Spark Plasma Sintering Technique for Cyclotron Solid Target Manufacturing

Sara Cisternino*

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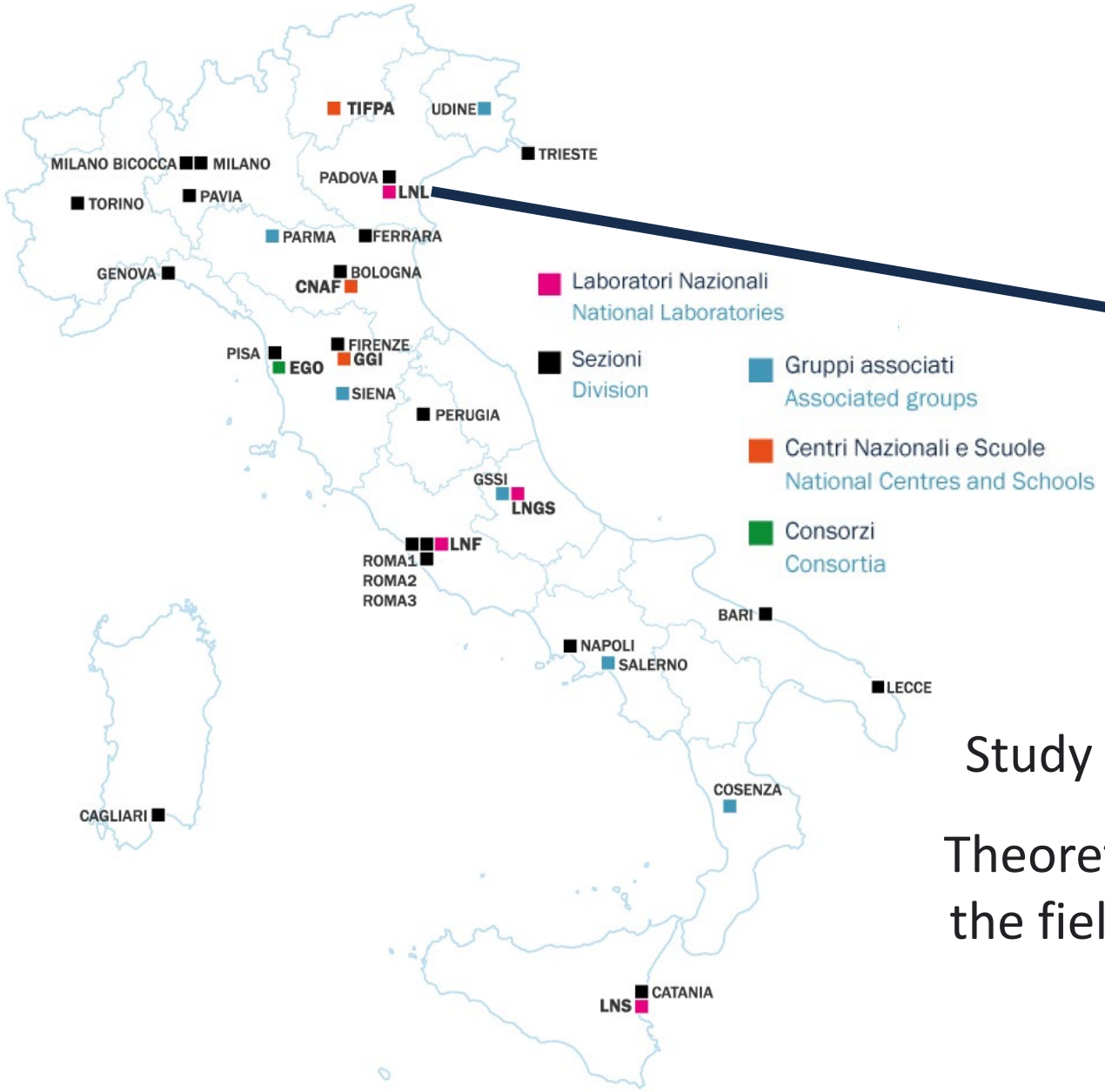
***PhD student in Materials Engineering**
DII University of Padova

***Research fellow**
INFN-LNL – LARAMED project

18th Workshop on Targetry and Target Chemistry (WTTC18)- Whistler, BC - August 22, 2022



INFN LNL
Istituto Nazionale di Fisica Nucleare
Laboratori Nazionali di Legnaro

MAIN activities:

Study of the fundamental constituents of matter

Theoretical and experimental research activities in the fields of subnuclear, nuclear, astroparticle and interdisciplinary physics

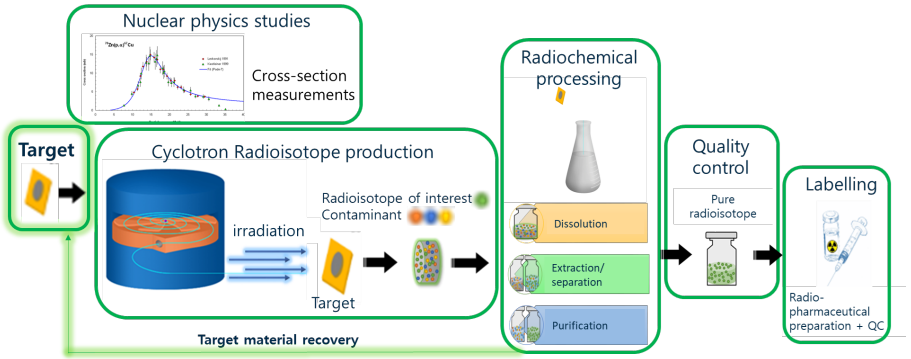
LARAMED project at INFN-LNL

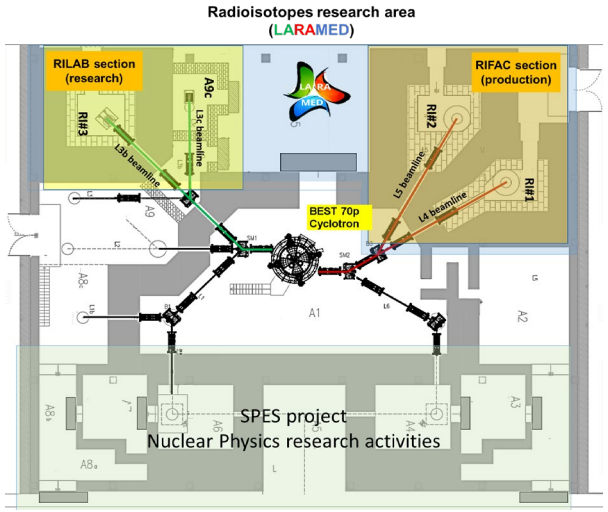
LARAMED project coordinator: J. Esposito

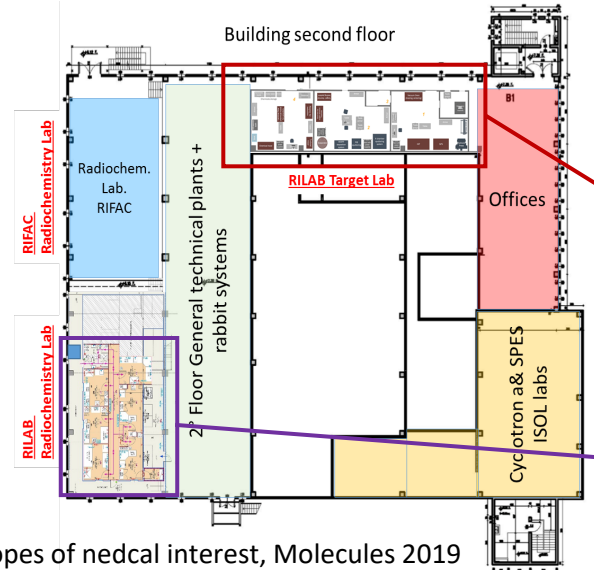
The BCSI 70p, p-cyclotron

LAboratory of RAdionuclides for MEDicine

Research and technological development for innovative radionuclide







RILAB chemistry / targetry labs

RILAB radiochemistry lab

Esposito et al., LARAMED: A Laboratory for Radioisotopes of nedcal interest, Molecules 2019

LARAMED project at INFN-LNL

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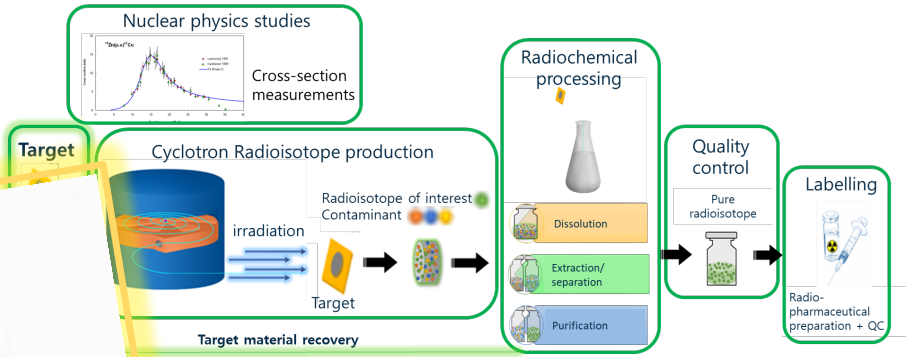

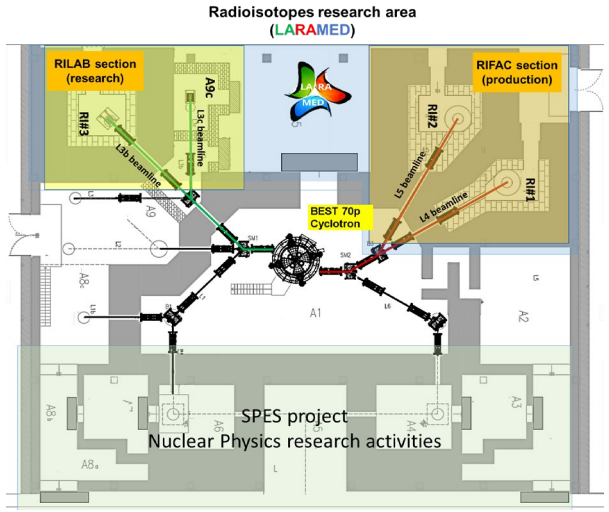

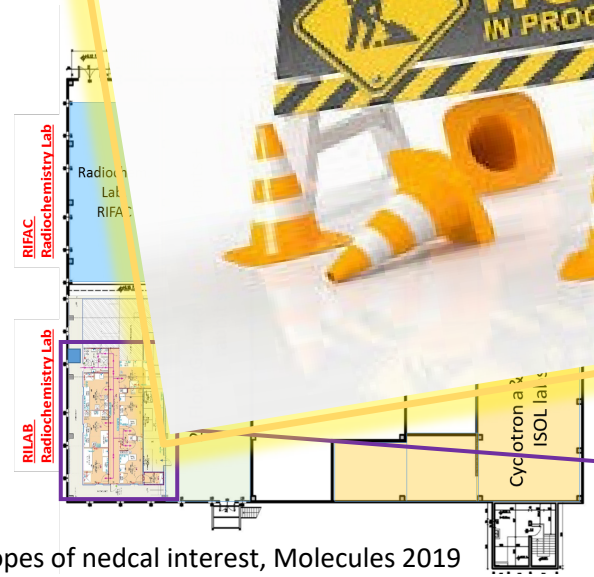

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RILAB radiochemistry lab

Esposito et al., LARAMED: A Laboratory for Radioisotopes of nedcal interest, Molecules 2019

LARAMED network

National collaborations

- Università degli Studi di Milano
- Consiglio Nazionale delle Ricerche
- INFN Pavia
- Università di Pavia
- Università degli Studi di Ferrara
- L.E.N.A.
- INFN Ferrara
- Università degli Studi di Padova
- INFN Padova
- Policlinico di Sant'Orsola
- Servizio Sanitario Regionale Emilia-Romagna, Azienda Ospedaliero - Universitaria di Bologna, IRCCS Istituto di Ricovero e Cura a Carattere Scientifico
- INFN Milano
- Sacro Cuore Don Calabria IRCCS

International collaborations

- Arronax Nantes
- IAEA
- UW-MADISON CYCLOTRON LAB
- PRISMAP Medical Radionuclides

LARAMED network

National collaborations

UNIVERSITÀ DEGLI STUDI DI MILANO

Consiglio Nazionale delle Ricerche

INFN PAVIA

UNIVERSITÀ DI PAVIA

INFN FERRARA

Università degli Studi di Ferrara

L.E.N.A.

INFN PADOVA

UNIVERSITÀ DEGLI STUDI DI PADOVA

POLICLINICO DI SANT'ORSOLA

SERVIZIO SANITARIO REGIONALE
EMILIA-ROMAGNA
Azienda Ospedaliero - Universitaria di Bologna
IRCCS Istituto di Ricovero e Cura a Carattere Scientifico

INFN MILANO

SACRO CUORE DON CALABRIA
IRCCS

TR19 cyclotron used for irradiations

Irradiation and radiochemistry steps

International collaborations

Arronax Nantes

IAEA

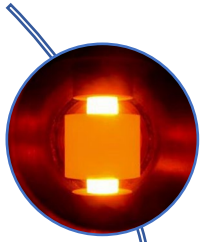
UW-MADISON CYCLOTRON LAB
Just another WiscWeb Wordpress Production Sites site

PRISMAP
Medical Radionuclides

R&D on innovative target manufacturing techniques

in the framework of the LARAMED project

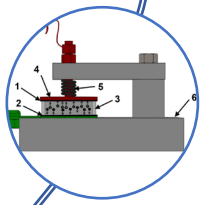
to overcome the limits of standard techniques



Spark Plasma Sintering

Magnetron Sputtering

Oral presentation by A. Kotliarenko – h.10:30



High Energy Vibrational Powder Plating

Oral presentation by S. Cisternino – h.9:40

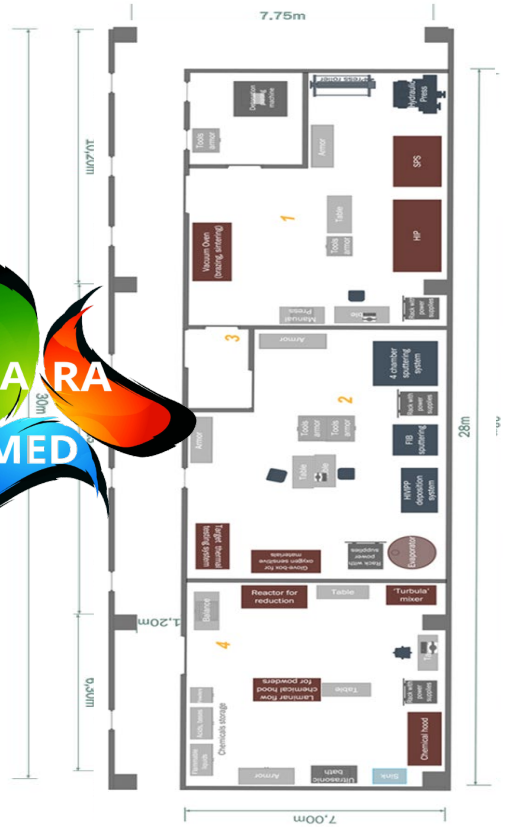
Thick targets for production

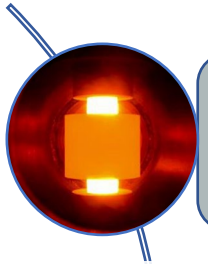
Thin targets for nuclear XS studies

R&D on innovative target manufacturing techniques

in the framework of the LARAMED project

to overcome the limits of standard techniques





Spark Plasma Sintering

Thick targets for production

Magnetron Sputtering

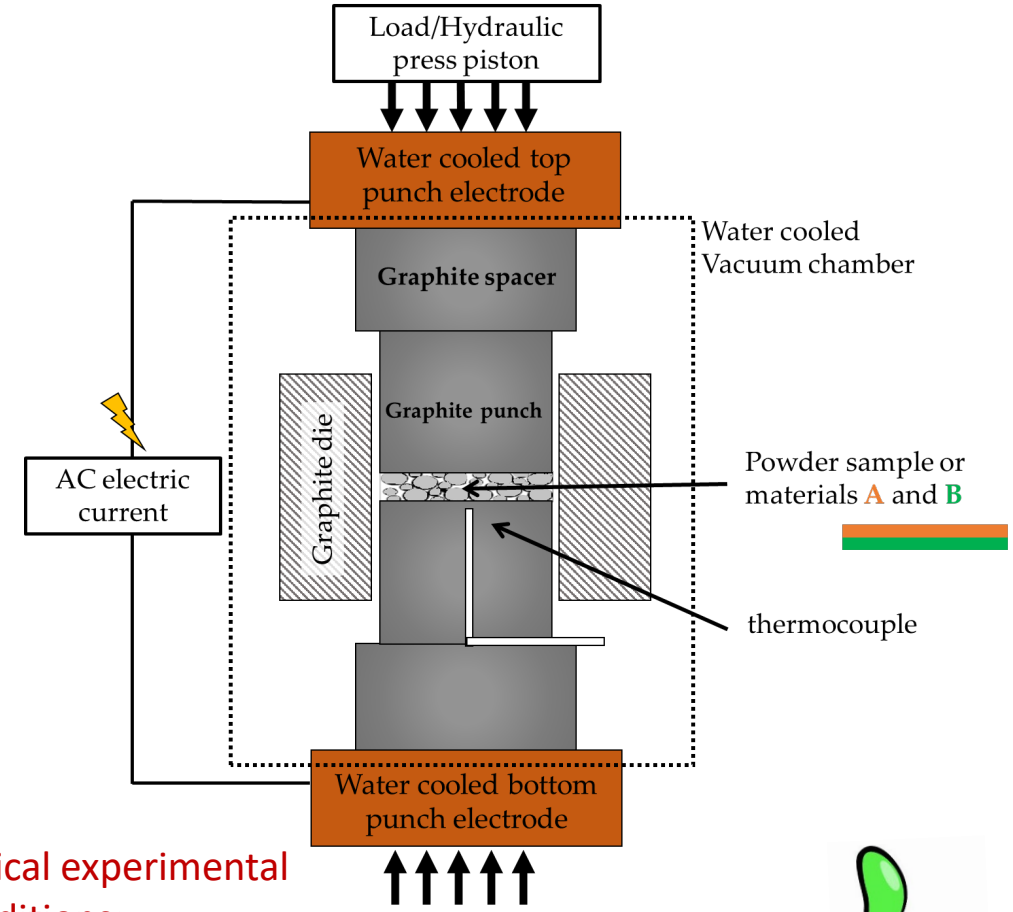
Oral presentation by A. Kotliarenko – h.10:30

High Energy Vibrational Powder Plating

Oral presentation by S. Cisternino – h.9:40

Thin targets for nuclear XS studies

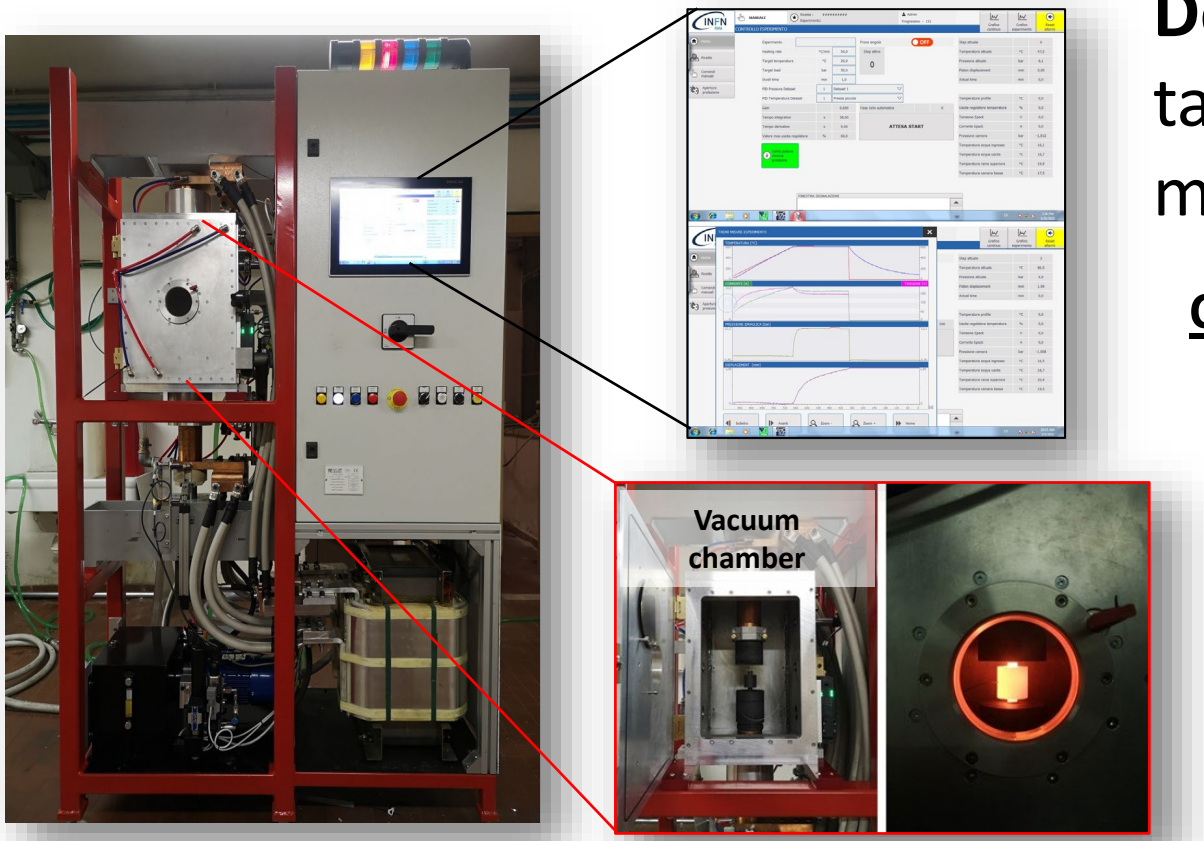
Spark Plasma Sintering (SPS) technique



Typical experimental conditions:
Voltage 5-10 V
Current intensity 10^3-10^4 A
Pressure 5-100 MPa

- ✓ **High heating rates (up to 1000 °C/min)**
- ✓ **Very short sintering times (min instead of hours)**
- ✓ Sintering of **high melting point materials (metal or oxide)**
- ✓ **99% efficiency→ No loss of isotope-enriched material** during manufacturing
- ✓ Starting materials→ **powder or foils**
- ✓ **Directly bonding of different materials→** (target and backing)
  - **Au protective thin layer** in between instead of costly Au backings
- ✓ 200 μm - mm thickness pellet→ targets for production

TT_Sinter SPS machine for LARAMED



Dedicated SPS machine

tailored for targets manufacturing for research

UNIVERSITÀ DI PAVIA

INFN Istituto Nazionale di Fisica Nucleare Sezione di Pavia

Components:

- water-cooled vacuum chamber
- vacuum system
- vertical single-axis hydraulic press
- specially designed water-cooled punch electrodes
- AC generator
- temperature and other sensors
- PLC controlling all the sintering parameters
- custom graphite punches and die

- ✓ Very fast process cycle
- ✓ Customizable tooling
- ✓ Easy to use and maintain
- ✓ Affordability

SPS technique…

…some examples

SPS Cr targets for ^{52}Mn production: different configurations

$^{52}Cr(p,n)^{52}Mn$

Target characteristics

Dr. SINTER® model SPS1050 machine of Sumitomo

Cr pellet: Ø10 mm x 410 µm; ρ_{Cr}~**90%** bulk
Au: Ø20 mm x 25 µm
Cu: Ø23.7 x 1.7 mm

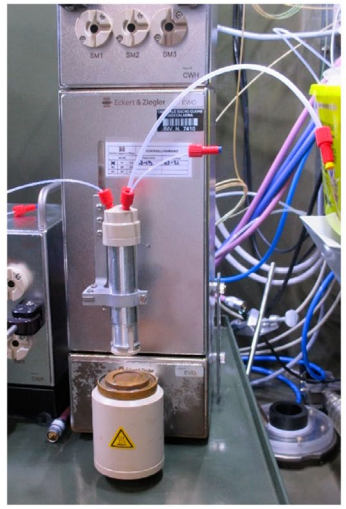
SPS prototype @ University of Pavia

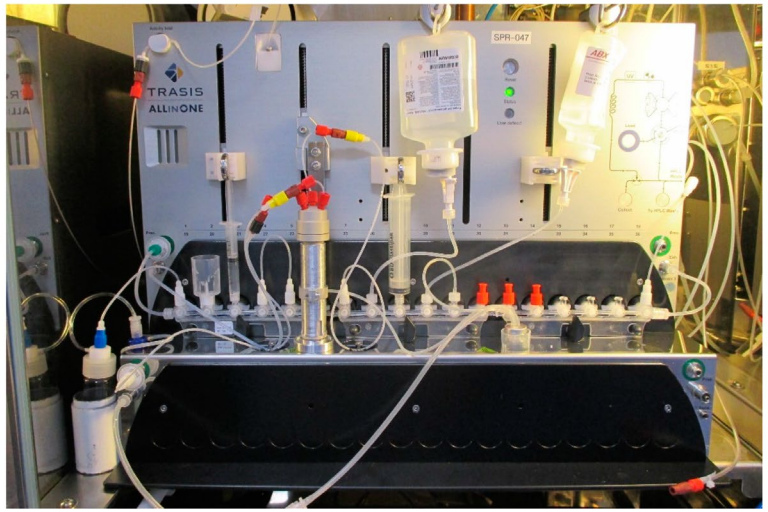
Cr pellet: Ø10 mm x 400 µm; ρ_{Cr}~ **50%** bulk
Nb: Ø23.5 x 1 mm

TT_Sinter machine (LARAMED)

Cr pellet: Ø10 mm x 430 µm; ρ_{Cr}~ **66%** bulk
Au: Ø20 mm x 25 µm
Nb: Ø23.5 x 1.6 mm

Dedicated dissolution system





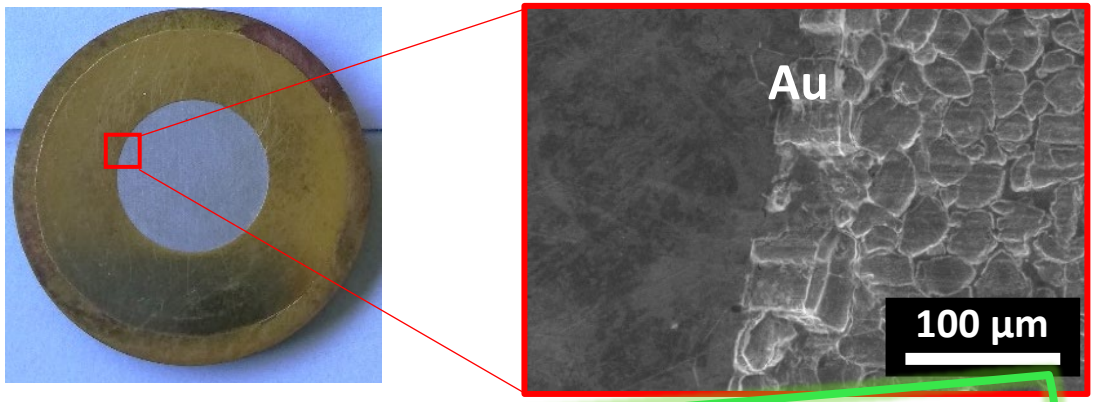
[Sciacca et al., Molecules 2021]
For more information contact G. Sciacca

SPS Cr target: Cr-Au-Cu

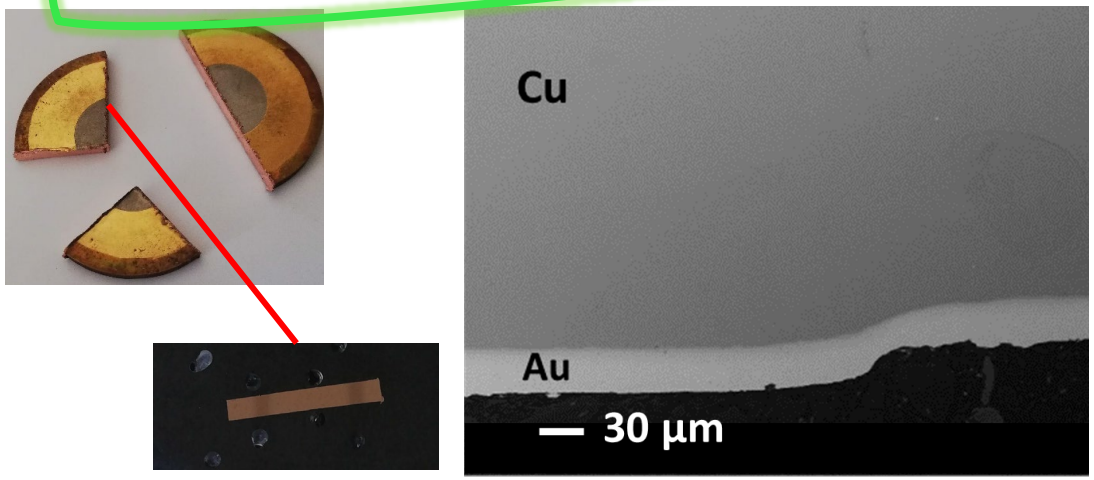
Target characteristics

Cr pellet: Ø10 mm x 410 µm; ρ_{Cr}~**90%** bulk
Au: Ø20 mm x 25 µm
Cu: Ø23.7 x 1.7 mm

Analysis after «cold» Cr dissolution



Au works as protective layer



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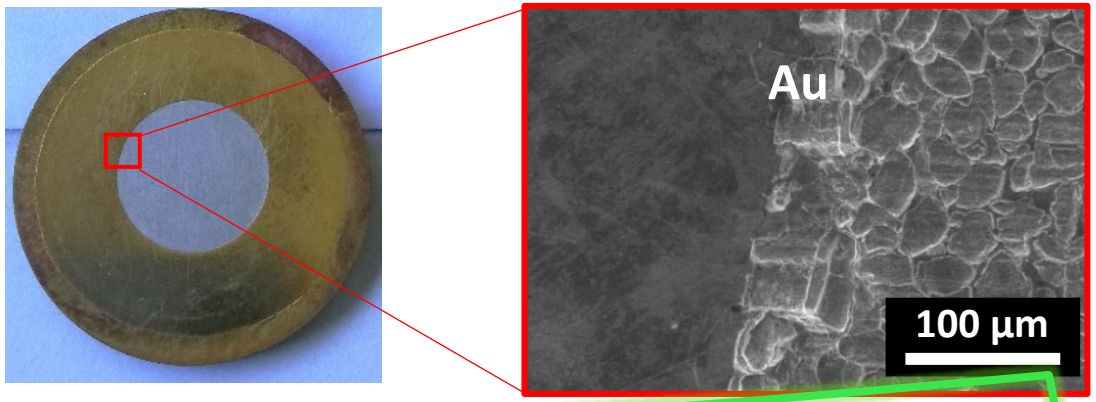
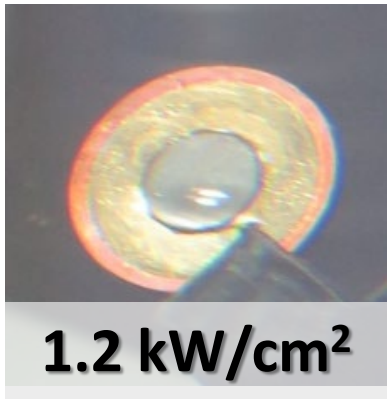
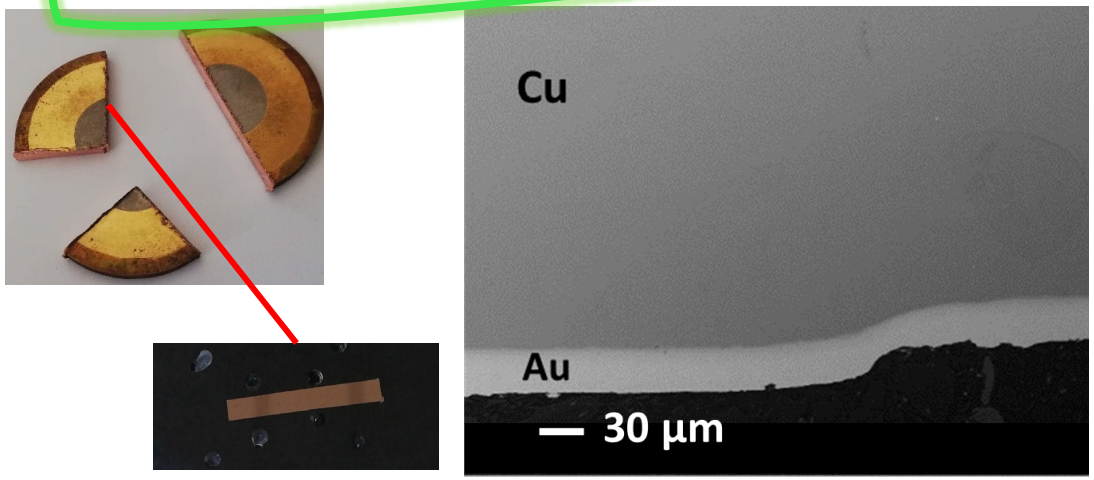


Au works as protective layer

Thermo-mechanical irradiations tests at maximum energy and current available



✓ Energy 19 MeV*,
p+ current 50 µA
10 min



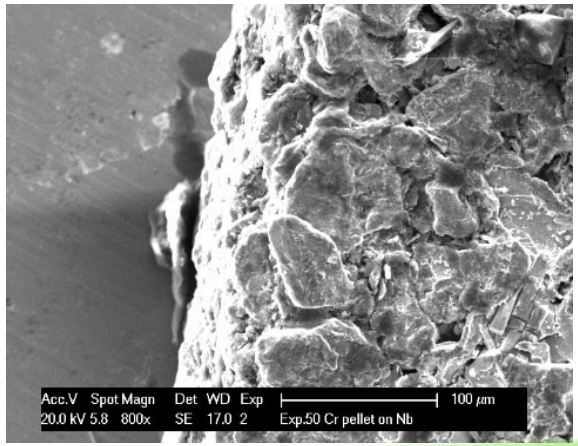
* This energy was used exclusively for the thermo-mechanical test

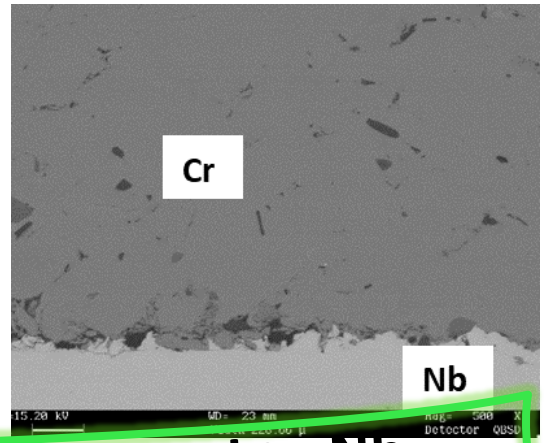
SPS Cr target: Cr-Nb

Target characteristics

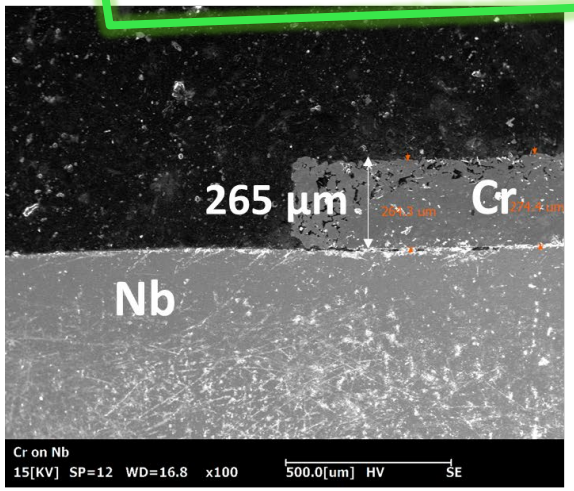
Cr pellet: Ø10 mm x 400 µm; ρ_{Cr}~ **50%** bulk
Nb: Ø23.5 x 1 mm

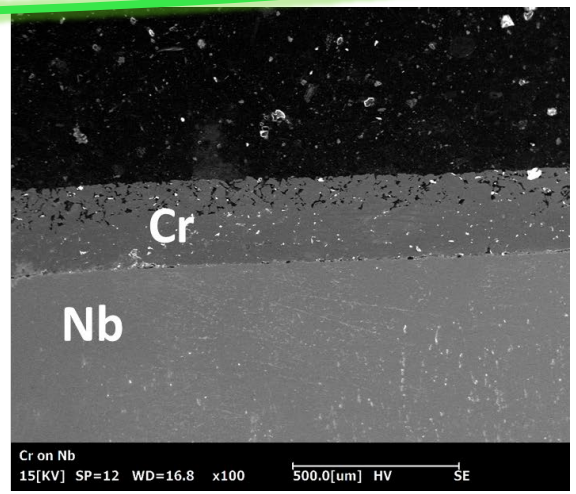
Metallurgical analysis





Cr is mechanically attached to Nb





SPS Cr target: Cr-Nb

Target characteristics

Cr pellet: Ø10 mm x 400 µm; ρ_{Cr}~ **50%** bulk
Nb: Ø23.5 x 1 mm

Thermo-mechanical irradiations tests at maximum energy and current available

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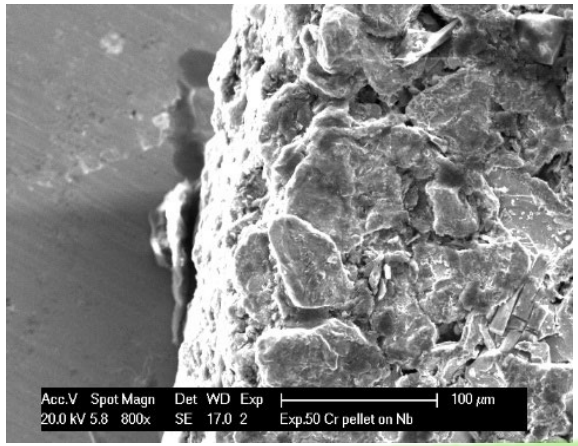
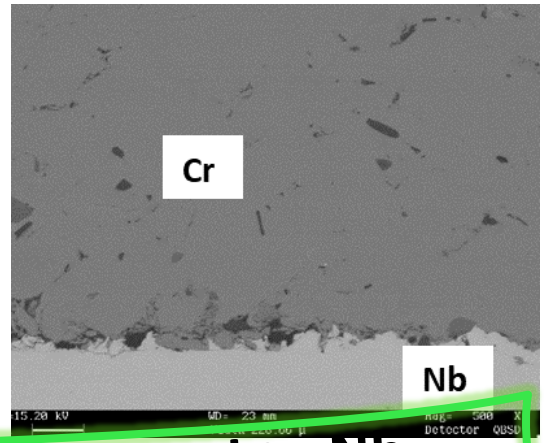
Metallurgical analysis





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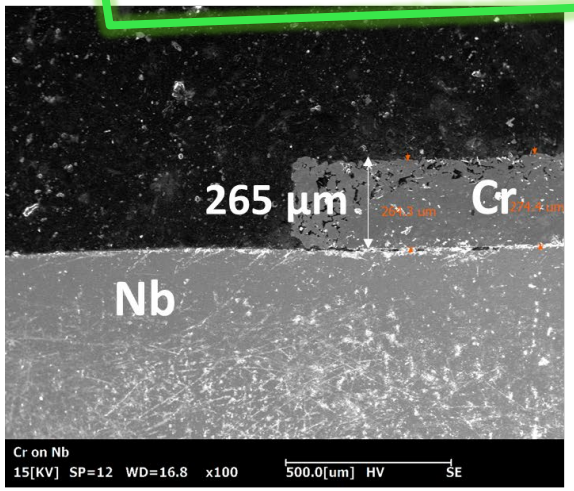
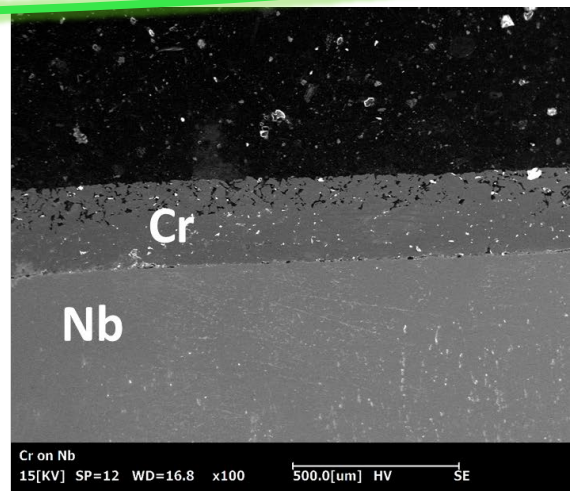


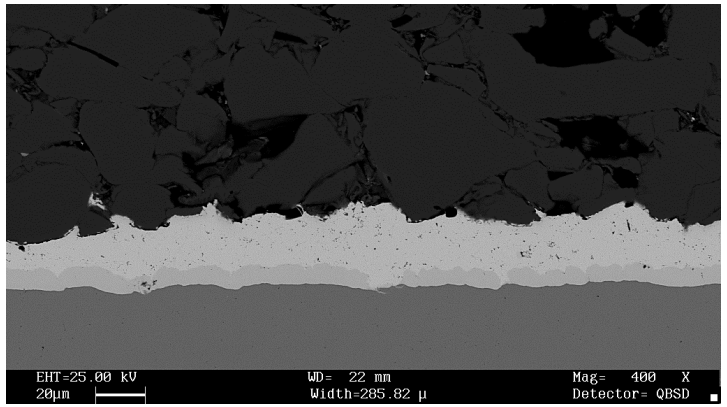


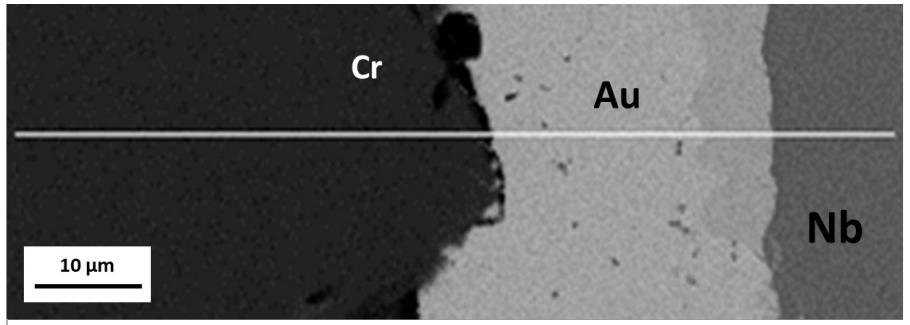
SPS Cr target: Cr-Au-Nb

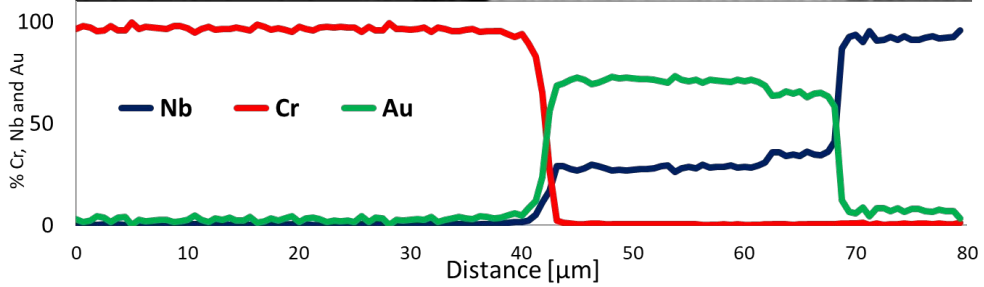
Target characteristics

Cr pellet: Ø10 mm x 430 µm; ρ_{Cr}~ **66%** bulk
Au: Ø20 mm x 25 µm
Nb: Ø23.5 x 1.6 mm

Metallurgical analysis







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Target characteristics

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Au: Ø20 mm x 25 µm
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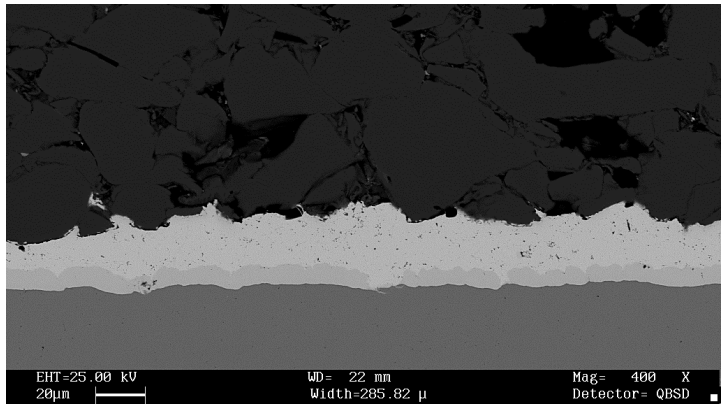

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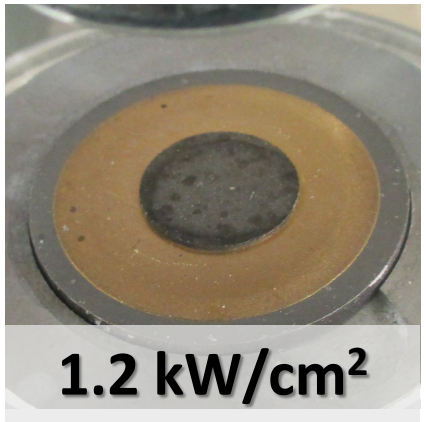

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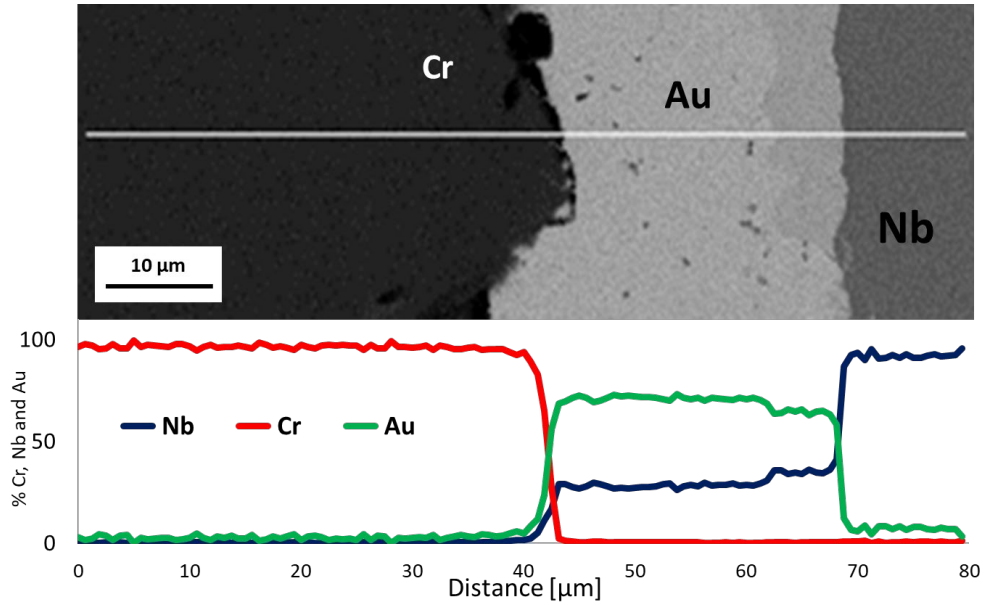
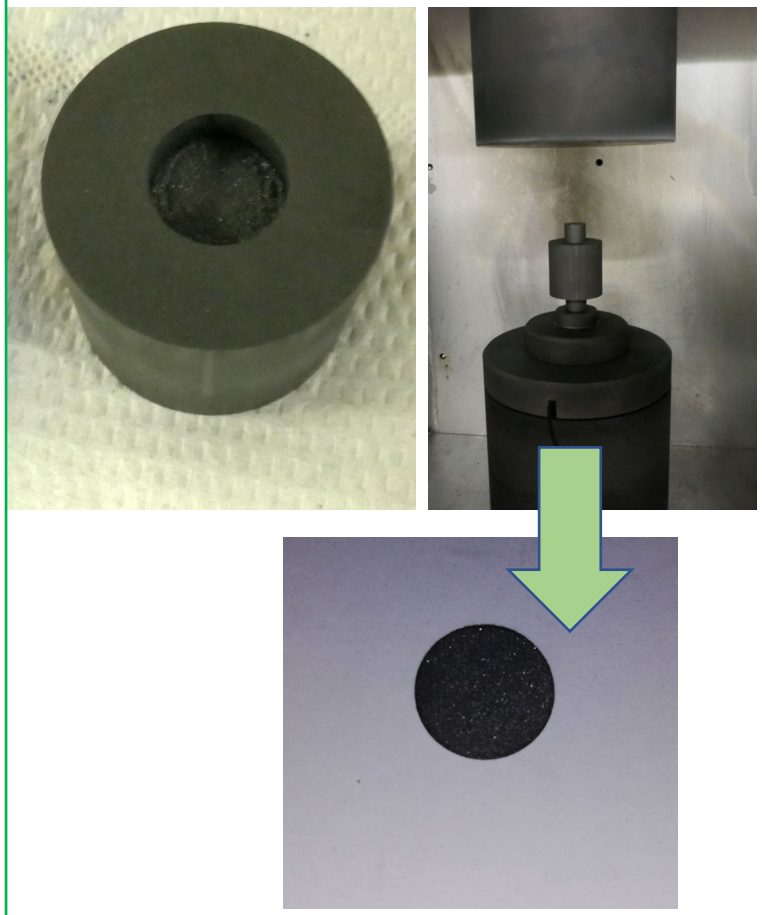
p+ current 50 µA
10 min



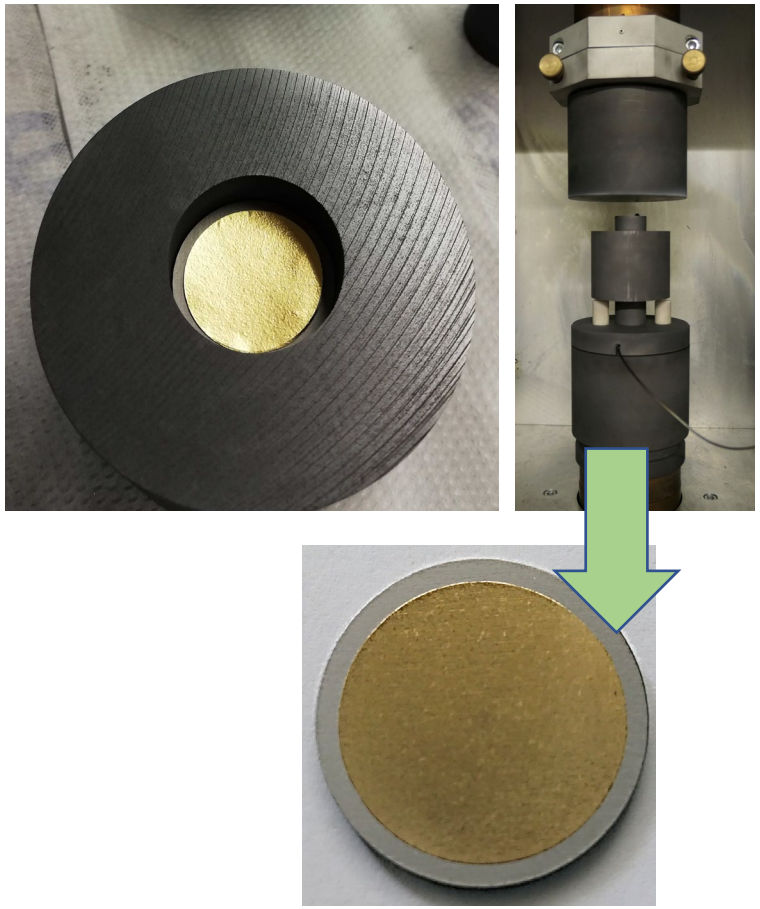
* This energy was used exclusively for the thermo-mechanical test

Typical SPS preparation steps

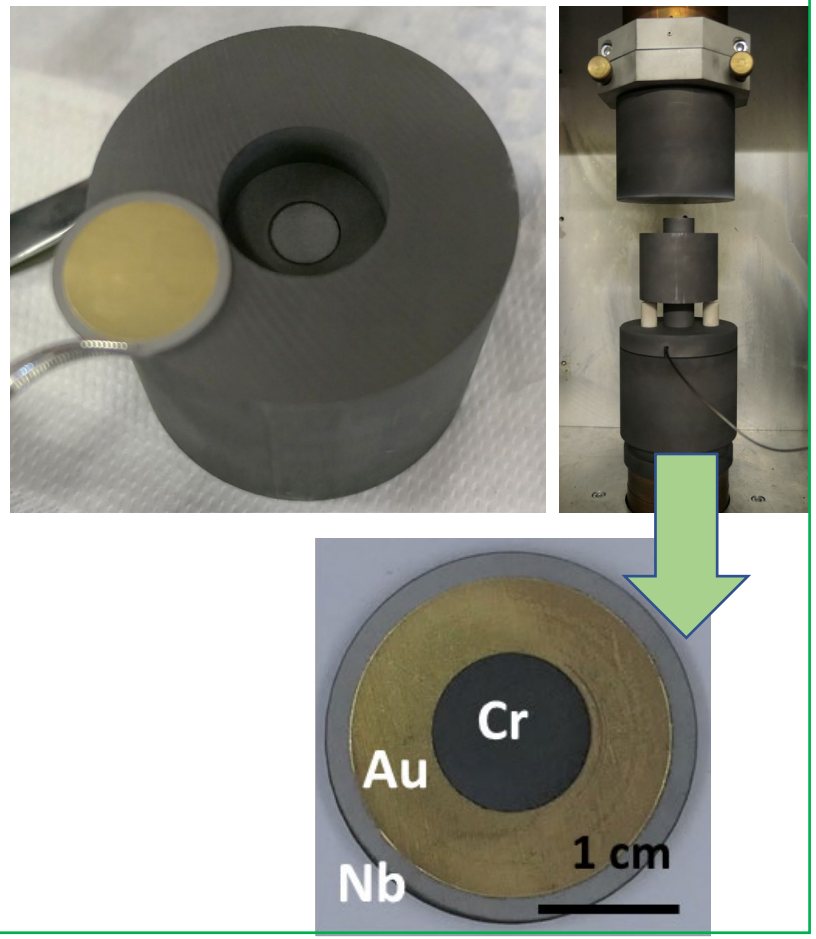
Pellet preparation starting from powder



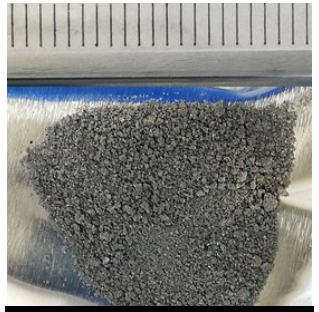
Support material preparation



Pellet-substrate bonding



Enriched ^{52}Cr targets



The smallest original powder

The biggest original powder

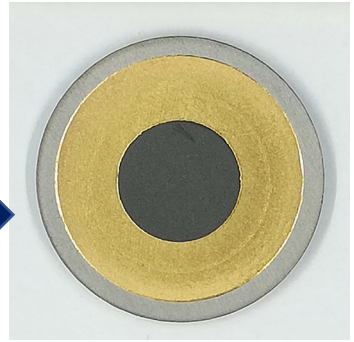
SPS

Cr pellet: Ø10 mm x 550 µm;
ρ_{Cr}~ ***50%*** bulk
Au: Ø20 mm x 25 µm
Nb: Ø23.5 x 1.6 mm

Cryomill

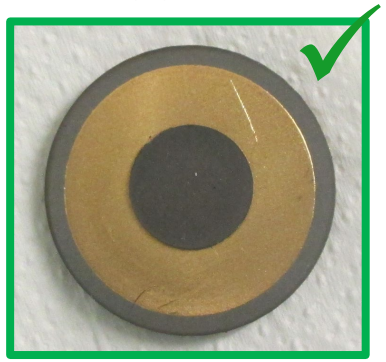
After milling

SPS



Cr pellet: Ø10 mm x 460 µm;
ρ_{Cr}~ ***60%*** bulk
Au: Ø20 mm x 25 µm
Nb: Ø23.5 x 1.6 mm

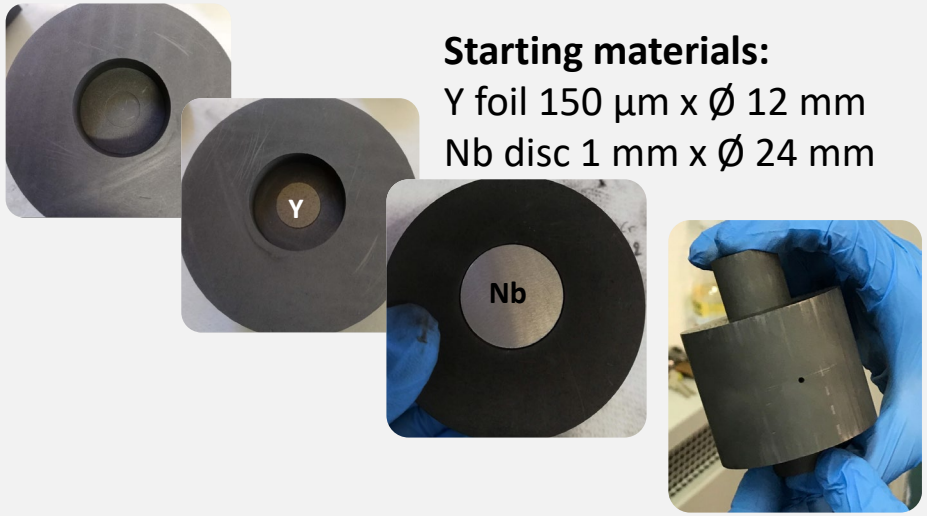
Irradiation at
16 MeV, **20 µA**
30 min



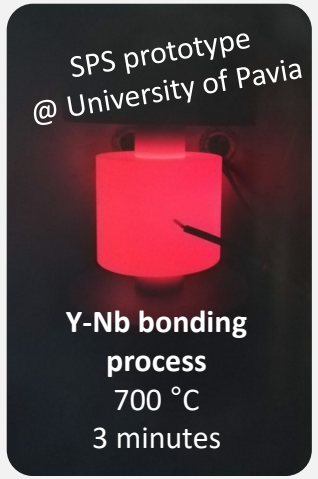
^{52}Mn yield and purity are under investigation

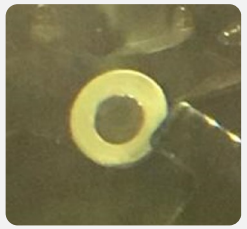
Y targets for ^{89}Zr production

$^{89}Y(p,n)^{89}Zr$

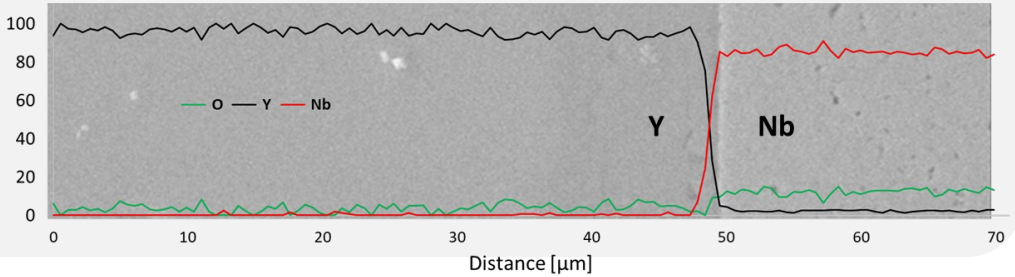


Starting materials:
Y foil 150 µm x Ø 12 mm
Nb disc 1 mm x Ø 24 mm

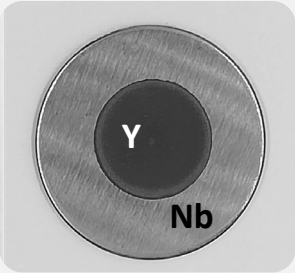




- ✓ Fast and efficient target manufacturing technique: 1 step
- ✓ Easy target handling with tele-pliers
- ✓ **Y-Nb high adhesion → improved heat dissipation efficiency to withstand higher currents**



SPS target



Y foil

Irradiation
12 MeV, 50 µA

Saturation yield [MBq/µAh]

14.12 ± 0.38 | 14.33 ± 0.14

^{89}Zr recovery

94 ± 4 % | 96 ± 0.14 %

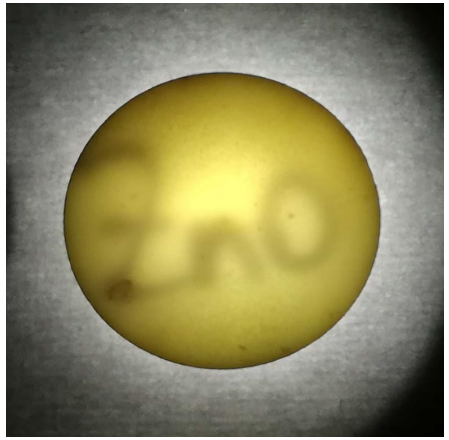
DFO-Trastuzumab labelling
Yield 100%

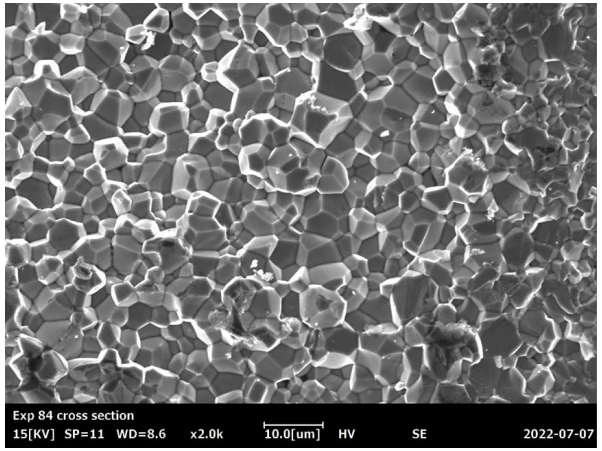
Cisternino et al., *Nuclear Medicine and Biology*, 2022

SPS ZnO targets for ^{67}Cu production: first feasibility study…

…in the framework of INTEFF_TOTEM project (2021-2022, PI S. Cisternino)

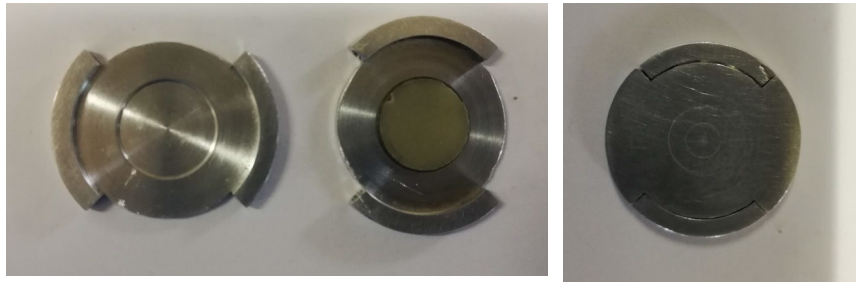
First pellets: Ø10 mm; thickness 600 µm; 100% density





	ZnO	Zn
Melting temperature [°C]	1975	419.5
Thermal conductivity [W/mK]	low	116

Pellet-holder Al prototype designed by G. Sciacca, inspired by Loveless et al. 2020



Future works and conclusions

SPS technology and TT_SINTER

LARAMED machine…

- Can be applied to different materials
- desired target material density
- negligible material losses
- fits the requirements for a suitable solid target

…towards systematic target supply for further studies

…for other emerging theranostic radionuclides ^{67}Cu ^{155}Tb

Thank you for the attention

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