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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 options advanced by the contributions.
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Institute of Atomic Energy, P.O. Box 275, Beijing, China

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John O. Stoner, Jr.
University of Arizona, Physics Department, Tuscon, AZ 85721, USA
and The Arizona Carbon Foil Co.,
Inc. 2239 E. Kleindale Road, Tucson, AZ 85719, USA

INTDS Membership List
Dear Colleagues,

I believe we can feel satisfied about the fact that our Newsletter has existed already 12 years. This informal means of information is supported directly by most members, so that the problem of insufficient input almost never arises. This makes the task of an editor easy!

I wish to thank them for this help and I hope to continue this way.

Mrs. De Coninck at CBNM puts the Newsletter together, polishes up the presentation by retyping parts and takes care of mailing. She always has to do an urgent job at the very last moment. She merits our appreciation for this important aspect of the Newsletter.

I also take this opportunity to thank all INTDS members and especially the board members for the years of loyal support and cooperation during my term as INTDS President.

I wish our new President: Harold Adair (Oak Ridge National Laboratory) success in leading INTDS not only along the lines of classical activities but perhaps also towards newer fields.

Let us all support him in this task.

Merry Christmas and Happy New Year

Jan VAN AUDENHOVE
Editor
Minutes of the INTDS Business Meeting
September 18, 1986
Deep River, Ontario, Canada

The meeting was called to order at 5:30 p.m. by the President, Jan Van Audenhove.

Joanne Heagney led the assembled members in a moment of silence in observance of the deaths of two INTDS members: Marcel Weishaar on October 18, 1985 and Don Ramsey on January 13, 1986.

Agenda, Actions and Resolutions
1. Minutes of the 1984 Business Meeting.
   There were no comments from the floor concerning the minutes of the meeting in Antwerp. The minutes were approved by the members.

2. Minutes of the 1985 Board Meeting
   Jan Van Audenhove commented that the Board held discussions during the meeting at ORNL and took no actions.

3. Secretary/Treasurer's Report
   Corresponding Secretary/Treasurer, Joanne Heagney presented a financial report and stated that it will be published in the December issue of the Newsletter.

4.1. The three member committee recommended nominees to fill the positions of outgoing board members Helmut Folger, Hans Maier and Dan Riel.

4.2. Proposals for changes in the By-laws
   A Committee composed of Jean Pauwels (Chair), Peter Maier-Komor and Joanne Heagney set up at the Board Meeting of September 16, 1986 discussed desirable changes in the INTDS by-laws, as e.g. terms of office for the board members and officers, election techniques which allow participation of the entire membership in the election of board members (presently restricted to members attending the World conferences) and biannual vs annual dues.
   On the basis of this discussion, Jean Pauwels was asked to elaborate a written proposal of modification of the by-laws, including application rules and transition measures for the period 1986 - 1988, to be sent to Harold Adair before October 18, 1986.
   Harold Adair will ensure that this proposal will be submitted for approval both to the board and to the membership before the 1987 Business Meeting so that it becomes valid from that time on.

4.3. Broadening the scope and membership base of the INTDS
   Harold Adair and Jan Van Audenhove, having agreed at the 1985 board meeting to approach other material groups regarding membership in the INTDS, reported that a new initiative which redefined the scope of the Society was necessary. A larger committee chaired by Harold Adair will attempt to find a suitable means of maintaining and enhancing the vitality of the Society. Each committee member (Jan Van Audenhove, Helmut Folger, Hans Maier, Geirr Sletten and Bill Lozowski) will summarize (by February of 1987) his thoughts on the scope of the INTDS (as it now exists); which groups the INTDS should try to attract; how best to define the society; changing the name; etc. The committee will report to the board before the 1987 meeting and a set of recommendations will be ready to submit to the general membership at the 1988 conference in Darmstadt. The recommendations will appear in the Newsletter before the 1988 conference.
4.4. Committee for Promotion of Recognition of Target Making
Committee members Harold Adair, Hans Maier and Dan Riel (not present) advised:
(a) There is not sufficient justification, presently, for the INTDS to undertake the task of writing a book on target making.
(b) The memoirs which retiring members are encouraged to present as talks at INTDS conferences (and published in the proceedings) promote the profession by illustrating the career of an individual for whom target making was a life's-work.

A discussion was conducted concerning the feasibility of providing INTDS funds to finance the instruction of target makers from developing countries at host facilities. The Board concluded that it is beyond the financial resources of the society to directly subsidize such hands-on instruction. The INTDS will act, however, as an intermediate whenever possible to locate an agency with the capacity to give instruction or accept a trainee.

Harold Adair proposed that this committee remain a standing committee. The proposal was sustained.

4.5. Industrial Relations Committee
The subject of industrial relations was not considered because the committee chair, Dan Riel, was not present.

4.6. Electromagnetic Isotope Enrichment Facility Users Group
Helmut Folger (a member of the Executive Committee of the EIEFUG) attended the 1986 annual meeting and reported:
(a) Steve Suchard (Hughes Aircraft) was elected chairperson.
(b) For the coming year, Joe Ratledge will be the liaison between the EIEFUG and ORNL.
(c) It will not be fruitful to petition Congress for the additional funds necessary to maintain stock equilibrium. The amount of money is "too small".
(d) A major problem is the need to "balance the books" each year. A year is not a realistic amount of time to recover the outlay involved in separating isotopes and preparing them for sale. An apparent solution would be to convince an agency such as NSF to subsidize the operation for a period longer than a year.
(e) Seventy percent of the separations are for "big users", e.g. medical diagnoses and treatment.

4.7. Newsletter
Jan Van Audenhove graciously volunteered to continue as Editor of the Newsletter and explained his intentions for forthcoming issues:
(a) The content will remain informal contributions from members.
(b) The cover will be changed to be more professional in appearance.
(c) Bibliographic references of interest to target makers will appear as a page in the Newsletter. Members should send them (not necessarily in English) to Jan.
(d) The cost of the publication will continue to be borne by CBNM.

4.8. Guidelines for hosts of INTDS world conferences
Joanne Heagney and Harold Adair will write a first draft of a policy manual for hosts. In January of 1987 it will be mailed to the other board members.
4.9. K. Okamoto's Proposal
K. Okamoto (International Atomic Energy Agency) proposed to the International Nuclear Data Committee that a meeting be held regarding "The Influence of Target and Sample Preparation on Nuclear Data Measurements" concurrent with the 1988 INTDS conference. Jan Van Audenhove, Helmut Folger, Harold Adair, and Gjerr Slatten met with Dr. Okamoto and agreed to talk again at a small meeting in Geel in 1987. The topic to be resolved is whether to conduct concurrent meetings in 1988 or to absorb the INDC meeting format as a session or sessions in the INTDS world conference. A consensus emerged from discussion among the board members: an effort should be made to keep this group with the INTDS. Jan stated that he will inform everyone of this situation as it develops.

4.10. Awards
A committee composed of Harold Adair, Peter Maier-Komer and Jean Pauwels will draft a set of guidelines regarding awards to INTDS members. Resolved:
(a) The guidelines are a desirable method of avoiding an evolution of criteria for the presentation of awards as the Board changes members over the years.
(b) Awards will be considered on a case-by-case basis by the Board as a normal business duty each time it meets.
(c) INTDS members must be encouraged to provide input to the Board regarding fellows who should be considered for awards.
(d) The Board, lacking prior knowledge of the recent retirement of Arthur Pack or John D. Stinson, considered both for awards. It was agreed to award an INTDS plaque to each.

Helmut Folger announced that the INTDS is welcome at GSI in 1988.

Barry Barthell of the Materials Science Technology Facility (Los Alamos Scientific Technology Facility (Los Alamos Scientific Laboratory) announced that the facility is willing to host the conference in Santa Fe.

7. Election of Board Members
Nominations to the Board to replace those members with expired terms (H. Adair, H. Folger, and D. Riel) were received from the Nominating Committee and from the floor. The members voted and elected Darrell Erikson, Hans Maier and Eugene Newman.

8. Jan instated the new INTDS President, Harold Adair. Harold's first "official act" was to ask Jan to be the master of ceremonies at the INTDS banquet.

The meeting adjourned at 6:35 p.m.

Respectfully submitted,

Bill Lozowski
Recording Secretary
IN MEMORIAM - DONALD RAMSAY

At a meeting last October in Oak Ridge, TN, the INTDS Board decided to present special awards to several members for exceptional service to the Society. One of those selected was Don Ramsay. We only regret that the Society must present this award to Don posthumously. Don Died at the age of 59 on January 18, 1986 after a two-year battle with lung cancer. He was the target-maker at Varian Labs., Stanford University from 1968 until severe funding cuts forced his early retirement in April, 1983.

Many of us remember Don as one of the founders of INTDS, for his service on the Board of Directors and for his creative, inventive work in the field. Many of us who knew Don more closely also remember him for his sharp wit and constant good humor.

This award will be presented to Don's family and will be sent to his eldest Daughter, Deborah, who lives in San Francisco, CA.
PRESENTED TO THE FAMILY OF

Don Ramsay

AT THE THIRTEENTH WORLD CONFERENCE OF INTDS
SEPTEMBER 17-19, 1986

CHALK RIVER, ONTARIO, CANADA

IN RECOGNITION OF HIS CONTRIBUTION TO THE FORMATION AND SUCCESS OF INTDS AND HIS CONTRIBUTIONS TO THE SCIENTIFIC COMMUNITY DURING HIS FIFTEEN YEARS AT STANFORD UNIVERSITY.

International Nuclear Target

[Signature]
INTDS Award Presentation to Joe Gallant

Joe Gallant was a teacher in the Canadian Public School System for three years before joining the Chemical Operations Laboratory of AECL in 1951. In 1967, until his retirement in August of 1985, he was in charge of the Target Preparation Laboratory at Chalk River. In his years at Chalk River, he developed his target laboratories into a world class, customer-friendly facility.

Those of us who remember Joe from the first INTDS conference here at Chalk River recall that he was open, friendly, candid and very knowledgeable. He never changed. Some of us will recall that he referred to himself as a "wet chemist" in a presentation he gave in the 1978 conference in Garching. To his colleagues he was, throughout his career and continues to be—a teacher. His published contributions date from 1970. The helpful, time-saving information he offered in them remain a legacy of his talents as an Educator.

Most of us learned in the August 1985 INTDS Newsletter that he had received a Special Discovery Award from AECL for "innovative development of tritium sources for document imaging. The tritium sources provide an economical and highly portable method for examining documents and currency for forgeries and alternations."

To state the obvious: Joe's retirement has been only partially successful in that he has continued to make outstanding, contributions to INTDS, Chalk River and the general public.
INTDS Award Presentation to Mrs. Katherine M. Glover

After War-time service at the Building Research Station, Watford, Mrs. K. Glover graduated with B.Sc from Nottingham University in 1949.

She joined the Atomic Energy Research Establishment, Harwell in November 1949 and retired after 34 years service in January 1984.

Responsible for the development of precise radiometric analysis — specialising in alpha counting, alpha spectrometry, and standard sources.

She has also published papers on integral neutron cross-section and nuclear decay data measurements on transactinium nuclides. She has created an international reputation for the production of very high quality targets of transactinium nuclides for use in many nuclear physics and chemistry experiments. Under her direction many environmental tracer isotopes and biological tracers have been prepared using the Harwell variable energy cyclotron.

As Secretary of the α-spectrometry sub-group of the International Committee for Radionuclide Metrology she organised the 1983 conference.

In 1972 the work was extended to involve stable isotope target production, γ-irradiation and γ-dosimetry.

In her later years at Harwell she was involved with safeguards requirements and the provision of nuclear reference materials for this particular requirement.

As well as her academic and scientific achievements she was also a leading light in the social activities of the Division and had a happy family life as well.

In 1977 she received the Queen's Silver Jubilee Medal.
INTDS Award Presentation to Mrs. Judith Gursky

Mrs. Judith Gursky was a B.A. in physics from Mount Holyoke College, 1947 when she started to work in the Physics Division at Oak Ridge National Laboratory from 1947 to 1951, on nuclear spectroscopy and development of scintillation spectrometry.

She graduated with M.S. in physics at Vanderbilt University, moved to Los Alamos with her husband, and joined Los Alamos Laboratory in 1955. She worked in Physics Division first part-time while raising five children, later full-time. She was involved in gas scintillators and other projects, but largely in vacuum evaporation of thin films. She produced targets for the Los Alamos cyclotron (the old Harvard cyclotron which was moved to Los Alamos during World War II), essentially all the isotopic and other targets for the Los Alamos vertical Tandem and three-stage Van de Graaff operations for a number of years.

With John Povelites she organized the 1976 Conference of INTDS at Los Alamos.

She made several important contributions to the growth and development of INTDS. Beside many publications she also made available a highly appreciated Index of Publications of INTDS covering the period 1971 - 1983.

Judith retired officially in 1985 but continues to operate Physics Division Target Laboratory as needed.
INTDS Award Presentation to Arthur A. Peck

Immigrated to Canada from the United Kingdom after war service with the Royal Air Force.

Attended a course in Industrial Chemistry at Ryerson Institute of Technology (as it was then known).

Received a diploma in Metallurgy from British Institute of Technology. In 1966 awarded a High School Teaching Certificate from the Ontario College of Education.

After 20 years in Industry including metallurgy, electroplating, anodizing and three years in the development of the Avro Arrow joined the Nuclear Physics group at Queens University making some of their first Germanium detectors under the direction of Dr. H. Evans. Finally becoming their target maker.

Joined the I.N.T.D. during their first years, attending most of the conferences until just before retirement in December 1984. Still associated with Queens University, making the occasional target and some consulting work.
Attended Cobourg Collegiate Institute for his high school education and subsequently graduated from Shaw's Business School in business administration. He also holds a diploma in electronics from the Eastern Ontario Institute of Technology.

His working life includes 2 years with the Bank of Toronto, 3 years with Eldorado Mining and Refining, 1 year with Gunnar Uranium Mines and 32 years with the National Research Council. His time spent with Eldorado Mining and Refining was spent in the preparation of radium medical units and in a physical chemistry measurements laboratory. His time spent at Gunnar Uranium Mines was as an assayer of uranium ores.

At the National Research Council his original mandate was to measure radium medical units for government certification. His last twenty years there was spent as a target maker.
INTDS Award Presentation and
RECOGNITION GIVEN TO JAN VAN AUDENHOVE FOR HIS LEADERSHIP
AS PRESIDENT OF INTDS

On behalf of all INTDS members, I want to express my sincere appreciation for all the many contributions Jan has made to INTDS. His leadership for the past three years as President of INTDS has been outstanding.

For many years as a member of the staff at the Central Bureau for Nuclear Measurements (CBNM) and since 1974 as the head of the sample fabrication group at CBNM, Jan has made many technical presentations at INTDS conferences and other technical meetings. The presentations have described various research and development aspects relating to target and special nuclear materials preparation. The presentations along with his numerous technical publications have made significant contributions to our understanding in these areas.

The sample fabrication group at CBNM is recognized throughout the world for their technical achievements which are directly attributable to the leadership of Jan Van Audenhove. The efforts of Jan and his colleagues at CBNM in supporting INTDS through their participation at the biannual conferences, in hosting the conferences, and by serving in various INTDS offices have helped the Society to become more visible.

During his tenure as President, several changes have been made, and several recommendations are being considered. The changes will help INTDS to become a stronger Society that will better be able to meet its objectives.

My job as the new President of INTDS has been made much easier as the result of Jan's efforts during his tenure as President.

Harold Adair
President, INTDS
WORKSHOP 1986

Nuclear Target Sub-society of China

Xu Guoji

The workshop of Nuclear Target Sub-society of China, NTSC, was held at IAE, Beijing, on Oct. 6-8, 1986. 33 participants attended the workshop, they came from Shanghai Institute of Nuclear Research, Shanghai, Institute of Modern Physics, Lanzhou, South-western Institute of Nuclear Physics and Nuclear Chemistry, Chengdu, Fudan University, Shanghai, Beijing University, Beijing, Lanzhou University, Lanzhou, Nankai University, Tianjin, Institute of High Energy Physics, Beijing, Sichuan University, Chengdu, Institute of Technology and Physics of Tianjin, Tianjin, and IAE, Beijing. Dr. H. Folger, GSI, Darmstadt, F. R. Germany, was invited to the workshop and give three invited lecturers.

The information exchange covered a wide range of topics including:

1. recent progress of target making,
2. reduction techniques for isotopic materials,
3. target preparation with vacuum evaporation,
4. survey on the existing high energy heavy-ion accelerators,
5. preparations and investigations of targets at GSI,
6. targets prepared by focused heavy-ion sputter-deposition,
7. carbon stripper foils,
8. electron beam evaporation,
9. rolling techniques and
10. spectrophotometer.

The NTSC board meeting was held during the workshop and president of NTSC was elected by the board of directors.

The third symposium of NTSC will be held in Lanzhou in 1988 or 1989.
Preparation of Nitrogen - 15 Targets as Vanadium Nitride*
William R. Lozowski
Indiana University Cyclotron Facility

Recently, \(^{15}\text{N}\) powder has been synthesized in a tube furnace, distributed in a 1.27 cm diameter disk, and pressed into a 35 mg/cm\(^2\) target disk. Less than 0.5 wt.% of binder (the pressure sensitive adhesive on common translucent adhesive tape) was added before pressing. No amount of the binder was detectable (by weighing) after firing at 600°C in a vacuum furnace.

The (p\(\beta\)) experiment for which the target was prepared revealed the \(^{15}\text{N}\) content to be 82% of the theoretical value and the \(^{14}\text{N}\) content to be 0.01 wt.% This less than optimum efficiency is most likely due to the 1200°C limitation of the tube furnace used. Clark [1] has reported a range of reaction temperature of 900°C-1300°C, for the formation of VN. H. V. Fruebel, H.J. Maice et al [2]; however, have recommended a minimum temperature of 1250°C for reasonable temporal efficiency. The three hours at 1200°C used for the present synthesis will be extended to six hours to increase our efficiency.

Furnace Procedure

1. A mullite combustion tube (25mm I.D., McDanel Refractory Porcelain Co.) and an \(\text{Al}_2\text{O}_3\) combustion boat (Thomas Scientific) were outgassed at 400°C under vacuum.

2. 150 mg of 325 mesh, 99.7% vanadium powder was placed in the combustion boat.

3. The tube was evacuated while being heated to 600°C.

4. A flow meter was used to admit three times the stoichiometrically required amount of \(^{15}\text{N}_2\) (100cc). A bourdon tube pressure gauge indicated 50.5 K Pa (1/2 atmos).

5. The combustion tube was maintained at 1200°C for three hours, after which an indicated pressure of 21.5 KPa (165 Torr) was recorded.

6. The furnace was allowed to cool overnight. Beyond these details the reader is referred to ref. 2 for guidance.

Binder

Petroleum ether (20-40°C) was used to dissolve the adhesive from Scotch Magic Transparent Tape®. The concentration of the solution was adjusted to yield 1.38 mg of adhesive after evaporation of ten drops. Two drops of this solution (~275 µg) were applied directly to 45 mg of \(^{15}\text{N}\) powder. After drying at room temperature, the clumped powder was finely divided by chopping it with a razor blade. Comment: this adhesive has been found to be an excellent binder (in negligible amounts) for a growing number of pressed powder targets at IUCF.

*Work supported in part by the National Science Foundation, Grant NSF PHY 8412177.
Pressing

The 1.27 cm I.D. die and two compacting rods (a three piece assembly) were made of tungsten carbide in a 6\% nickel matrix. It was found necessary to coat the contact surfaces of the compacting rods with 5 \( \mu \text{g/cm}^2 \) carbon foils to prevent sticking during the pressing operation. This was accomplished by floating vacuum-evaporated carbon foils in water and simply lifting them (from the underside) with the rods. When dry, the carbon adhered, did not come off on the pressed targets, and could be used for several pressings.

The die cavity was adjusted to be slightly higher than the bottom compacting rod, forming a shallow die cavity. Using a razor blade and a vertical chopping motion, the powder was carefully distributed within. By raising the die, the distributed powder was lowered deeper into the die cavity before the top compacting rod was added. A four column press was used to apply 35 MPa (1000 lbs force on 1.27 cm\(^2\)) for 30 seconds. The target disk had sufficient strength to withstand moderate handling during transfer and weighing.

Firing

Thermal decomposition of the binder was done in the target lab vacuum evaporator at 0.1 mPa (8\times10^{-7} \text{Torr}). The vacuum oven consisted of two Al\(_2\)O\(_3\) coated evaporation sources. (R.D. Mathis S36-A0-W) placed one over the other with the Al\(_2\)O\(_3\) coated sides facing inward. After resistance heating the disk within the oven for thirty minutes at 600°C, the temperature was decreased to ambient over a period of 20 minutes.

Useful spectra were obtained from both the \(^{15}\text{N}\) target and a VN calibration target, made using the same binding and pressing techniques. Further attempts to increase the content of \(^{15}\text{N}\) from 82\% (of theoretical) to beyond 90\% will be made by either increasing the reaction time at the 1200°C furnace limit, or if necessary, using a furnace with a higher limit.

References


DEVELOPMENT OF LARGE-AREA THIN FILMS FOR
GENERATING NEUTRAL PARTICLE BEAMS*

W. S. Aaron, L. A. Zevenbergen, H. L. Adair, Oak Ridge National Laboratory
W. H. McCulla, Los Alamos National Laboratory
T. A. Nolan and M. R. Hughes, Oak Ridge Gaseous Diffusion Plant

For approximately six months, the Isotope Research Materials Laboratory (IRML) at the Oak Ridge National Laboratory has been involved in the development and characterization of large-area thin aluminum foils to be used in generating neutral particle beams as part of a program conducted by the Los Alamos National Laboratory. Originally, foils of aluminum and carbon were prepared and supported on 7.6-cm-diam nickel grids mounted on aluminum frames. Foil thicknesses ranging from 5 to 20 μg/cm² were prepared to assess the survivability of this type of neutralizer and determine the optimum thickness to maximize neutralization efficiency. The foils were tested at the Argonne National Laboratory's Neutral Particle Beam Test Facility by experimenters from Los Alamos and IRML. Foils were exposed to a 50-MeV H⁻ beam with a peak current density of 15.3 mA/cm² which was pulsed for 50 μs at a frequency of 1 to 3 Hz. The first two foils that were tested had neutral fraction production rates of 56% and 38%, respectively, for thicknesses of 5 to 6 μg/cm² and 12 μg/cm². With subsequent refinements in film thickness measurement capabilities, it is felt that these foils were actually 10 μg/cm² and 18 μg/cm² thick. Examination of the foils after beam exposure using transmission electron microscopy could detect little or no changes in the foils from the "as-prepared" condition. With the success of these tests, the program for the development of this type of neutralizer foil was expanded. Currently, foils 25 cm x 25 cm with thicknesses ranging from 5 μg/cm² up to 12 μg/cm² are being

*Research sponsored by the Division of Basic Energy Sciences and Division of Nuclear Sciences, U.S. Department of Energy, under contract DE-AC05-84OR21400 with the Martin Marietta Energy Systems, Inc.
routinely prepared and used for a variety of tests. These tests, performed by other groups, include further in-beam experiments, vibrational testing for space launch survivability, heating (heat-dissipation) characteristics, and compatibility with space environments. Further developments needed include refinements to preparative techniques, improved characterization of the foils, the preparation of much larger foils, and the investigation of integral evaporated aluminum support grids instead of the separate nickel grids.

Foil Preparation Details

Large-area aluminum neutralizer foils are being prepared by flash evaporation of known quantities of aluminum from a multistrand tungsten ring filament. Glass plates of the appropriate size are coated with a Betaine-sucrose parting agent to serve as the foil substrate. The surface is deliberately left rough to impart texture to the foil, which apparently improves its strength since a rough texture produced much better and stronger foils than those deposited on Betaine-sucrose layers that had been polished. Applying satisfactory coatings to 25 cm x 25 cm or larger areas is, at best, an art, and more reproducible methods to apply the parting agent are being sought. The substrate is then placed in the same 81-cm cubical box coater that was described in the INTDS Newsletter dated June 1986 when it was being used for large-batch (100-g) calcium reduction-distillation processing. Evaporations are performed in 5 to 10 s and are made in the 10^{-6}-torr pressure range. The substrate is then removed from the evaporator and placed on an incline in a large water tank. The foil support, which consists of a very fine, commercially available nickel grid (90% transmittance and 3 μm thick) attached to a heavy aluminum frame, is placed vertically in one end of the tank. Distilled water is slowly added to the tank, and as the water level rises, the foil is floated off the substrate. The 25-cm x 25-cm foil is then guided to the end of the tank where the grid and frame assembly is just barely submerged. Water is slowly drained from the tank, allowing the foil to drape onto the support grid. The foil is allowed to dry in place, after which it demonstrates remarkable strength and durability.
Thickness and uniformity measurements agree well with those calculated from ring-source models, so it is felt that the foils can be scaled up to much larger sizes as preparative techniques are developed. A limited number of foils have been prepared with an evaporated aluminum grid structure (approximately 1-mm-wide bars on 1-cm centers) and show a great deal of promise.
X-ray Photoabsorption Filter Foils Fabricated by Magnetron Sputtering

Leslie M. Jones II, John W. Weed

Lawrence Livermore National Laboratory
Livermore, Ca. 94550

INTRODUCTION

Foils for x-ray photoabsorption filtering were produced in a magnetron sputtering deposition system. Elemental coatings were deposited on frame mounted polypropylene films that were 5000 Angstroms thick. The coated areas measured approximately 15 to 55 square centimeters; some were circular, others were rectangular. Deposition thickness ranged from 1500 to 6000 Angstroms. Elements included: copper, iron, nickel, germanium, manganese, and cobalt. Aluminum foils were also fabricated for the same purpose, but by a different technique which is to be reported on separately. The iron was deposited using a source designed for magnetic materials.

EQUIPMENT

Deposition took place in a stainless steel chamber which measured 60 X 60 X 92cm (see Figure 1). Pumping was done with a cryogenically trapped, 1500 l/sec turbomolecular pump backed by a 17 cfm mechanical pump. A base pressure of 5x10^{-7} torr was typically achieved. The process gas was maintained at 5 millitorr by a closed loop, capacitance manometer system.

Two vapor sources were used. Most of the elements were sputtered from a 10 kilowatt, circular, magnetron sputter gun. The iron was sputtered from a 15 kilowatt, 10 cm X 20 cm, rectangular source designed for magnetic materials. Both sources were mounted diametrically opposed on a rotating fixture which allowed movement of the sources during deposition. Sputtering power levels were on the order of 300 to 2000 watts depending upon the given element.

PROCEDURE

Several substrates were mounted on a rotating fixture which simultaneously rotated them on a horizontal circle, perpendicular to the source, and spun them on a diametric axis. This motion is believed to provide two benefits. First, both sides are coated during a single pumpdown. Second, the angle of incidence of the arriving vapor is continuously varied. This tends to produce deposited films that have lower residual stress.

After deposition, the total thickness and uniformity of the films was established using an x-ray fluorescence analyzer programmed to inspect ten different places on each film. The initial thickness determination and the establishment of standards for each element was done by cutting out a number of the films, then weighing and calculating their areal density. The cut-out foils were then remounted on frames and used as reference standards for subsequent x-ray fluorescence measurement.

*Work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48.
FIGURE 1

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Fabrication of Multilayered X-ray Photoabsorption Targets

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Livermore, CA 94550

INTRODUCTION

Free standing targets were produced to measure the sub-keV photoabsorption cross section of numerous elements. The targets were fabricated with a computer controlled, magnetron sputter deposition system. The targets are structured of multiple layers of a given element interspersed with layers of carbon. The purpose of the multilayering is two-fold: to improve the mechanical integrity and hardness of the targets; and to protect the given element from exposure to the atmosphere, using the outermost C layers as a seal.

The targets are 1.9 cm in diameter and their thicknesses range from 4000 Angstroms to 27,000 Angstroms depending upon the design requirements for the given element. The total thickness of the C layers in each target is 2000 Angstroms. The first and last C layers, the sealing layers, are 500 Angstroms thick and the remaining 1000 Angstroms are divided among the interspersed layers. Multilayer targets were successfully produced in which the given element was either Al, Ti, V, C, Cr, Ni, Cu, or Au. Difficulties were encountered in attempting to do multilayers of Fe and of B. Because of scheduling considerations, these were deferred for future effort. Multilayer targets of Ge and of Si are also planned.

APPARATUS

The deposition was done in a stainless steel chamber which measures 76 x 76 x 107cm. The chamber is pumped by a both a turbomolecular pump and a cryogenic pump which together typically produce a system base pressure of 2x10^-7 Torr. A closed loop, process gas control system was used to maintain a constant argon pressure of 5.0 millitorr. The control system consisted of a capacitance manometer, a piezoelectric valve, and the associated electronic controls.

Two, 10 kilowatt planar magnetron sputter guns were used as the vapor sources. These are mounted on opposing arms of a rotary feedthrough assembly that also provides for distribution of source power and water cooling (see Figure 1). Power levels were maintained at between 80 and 2000 watts depending on the given element.

Glass slides, measuring 5.1 x 7.6cm, were used as substrates. These were first coated with a release agent and 500 Angstroms of C. Each substrate was sandwiched between a holder and a mask that contained four holes. Three of the holes were used to produce the

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targets. The remaining hole was masked by a shutter during the deposition of the given element and exposed during C deposition. This allowed subsequent determination of the total amount of C contained in the multilayer structure. The substrate assembly was contained in a water cooled Cu box having a second shutter (see Figure 1). The source to substrate distance was 13cm.

A microcomputer linked to a data acquisition/control unit was used to control the deposition process. Control was provided for source and shutter positioning, source power, and deposition time.

PROCEDURE

In preparation of a deposition run, the computer was fed: 1) the predetermined deposition rate of both the given element and the C, and 2) the desired number of layers and the thickness of the layers for each of the two materials. The substrate box shutter was then closed and both sources were warmed up.

After the warm up was complete, the source containing the given element was positioned directly in front of the substrate box. The shutter inside the Cu box (for C only) was closed. Then the substrate shutter was opened, starting deposition of a layer of the given element. At the appropriate time, as determined by the computer, to deposit the correct thickness, the substrate shutter was closed and the C source was positioned in front of the substrate box. Both the internal and the substrate shutter then opened, commencing the deposition of a C layer. At the appropriate time, again as determined by the computer, the substrate shutter was closed. The cycles were repeated until all the layers were deposited.

After completion of the deposition process, the substrate, mask, and holding fixture were removed from the deposition system. A scalpel was used to scribe a line along the inner circumference of the mask holes, separating the targets from the rest of the C film deposited on the substrates. The targets were floated off the glass slide in a distilled water bath. Areal density and Rutherford backscatter measurements were performed to establish the total amount of the deposited element.
Several $^{40}$Ca targets were produced. We started with calcium carbonate, which was heated in vacuum in an aluminium oxide crucible until there was no outgassing any more. The resulting calcium oxide was then mixed with four times its weight of tantalum pressed to a pellet and heated in a tantalum tube of 5 mm height and 3 mm inner diameter. As even a vacuum of $2 \times 10^{-7}$ mbar obtained with an oil diffusion pump filled with Santovac oil resulted in too much oxygen in the targets, some titanium was evaporated in an edge of the bell before the calcium was evaporated. 24 mg of calcium oxide resulted in a 2 mg/cm$^2$ target on a 50 mg/cm$^2$ bismuth backing rolled to a copper foil and fixed on a large piece of copper (for cooling) at a distance of 2 cm from the crucible during the evaporation.

In the same way 0.3 mg/cm$^2$ calcium targets on a 80 µg/cm$^2$ gold backing were produced at 6 cm from the crucible. All targets were coated in the same vacuum on the front with a thinn layer of bismuth or gold to slow down the oxidation when handling them.

$^{138}$Ce: Cerium oxide was reduced with the method of Bennink and Tuinjtjer 1). In the transformation from cerium dioxide to sesquioxide we got only half the amount, which one should get according to their description, but we worked with a smaller quantity (only 30 mg than 100 mg). As it turned out, that our thorium, used for the reduction from sesquioxide to metal, had a very small but for the experiment very disturbing content of aluminium, we used also tantalum. One needs approximately 6 times as much power to evaporate the cerium from a mixture of the oxide with tantalum than with thorium. 1 mg/cm$^2$ targets were produced on a cooled bismuth copper sandwich in 2 cm distance from the tungsten tube in which the reduction was performed.

$^{138}$BaO: Barium carbonate mixed with the same weight of tantalum was pressed and put in a tantalum tube of 5 mm height and 3 mm inner diameter. In 4 cm distance a 20 µg/cm$^2$ carbon foil was rotated in such a way that the barium could condense on both sides. This helps to avoid stresses which brake the foil. It was possible to make 140 µg/cm$^2$ targets with 1 cm diameter in this way but not with 2 cm diameter. Of course those targets oxidize immediately when they come into contact with air.

Measurement of Foil Thickness With Spectrophotometer

Xu Guoji Meng Xiangjin and Luo Xinghua

Tandem accelerator HI-13 at IAE needs carbon stripper foils from 0.005 to 0.01 mg/cm². It is difficult and time consuming to determine the areal density of stripper foils by weighing. On the other hand, α-absorption is ruled out due to its very low absorption. RBS is a good method to measure the thickness of such thin foils, but the beam time is expensive and the method is also time consuming.

We found that calibration curve of optical absorption of carbon foil is particularly useful in the preparation of carbon foils by ethylene cracking. The measurements were carried out using a single beam visible light spectrophotometer. The reference thickness was determined by weighing. The calibration curve obtained at λ=680 nm is given in Figure 1.

The uniformity in thickness of carbon stripper foils was measured by moving the foils in 2 mm steps in X and Y directions. The light spot size of the spectrophotometer is 1.2 mm in width and 1.6 mm in height. Table 1 shows the uniformity of a batch of carbon foils. The ununiformity of each foil is less than 5%, and the unhomogeneity among 26 foils is less than 10%.

In order to control thickness of carbon foils, the relation between quantity of electricity of glow discharge and thickness of carbon foils was measured with the spectrophotometer and is given in Fig. 2. When the quantity of electricity is 40 mA minute, carbon foils of 0.006 mg/cm² could be obtained.

The relation between wavelength and absorption for gold foil was first determined (Fig. 3) to find a fast thickness measurement way. It is clear that there is an interference peak at 500 nm. The calibration curve got for λ=500 nm is given in Fig 4.
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<tr>
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Table 1 Uniformity of a batch of carbon stripper foils
Fig. 1 Relation between thickness and absorption for ethylene cracking carbon foils

Fig. 2 Relation between quantity of electricity and thickness for ethylene cracking carbon foils
Fig. 3 Transmission spectrum of a 116 mg/cm² gold foil
Fig. 4 Relation between thickness and absorption at $\lambda=500$ nm for gold foils
De-Stressing and Trimming Thick Carbon Foils

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Carbon foils having surface densities greater than about 100 micrograms per square centimeter pose special problems when the experimenter wishes to cut them to size for mounting on target holders. An evaporated carbon foil is brittle, and tends to shatter along the cut when a sharp-edged tool is used to mark the foil prior to floating it off of its substrate. The problem is worse when the foil does not adhere tightly to its substrate. The resulting damaged edge is more fragile than the remainder of the foil. It is not unusual for cracks started in the cutting process to propagate into the foil later. Cutting the foil can be avoided by masking the substrate during the evaporation process so that pieces are formed in the size desired, but this is not always convenient.

Foils often show a tendency to curl when floated off of their substrates. Sometimes the stresses that cause this phenomenon are so great that the foils break up and sink as they are released. Even when the stresses are milder, foils may spontaneously break away from their target holders after they have been mounted and allowed to dry. The stresses in foils of several hundred micrograms per square centimeter are often great enough to cause them to break loose from their substrates while they are in storage before they are floated off, and curl into tight little tubes that have no known use.

The cutting and curling problems can be cured simply. It is necessary to use carbon foils that are evaporated onto a glass substrate previously coated with soap and then polished. If such a foil is placed horizontally and baked in an oven in air at 260 degrees Celsius for an hour, its stresses are almost completely relieved. The foil separates almost completely from its substrate in this process, so that it is easy to float it off. The tendency to curl nearly disappears and it floats limply on the water surface. Such a foil can be picked up on a U-shaped frame so that the foil has an unsupported edge of length 50 millimeters or more.

It is easy to trim such a foil with sharp scissors and straight-line cuts while it is floating. It is only necessary to make sure that the scissor blades are longer than the length of foil to be cut, so that the points do not close on the foil, and to keep the lower blade close to but below the water surface.