Research Square

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# Fabrication and Characterization of Variable-Thickness 94,98,100 Mo targets for Heavy-Ion Fusion Experiments

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# Abstract

A series of self-supporting, enriched Molybdenum targets 94,98,100Mo featuring a wide range of areal densities from thin films of 90 μg/cm2 up to thicker foils of 600 μg/cm2 were successfully fabricated using high vacuum physical vapor deposition. Through optimized deposition parameters, the overall target preparation was achieved with high material efficiency, consuming as little as 70 mg of rare isotopic material. These targets are prepared specifically for low energy heavy-ion nuclear physics experiments, including multi nucleon transfer reaction measurements and fusion barrier distribution studies at the Inter University Accelerator Center (IUAC), New Delhi. This paper presents the novel single evaporation technique used to synthesize targets of varying thick nesses simultaneously, the thermal annealing protocols optimized for mechanical stress relief, and the quantitative characterization of chemical purity and stoichiometry performed via Rutherford Back scattering Spectrometry (RBS) and Energy Dispersive X-ray Spectroscopy (EDX).


# Introduction

Target fabrication plays a vital role in nuclear reaction experiments, as a stable, highly uniform, and chemically pure target is an absolute necessity for extracting nuclear structural information and studying reaction mechanisms at energies around and below the Coulomb barrier. A suitable target must possess sufficient mechanical strength to withstand intense heavy-ion beam irradiation over extended run times while maintaining precise areal density. In low energy fusion experiments, cross-sections are determined by measuring compound nucleus evaporation residues (ERs), which are directly identified based on their mass to charge (M/q) ratios using recoil mass spectrometers like the Heavy Ion Reaction Analyzer (HIRA) at the Inter University Accelerator Centre (IUAC), New Delhi [1–3]. The present study aims to investigate fusion barrier distribution dynamics alongside multinucleon transfer reaction mechanisms for $^{32}S + ^{94,98,100}Mo$ systems using the HIRA facility. Thin targets of approximately 90 μg/cm$^2$ are essential for the barrier distribution measurements to minimize energy straggling and maintain high energy resolution, whereas relatively thicker targets of about 600 μg/cm$^2$ are needed for transfer reaction studies to enhance the experimental reaction yield [4, 5]. While Molybdenum targets thicker than 1 mg/cm$^2$ could be typically produced through mechanical cold rolling, thinner targets below 1 mg/cm$^2$ must be synthesized using physical vapor deposition techniques. A significant hurdle in this process is the scarcity and extreme cost of enriched isotopic precursors, given that the natural abundances of $^{94}Mo$,$^{98}Mo$ and $^{100}Mo$ are only 9.2%, 9.4% and 9.6%, respectively. Minimizing material consumption during evaporation was therefore a primary goal. To achieve this, a range of self-supporting foils as well as carbon-backed targets spanning 90 μg/cm$^2$ to 600 μg/cm$^2$ were successfully fabricated. Carbon was selected as a thin backing material owing to its low atomic number, which keeps beam energy loss and background contamination to a minimum. The target synthesis was executed using a dedicated two system setup a turbo molecular pump based high vacuum coating unit for clean Molybdenum deposition, and a diffusion pump based unit for carbon backing preparation.

## Experimental procedure

# Development of self-supporting targets:

Self-supporting molybdenum targets were the ideal choice for the planned transfer reaction and fusion barrier distribution measurements. Several research groups have previously reported the fabrication of self-supporting Mo targets using various parting agents, such as NaCl, $BaCl_2$, and CsI [6]. Initial trial runs at IUAC using $BaCl_2$ ,NaCl, CsI, and Teepol as parting agents were unsuccessful, as the deposited films consistently broke during the water-floating process. However, trial runs utilizing Kcl as a parting agent showed marked improvement, allowing several self-supporting Mo target foils to be successfully floated and mounted onto target holders with 10 mm diameter apertures. The fabrication was carried out using high-purity glass microscope slides (Cole-Parmer) as substrates. The sequential deposition of the KCl parting layer via resistive thermal evaporation, followed by Mo deposition via electron beam evaporation, was performed inside a single coating unit without breaking the vacuum. Following deposition, the coated substrates were annealed in a tubular furnace, as shown in Fig. 1, under an argon atmosphere at an optimized temperature of $235^0$ C to relieve internal tensile stress in the molybdenum films. Process trials revealed that annealing temperatures below $235^0$ C were insufficient for effective stress relief, whereas temperatures exceeding $235^0$ C caused thermal degradation of the underlying KCl layer. Post annealing, the Mo films were released by floating them in warm distilled water. Despite this successful release, the resulting self supporting Mo targets exhibited a limited shelf life of only a few days before degrading. While further systematic studies are required to improve the long term mechanical stability of thin self supporting Mo targets, carbon backed Mo targets demonstrated significantly greater durability over extended periods and will therefore be utilized for the upcoming beam line experiments [7, 8].

## Preparation of Mo targets with carbon backing:

# Fabrication of carbon backing:

Carbon backing films were synthesized using a diffusion pump based coating unit, as shown in Fig. 2, maintained at a base vacuum level of $10^{-7}$ mbar. High purity glass slides were thoroughly cleaned and used as substrates. Following the procedure described in [9, 10]. A 6 mm diameter KCl pellet, prepared using a hydraulic pellet press, was first evaporated onto the substrates via resistive thermal heating to act as a parting layer. Immediately following this, a 9 mm graphite pellet was evaporated using electron beam bombardment. To maintain interfacial cleanliness, both the KCl parting agent and the carbon layer were deposited sequentially within the same vacuum cycle without breaking vacuum. The nominal deposited thicknesses were 100 nm for the KCl layer and 15 μg/$cm^2$ for the carbon film, with a controlled deposition rate of 0.1 nm/s at a target to substrate distance of 16 cm. Upon completion of the evaporation sequence, the vacuum chamber was allowed to cool under vacuum for 4 to 5 hours prior to

venting with air. To relieve internal stress, the carbon coated films were subsequently thermally annealed in a tubular furnace at $235^0$ C for 1 hour under an argon gas flow, as illustrated in Fig. 1.

## Deposition of Mo on carbon backing:

The deposition process was monitored in real time using a dual crystal thickness monitor. To optimize evaporation parameters, preliminary trial runs were conducted using natural Molybdenum prior to evaporating the enriched isotopes using e-gun. Because the natural Molybdenum material was in powder form, a hydraulic pellet press was utilized to compress the powder into 3 mm pellets. For typical trial runs, the e-beam gun emission current was ramped gradually to 205 mA to deposit Mo at a rate of 0.1 $A^0$/s under a base vacuum of $10^{-8}$ mbar. Following deposition, the Mo coated slides were annealed at 235◦ C for 1 hour under an argon gas flow for stress relief, after which the Mo films were successfully released by floating in warm distilled water. For the upcoming nuclear structure experiments, multiple targets spanning thicknesses from 90 μg/$cm^2$ to 600

μg/$cm^2$ were required, but the available isotopic material was strictly limited to 100 mg. The principal technical challenge was to fabricate targets of varying thicknesses while maximizing material collection efficiency within a single run. To achieve this, carbon-coated glass substrates were strategically arranged at different source to substrate distances ranging from 5 cm to 11 cm. Once the vacuum chamber reached $7.0 \times 10^{-8}$ Torr, the electron gun was energized, and the current was incrementally raised from 70 mA to 205 mA in 5 mA steps while maintaining a steady deposition rate of 0.1 $A^0$/s. Upon reaching 205 mA, the total deposited Molybdenum thickness recorded by the quartz crystal monitor reached 0.177 $kA^0$. After a cooling period of 7 to 8 hours, the chamber was vented with air and the deposited slides were retrieved. Using this multi-distance configuration, ten $^{94,98,100}$Mo targets with thicknesses spanning 90 μg/$cm^2$ to 600 μg/$cm^2$ were successfully synthesized in a single evaporation batch as shown in Fig. 3. To relieve internal stresses in the target films, the slides were thermally annealed in a tubular furnace under an argon atmosphere. The furnace was heated at a ramp rate of $5^0$ C/min to $235^0$ C and held at this temperature for a dwell time of 1 hour. Following annealing, the Molybdenum targets were successfully floated in warm distilled water and mounted onto stainless steel target frames with a 10 mm inner diameter, as shown in Fig. 4.

## Characterization

# Rutherford back-scattering technique:

The fabricated targets were subsequently characterized using the Rutherford Back-scattering Spectrometry (RBS) facility at IUAC. The measurements were carried out utilizing a 1.7 MV tandem accelerator with a 1.7 MeV He + ion beam. The back scattered He+ ions were detected at a scattering

angle of $166^0$ using a silicon surface barrier detector (SSBD) with a solid angle of ~ 3.6 msr and an energy resolution of 15 KeV for 1.7 MeV He+ ions.

The beam current was maintained at 11 nA, collecting a total charge of 10–15 μC per spot at various positions across the target surface. Quantitative analysis of the RBS spectra, carried out using SIMNRA software [11–14], confirmed that the thickest targets achieved an areal density of 600 μg/$cm^2$, while the thinnest targets measured 90 μg/cm. The representative RBS spectra for the $^{94,98,100}$Mo targets verified high elemental and isotopic purity, with no significant high-Z or low-Z impurities detected in the analysis. The thickness achieved are calculated using various techniques which are listed in Table 1.

Table 1
Thickness of molybdenum targets measured by different techniques.

| Target | Thickness ( μg/$cm^2$) | | |
|---|---|---|---|
| | QCM | α-loss | RBS (SIMNRA) |
| $^{94}$Mo | 225 ± 5 | 200 ± 8 | 232 ± 5 |
| $^{98}$Mo | 596 ± 10 | 600 ± 25 | 605 ± 12 |
| $^{100}$Mo(Thick) | 416 ± 10 | 409 ± 24 | 420 ± 11 |
| $^{100}$Mo(Thin) | 86.1 ± 4 | 95 ± 4 | 90 ± 4 |

## Energy dispersive X-ray analysis:

**Figure 6** EDX spectra of $^{94}$Mo thick and thin targets. Inset shows the weight percent of the

elements present in targets

Energy Dispersive X-ray (EDX) spectroscopy, performed in conjunction with Scanning Electron Microscopy (SEM), is a widely recognized technique for analyzing the elemental composition and chemical purity of target materials. In this setup, high energy incident electrons interact with the atoms in the target sample, generating secondary electrons and characteristic X-rays [15]. An Energy Dispersive Spectroscopy (EDS) detector measures the energies of these emitted X-rays. Because the intensity of an element's characteristic X-ray peak directly correlates with its relative concentration in the sample, the resulting spectral intensities can be converted into quantitative elemental weight percentages. The characteristic line designations (e.g., K or L shells) represent the specific photon wavelengths emitted when electrons transition from outer atomic shells to fill lower energy inner shell vacancies. To qualitatively evaluate potential impurity levels, EDX spectra were acquired for two representative $^{94}$Mo target samples. The resulting spectra primarily exhibited the characteristic L lines of Molybdenum, alongside minor K lines corresponding to carbon and oxygen. The trace oxygen signature is attributed to

ambient surface oxidation of the thin films following atmospheric exposure, while the minor carbon background likely originates from residual hydrocarbons within the evaporation chamber. Beyond these expected surface and substrate contributions, no additional high-Z or low-Z elemental impurities were detected within the sensitivity limits of the EDX apparatus. The relative weight percentages of all detected elements are summarized in Fig. 6.

## Summary and Conclusion

A reliable method was established for fabricating molybdenum ($^{94}$Mo,$^{98}$Mo and $^{100}$Mo) targets across both thin and thick regimes while minimizing raw material loss. Because enriched stable isotopes are exceptionally rare and costly, standard physical vapour deposition techniques were refined by optimizing evaporation distance and geometric parameters. This careful alignment ensured maximum target yield per milligram of natural material, providing precise thickness control without excessive loss on chamber walls or substrate fixtures. To facilitate clean target recovery, carbon coated glass slides were prepared using potassium chloride (KCl) as a water-soluble releasing inter layer. The thin carbon layer acts as a structural backing for the deposited molybdenum foil, while the underlying KCl film enables effortless floating. When the assembly is immersed in distilled water, the salt layer dissolves cleanly, allowing the carbon backed molybdenum target to detach from the glass substrate without mechanical tearing or distortion. Thermal processing played a critical role in maintaining structural integrity prior to target detachment. Annealing the deposited target assembly at $235^0$ C effectively relieved internal mechanical stress induced during high energy vapour condensation. This specific thermal treatment prevented micro cracking, curling, and layer delamination during the floating phase, while simultaneously driving off surface moisture and organic residues. Compositional and structural quality were confirmed through non-destructive analytical techniques. Rutherford Backscattering Spectrometry (RBS) verified target thickness profiles and layer stoichiometry across both thin and thick variations, confirming uniform thickness and an absence of heavy element contamination from the deposition environment. Additionally, Energy Dispersive X-ray Spectroscopy (EDX) confirmed high chemical purity within the molybdenum matrix, demonstrating that no significant elemental impurities, such as oxygen or sulfur, were introduced during deposition, annealing, or substrate handling.

## Declarations

# Conflict of Interest

The authors have no conflicts of interest to disclose. No financial interests are associated with this work. Furthermore, this manuscript represents original research, has not been previously published, and is not under concurrent review elsewhere.

# Author Contribution

The authors declare that they have no conflicts of interest to disclose. All authors are supported for research work, and no financial interests are associated with this work. Furthermore, this manuscript represents original research, has not been previously published, and is not under concurrent review elsewhere

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## Figures




**Figure 1**

Tubular Furnace

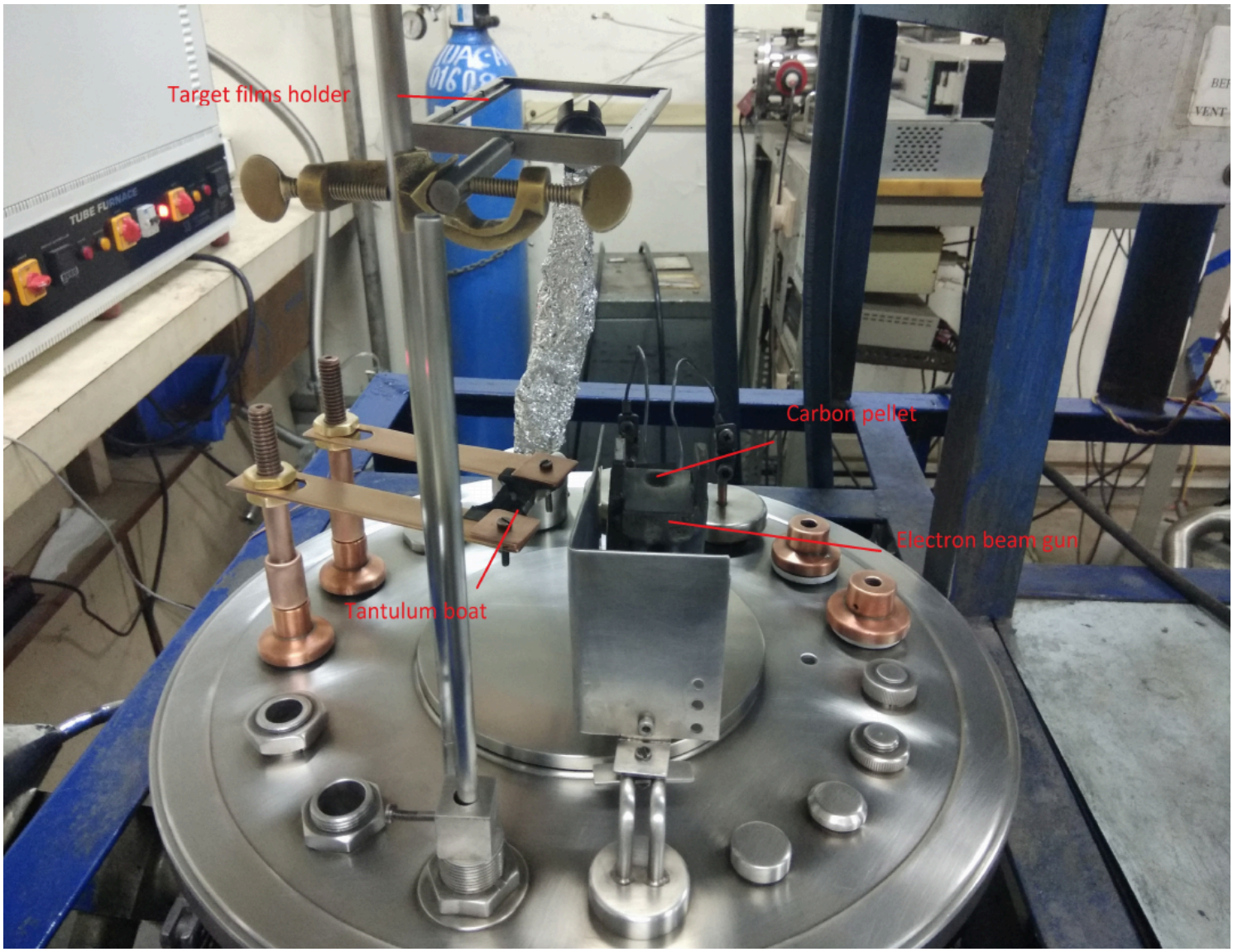



**Figure 2**

Inside view of diffusion pump based coating unit

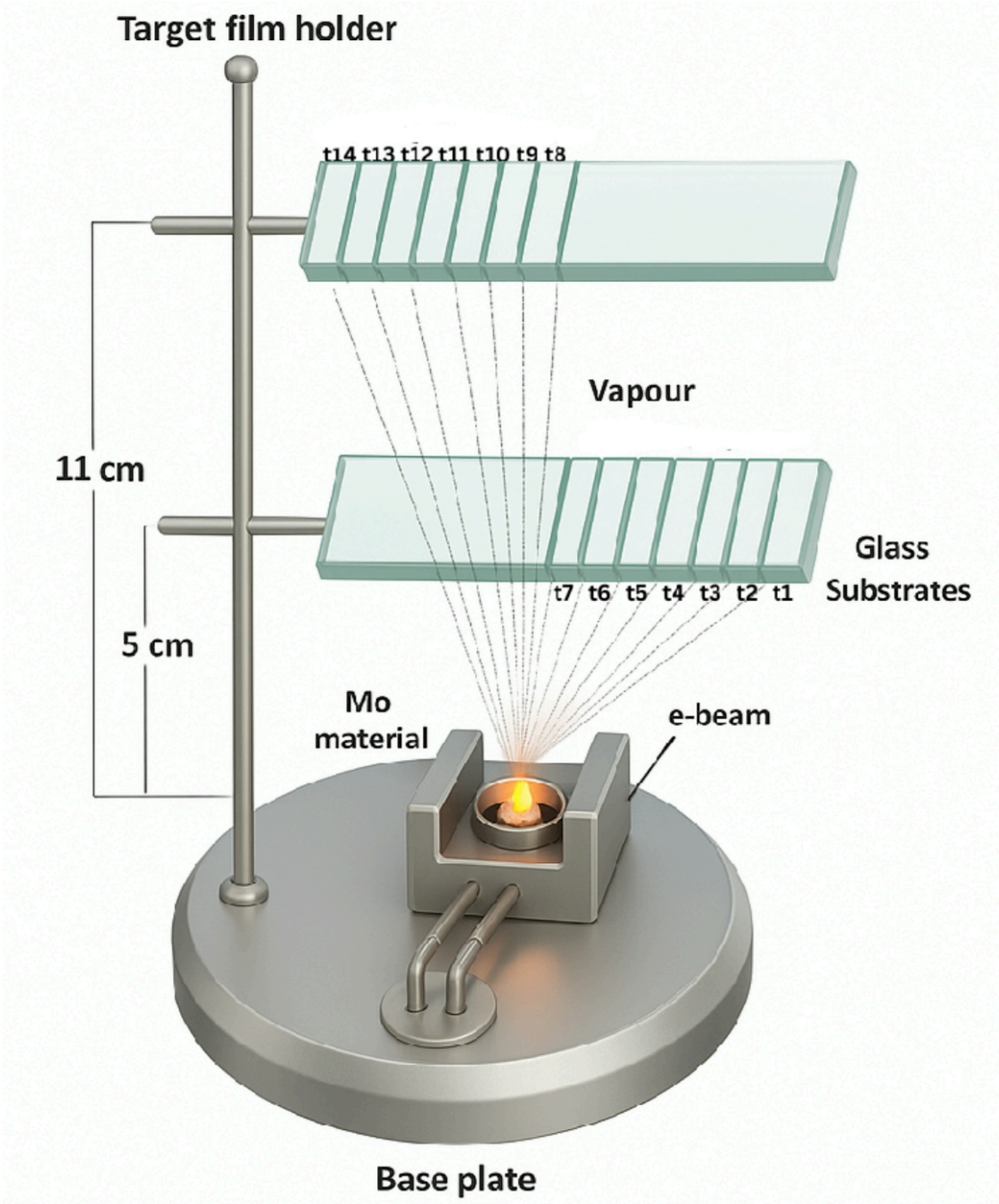



**Figure 3**

3D schematic diagram illustrating the target evaporation technique for preparing multiple thin nuclear reaction targets with different thicknesses simultaneously.

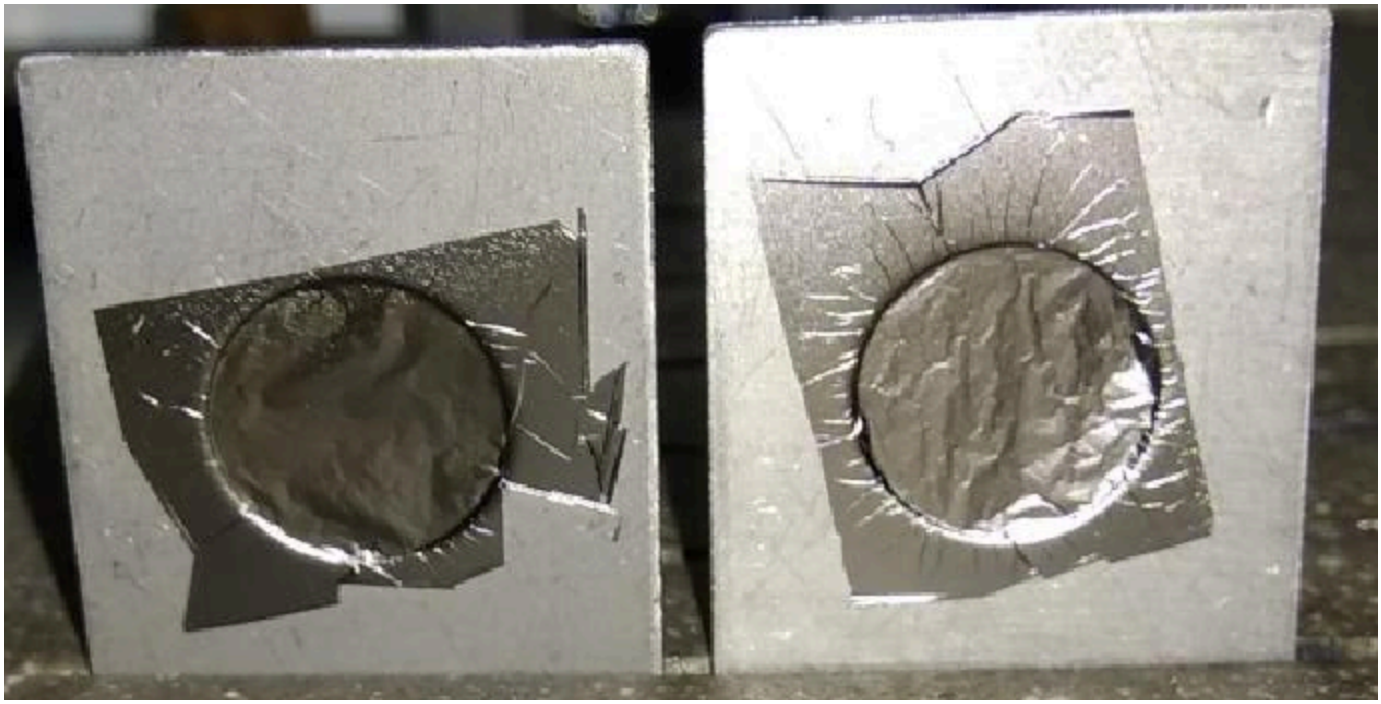


**Figure 4**

Multiple thickness of Mo targets

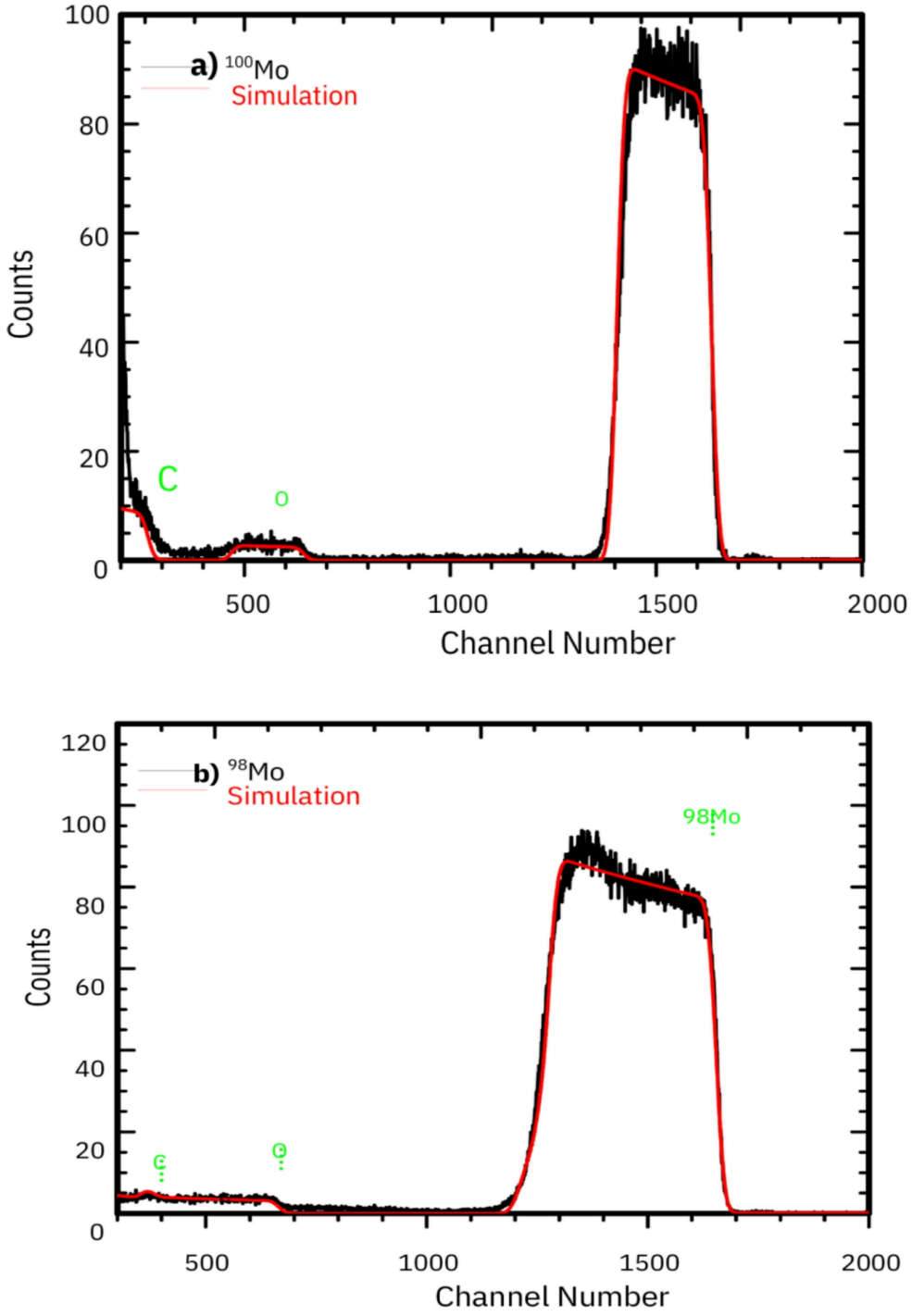



**Figure 5**

Experimental spectra obtained from RBS measurement along with the simulated results (red line) for (a) $^{100}$Mo and (b) $^{98}$Mo film with a carbon backing .

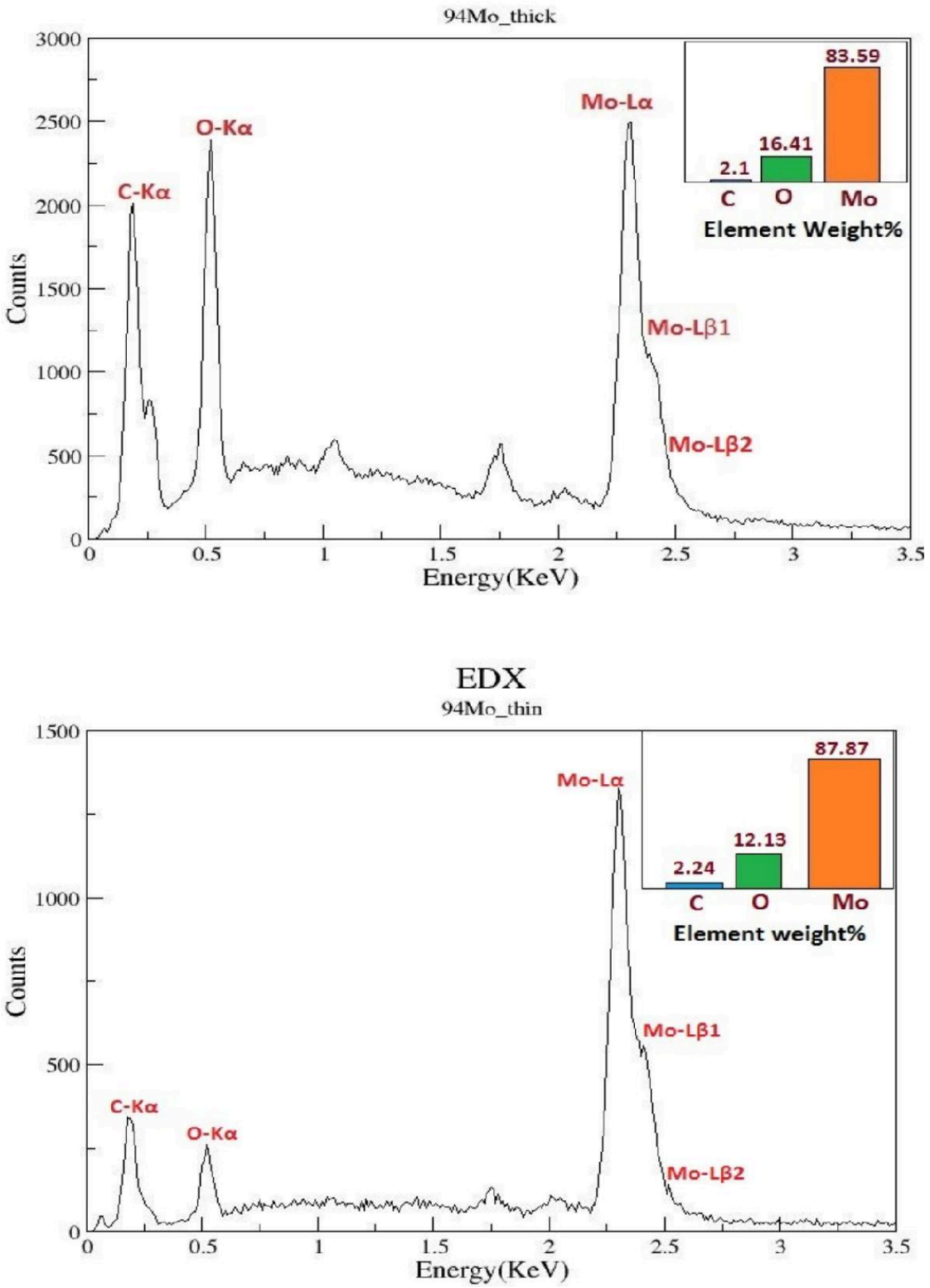



**Figure 6**

EDX spectra of $^{94}$Mo thick and thin targets. Inset shows the weight percent of the elements present in targets