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ISOTOPES DEVELOPMENT CENTER

PROGRESS REPORT

TARGET DEVELOPMENT

October - December 1962

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PROGRESS REPORT

TARGET DEVELOPMENT

October - December 1962

Compiled by:

E. H. Kobisk

Contributions by:

H. L. Adair
H. G. Hunter
E. H. Kobisk
E. B. Olszewski

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SUMMARY

Several materials containing either isotopically enriched or normal elements were converted into target forms, particularly thin films or foils, for the first time. Continued emphasis on the handling of alpha-emitting isotopes proved that targets could be fabricated from such materials.

Evaporation technology continued to be emphasized in developmental efforts. Filament and electron bombardment heating methods and improvement of vacuum systems were studied.

A total of 331 targets (292 stable isotope targets, 23 tritium targets, 16 radiotargets) were fabricated during the quarter; this figure represents a 58% increase over the previous quarter.

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1. STABLE ISOTOPE TARGET DEVELOPMENT

Electron Bombardment Evaporation

Self-supporting films of chromium, iron, and nickel have been prepared by electron bombardment of the parent metal in a vacuum. This method has been previously used to evaporate boron films. Although it is possible to evaporate chromium, iron, and nickel from a filament, each of the metals possesses alloying properties which cause failure of tungsten, tantalum, or molybdenum filaments. Furthermore, losses of valuable enriched material are incurred due to alloying with the filament and collimation of the evaporated molecules.

In the case of chromium, minute quantities of impurities cause embrittlement, which results in very poor thin-film strength. But by electron bombardment and application of the collimation technique,[1] good film integrity was achieved. Thin, self-supporting films (0.5 to 2 mg/cm^2 thick) having a highly reflective surface were prepared. The superiority of the electron bombardment evaporation method to electroplating is illustrated by the fact that the preparation of chromium films of equal thickness by electrodeposition requires at least 20 g of chromium in the plating solution as compared to only 0.5 g by electron bombardment evaporation.

Chromium films are deposited on stainless steel backing previously coated with sodium chloride which is used as a parting agent to remove the chromium from the substrate by water flotation. Although most evaporated materials condense at ambient temperatures into thin films having sufficient strength and continuity, some materials such as chromium form good films only when the substrate is heated or cooled. When the substrate is heated to 70°C, the deposited chromium film is bright and continuous, but friable; at ~250°C the condensed film achieves uniformity and appears nearly as brilliant as electrodeposited chromium; at 400°C the film is a dull amorphous-appearing coating. Further study of condensation conditions is required, since many very small pinholes are usually found in the self-supporting film produced under these conditions.

In the preparation of self-supporting films of iron by electron bombardment, substrates of tungsten and aluminum were found inferior to stainless steel (all coated with sodium chloride). The required substrate temperature was >400°C, for at lower temperatures the iron film became very fragile and cracked into narrow strips during water flotation. Films, 115 μg/cm^2 thick, were obtained from 100 mg of iron. The largest self-supporting film of iron which could be mounted on a suitable frame was only 1/2 in. in diameter (1-1/2-in.-diam chromium films obtained).

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[1] E. H. Kobisk, Target Development, July-September 1962, ORNL-TM-442.

Rolled, Pressed, and Cast Targets

Thin foil targets of tungsten metal were prepared by suspension in a copper metal matrix and by rolling to a minimum thickness of 1 mg/cm^2. Using this same technique, isotopically enriched, powdered molybdenum metal was suspended in molten copper in the radiofrequency furnace, cooled, and subsequently rolled. Molybdenum concentrations of >40 wt % caused the rolled foil to be brittle, and cracking became a serious problem, even though the foil was annealed after each pass through the mill. A satisfactory rolled foil of 40 wt % molybdenum was obtained, but annealing was still required. Similar techniques using as low as 10 wt % ruthenium (in a copper matrix) resulted in highly brittle mixtures which could not be rolled. The embrittlement was probably caused by limited alloying of the copper and ruthenium.

A solid cylinder of Li^7, 9 in. in diameter and 5.5 in. high, was cast. Since lithium metal contracts during cooling, a conical void is usually formed in casting. By casting the metal in a stainless steel container with a 1-in.-diam stainless steel rod placed parallel to the axis of the casting cylinder, this void was eliminated. After the stainless steel rod was removed, the resulting hole was filled with additional molten metal. The finished casting was smoothed on its parallel faces by pressing with a force of 10 tons on a 6-in.diam ram. All casting was performed using mineral oil to retard oxidation. The resulting block was canned in aluminum, heliarc welded, and finally coated at the weld joint with Apieson W to close any pinholes.

Another lithium fabrication problem involved formation of hollow cylindrical cups, 1/2-in. in diameter by 1/2 in. high and 1/2-in. in diameter by 1/4 in. high. Although the pressing operation was straight-forward, lubrication of the dies was difficult. Even when pure mineral oil was employed as lubricant, lithium metal caused severe galling and scoring of the dies after the pressing of several pieces. Lithium metal, oxide, and nitride wastes obtained from the casting operation were agitated with mineral oil and then allowed to settle. Traces of these materials remaining in the oil as a suspension provided excellent lubrication without contamination of the surface with other elements. The oil was free from moisture by reaction with the lithium metal.

2. EQUIPMENT DEVELOPMENT

Because of the increasing number of target requests, more efficient vacuum systems are needed to provide adequate evaporation facilities. Four commercial 4-in. oil-diffusion pumping stations were purchased, and special baseplates were designed and obtained. Each pumping station is capable of producing a vacuum level of $\leq 1 \times 10^{-7}$ Torr. Small 12-in.-diam Pyrex bell jars will be used, in contrast to the existing 18- to 24-in.-diam stainless steel bell jars; thus, rapid pump-down and lower residual vacuum can be attained. Economy of equipment utilization and of cost is being accomplished by special design of multipurpose electronic vacuum-measuring apparatus and by reducing the number of rotary pumps required.

Although the number of evaporation vacuum systems is being increased, almost all the systems will be used for production. An Edwards evaporator was purchased and is being installed for developmental studies of evaporation by electron bombardment, filament heating, sputtering, and radiofrequency heating. Also observation of film growth will be possible.

A small demountable evaporation system was developed which should significantly contribute to the formation of targets using alkali or alkaline earth metals. The system was developed using a 4-in.-diam Pyrex pipe tee as the vacuum enclosure which is evacuated by a 2-in. air-cooled oil diffusion pump. The equipment can be easily contained in an inert-gas, refrigerated, vacuum glove box in which the targets can be prepared and packaged for shipment with minimum corrosion or contamination. The technique should enhance target integrity and produce improved results in nuclear experiments because of the low number of side reactions and low extraneous scattering.

3. RADIOISOTOPE TARGETS

Design and construction of a double containment enclosure for a filament-type evaporator was completed. The evaporator is similar to that noted above for use with alkali metals but has minor changes in the diffusion pump and target holding device. Using this system, 16 targets (10 to 20 $\mu g/cm^2$ thick) were prepared from $U^{235}F_4$, $U^{238}F_4$, and $Pu^{239}F_3$ by depositing the metal fluoride on a 35-$\mu g/cm^2$ carbon film (self-supporting).

Excellent film uniformity was attained and light interference bands were almost completely absent. Contamination encountered using this evaporation device was negligible. Double bagging the targets in plastic envelopes when transferring them from the containment enclosure proved successful.

Targets will be prepared from $Ra^{226}Br_2$, $Pu^{241}F_3$, $U^{233}F_4$, $Th^{232}F_4$, $Am^{241}F_3$, and $Cs^{137}F$.

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