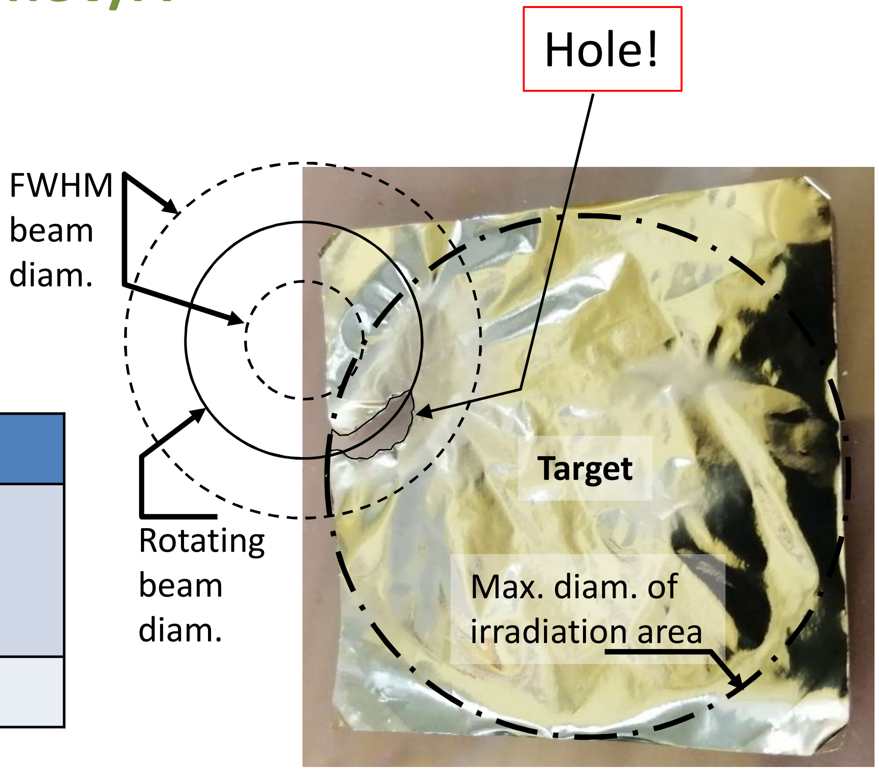
ANR cnrs TULIP GANIL SPIRAL2 IPN

On line test: ^{22}Ne Beam @ 4,5 MeV/A

Fluency: 1,7 x 10^{16} part/cm^2 in 9 hours
Power : up to 111 W

Ni emissivity : 0,19
Mo emissivity : 0,35

	Temperature (°C)	
Max. Target Power density (W/cm2)	ε=0.19	ε=0.35
8	1120	920



Excessive focussing or power density of the beam
(sputtering excluded)

⇒ Beam characteristics must be better controlled

Preliminary conclusion

Mo(0,3µm)-Ni (4µm)-Mo (0,3µm) sandwich

- seems to satisfy the stability requirement of the target if used at 1100°C
- allows to work for few hours (and to produce neutron deficient Rb^+ ions)

- Beam power density must be better controlled (or/and reduced) to test its life-time

- Next on-line test is expected by Spring 2023

Thank you for your attention

***Pascal* Jardin[1]**, *Vincent* Bosquet[1], *Samuel* Damoy[1], *Georges* Frémont[1], and *Marion* MacCormick[2]

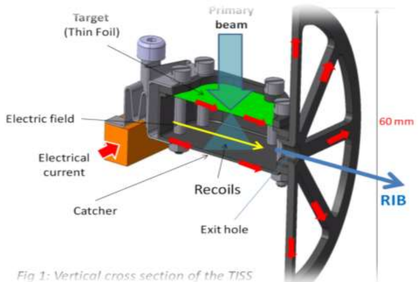
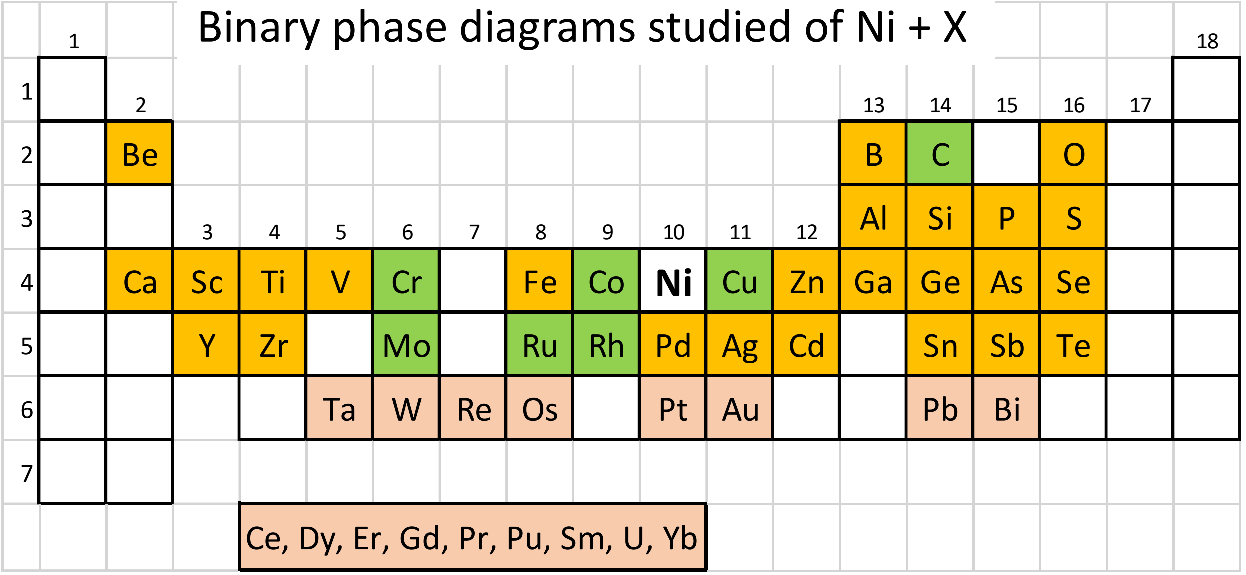


Fig 1: Vertical cross section of the TISS

Which material could be associated to Ni?



Pink: need a specific safety study

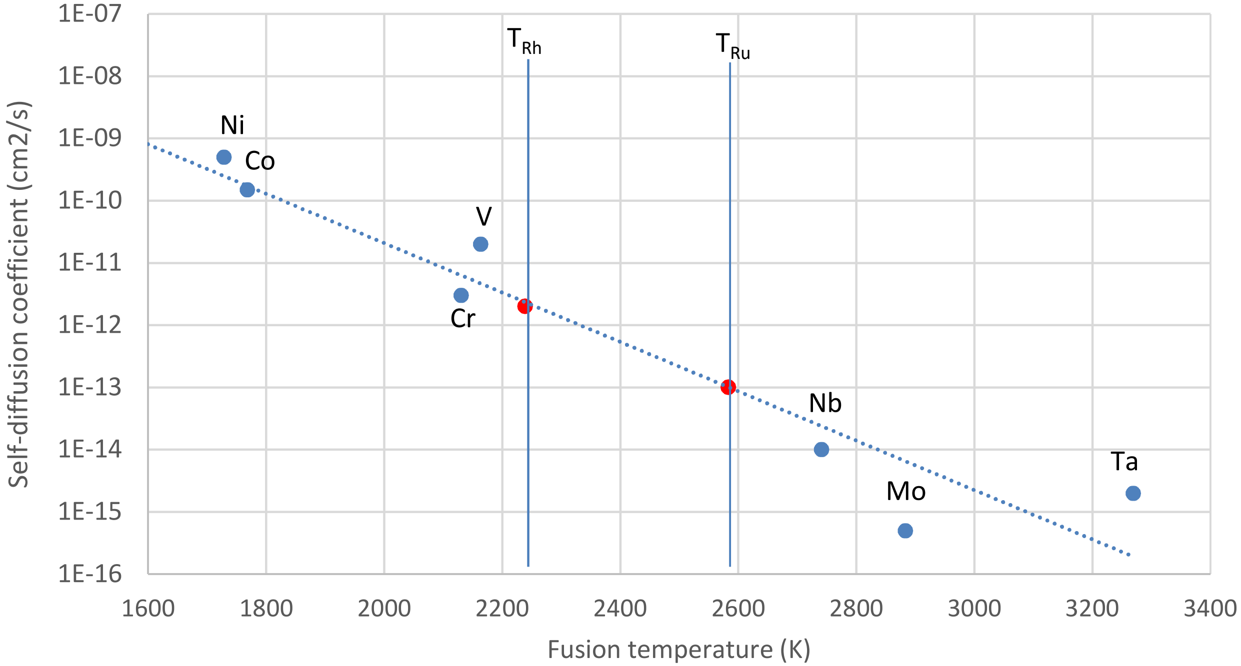
Orange: complex phase diagram or too low fusion temperature (<1570K)

Green : phase diagram ok and alloy fusion temperature > 1573K

Cr, Co and Cu are too close to Ni

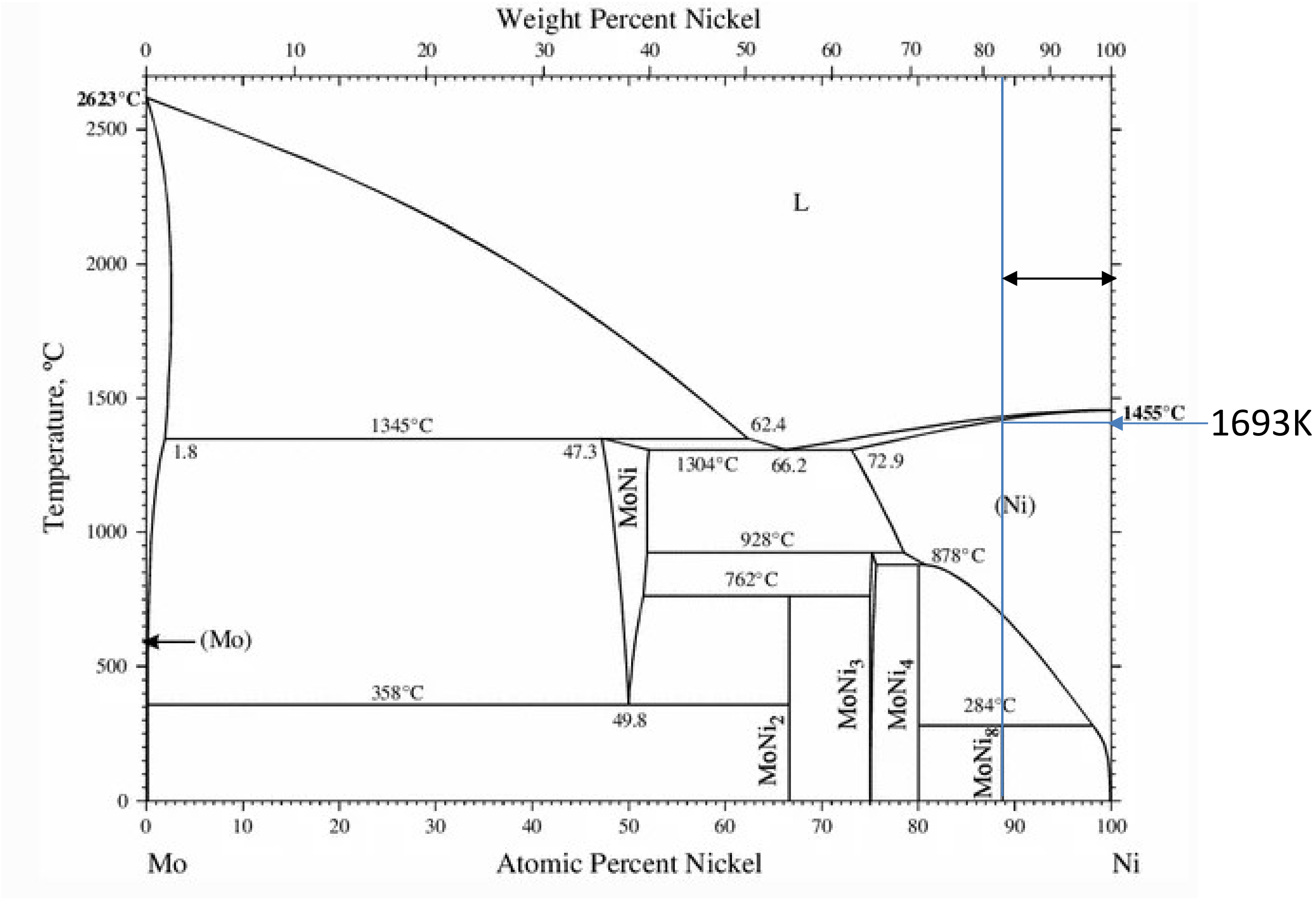
➔ Mo, Ru and Rh could be good candidates

Mobility or self-diffusion versus fusion temperature



	1																	18
1		2											13	14	15	16	17	
2		Be											B	C		O		
3			3	4	5	6	7	8	9	10	11	12	Al	Si	P	S		
4		Ca	Sc	Ti	**V**	**Cr**		Fe	**Co**	**Ni**	Cu	Zn	Ga	Ge	As	Se		
5			Y	Zr	**Nb**	**Mo**	Tc	Ru	Rh	Pd	Ag	Cd		Sn	Sb	Te		
6					**Ta**	W	Re	Os		Pt	Au			Pb	Bi			
7																		

Ni-Mo phase diagram



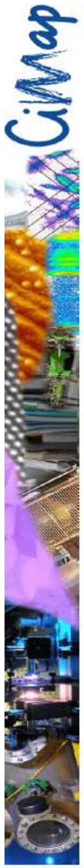
Okamoto, H. Supplemental Literature Review of Binary Phase Diagrams. Phase Equilib. Diffus. ***35****, 208–219 (2014).*

TULIP

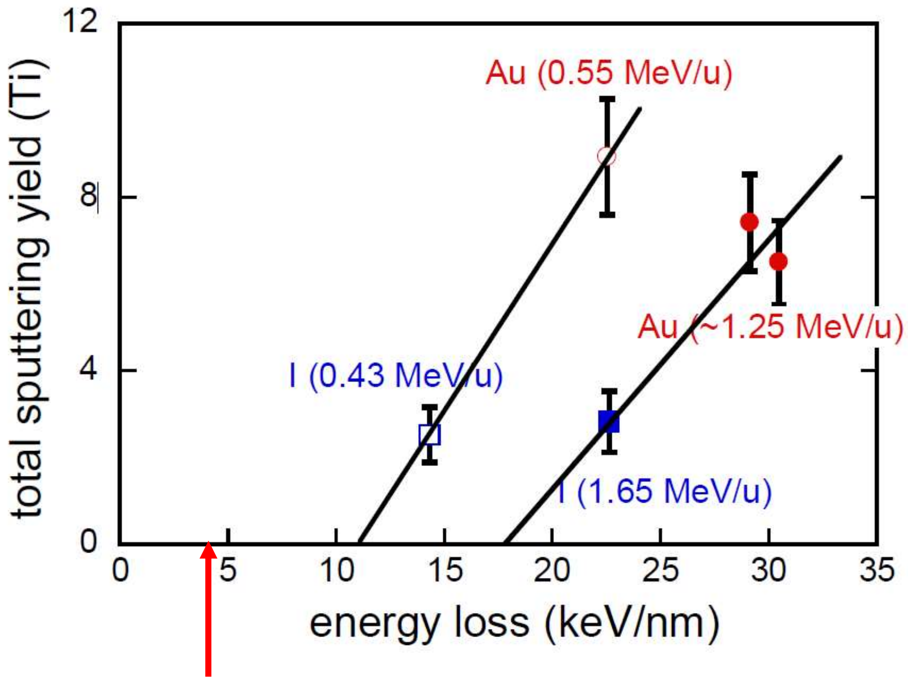
What about sputtering?

M. Toulemonde, C. Dufour , CIMAP, Caen (France)

Toulemonde et al. Phys. Rev. B46(1992)14362
Dufour et al. J. Phys.: Condens. Matt. 5(1993)4573
Toulemonde et al. Nucl. Instr. Meth. B66-167(2000)903



Titanium sputtering



^{22}Ne @ 4,5 MeV/A in Ni

Sputtering rate required to explain the hole: ~ 2000 at / ion
Sputtering damage negligible for an energy loss of 4 keV/nm.
Can not explain the hole
Confirmed by A. Benyagoub (CIMAP)

MSU
MAY
2013