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Development of a pulsed laser deposition system suitable for radioactive thin films growth

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Astract: Radioactive thin films have a direct application in the development of beta-voltaic batteries. The main advantage of that kind of nuclear battery is its durability, which can range from a hundred years, depending on the half-life of the radioisotope used. In this context, Pulsed Laser Deposition (PLD) is an important tool. A relevant aspect of a system using this technique is that the main equipment is outside the chamber where the material is processed. Consequently, this feature allows the growth of radioactive thin films, as it enables the development of an arrangement where the contaminated area is controlled. In this way, the present work proposed the development of a PLD system for the growth of radioactive thin films. The PLD system was then implemented and radioactive copper targets were processed for 60 min and 120 min, resulting in radioactive thin films with an average thickness of (167.8 ± 3.7) nm and (313.5 ± 9.2) nm, respectively. Then, a study was performed about the radioactive contamination spread in the PLD system in order to prove if the filtering implemented was effective in retaining the contamination inside the vacuum chamber. Thus, it is demonstrated for the first time the feasibility of using the PLD technique in the growth of radioactive thin films, making its use possible in future studies on the development of beta-voltaic nuclear batteries.

Keywords: laser ablation, pulsed laser deposition, thin film, nanosecond laser, radioactive target.

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Desenvolvimento de um sistema de deposição por laser pulsado para o crescimento de filmes finos radioativos

Resumo: Os filmes finos radioativos desempenham um papel crucial no avanço das baterias beta-voltaicas. A principal vantagem dessas baterias nucleares reside em sua durabilidade, podendo se estender por até cem anos, dependendo da meia-vida do radioisótopo utilizado. Nesse contexto, a Deposição por Laser Pulsado (PLD) é uma ferramenta importante. Um aspecto significativo de um sistema que emprega essa técnica é que os equipamentos principais ficam fora da câmara onde o material radioativo é processado. Consequentemente, essa característica viabiliza a criação de um arranjo em que a área contaminada é estritamente controlada, permitindo o crescimento de filmes finos radioativos. Assim, este trabalho propôs o desenvolvimento de um sistema PLD dedicado ao crescimento de filmes finos radioativos. O sistema PLD foi implementado com sucesso e alvos de cobre radioativos foram processados por 60 minutos e 120 minutos, resultando em filmes finos radioativos com espessura média de (167,8 ± 3,7) nm e (313,5 ± 9,2) nm, respectivamente. Em seguida, conduziu-se um estudo minucioso sobre a propagação da contaminação radioativa no sistema PLD, visando comprovar a eficácia do filtro implementado em reter a contaminação dentro da câmara de vácuo. Este estudo apresenta, pela primeira vez, a viabilidade do uso da técnica PLD no crescimento de filmes finos radioativos, abrindo caminho para futuras pesquisas no desenvolvimento de baterias nucleares beta-voltaicas.

Palavras-chave: ablação a laser, deposição por laser pulsado, filme fino, laser de nanossegundos, alvo radioativo.

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1. INTRODUCTION

The constant technological development achieved in the last decade has led to the miniaturization of various components and electronic devices. As a consequence, there is an increasing demand for batteries that provide significant energy densities and are also small, light, reliable, and autonomous [1, 2]. For specific applications, such as remote monitoring, radars, satellites, spacecraft, and deep-water probes, the impossibility of recharging the power supply device is a factor that makes it difficult or even impossible to use traditional chemical batteries [3-6].

Some of these applications resort to radionuclides as energy storage media. Since there are a great variety of isotopes, each with unique decaying properties, it is possible to tailor-make energy sources [7] with energy densities hundreds of times greater than traditional batteries and longevity of many decades: they are known as nuclear batteries or radioisotope generators.

A portion of these energy sources are thermally driven while others are denominated as "direct conversion", which uses a solid-state device such as a PN junction, PIN junction, or Schottky barrier, as well as a thin film (layer) of an alpha or beta (-) emitter, for the generation of electric current [5, 8, 9]. These are called alphavoltaic or betavoltaic batteries, depending on the emitted particle in the radioisotope decay. One that is constantly requested in remote applications is the battery based on ^{63}Ni, which has a life cycle of hundreds of years [10].

Direct conversion nuclear battery is composed basically of a thin film of a beta (-) or alpha emitter, which is deposited on the surface of a semiconductor device so that the radiation when penetrating the semiconductor promotes the generation of electric current [11, 12].

A radioactive thin film is an appropriate definition of the layer used in such batteries. In general, the thickness of these films can be on the micrometer or nanometer scale. Thin films can be produced with the deposition of a material on a specific surface (substrate),

using several methods, such as Electrodeposition [13], Sputtering [14], Electron-beam Physical Vapor Deposition (EB-PVD) [15], and Pulsed Laser Deposition (PLD) [16].

Although there is a wide variety of techniques aimed at the growth of conventional thin films, when it comes to the growth of radioactive thin films, there is a certain shortage, since this process can very easily contaminate the equipment involved. In the PLD technique, a laser beam can be propagated through an optical window into an airtight chamber, where the growth of the film can be done and only the interior of the chamber is exposed to the radioactive material, freeing the rest of the equipment contamination. It is worth mentioning that the use of this technique for the growth of radioactive thin films is something that has not been reported in the literature.

The advantage of growing a film from a radioactive target, instead of the other way around (activating the grown thin film by neutron irradiation), comes from the fact that for a betavoltaic type battery, the semiconductor would be damaged in the process.

In order to be able to grow radioactive films to test betavoltaic batteries the objective of the present work [17] is to design and test a PLD aimed to produce radioactive thin films.

2. PLD SYSTEM DESIGN

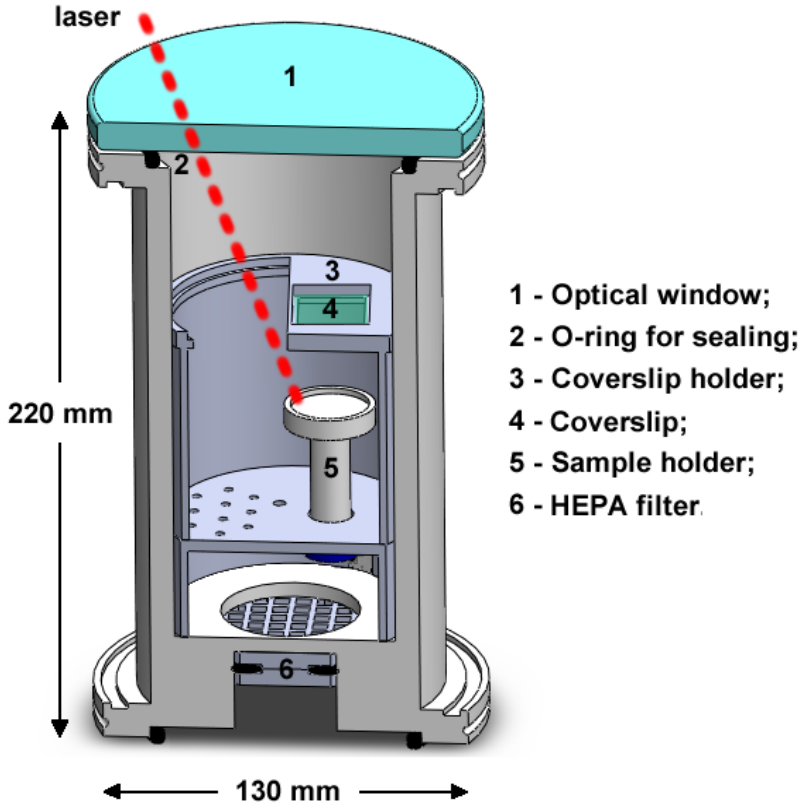
Some considerations were assumed as constraints in the design concept: 1) parts needed to be low cost and disposable; 2) compatible with vacuum piping market standards; 3) easy to produce.

The vacuum chamber served as the basis for the conception of the system. To facilitate its production, it was conceived as having a cylindrical form, so it could be lathed. The dimensions of the chamber were defined according to the space required to accommodate the components necessary for the growth of thin films, in addition to the need for a minimum space for the angular incidence of the laser beam on the target surface. Therefore,

it was chosen to engineer a chamber that was compatible with the ISO-100 standard of vacuum equipment.

The PLD chamber was designed as the scheme shown in Figure 1. An optical window (1) made of flint glass was used to allow the laser to enter; (2) O-rings were used to seal the flange connections; (3) a substrate holder was 3D printed and used to support the microscope slide (coverslip) above the target; (4) an opening was left to allow the laser to reach the target; (5) a sample holder was coupled to a stepper motor to allow the target to rotate; and (6) a High-Efficiency Particulate Arrestance (HEPA) filter was attached to the system so that all radioactive material could be retained inside the chamber.

Figure 1: This illustration represents the designed PLD (Pulsed Laser Deposition) system, taking into account concerns related to contamination and replacement costs arising from the use of radioactive materials. The cross-sectional 3D model depicts the vacuum chamber and its constituent parts, with dashed lines illustrating the path of the laser beam.



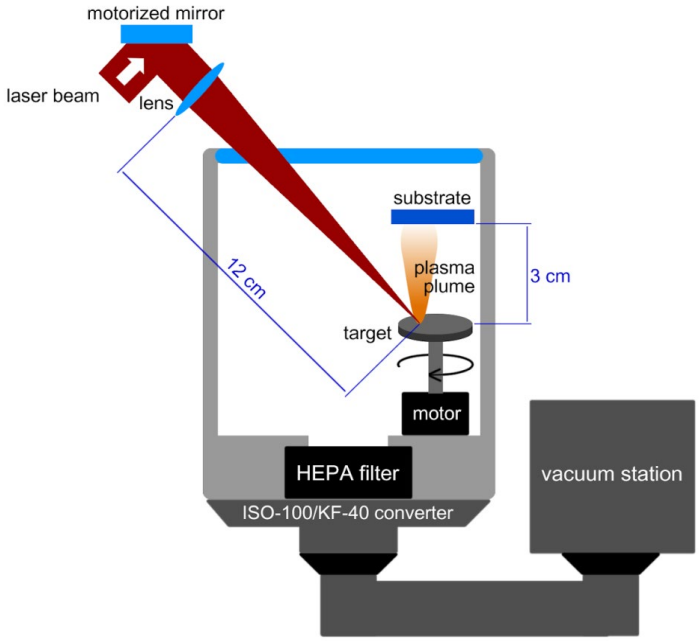
3. MATERIALS AND METHODS

To deposit thin films, the Pulselas-P-1064-150-HE laser was utilized, which generated pulses with 1.5 mJ of energy, 0.6 ns of temporal width, at a wavelength of 1064 nm, and a repetition rate of 100 Hz. A 120 mm lens was employed for ablation, resulting in a fluence of 65 J/cm^2 at the focal point.

Figure 2 shows a schematic model of the PLD system that was developed for the production of thin films. To ensure the retention of radioactive material within the vacuum chamber, a filtering module was incorporated into the system. The module features a HEPA WhatmanTM EPM 2000 filter, which has a retention capacity of 99.95% for particles as small as 0.3 μm.

Microscopic coverslips (24 × 32 mm) were used as a substrate for the growth of the films. In order to remove any impurities present on the surface of the coverslips, they were subjected to a cleaning process using isopropyl alcohol and an ultrasonic cleaner.

Figure 2: Schematic illustrates the PLD setup employed in the current study. The target surface is vaporized, and the resulting material condenses onto the substrate, forming a film. To ensure thorough surface ablation, the coordinated movement of the motorized mirror for beam positioning and the target's rotational motion is utilized. All ablated material is contained within the chamber by a HEPA filter.



To evaluate the performance of the designed PLD system, stable thin films were initially grown using a stable Nickel target. Nickel was selected for this purpose because its isotope ^{63}Ni can be utilized in the development of betavoltaic batteries [18]. After PLD processing, nickel films were analyzed through SEM and 3D optical profilometry, to evaluate thickness, homogeneity, and morphology.

Then, experiments were conducted to verify the efficacy of the filtering system, as it is imperative that all radioactive waste generated during the PLD process was confined within the vacuum chamber. To investigate this, following the production of radioactive thin films, the chamber was dismantled, and the level of activity was measured for each component.

Performing tests on the PLD system using radioactive materials would be beneficial with isotopes having a short half-life. This is due to the fact that, in case of any material leakage from the vacuum pump, the activity would decay considerably within a few days, making it possible to make necessary adjustments and conduct further tests.

Copper was chosen to be neutron-activated at the IPEN nuclear reactor (IEA-R1) due to its isotopes' direct decay into stable elements and short half-lives, specifically 12.7 hours for ^{64}Cu [19] and 5.1 minutes for ^{66}Cu [20]. The target decayed for a few hours to allow the ^{66}Cu to completely decay. Two experiments were conducted to grow copper radioactive thin films, with deposition times of 60 and 120 minutes.

In measuring the radioactivity of the Cu thin films and PLD system components, were utilized an analog Geiger-Muller (Eberline model E520) counter and an HPGe gamma spectrometer (Canberra, model GX2020) coupled to a digital acquisition system. The Geiger-Muller counter was used immediately after the first production of the radioactive thin film, whereas the HPGe was utilized after the second production. In the second dataset acquired using the HPGe, to ensure that we could identify only the transitions of interest (^{64}Cu transitions), the activity was determined by integrating the peaks of the emission spectrum.

The methodology used for thin film growth, employing the engineered PLD system, comprised the following steps: firstly, the target material (Ni or radioactive Cu) was placed in the sample holder, and the chamber was then sealed. Next, the laser was activated and focused. A five-minute sweep was performed to eliminate any impurities from the target surface. Once complete, the chamber was opened, and the coverslip was positioned 3 cm above the sample. The vacuum was then created, and the pressure stabilized at 6.1×10^{-3} mbar (mean free path of approximately 52 cm for Cu and Ni). Subsequently, the laser was switched on, and the deposition process began.

4. RESULTS AND DISCUSSIONS

4.1. Stable Nickel thin films

Initially, films were grown using a stable Ni (99%) target to evaluate the effectiveness of the PLD system for deposition. This allowed measurement of the deposition rate (nm/min) for Ni and assessment of the engineered PLD system's performance before working with radioactive materials.

Figure 3 shows the engineered PLD system in a) and the system in operation producing a thin film of Ni in b). The plasma plume generated from the ablation process is visible, as schematized in Figure 2.

The natural Nickel thin films were grown in triplicate at three deposition times (30, 60, and 90 minutes) for statistical purposes. Figure 4 shows the SEM analysis (item a) and Optical Profilometry (item b) conducted on the thin film. The optical profilometry analysis enables the measurement of the thickness profile of the thin film.

Figure 3: a) A photograph of the PLD prototype; and b) A photograph of the thin film being produced, it is possible to see the plasma plume (in blue) expanding from the target.

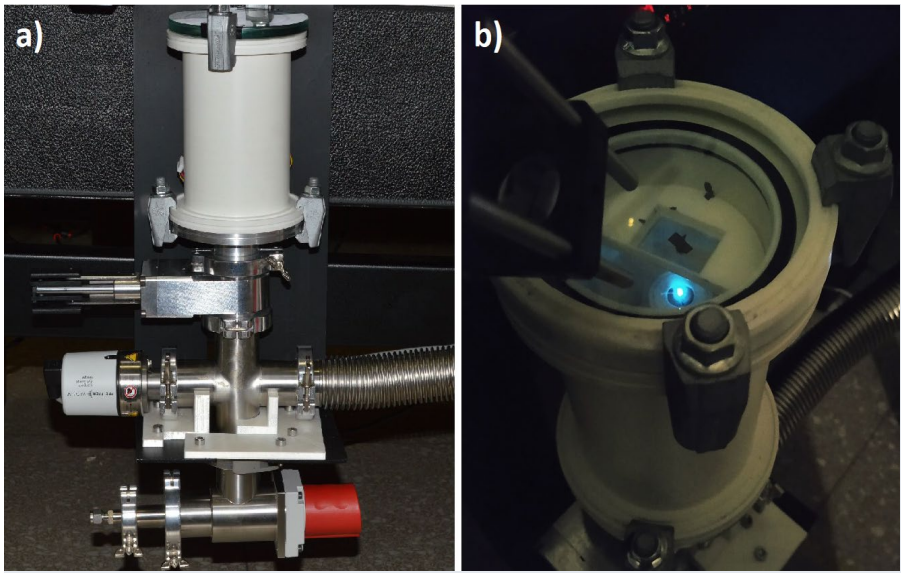


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Figure 4: Nickel film: a) SEM morphology analysis, and b) thickness analysis through optical profilometry.

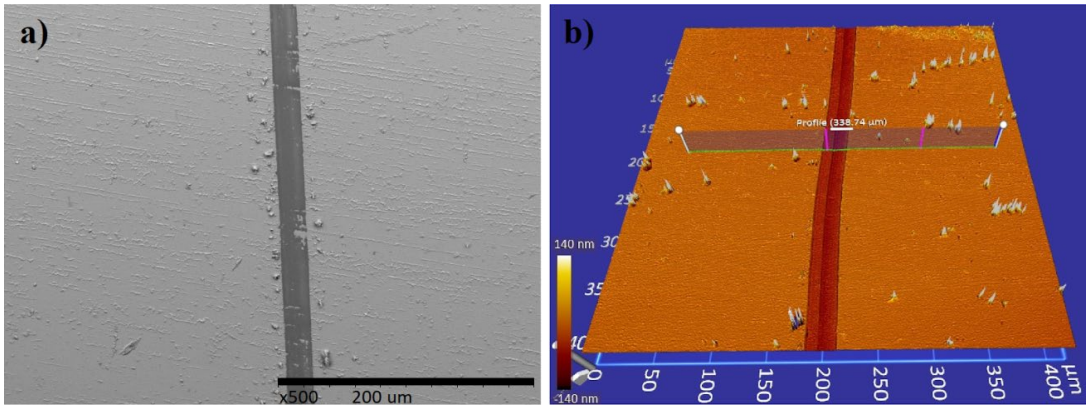
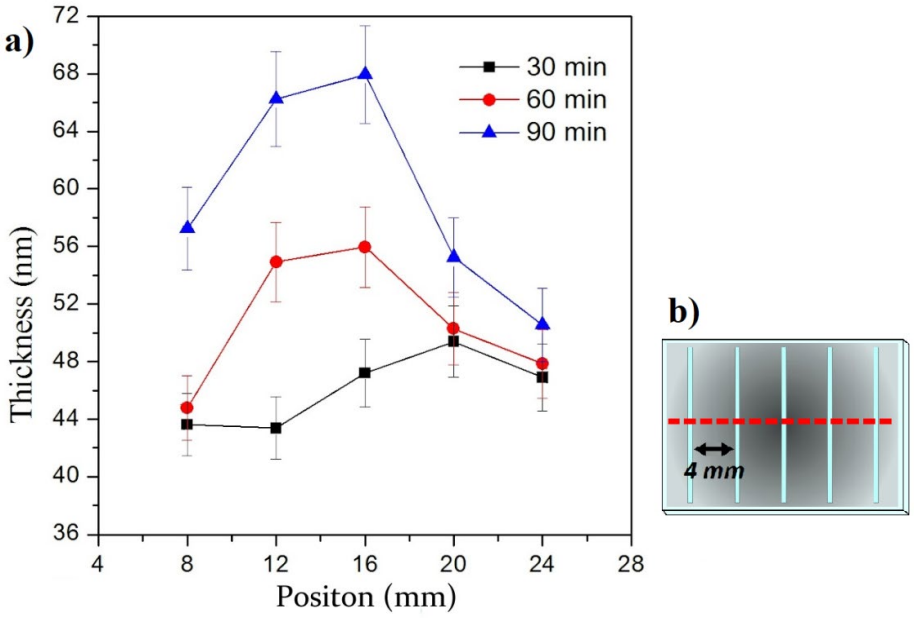


Figure 5 shows the thickness profile of Ni films grown, the abscissa axis represents the horizontal line dashed in red in the film illustration and each point on the graph corresponds to the film thickness.

Figure 5: a) Thickness profile of Ni films for a deposition time of 30, 60, and 90 minutes, and b) thin film illustration, the measurements were taken at five different points on the axis dotted in red.



Based on Figure 5, it can be seen the thickness profiles are not homogeneous in thickness, as expected, since the plasma plume expands from a single point. Considering the assembled setup, in which the metallic target was aligned with the center of the coverslip, and the ablation also performed close to the central region of the target, a thicker film at the 16 mm region was an expected result.

According to simulations carried out by Bormashov, the ideal ^{63}Ni thickness for the development of betavoltaic batteries is about 2 μm [18] which leads to the best efficiency. As can be seen in Figure 5, the preliminary films grown from a stable Nickel target resulted in thicknesses much less than 2 μm, of which the greatest thickness obtained was (70.0 ± 3.4) nm.

Considering this point with greater thickness, the deposition rate obtained was (0.75 ± 0.04) nm/min, since this study could later serve as a basis for the production of ^{63}Ni films.

Regarding the non-uniformity of the film it is remarkable that, if there were no beam sweeping and sample rotation, the deposition would be much more accentuated in the central

region of the coverslip. If there was a need to obtain a more homogeneous thickness, it would be possible to develop a system to carry out the coverslip movement, this would significantly improve deposition, generating films with more regular thicknesses.

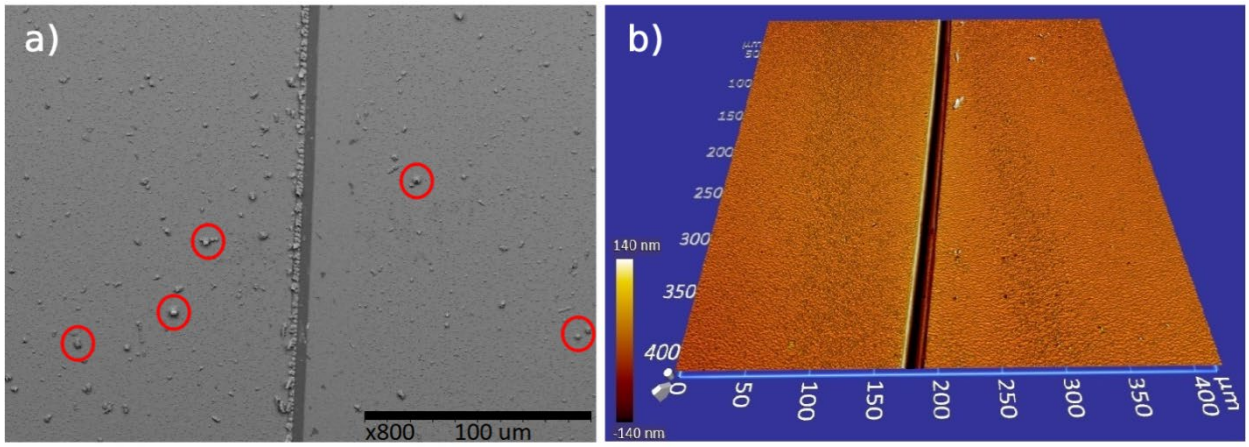
4.2. Radioactive Copper thin films

Once the effectiveness of the system for producing conventional thin films was validated, the next step was to utilize it for the production of radioactive thin films. Therefore, the Cu samples (99.7%) were neutron-activated in the IPEN nuclear reactor (IEA-R1) and after that, they were processed in the PLD system for radioactive thin film growth. Copper radioactive thin films were grown in two experiments with deposition times of 60 and 120 minutes, respectively.

In Cu films, a certain unevenness in the thickness profile along the horizontal axis was identified, similar to the Ni thin films. The central regions had the greatest thicknesses, (167.8 ± 3.7) nm and (313.5 ± 9.2) nm, for films grown with one hour and two hours of deposition, respectively. In addition, concerning the copper processing in the PLD system, a deposition rate of (2.70 ± 0.10) nm/min was obtained - this means that if it were necessary to grow a 1 μm film, 6.1 h of deposition would be necessary, for the given condition.

As shown in Figure 6 (a), the growth process created some irregularities on the Cu film surface, which were eventually caused by the deposition of small amounts of molten material (droplets).

Figure 6: Copper Radioactive thin film surface analysis. a) Surface characteristics were analyzed with SEM and, b) the film thickness was measured with optical profilometry.



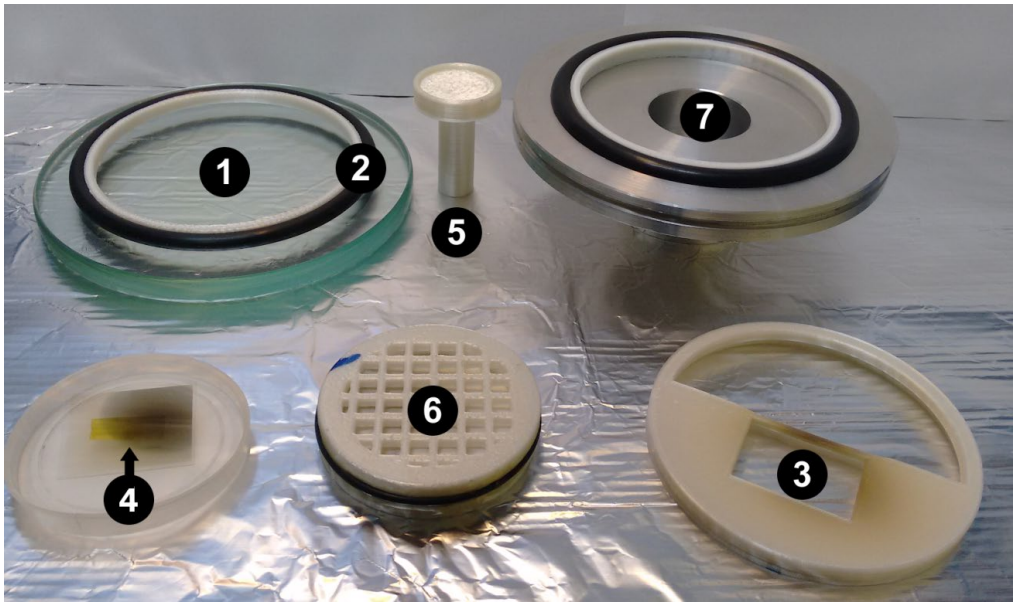
The presence of droplets was more pronounced in Cu films in comparison to Ni films, which can be primarily attributed to the difference in their thermal conductivities. Given that copper exhibits a higher thermal conductivity than nickel, the ablation process resulted in the formation of regions with a larger volume of molten material. This, in turn, led to the creation of a plume where the occurrence of droplets was more prominent. On the other hand, in Nickel due to the lower thermal conductivity, the laser pulse energy introduced into the sample is restricted to a smaller volume, allowing the formation of a plume in which there is much more vapor than droplets.

4.3. Assessment of Contamination in the Vacuum Chamber and Its Components

The PLD system underwent tests to identify the location of the radioactive material. The first aspect examined was the filtering system's efficiency in containing all radioactive waste produced within the vacuum chamber. To investigate, the chamber was disassembled after the production of Cu radioactive thin films, and the level of contamination was measured for each piece. Some of the parts analyzed individually with the Geiger-Müller counter are shown in Figure 7.

In the context of investigating the retention capacity of radioactive material, the radioactivity analysis of the KF-40 to ISO-100 converter (item number 7 in Figure 7) is of utmost importance. This is due to the fact that it is directly connected to the vacuum chamber, positioned immediately below the filtering module. Thus, in case of any leakage of radioactive material from the chamber, this component would be the first to be contaminated.

Figure 7: Parts of the PLD prototype analyzed: 1) the optical window; 2) O-ring used for sealing; 3) the cover slip support; 4) the radioactive thin film; 5) the sample holder; 6) the filter used; and 7) the ISO-100 converter for KF-40.



Although some parts had little contamination, it was possible to obtain some counts when the equipment was approached to a few millimeters away from the surface. Table 1 presents a list of the measured radioactivity from the pieces, the sample, and the film after the deposition process.

Table 1: Measurements of radioactivity in PLD system components following radioactive thin film growth. The initial dataset was obtained using the Geiger-Müller, while the subsequent dataset was acquired utilizing an HPGe.

CHAMBER PIECE	1º RADIOACTIVE THIN FILM (GEIGER-MÜLLER)		2º RADIOACTIVE THIN FILM (HPGe)	
	COUNTS/MIN	TOTAL ACTIVITY	COUNTS/MIN	TOTAL ACTIVITY
Sample	12,000 ± 500		279,254.1 ± 1846.4	
Radioactive thin film	230 ± 50	24.5%	146.6 ± 1.4	44.8%
Sample holder	250 ± 50	26.6%	75.0 ± 1.0	22.9%
Coverslip holder + Optical window	240 ± 10	25.5%	62.5 ± 1.1	19.1%
Chamber wall	130 ± 5	13.8%	29.6 ± 0.5	9.0%
Filter	90 ± 5	9.6%	13.8 ± 0.3	4.2%
ISO-100/KF-40 converter	Background	0%	Background	0%
Vacuum station	Background	0%	Background	0%
Vacuum piping	Background	0%	Background	0%
Total activity	940	100%	327.5	100%

A comparison between the initial and subsequent productions of radioactive thin films reveals a substantial rise in the total thin film activity, from 24.5% to 44.8%, denoting a percentage increase of roughly 183% in the quantity of deposited material. This enhancement is attributable to an improvement in the motion of the motorized mirror after the initial experiment, which resulted in improved alignment of the plasma plume with the substrate, leading to an elevated deposition rate and reduced contamination of other vacuum chamber components.

The ISO-100/KF-40 converter was a key part to evaluate the efficiency of radioactive material retention inside the chamber, this part of the system did not present contamination by radioactive material in any of the productions of radioactive thin films.

The analysis of the activity percentage of each component reveals that the majority of the radioactive material, removed via ablation from the target, was concentrated on the surfaces parallel to the target. Specifically, the activity percentage for the parts above the target was 76.6%

and 86.8% for the first and second experiments, respectively. As the plasma plume moves in a direction perpendicular to the target, these findings are consistent with expectations.

Despite this, there was a percentage of 23.4% and 13.2% of radioactive material, for the first and second experiments, which were deposited on the chamber wall or reached the filtering module.

However, when the plasma plume was more accurately aligned with the deposition substrate, the deposition efficiency improved, resulting in less radioactive material reaching the filtering module. The percentage of filter activity decreased from 9.6% of total activity in the first experiment to 4.2% in the second.

Basically, the cooling of the plasma plume that arises from the emission of radiation and heat transfer enables the generation of nanoparticles [21-23] that may eventually scatter in the chamber via inelastic collisions and subsequently converge toward the bottom of the chamber due to the interplay between the gravitational force and the suction force exerted by the vacuum station. As a result, a fraction of the ablated material is captured and retained by the filtering module.

5. CONCLUSIONS

This study aimed to develop a pulsed laser deposition (PLD) system for processing radioactive targets and producing radioactive thin films, which to the best of our knowledge, has not been attempted before. The system was engineered to prioritize safety, easy replacement/disposal of parts, and effective vacuum management.

Firstly, a nonradioactive film of Ni was grown with a maximum thickness of (70.0 ± 3.4) nm, at a rate of (0.75 ± 0.04) nm/min for the laser parameters applied (100 Hz and 65 J/cm^2 fluence). The system performed as expected, enabling the system to be tested with radioactive material.

The Cu radioactive targets, necessary for the growth of the radioactive thin films, were obtained via neutron activation. Analysis of the thin film central region revealed a thickness of (167.8 ± 3.7) nm for a deposition of 60 minutes and (313.5 ± 9.2) nm for a deposition of 120 minutes. The analyses of the thicknesses indicate an average deposition rate of (2.70 ± 0.10) nm/min within the central region, considering the specific laser parameters applied.

The contamination of the system parts was measured, besides the film, the radioactive copper mainly stayed around the target area. In addition, the vacuum pump was found to be free of radiation counting, indicating the effectiveness of the HEPA filter which separates the "hot" and "cold" areas.

The processing time could be significantly reduced by using a laser with a higher repetition rate and/or pulse energy. Since no complex or expensive systems or equipment are located in the "hot" area, the operational and maintenance costs of the system can be considered low.

As an extension of this study, the implemented and validated system exhibits promise in exploring the development of betavoltaic nuclear batteries based on solid-state devices and radioactive thin films.

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CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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