

Recent target development activities at Inter-University Accelerator Centre

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Abstract The target development laboratory at Inter-University Accelerator Centre (IUAC) is one of the main facilities for the fabrication of nuclear physics targets in India. Vacuum evaporation and cold rolling technique are mainly used for the production of isotopically pure targets at IUAC. Targets of Gd, Pt, W, Mo, Ta, Ni, Ag and Sn have been developed in the form of self-supporting targets and with thin carbon backing. Recent progress in target development laboratory at IUAC will be discussed in the report.

Keywords Rolling technique · Electron beam evaporation · Thermal evaporation · Isotopic targets

Introduction

The target development laboratory at IUAC plays an active role in fabrication and supplying of targets and thin films for researchers in nuclear physics, material science and atomic physics in India. Main focus of this report is to discuss the recent development of isotopically pure targets for nuclear physics applications and further improvements in target preparations which were already reported. At IUAC, targets are prepared by physical vapor deposition (PVD) techniques such as thermal evaporation and electron gun evaporation or by cold rolling. Thick targets with a thickness in the order of mg/cm² are usually prepared by cold rolling and thin targets with a thickness in the order of μ g/cm² are prepared by PVD. Thickness of the targets fabricated in PVD technique is measured by an in-situ quartz crystal monitor. Thickness of the rolled foils is calculated by mass and area of the foil. Profilo meter, α energy loss method, XRF technique and RBS technique are also used for the further verification of the thickness and impurities in the targets. The maximum variation in thickness of rolled foils of size of 5×5 cm² is within 20 %. Thickness variation in rolled targets and evaporated targets of size of 1×1 cm² is measured within 5 %.

Experimental procedure

Targets of ⁹⁴Mo and ¹⁰⁹Ag by cold rolling

Mo targets in the form of self-supporting and with thick Au backing have been fabricated by rolling. Fabrication of Mo targets by different methods was reported by several investigators [1-12]. Kheswa et al. [9] and Karasek [10] have described the possibilities of cold rolling in Mo target preparation. The thin self-supporting targets of Ag have also been reported by Gallant [13].

For the preparation of Mo and Ag targets for each material less than 100 mg and only as a powder were available. Targets of Ag and Mo were regularly fabricated by PVD. However an amount of 100 mg of material was not sufficient for preparing targets with a thickness in the order of mg/cm² by vacuum evaporation. Minimum material loss in rolling and the good malleability as well as high ductility of Mo and Ag were the major factors for choosing the

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rolling technique. In the following the different steps for the production of a foil are described.

Initially, the powder was pressed by a hydraulic press to form a pellet of 3 mm diameter which was rather fragile. The pellet was melted by electron beam heating in a vacuum coating unit several times with intermediate flipping to form a single uniform solid bead. Since a sudden cooling of material by the chilled water supply in crucible may adversely affect the rollability of material, the water supply was minimized and the beam current of the electron gun was gradually reduced. The resulting piece was gradually thinned down by rolling in a highly polished stainless steel folder. The homogeneous metal bead and a resulting rolled Mo foil are shown in Fig. 1. With this technique, target thickness in the order of 1 mg/cm² was achieved for ⁹⁴Mo and ¹⁰⁹Ag.

For Mo targets with Au backing, initially the Mo (1.5 mg/cm^2) and Au (10 mg/cm^2) were rolled separately. An In layer of 100 nm was deposited by PVD on the rolled foils. The foils were subsequently rolled together to form a single piece of Mo target of 1.4 mg/cm² with a Au backing of 9.6 mg/cm².

Ni foils by cold rolling

Several investigators have reported the fabrication of Ni foils by electron beam evaporation and sputtering [14, 15]. Karasek [10] has already described the importance of heat treatment in rolling of ductile foils in the order of μ g/cm². Additional features like glove box and Ar gas supply are also recommended for maintaining a neutral and dust-free environment for rolling.

Latest experience with Ni foil in rolling without any heat treatment indicated that gradual reduction of the foil thickness with extreme care and careful handling can result in thin foils of 450 μ g/cm² thickness. Several Ni foils were fabricated from the initial thickness of 22 mg/cm² (Fig. 2; Table 1) with the help of a mirror polished stainless steel folder. No additional features like glove box or Ar gas supply were used in the fabrication. These foils were used to separate the gas-filled magnetic region from beam line vacuum in HYbrid Recoil mass Analyzer (HYRA) at IUAC. All the rolled Ni-foils were stable enough to withstand a pressure difference between 1.3 mbar and 1.7 × 10⁻⁵ mbar.

Targets of rare earth elements

In the past, the isotopes of gadolinium and other rare earth elements were mainly available in oxides forms. Conversion of oxide form into elemental form was a major task in target fabrication of rare earth elements. Many investigators have reported the preparation of Gd by reduction and vacuum evaporation [16–23].

Recently suppliers like Oak Ridge National Laboratory (ORNL),Oak Ridge, USA and ISOFLEX, Moscow, Russia started supplying isotopes of Gd and other rare earth materials in elemental forms like sheets and shots which can be rolled. Sapir [24] has reported the fabrication of Gd target of 3 mg/cm² by cold rolling.

Gd enriched in Gd-160 is commercially available as foil with a thickness between 0.25–1 mm. Gd foils were thinned down to the thickness of 500 μ g/cm² in cold rolling. Oxidation and burning of the foils during the rolling were the main difficulties. It was noticed that, the burning of foil can occur due to an electric discharge as can be observed between the foil and the forceps while handling the foil. The electric charge accumulated due to the friction on the surface of foil by the hard rollers of the rolling machine. Usage of an anti-static wrist strap was very effective in protecting the foils from burning. An uninterrupted flow of



Fig. 1 Molybdenum metal bead (*left*) and rolled foil (*right*)



Fig. 2 Rolled Ni foil

Table 1 Properties of thin Nifoils produced by cold rolling

Area (cm ²)	Thickness	
1.8 × 2.5	450 μg/cm ²	
2.5×2.0	540 μ g/cm ²	
5.6×2.5	680 µg/cm ²	
2.0×2.5	$700 \ \mu g/cm^2$	
2.1×2.7	810 μg/cm ²	
5.0×3.0	1 mg/cm^2	

argon in the surrounding of roller was helpful in minimizing the oxidation of target surface during rolling.

Ho, Tb, Tm, Gd, Nd, Sm, Ce, Pr, Sm, Dy, Yb and Lu targets in the order of $\mu g/cm^2$ thickness were frequently fabricated in IUAC by evaporation. In addition to Gd, rare earth elements like Ho, Tb and Tm targets in the order of mg/cm² have been fabricated by cold rolling.

Isotopic Sn targets by rolling

Sapir [25] has described the fabrication of Sn targets of 500 μ g/cm² thickness by rolling in triple sandwich of stainless steel, beryllium copper and wax paper or mylar. Manente et al. [26] have also reported the fabrication of Sn targets of 1 mg/cm² thickness by rolling in stainless sheet with silicone oil layer.

At IUAC, many targets of ¹¹⁶Sn, ¹¹⁸Sn, ¹¹⁹Sn, ¹²⁰Sn, ¹²²Sn and ¹²⁴Sn with thickness exceeding 1 mg/cm² as self-supporting foils as well as with backing have been fabricated by rolling. Stainless steel folders were used for thickness up to $\sim 5 \text{ mg/cm}^2$. Heating the sandwich steel folder and foil was helpful for the successful rolling of Sn foils. Since Sn foils started sticking to steel sheets at $\sim 5 \text{ mg/cm}^2$, stainless steel sheets were replaced by Teflon sheets for the further reduction down to $\sim 1 \text{ mg/cm}^2$.

Isotopic Sn targets by evaporation

Thin Sn targets of ¹²²Sn and ¹²⁴Sn of thickness not exceeding 1 mg/cm² were successfully fabricated by thermal evaporation in the form of self-supporting film and with backing. For self-supporting targets, 100 nm NaCl was deposited as a parting agent. NaCl and Sn are evaporated without breaking the vacuum, since degradation of NaCl film in the moist air reduces its solubility in water. Floating of Sn within few hours after the evaporation was also helpful for an easy separation of films from the glass slide. Thickness of self-supporting Sn targets fabricated by evaporation was $\sim 200 \ \mu g/cm^2$. The Sn targets were also made with thin carbon and thick gold backing. The thickness of Sn targets with backing varied from 200 to 700 μ g/cm². The distance between the evaporation source and substrate during the evaporation was maintained above 6 cm to minimize the transfer of heat from the source to substrate.

Isotopic W targets by evaporation

Thin targets of tungsten isotopes of ¹⁸²W, ¹⁸⁴W and ¹⁸⁶W were frequently fabricated in IUAC, but the process had drawbacks regarding the achievable minimum thickness and the reliability of the carbon backing foil [27]. Therefore we optimized our process. Now ¹⁸²W was directly evaporated by electron beam on the glass slides already coated with 25 μ g/cm² of carbon and 100 nm of BaCl₂ as a parting agent. Before and after the evaporation of W, the carbon coated glass slides were annealed at 325 °C in argon environment in order to relieve the stress from the foil. The glass slide having now films of W, C and BaCl₂ were floated in distilled water. Finally the tungsten film was mounted on a target frame. With this method more than 15 targets of ¹⁸²W of 100 µg/cm² thickness were prepared in a single evaporation run by using 80 mg of starting material.

Ta targets by evaporation

Several targets of Ta were fabricated and supplied for nuclear physics experiments in IUAC. Self-supporting Ta targets of 350-550 µg/cm² thickness and Ta targets of $250 \text{ }\mu\text{g/cm}^2$ thickness with carbon backing were successfully fabricated by electron beam evaporation in high vacuum environment. For self-supporting targets, Ta was deposited on copper foil of 1.5 mg/cm². A substrate heater was also used to maintain the copper foil at 300 °C. Ta coated copper foil was then cut into the sizes of the target frame and the copper was removed slowly by etching in a nitric acid solution having 1:3 ratios of nitric acid (assay 68-72 %) and deionized water. It was observed that many thin targets less than 500 μ g/cm² had short life time after
 Table 2
 Targets recently
fabricated at IUAC for nuclear physics experiments

Sl. No	Description of target	Method	Sl. No	Description of target	Method
1.	¹⁴² Nd, 250 µg/cm ²	E-Gun	15.	²⁰⁶ Pb, 100 µg/cm ²	Thermal heating
2.	¹⁵⁰ Nd, 250 µg/cm ²	E-Gun	16.	²⁰⁸ Pb, 100 µg/cm ²	Thermal heating
3.	¹⁸² W, 100 µg/cm ²	E-Gun	17.	²⁰⁴ Pb, 400 µg/cm ²	Thermal heating
4.	¹⁷⁶ Yb, 150 µg/cm ²	E-Gun	18.	¹⁴² Nd, 250 µg/cm ²	Thermal heating
5.	¹⁷⁴ Yb, 800 µg/cm ²	E-Gun	19.	¹²² Sn, 250 µg/cm ²	Thermal heating
6.	⁹² Zr, 300 µg/cm ²	E-Gun	20.	¹²⁸ Te, 400 µg/cm ²	Thermal heating
7.	⁹⁴ Zr, 520 µg/cm ²	E-Gun	21.	144Sm, 150 μg/cm ²	Thermal heating
8.	¹⁹⁸ Pt, 100 µg/cm ²	E-Gun	22.	¹³⁰ Te, 400 µg/cm ²	Thermal heating
9.	¹⁹⁴ Pt, 100 µg/cm ²	E-Gun	23.	¹⁶⁰ Gd, 500 µg/cm ²	Rolling
10.	¹⁹⁶ Pt, 200 µg/cm ²	E-Gun	24.	¹⁵⁴ Sm, 1200 µg/cm ²	Rolling
11.	¹⁸⁴ W, 200 µg/cm ²	E-Gun	25.	¹¹⁶ Cd, 1100 μg/cm ²	Rolling
12.	¹⁴⁸ Nd, 250 µg/cm ²	E-Gun	26.	⁹⁴ Mo, 1000 μg/cm ²	Rolling
13.	¹⁵⁰ Nd, 250 µg/cm ²	Thermal heating	27.	¹²⁴ Sn, 4000 µg/cm ²	Rolling
14.	¹⁵⁴ Sm, 150 μg/cm ²	Thermal heating	28.	-	_

mounting on the target frame. However targets of more than 500 μ g/cm² showed a long life time with high stability. Ta targets on a carbon backing were prepared by same procedure which was adopted for W target.

Pt targets by evaporation

Targets of ^{194,196,198}Pt had been fabricated for nuclear physics experiments in IUAC. Singh et al. [28] has reported the fabrication of self- supporting targets of Pt by evaporation and etching. Gursky et al. [29] and Singh et al. have also reported that KI is the most suitable releasing agent in Pt self-supporting target fabrication.

Since isotopic Pt is rather expensive, more work is continued to optimize the material consumption and success rate by evaporation. It was experienced that stress developed in the film was having prominent role in developing cracks and damages on self-supporting film. In the recent attempts, $300 \ \mu g/cm^2$ of Pt was evaporated by electron beam on a glass slide having 100 nm of BaCl₂ coating. The slides were annealed at 425 °C for 2 h in nitrogen environment in order to remove the stress from the foil. The annealed slides have shown significant improvement in floating and life of Pt targets. The targets of ^{194,196}Pt were also successfully prepared with thin C backing of 15 μ g/cm² by electron beam evaporation.

Summary

The target development laboratory in IUAC has fabricated and supplied many targets to nuclear physics research community in India. Table 2 shows some of the recently fabricated targets in IUAC. More works have done in the direction of improving or modifying the existing process to J Radioanal Nucl Chem (2015) 305:749-753

reduce the cost and increase the efficiency of target fabrication.

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