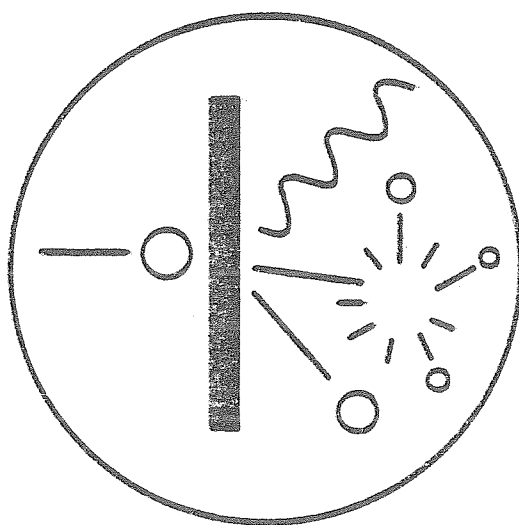


**INTERNATIONAL NUCLEAR  
TARGET DEVELOPMENT SOCIETY**

**NEWSLETTER**



**DECEMBER 1984**

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The INTDS assumes no responsibility for the statements and opinions advanced by the contributions.

E D I T O R I A L

Dear Colleagues,

First of all , I would like to express my appreciation to all the attendees of the 12th I N T D S Conference in Antwerpen.

The written papers submitted at the Conference were reviewed end December as planned, so that the proceedings appearing as a single publication of Nuclear Instruments and Methods can reach the Members as soon as possible (Mid 1985).

I especially thank the reviewers : H. Adair, H. Maier and J. Pauwels for their efforts.

I extend my best regards and wishes to you all and hope your Holiday Season was a happy one and that your New Year will be prosperous.

J. VAN AUDENHOVE

CLOSING REMARKS - TWELFTH INTDS CONFERENCE

H. L. Adair, Oak Ridge National Laboratory, Oak Ridge, Tennessee

I would like to take this opportunity to thank Jan Van Audenhove and his fine staff for hosting a superb International Conference. It has truly been an International Conference in which some 16 countries participated. Forty-five papers have been presented in eleven sessions which covered a wide range of topics including:

- (1) history and progress of target making,
- (2) isotopes,
- (3) target preparation techniques,
- (4) assaying techniques,
- (5) target properties,
- (6) neutron dosimetry, and
- (7) carbon foils.

Jan, again thanks to you and your fine staff for the many long hours required for preparing and for conducting the meeting. Thank you for taking care of all participants in a marvelous way.

Before closing, I would like to say a few words about our Society. As you know, we are a fairly young society being formed in a formal way in 1971. A lot of efforts directed at target making of course existed prior to our formation of INTDS but I know all target makers have benefited greatly from the formation of INTDS and from the cooperation that has and continues to exist among its members. I know I have received tremendous benefits. I'm reasonably sure the exchange of information at the conferences and workshops, as well as visits to other laboratories, have benefited each of us. Cooperative exchange programs between various target laboratories, many of

which are directly traceable to problems discussed at INTDS meetings or through associations established at INTDS meetings, have proven to be mutually beneficial and have provided the research community with samples they need in a more timely manner. Such exchange efforts should continue to be encouraged, and possibly the INTDS Board of Directors will have a little more to say on this subject at the next meeting.

As a member of INTDS, I would like, as I am sure you would, to increase the image of the Society. Spread the word about our Society and why we exist. Support our Society in every way you can by attending meetings, giving reports on your target making efforts at these meetings, publishing in recognized journals, and if possible, host meetings or assist in hosting meetings. Please provide input to the Board of Directors on how the Society can function better. For example, a few members have expressed the idea that one approach to increasing the image of the Society is to publish a book on target making. Of course, we realize that this would be a very difficult undertaking, but I know the expertise exists in the Society, and I believe it is something we could accomplish in approximately two years. At this point this is just an idea. If you have input concerning this idea or have other ideas, please don't hesitate to communicate them to the present committee responsible for coming up with ideas to increase the image of INTDS and the target maker. This committee is presently composed of Dan Riel, Hans Maeir, and myself.

At each INTDS meeting I seemed to get recharged with new ideas or new approaches to solving existing target-related problems. I also become a little saddened because it seems that at each meeting we learn of those who have retired or will be retiring from target making. These individuals are very hard to replace. As a Society, a Society which wants to grow in quality and number, we want to aggressively recruit new members. I think we have

much to offer. I also am sure there are many people who are not members who can offer a lot to this Society.

As target makers, we have made tremendous progress through the years, but fortunately, we haven't solved all the problems. Thus, we still have some challenges left. One challenge we have always had before us is that no matter what equipment we have (if we have every possible piece of target making equipment) we still can't prepare the target if we don't have the isotopic material. This has always been a problem and will probably continue to be a problem. However, for those of you who attended the ANL INTDS Workshop in 1983, we see that there is hope for the target maker with regard to the supply of isotopic material. That is, in some instances in the not to distant future, the supply of specific isotopic materials may rest with the individual target maker and not at ORNL or other places that supply enriched isotopic materials. By the use of the laser isotope separation technique, it will be possible to vaporize the desired normal material by conventional techniques with which we are all familiar and excite the specific isotope required with the laser. The specific isotope can then either be collected on the required substrate or collected for use in other processes. Hopefully, in many future INTDS meetings this will be a chief topic of discussion.

In closing I want to again thank Jan, his staff, and all participants for a splendid meeting.



PRESENTED TO

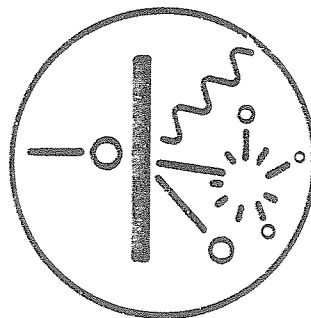
George Thomas

AT THE TWELFTH WORLD  
CONFERENCE OF INTDS  
SEPTEMBER 25-28, 1984

ANTWERP, BELGIUM

IN RECOGNITION OF HIS SCIENTIFIC  
ACHIEVEMENT AND MANY LOYAL  
YEARS OF SERVICE TO INTDS

*International Nuclear Target*



*Development Society*

# Lawrence Livermore National Laboratory

## MECHANICAL ENGINEERING DEPARTMENT

E. Darrell Erikson  
Materials Fabrication Division

### VACUUM COATINGS

#### Polypropylene Substrates.

For the past several years, thin films of unsupported Parylene have been the principal backing material for metallic coatings used as detector targets. Recently, requesters were selected polypropylene as a substitute for Parylene. The requester provided the polypropylene already mounted on target foil support frames. The frames were of two different configurations. In one configuration the 500 nm polypropylene was stretched across a 76.2 mm ID opening - in the other it was stretched across a 38.1 X 63.5 mm rectangular opening. The switch to polypropylene resulted in two significant advantages - we were dependably provided substrates to coat, and the bare polypropylene was relatively easy to handle because it is mechanically stronger than Parylene. However, polypropylene is much more subject to thermal degradation than previously used substrate materials. The polypropylene had to be limited to a temperature of 66°C during any part of the coating process. This limitation, coupled with very short lead time requests, required that extensive process development be conducted on a very short turnaround basis.

#### Manganese on Polypropylene.

During our initial attempts to deposit manganese on polypropylene by magnetron sputtering, the brittle manganese sputter targets broke at minimum power levels. Reverting back to resistance heated evaporation, we were able to successfully coat the polypropylene foils with the requested 500 nm of manganese. The deposition was accomplished at a rate of 0.05 nm/sec. To achieve coating uniformity the polypropylene substrates were mounted on a rotating fixture, with a source to substrate distance of about 30.48 cm.

#### Magnesium and Zinc on Polypropylene.

Preliminary test runs were done using magnetron sputtering sources to determine the feasibility of depositing either magnesium or zinc on polypropylene foils by magnetron sputtering. The initial tests showed that either material could be made to nucleate and deposit on the polypropylene foils. Unsuccessful, parallel attempts were made to produce zinc (at LLNL) and magnesium (at LBL) coatings on polypropylene foils by evaporation. The evaporated zinc would not nucleate and form a coherent coating on the polypropylene. Magnesium coated foils were produced (at LBL) by evaporation, but they rapidly decomposed after a few days in storage. Magnesium and zinc targets for magnetron sputter guns have been obtained, and magnesium coating runs have been made to test and calibrate the process. The initial magnesium coated foils produced had flaws which we feel are correctable. The flaws consisted of small uncoated areas ("windows") and color striations on the deposit. It appears that the "window" problem can be overcome by adjusting the sputtering gas pressure parameters. The system was cleaned and baked in an attempt to reduce the possibility of unknown contaminants causing the color striations - though it is possible that pre-existing contamination of the polypropylene substrates may have caused this problem. Some fine tuning of the process is still in order, but the magnesium deposits made so far look very good and are of acceptable quality.



### Iron on Polypropylene.

Coatings of iron were requested on polypropylene foils. Initial attempts were made to magnetron sputter the iron by changing the sputter target geometry to compensate for the magnetic nature of the iron. Though the results of these attempts seemed promising, no usable foils were being produced. We decided also to attempt to produce the coatings by electron beam evaporation while in parallel continuing to develop a capability to magnetron sputter coat magnetic materials. The iron coatings were successfully accomplished by implementing two techniques. A wire mesh was placed between the source and the rotating substrate holder. The mesh served to reduce the heating of the substrates by radiant energy from the molten iron in the electron beam gun hearth and it also served to protect the substrates from the heating effect of bombardment by stray electrons.

### Copper on Polypropylene.

Copper for use as a detector material was vapor deposited on approximately 150 polypropylene foils. Two vacuum process systems, VDS-106 and M-3, were employed. In both systems, "Torus-10" magnetron sputtering sources were used and the processing pressure was maintained at 18 millitorr of argon. The initial testing of the concept and the establishing of coating parameters to accommodate low substrate temperature requirements were accomplished in VDS-106, because it was already setup to do magnetron sputtering (for a different project). For schedule considerations and to improve the coating uniformity the source to substrate distance was doubled in the M-3 system. The power level was raised from 300 watts in VDS-106 to 1600 watts in M-3. X-ray calibration data showed us to be within 10% of the requested thicknesses. All of the requested copper coated foils were delivered by mid September.

### Germanium on Beryllium.

Germanium coated beryllium foils were required for use as detectors. In the time allotted we could not solve curling and cracking problems associated with intrinsic stresses encountered at the original requested coating thickness. We therefore negotiated a reduction in coating thickness to 1000 nm. The coatings were then successfully accomplished by: evaporation from a 50.8 mm hearth pocket electron beam gun source, a source to substrate distance of approximately 35.56 cm (in the VDS-103 system), and substrate back-heating to a temperature of 250°C.

### Cesium Iodide Coatings.

In our last progress report we described having assembled a work station to permit transferring, measuring, and packaging cesium iodide coatings without exposure to ambient atmosphere. In this report period the new work station, which consists predominantly of an inert atmosphere glove box that encloses the tops of two vacuum deposition systems, was put to good use. Cesium iodide coatings of higher quality than previously possible were accomplished with

much less effort. The very hygroscopic coatings of cesium iodide were applied to substrates of three different types on X-ray detectors: One application involved the coating of aluminum shapes; a second includes the over-coating of gold coatings on micro-channel plates; a third application included the coating on Formvar Pellicles for a few special experiments. In all cases the cesium iodide salt was evaporated by use of a resistance heated, cylindrically shaped source, and deposited in the range of 100 nm to 300 nm.

#### Copper and Aluminum on Tantalum.

The coating of a type of detector called a "race track" was requested. The substrate consisted of a tantalum disk, 0.051 mm thick X 76.2 mm diameter, having a 6.35 mm X 31.75 mm slot in its center. The coating requirements were for a 0.051 mm thick base layer of copper, over-coated by 0.051 mm of aluminum, deposited over one entire side and all surfaces of the slot. This was accomplished by holding the substrate in a specially fabricated stainless steel fixture, sputter etching the substrate, ion-plating the initial layers of copper, and then sputter depositing the copper and finally the aluminum. The coating parameters were: 18 millitorr argon pressure (for both materials), 9 kW of power for copper and 6 kW for aluminum, 410 nm/min. for copper and 225 nm/min. for aluminum.

#### Chrome on Beryllium.

Chrome coatings, 1000 nm thick, were requested on beryllium detector substrates for use as detectors. The coatings were produced using a Torus-10 magnetron sputtering source. The processing parameters were: 3 kW gun power, 10 millitorr partial pressure of argon, ambient substrate temperature, and a deposition rate of 45 nm/min.

#### Aluminum on Parylene.

Aluminum coatings 100 nm thick were deposited on 500 nm thick Parylene and subsequently mounted on aluminum frames. Two different techniques were tried. First, the Parylene was mounted on rings and then coated. It proved difficult to try to get the Parylene taut enough but not so taut as to break during coating. The preferred technique proved to be coating the as received Parylene pellicle first, and then floating it on water to effect the transfer onto the rings. Subsequent coatings of aluminum were then transferred to LBL to accommodate programmatic schedule requirements.

#### Titanium on Aluminum.

Titanium at a specified areal density of 50 mg/cm<sup>2</sup> was deposited onto 25.4 mm aluminum substrates for nuclear chemistry accelerator experiments. This was accomplished by vacuum evaporation from a 50.8 mm "Super-Source" electron beam gun.

## VACUUM ENGINEERING AND MATERIALS TESTING

### X-Ray Calibration and Standards Facility.

Work has been completed on the preparation of procedures for cleaning of ultra-high vacuum components for the XCSF Project. SLAC/SSRL personnel have given approval of our procedures for the cleaning of aluminum, copper, and stainless steel (ENN84-12, -13, and -14, respectively). By pursuing the approval of LLNL cleaning methods, we've avoided the need for a dedicated facility; the cost of such a facility was estimated to be greater than \$500 K.

Work continues on the preparation of similar engineering notes which will outline procedures for welding, handling, machining, assembling, and testing UHV components as well as the establishing of a quality assurance program to track and document the processing of hardware needed by the project.

At the present time, conceptual design reviews are being presented to SLAC/SSRL for those components which are scheduled for installation during the three month shut-down of the Synchrotron in Summer of '85. Upon approval of these designs, detailing and fabrication will proceed through Spring of '85. During the period of fabrication, the impact upon MFD facilities is estimated as about 500 man-hours of cleaning and about 800 man-hours of welding.

### POCO Graphite Outgassing Test.

We were asked to identify the gas constituents evolved from "Poco" graphite in vacuum at elevated temperatures - to the upper limit of our existing equipment capability. To accommodate this request we attached our portable residual gas analyzer/data acquisition system, MS-2, to our radiant heating vacuum furnace, VF-100, which in its present form is limited to 1100°C. To establish the system background, outgassing tests were done of the empty system both before and after the graphite was tested. The tests clearly demonstrated that the graphite samples from two different vendors were heavily contaminated with what appeared to be a hydrocarbon oil. At temperatures of about 500-750°C the gas load would start to decrease significantly. Above 750°C the gas load would decrease and the residual gas spectrum would start to resemble that of the empty system. We recommended to the requester that, all newly received graphite be baked at about 1000°C prior to being placed in their vacuum system.

### Mylar Properties Test.

Data for the permeation rate of hydrogen through Mylar barriers was requested. A 1.5 micron thick Mylar barrier was installed on a vacuum system and both sides of the Mylar were pumped down to less than 30 millitorr. One side of the Mylar was then subjected to hydrogen at various pressures and the low pressure side of the Mylar was continually pumped through a 0.457 mm orifice by a turbo pump. Measuring the pressures drop across the orifice gave the following permeation data: with 20 Torr of hydrogen the permeation rate was  $2.2 \times 10^{-4}$  Torr-liters/sec-cm<sup>2</sup>, with 40 Torr of hydrogen the permeation rate was  $6.6 \times 10^{-5}$  Torr-liters/sec-cm<sup>2</sup>, and with 60 to 100 Torr of hydrogen the permeation rate was  $9.5 \times 10^{-3}$  Torr-liters/sec-cm<sup>2</sup>.

The burst strength of the Mylar barriers was also requested. For safety, nitrogen was used in place of hydrogen. Data indicate the burst pressure to be on the order of 135 Torr for the samples.

### Presentations

Integrated Contractor's Meeting on Coatings, Sandia, Albq., June 26 & 27.

J. W. Weed, J. G. Romo, and G. E. Griggs, "Nuclear Target Foil Fabrication for the Romano Event."

W. K. Kelley, "Electropolishing Very Large Spatial Filter Tubes."

M. A. Benaffl, T. G. Beat and R. H. Reiss, "Ion Plating Copper on Molybdenum."

W. K. Kelley, Alpha 1 Cladding Status Review, September 12, 1984, Redondo Beach, California.

J. W. Dini, W. C. Cowden, and W. K. Kelley, "Engineering Properties of Some Coatings," American Electroplaters' Society Sur/Fin 84, July 16, New York.

E. D. Erikson, P. L. Tassano, R. H. Reiss and G. E. Griggs, "The Preparation of Selenium Coatings and Beryllium Foils," 12th International Conference of the International Nuclear Target Development Society, Antwerp, Belgium, September 25-28, 1984.

### Publications

J. W. Weed, J. G. Romo and G. E. Griggs, "Nuclear Target Foil Fabrication for the Romano Event," UCRL-90552, June 19, 1984.

W. K. Kelley, H. G. Patton, D. H. Fitch, B. Freitas, D. E. Hoffman and E. M. Lopez, "Electropolishing Large Spatial Filter Tubes for the NOVETTE and NOVA High-Energy Laser System," Plating and Surface Finishing 58, 61 (September, 1984).

D. D. Berger and E. D. Erikson, "A System and Procedure for Outgassing Tests," LLNL Engineering Note ENF84-04.

J. Gallagher, W. K. Kelley, and E. D. Erikson, "X-ray Calibration and Standards Facility; Cleaning Specifications for Aluminum, Copper, and 300 Series Stainless Steel Alloys," LLNL Engineering Note. ENN84-12, -13, and -14, respectively.

J. W. Dini, "Fundamentals of Chemical Milling," American Machinist, Special Report 768, July 1984.

J. W. Dini and H. R. Johnson, "Plating on Some Difficult to Plate Metals and Alloys," Fortschritte Bei Der Galvanisierung Problematischer Grundwerkstoffe, May 1984.

E. D. Erikson, D. D. Berger, and B. A. Frazier, "A Comparison of the Outgassing Characteristics of Several Solar Absorbing Coatings," UCRL-90097.

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L8S 4K1  
Canada

### 1. Self-supporting Tantalum Targets

At our target lab, self-supporting tantalum targets of thicknesses of 100 to 300  $\mu\text{g}/\text{cm}^2$  are being produced.

The procedure involves the electron beam evaporation of tantalum with a 2 kw Varian electron gun. The tantalum is evaporated onto 0.001" thick copper foil which has been previously cleaned at 500<sup>0</sup> C by resistive heating in a high vacuum. The copper substrates are heated to 350<sup>0</sup> C during the deposition to reduce any stress in the deposited tantalum foil. The deposition rate of the tantalum onto the copper substrate is ~ 100 Å/min.

When the substrate has cooled to room temperature, it is removed from the vacuum system. The target and substrate is then placed onto the surface of a solution of trichloroacetic acid, ammonium hydroxide and water which will slowly dissolve the copper substrate. When all of the copper has been removed, the tantalum target is cleaned by floating it on distilled water. It can then be mounted on a suitable target frame. For some of our experiments, the target material is implanted with <sup>20</sup>Ne prior to removing the copper substrate.

Investigation into the minimum or maximum target thickness by this method has not been carried out.

The solution for removing the copper substrate consists of:

50 g	trichloroacetic acid
250 ml	ammonium hydroxide
250 ml	distilled water.

## 2. Low Fluorine Background Target Substrates and Chambers

It has been observed that commercially available tungsten used as target substrates contains residual fluorine which causes undesirable background in (p, $\gamma$ ) experiments.

In an effort to remove the fluorine, a small electron gun has been designed and fabricated to uniformly heat the 5.5 cm<sup>2</sup> tungsten substrates to temperatures of 1600 to 2000<sup>o</sup> C. Uniform heating is required to maintain a flat substrate which is needed to ensure efficient contact cooling under beam conditions. The tungsten substrate is slowly raised in temperature to ~ 1800<sup>o</sup> C in high vacuum and maintained at this temperature for 30 minutes. When cooled, the target material may be evaporated onto the substrate.

To further reduce the effects of a fluorine background, a small vacuum furnace was fabricated to form tungsten sheet into cylindrical shaped sections and to out-gas the fluorine in one step. The furnace allows out-gassing in high vacuum at temperatures of 600<sup>o</sup> C for periods of 4 to 6 hours. When cooled to room temperature, the tungsten sheets retain their cylindrical form and are inserted into the 2 inch stainless steel beam line and target chamber to form a low fluorine tungsten lined target chamber.

In addition, beam line apertures are also fabricated from tungsten sheet and out-gassed in a similar manner.

Figure 1 shows a spectrum of the reaction  $^{50}\text{Cr}(p,\gamma)^{51}\text{Mn}$  with a tungsten substrate which was not out-gassed. The reactions from fluorine obscure the peaks which are of interest.

Figure 2 shows the spectrum of the same reaction with a tungsten substrate which has been out-gassed to lower the fluorine. It will be noticed that the peaks caused by fluorine have been reduced by a significant amount and no longer obscure the peaks of interest.

### 3. Isotopic Rare Earth Targets

The target lab is now fabricating thin isotopic rare earth targets from the oxides of 50 to 100  $\mu\text{g}/\text{cm}^2$  which are supported by a Collodion film of  $\sim 7$   $\mu\text{g}/\text{cm}^2$ . The targets are prepared using the procedure to produce 100 to 300  $\mu\text{g}/\text{cm}^2$  self-supporting targets described at the 1983 workshop held at ANL.

The difference in the procedure is to remove the  $\text{H}_2\text{O}$  with methanol as described, but the collodion film is not removed from the rare earth film. Under beam conditions, the collodion is removed by beam heating only over the beam spot area, leaving the remaining collodion to support the target material.

- 1) D.J. Yaraskavitch and Y.K. Peng, ANL/PHX-84-2, 231-234.

DATA AREA : 2.

RUN NUMBER: 3.

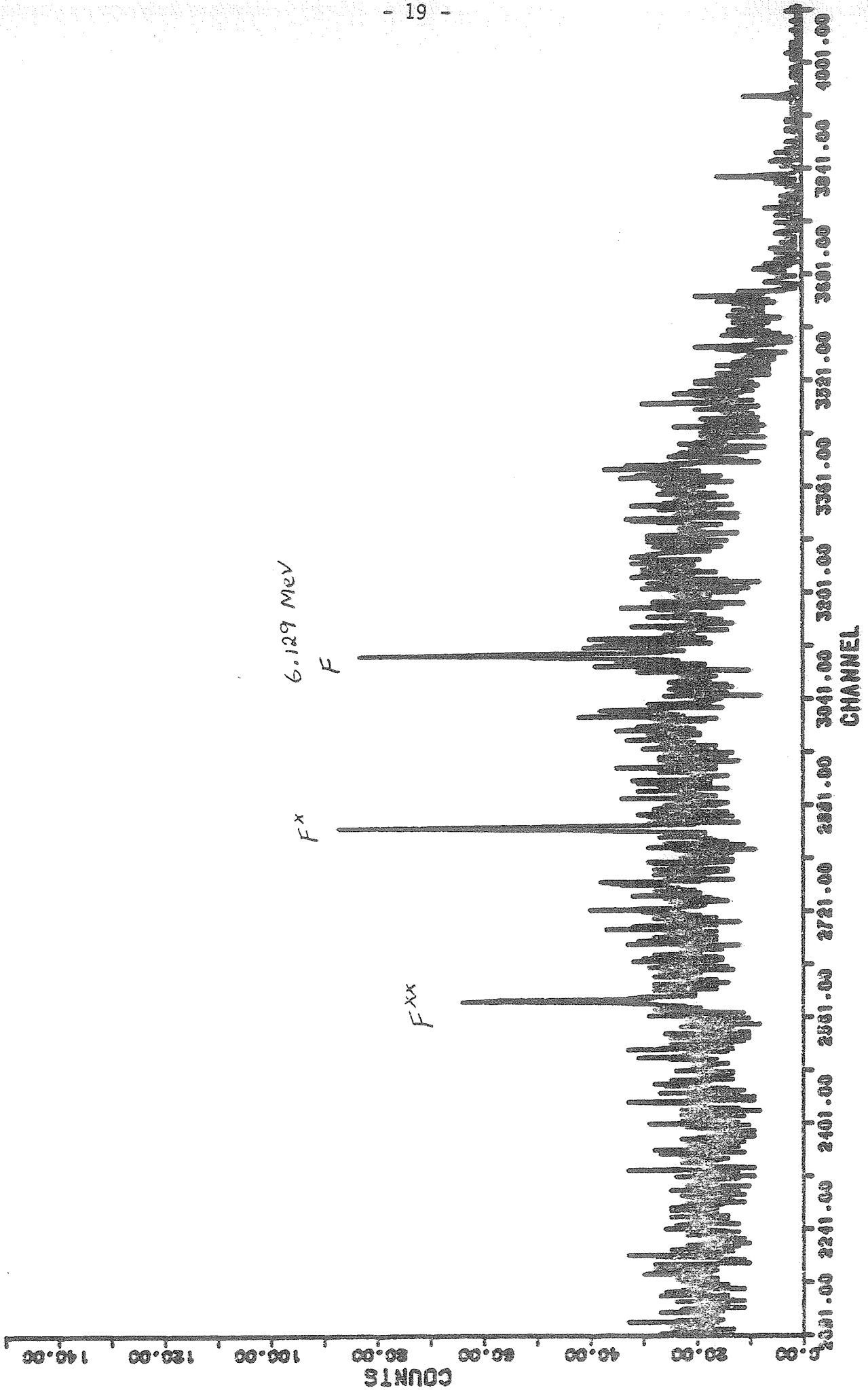


Figure 1. Spectrum of the reaction  $^{50}\text{Cr}(p,\gamma)^{51}\text{Mn}$  with the target material on a tungsten substrate which has not been outgassed.



DATA AREA : 1.

RUN NUMBER: 3.

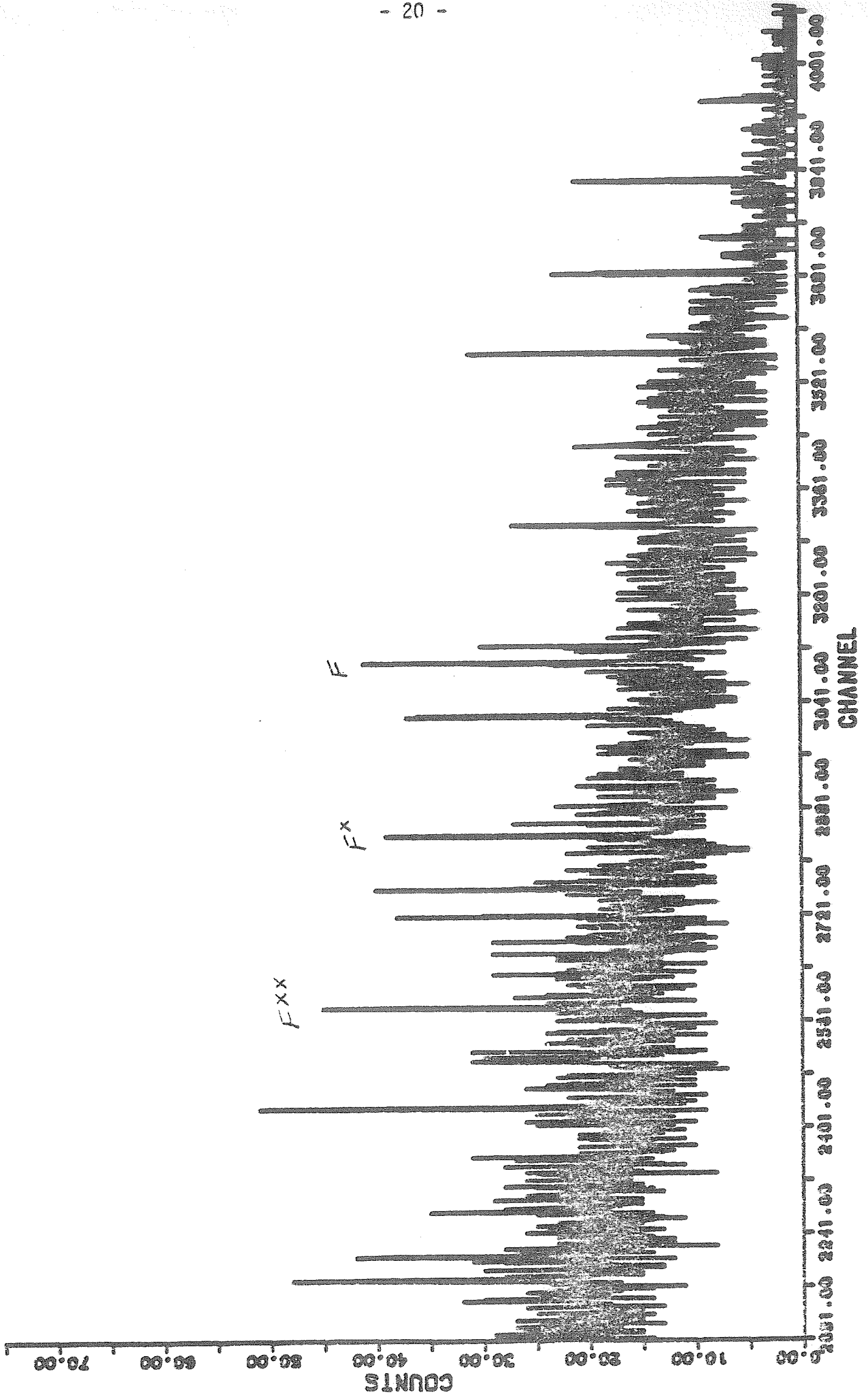


Figure 2. Spectrum of the reaction  $^{50}\text{Cr}(p,\gamma)^{51}\text{Mn}$  with the target material on a tungsten substrate which has been outgassed to lower the residual fluorine.

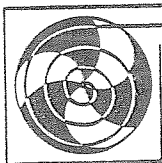
K.O. Zell

Institut für Kernphysik der Universität Köln

Zülpicher Straße 77, D-5000 Köln 41, West Germany

- Cu CuO has been reduced to Cu at 300 °C in a H<sub>2</sub> stream in 2 h and molten afterwards by an electron gun. It has been rolled down to 4 mg/cm<sup>2</sup> and then rolled together with bismuth to 1 mg/cm<sup>2</sup> Cu on 4 mg/cm<sup>2</sup> Bi. 0.3 mg/cm<sup>2</sup> Cu targets were prepared by evaporation on betain.
- Os 50 µg/cm<sup>2</sup> Os targets on 1 mg/cm<sup>2</sup> Al backing have been prepared by evaporation with an elektron gun starting with a 50 mg pill. The distance to the backing was 10 cm. The targets were coated with a 10 µg/cm<sup>2</sup> Al layer to prevent oxidation.
- Li 0.3 mg/cm<sup>2</sup> Li targets on 10 µg/cm<sup>2</sup> C were prepared by evaporating the Li on to the backings. The C foils did not break only if they were rotated in such a way that the Li went to both sides of them.
- Sn Several 0.5 mg/cm<sup>2</sup> Au plunger targets were made by evaporating the Sn from a W tube shielded with a diaphragm in 2 cm distance on the rolled Au foil.
- It has been tried to roll 8mg/cm<sup>2</sup> Sn on 12 mg/cm<sup>2</sup> Ta but at this thickness there was no sticking.
- Ag 30 µg/cm<sup>2</sup> Ag targets were made by evaporation on betain. This was the thinnest layer from which selfsupporting targets could be made.

K.V.I.



KERNFYSISCH VERSNELLER INSTITUUT DER RIJKSUNIVERSITEIT  
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T.W. TUINTJER

#### Improved efficiency in the production of calcium targets

The method of making Ca targets<sup>1)</sup> on the high-gloss chromium coated metal plate (Kindermann plate) yields only 10-20%. A reasonable amount, however, can be regained as oxide/carbonate from the used glass shield. In view of the high cost of  $^{48}\text{CaCO}_3$  and the demand for thicker targets there was a need for improving this direct yield. An effective output of over 60% was obtained by taking four measures:

- i) Using a 2 mm instead of a 2.5 mm aperture on top of the crucible.
- ii) Reducing the distance between substrate and crucible from 6-8 cm to 1 cm.
- iii) Doubling the quantity of Zr powder (reducing agent) to 2.4 times the stoichiometric amount.
- iv) Slowly raising the source temperature from 1300° C till 1850° C during evaporation.

With 10 mg CaO + 20 mg Zr in the crucible the evaporation took about 15 minutes. This resulted in a piece of Ca on the substrate which had an average thickness of 29  $\mu\text{m}$  and a weight of 4.5 mg, i.e. an efficiency of 63%. Of course it is possible to use a glass shield for catching the rest of the material.

1. KVI Annual Report 1980, p.154.

#### The preparation of metallic mercury targets

A new and simple method for the production of carbon backed Hg targets was developed. The procedure consists of three steps:

- i) Preparation of mercury oxalate: The isotopic material, usually HgO, is solved in diluted  $\text{HNO}_3$  (3.5 mg HgO in 100 mg  $\text{HNO}_3$  (conc) and 2 g  $\text{H}_2\text{O}$ ). Fine crystalline  $\text{HgC}_2\text{O}_4$  precipitates after the addition of 100 mg oxalic acid and 2 g  $\text{H}_2\text{O}$ . The liquid phase is pipetted off, the precipitate is washed three times with ca. 5 ml demineralized water and three times with ca. 5 ml ethanol. Each time the liquid is removed with a Pasteur capillary pipette. The oxalate is dried in a furnace at 60° C. Yields are in the range of 95-97%.

- ii) Decomposition of the oxalate and evaporation of Hg: These processes were simultaneously carried out at atmospheric pressure in the fume-cupboard. About 2 mg Hg oxalate is divided equally over the bottom of a pyrex crucible which is placed on a gauze with asbestos centre. A glass-slide with a  $30 \mu\text{g}/\text{cm}^2$  C-coating is put on the crucible. The crucible is heated for exactly 2 min. by a Bunsen burner established for maximum-heating. Then the glass-slide is immediately removed for cooling. With the microscope it was observed that the Hg-vapour is condensed into very fine drops. No large differences in the distribution pattern are observed when the slide is placed in vertical position, even not after a week. The average thickness was  $680 \mu\text{g}/\text{cm}^2$  which means a 55% efficiency.
- iii) Protection: To prevent the re-evaporation of Hg, especially in vacuum, the C-Hg target is put in a 2% solution of Styron-666 (DOW) in chloroform which results in a protecting coating of  $12\text{-}25 \mu\text{g}/\text{cm}^2$  polystyrene. Finally, the sandwich target is floated off on water, picked up on a frame and mounted.

A simple high-yield method for evaporation of Zn, In, Mg, S and Tl

A slight modification of our method for evaporation of Cd, Se and Te with high efficiency<sup>1)</sup> turned out to be suitable for Zn, In, Mg, S and Tl. This modification implies a more indirect heating of the crucible which enables a better control of evaporation rates.

A tungsten spiral (w-Kegelwendel 490105, Balzers) is clamped between the electrodes of the resistance-heater of a vacuum installation. A glazed porcelain 5 ml crucible (Rosenthal 101/20) with 5-50 mg of the desired element is placed in the spiral. The elements were prepared in various ways with yields in the order of 70-100%:

- i) Zinc: crystalline powder prepared by electroplating according to ref.<sup>2)</sup>.
- ii) Indium: "dendritic" crystals and powder prepared by electroplating, see section e.
- iii) Magnesium: pieces of foil obtained by reduction and evaporation according to ref.<sup>3)</sup>.
- iv) Sulphur and Thallium: as powder in stock.

In the case of Mg, S and Tl the material is covered by one or two Ta or W gauzes. A microscope slide is placed on top of the crucible. This slide is cleaned (Zn), very thinly greased (Mg) or provided with a suitable backing (In, S and Tl). For cooling purposes the slide may be covered with one or two extra slides. After evacuating at appr.  $10^{-6}$  Torr the heating is slowly raised until the evaporation starts. Then the heating is continued during 5-50 minutes, depending on the element and the amounts of material. The result is a nice circular coating of uniform thickness. In the case of Zn and Mg the metal foil is lifted from the slide with help of a surgery knife and can be easily rolled to the thickness desired. In the case of In polystyrene (Styron-666, DOW) is used as backing. The combination is floated on water. Afterwards the polystyrene is solved in chloroform. Tl must be handled under vacuum or argon atmosphere to prevent oxidation.

1. A.H. Bennink et al., INTDS Newsletter, July 1979.  
2. J.M. Heagney, J.S. Heagney, Proc. INTDS Conf., Berkeley, USA, 1977, p.35-36.  
3. KVI Annual Report 1981, p.99.

Electrolytical reduction of  $^{115}\text{In}$  oxide

Corresponding to the formula:  $\text{In}_2\text{O}_3 + 3\text{H}_2\text{SO}_4 \rightarrow \text{In}_2(\text{SO}_4)_3 + 3\text{H}_2\text{O}$  we prepared an electroplating solution of 120 mg  $^{115}\text{In}_2\text{O}_3$  in 1200 mg diluted  $\text{H}_2\text{SO}_4$  (conc. 9.8%), completed with 5.7 g. water. This solution with  $\text{pH} \approx 2.5$  was transferred into a standard plating cell<sup>1)</sup>. The cathode was a strip carbon filament of  $25 \times 5 \text{ mm}^2$ , cleaned with alcohol. The immersed part had an effective area of  $(2 \times) 15 \times 5 \text{ mm}^2$ .

The process started at room temperature with a current density of  $40 \text{ mA/cm}^2$ . During the plating this density increases to about  $200 \text{ mA/cm}^2$  and the temperature rises to  $60-70^\circ \text{C}$ . The process can be interrupted for weight-control. After  $2\frac{1}{2}$  hours about 75% of the indium was plated on the electrode. The reduced material together with the carbon-cathode is put in a crucible and evaporated using resistance-heating.

1. KVI Annual Report 1978, p.146.

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1.) Preparation of Selfsupporting Zr-Targets

During the 12<sup>th</sup> World Conference of INDTS in Antwerp some doubts remained about making selfsupporting Zr-targets, because I couldn't remember exactly the conditions or preparation of these targets, I had made 3 years ago.

Therefore I consulted my former notes and made some repetitions with reproducible results:

Zr-powder was pressed to a pill and molten to a ball by an electronic gun (Leybold-Heraeus ESV 6). The evaporation was also done with the electronic beam of this gun on to a pane of Tempax glass of 2mm thickness, coated with a Betainmonohydrate layer and  $100 \mu\text{g}\cdot\text{cm}^{-2}$  NaCl or with a layer of  $100 \mu\text{g}\cdot\text{cm}^{-2}$  KJ.

The distance from the crucible of the electronic gun to the substrate was varied between 6 and 12 cm, the evaporation rate was in the range of  $10 \mu\text{g}\cdot\text{cm}^{-2}\cdot\text{min}^{-1}$ , determined by Veeco QM-301 with FTT-4.

After floating off in warm distilled water (about 50°C) the foils were picked up with frames.

The thickness of the several charges was controlled by measuring the energy loss of  $\alpha$ -particles and found to be

140 to 270  $\mu\text{g}\cdot\text{cm}^{-2}$ , which was in good conformity with the values of the quartz crystal thickness monitor.

Some of the targets made in 1981 are still intact.

2.) Recent activities are:

evaporation of Au and various organic chemicals with low melting-points on to thin formvar foils  
selfsupporting Be-targets, 15 and 30  $\mu\text{g}\cdot\text{cm}^{-2}$   
selfsupporting  $\text{SiO}_2$ -targets, 80  $\mu\text{g}\cdot\text{cm}^{-2}$   
C-stripperfoils by glow-discharge technic  
C-backings of various thicknesses by electron beam evaporation  
preparation of reflecting surfaces by means of evaporation of Al and Ag.