

# **Carbon-backed thin tin (116Sn) isotope target fabrication by physical vapor deposition technique**

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#### **Abstract**

In nuclear reaction experiments, the thin targets are required. In the present work, 30 thin <sup>116</sup>Sn targets were prepared using physical vapor deposition technique (preferable for thin film fabrication) on carbon backing with usage efficiency of 98%. The carbon-backed thin target flms along with the parting agents are deposited on the particular substrates using a difusion pump based coating unit. The thicknesses of the targets were verified using  $\alpha$ -energy loss and RBS technique and they were in good agreement with each other. The purity of the target, verifed using RBS, EDS and XRD techniques, were also tested with confrmation.

**Keywords** Physical vapor deposition · Carbon-backed 116Sn target · Rutherford backscattering spectroscopy · Energy dispersive X-ray spectroscopy · Thin flm · X-ray difractometer

### **Introduction**

The nuclear reaction requires a projectile beam and the target of very good quality with uniform and small thickness, good tensile strength, good adhesion between the thin flm and the substrate, low enmesh of gas and minimum contamination [\[1](#page-6-0)], which makes its fabrication quite arduous. For heavy ion induced nuclear reaction studies using mass analyzer, where the reaction products are separated from the beam like particles and disperses the nuclei of interest at its focal point with good mass resolution, thin self-supporting targets (few mg/cm<sup>2</sup> to some  $\mu$ g/cm<sup>2</sup>) are called for to obtain the best results [[2\]](#page-6-1). This will reduce the energy loss of projectile beam and the encapsulation of reaction products within the target. This will give clear spectrum with good

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segregation of various reaction products. In order to measure fusion excitation function precisely and to determine barrier distribution, small energy steps are necessary. And the energy lost by the incident beam must be lesser than the energy steps. To prepare such targets, thermal evaporation (one of the types of physical vapor deposition) is the most effective method  $[3, 4]$  $[3, 4]$  $[3, 4]$  $[3, 4]$ . But due to difficulties in obtaining such thin self-supporting target foil (mostly for high Z elements), target materials are evaporated on a thin lower Z backing material, viz., carbon foil ( $\approx 30 \mu g/cm^2$ ) which will minimize the energy loss and energy straggling effects of the projectile beam. Carbon remains inert with most of the metals, thereby allowing it to be the frst choice as the backing material.

Diferent ways of fabricating Sn targets are already being carried out. Long back, Zell  $[5]$  $[5]$  prepared Sn ( $\sim$  1 to 2.5 mg/ cm<sup>2</sup>) target on Bi (~50 to 75 mg/cm<sup>2</sup>) backing by rolling and on Cu ( $\sim$  20 mg/cm<sup>2</sup>) backing using evaporation technique. In another case, Manente and Pengo [[6](#page-6-5)] and Abhilash et al. [[7\]](#page-6-6) prepared self-supporting Sn ( $\approx$  1 mg/cm<sup>2</sup>) target using rolling techniques. Sood et al. [\[8](#page-6-7)] could fabricate self-supporting thin  $\frac{116,118}{5}$ Sn films (250–600 µg/cm<sup>2</sup>) using the resistive heating method. Singh et al. [[9\]](#page-6-8) reported the fabrication of gold-backed Sn targets of thickness  $(0.5-2 \text{ mg/cm}^2)$  using rolling technique and evaporation technique. Abhilash et al. [\[7](#page-6-6)] and Sharma et al. [[10\]](#page-6-9) prepared thin carbon backed <sup>122</sup>Sn

targets using the resistive heating method with parting agent NaCl and BaCl<sub>2</sub> respectively. Giri et al.  $[11]$  $[11]$  and Ali et al. [\[12](#page-6-11)] fabricated <sup>124</sup>Sn target on Aluminium backing and <sup>112</sup>Sn target on Lead backing respectively. Pai et al. [[13](#page-6-12)] prepared the self-supporting and lead-backed  $112$ Sn target but those were thicker one prepared for in-beam  $\gamma$  ray spectroscopy experiments. In this paper, preparation of a large number of carbon backed thin enriched <sup>116</sup>Sn ( $\approx$  150 µg/cm<sup>2</sup>) target with KCl as parting agent, with only 36 mg material, using physical vapor deposition technique (ultra-high vacuum thermal evaporation) has been reported along with the essential steps and the precautionary measures reckoned for successful fabrication.

For fusion excitation function measurement in the subbarrier region using recoil mass spectrometer through Heavy Ion Recoil Analyzer (HIRA) [\[14](#page-6-13)] at Inter University Accelerator Centre (IUAC), New Delhi, India, <sup>116</sup>Sn targets were fabricated. 100 mg of 99.6% enriched  $116$ Sn isotope was imported from Oak Ridge National Laboratory, Tennessee, USA, which was in the form of thin metal fakes. Due to the expensive and limited isotope material, direct depositions and hence, direct isotope flm fabrication were eschewed. Alternatively, prior to isotope deposition, some trials were done with the natural Sn (which was in the form of metal shots) to calibrate and optimize the various parameters required for its fabrication process. We went for the fabrication of self-supporting thin flm using natural Sn which is ideal in our case. But the effort to produce it was futile due to the obstacles faced during foating of the evaporated target film on the stainless steel target holder as the film so produced was inconsistent. It lasted for the transient time. This led us to go for evaporation of the material on some appropriate backing which is chosen to be carbon. This eliminates the risk factor of fabricating unstable target as the available isotope is very limited. The deposition of carbon was carried out inside the difusion pump based coating unit (DP) with a vacuum around 10−6 mbar. The deposition of Sn-isotopes over the carbon backed substrate was carried out inside the turbo-pump based coating unit (TP) with a vacuum of the order of  $10^{-9}$  mbar. In the following sections, the description of the experimental setup, fabrication processes and the characterization techniques are discussed along with the results and conclusions.

#### **Experimental methodology**

Here we briefy discuss the experimental set up used in our work and then present a complete fabrication procedure of carbon-backed thin tin isotope by thermal evaporation method which involves the following steps: fabrication of carbon backing followed by that of tin isotope over the carbon flm. The C-backed tin isotope flm is then foated which is also described below.

### **Physical vapor deposition setup**

Physical vapor deposition (PVD) technique is one of the important and efective methods for the thin flm fabrication involving heavy ion beams. PVD is a coating technique in which the material to be deposited is transferred from the source to substrate on an atomic level under vacuum. During evaporation the material is bombarded by a high energy beam of electrons/ions which dislodge the atoms from its surface. The vaporized atoms are then transported in straight line to the substrate. The metal atoms react with gas while being transported which will not be the case in vacuum condition. The deposition then takes place due to which coating build up on the surface of the substrate. The images of DP and TP in the target laboratory of IUAC, one of the main facilities for the fabrication of nuclear targets in the country, can be seen in Ref. [\[15,](#page-6-14) [16\]](#page-6-15). Banerjee et al. [[1\]](#page-6-0) gave their schematics in detail. DP is connected with a difusion pump, 2 kW single pocket electron gun (e-gun) and resistive heating arrangement [\[15](#page-6-14)]. Liquid nitrogen trap is ftted between the chamber gate valve and the difusion pump to condense oil molecules of difusion pump from moving towards chamber. This gives better vacuum pressure. TP is connected with a turbo molecular pump, a scroll pump and 6 kW multi-pockets e-gun [\[16](#page-6-15)]. Chilled water is circulated throughout the chambers to maintain them at room temperature. A quartz crystal (Edwards FTM 5 crystal with 6 MHz as its unloaded frequency) thickness monitor [[17\]](#page-6-16) was kept inside both the chambers to monitor the deposition of thin flms with time.

#### **Fabrication of carbon backing**

Before going for the carbon  $(C)$  deposition, which is the first step towards preparation of carbon-backed targets, a parting agent deposition is required. A parting agent selection is very important as it helps in separating the target flm from the substrates after deposition. Moreover, the parting agent should not contribute in contamination or degradation of the isotopic flm. Braski [[18\]](#page-7-0) found that alkali halides are more efective as a parting agent than others by using carbon replicas for comparing surface structure and separating ability of diferent parting agents like alkali halides, Victawet, detergents, plastics. In the present case potassium chloride (KCl) is considered to be the most reliable and eligible parting agent, (unlike in Refs. [\[7](#page-6-6), [10\]](#page-6-9)) because the diference of K mass and Sn mass is enough to separate its energy spectrum which will ease the analysis work after the experiments, provided if K is responsible for the contamination on the metal. Moreover, regarding other metal halides, the degradation of NaCl flm in the moist air reduces its solubility in water [[7](#page-6-6)], CsI being unstable cannot be used here [[1\]](#page-6-0) and  $BaCl<sub>2</sub>$  leads to more contamination with carbon film compared to KCl [[19\]](#page-7-1). To begin with, DP chamber is thoroughly cleaned with propanol. A thin pellet of KCl powder is prepared using hydraulic press and is enclosed in the DP chamber along with a piece of graphite rod (carbon source) of approximately 5 mm diameter simultaneously. KCl is kept on rectangular Molybdenum boat [[15\]](#page-6-14) and graphite is kept on the water cooled Copper crucible. 120 nm thickness of KCl was deposited on the clean glass slides (which is taken as the substrate), kept on the dice fastened at 18 cm from the source, using resistive heating arrangement, after acquiring the vacuum of  $10^{-6}$  mbar. The deposition was done slowly with the rate of around 0.1 nm/s at 188 A current and 1 V voltage. After about 15 min, C of thickness  $\approx$  30 µg/cm<sup>2</sup> was deposited over KCl on the slides (kept at 17 cm away from the C source) by e-gun without disturbing the vacuum with 0.1 nm/s at 220 V voltage and 120 mA current. The current was increased gently from 0 to 55 mA for outgassing process. At 55 mA the deposition commenced following which the current was increased gradually till 120 mA to minimize the internal stress. E-gun is used for C because its melting point is very high (3550 °C). In the DP chamber, the sequential deposition of KCl followed by carbon was done on the substrate without splintering the vacuum to prevent the degradation of the parting agent due to the moisture in the air, if any. After cooling the chamber, it is vented and the C slides were taken out. They were then annealed in a tubular furnace at a KCl annealing temperature of 250 °C for 1 h in the Argon gas environment to relieve the internal stress developed during flm growth due to various reasons like lattice misfts, chemical interaction with the substrate, the adhesive force acting between the evaporated materials and the substrate or the thermal expansion of the flms and the substrate [[20](#page-7-2)]. Annealing is followed by foating where one of the slides is foated to separate it from the C layer by dissolving the parting agent through the slide. Floating was successful (the flm so obtained resembled exactly as is seen in Ref. [[15\]](#page-6-14)) due to which the remaining carbon slides were used for further material deposition. We strictly adopted the foating procedure described properly by Banerjee et al. [[1\]](#page-6-0) pictorially.

# **Fabrication of thin natural Sn on carbon backing**

Using propanol again, the TP chamber is thoroughly cleaned in which the carbon deposited slides were kept on the dice clamped at 5 cm from the source with the carbon surface facing it. The quartz crystal was adjusted at 5 cm from the source to make the tooling factor unity. Tooling factor is the ratio of substrate distance from the source to the crystal distance from the same source. This will help in monitoring the deposition thickness on the substrate accurately. The source, that is, natural Sn, is kept on the annular Tantalum boat of height 4 cm [[21](#page-7-3)]. Such boat, having lesser solid angle, will allow limited deposition of the source efectively which will reduce the wastage of expensive isotopic source. With the vacuum of  $10^{-9}$  mbar, the deposition of 35 mg of the natural Sn was done using thermal evaporation with resistive heating technique till the desired thickness of around  $150 \mu g/cm^2$ is obtained. The deposition rate was kept around 0.1 nm/s at 250 A current and 1 V voltage. The current was increased slowly from 0 to 180 A where the evaporation started. From 180 A, the current is slowly increased till 250 A and is then kept constant following which the deposition became steady. After completion of the deposition, the chamber is then vented after about 4 h and then it is opened. The glass slides were taken out. Floating was done in warm distilled water as discussed in Ref. [[1](#page-6-0)]. Sn layers then foats on the water. From the water surface, using target holder of 1 cm diameter hole, the flm layer is then grabbed. The carbon-backed natural Sn target fabrication, therefore, was successful. The natural target flms so obtained were intact, consistent and stable. The parameters used for fabrication are then noted down. The contriving procedure used here is diferent from that of the conventional technique [\[2](#page-6-1)], where the carbon flm is foated frst. The carbon flm was then grabbed by the appropriate target holder followed by the deposition of the isotope over the carbon flm. But in the present procedure, the risk of the carbon flm rupture due to the mechanical disturbance or any kind of bad handling is minimized considerably, thereby reducing the wastage of an isotope. In this method foating is done only after complete deposition of isotope over the carbon flm within the substrate itself.

# **Fabrication of thin 116Sn isotope on carbon backing**

Following the successful fabrication of the natural Sn targets and using clean TP thermal evaporation, 36 mg of 116Sn was deposited on the fresh annealed C slides, keeping all the parameters same as discussed in the previous section. <sup>116</sup>Sn was deposited till the thickness reading in the crystal monitor was around  $150 \mu g/cm^2$ . After evaporation, the chamber was left for few hours for cooling followed by venting after which the deposited material was taken out. The foating was successfully done, and the targets of required thickness were obtained which is displayed in Fig. [1](#page-3-0).



**Fig. 1** The target holders containing stable  $116$ Sn film

# <span id="page-3-0"></span>**Characterization of target**

After fabrication of the targets, their thickness and purity need to be verifed. Using various techniques, the target thickness is verifed. To check its purity, the targets are characterized. This is very much essential for the nuclear reaction experiments.

# **Alpha particle energy loss technique**

In this technique, 5.486 MeV alpha (α)–radiation from a strong 50  $\mu$ Ci radioactive <sup>241</sup>Am source is transmitted through the foil [\[22\]](#page-7-4). The energy of the radiation will then be reduced from which the target thickness can be estimated. The energy of alpha passing through the flm is recorded in the U126 CANBERRA MODEL silicon solid-surface barrier detector kept on the opposite side of the target flm in vacuum. As the target in this case is carbon backed, so we frst measured the thickness of the carbon flm followed by the carbon backed target flm. By measuring the diference in the centroid of the peak obtained from the energy spectrum of the alpha particles from the carbon and that of the background, the energy loss through the carbon foil was determined [\[15](#page-6-14), [23](#page-7-5)]. Similarly, the energy loss through the carbon-backed <sup>116</sup>Sn target was measured separately and then the calculation was corrected with the energy loss due to carbon foil. The energy loss of α-particles in the  $116$ Sn targets and the carbon foils were extracted from the SRIM code [[24](#page-7-6)].

# **Energy dispersive X‑ray spectroscopy (EDS) measurement**

The involvement of other non-desirable material as impurities is tested using this technique. It is a qualitative and quantitative X-ray micro-analytical technique used



<span id="page-3-1"></span>**Fig. 2** The energy spectra of  $\mathbf{a}$   $^{116}$ Sn and **b** carbon with the energy channel along the *x*-axis and the counts of event along the *y*-axis

under scanning electron microscope (SEM) to determine the elemental concentrations in the sample. The EDS measurement was done for  $116$ Sn target at IUAC using feld emission SEM (FESEM) facility of model number FESEM—JOEL's JSM-7610F and EDS model used is EDAX with Peltier cooled—silicon-drift detector (SDD) [[25](#page-7-7)]. SDD in this setup is of Octane series having excellent typical energy resolution of 129 eV at 5.9 keV of Mn line [\[19](#page-7-1)]. The setup is also equipped with user friendly and compatible analyzing software. The present measurement was performed with the low current of 9 nA. The chamber vacuum was of the order of  $10^{-5}$  Pa. This measurement was done by irradiating the sample with 5 kV electron beam energy. The spectra of the samples so obtained are illustrated in Fig. [2](#page-3-1)a, b. The letters K, L and M in the spectrum are the wavelength of the characteristic X-rays according to the electron jump that takes place within the atoms.

The surface topologies of  $116$ Sn target and the carbon film, measured with scan rate being  $6.4 \text{ s}^{-1}$ , are shown in Fig. [3](#page-4-0)a,



**Fig. 3** The surface morphology of  $\mathbf{a}$  <sup>116</sup>Sn and **b** carbon films

<span id="page-4-0"></span>b respectively. These SEM images ensured the absence of any other impurities within the flm.

# **Rutherford back‑scattering (RBS) characterizations**

RBS is the only technique which analyzes the test target flm quantitatively without the application of any standard reference target [[26\]](#page-7-8). This technique is an elastic collision between the energetic projectile  $(2 \text{ MeV } \alpha\text{-particles from})$ 1.7 MV 5SDH-2 tandem accelerator at PARAS, IUAC in our case) and the fxed target nuclei. After collision with the target, α-particles is then backscattered which allows us to determine atomic mass and elemental concentration against the depth below the surface. The backscattered particle is detected using Silicon surface barrier detector (SSBD) of 3.6 mSr solid angle and 500 µm effective area of silicon crystal kept at around 165° with respect to the principal axis of



<span id="page-4-1"></span>**Fig. 4** The RBS spectrum of <sup>116</sup>Sn

beam transmission. Energy yield and spectra from SSBD is then analyzed using RUMP simulation software [[27\]](#page-7-9). RBS spectrum so obtained is shown in Fig. [4](#page-4-1).

### **X‑ray difractometer (XRD) characterizations**

XRD is an analytical technique to identify the crystalline material. Here,  $116$ Sn targets were analyzed qualitatively, as shown in Fig. [5,](#page-5-0) using EMPYREAN—011220659 model XRD by PANalytical with Cu-K $\alpha_1$  source ( $\lambda$  = 0.154 nm) at XRD laboratory, Department of Instrumentation and USIC, Gauhati University. The XRD generator settings were kept at 40 mA current, 45 kV voltage and 25 °C temperature. The XRD setup is equipped with user friendly ideal software for phase identifcation—'HighScore plus' inbuilt from PANalytical with PDF-4 (Powder Difraction File) database maintained under ICDD (The International Centre for Diffraction Data).

### **Results and discussion**

Thin, uniform and pure targets of  $116$ Sn are required for the nuclear reaction studies. Due to the limited availability of the expensive isotope, the condition for the preparation is optimized frst with the help of the natural element of the same atomic number. Using the same parameter and the procedure, the isotope 116Sn deposition was done. Successfully the target flm was obtained. 30 number of such targets could be extracted carefully. The flms prepared, therefore, need to be verifed for thickness and purity. Using alpha energy loss technique and RBS technique, the thickness were

<span id="page-5-0"></span>



determined which are found to be (within the experimental uncertainties) in close agreement with each other along with the thickness obtained during deposition process by the crystal monitor in the coating units. All the values are charted in Table [1.](#page-5-1) The thicknesses calculated accordingly using alpha energy loss technique are found to be  $\approx 30.05 \pm 0.90$  µg/cm<sup>2</sup> for carbon and  $\approx 150.98 \pm 3.10 \,\mu g/cm^2$  for  $^{116}Sn$ . The thickness value calculated using this technique and presented in the Table [1](#page-5-1) is of the average result of 30 targets so obtained. The reason for uncertainty here is mainly the source-targetdetector geometry. The thickness of the target is further calculated using RBS technique. The thickness so obtained from this technique is found out to be  $\approx 147.2 \pm 1.2$  µg/ cm<sup>2</sup> . Hence, the mean value of the thicknesses calculated by averaging the thickness values obtained from these two techniques along with the crystal monitor readings comes out to be  $149.39 \pm 1.92 \,\mu g/cm^2$  for carbon-backed  $116$ Sn film and  $30.03 \pm 0.44$  µg/cm<sup>2</sup> for carbon film. As many as 30 such isotopic target flms were obtained from the substrates where deposition was done over the surface of area  $235 \text{ cm}^2$ which gives usage efficiency of 98%. Such thin targets can withstand a beam intensity of  $\sim$  1 pnA during the experiment and maintain a good spectroscopic resolution.

After confrming the target thickness, the purity of the target flms are further checked with the help of three techniques—the EDS, the RBS and the XRD techniques.

Figure [2](#page-3-1)a, obtained using EDS technique, indicates the presence of Sn element along with some fractional amount of oxygen and the backing carbon. No other impurities are found in the target material. The EDS measurements are done even for the carbon flm which did not revealed any impurities within it as is shown in Fig. [2b](#page-3-1). Also SEM pictures (Fig. [3](#page-4-0)a, b) could not detect any impurity in the flm. In the RBS spectrum (Fig. [4](#page-4-1)), three peaks of carbon  $(C)$ , oxygen (O) and tin (Sn) are there. C is because of backing element; O indicates the oxidizing of the target which is very minimal indicating negligible amount as impurity content. No other signifcant high or low Z contaminants are seen in the spectra. The bulk of compound nucleus leading to the desired spectrum will be mainly due to the contribution of Sn. The difraction peaks obtained using XRD technique are almost sharp indicating the good crystalline of the target and low residual strain in most orientations. These sharp peaks were obtained without any refnement on the measured data. The XRD peaks along with the miller index (ICDD Card Nos. 04-7744 and 07-4906) corresponding to each peak [\[28](#page-7-10)–[30\]](#page-7-11) within the XRD detection limit are shown in Fig. [5.](#page-5-0) The (220) and (211) peaks in the XRD plots are relatively broad which is due to peak overlap. Also, a little hump is seen near 25° (2*θ*) which is due to the presence of carbon  $[31]$  $[31]$  and it is due to the

Film	Density (gm/ $\text{cm}^3$	Melting point $(^{\circ}C)$	Thickness ( $\mu$ g/cm <sup>2</sup> ) using			Mean thick-
			Crystal monitor	$\alpha$ -energy loss	<b>RBS</b>	ness $(\mu g)$ $\text{cm}^2$
$Natural C$ (graphite)	2.3	3550.0	30.0	$30.1 \pm 0.9$		$30.0 \pm 0.4$
$Natural C$ (amorphous)	1.8					
$116$ Sn	7.3	231.9	150.0	$150.9 + 3.1$	$147.2 + 1.2$	$149.4 \pm 1.9$

<span id="page-5-1"></span>**Table 1** Chart of the target thickness obtained from various characterizations

refection of plane (002). No other elements are present in the spectrum indicating the purity of the target flm.

Hence, the target developed is free from any substantial impurities in this work. Apart from the carbon, which is the backing foil, and a slight amount of oxygen, all the techniques used in the present work could not detect any other contaminants in the notable amount in the flm prepared indicating their purity. The targets are impurity free as these impurities might contribute to spurious reactions with inclusion of arbitrary energy losses thereby leading to large uncertainties in the masses of the reaction product. Therefore, these target flms can be considered as the suitable product for the experiments to be performed in future. 30 numbers of such target flms are prepared which is then kept in the desiccators properly for the upcoming experiments. In the whole procedure, it is to be noted here that the target prepared for fusion is very thin. Moreover, the targets are successfully prepared with very less amount of such expensive isotope. Using this process, the targets prepared were found to be uniform and pure.

# **Conclusions**

With usage efficiency of 98% of enriched  $116$ Sn isotope, as much as 30 thin and uniform carbon-backed targets of  $116Sn$ is fabricated. Although it is preferable, but self-supporting targets could not be prepared because of its inconsistency and due to which carbon-backed targets were considered for preparation using thermal evaporation or resistive heating method. Following the fabrication of the targets, their thicknesses are verifed using the alpha energy loss technique and the RBS technique. The thickness values are  $\approx 149.39 \pm 1.92$  µg/cm<sup>2</sup> for Sn target and the thickness of carbon-backing is  $\approx 30.03 \pm 0.44$  µg/cm<sup>2</sup>. EDS and RBS techniques were then used to verify the uniformity and the purity of the fabricated targets. From EDS result, it was found out that the surface topology is uniform and the negligible amount of impurity is sustained by the target material. Even from the RBS spectrum, same conclusion is drawn. These targets are, therefore, the feasible option for the nuclear reaction studies.

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