Nuclear Instruments and Methods in Physics Research A 709 (2013) 72–75

Contents lists available at SciVerse ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

Preparation of ^{6}LiF deposits and characterisation via Monte Carlo simulations and Neutron Depth Profiling

R. Bencardino *, G. Giorginis, D. Sapundjiev

European Commission, Joint Research Centre, Institute for Reference Materials and Measurements, Nuclear Physics Unit, Retieseweg 111, B-2440 Geel, Belgium

ARTICLE INFO

Article history:
Received 19 April 2012
Received in revised form
19 December 2012
Accepted 31 December 2012
Available online 18 January 2013

Keywords:
^{6}LiF
Transparent backings
Monte Carlo simulations
Vacuum deposition
Neutron Depth Profiling

ABSTRACT

The Institute for Reference Materials and Measurements (IRMM) is measuring the ^{6}Li(n,t)^{4}He cross-section aiming at extending its status of standard over the MeV energy range. We developed a protocol to stretch-mount 0.75 μm, 1.5 μm, 8 μm, and 20 μm thick aluminium foils onto 0.5 mm thick tantalum rings. ^{6}LiF samples were produced depositing, by vacuum evaporation onto aluminium backings, a layer of lithium fluoride 95.5% enriched in ^{6}Li. We engineered dedicated tools and containers to handle and transport the resulting samples. These were characterised first at IRMM by differential weighing, then by Neutron Depth Profiling (NDP) at the TU Delft. These two measurements were found to be consistent for a selected sample, probed by a thermal neutron beam in three different regions to measure the ^{6}LiF layer thickness and uniformity (defined as variation of the thickness relative to its average). The latter was found to be 0.8%, and the ^{6}Li thickness to be 7.30 ± 0.12, 7.35 ± 0.12, and 7.29 ± 0.12 μg/cm^{2} in the three regions. We performed Monte Carlo simulations to estimate the uniformity of the ^{6}LiF layer, and benchmarked the calculation against the NDP measurements. They were consistent with respect to the deposit uniformity although the simulations were found to overestimate the thickness of the layer.

1. Introduction

The ^{6}Li(n,t)^{4}He reaction is relevant for the possible use as neutron beam monitor and breeder of tritium, and its cross-section is a standard for neutron energy up to a few hundred keV.

In the framework of the International Atomic Energy Agency (IAEA) Nuclear Data High Priority Request List (HPRL), the Nuclear Physics unit of IRMM is investigating the ^{6}Li(n,t)^{4}He reaction kinematics and measuring its cross-section in the MeV neutron energy region [1,2].

Cross-section measurements suitable for a standard need highly accurate preparation and characterisation of ^{6}LiF deposits. At IRMM, high quality ^{10}B and ^{6}LiF reference deposits were produced in the first half of the nineties for accurate neutron lifetime measurements [3] and high accuracy neutron counting [4]. Based on the experience of these successful campaigns, sets of ^{6}LiF deposits on transparent backing were recently prepared at IRMM by vacuum deposition via evaporation of 95.5% ^{6}Li-enriched lithium fluoride powder.

This work reports on the preparation of the substrates for the ^{6}LiF deposits, the process of material deposition, the characterisation of the deposits via Neutron Depth Profiling (NDP), and the benchmark of the experimental results against Monte Carlo simulations. The observable of interest in the calculation was the uniformity of the ^{6}LiF layer, defined as the percent ratio between the thickness variation across the deposit and its average thickness.

2. Experimental considerations

2.1. Sample backings

The sample backings consisted of aluminium foils stretch mounted onto tantalum rings. We selected four different foil thicknesses, namely 0.75 μm, 1.5 μm, 8 μm, and 20 μm, to be transparent to different extents to the ^{6}Li(n,t)^{4}He reaction products induced by 2 MeV incident neutrons [1]. The rings were 0.5 mm thick, having 5 cm and 7 cm inner and outer diameter respectively.

A protocol for mounting the foils onto the rings was developed to engineer a flat surface suitable for vacuum deposition of ^{6}LiF layers. It consisted in stretching the aluminium foil prior to gluing the rings. As shown in Fig. 1, the foil at ambient temperature was locked between pre-cooled PVC rings, then positioned onto a flat surface for one hour at ambient temperature to allow for thermal expansion. Afterwards, the tantalum washer was glued using epoxy glue onto the aluminium surface, which was flat-stretched against a PVC support. The application of a silver glue (E-solder 3021)

* Corresponding author. Tel.: +32 621517027.
E-mail address: Raffaele.BENCARDINO@ec.europa.eu (R. Bencardino).

0168-9002/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.nima.2012.12.125

shorting bar, in addition to the epoxy glue, ensured the electric contact between the foil and the washer.

The protocol was tuned using the coefficients of linear thermal expansion (TC) reported in Table 1. The stability of the coefficients over the operational temperature range ensured the functional equivalence between linear and areal thermal expansion. The PVC rings pre-cooling temperature was set to 20 °C lower than the ambient temperature. The expansion of the PVC rings was dominant over the deformations due to ambient temperature variations and foil initial cooling by the thermal contact with the PVC rings. This procedure guaranteed the optimal stretching of the aluminium foil.

The backings produced appeared to be stable yet delicate, especially the 0.75 μm thick ones. Therefore, dedicated sample handlers and containers were designed and fabricated. The handler was produced engineering specific compass pins as shown in Fig. 2, dedicated to fit into side grooves machined in the tantalum washers. The sample containers were grooved to limit pressure differences between the air volumes above and below the sample to avoid air suction.

Each sample backing produced was used as a substrate for the vacuum deposition of a layer of lithium fluoride, over one or more evaporation runs.

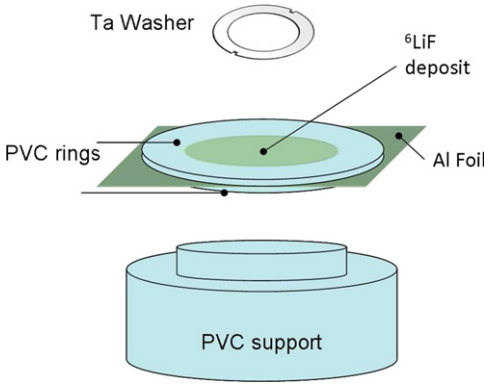

Fig. 1. Preparation of the sample backings.

Table 1
Coefficients of linear thermal expansion.

Material	TC (μm/mK)
Aluminium	23.1
PVC	50.4
Tantalum	6.3

2.2. Material deposition

A total of 15 layers of lithium fluoride were deposited by vacuum deposition via evaporation onto aluminium substrates. As reported in Table 2, pairs of layers were deposited during the same evaporation run (ER) when possible, while the thickest layer was deposited over three runs.

The ^{6}LiF source material, 95.5% enriched in ^{6}Li, was charged into a tantalum crucible. The experimental set-up is illustrated in Fig. 3. The crucible was heated by electric current to the ^{6}LiF evaporation temperature of 900 °C and monitored by a Raytek Marathon Infrared Pyrometer. The temperature throughout the crucible varied by 60 °C. The pressure of the chamber during the evaporation was 5.10^{-3} Pa, measured by a combined cold cathode/Pirani vacuum gauge. During the evaporation, the backings were rotated by an electric motor via a carousel. The deposition was controlled by an acoustic impedance quartz crystal microbalance deposition monitor [5] (see Fig. 3).

Table 2
^{6}LiF deposits produced at IRMM in 2010 by vacuum deposition onto aluminium backings.

Sample ID	ER	Al foil thickness (μm), $\delta_{rel} = 30\%$	^{6}LiF thickness (μg/cm^2)	^{6}LiF total mass (mg)
TP-2010-006-1	1	1.5	30.2 ± 0.2	0.21 ± 0.04
TP-2010-006-2	2	0.75	30.4 ± 0.2	0.31 ± 0.05
TP-2010-006-3	2	8.0	30.4 ± 0.2	0.31 ± 0.05
TP-2010-006-4	3	0.75	80.0 ± 0.2	0.49 ± 0.04
TP-2010-006-5	3	1.5	80.0 ± 0.2	0.54 ± 0.05
TP-2010-006-6	4	8.0	80.4 ± 0.2	0.54 ± 0.05
TP-2010-006-7	5	20.0	20.1 ± 0.2	–
TP-2010-006-8	6	20.0	50.3 ± 0.2	–
TP-2010-006-9	5, 6, 7	20.0	100.2 ± 0.2	–
TP-2010-006-10	8	0.75, broken	–	–
TP-2010-006-11	9	0.75	30.49 ± 0.14	0.17 ± 0.06
TP-2010-006-12	10	1.5	10.44 ± 0.14	0.18 ± 0.06
TP-2010-006-13	10	1.5	10.44 ± 0.14	0.11 ± 0.05
TP-2010-006-14	9	1.5	30.49 ± 0.14	0.20 ± 0.05
TP-2010-006-15	11	1.5	50.51 ± 0.14	0.47 ± 0.05

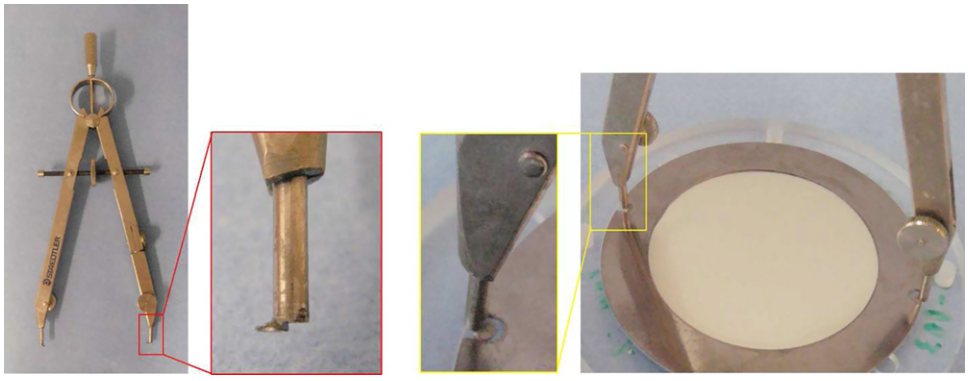

Fig. 2. Sample handling tool and container.