ISSN 0020-4412, Instruments and Experimental Techniques, 2008, Vol. 51, No. 1, pp. 147–149. © Pleiades Publishing, Ltd., 2008.
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Measuring the Lithium Layer Thickness

B. F. Bayanov[a], **E. V. Zhurov**[b], **and S. Yu. Taskaev**[a,b]

[a] *Budker Institute of Nuclear Physics, Siberian Branch, Russian Academy of Sciences, pr. Akademika Lavrent'eva 11, Novosibirsk, 630090 Russia*
[b] *Novosibirsk State University, ul. Pirogova 2, Novosibirsk, 630090 Russia*

Received May 3, 2007

Abstract—An epithermal neutron source based on a compact tandem accelerator is being built at the Budker Institute of Nuclear Physics for use in neutron capture therapy at a cancer clinic. It is expected that a lithium neutron-generating target will act as a neutron source. An original technique for determining the thickness of the lithium layer is proposed, and the measured radial distribution of the evaporated lithium layer thickness is presented.

PACS numbers: 06.30.Bp

DOI: 10.1134/S002044120801020X

Clinical trials of the neutron capture therapy of malignant tumors [1] on nuclear reactors have demonstrated its considerable promise [2]. A compact source of epithermal neutrons is currently being developed by the Budker Institute of Nuclear Physics (Novosibirsk, Russia) on the basis of a proton accelerator [3] for use in a cancer clinic. Neutrons are generated in the threshold reaction ^{7}Li(p, n)^{7}Be upon spill of the proton beam with an energy of 2.0–2.5 MeV on a lithium target. The target is a water-cooled thin metal disk [4] with a thin lithium layer applied on the proton beam side. Protons with initial energies of 2.5 and 1.915 MeV are decelerated to a threshold neutron generation energy of 1.882 MeV in lithium layers 88 and 4.3 μm thick, respectively [5]. Therefore, for neutrons to be generated, it is necessary that the thickness of the deposited lithium layer range between 10 and 100 μm. The use of a thicker layer results in an increased sublimation of lithium (which is dangerous through propagation of the radioactive beryllium isotope produced in the reaction) and a substantial increase in the concomitant undesirable flux of 0.477-MeV γ rays produced by inelastic proton scattering from lithium nuclei.

In this paper, we propose a new technique for determining the thickness of the lithium layer and present the measured radial distribution of the layer thickness over the target.

LITHIUM EVAPORATION

A lithium layer is evaporated onto a target by means of a special device designed on the basis of a commercially produced valve. A heater is placed upon a valve plate; a circular-slot evaporator, which houses a container with lithium, is installed under the plate. Before the evaporation, the valve plate is inserted into the proton guide and, using the valve sealing mechanism, is shifted by 5 mm toward the target. When shifted, the plate presses against the bellows, which limits the evaporation volume. Then, the heater is turned on, and evaporation is effected. Since the bellows is heat-insulated and its temperature during evaporation is high, lithium does not deposit on it. Lithium is evaporated only onto the target, which is cooled with water.

A TECHNIQUE FOR MEASURING THE LITHIUM LAYER THICKNESS

A special technique has been developed to measure the radial distribution of the evaporated lithium layer thickness. The need for this technique is dictated by two facts. First, lithium is a very active metal, which promptly forms compounds after being placed in an air medium. Second, most standard techniques for measurements in vacuum are inapplicable in our case, since a hot evaporator is located in the immediate vicinity of a cooled target. For example, it is impossible to use the well-known method for determining the mass of a lithium sample evaporated on a piezoelectric ceramic substrate from a change in the natural frequency of the ceramics, because the process of evaporation onto ceramics differs substantially from the evaporation onto a cooled substrate of the target in consequence of a significant difference in temperature.

The idea behind the proposed technique consists in measuring the conductivity of distilled water, in which lithium layers evaporated onto thin plates ("spectators") are dissolved. The spectators, which are made of the target material, are located on the target surface and are held in good thermal contact with it during evaporation. As is shown in what follows, the obvious advantage of this method consists in that the conductivity of the water solution produced thereby depends only on the mass of the dissolved lithium and is independent of

Table 1. Dependence of the conductivity of water (250 ml) on the volume of the primary lithium solution

Volume of the primary solution	Electric conductivity, μS/cm	Temperature of the solution, °C
1	320	19.5
2	643–646	19.5
3	942–946	19.5
4	1240–1247	19.4
5	1548–1558	19.4
10	3040	19.1

Table 2. Dependence of the water conductivity on the residence time of lithium in air prior to being dissolved

Sample no.	Exposure time in air	Water conductivity, μS/cm
1	5 min	2000
2	4 h	2000
3	28 h	2020
No sample	–	1

whether this lithium is pure or is a component of any compound (lithium hydride, nitride, or oxide). Measurements of the conductivity were performed using standard equipment—an Anion 410 ion meter–conductometer.

To calibrate the method, the following experiment was carried out. A portion of lithium shaped as a 4-mm-diameter 34.5-mm-long rod was squeezed out of the bellows in which lithium was stored. The rod was wrapped in aluminum foil of a known mass and then weighed. Since the lithium sample mass calculated from the known volume of the rod coincided with the result of weighing, one can assert that the major part of the lithium sample has not been transformed into lithium nitride or oxide in a short period of time when the lithium was exposed to air, which has also been indicated by the outward appearance of the rod. The whole of the lithium (0.234 g) was dissolved in 100 ml of water. A sample of this solution was taken, the volume was increased to 250 ml, and the electric conductivity was measured at a controlled temperature (Table 1). Results of these measurements showed that the conductivity of the solution was proportional to the dissolved lithium mass.

The effects that the other factors had on the conductivity of the solution were also studied. In particular, we investigated the dependence of the conductivity on the type of lithium compound and the possible contribution to the conductivity made by the indium–gallium paste, which was used to ensure reliable thermal contact between the spectator and the target and a small portion of which permanently remained on the spot at the rear side of the spectator.

An experiment was carried out to reveal the effect of a lithium compound on the conductivity of water in which it was dissolved. A rod was squeezed out of the bellows in which lithium was stored, and three identical samples were cut off by a special device. The first sample that retained metallic luster was immediately dissolved. The second sample was dissolved in 4 h. By that time, it has acquired black color, which may be explained by the appearance of lithium nitride on its surface. The third sample was immersed in water a day later; at that time, its color was white-gray, which might be associated with the substitution of nitrogen by oxygen and transformation of the whole of lithium or a larger portion of it into lithium oxide. The measured values of the conductivity of water with lithium dissolved in it (Table 2) show that the conductivity is inde-

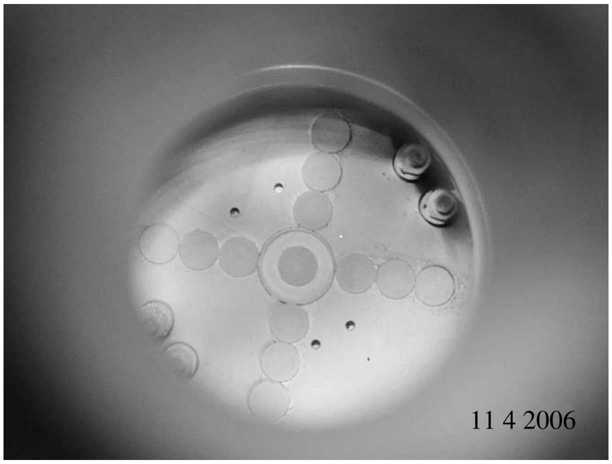

Fig. 1. Photograph of the target with spectators attached to its surface, which was taken through the window in the vacuum chamber immediately after the lithium evaporation.

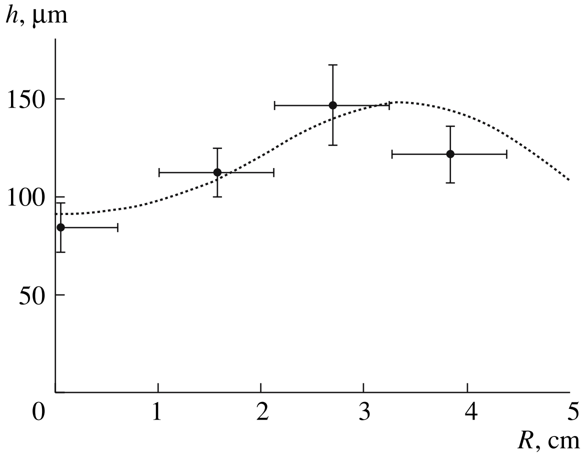

Fig. 2. Radial distribution of the lithium layer: the experimental results are shown with dots, and the theoretical values are presented with a dashed line.

pendent of the lithium sample composition, which renders this method undoubtedly attractive.

A very important advantage of this technique consists in the fact that the indium–gallium coating applied to the rear side of the spectator does not contribute to the conductivity and, therefore, does not affect the results of dissolved-lithium measurements. To verify this conclusion, 1 g of the indium–gallium paste was placed in 250 ml of water. The water conductivity remained virtually unaltered, though the introduced amount of paste was an order of magnitude greater than could be present on a spectator.

EXPERIMENTAL RESULTS

The lithium layer thickness was determined in the following way. Copper disk–spectators, each with a thickness of 0.5 mm and an area of 1 cm^2, were preliminarily fastened in two rows (crosswise) on the surface of a cooled target 10 cm in diameter. The indium–gallium paste ensured reliable thermal contact with the target. It should be noted that if a spectator is merely put on the target surface, a significantly thinner layer is evaporated onto it, which can be attributed to an increase in the spectator temperature. The container with lithium was placed in the evaporator on the valve plate, the system was pumped down to a pressure of 10^{-5} Torr, and the heater was turned on. The lithium evaporation time was 40 min. A photograph of the target with spectators attached to its surface is shown in Fig. 1. The photograph was taken at the end of the evaporation procedure. After the evaporation, the working volume was filled with air and the target with the evaporated lithium layer was removed. The disk–spectators were detached from the target and sunk into a vessel with distilled water. The amount of lithium deposited on each spectator was calculated from the conductivity of the solution formed in the vessel.

The measured distribution of the lithium layer thickness along the radius of the target is shown in Fig. 2. It is evident that the results of measurements are in good agreement with the simple calculation made under the assumption of collisionless spreading of lithium vapor from the circular orifice in the evaporator.

Table 3. Results of measurements of the lithium layer thickness using both a micrometer and the water conduction method

Spectator no.	Lithium layer thickness measured with a micrometer, μm			Lithium layer thickness determined by the water conductivity, μm
	spectator with lithium	spectator without lithium	lithium layer	
1	825	735	90	97.6
2	855	745	110	94.7
3	825	730	95	94.5

Direct measurements of the lithium layer thickness were also performed. The spectator thickness was measured using a micrometer before and after dissolving of the layer in water, and these readings were compared. The results of these measurements and the layer thicknesses determined from the water conductivity are presented in Table 3. The coincidence of these data indicates that a continuous lithium layer is formed during evaporation, and its density corresponds to the metal lithium density (0.54 g/cm^3). In paper [6], in which the influence of the residual gas on the lithium layer composition was investigated, it was ascertained that the evaporated layer consisted of pure lithium.

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