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Characterization of LiF thin layer by nuclear reaction techniques

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HIGHLIGHTS

- A thin LiF target was fabricated by thermal evaporation technique onto self-supporting Ag film.
- The thickness and the stochiometric ratio of the thin LiF target was measured using EBS, PIGE, and NRA techniques.
- The values of the thickness of the target obtained by these techniques are in good agreement.
- All results are presented in an absolute approach by using the experimental cross-section values.

ABSTRACT

To measure excitation functions of particle-induced prompt gamma-ray production reactions on Li, a thin LiF target was fabricated by thermal evaporation technique onto self-supporting Ag film. The thickness and the 'stochiometric' ratio of the thin LiF target was measured using Elastic 'Back scattering' Spectroscopy (EBS), Particle Induced Gamma-ray Emission (PIGE), and Nuclear Reaction Analysis (NRA) techniques. The target was characterized to check uniformity and its stability under beam bombardment. Carbon and molybdenium contamination in the target also were examinated. The values of the thickness of the target obtained by EBS, PIGE and NRA techniques are in good agreement with each other, within the estimated uncertainties. Measurements were conducted using the proton/deuteron beams of the 3 MV 'Van de Graaff' electrostatic accelerator of Nuclear Science and Technology Research Institute (NSTRI). All results are presented in an absolute approach by using the experimental cross-section values available through the Ion Beam Nuclear Data Library (IBANDL).

KEYWORDS

Thin LiF target
Thermal evaporation technique
Target characterization
Nuclear reaction analysis

HISTORY

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1 Introduction

Lithium fluoride (LiF) is an interesting material because of its low refractive index and large band gap (Damache et al., 2013). LiF has widely been used in many fields of applications, for instance in optic physics (Hennessy and Nikzad, 2018) and in nuclear physics as a thin-film detector and as a radiation dosimeter (Montereali et al., 2010).

LiF thin target also commonly is used for measuring the Li and F cross section data in low-energy charged particle beams because of its high stability under bombardment with high intensity ion beams (Abriola and Pedro de Jesus, 2011). Since more excitation functions of particle-induced prompt gamma-ray production reactions consist of the several narrow resonances, the energy loss of projectile beam into the target should be smaller than the energy separation of the neighboring resonances. Therefore, the target thickness plays a crucial role in the quality

and reliability of the experimental gamma-ray production cross section data (Noor et al., 2021). The target thickness should also be uniform if the beam spot has a smaller diameter than the target (Deb et al., 2019).

A search of the literature revealed that for measurement of cross sections of proposed reactions of $^7\mathrm{Li}(\alpha,\alpha\gamma)^7\mathrm{Li}$ ($E_r=1900\pm10$ keV, $\Gamma=130\pm30$ keV and $E_r=2480\pm50,~\Gamma=150\pm40$ keV) (Ajzenberg-Selove and Busch, 1980) and $^7\mathrm{Li}(p,p\gamma)^7\mathrm{Li}$ ($E_r=1030\pm5$ keV, $\Gamma=168\pm1$ keV) (Ajzenberg-Selove, 1984), LiF target thicknesses less than 5.2×10^{18} atoms.cm $^{-2}$ ($\sim120~\mu\mathrm{g.cm}^{-2}$) are required.

The use of nuclear reactions induced by low-energy charged particles provides several most suitable techniques to analyze thin films in the range of tens to hundreds of nanometers thickness. Among these techniques EBS, PIGE and NRA methods are preferably used, due to their high analytical power for accurate and simultaneous de-

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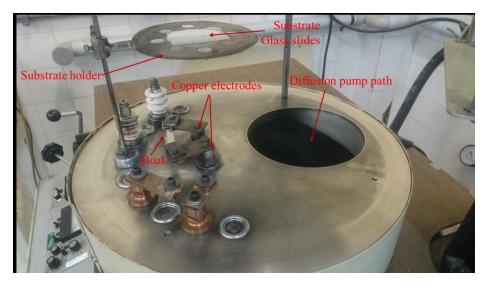


Figure 1: The inside view of thermal evaporation chamber.



Figure 2: a) Floating the Ag film on water, b) picking up the Ag film from the water surface and the Ag film on the target holder.

termination of several light elements (Nastasi et al., 2014).

LiF thin layers have been deposited mostly by physical vapor deposition (PVD) methods such as thermal evaporation or electron beam evaporation (Kumar et al., 1997). It should be noted that when chemical compounds are evaporated the elemental composition and stochiometry of the deposited layer may be not the same as those of the original compound.

The aim of this research is to determine the thickness and the stochiometric ratio of the thin LiF/Ag target fabricated by thermal evaporation technique, using proton/deuteron induced nuclear reactions. This research also presents a comparison among the results obtained by EBS, PIGE and NRA techniques.

2 Materials and Methods

2.1 Target Preparation

Thermal evaporation chamber set-up used for fabrication of the LiF/Ag target can be seen in Fig. 1. The material are kept on a suitable boat while its ends were screw tightened to the Copper electrodes. An optimized current is passed through the electrodes till all the material inside the boat got evaporated. In this high vacuum evaporation system, the target material with low melting point such as LiF and Ag can be evaporated by resistive heating technique. The chamber is equipped with a diffusion pump

and a rotatory oil pump used as the backing. Liquid nitrogen (LN2) trap is fitted between the chamber gate valve and the diffusion pump to condense oil contamination generated by diffusion pump. A pressure around 1×10^{-6} mbar can be achieved in the chamber.

In order to remove uncertainties due to improper charge collection during cross-section measurments the LiF was evaporated on a high atomic number self-supporting film (as Ag film) for charge normalization purposes (Jokar et al., 2016). It is worth mentioning that within the energy range available at the NSTRI 'Van de Graaff' accelerator, the elastic scattering cross section of incident particles by Ag nuclei is purely Rutherford.

To begin with, fabrication of self-supporting Ag film was done. Clean glass slides were taken and placed into a beaker containing homogeneous solution of distilled water and colorless and dense dish washing liquid. In order to make a thin and homogeneous dish washing liquid on slides, the beaker was fitted into a ultrasonic bath for about 20 minutes. Then dish washing film was allowed to dry in the fresh environment for about 30 minutes. It should be noted dish washing liquid will act as parting agent in this case as it is readily soluble in water. After being completely dried, the coated glass slides were kept on the substrate holder in the chamber at a distance of 20 cm from the source (see Fig. 1). Afterwards, 55 mg of the high-purity Ag was kept on the Tungsten boat of diameter

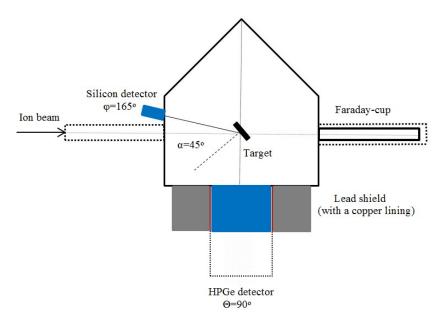


Figure 3: Schematic diagram of the experimental setup (not to scale).

3 mm and then thin layer of Ag was deposited on the opposite side of glass slides. After deposition, the deposited glass slides were taken out and immersed into warm distilled water gradually at 45° with the water surface (Fig. 2-a). So that the releasing agent is dissolved, allowing the Ag film to float on the water. Finally, With a target holder having 9 mm diameter hole, the floated film was gently picked up from the water surface (Fig. 2-b). In order to fabricate the LiF thin target on the self-supporting Ag film, Ag films mounted on target holders were kept on the substrate holder in the chamber at a distance of 12 cm from the source. 26 mg of the high-purity anhydrous LiF was kept on the Molybdenum boat of diameter 3 mm. The vacuum of the order of 10^{-6} mbar was obtained in the chamber and then the deposition of LiF was started using thermal evaporation technique. After evaporation, the prepared LiF/Ag targets were saved in vacuum chamber till implementing the target characterization.

2.2 Experimental procedure

The thickness and the stochiometric ratio of the thin LiF/Ag target fabricated by thermal evaporation technique was measured by EBS, PIGE and NRA techniques and then the target was characterized to investigate its uniformity, stability and purity. All measurements were carried out on the 45° right beamline of the 3 MV 'Van de Graaff' electrostatic accelerator of NSTRI. Our experimental setup includes a coaxial type HPGe detector at a right angle with respect to the beamline direction, a silicon charged particle detector at an angle of 165°, an isolated target holder and a Faraday cup electrically connected to the target to measure the incident beam current. The target was fitted in reaction chamber immediately after taking out of the thermal evaporation chamber in order to minimize contamination absorption during target handling. The target was oriented inside the reaction chamber so that the incident beam direction made an angle of 45° with the normal to the target. In reaction chamber a vacuum of the order of 1×10^{-5} mbar was achieved using a 'turbopump'. The beam spot size on the target was about 3 mm in diameter. Figure 3 shows a schematic diagram of the experimental setup. More description of the employed experimental setup can be found in Ref. (Jokar et al., 2016).

The incident beam current was selected 100 nA to keep the counting rate of 'HPGe' detector at about 1000 counts.s $^{-1}$. In this way the pile up was negligible and required correction for dead time of 'HPGe' and particle detector was less than 10% and 1%, respectively.

3 Results and discussion

The LiF/Ag thin target was characterized by implementing the EBS, PIGE and NRA techniques. The 'stoichiometry' as well as the thickness of the thin target were measured by applying simultaneously the EBS and NRA techniques with 2.2 MeV proton beam. By simulation of the spectrum with the SIMNRA code (Mayer, 1997) and experimental cross sections downloaded through IBANDL (www-nds.iaea.org/ibandl), composition of the LiF layer was obtained to be 45% natural Li and 55% F. The thickness of the Ag film was obtained to be (1.15 \pm $0.05) \times 10^{18}$ atom.cm⁻². The experimental spectum along with simulation result is presented in Fig. 4. The absolute number of Li and F atoms per cm² in the LiF layer was determined by employing the elastically back scattered protons from the ${}^{7}\text{Li}(p,p){}^{7}\text{Li}$ and ${}^{19}\text{F}(p,p){}^{19}\text{F}$ reactions and the alpha particles from the ${}^{7}\text{Li}(p, p\alpha_0)^{4}\text{He}$ and $^{19}{\rm F}(p,\alpha_{1,2})^{16}{\rm O}$ reactions. Table 1 presents the measured results from these reactions. As can be seen in this table, two independent analyses are in good agreement within regard to the uncertainties.

In Fig. 4, beside the elements referred above, a slight impurity of carbon is shown. The carbon peak can be

Table 1: Measured LiF thicknesses.

	EBS	NRA	p-PIGE	DIGE
Li $(10^{18} \text{ atoms.cm}^{-2})$				
$F (10^{18} \text{ atoms.cm}^{-2})$	2.63 ± 0.16	2.95 ± 0.24	2.84 ± 0.25	2.89 ± 0.26

attributed to carbon buildup on the target during measurements and carbon contamination during evaporation and floating process. Amount of carbon in target was determined to be less than 2%. It has to be mentioned that at low incident energies there are no interference between the gamma-rays originating from Lithium isotopes with the gamma-lines of isotopes of carbon contamination when the target is bombarded with light particles such as p/d/He. Hence, low amount of the carbon contamination is not a severe problem in the cross section measurements. As can also be seen in the Fig. 4 there is no sign of contamination of Molybdenum from the evaporation boat.

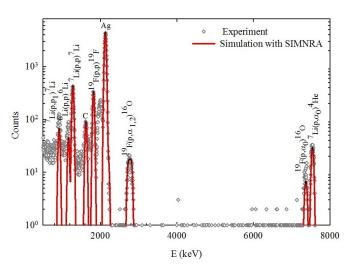


Figure 4: Experimental and simulated Spectrum obtained at 165° by bombarding the thin LiF/Ag target with $E_p = 2.2$ MeV. A distinct carbon contamination peak can also be seen. Amount of carbon in target was determined to be less than 2%.

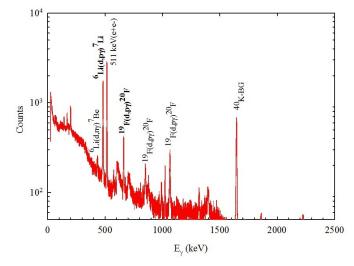


Figure 5: Gamma-ray spectrum collected at 90° by bombarding the thin LiF/Ag target with a 1.15 MeV deuteron beam.

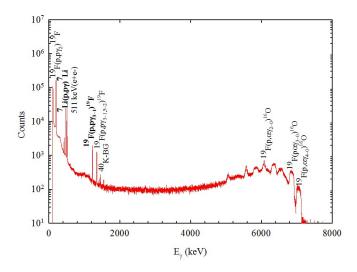


Figure 6: Gamma-ray spectrum of the thin LiF/Ag targe at 90° , bombarded with 2.4 MeV protons.

DIGE (Deuteron Induced Gamma-ray Emission) and p-PIGE (Proton Induced Gamma-ray Emission) measurements with the 1.15 MeV deuteron and with the 2.4 MeV proton beam were also performed on the thin LiF/Ag target. The experimental spectra are shown in Figs. 5 and 6 for $E_{d,lab}=1.15$ MeV and $E_{p,lab}=2.4$ MeV, respectively.

In the gamma spectra we focused on utilization of the intense prompt gamma-rays of 478, 656, 478, and 1236 keV from the respective nuclear reactions $^6\text{Li}(p,p\gamma)^7\text{Li}$, $^{19}\text{F}(d,p\gamma)^{20}\text{F}$, $^7\text{Li}(p,p\gamma)^7\text{Li}$, and $^{19}\text{F}(p,p\gamma_{3-1})^{19}\text{F}$. In Figs. 5 and 6, the gamma-rays corresponding to the studied reactions are labeled in bold. The results of the DIGE and p-PIGE measurements are also presented in Tabel 1.

All the values of the thickness of the thin LiF target obtained by EBS, NRA, p-PIGE and DIGE techniques are found to be in good agreement with each other which are tabulated in Table 1. The absolute concentrations of LiF layer obtained from these techniques are averaged, hence the concentration of Li and F are found to be $(2.36\pm0.19)\times10^{18}$ and $(2.83\pm0.23)\times10^{18}$ atoms.cm⁻², respectively. Based on SRIM calculations (Ziegler et al., 2010), the beam energy loss in LiF layer at $E_{\alpha}=2480$ and $E_{p}=1030$ keV are obtained to be 130 keV and 24.2 keV, respectively.

For the measured thicknesses, uncertainties are obtained considering the total uncertainties in the cross sections, in the stopping power for particles and in detector absolute efficiency for the gamma-rays.

In addition to measure the thickness and the stochiometry of the thin LiF film, the target was characterized to check uniformity and its stability under beam bombardment. The target uniformity was checked by changing the beam spot position for two different points on the target surface through the adjustment of target location. On the basis of comparison the gamma-ray and proton yields measured for two points, difference between yields were found be less 4%, so within estimated uncertainties. The gamma-ray yield measurements were repeated at 2.4 MeV proton with high beam intensity after finishing the target characterization to ensure the stability of the target. No sign of damage was observed in the target.

4 Conclusions

For nuclear reaction cross section measurements, a thin LiF/Ag target was prepared by thermal evaporation of the high-purity anhydrous LiF powder. The thickness and the stochiometric ratio of the fabricated LiF/Ag target was obtained using EBS, NRA, p-PIGE and DIGE techniques. The results of these techniques are consistent if the estimeted uncertainties are taken into account. The average of the absolute concentrations of Li and F obtained from these techniques were found to be $(2.36 \pm 0.19) \times 10^{18}$ and $(2.83 \pm 0.23) \times 10^{18}$ atoms.cm⁻², consisting of 45% natural Li and 55% F, respectively. The uniformity, the purity and the stability of the target were examined. From the results of the gamma-ray and the scattered proton yields obtained for two different points on the target surface, it was found that target thickness is uniform. The EBS measurements showed that carbon contamination exist within the target is less than 2%. The carbon contamination does not introduce significant error in the cross section measurements due to the gamma-rays interference. No other heavy and light impurities were detected. Target stability against beam bombardment confirmed by high beam intensity. The gamma-ray yield measurements did not show any damage in the target as an effect of beam bombardment. Therefore, the fabricated LiF/Ag target is most suitable for measuring the prompt gamma-ray production cross sections of Li isotopes.

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