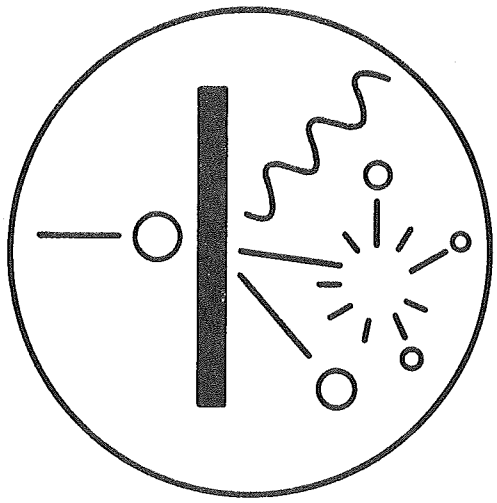
INTERNATIONAL NUCLEAR

TARGET DEVELOPMENT SOCIETY

NEWSLETTER



AUGUST 1980

INTDS Newsletter

August 1980

We are putting out the second issue of the INTDS Newsletter this year. The response for contributions has been much appreciated.
We would further encourage all members to make contributions. The following items could be of interest :

1. Description of recent work.
2. Information of any newly developed technique or progress made.
3. Request for advice on, for example : - making specific targets
   - obtaining specific material (e.a: enriched isotopes)

Our thanks are due to the Direction of the Central Bureau for Nuclear Measurement for their support in producing the Newsletter.

Editor :

Jan Van Audenhove
Central Bureau for Nuclear Measurements
B - 2440 GEEL (Belgium)

Some Papers of interest

- A.H.F. Muggleton
  Preparation of thin nuclear targets
  Journal of Scientific Instruments (September ,1979)

- M. Pickering, N.R.S. Tait and D.W.L. Tolfree
  Electron diffraction studies of carbon stripper foils (DL/NUC/P 105A)
  Submitted to Phil. Mag.

- N.R.S. Tait and D.W.L. Tolfree
  Daresbury Laboratory
  P. John, I.M. Odeh, M.J.K. Thomas, M.J. Tricker and J.I.B. Wilson
  Departments of Chemistry & Physics, Heriot-Watt University
  and
  J.B.A. England and D. Newton, Department of Physics, University of Birmingham
  The hydrogen and oxygen content of self-supporting carbon foils prepared by D.C. Glow discharge in ethylene. (DL/NUC/P 106A)
  Submitted to Nucl. Inst. & Meth.

Request for advice

LARS EINARSSON, Tandem Accelerator Laboratory, Box 533-S/75121 UPPSALA, Sweden has to prepare a metallic ^{236}U target with a diameter of at least 6 mm and a thickness of 10 mg/cm^2. The uranium base material is now as an oxide.

RESEARCH SAMPLES OF THE TITANIUM ISOTOPES*

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Several members of the INTDS have indicated that it would be highly beneficial for their research if they could obtain a small quantity of ^{50}Ti with an isotopic purity greater than 95 %. We have recently completed a research project directed toward understanding the causes which limited the titanium isotopic assay in previous separations. The experimental results indicated that the chemical compound used as feed material in the ion source was the major problem. The previous feedstock was giving a high neutral vapor background pressure in the calutron vacuum system which, in turn, contributed to the reduction in isotopic purity in the collectors. Following the R&D effort, a small amount of ^{50}Ti was collected for those who had previously requested the material. As part of the same separation, we have collected research quantities of the other isotopes of titanium which will be available on a limited basis. Their estimated isotopic assay is as follows :

Titanium Isotope	Assay (at. %)
46	~ 99
47	~ 99
48	> 99.4
49	~ 98.5

* Research sponsored by the Office of Basic Energy Sciences, U.S. Department of Energy under contract W-7405-eng-26 with the Union Carbide Corporation.

Bill Lozowksi and Tina Rife
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We have recently made fused, homogeneous Melamine targets up to 13 mg/cm^2 by downward vacuum evaporation onto 100 μg/cm^2 carbon foils, Our previous attempts at upward evaporation of dried and pressed Melamine powder pellets resulted in stress cracked films with poor adhesion to the carbon foils.

The additional problem of Melamine decrepitation (which causes gross non-uniformities in the deposit) was solved by placing a 3/16" dia. pellet into a "sock" of 325 mesh stainless steel wire cloth. This "sock" was tied with resistance wire and placed inside the evaporation source. The source, an R.D. Mathis no. S31-.010 Ta, consists of two 1/8" deep dimpled strips of tantalum with a 3/16" hole for evaporation. Two separate loops of 0.020" dia. resistance wire were wrapped tightly around the source to close the horizontal gap between the strips. A glass slide was used to determine when evaporation began and the source temperature was not increased beyond this point. The distance from source to substrate was 2.6 cm. Prior to the evaporation, the carbon foil substrate was mounted on an aluminum frame over a 3/4" dia. hole. A mask, also with a 3/4" dia. hole, was spaced 0.020" from the carbon foil.

Test of Polyimide Foils for Beam-Foil Spectroscopy

John O. Stoner, Jr., Peter W. Rathman, David J. Walker,
John A. Leavitt, and Stanley Bashkin
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We introduced polyimide foils in a 3 MeV beam of Kr^+ to test their suitability for excitation of beam-foil spectra. The ion beam flux was approximately 4 microamperes through a 5-millimeter hole. Foils with a nominal thicknessof 24.5 micrograms/cm^2 were kindly provided by J. Pauwels, who reported on some of the properties of such foils at the 1979 meeting of the International Nuclear Target Development Society.

Although conventional arc-evaporated carbon foils with surface densities of about 6 micrograms/cm^2 lasted about 3 minutes in our beam, the polyimide foils broke within ten seconds. The heavily-bombarded parts of the foils were completely removed in that time. Parts of the foils that sustained only momentary bombardment became brown-colored, and the boundaries of the heavily-bombarded parts of the foils were curled and brown-colored.

We suggest that such foils will be useful only in beams for which little heating occurs (e.g., the 6 MeV alpha-particle beams used by Van Gestel and Pauwels).

Uniform Carbon Coatings Over Long Narrow Substrates

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We recently needed to coat a flat Pyrex substrate 64 mm x 305 mm (x 44 mm thick) with a uniform arc-deposited carbon coating having surface density near 20 micrograms/cm^2.

With an arc-to-substrate distance of 60 cm (about the maximum we could hope to obtain in a standard bell jar) and assuming a cosine distribution of evaporant emitted by the arc, we would expect at least a 13 % variation in thickness from center to end. This would have been unacceptably large. We therefore calculated the thickness variations to be expected with the use of two evaporation cycles, with the arc symmetrically-located at two different places relative to the substrate. The results of these calculations showed that, for an arc-substrate separation equal to the length of the substrate, and with the arc located at $\pm \tan^{-1}(1/2) = \pm 26.6°$ from the normal to the center of the substrate, surface-density variations should be less than 4 % across the substrate. In fact when we carried out this process, we obtained a range of surface densities from 19.0 to 20.8 micrograms/cm^2, i.e., varying by about $\pm$ 4.5 %, significantly better than the 13 % variation expected from a single evaporation. The fact that this variation was about twice as great as we expected was traced to the nonideal emission properties of the arc. Such effects can presumably be reduced substantially by increasing the arc-substrate separation somewhat while keeping the angle (as defined above) unchanged.

Marie Antoinette SAETTEL

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During the last semester, the preparation of ^{232}Th layers was the main part of my work. These targets were made by electrolysis and it will be the subject of my paper for the next conference in Oak-Ridge. The method can also be extended to uranium deposits.

Beside this, many usual targets were prepared such as ^{18}O (as Nb_2O_5 and Ta_2O_5 self-supporting), ^{15}N, ^{68}Zn on carbon backings, Ag_2S self-supporting with natural S, ^{44}Ca, ^{24}Mg and ^{26}Mg by evaporation and rolling.

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Target laboratory activities of the last year

A. Hydrogene targets

1. By implantation of hydrogene in a backing

A proton beam made in our ion-beam sputtering unit (Danfysik, Denmark), was accelerated to the desired voltage (2-10 KV) and defocussed until the desired diameter (variable from 1 mm Ø till 20 mm Ø). The backing has been put on the beam-stop. Hydrogene has been implanted in this way in the next backings : 0.2 mm thick zirconium and 25 μm thick gold.

2. By reactive sputtering of titanium with a mixture of 25 % argon and 75 % hydrogene

A Ti H_x-layer is formed in this way on the backing. The "x" was not yet estimated, but is probably about 1.8.
I used the above mentioned gas-mixture, to obtain a complete hydrogenated Ti-layer with an areal density of $\approx$ 200 $\mu g/cm^2$ within an acceptable time (12 hours). It is possible to procedure Deuterium- and Tritium-targets in the same way. As the consumption of the gas-mixture is very low, it is cheap to prepare D- and T-targets in this way. The targets, described under 1 and 2, have been used for the reaction $^{1}H(^{18}O,p\gamma)^{18}O$ $E(^{18}O)$ = 48.5 MeV.

B. Targets of noble gas isotopes

These targets were prepared by ion-implantation (defocussed ion-beam) in a metal substrate with acceleration-voltages varying from 2-10 KV. The following targets have been fabricated : Neon-22 and Argon-40 in 0.3 mm thick Tantalum; Helium-4 in Magnesium-(25 μm), Gold-(15 and 25 μm), Silver-(25 μm), Titanium-(3 μm) and Nickel-foils (2 μm).
I didn't succeed in an attempt to implant helium-4 in a 25 μm thick gadolinium-foil.

C. Germanium-targets

Germanium-powder has been sputtered with an Ar^+-beam on a glass slide, on which $BaCl_2$ has been evaporated. ($BaCl_2$ according to D. Ramsay, Proceedings of the 1974 Conference of I.N.T.D.S.). About 50 % of the Ge-layers (35-300 $\mu g/cm^2$) could be mounted on the frames, without damaging the Germanium-targets.
When I used KCl as releasing agent, 100 % of the Germanium-targets (same thickness) could be mounted on the frames without damage to the targets.

Albert H.F. Muggleton

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Target Laboratory Activities, November 1979 - June 1980

1. Heavy-Ion Sputtering Assembly

The construction of a heavy-ion sputtering facility is still progressing : initial teething troubles due to electrical breakdown have been eliminated by design modifications. Problems are still being experienced with obtaining a sharply focussed beam spot but modifications to the lens system are confidently expected to overcome this difficulty.

2. Stripper Foils

Efforts to exploit the apparent superiority of glow discharge ethylene foils have been disappointing. The glow discharge foils hastily installed at the last tank opening of our 14UD Van de Graaff accelerator were inferior in performance to standard foils produced by vacuum evaporation. It was apparent that the glow foils were too thick. As measured by use of the light transmission device used for standard foils, the glow foils had transparencies of 37 % to 71 %. Experience with similar foils at Oak Ridge suggests that glow foils absorb half the light of equal thickness evaporated foils. Some desultory efforts to confirm this were inconclusive. Nevertheless, accepting it as a "rule of thumb", this implies that the foils previously tested varied from 7 micrograms/cm^2 to 20 micrograms/cm^2. Since our standard evaporated foils of 3 to 5 micrograms/cm^2 have 60 % to 74 % light transmission the poorer performance of the glow foils can be ascribed to their excessive thickness.

Further batches of glow foils have been made using a bell jar based system. The only ones checked to date have light transmissions

of 87 %, corresponding to a thickness of 3 micrograms/cm^2.
These foils were tested with a silicon beam and had apparent lifetimes (ie 50 % beam) of about 12 hours, quite comparable to standard evaporated foils.
To date the strengthening layer of collodion applied to all stripper foils has been removed from the centre of the foil using the heat from a focussed projector lamp. This technique is laborious, (as each foil is handled individually) and unreliable because the temperature to which the foil rises under the lamp depends on the opacity, ie the thickness of the carbon. Consequently, if the lamp intensity is high enough to burn collodion off a 3 microgram/cm^2 foil than for a 5 microgram/cm^2 foil the lamp will tend to burn the carbon itself.Such "pockmarking" of foils is easily observed. Other laboratories have found that heating the mounted foil in air at 250°C was sufficient to remove the collodion. An adaptation of this procedure was used. After the foils had been mounted in the changer mechanism the end of the device containing about 50 foils was inserted into an oven at 320°C and the changer actuator operated sequentially to cycle through the full load of foils.
The criterian used to date has been to observe relaxation of the foil where collodion has been removed. Tests performed with an oxygen beam and an Enge spectrometer indicate that even these burnt-off foils still have a residual collodion thickness equal to about 20 % of its 10 microgram/cm^2 initial value. With continued exposure to beam of sufficient intensity the residual collodion was removed. Subsequent exposure to beam resulted in a gradual decrease in elastic scattering counting rate. This apparent initial reduction in foil thickness with exposure is in contradiction with the experience of other laboratories using much higher intensities. Whether this is an intensity dependent effect, or the foils made elsewhere were in an important way different to those made at ANU is not known. At present we do not know the efficiency of our technique. Further work is being undertaken to repeat absolute thickness determination for all our foil types.

3. Thin Au and Ag Single Crystal Films

Thin, single-crystal films of gold or silver are required to test quantum mechanical predictions for the scattering of channelled particles in thin single-crystals.

It is necessary to restrict the thickness of these single-crystal films to that at which there is a small probability that a particle, travelling at some maximum angle of interest θ_{max} to the crystal axis will cross to a neighbouring channel. For gold this thickness is ~ 300Å (~ 60 microgram/cm^2).
A second requirement for these single-crystal films will be to look for coherent x-rays. The thickness requirements in this case are less critical than those needed for the observation of channelling effects. However, the film thickness must be small enough so that the channelled particle energy loss in passing through the crystal is small compared with the incident energy (~ 5000Å, ie ~ 1 mg/cm^2 for 1 % loss of 20 MeV proton).
Attempts to date at producing good, single-crystal films of gold or silver have been disappointing. All attempts have been to obtain epitaxial growth by evaporating gold or silver onto single crystal NaCl or freshly cleaved mica. The resulting films were stripped from the substrate and tested for crystallinity and defect density using electron microscopy and high energy electron diffraction. All samples, to a greater or lesser extent showed some degree of crystallinity but also exhibited a higher density of random and twinned material. Most of the work has concentrated on a pulse-and-anneal technique. However, no improvement over straight deposition at elevated temperature has been observed.
Deposition parameters are typically :

NaCl substrate

Anneal NaCl crystal in a good vacuum at 300°C for 1 hr. Pulse gold for 0.1 sec at a deposition rate of ~ 100Å/sec, with substrate maintained at 300°C. Anneal for 30 minutes at 400°C. Cool to 300°C and deposit remainder of gold to the required thickness at ~ 5Å/sec.

Mica substrate

Same technique used for gold and silver. Conditions similar to those used for NaCl except higher (350°C) and lower (250°C) substrate temperatures were maintained during deposition.
Gold on silver and gold on NaCl on mica were tried without success.
Work is continuing, using different combinations of the parameters outlined above. Any useful advice or experimental suggestions from other workers in this field would be welcomed.

4. Thin, High Purity, Self Supporting ^{24}Mg and ^{28}Si Films

In the $^{24}Mg(^{24}Mg,^{20}Ne)^{28}Si$ reaction a significant background from carbon and oxygen impurities in the ^{24}Mg target obscures ^{20}Ne reaction products. Attempts to fabricate self-supporting carbon and oxygen free ^{24}Mg targets (approx. 100 $\mu g/cm^2$ thick) have to date been unsuccessful. I would welcome any information on making such a target.

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Target Laboratory activities 1st Semester 1980

1. Services.

Sample preparation is a traditional activity of CBNM primarily conceived as a public service support, not only to the nuclear data programme of CBNM, but also to the national laboratories of the European Communityas well as to many universities and other laboratories.
During this reporting period 466 samples were delivered covering 67 different applications. Details are given in Table 1.
Samples in the form of thin films or foils were prepared by electrospraying, including suspension spraying, spraypainting, vacuum deposition, cathodic sputtering or rolling.
Alloys with certified composition or bulk samples were produced by melting, including high frequency levitation melting, rolling, powder metallurgy and conventional machining techniques.
The degree and quality of the characterization of the samples and targets depended on the application. It varied from a crude film thickness measurement by an oscillating quartz device to the extremely accurate mass assay by α- counting and isotope dilution mass spectrometry.

2. New preparation technique for deposits

It is the objective of this work to adapt a commercial piezo-electric pulsed jet device - developed by GOULD Instrument Systems Div. for character printing with ink - to the quantitative preparation of uniform deposits of scarce isotopes.
In this jet device a tubular ceramic piezo-electric transducer surrounding a glass tube terminating in a nozzle and filled with a metal acetate solution in methanol instead of ink is used.

Each electric pulse causes contraction of the transducer and the glass tube and results in the production of a droplet.
Preliminary results with Cu-acetate indicate that the drop size (1.10^{-6}ml) is constant and that uniform deposits with high packing density can be prepared by using a suitable programmed X-Y stepper motor driven table. A counter registering the number of pulses has been built so that the number of drops, is known.
There remain problems to be solved mainly concerning the compatibility of the components of the jet device with the acetate methanol solutions and the reliability of the technique especially for glove box work.

3. Spectrophotometric definition of targets

The non-destructive determination of the thickness of thin plastic foils, used as substrate in nuclear target preparation and of thin vacuum deposited layers was investigated by visible light spectrophotometry. Polyimide, polysulfone, vyns, formvar and parylene were measured in transmission mode.
Calibration of the method was carried out using sets of foils characterized by weighing and alpha particles energy loss. In the range of 10 to 100 $\mu g/cm^2$, reproducibilities of $\pm$ 0.5 $\mu g/cm^2$ (standard deviation on a single measurement) can be obtained. The obtainable accuracy is presently evaluated at $\pm$ 2 %. Using a reflection device the method could also be used for the thickness determination of evaporated UF_4, ThF_4, PuF_3 and LiF layers on metallic substrates. In the range of 20 to 500 $\mu g/cm^2$ reproducibilities of $\pm$ 5 $\mu g/cm^2$ can generally be achieved. Calibration was based on weighing for LiF and on alpha activity measurements for actinides.
Although further optimalisation of the method remains necessary, it already proved to be of high practical interest in target production control because of its simplicity and speed. A measurement, including spectrum evaluation and calculation of the thickness, can be carried out in approximately 5 minutes.

TABLE 1

Samples delivered first semester 1980

Materials	Applicants (See list (a))	Number of samples	Definition Method (See list (b))	Preparation Method (See list (c))
U_{nat}	(1) (2)	16	2πa - IDMS	VD
^{233}U	(2)	4	a-count - IDMS	VD
^{235}U	(1) (2)	7	a-count - IDMS	VD
^{235}U	(2)	4	a-count - IDMS	ES
^{235}U + Ta	(3) (4)	60	a-count - IDMS	P - CS
^{238}U	(2)	18	2πa - IDMS	VD
^{237}Np	(2)	3	a-count.	VD
^{239}Pu	(2)	2	a-count - IDMS	VD
^{240}Pu	(5)	1	MD	Canning
^{240}Pu	(2)	2	a-count - IDMS	VD
^{241}Pu	(6)	1	a-count - IA	C.P
^{242}Pu	(1)(2)(5)(7)(8)	30	a-count - IDMS	VD
^{242}Pu	(9)	1	MD	Canning
^{244}Pu	(9)	1	MD	Canning
^{104}Pd	(2)	1	MD	M - R - Mach.
^{108}Pd	(2)	1	MD	M - R - Mach.
^{54}Fe	(2)	3	MD	Pr - Canning
^{56}Fe	(2)	1	MD	Pr - Canning
^{53}Cr	(2)	1	MD	Pr - Canning
^{208}Pb	(2)	2	MD - DC	Red - M - R - Mach.
U-0.5% ^{242}Pu	(2) (10)	39	QA	LM
V-2% ^{238}U	(11)	20	QA	LM - S
V-30% Ga	(12)	2	QA	LM
V-15% Si	(12)	3	QA	LM
Cr-30% Mn	(13)	2	-	M
Cr- 7% Si	(13)	2	-	M
Pb- 8% In	(1)	4	Cal. M.P.	M - R - WD
Pb	(1)	15	Cal. M.P.	M - R - WD
Bi	(1)	15	Cal. M.P.	M
Zr-20 ppm Au	(14)	1	QA	LM
Al-0.1% Au	(14)	1	QA	LM
Zr	(13)	80	-	CS
^{6}Li	(2)	8	DC	M - R - Mach. - Cann.
^{7}Li	(2)	3	DC	M - R - Mach. - Cann.
^{6}LiF	(8)	14	Spectrophot.	VD
nat. LiF	(2)	2	MD	VD
C	(2)	2	MD - DC	Mach.
^{96}Ru	(1)	2	MD	ES
Cr-86.5% ^{62}Ni	(15)	1	QA	LM - R
Al	(2)	40	-	CS
Polyimide	(16)(17)(18)	51	Spectrophot.	C.P.

(a) List of applicants

(1) S.C.K., Mol
(2) C.B.N.M., Geel
(3) K.F.A., Jülich
(4) I.L.L., Grenoble
(5) C.E.A., Bruyères-le-Châtel
(6) K.F.K., Karlsruhe
(7) N.B.S., Washington
(8) H.E.D.L., Hanford
(9) T.H., Darmstadt
(10) IDA-80 - WG
(11) Dosimetry - WG
(12) U.I.A., Antwerpen
(13) Electronite, Houthalen
(14) A.T.E.C., Turnhout
(15) R.U.G., Gent
(16) Univ. Arizona
(17) T.U., München
(18) Univ. Bordeaux

(b) List of Definition Methods

(B) a-count : Counting of a-particles
(JRC) IDMS : Isotope Dilution Mass Spectrometry
(FRG) MD : Mass definition
(F) Spectrophot. : Spectrophotometry
(F) QA : Quantitative Alloying
(FRG) DC : Dimensional Control
(USA) IA : Isotope Analysis
(USA) Cal.M.P. : Calibration of Melting Point
(FRG)
(Intern.)
(EC)

(c) List of Preparation Methods

(B) ES : Electrospraying
(B) VD : Vacuum Deposition
(B) R : Rolling
(B) LM : Levitation Melting
(USA) Pr. : Pressing
(FRG) P : Painting
(F) M : Melting
CS : Cathodic Sputtering
Red. : Reduction to metal
Mach. : Machining
C.P. : Chem. Preparation
S : Swaging
WD : Wire drawing