International Nuclear Target Development Society

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Editors Note

Dear Colleagues,

Thanks to the regular Newsletter contributors who continue to be highly productive and find time to write about it.

Thanks also to Satu Laakkonen who has compiled and distributed recent editions of the Newsletter (the real work).

Good holidays.

Chris Ingelbrecht
Editor
Calcium target preparation for the Tandem SNICS source

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NFI - Laborator Nazionali di Legnaro

Ca beams are of special interest for nuclear physics because of the exceptionally large neutron excess of $^{48}\text{Ca}$ and of the double closed shell structure of $^{40,48}\text{Ca}$ nuclei.

An effort was made to prepare metallic Calcium to be used in the XTU Tandem SNICS source. It was produced following the reduction-distillation method, developed by Kobish and Grisham\textsuperscript{11}, which allows to obtain metals with a purity up to 99.95%. This involves a high temperature both for CaO reduction, using Zr as reducing agent, and for the distillation of the produced metal.

The conversion of about 20 mg of Calcium in the appropriate form for a sputter cone, was made in three steps:

1) 100 mg of calcium carbonate (CaCO$_3$), corresponding to 40 mg of metallic Calcium, were converted into CaO with thermal treatment in an electric oven in open air at 1000 °C for two hours. As the result of this procedure, 56 mg of a very dry powdered calcium oxide was obtained through the process:

$$\text{CaCO}_3 \rightarrow 1000 °C \text{ in air} \rightarrow \text{CaO} + \text{CO}_2$$

2) In a second step, about twice stochiometric amount of high purity Zr metal was mixed to CaO. The mixture was pressed under vacuum with 1.5 Tons producing a 5 mm diameter pellet which was put in a tantalum tube crucible with the following characteristics: locked ends, 6.5 mm diameter, 0.2 mm wall thickness, 4.5 cm length with a 1 mm diameter hole drilled in the middle. At about 0.4 mm over the hole, a water cooled copper block was put with the aim of condensing Ca vapours. These are produced in the crucible at 1200 °C in a vacuum chamber with $p < 10^{-7}$ Torr, according to:

$$\text{CaO} + \text{Zr} \rightarrow 1200 °C \text{ at } 10^{-7} \text{ Torr} \rightarrow 2 \text{Ca} + \text{ZrO}_2$$

First vapours were used as a getter to capture oxygen and nitrogen gases contained in the crucible, then they condense closing the hole and bringing into contact the cold copper plate and the crucible and a metallic Ca stalactite grows inside. An optical pyrometer was used to read and control the crucible temperature. An amount of 23 mg of metallic Ca was obtained with an efficiency near 58%.

3) In the last step the stalactite was put in an electric furnace at 650 °C for two hours in a hydrogen flux with a pressure of 200 g/cm$^2$. The hydrogenated Ca was put in the source target and successfully used in the production of a Ca beam.

References
THE IUCF TARGET LAB TECHNICAL STATUS

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Being hosts of the 17th world Conference of the International Nuclear Target Development Society (INTDS), October 17-21, was the event for the target lab. The success of it must be credited to the support and work of many individuals from Indiana University, IUCF, and INTDS. The proceedings will be available as NIM-A362 in late summer of 1995.1

Along with the normal production of various targets and diagnostic foils for the accelerators, lab effort was applied to the development projects summarized below.

- Micro-ribbons and skimmer foils for CE-46: The methods to produce carbon micro-ribbons were extended to provide six targets with requested linear densities ranging from $1.8 \times 10^{14}$ to $3.0 \times 10^{16}$ atoms/cm. For direct comparison of the data-taking characteristics of carbon micro-ribbons vs. skimmer foils, open-edged carbon foils were produced with use of the substrate surface texturing techniques reported last year (and more fully in ref. 2). Also for this experiment, 25 mm open-edged skimmer foils of polystyrene (the beam monitor on all six ladders), Al, Sc, Ni, Zr, and Pb were provided in thicknesses between 200 and 500 $\mu$g/cm².

- For a neutron flux measurement experiment to be run at NIST, a $^6$Li$_{0.34}$Mg$_{0.66}$ alloy disk of 4 cm diameter and 2.8 mm thick was made. This project had a lead time of one year, so it was researched reasonably well and developed as the other target work allowed. The requirements addressed were the need to obtain at least 3.6 grams of uniformly mixed alloy with a $^6$Li content near (but not to exceed) 74 atomic %, a "very low" content of hydrogen, and a low oxygen content. These, along with the chemical activity and vapor pressure of Li and Mg at the formation temperature (±925 K), guided the development toward a reaction "bomb."

Trials conducted with stainless nut-and-ferrule constructions revealed they were unreliable for the number of temperature cycles and the temperatures necessary. Thus, a welded bomb design was used: a stainless steel tube with tightly fitting recessed end caps, pressed into place then sealed by TIG welding. A small stainless tube welded to/through one end allowed a protective blanket of argon to be removed and the interior to be pumped to high vacuum. After that, the small tube was flattened and sealed by spot-welding. The volume of the bomb was designed to allow for the calculated expansion of the 4.5 g charge of Li and Mg, and to provide 1.3 cm³ of headroom for mixing.

An old induction furnace (Lapel model T-1-B, 2.5 kW) was brought into service and modified to couple adequately with the bomb to reach a maximum temperature of 1100 K. This type of heating allowed the reactants to be brought quickly to the required temperature, and for thorough mixing to occur by shaking the bomb back-and-forth at elevated temperatures. It also allowed the process to occur easily in a surrounding atmosphere of deoxidized argon, in case a weld did not hold.

In the present form, the furnace should be quite useful in the target lab.

The welds held and the alloy stripped from the interior of the bomb initially had no visible signs of oxidation. Judged from its behavior during extensive rolling in argon, annealing in high vacuum, and trimming to a disk of 4 cm diameter, the alloy had no gross composition inhomogeneities.

- Follow-up work was done to prepare a contribution on the development of thin Teflon film walls for the polarized-gas target cell of CE-35. The technique of producing good foils was brought
to being a routine procedure. Employing a $^{228}$Th source, silicon detector, and the usual electronics, a setup was devised to check quickly the thickness of foils mounted on the gas-cell quadrants. For the three films measured in their middle areas and at near their ends, the areal weights were $450 \pm 20 \mu g/cm^2$.  

- Development of a prototypical carbon stripper foil for CIS was begun. One successfully mounted was a 7 mm-wide strip of 4.5 $\mu g/cm^2$, supported only at the ends. It spanned a 25-mm wide opening in a “C-shaped” frame. A satisfactory foil-strip stripper for CIS is thought currently to be only 0.5 $\mu g/cm^2$ thinner. Also, for in-beam tests, 4.5 $\mu g/cm^2$ carbon films were mounted successfully on IUCF’s normal open-edge stripper-foil frames (32 mm wide). The most-used stripper foils for the Cooler have been 6-8 $\mu g/cm^2$.

- For NIKHEF, a 6 $\mu g/cm^2$ carbon stripper foil, produced in the target lab, was mounted on NIKHEF’s standard stripper-foil frame. The opening in that frame is 31 mm by 52 mm. There is interest in these foils because they are a factor-of-five thinner than the foils used thus far on these rather-large frames. The mounted foil survived the journey to the Netherlands and more have been requested.

- For an experiment at TRIUMF, moderately rugged $^{13}$C pressed-powder targets of 100 mg/cm$^2$ by 1.27 cm by 4.5 cm were made. Enhanced target strength was necessary to enable the pellets to be mounted here at IUCF on TRIUMF’s oversize frame and to survive travel. This was provided by a cage made of thin Mylar sheet glued around the perimeter of each target. The Mylar was positioned edge-on to the beam direction and was not detected in the experiment.

- For a beam proposal submitted at Bates, Na target feasibility trials were done. A metallic Na disk of 7.3 cm diameter and 20-25 mg/cm$^2$ was required. By coating Na with high-grade mineral oil, rolling between Mylar sheets, and handling in argon, a very clean $22 \pm 1$ mg/cm$^2$ disk 9 cm in diameter was produced in a short time.

Producing Heat-Resistant CD₂ Targets of 150 mg/cm² in 3 Layers of Equal Thickness

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For a forthcoming (p, n) experiment at IUCF, commercially available CD₂ powder was sequentially hot pressed to 60 ± 5 mg/cm², rolled to 50 ± 1 mg/cm², and flashed with vacuum-condensed Au. Three pieces prepared in that way were cut in thirds, each 13 mm by 15 mm, to provide three targets of 150 mg/cm². The Au flashing (≈ 30 μg/cm², on both sides) and the stacking of the pieces were done to increase the ability of the targets to withstand days-long exposures to 100-125 nA beam currents of 135-200 MeV protons. The layers were mounted on a target ladder by trapping their corners between Al target frames, each with a 15.9 mm diameter center hole. This provided a spacing of ≈ 0.85 mm between the layers and a secure thermal contact to the frames and the target ladder.

The as-purchased starting material, commonly available CD₂ powder (98 % enriched deuterium), usually contains particles which vary in size from ≤ 10 μm to ≥ 3 mm. To promote better particle distribution in the pressing die, the disparity in sizes was reduced with the use of two single-edge razor blades. Each of these was hand-held with the cutting edge down, the plane of the blade at an angle of ≈ 15° to vertical, and with the lengths of the cutting edges in contact. The cutting action was delivered by repeatedly sliding the blades apart by pulling one’s right hand rightward and left hand leftward as the blades pressed into the powder on a cutting surface. Sheet glass (18 cm by 13 cm) was used as the cutting surface. Jumping particles were retained with aluminum foil extending under and beyond the edges of the glass. The cutting was continued until casual examination revealed that no particle was larger than ≈ 0.5 mm.

The pressing die used (WC, precision dimensions, mirror-finished pistons) produced pellets of 12.7 mm by 38.1 mm in area. It was loaded with ≈ 337 mg of the prepared powder by use of a razor blade in one hand and an anti-static pistol (ZeroStat®, England, USA distribution by Discwasher®). This was done with moderate care in about 3 minutes.

The first pressing was done with the loaded die at room temperature and using 13.8 kN/cm² applied smoothly in about 30 s, held for 5 minutes, and released in about 1 minute. Then the die was heated in an air oven to ≈ 395 K in about 1 h. At which temperature, 13.8 kN/cm² was again applied through the die pistons to the CD₂. The pressure was allowed to leak down as the die cooled to room temperature in about 2 h. The CD₂ separated easily from the die pistons and was a light green color.

A density of 1.05 g/cm³ was recorded for CD₂ sheet fused at 394-401 K. A piece hot-pressed at 382 K had a density of only 0.52 g/cm³, but after hot rolling this increased to 1.04 g/cm³. During pressing/fusing, 8-12 % of the CD₂ loaded into the die squeezed into the 12-μm-wide clearance space between the pistons and the die body. It was, of course, recoverable.

Hot rolling of the fused pieces was done to decrease thickness variations (up to 90 μm) recorded after pressing, and to obtain CD₂ target stock material larger than the die area. The rolling was done in a mirror-finished stainless steel pack after the pack was heated to 398 K in an air oven. To minimize the exposure time of the CD₂ to the elevated temperature, it was placed in the pack after the pack was removed from the oven. The rolling speed of a motor-driven rolling mill was set to about 6 mm/s, and the rolls initially were at room temperature. By reversing the direction of the motor shortly after the CD₂ sheet cleared the rolls, two roll passes (at the same roll setting) were possible before the pack needed reheating. Target stock of ≈ 63 mg/cm² was reduced to 50 ± 1 mg/cm² in 2-4 passes through the rolls.

If these targets perform especially well, or poorly, it will be reported in a later issue of the Newsletter.
Improved Electrodeposited Actinide Targets
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Electrodeposition has the significant advantage of very high yields (approaching 100 %) and is an attractive method when very limited quantities of starting material are available. Targets of $^{238}\text{Pu}$, $^{239}\text{Pu}$, $^{240}\text{Pu}$, $^{242}\text{Pu}$, $^{244}\text{Pu}$ and $^{234}\text{U}$ were requested and an improved electrodeposition method from an organic medium was developed to carry out this work. The main refinement was in the cell design (Fig. 1.), using a "knife-edge" compression of a PTFE ring to provide the liquid seal. A thin Al ring between the PTFE and the substrate was used to reduce the border effect that was otherwise noticed. The cell material was polyacetal (Delrin), replacing PVC, which is undesirable in the waste. A platinum grid anode rotating at 6 rpm was used. The deposition conditions for both U and Pu were:

Starting solutions: Pu or U 10 to 20 µg/µl in 0.75 M HNO$_3$
Electrolyte: 20 to 200 µl HNO$_3$ solution in 10 ml isopropyl alcohol
Current density: 0.5 to 2 mA/cm$^2$ at 100-300 V

Targets of up to 500 µg/cm$^2$ on metallic backings with good edge definition and colour uniformity were produced by this method and work is continuing to check homogeneity and to deposit onto plastic foil substrates.

![Diagram of electrodeposition cell](image-url)