HORIA – HULUBEI NATIONAL INSTITUTE FOR PHYSICS AND NUCLEAR ENGINEERING (IFIN-HH)

Isotopic thin films preparation laboratory for nuclear physics

DOMAINS OF ACTIVITY:
Thin-film fabrication technology

SCIENTIFIC & TECHNICAL TEAM:
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RESEARCH SERVICES:
Manufacturing and characterization of different naturally or stable enriched isotopic thin films for different types of nuclear physics experiments.

The laboratory was developed to support the nuclear physics experiments performed mainly at the IFIN-HH 9 MV Tandem Accelerator (see https://eeris.eu/ERIF-2000-000S-4963) or at other

international research facilities in the frame of scientific collaborations (CERN, IN2P3, TUM, IKP, JINR, etc).
The laboratory provides thin films of various thicknesses from several tens of μg/cm^2 to several hundreds of mg/cm^2 made from both naturally occurring materials and stable enriched isotopes. Such thin films (called “targets”) are dedicated for different types of nuclear structure experiments: nuclear structure and lifetimes measurements of excited nuclear states using γ-ray spectroscopy techniques (Doppler shift attenuation, plunger, and fast-timing), nuclear structure studies using the activation technique, cross-section measurements of interest for nuclear astrophysical processes, etc.
To assure good target quality the laboratory includes state-of-the-art equipment used for thin-film fabrication technology and consist of Physical Vapor Deposition (PVD) and mechanical rolling techniques.

RESEARCH EQUIPMENT:

Physical Vapor Deposition (PVD)

- used for creating self-supporting or backed targets with a wide range of thicknesses from several tens of μg/cm^2 to several hundreds of mg/cm^2.

1. TE18-High Vacuum Deposition System produced by Intercovamex Company

(A) TE18-High Vacuum Deposition System produced by Intercovamex Company;
(B) interior of the deposition chamber, showing: **(a)** resistive heating; **(b)** electron bombardment evaporation; **(c)** electron beam evaporation

TE18-High Vacuum Deposition System, is a physical vapor deposition device, used for creating thin films by resistive heating, electron beam and electron bombardment evaporation.
The system features a stainless steel cylindrical chamber (45 cm diameter and 53 cm high), placed on the top lid together with the rotary heater and the substrate. The heater of the system is specially configured to reach a maximum temperature of 700 °C. The chamber includes a frontal 4-inch viewport that is placed in an ISO-KF DN100 flange. The chamber has a motorized hoist to raise and lower the top lid and the vacuum chamber.
The setup is equipped with a high-vacuum system capable of pumping down the chamber to a pressure level below 3.5E-5 Torr in < 1 h.
The system has also one resistive evaporation kit with a 3.6 kW power supply (6 V and 600 A) for evaporation of materials with melting points not higher than 1800 °C.
For making targets of high melting point materials (up to 3800 °C) and dielectrics, the system is equipped with an electron bombardment evaporation system and a 4-pocket 9 x 2 cc volume rotary water-cooled copper crucible which allows sequential evaporation of up to 4 materials. The electron bombardment evaporation system has a 270° deflection angle, 5 kV acceleration voltage and 600 mA maximum current. The system is also equipped with a XY sweep controller.

An electron beam evaporation system produced by Mantis Deposition, was also installed in the vacuum deposition chamber of the device. The system is equipped with a QUAD-EV-C source with a power supply of 500 W that can be used to heat the target material to temperatures up to 3000 °C. The target rod or crucible is located close to the W filament. When sufficient current is passed through the filament, the electron emission temperature is reached and a stream of electrons is drawn from the filament towards the rod or crucible.

For monitoring the thickness of the deposited material and the rate of deposition, the system has an Inficon SQM160 Thin Film Deposition Monitor.

2. Quorum Technologies E6700 Bench Top Evaporator

This system is equipped with power supplies for both carbon and metal evaporation. It includes sputtering, glow discharge, low angle shadowing, and film thickness monitoring. The work chamber with a diameter of 30 cm can be evacuated to less than 10^{-5} mbar. In standard operation we apply up to 10 V at 200 A for carbon coating and 20 V at 100 A for metal evaporation.

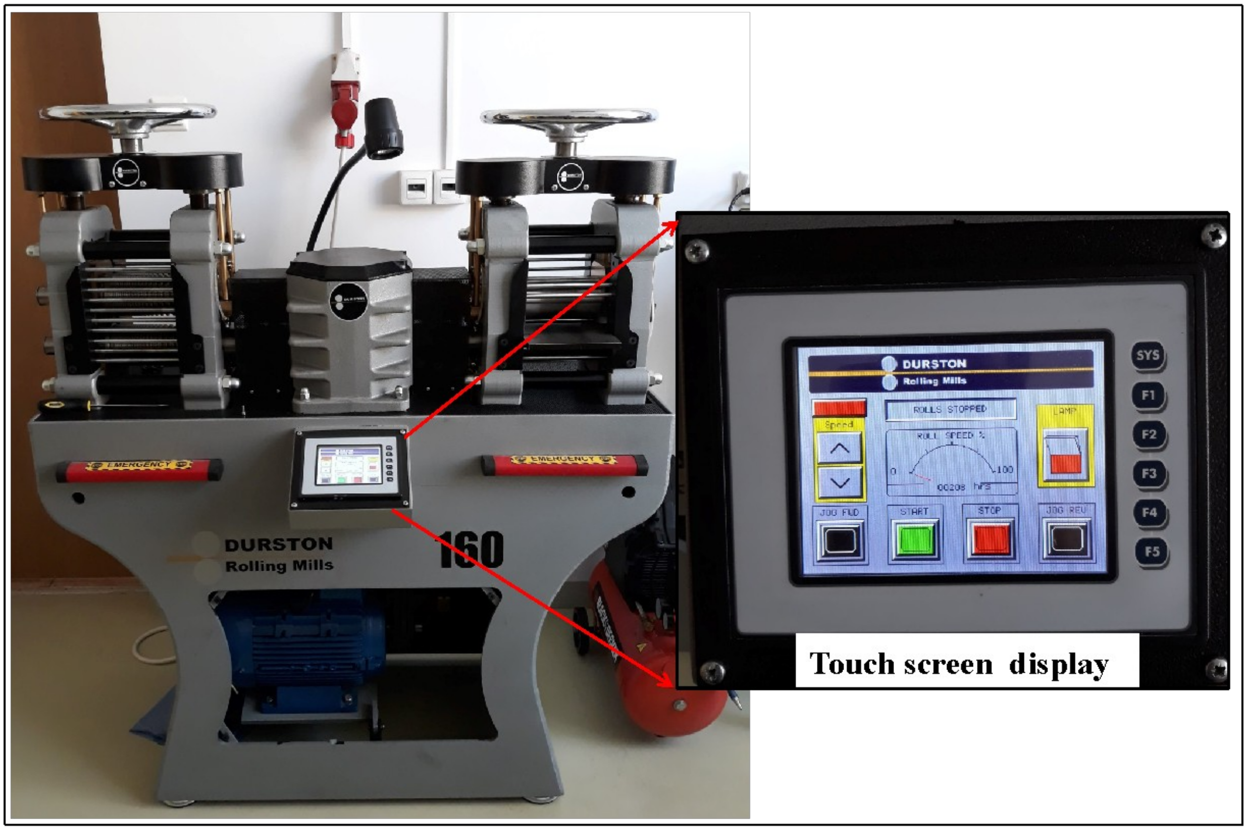
Mechanical rolling technique

- extremely efficient for self-supported metallic foil production using small amounts of starting material. It allows preparation of metallic foils with areal densities ranging from several hundreds of $\mu g/cm^2$ to hundreds of mg/cm^2.

1. Electrically controlled Durston type DRM 100 rolling mill

Electrically controlled Durston type DRM 100 rolling mill with variable speed. The rolls have a size of 100 x 50 mm and are made of specially selected roll steel, heat-treated on the main working surface.

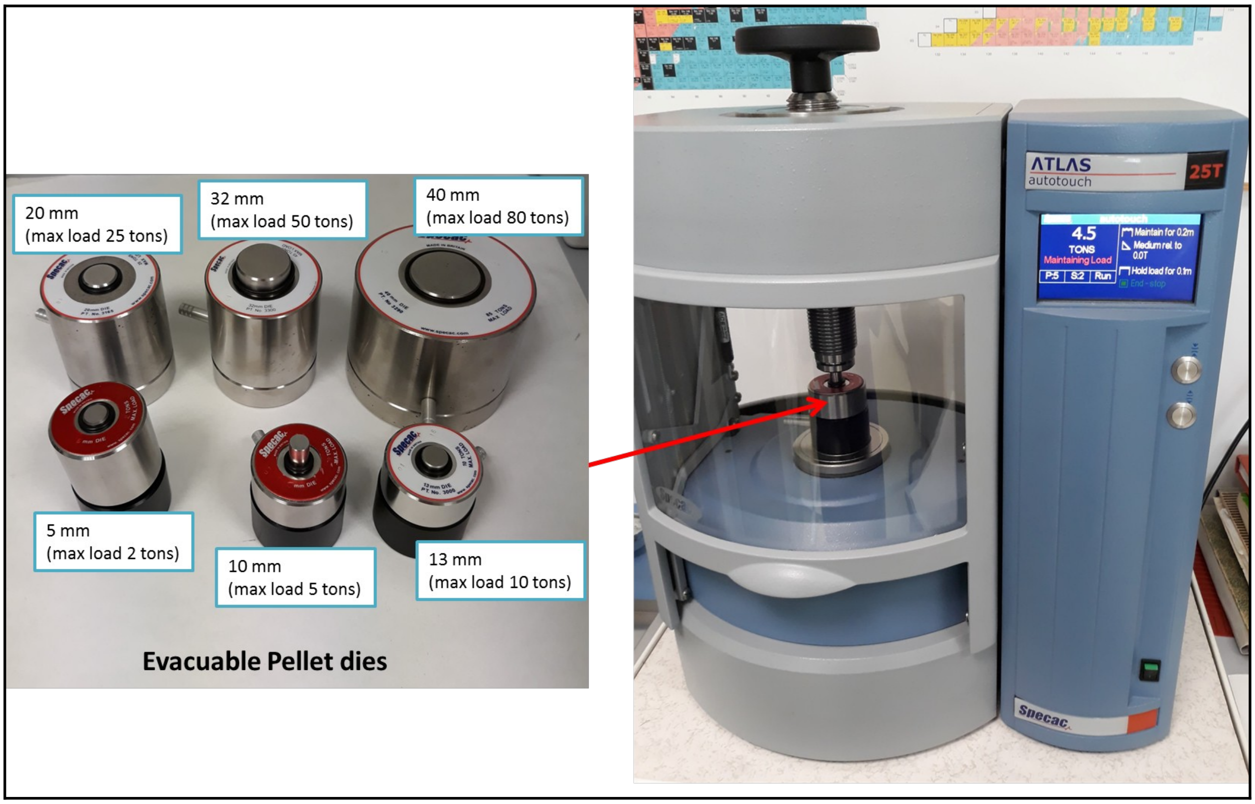
2. Electrically controlled Durston type FSM 160 VCC power rolling mill



It is a more powerful, double sided, electrically controlled and with touch screen display Durston type FSM 160 VCC rolling mill with variable speed (5-20 rpm). It is a 3 phase voltage, floor standing system with a 4kW electric motor power equipped with 160 x 86 mm rolls, made of specially selected roll steel, heat-treated on the main working surface. It has a maximum roll gap opening of 25 mm and the lubrications to roll bearings is automated.

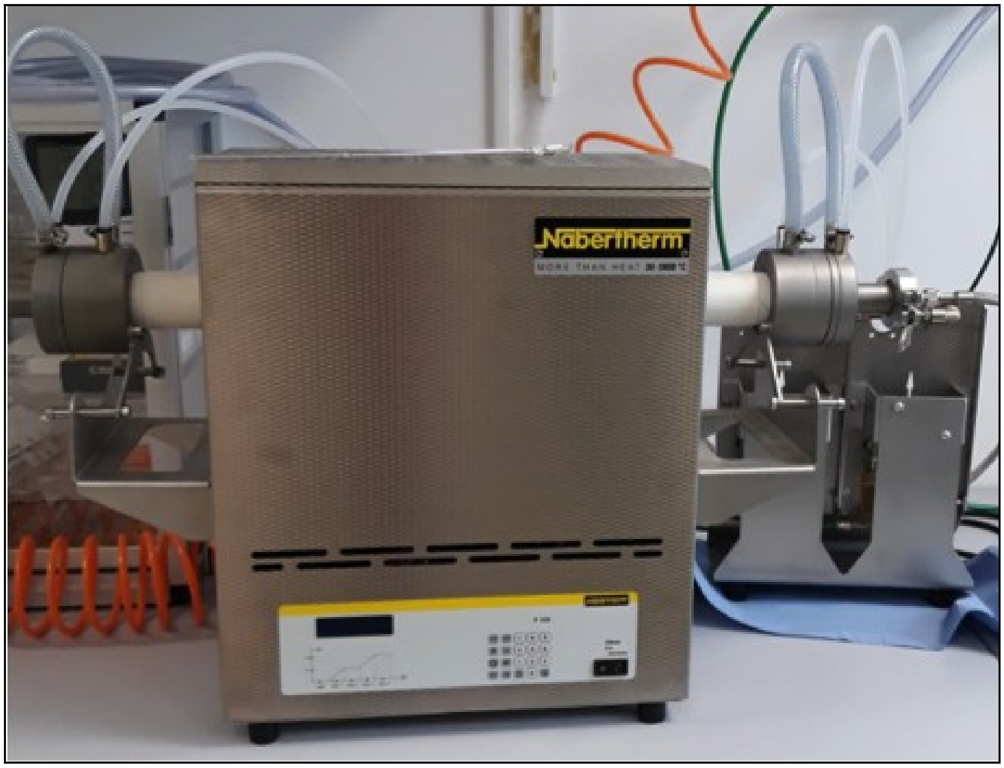
Auxiliary equipment is also used to provide support for the numerous other activities required for target fabrication.

1. Atlas™ Automatic 25Ton (25T) Hydraulic Presses equipped with 40 mm, 32 mm, 20 mm, 13 mm, 20 mm and 5 mm evacuable pellet dies, produced by Specac. All the parts of the die are manufactured from hardened stainless steel for optimum quality and durability, and highly polished surfaces for contact with the sample. The applied pressure and release are well controlled. The LCD display show press status and load conditions giving a digital display of the load applied. The hydraulic press is mainly used for producing self-supported pellets, which can be further used in experiments as thick targets.



2. Mettler Toledo XP56/M analytical balance, offers a weighing capacity of 52 g with 0.001 mg readability.The weighing method is very important in the determination of the areal density of foils or deposited targets, combined with the precise surface measuring using a digital optical microscope (Dino-Lite Pro Digital Microscope AM-413TL).

3. Nabertherm tube furnance equipped with a gas supply systems for protective gas or vacuum operation which can be operated at temperatures up to 1300 °C is used mainly for chemical processing of isotopic material.



4. Ohaus Micro Centrifuges, FC5515R 230V - for high-speed laboratory applications as ultrafiltration.

Speed Range: 200 rpm – 15200 rpm;
Maximum Relative Centrifuge Force (x g): 21,953 g;
Maximum Capacity (Rotor): 44 x 1.5 / 2.0 ml; 12 x 5 ml;
Refrigeration Range: - 4 °F – 104 °F (-20 °C – 40 °C).

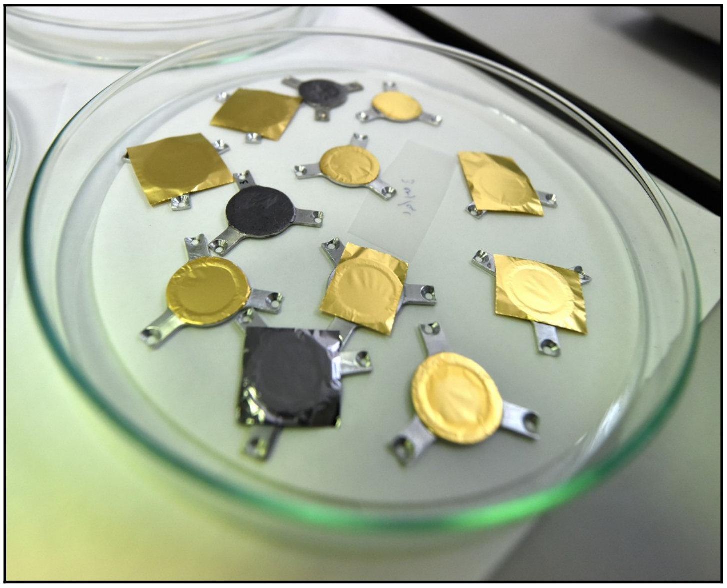
5. Ohaus Incubating Orbital Thermal Shaker, ISTHBLHTS - for heating applications that require consistent and precise high-speed shaking .

Motion: Orbital, 3 mm;
Temperature Range: Ambient +4 °C – 100 °C;
Speed Range: 300 rpm – 3000 rpm.

Both equipment (**Ohaus Micro Centrifuges, FC5515R 230V and Ohaus Incubating Orbital Thermal Shaker, ISTHBLHTS)** are used for obtaining high purity isotopes in elemental metallic form. In metallothermic reduction reactions of different oxides with dedicated reducing agents (Zr, Hf, La) the final reaction product is a mixture of an isotope used further for the target preparation and a chemical compound that should be completely removed without loosing the isotope. The most efficient method used to separate the particles from a solution is centrifugation.

An **inert-gas glove box (Labconco)** is also available for handling of targets that may oxidize quickly.

TARGET DEVELOPMENT



In the last years, different approaches and experimental protocols were applied for the preparation of high quality isotopic thin films with selected properties that perfectly answer nuclear physics experiments requirements.

Some examples: Self-supported ^{13}C solid thick targets (400 mg/cm^2 thickness and 2 cm diameter) using amorphous ^{3}C powder and also N^{14} target with similar geometry features using $LiNH_2$ compound, for IKP Poland were prepared by pressing using a hydraulic 25T press. Another methods involves metallothermic reduction reaction of different oxides with dedicated reducing agents (Zr, Hf, La, CaH_2, Mg, C, H_2), which subsequently leads to obtaining of high purity metallic form of the deposited thin film ($^{144,147,149,152,154}Sm_2O_3$, $^{140}CeO_2$, $^{46}TiO_2$, $^{30}SiO_2$, $121Sb_2O_5$, $^{72,73}GeO_2$, etc.). Reliable thin films made of ^{nat}Ca, ^{nat}Ba, ^{nat}Sr, ^{24}Mg in metal form that can resists oxidation by air were also produced.

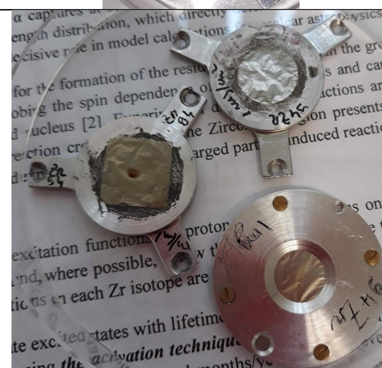
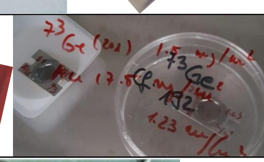
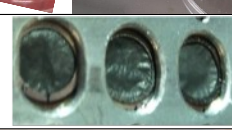
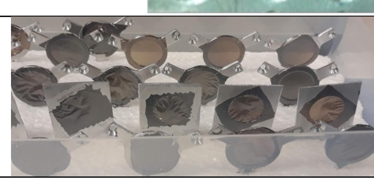
To obtain durable ^{82}Se layers that preserve their physical and chemical properties while irradiated with intense accelerated particle beams, a thermal treatment to convert the condensed ^{82}Se layers that exhibit unstable amorphous structure to hexagonal crystal structure was performed. This is very important because an intense fast heating of Se films with amorphous structure rapidly induces the conversion to hexagonal crystal structure and so the obtained foils shrink and tend to crack or curl.

To remove the oxygen layer (as contamination) from the surface of different metallic isotopic foils (^{64}Ni, ^{65}Cu, ^{208}Pb) resulted during the fabrication process a thermal treatment using a hydrogen oven was successfully applied. The process of removing the surface oxygen contamination is very useful for sub-barrier neutron transfer reactions with ^{18}O or ^{13}C beams.

To produce reliable thick targets from refractory materials as Os, W with extremely high vaporization temperatures (4500-5500°C) using Pulsed Lased Deposition (PLD) technique was also developed in collaboration with the National Institute for Laser, Plasma and Radiation Physics (INFLPR).

Other self-supported or backed targets were also produced through high vacuum evaporation methods or by mechanical rolling: ^{130}Te, $^{182,184}W$, Au, Ta, Y, Nb, Sn, etc.

Examples of isotopic targets for experiments performed at the Tandem 9MV

Isotopic targets	
^{58}Ni (1 mg/cm^2) **^{58}Ni (0.3 mg/cm^2)**	
^{46}Ti (0.4 mg/cm^2) / Au (2mg/cm^2)	
^{94}Zr (8 mg/cm^2)	
^{30}Si (0.2 mg/cm^2) / Ti (3 mg/cm^2) **^{30}Si (0.2 mg/cm^2) / Ta (13 mg/cm^2)** **^{30}Si (117 mg/m^2)**	 
$^{72,73}Ge$ (1.6 mg/cm^2) / Au (8 mg/cm^2) **^{74}As (500 mg/cm^2)**	 
$^{182,184}W$ (0.2 mg/cm^2) / C (0,03 mg/cm^2)	
^{13}C (200 mg/cm^2) **^{13}C (0.5 mg/cm^2)**	 
$Li^{14}NH_2$ (200 mg/cm^2)	

$^{142,143}Nd_2O_3$ (0.25 mg/cm^2) / C (0.03 mg/cm^2) **$^{144,154}Sm_2O_3$ (0.25 mg/cm^2) / C (0.03 mg/cm^2)**	

ARTICLES:

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NATIONAL PROJECT:

Romania’s solution to the global crisis of Technetium-99m: Method development for 100Mo solid targets to use in high current cyclotron production of medical 99mTc radionuclides; PN-III-P1-1.1-TE-2019-0337.