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OAK RIDGE NATIONAL LABORATORY

TARGET PREPARATION PROGRAM

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OAK RIDGE NATIONAL LABORATORY

operated by

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OAK RIDGE NATIONAL LABORATORY
TARGET PREPARATION PROGRAM

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OAK RIDGE NATIONAL LABORATORY TARGET PREPARATION PROGRAM

ABSTRACT

In early 1961 a Target Fabrication Center was established at Oak Ridge National Laboratory to carry out the routine preparation of a wide variety of targets and to provide research and development support to the target program. Although most of the effort concerns enriched stable isotopes, both normal and radioactive materials are also handled. With the aid of rolling mills, resistance-heat and electron-bombardment vacuum evaporators, presses, and electroplating equipment, targets of elements and compounds are prepared which cover a broad range of thicknesses and sizes, in both supported and self-supporting forms. Thicknesses vary from a few micro-inches to dimensions limited in many cases only by the amount of material available. Although there are certain limitations imposed by chemical reactivity, the development effort is being directed toward the ultimate availability of self-supporting targets of most elements and compounds.

1. INTRODUCTION

There is an increasing need in present-day research and development for separated isotopes, radioactive isotopes, and ultrapure elements and compounds in a diversity of physical forms. "Targets" which, for a variety of reasons, are to be irradiated by electromagnetic radiation, neutrons, or charged particles represent an important group of these special research tools. In this report the preparations of nonradioactive and low-activity targets, the latter containing less than 10 curies of radioactive material, are discussed.

For more than 10 years ORNL has been engaged in the fabrication of isotopic targets in geometric forms ranging from films only a few hundred angstroms thick to cylindrical or rectangular parallelepipeds weighing several kilograms. By early 1961, numerous demands for a wide variety of targets made it necessary to establish a Target Preparation Center at ORNL.¹ The early operations of the Center were confined to routine preparations to meet the large volume of requests; as a result, there was little opportunity for development. Therefore a small development staff was added to support the production effort. At the same time improved, centralized facilities were obtained for the specialized personnel and equipment required to accomplish the variety of tasks assigned the group. Lack of facilities has, so far, prevented centralization of the radioactive target work.

¹Letter, S. R. Sapirie to C. E. Center, "Irradiation Target Preparation Center at ORNL," May 1, 1961.

The activities carried out by the ORNL Target Preparation Center include:

1. Preparation of isotopes of sufficient chemical and isotopic purity to meet particular requirements
2. Preparation of forms suitable for target fabrication
3. Fabrication of targets to meet exacting physical specifications, and packaging to assure shipment without damage
4. Measurement of the chemical and physical characteristics of the targets to meet specifications
5. Recovery of enriched material not actually shipped as target
6. Development of procedures to minimize target cost
7. Publishing of new developments in the fabrication of targets

Centralization of the target preparation function permits analysis of the services and manpower needed by this activity. During FY 1962 orders were received from more than 40 universities and research centers in the United States, excluding ORNL, and from 15 other organizations throughout the world. All requests were filled except those for which the specific isotopes desired were not available or for which there was insufficient technology and/or equipment to permit target fabrication to meet specifications. More than 400 targets were prepared during this period, nearly one fourth of them during May and June.

Some radioactive isotopes were also converted into target form, e.g., ^{14}C ; ^{99}Tc ; ^{238}U ; H^3 sorbed on Y, Ti, or Zr; most of these were tritium targets. Only a few orders for radioactive targets could be accepted because of the undesirability of contaminating existing equipment and the unavailability of suitable hot-cell or glove-box facilities.

Techniques that have been used in target preparations include vacuum evaporation (thin films), rolling (metal foils), electrodeposition, pressing, and casting (metals and compounds).

2. PRESENT STATUS OF TARGET FABRICATION

2.1 Equipment and Personnel

The building allotted to fabrication of targets is a one-floor structure of 700 sq ft with equipment as shown in Figs. 2.1 through 2.3. Future requirements for additional equipment and for handling research-and-development or quality-control functions are discussed in Sect. 3.

The following specialized equipment is now available for use in fabrication of stable targets:

Fig. 2.1. Electrodeposition Apparatus.

Fig. 2.3. Rolling Mill, Glove Box, and Evaporators.

Fig. 2.2. Induction Heater and Vacuum Evaporation Equipment.

1. One 5-in. Fenn rolling mill and one 3-in. hand rolling mill
2. Three commercial resistance-heat vacuum evaporators
3. One electron-bombardment vacuum evaporation furnace
4. One kc-Mc radiofrequency heater
5. One 30-ton press
6. One aqueous-solution electroplating apparatus
7. One 20X to 2000X metallographic microscope
8. One refrigerated vacuum glove box

In addition, limited electroplating equipment, two sintering furnaces, and one small vacuum evaporator are available on a part-time basis for radioactive target fabrication. The large-volume commercial vacuum evaporators are needed, in spite of slow operation, to allow bulky supports and unusual filament devices to be used within the evacuated bell jar. One small bell jar system is used for evaporations of mildly radioactive materials such as Tc⁹⁹; however, its low potential vacuum level, 10^{-4} Torr, restricts its application.

The nucleus of the Target Preparation Group comprises four professional people (three chemists and one physicist). In addition, four technicians perform most of the routine evaporation and rolling operations. Currently both production and development work are carried out by this group. Radiotargets are prepared by technical people as a part-time activity; about one man-year is involved in this function, and development effort in this area is directed toward fulfilling specific requests. Future plans for development work on radiotarget preparations are outlined in Sect. 3.

Experience has indicated that about one man-year of analytical chemistry support and one man-year on the part of technical personnel to prepare the various isotopes in requisite chemical forms are required for target preparation. Thus a total of about seven professional people and four technicians is now assigned to the program.

2.2 Target Preparations

More than 52 elements, excluding the transuranium elements, have been prepared in target form. Most of these elements are used in elemental form, but more reactive metals, e.g., Li and U, are prepared as chemically stable compounds. Table 1 summarizes the elements whose isotopes have been prepared in target form and the methods by which these targets are fabricated. Targets and foils are fabricated to customer order when specifications are within the range of those noted for the elements listed in the table. Processes used to fabricate these targets

and the various substrates or backing materials used are noted in the table according to the following number or letter codes:

a. Evaporation	1. Self-supporting
b. Rolling	2. Metal backing
(specified by customer)
c. Electrolytic deposition	3. Thin carbon: 20-100 $\mu\text{g}/\text{cm}^2$
d. Pressing or casting	
e. Vapor plating	
f. Miscellaneous	

Table 1. Target Fabrication Data

Element	Range of Thickness	Method of Preparation	Backing
H	Zr, Y, or Ti: 100 to 5000 $\mu\text{g}/\text{cm}^2$	f Zr, Y, Ti on 10-mil Pt followed by H^3 sorption	2
Deuterated polyethylene	>0.2 mil	f	1
Li	1 mg/cm^2 to any cast size	b, d	1
LiH	200 to 500 $\mu\text{g}/\text{cm}^2$	a	3
Be	25 to 500 $\mu\text{g}/\text{cm}^2$	a	3
B	20 to 500 $\mu\text{g}/\text{cm}^2$ 500 to 1000 $\mu\text{g}/\text{cm}^2$	a a	1, 2 2
C	10 to 100 $\mu\text{g}/\text{cm}^2$	a normal f isotopic	1 normal 2 isotopic
F (as fluoride)	25 to 500 $\mu\text{g}/\text{cm}^2$	a	2
Na	7.5 mg/cm^2 to any cast size	b, d	1
Mg	5 to 8000 $\mu\text{g}/\text{cm}^2$	a	3
Al	5 to 12,000 $\mu\text{g}/\text{cm}^2$ 250 $\mu\text{g}/\text{cm}^2$ up	a b	2 1
Si	30 to 100 $\mu\text{g}/\text{cm}^2$ 10 to 100 $\mu\text{g}/\text{cm}^2$	a a	1 2

Table 1. - continued

Element	Range of Thickness	Method of Preparation	Backing
S	5 mg/cm ² up	d	1
Cl (as compound)	5 mg/cm ² up	a d	2 1
K	7.5 mg/cm ² to any cast size	b, d	1
Ca	1 mg/cm ² up	b	1
Sc	1 mg/cm ² up	b	1
Ti	1 mg/cm ² up	b	1
V	1 mg/cm ² up	b	1
Cr	10 to 5000 µg/cm ²	c	2
Fe, Co, and Ni	1 mg/cm ² up 800 mg/cm ² up 10 to 1000 µg/cm ²	b d c	1 1 2
Cu	1 mg/cm ² up 10 to 1000 µg/cm ²	b c	1 2
Zn	1 mg/cm ² up 10 to 1000 µg/cm ²	b c	1 2
Ge	10 to 200 µg/cm ²	a	2
As	10 to 200 µg/cm ²	a	2
Se	300 to 5000 µg/cm ² 10 to 5000 µg/cm ²	a a	1 2
Br (as bromide)	5 mg/cm ² up	a d	2 1
Rb (salt)	10 to 1000 µg/cm ² 40 mils up	a d	2 1
Y	10 to 5000 µg/cm ²	a	2
Zr	1 mg/cm ² up 10 to 5000 µg/cm ²	b a	1 2
Nb	2 mg/cm ² up	b	1

Table 1. - continued

Element	Range of Thickness	Method of Preparation	Backing
Mo	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
Tc	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
Pd	1 mg/cm^2 up	b	1
Ag	1 mg/cm^2 up	b	1
	100 to 5000 $\mu\text{g}/\text{cm}^2$	u	1
	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
	40 mils up	d	1
Cd	1 mg/cm^2 up	b	1
In	5 mg/cm^2 up	b	1
Sn	3 mg/cm^2 up	b	1
	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
	0.5 to 3 mg/cm^2	a	1
Sb	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
Te	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2, 3
Cs (salt)	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
Ba (salt)	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
La (metal) and rare earth oxides	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
Hf	Undetermined	b	1
Ta	2 mg/cm^2 up	b	1
	10 to 1000 $\mu\text{g}/\text{cm}^2$	a	2
W	In Cu matrix: $>2 \text{ mg}/\text{cm}^2$	b, d	2
Re	Undetermined	a	2
Ir	Undetermined	a	2
Pt	3 mg/cm^2 up	b	1
Hg	1 to 4 mg/cm^2	c	2 on Au
Tl	8 mg/cm^2 up	b	1

Table 1. - continued

Element	Range of Thickness	Method of Preparation	Backing
Pb	8 mg/cm ² up	b	1
	1 to 8 mg/cm ²	a	1
	10 to 1000 µg/cm ²	a	2
Bi	Undetermined	a	2

Purity of material in any target is governed largely by the initial purity of the substance used. A mass analysis and a spectrochemical analysis of the isotope production lot, and a spectrochemical analysis of the final target, are provided upon request. Uniformity of targets has been found to vary from $\pm 5\%$ for rolled foils to $\pm 20\%$ for evaporated films.

To protect evaporated films on thin carbon backing and those to be stripped into self-supporting form, the films are shipped either on glass plates (3.75 by 4 in.) or on aluminum sheets (2 by 3 in.) with complete instructions for stripping. This prevents damage to the thin materials during handling and packaging. For the materials in the preceding table, the standard geometric areas of targets are:

Rolled foil: 1 by 1 in.

Evaporated targets on glass slides: 3.75 by 4 in.

Evaporated targets on metal plates: 2 by 3 in.

Pressed targets: 0.25-in. diameter to 2.25-in. diameter in
0.25-in. increments

Quality of films and foils is controlled entirely by weight and spectrochemical measurements. An apparatus is being constructed to measure film thicknesses in the µg/cm² region by the attenuation of the energy of alpha particles having known initial energy. A 20X to 2000X bright and dark field metallographic microscope has been purchased for further support of film studies. Future quality-control efforts will be described in Sect. 3.

2.3 Budget

The current annual research and development budget for target preparations is \$150 thousand; it is estimated that expenditures will continue at this level for another year. In addition, \$100 thousand is budgeted for the production of targets, which is covered by income from sales.

3. FUTURE TARGET PROGRAM

It appears that within the next two years it will be necessary to double the production of targets. This will require increased equipment, increased personnel, additional space, and increased effort on development of techniques and quality control.

3.1 Target Fabrication

If the number of targets now being fabricated monthly (~100) is doubled, it will be necessary to add three vacuum evaporators for the stable target preparation work, three additional evaporators for the fabrication of radioactive targets, and auxiliary apparatus for both operations. It is estimated that the total increase in equipment will cost approximately \$150 thousand. Four more technicians will be required to operate the additional equipment at maximum capacity.

3.2 Quality Control

Quality control is the basis for reproducibility of target characteristics, and it will be necessary to expend effort in this direction. Microscopic examination of film structures and thicknesses, careful mapping of films by use of alpha-, beta-, or gamma-ray energy attenuation as a measure of film thickness, and precise weight measurements in the microgram region are being or will be used. Knowledge of target uniformity, thickness, and purity is required by the researcher in order to provide meaningful results.

3.3 Research and Development

Research and development effort is required to develop procedures for quality control of stable and radioactive targets and to develop techniques for the preparation of targets of all isotopes.

Equipment such as small vacuum and inert-gas systems will be required for experimental work with vapor plating and plasma spraying. Vapor plating will be an extremely valuable technique for bridging the thickness gap between 10,000 angstroms and 1 mil, and should be well suited to the deposition of refractory metals such as Ru, Re, Mo, and W. Plasma heat sources will be investigated for spraying of refractory compounds and elements. The orientation of films formed by vapor deposition will be studied. Requests have been received for single-crystal films to be used in Mössbauer studies, and requests for targets of transuranium elements are increasing. Of most interest are targets of Th^{228} , Th^{229} , Pa^{231} , U^{232} , U^{233} , Np^{237} , Pu^{238} , Am^{241} , Am^{243} , Cm^{242} , and Cm^{244} . Development work is required to determine the best methods of fabricating such targets. Since present facilities for handling radioactive materials -- particularly alpha materials -- are inadequate, it will be necessary to design equipment to permit safe handling.

3.4 Space Requirements

It is estimated that approximately 7000 sq ft of floor space will be required for the fabrication of radioactive and stable targets. The area utilized for handling radioactive materials must be contained to control the spread of contamination. Shielded and unshielded glove boxes will be required to control contamination and radiation within the contained area. The cost of renovating an existing structure for this work is estimated to be \$150 thousand.

3.5 Equipment

The cost of additional equipment for the fabrication of stable targets and for the development of fabrication techniques is estimated to be \$150 thousand. In addition to the equipment needed for stable targets, it is estimated that another \$150 thousand will be required to provide shielded and unshielded glove boxes, vacuum evaporators, rolling mills, and presses for the fabrication of radioactive targets; therefore a total expenditure of approximately \$300 thousand is anticipated for future equipment needs.

3.6 Personnel Requirements

It is anticipated that four additional technical people and four technicians will be needed for developing fabrication techniques and for the actual fabrication of targets.

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