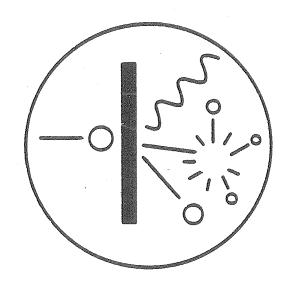
# INTERNATIONAL NUCLEAR TARGET DEVELOPMENT SOCIETY

# NEWSLETTER



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The INTDS Newsletter is an informal source of information for and from the Membership.

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

# CONTENTS

		<u>Page</u>
	In Memoriam - Darrell Erikson	1
•	Future Directions of INTDS	2
	Production and Development Activities of the Isotope Research Materials Laboratory at Oak Ridge National Laboratory (ORNL) during October 1987 through March 1988 W.S. Aaron Oak Ridge National Laboratory, USA	4
	Targets Prepared at Daresbury Laboratory during 1987  J.B. Reynolds  Daresbury Laboratory, UK	12
	Laminar Structure in Strained Evaporated Carbon Target Foils John O. Stoner, Jr. University of Arizona, Phys. Dept., Building 81, Tuscon, AZ 85721 and ACF - Arizona Carbon Foil Co., Inc., 2239 E. Kleindale Road, Tuscon, AZ 85719, USA and Robert B. Stoner ACF - Arizona Carbon Foil Co., Inc., 2239 E. Kleindale Road, Tuscon, AZ 85719, USA	13
	Activity Report A.H.F. Muggleton, L.K. Fifield and N.A. Orr Dep. of Nuclear Physics, Research School of Physical Sciences; The Australian National University, GPO Box 4, Canberra, Act 2601, Australia	17
	Activity Report at ISN Grenoble R. Blanc, M. Bouriant, J.P. Richaud Institut Sciences Nucléaires, 53, Avenue des Martyrs, 38026 Grenoble, France	20

- Statement of Accomplishments
   John P. Green and George E. Thomas

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   9700 South Cass Avenue, Argonne, III 60439, USA)
- INTDS Membership list of June 17, 1988

23

#### IN MEMORIAM - Darrell ERIKSON

We are sad to inform you of the death of our colleague Darrell Erikson.

Darrell died in an automobile accident in February of this year. He is survived by his wife Pat and their children Lynn and Reid. He lived in Castro Valley, California. Darrell was born in Fargo, North Dakota in 1940. He received a BS and MS in mechanical engineering from the University of California at Berkeley. Darrrell had worked at the Lawrence Livermore National Laboratory since 1981. He was a member of the Vacuum Processes Group and was well known for his expertise in vacuum science and engineering.

He was also a member of the American Vacuum Society.

In 1986 Darrell was elected on the Board of Directors of the INTDS.

Our special thoughts go to his wife and two children. His numerous friends of INTDS will miss his friendliness and professionalism.

Woody Weed LLNL J. Van Audenhove Editor INTDS

#### FUTURE DIRECTIONS OF INTDS

The INTDS was officially formed at the University of New York at Stony Brook in 1973. The purpose for forming the Society was to bring together, on a regular basis, those individuals interested in exchanging ideas relating to materials availability (typically highly enriched isotopic materials), sample fabrication techniques, sample characterization, and associated related subjects of interest. The INTDS has answered a need for communication of information in many areas of material science, including target and special nuclear materials preparation, through publication of newsletters and conference proceedings.

For the past few years, there has been a general concern on the part of many INTDS members that continued decreases in funding for atomic and nuclear physics research may result in our Society membership decreasing to a subcritical number. This resulted in discussions at our 1985 INTDS Board meeting concerning the desirability of broadening the membership base of the INTDS. At the 1986 INTDS Board meeting, a committee was appointed to formulate a proposal on this subject and present this proposal at the 1987 INTDS Board meeting. The members of the committee are listed below.

Harold Adair, Chairman Jan Van Audenhove Helmut Folger Hans Maier Geirr Sletten Bill Lozowski

Suggestions on specific directions the Society should go or groups the Society should try to attract to improve its viability were received before and after the 1986 INTDS Board meeting. A summary of the areas of interest of individuals for which the INTDS may wish to try to attract is given below.

- 1. Thin films
- 2. Metals and alloys
- 3. Ultra high-purity materials
- 4. Single crystals
- 5. Isotope production
- 6. Preparation of standards and sources
- 7. Research sample fabrication and characterization

As you are well aware, many of these topics are very broad; however, I think you will also agree that as target makers we really have an interest in all these areas. The samples we prepare have primarily in the past been used in the area of nuclear research. However, the expertise we have developed should be of interest and benefit to many areas of material science. Although the list of areas of interest above is not an exhaustive one, I think it should be evident that perhaps a name change for our Society would be in order. The title implies that our main interest is in nuclear targets, which has been our main thrust in the past. However, I think we should change the name of our Society to perhaps the "International Research Materials Society," which would be more in align with the direction I think the Society should be going.

I don't believe it is our objective as a Society to grow to a size where we will lose the benefits we have enjoyed through personal knowledge of the activities and expertise of the membership. However, it should be recognized that if our scope remains too narrow, our survival as a viable Society for many years to come may be questionable.

# PRODUCTION AND DEVELOPMENT ACTIVITIES OF THE ISOTOPE RESEARCH MATERIALS LABORATORY AT OAK RIDGE NATIONAL LABORATORY (ORNL)

DURING OCTOBER 1987 THROUGH MARCH 1988\*

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#### W.S. AARON

S. D. BRANDEL J. R. GIBSON
K. B. CAMPBELL W. B. GRISHAM
J. M. DAILEY L. A. ZEVENBERGEN
M. PETEK C. B. FINCH<sup>1</sup>

The Isotope Research Materials Laboratory (IRML) at the Oak Ridge National Laboratory (ORNL) serves a number of functions, including the production and sale of research materials, research and development for improved enriched isotopic materials processing, and a wide variety of production and/or development efforts in materials preparations. IRML's activities during the six-month period of October 1987 through March 1988 are summarized below.

#### RESEARCH MATERIALS PREPARATIONS

The IRML completed and/or shipped during this period a total of 271 research materials preparations through the Isotope Distribution Office. The specific preparations are shown in Table 1.

Highlights among those preparations include an ingot of <sup>6</sup>Li-Mg weighing over 140 g. Special melting, casting, and characterization techniques were developed or used to produce a homogeneous alloy. The dissimilar melting points and densities of these two metals, as well as their reactivities, were the source of alloying problems overcome in this effort. A total of 20 discs of <sup>7</sup>Li metal that were 3 cm in diameter by 2, 3, and 5 mm thick were also prepared in IRML's lithium facility.

Pyrochemical reduction/distillation, followed in some cases by rolling, was used to prepare samples of magnesium and calcium isotopes. In the case of

<sup>&</sup>lt;sup>1</sup>Solid State Division (ORNL)

<sup>\*</sup>Research sponsored by the Division of Basic Energy Sciences and Division of Nuclear Sciences, U.S. Department of Energy, under contract DE-A05-840R21400 with the Martin Marietta Energy Systems, Inc.

Table 1. Research materials prepared during October 1987 through March 1988

Isotope	Quantity of Material Shipped (g)	Number of Preparations
6 <sub>Li</sub> _N <sub>Mg</sub>	141.670	1
7 <sub>Li</sub>	20.191	20
$^{24}$ Mg	0.932	2
26 <sub>Mg</sub>	0.171	6
40 <sub>Ca</sub>	0.932	1
48 <sub>Ca</sub>	0.002	1
$48_{ ext{Ti}}$	0.369	1
52 <sub>CT</sub>	0.002	3
64 <sub>Zn</sub>	0.020	2
68 <sub>Zn</sub>	0.007	1
86 <sub>Sr</sub>	36.320	1
87 <sub>Sr</sub>	0.080	1
90 <sub>Zr</sub>	1.006	3
91 <sub>Zr</sub>	0.285	1
96 <sub>Zr</sub>	0.019	2
113 <sub>In</sub>	0.031	1
142 <sub>Nd</sub>	0.018	4
144 <sub>Nd</sub>	0.026	6
146 <sub>Nd</sub>	0.597	1
148 <sub>Nd</sub>	0.055	2
152 <sub>Sm</sub>	0.022	2
174 <sub>Yb</sub>	0.033	2
176 <sub>Yb</sub>	0.023	2
233 <sub>U</sub>	0.059	1
235 <sub>U</sub>	104.064	17
238 <sub>U</sub>	45006.932	180
238 <sub>Pu</sub>	0.001	1
243 <sub>Am</sub>	0.761	2
252 <sub>Cf</sub>	6.4 ng	2

<sup>48</sup>Ca (\$280/mg), a 1-mg/cm<sup>2</sup> <sup>48</sup>Ca foil was overcoated on one side with a 1-mg/cm<sup>2</sup> layer of gold using radio-frequency sputtering at a slow deposition rate. Precision rolling techniques were used to prepare a variety of foils from isotopes of titanium, zinc, zirconium, and indium as well as several rare-earth metals that had been reduced by the reduction/distillation process.

A large-scale reduction/distillation of <sup>86</sup>Sr was performed with a yield of 91% to produce 36.32 g of metal. This metal was then loaded as chunks into an 8.6-mm-I.D. stainless steel tube and repeatedly melted to ensure maximum density of the metal within the tube, eliminating any porosity or voids.

Electron bombardment evaporation was used to prepare  $250-\mu g/cm^2$ -thick deposits of  $^{142}Nd_2O_3$  and  $^{144}Nd_2O_3$  on  $30-\mu g/cm^2$  carbon backings. Each set of deposits was made on four microscope slides (2.5 cm x 7.5 cm) for the convenience of the customer in floating and mounting these targets on frames. Similar methods were used to prepare eight lantern slides with a 40- to  $50-\mu g/cm^2$  carbon layer followed by electron beam evaporation of approximately  $420~\mu g/cm^2$  of  $^{238}UO_2$  and finally a  $10-\mu g/cm^2$  carbon overlayer. Electron beam evaporation of  $^{238}UO_2$  was also used to prepare 150 fast-flux neutron monitors as a  $^{238}UO_2$  deposit on nickel backings.

The preparation of electron-beam evaporated deposits of  $^{235}\text{UO}_2$  of various thicknesses up to 5 mg/cm² on 7-cm x 30-cm aluminum plates continued with the production of an additional 16 samples for use in Sandia National Laboratory's Falcon Laser Development program.

Two foils each of  $^{238}$ U metal, 2.5 cm diam x 0.254 and 0.127 mm thick, having better than +/-5% uniformity, were prepared by rolling and then overcoating with aluminum to minimize oxidation. IRML was requested to clean and overcoat with aluminum a total of 45 kg of depleted uranium in the form of 18 circular plates that were procured by ORNL's Engineering Physics and Mathematics Division from an outside vendor. The plates are to be used as neutron filters. The plates were cleaned by chemical etching and immediately overcoated with  $50-\mu g/cm^2$  of aluminum metal by resistance evaporation. The aluminum overcoating is intended to minimize oxidation of the uranium metal and reduce contamination during handling.

One gram of  $^{243}$ Am as oxide was converted to metal for use as a safeguards standard using the reduction/distillation process. A reduction yield of over 80% was obtained, and after arc melting and sampling, a 0.760-g bead of metal was shipped to the customer. This metal has an isotopic purity of

98.045 at. % <sup>243</sup>Am and total impurities of approximately 2,500 ppm. Severe equipment problems were encountered in preparing this metal since IRML's actinide reduction/distillation system is approximately eight years old and only minimum levels of maintenance on the vacuum pumps can be performed because of the extremely high contamination levels present in the system. A request for replacement of this equipment with a more maintenance-free vacuum system was submitted and approved.

Two <sup>252</sup>Cf sources were prepared by the self-transfer process from a mother source. Platinum substrates were prepared by rolling to 0.25 mm thick, punching to the approximate diameter, and machining to a final diameter of 2.159 cm. Deposits of 1 ng and 5 ng were then made on a 1-cm-diam spot centered on the platinum substrates. Following counting of the deposits to quantitatively determine the amount of <sup>252</sup>Cf present, the sources were welded inside special chambers supplied by the customer.

#### RESEARCH AND DEVELOPMENT

Development of methods to plate relatively thick  $(0.5\text{-mg/cm}^2)$  deposits of uranium oxide on thin carbon foils  $(40\text{--} \text{to } 50\text{--}\mu\text{g/cm}^2)$  continued this period. It became evident that in order to obtain quality targets by electroplating, in contrast to vacuum deposition, the carbon film preparation technique and, specifically, the nature of the parting agent are of paramount importance. Carbon foils prepared with different parting agents were found to form distinctly different  $U_XO_Y$  deposits, particularly in regard to their adherence and morphology. When soap or NaCl was used as the parting agent, the deposits were bright yellow flakes which would fluff off easily by merely shaking the specimen.

Better adhesion was obtained when betaine-sucrose was used as the parting agent, but the deposit could be wiped off with gentle swiping with paper. In order to affect the mechanism of the uranium deposition, small amounts of acetylacetone were added to the solution. Acetylacetone is known to form complex compounds with uranium, which was evident by the yellow coloration of the plating solutions containing acetylacetone. The reduction kinetics of the uranyl acetylacetone complexes are different than that of the free uranyl ion, and it was expected that this might have a positive effect on the deposit morphology and adherence. It was found that the rate of uranium deposition

diminished with increasing acetylacetone concentration. At 0.3 vol % acetylacetone, no uranium plating was observed after a 30-min plating at 1 mA/cm². The best deposit adherence was obtained for 0.015 to 0.03 vol % acetylacetone. While at concentrations of <0.003 vol %, the plating rate approached that of solutions without acetylacetone, and the deposit was no longer sufficiently adherent. Very good deposits were obtained at the following conditions:

4.6 mg of UO<sub>2</sub> in 10-mL isopropanol, 0.02 vol % acetylacetone, 30 min at 1.5 mA/cm<sup>2</sup>, 15<sup>o</sup>C, and anode rotation 200 rpm.

When floating off such a deposit, about 5% of the material was removed from the target by flaking or other physical losses. It appears that a thin overcoat of  $10-\mu g/cm^2$  carbon would alleviate such losses.

In similar experiments with soap as the parting agent and concentrations of 0.3 to 0.003 vol % acetylacetone in the plating solution, the carbon tended to peel off during or immediately after the electrodeposition. Further experiments with NaCl are not being considered at this time because of concerns over possible residual sodium and chlorine in the targets.

As a result of limited supplies of some barium isotopes, electroplating is the preferred method for preparing barium targets on carbon foils. Although acetone was recommended in the literature as the best medium for electroplating barium on carbon, it was found that better adhering deposits were obtained with isopropanol. It was observed that some of the BaO deposit dissolves during the floating process, but this can be prevented by overcoating with a thin layer of carbon  $(10-\mu g/cm^2)$  prior to floating off. The carbon overcoating effectively seals off any defects that would permit water to get through the substrate foil during the brief floating period. A method other than weighing the resulting electroplates is needed to evaluate the plating thickness since changes in overall weights are unreliable because of parting agent weight changes during electrodeposition.

Adherence problems of another type were encountered with  ${\rm UO}_2$  and boron deposits prepared by electron beam deposition. In both cases, the substrates being coated were stainless steel foils, and adherence problems were fairly random. Without any detectable changes in surface preparation or deposition parameters, a significant number of deposits were peeling off either during or

after the deposition. It was found that applying a thin (10- to  $30-\mu g/cm^2$ ) layer of aluminum eliminated the random problems while not interfering with the use of the deposits.

The limited availability of highly enriched <sup>236</sup>U precludes its preparation as a thin film by evaporation techniques, and its activity level cannot be handled in currently available focused ion-beam sputtering equipment. Electrodeposition of oxide films was, therefore, investigated as a means to prepare very thin films of this material on stainless steel substrates. As a result of development efforts, very small (milligram) starting quantities of <sup>236</sup>U can be used to prepare oxide thin films with minimal losses. electrodeposition technique was successfully applied to the preparation of two alpha sources for the Health and Safety Research Division at ORNL. The first source was to have a 1-mm-diam spot centered on a stainless steel disc that produced one count per second. It was estimated that 0.86  $\mu$ g of  $^{236}$ U would be required to produce this count rate. The second source consisted of a 3.8-mm-diam x 3.2-mm-tall stainless steel cylinder with a polished end face onto which was deposited enough <sup>236</sup>U to produce a count rate of 50 counts per second. It was estimated that 43  $\mu$ g of  $^{236}$ U would be needed to produce this count rate. The <sup>236</sup>U was electrodeposited from an isopropanol solution containing 0.02% acetylacetone. Because of the small geometry of the electrodeposits, modifications to the plating cell and ammeter on the plating power supply were required. The ammeter was modified to allow accurate current readings below 2 mA since the plated area was so small. High current densities at low overall currents were produced but could not be accurately monitored. In the case of the 1-mm-diam spot, lacquer was used to coat the entire disc surface, and a 1-mm-diam hole was machined through the lacquer to expose the desired substrate spot. For the cylinder, an interference fit hole in a Teflon disc was used to limit electrodeposition to one end face of the cylinder. Counting of the final sources indicated that the desired quantities of <sup>236</sup>U had been deposited. The sources are currently being evaluated by researchers in their particular application.

#### OTHER ACTIVITIES

IRML's extensive capabilities in electron beam evaporation of metals and oxides and past experience with thermographic phosphors resulted in an order

for development work on the preparation of a series of rare-earth phosphor coatings for use by Los Alamos National Laboratory (LANL) in neutral-particle-beam sensing studies. Coatings of  $Y_2O_3$  doped with 6.8 wt % europium were deposited to thickness of 5, 10, and 20  $\mu$ m on 20-cm x 20-cm aluminum sheets, 2.5-cm x 20-cm aluminum strips, and a variety of metal ribbons and wires as well as graphite fibers. Attempts to coat glass plates and highly polished surfaces of chromium-plated stainless steel were suffered from oxide film adherence problems that could not be overcome without degrading the smooth and/or polished substrate surfaces. Even the use of intermediate bonding layers, such as aluminum, did not significantly alter adherence. These problems were particularly prevalent at thicknesses greater than 1 to 2  $\mu$ m. An 18-cm x 18-cm nickel grid, identical to those used for neutralizer foils, was successfully coated with a 12  $\mu$ m layer of this phosphor.

This same phosphor will be used, at least initially, in a new Isotope Production Department development project in which IRML will be participating. The new project involves the preparation of phosphor layers that are in intimate contact with titanium or other hydride-forming metals which will then be tritiated at IRML's Tritium Target Fabrication Facility in an effort to produce improved radioluminescent (RL) lights that are powered by a solid tritide rather than a gaseous tritium source.

In cooperation with the Solid State Division of ORNL, IRML continued development efforts on the preparation of  $^{147}\text{Pm}\text{-doped}$  single crystals and glasses. This project began in September 1986 with the high-pressure ion exchange separation of  $^{147}\text{Pm}$  from its samarium decay product. This  $^{147}\text{Pm}$  has since been used to dope a variety of crystals and glasses in an effort to develop a  $\text{Pm}^{3+}$  laser at 933 and 1100 nm. During this period, two  $\text{CaF}^2$  chips, (m.p.  $^{-1425^{\circ}\text{C}}$ ) single crystals doped with 0.5 wt %  $^{147}\text{PmOF}$  were prepared using the Bridgman technique. Typically, an initial charge of 4.00 g of  $\text{CaF}_2$  chips, 0.020-g of  $^{147}\text{PmOF}$ , and 100-mg of  $\text{NH}_4\text{HF}_2$  is added to a vitreous carbon crucible and lowered through an induction heater hot zone at a rate of 1.5cm/h under flowing argon. The  $\text{NH}_4\text{HF}_2$  was added as a fluoridizing agent that is lost during heat-up by vaporization. The resulting single crystals were a transparent green in color.

A series of BZILT glass samples were doped with 0.5, 1.0, 2.5, and 5.0 wt % PmOF by melting the BZILIT<sup>2</sup> glass frit and PmOF at 850°C in a vitreous carbon crucible under an atmosphere of argon - 3 vol % NF3. The NF3 served to convert the PmOF dopant to PmF3 and to eliminate oxide and hydroxide impurities during fusion. The samples were held in a molten state for 1 h at 850°C in the vitreous carbon crucible under a 100-mL/min flow of Ar - 3 vol  $% NF_3$ . The glass was then cast into brass molds at 250°C. All of the glass samples displayed intensities of reddish self-luminescence which were commensurate with dopant content. They were transparent lavender on initial casting but rapidly darkened with radiation damage. As expected, this damage could be removed by annealing the samples.

<sup>&</sup>lt;sup>2</sup>BZILIT GLASS MOL %

BaF<sub>2</sub> 30  $ZnF_2$ 20 InF<sub>3</sub> ThF<sub>4</sub>  $LuF_3$ 30 10

#### **Activity Report**

#### Daresbury Laboratory

#### J.B. Reynolds

Targets prepared during 1987:

B, Mo 92,94,96,98, Er, Mg<sup>24</sup>, Dy<sup>162</sup>, Hg<sup>204</sup> (at last !), Pb<sup>208</sup>, Cr<sup>50,52</sup>, Sc, Fe<sup>56</sup>, Ni<sup>58</sup>, Te<sup>130</sup>, Ge<sup>74</sup>

The reductions of the oxides of Ga, Ge, Ti, Si isotopes to element were performed.

#### Laminar Structure in Strained Evaporated Carbon Target Foils

John O. Stoner, Jr.

Physics Department, University of Arizona, Tucson, AZ 85721, and ACF - Arizona Carbon Foil Co., Inc., 2239 E. Kleindale Road, Tucson, AZ 85719

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Arc-evaporated carbon films used for accelerator targets and for substrates in electron microscopy usually have no long-range structure, i.e., they are completely amorphous. This assumption is supported by x-ray and electron diffraction measurements, which show only one or two diffuse rings, and by conventional transmission electron micrographs using carbon-film substrates. Except for phase noise due to interference within the electron beam, such micrographs typically show no structure in the films.

We recently had occasion to study via high-magnification scanning electron microscopy the torn edges of a carbon film. This film was evaporated in the conventional manner<sup>2</sup> from a carbon arc at a pressure of 3x10<sup>-5</sup> torr. The substrate was a microscope slide that had been previously coated with detergent and then polished until no trace of detergent could be seen by eye. Optical calibration<sup>3</sup> showed the surface density to be 20±2 µg/cm<sup>2</sup>, with a measured density of 2.00±0.02 g/cm<sup>3.4</sup> this corresponded to a thickness of 100±11 nm. The foil was floated onto a distilled water surface and then torn into small pieces while floating. Fragments were picked up on polished silicon substrates and dried; they were then coated with tungsten to a thickness of 2 nm at an angle of 18 degrees to the surface plane while the samples were rotated continuously about the surface normal. They were then studied in a Hitachi S-800 scanning electron microscope operating at 25 kV; the samples were at room temperature.

Observation of several torn edges verified that the thickness of the film was 100±20 nm. One cross section showed clear indications of laminae whose normals lay in (or nearly in) the plane of the film (Fig. 1). The laminae were estimated to have thicknesses of the order of 5-10 nm. There were no signs of laminae parallel to the plane.

The layering appears to have been created by bending of the film. From Fig. 1 it can be seen that the normals to the laminae lie perpendicular to the axis of the approximately cylindrical bending of the film in the neighborhood of the torn edge.

The purpose of this note is to point out that development of layered structure in strained carbon films is not only of interest in itself, but may have consequences in accelerator targetry and in specimen analysis in electron microscopy. Evaporated carbon films are usually in compressive stress on their substrates and, when exposed to humid atmospheres, often wrinkle, forming closely-spaced, narrow, and strongly bent ridges. If laminae are formed in these, energy losses of particles passing through them may be affected, increasing energy straggling for the foil as a whole. Biological specimens attached to carbon substrates and then dried often cause the foil to crinkle as they dry. Where carbon specimen mounts have or have had strong curvature, one may suspect the presence of artifacts in electron micrographs due to such laminae.

K.-R. Peters, J. Krupp, and D. J. Beckler provided valuable assistance. The authors were guests of the University of California at Santa Cruz at the time this work was done.

#### REFERENCES

- 1. S. McLintock and J. C. Orr, "Evaporated Carbon Films," in Chemistry and Physics of Carbon, Vol. 11, P. L. Walker and P. A. Thrower, eds. (Marcel Dekker, 1973).
- 2. John O. Stoner, Jr. and S. Bashkin, "Production of a large unsupported carbon-film ultraviolet filter," Appl. Optics 17, 321-324 (1978).
- John O. Stoner, Jr., "Accurate measurements of carbon-foil surface densities," J. Appl. Phys. 40, 707 (1969); John O. Stoner, Jr., "Optical constants of evaporated carbon target foils," Nucl. Instrum. Methods A236, 662-664 (1985).
- 4. Robert B. Stoner, private communication (1985).
- 5. G. Gille and B. Rau, "Buckling Instability and adhesion of carbon layers," Thin Solid Films 120, 109-121 (1984).
- D. N. Braski, "A study of various parting agents for producing self-supporting thin films," Nucl. Instrum. Methods 102, 553-566 (1972).

#### FIGURE CAPTIONS

Fig. 1. Scanning electron micrograph of torn edge of carbon foil nominally 100 nm thick, coated with tungsten to nominal 2 nm thickness, 25 kV.

#### ACTIVITY REPORT

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#### A.H.F. MUGGLETON, L.K. FIFIELD and N.A. ORR

Following modifications to the laboratory's fine beam saddle field ion-source, system stability has been improved and HV breakdown considerably reduced. This ion source has been used regularly to produce targets by heavy-ion sputtering where more conventional techniques of thin film deposition by either vacuum evaporation or electroplating are impracticable.

Comparisons have been made between carbon stripper foils produced in the laboratory by the standard carbon arc technique and those produced by a glow discharge method. Sets of glow discharge foils made by two external organisations were also evaluated. Laboratory foils made by the conventional carbon arc technique gave a greater beam current and lifetimes comparable to those made by the glow discharge process, consequently it was decided to continue using the carbon arc foils.

Detailed investigations were undertaken to minimise isotopic ratio degradation and reduce unwanted contaminants in  $^{40}$  Ca,  $^{48}$  Ca and  $^{18}$  O, targets.

#### Production of Implanted Sulphur Targets

Previous work in this laboratory employing reactions on  $^{36}$  S has used Ag<sub>2</sub>  $^{36}$  S targets produced from sulphur enriched to 82% in  $^{36}$  S. Backgrounds due to the 18% of  $^{34}$  S and silver have, however, prevented the study of low-yield reactions such as  $^{36}$  S( $^{18}$  O, $^{17}$  F) $^{37}$  P. Consequently a system has been developed for

producing very pure targets by implanting mass-analysed  $^{36}\,\mathrm{S}^-$  beams into carbon foils.

Implantation is performed during periods of accelerator inactivity and utilises the sputter source to produce negative sulphur ions which are mass-analysed by the 90° inflection magnet. The 30  $\mu \rm g/cm^2$  carbon foils are inserted into the path of the beam and the beam current measured in order to monitor the progress of the implantation. The source material is  $\rm Ag_2\,S$  enriched to ~90% in  $^{34}\,\rm S$  but which also contains 0.7%  $^{36}\,\rm S$ . It was found that implantation at energies greater than ~100 keV resulted in an unacceptably high foil failure rate. Thus, despite the poorer beam transmission, all subsequent implantations have been performed at 50 keV. Foils produced via a glow-discharge technique proved more robust than regular evaporated foils. However it proved very difficult to prevent contamination of these foils by the NaCl release agent, therefore their use was abandoned.

Ions are implanted over an area 5 mm in diameter. A uniform distribution over this area is ensured by rastering the beam using an orthogonal pair of magnetic steerers. The currents in these are varied under computer control. Typically, a carbon foil is exposed to 10-20 nA of  $^{36}\,\mathrm{S}^-$  beam for 24-36 hours, resulting in a total exposure of 2-3 mC. Targets produced in this fashion have thicknesses of ~5  $\mu\mathrm{g/cm^2}$ . Measurements using the  $^{36}\,\mathrm{S}\,(^{13}\,\mathrm{C},^{12}\,\mathrm{C})^{37}\,\mathrm{S}$  reaction have demonstrated that, provided chloride contamination of the source material is avoided, this is almost exclusively  $^{36}\,\mathrm{S}$ . It should be possible to produce targets in this manner with thicknesses up to ~10  $\mu\mathrm{g/cm^2}$ .

#### Targets produced

Targets listed below have been prepared in the target laboratory during the year. Over 250 targets have been produced by various techniques, including vacuum evaporation, ion beam sputtering, cold rolling and pressing.

TABLE F.3 Targets Prepared in 1987						
Isotope		ical Form L Initial	Backing	Target Thickness (µg/cm²)	Method	
Au197	Au	Au	C,SS	2-1000	a	
C12	С	С	SS	2-50	a	
Ca40,48	Ca	CaCo <sub>3</sub>	С	100 <sup>e</sup>	a	
F19	CaF <sub>2</sub>	CaF <sub>2</sub>	С	20-50	a	
Ce142	Ce	CeF <sub>3</sub>	C	5-50	a	
Li6,7	LiF	LiF	С	50-70	a	
Li7	Li	Li	Ni	500	a	
Mg24	Mg	MgO	С	20-50	a	
Nd150	Nd	Nd	SS + In	1500	b	
Os192	Os	Os	С	20	đ	
016,18	SiO <sub>2</sub>	SiO <sub>2</sub>	С	120	a	
Pb204,206	Pb	Pb	SS	3000	d	
Pt194	Pt	Pt	Ag	1000	đ	
Pt196	Pt	Pt	Pb+In,Sn124	1000	a	
Pt198	Pt	Pt	SS	50	d	
Pt198	Pt	Pt	SS	3000	b	
Rh103	Rh	Rh	Pd108-Fe+Pd	1000	a	
Si28,30	SiO <sub>2</sub>	SiO2	С	120	a	
Si30	SiO <sub>2</sub>	SiO <sub>2</sub>	SS	200	a	
Sm144,149	Sm	SmO <sub>3</sub>	SS+Pb208	9000	С	
Sm150	Sm	SmO <sub>3</sub>	SS+Pb	3000	С	
Te124,126 128,130	Te	Te	С	100	a	
Th232	Th	Th	С	20-200	d	
W183, natural	W	W	Gđ	1000	đ	
Yb171	dY	Yb	SS	1200	b	

a. Vacuum evaporation.

b. Cold rolling between stainless steel plates.

#### Carbon Foils

Approximately 500 stripper foils, 3-5  $\mu \rm g/cm^2$  thick, were produced as well as carbon foils for target backings.

c. Reduction of the oxide using Th and La and simultaneous vacuum evaporation of the resulting metal. Metal condensed, collected and rolled between stainless steel plates.

d. Sputtered using fine beam saddle field ion source.

e. Carbonate converted to oxide which was reduced to the metal by mixing with Y powder. Metal then evaporated.

#### Activity report at ISN Grenoble

#### R. BLANC, M. BOURIANT, J.P. RICHAUD.

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During this last period, the activity of our group was mainly devoted to preparation of thin targets used in heavy ion nuclear reaction experiments. Most of the 350 targets prepared are reported in table I, with specificities, methods and results.

In addition, new techniques have been developed and improved for the various applications mentionned below.

## 1) Carbon strippers

Properties of thin carbon foils used under heavy ion beams at SARA and GANIL accelerators have been systematically studied for more than 250 samples. (50 - 100 - 150 - 200 µg/cm thicknesses).

A comparaison with commercial products is in progress.

## 2) B<sub>2</sub> O<sub>3</sub> targets

Experiments based upon (d, n) reactions on  $B_2$   $O_3$  targets have been developed at ISN in nuclear medicine.

As the <sup>11</sup>CO<sub>2</sub> produced is used to synthetize organic acids primary B<sub>2</sub> O<sub>3</sub> samples have to be free of CO<sub>2</sub>. The method used involves a fusion under vacuum followed by a fusion under helium.

By use of a new tubular, horizontal oven heating up to 1400°C, series of 15 targets have been prepared and 170 samples have been obtained.

# ARGONNE NATIONAL LABORATORY

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May 12, 1988

#### STATEMENT OF ACCOMPLISHMENTS

John P. Greene and George E. Thomas

Argonne Nuclear Target Development Facility

#### Achievements: (October 1987 - March 31, 1988)

- 1. Begin work in target development laboratory
  - a) Repair and maintain existing vacuum equipment all three systems now routinely kept under high vacuum (10<sup>-8</sup> torr).
  - b) Upgrade of existing equipment, including;
    - i Quartz crystal thickness monitors in all systems (awaiting backordered feedthru)
    - ii Ordered necessary hardware for installation of thermocouple temperature sensors in all systems (completed)
    - iii Initiated work project for new electron beam source
    - ) Acquisition of auxiliary equipment, including;
      - i Inert atmosphere glove box
      - ii Positive flow laminar clean bench (ordered)
      - iii Glow-discharge apparatus (turbo-pumped)
  - d) Construction of 2<sup>nd</sup> target storage chamber.
- 2. Development of vacuum deposition techniques
  - a) Resistive heating using the NRC 3117 evaporator.
  - b) Sputtering high melting point metals using the ultra-clean cryopumped evaporator system (Ti, Cr, Pt, W, Zr, etc.).
  - c) Routine use of the electron beam gun for vacuum deposition (<sup>11</sup>B, C and Mo).
  - d) Maintain for immediate use, the Veeco system (primarily used for visitors or "quick evaporations".)

#### 3. Production Record

- a) All targets produced for experimenters on time and as requested!
- b) FY88 275 targets made to date.
- c) Computer operated target storage system working without problems.
- d) No lost time due to equipment failures.

#### 4. Scientific Research

- a) Explored a variety of release agents for producing self-supporting isotopic tin foils.
- b) Developed techniques for making 122 Te targets on various substrates.
- c) Made self-supporting isotopic Mg foils using a formvar substrate.
- d) Routinely rolling simple foils, including; Gd, Sn, etc.
- e) New techniques for evaporating boron targets.
- f) Produced stripper foils when needed.

#### 5. Collaborations

- a) Physics Division
  - i Ordering targets, isotopes, etc.
  - ii Working with Physics Division staff on ordering high purity chemical for the ATLAS ion source
  - iii Doing chemical reductions in the high temperature furnace
- b) Other Divisions
  - i Rich Reyors CMT (vacuum annealed sample cells for IPNS)
  - ii Marian Thurnauer CHM
     Peter Gast U of C (gold coating of a delicate μ-wave cavity)
  - iii Sol Zaromb ER
     Jerry Tull (student) (developing evaporation techniques for a multi-layered solid-state methane detector)