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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

PREPARATION OF FOILS FOR NUCLEAR PARTICLE TARGETS

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PREPARATION OF FOILS FOR NUCLEAR PARTICLE TARGETS

by

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ABSTRACT

Methods for preparing thin foils for use as nuclear particle targets are discussed. A number of specific foil preparations, including elements from each group of the periodic table, are given.

SECTION 1

INTRODUCTION

The usual foil used as a target for particle bombardment consists of a thin deposit ($\mu g/cm^2$ to mg/cm^2) of the desired material, on a flat backing. Common backings are of nickel, gold, platinum, glass, or DuPont "Mylar" film. Self-supporting foils with no backing are made when the backing material would interfere with the experiment.

This paper is divided into several sections dealing with the treatment of the backing, preparation of the deposit, and determination of the thickness of the deposit. Specific directions are given for preparing foils of 34 different elements, some of them by several methods. The preparation of foils of most other elements would be similar to at least one of the elements discussed. Table I lists methods which may be used to prepare foils of many of the elements. A method which is not discussed, that of electrospraying, has been reported previously.[1]

SECTION 2

BACKINGS

To obtain uniform and adherent deposits, the backing must be free

TABLE I

Methods Used to Prepare Foils

Element	Electro-deposition[a]	Vacuum Evap.[a]	Vapor Deposition[b]	Other[a]
H				As hydrides
Li		X		Machining
Be		X	$BeCl_2$	
B		X	BCl_3	
C		X	CH_3I	Painting Machining
N				As nitrides
O				As oxides
Na		X		Machining
Mg		X		
Al		X	AlCl	
Si		SiO	$SiCl_4$	
P				Machining
S		CdS		Machining
Halides		AgCl		
K		X		Machining
Ca		X		
Sc		X		
Ti		TiO_2	$TiCl_4$	Melting
V		X	VI_2	

TABLE I, contd.

Element	Electro-deposition[a]	Vacuum Evap.[a]	Vapor Deposition[b]	Other[a]
Cr	X	X	$CrCl_3$	
Mn		X		
Fe	X	X	$FeCl_3$	
Co	X	X		
Ni	X	X	$Ni(CO)_4$	
Cu	X	X	$Cu(CHO_2)_2$	
Zn	X	X		
Ga		X	$GaAs_3$	
Ge		X	GeI_2	
Se		X		
Sr		X		
Zr		X	$ZrCl_4$	Melting
Nb			$NbCl_5$	
Mo			$MoCl_5$	
Ru	X		$RuCOCl_2$	
Rh	X	X	$RhCOCl_2$	
Pd	X			
Ag	X	X		
Cd	X	X		
In	X	X		
Sn	X	X	$SnCl_2$	

TABLE I, contd.

Element	Electro-deposition[a]	Vacuum Evap.[a]	Vapor Deposition[b]	Other
Sb	X	X	$SbCl_3$	
Te		X		
Ba		X		
La + Rare Earths		X		
Hf		X	HfI_4	
Ta			$TaCl_5$	
W		X	WCl_6	
Re			$ReCl_3$	
Os	X		$OsCl_4$	
Ir	X		$Ir(CO)_2Cl_2$	
Pt	X	X	$Pt(CO)_2Cl_2$	
Au	X	X		
Hg		HgI_2		
Tl		X		
Pb	X	X	$Pb(CH_3)_4$	
Bi		X	$BiCl_3$	

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[a] Elements are used unless otherwise noted.

[b] The compounds listed are those which are decomposed to yield the element. Preparations of the compounds are given in reference 2.

of dirt and grease. Different cleaning procedures are used, depending on the backing material. It is assumed that the backing is free of gross contamination.

Platinum is rinsed in trichloroethylene, dried, and then heated red-hot in a flame. This is one of the better cleaning methods, but it is unfortunately not generally applicable because many metals oxidize during the heating.

Gold and nickel are cleaned in successive baths of boiling trichloroethylene, boiling acetone, and warm benzene. The solvents should be as pure as possible, preferably those specifically made for use in the transistor industry. Deposits will sometimes adhere better to gold if its surface is lightly etched with aqua regia before cleaning.

Thin backings, such as 50-microinch-thick nickel, will tear if cleaned in liquid solvents. This is apparently due to surface tension; the backing will not support the weight of solvent remaining on it as it is removed from the bath. These backings can be cleaned in the vapors of boiling solvents. The backing is held vertically over a beaker of boiling trichloroethylene; the vapors condense on it and fall back into the beaker. Isopropyl alcohol is sometimes used instead of trichloroethylene.

Thin backing films of Mylar are cleaned by wiping with a tissue moistened with trichloroethylene.

Glass backings are boiled for several minutes in "Alconox" (Alconox, Inc.) or a similar cleaner. After being rinsed in distilled water and acetone, the glass is held in vapors of trichloroethylene.

These cleaning procedures certainly do not remove or prevent a monolayer of contaminates from remaining on the backing, but do clean it sufficiently for preparation of foils. If the ultimate in cleaning is desired, a vacuum glow discharge may be used.

To prevent contamination, the backings are put into the vacuum chamber of the deposition apparatus, which is pumped down as quickly as is practical.

SECTION 3

PREPARATION OF FOILS BY VACUUM EVAPORATION

The vacuum evaporation method of foil preparation consists of evaporating a material in a vacuum and condensing the vapors on a backing. General discussions of the procedures, apparatus, and problems are given by Holland,[3] Dushman,[4] and Strong.[5]

The apparatus[6] used in this Laboratory is an all metal, water-cooled vacuum system (Fig. 1). It consists of a 2.3 l/sec mechanical pump and a 700 l/sec oil diffusion pump connected to the evaporation chamber through a 6-inch Freon-cooled pumping throat. The evaporation chamber is a brass cylinder, 12 inches long and 8 inches in diameter, with a quartz viewing port. It fits into an O-ring groove in a bottom plate attached to the pumping throat. The system is capable of attaining a pressure of 10^{-7} mm Hg.

The material to be evaporated is placed in a tungsten or tantalum

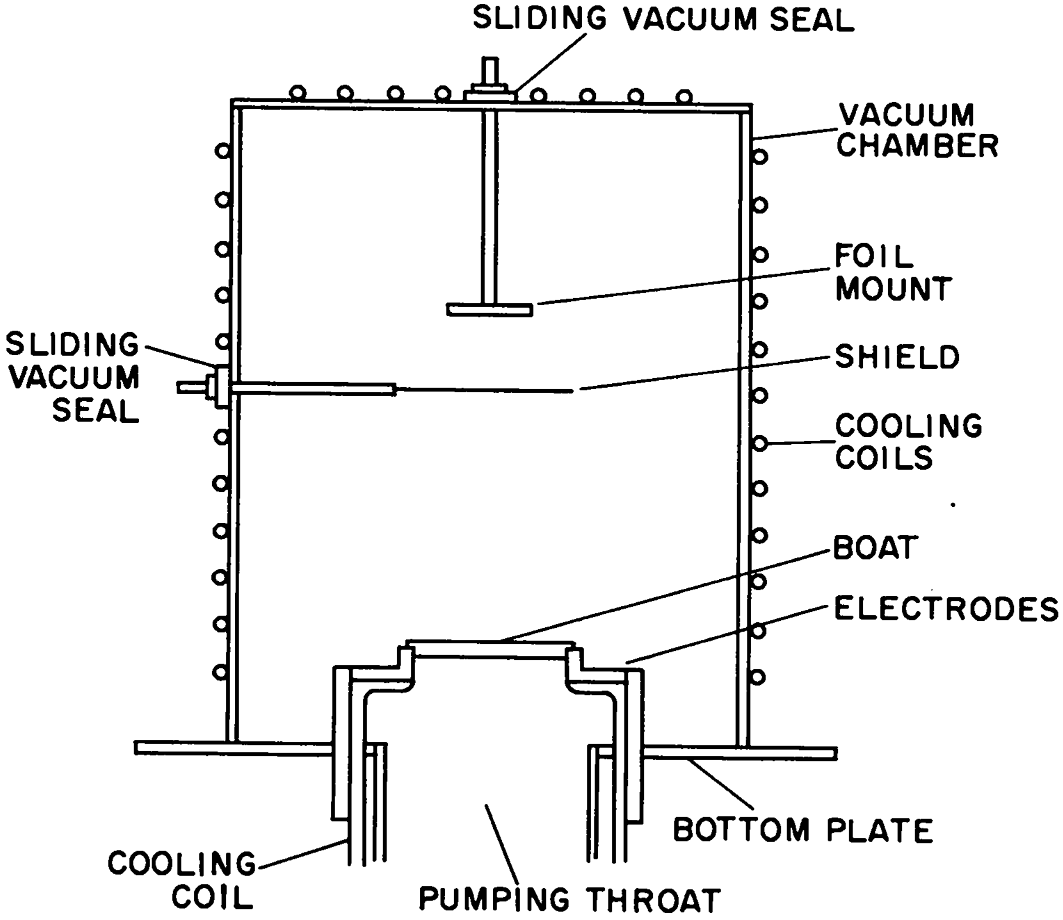


Fig. 1 Vacuum Evaporation Apparatus

boat 5 mils thick, 1 inch wide, and 2 inches long. The boat is bent into a "V" shape to hold the material and is clamped between two water-cooled electrodes which extend (via vacuum-tight Teflon gaskets) out of the bottom plate to the power supply. Although normally either tantalum or tungsten boats are used, some cases require special materials or shapes. These are discussed under the elements that require them.

The power supply is a 5-KVA, 240-volt primary, 24-volt secondary, transformer. The inlet voltage to the transformer is controlled by a 15-KVA Powerstat. This supply has sufficient power to heat a tungsten boat to its melting point. Low voltages are used to prevent power losses through the circulating cooling water.

The backing is suspended 4 to 6 inches above the boat. This distance will usually prevent the backing from becoming too hot and gives good uniformity on a disk 1 inch in diameter. Longer distances will give good uniformity over a larger area but require evaporation of more material. Evaporation occurs over a 2π solid angle, so that the thickness of the deposit varies inversely as the distance squared for the same amount of material evaporated.

The evaporating chamber is pumped down to a maximum pressure of 5×10^{-5} mm Hg before heating of the boat is begun. As the boat is heated, the material will outgas. The temperature is raised slowly so that the material is completely outgassed before evaporation begins. Pressures are allowed to increase to 10^{-4} mm Hg during degassing. About 1/2 hour is taken to reach evaporation temperature. This is for an average case (less than 1 gram of material and an evaporation temperature of 1000 to 2000°C). Raising the temperature slowly also warms the backing before evaporation starts. Warm backings, on the order of several hundred degrees, contribute to adherent deposits.

The temperature is increased after degassing until a visible deposit begins to form on the backing. This temperature is such that the vapor pressure of the material is about 10 microns Hg. Evaporation temperatures or vapor pressures are not normally measured; the temperature is simply increased until deposition occurs at an observable rate. Again, for the average case, evaporation is continued for about 1/2 hour. The thickness of the deposit is determined by the amount of material present in the boat. After evaporation is completed and the power is shut off, the foil is cooled to room temperature before being removed from the vacuum.

Lithium, Sodium, and Potassium Foils

About 1 gram of lithium is washed in hexane and loaded in an iron boat. The backing is covered with a removable shield. At a pressure of 10^{-5} mm Hg, the boat is heated to 500°C, and lithium is evaporated for several minutes onto the shield in order to clean the lithium. The shield is then removed and evaporation continued onto the backing. When the evaporation is complete, the foil is cooled to room temperature and the evaporating chamber filled with dry argon. The chamber and the foil are transferred as quickly as possible to a drybox, where the foil can be removed and placed in a protective container.

Generally the foil must be exposed to the atmosphere for short periods while loading and unloading it from the machine in which it is to be used. Several methods are used to protect the exposed surface of the lithium.

1. The foil is coated with carbon before it is removed from the vacuum chamber. A thin coat of carbon (25 $\mu g/cm^2$) is evaporated over the lithium (see Carbon Foils, this section).

2. The foil is converted to Li_3N before it is removed from the vacuum chamber. This is done by admitting a small quantity of pure nitrogen into the chamber for several minutes. The Li_3N will oxidize if exposed to air for several hours, but it can be handled for short periods.

3. The lithium can be evaporated as a salt, LiF, rather than as the metal. Lithium fluoride is evaporated from a tantalum boat at 1250°C.

Foils of sodium and potassium are prepared in a similar manner. Evaporation temperatures are given below.

Na - - - - 500°C
K - - - - 500°C
NaF - - 1400°C
KF - - - 1300°C

Sodium and potassium oxidize more rapidly than lithium, and it is almost impossible to remove the foils from the vacuum chamber without tarnishing the metal. Sodium and potassium do not form nitrides unless heated by a glow discharge in the presence of nitrogen. Foils of sodium and potassium are generally more acceptable if they are prepared directly by evaporation of the fluorides.

Magnesium and Calcium Foils

Foils of magnesium and of calcium up to 15 mg/cm^2 have been prepared by sublimation of the metal in the vacuum evaporation apparatus. In order to obtain chemically pure foils, redistilled metal which has been stored in an inert atmosphere is used. This metal is quite gassy and spatters out of the boat as it is heated. To prevent this, the boat is covered with a stainless steel screen. The temperature is increased very slowly so that the pumps have sufficient time to pump away the gases evolved. A tantalum boat at a temperature of 450°C for magnesium and 600°C for calcium is used.

Neither magnesium nor calcium adhere well to the backing, especially if the backing is well polished. Adherence is improved by etching the backing in an acid bath before cleaning.

The poor adherence of these metals to the backing makes it relatively simple to prepare self-supporting foils. A clean piece of glass is used for a backing while subliming. When the foil is removed from the evaporating chamber, the metal usually pops off of the glass; if it does not pop off, the edges are loosened with a knife until the metal comes away from the glass.

Magnesium and calcium do not oxidize as quickly as the alkali metals and can be handled in the atmosphere for short periods with no precautions. The foils are stored in a vacuum desiccator when not in use.

Carbon Foils

Very thin carbon foils, approximately 100 μg/cm^2, are prepared by vacuum evaporation. A setup similar to the electrodes of an arc lamp is used. Two rods of graphite lightly touching each other at one end to form a high resistance junction are clamped in the electrode holder. When current is flowing through the graphite rods, a high temperature arc occurs at the junction and evaporates a small amount of carbon. A temperature of about 2700°C is required.

Silicon Foils

Silicon can be evaporated from a beryllia boat at 1400°C, but the deposits are always contaminated with oxides. Consequently, silicon foils are usually prepared directly as the oxide (SiO) so that the chemical form is known. A 1:1 mixture of silicon and silicon dioxide is placed in a quartz tube. The tube is evacuated to less than 10^{-4} mm Hg and the lower portion of the tube heated to 1250°C in a furnace. Silicon monoxide is formed from the reaction of Si and SiO_2 and is distilled to the upper, cooler portion of the tube during the heating. The SiO is removed from the tube and placed in a tungsten boat. SiO is evaporated, in the usual manner, from the boat onto the backing, using a temperature of 1250°C. Higher temperatures cause the SiO to decompose partially to the element.

Halogen Foils

Foils of fluorine, chlorine, bromine, and iodine are prepared by evaporation of the respective silver halides. The evaporation is straightforward, using a tantalum boat at about 1000°C.

Chromium, Iron, and Nickel Foils

When heated, these metals alloy rapidly with the boat and quickly destroy it. Thin foils, in the $\mu g/cm^2$ region, can be prepared by the following procedure.

1. A thin deposit of the metal is electrodeposited onto a tungsten wire 10 mils in diameter. Any of the standard electroplating solutions may be used. The idea is to keep the ratio of the weight of metal to the weight of the filament very small, rather than to make a fine-appearing deposit.

2. The wire is then clamped in the electrode holders of the vacuum evaporation apparatus and heated by resistance to evaporate the deposit. The following temperatures are used:

Cr - - - - 1200°C
Fe - - - - 1450°C
Ni - - - - 1500°C

Vanadium Foils

Vanadium alloys with the boat when molten, but if the temperature is kept only slightly above the melting point, foils up to 500 $\mu g/cm^2$ can be prepared. A molybdenum boat is used at a temperature of about 1800°C. The melting temperature of vanadium varies upward from 1715°C, depending on small amounts of impurities; the evaporation temperature used is 100°C above the observed melting temperature.

Zinc Foils

Zinc foils cannot be prepared by evaporation from an open boat or from a deposit on a wire. Though the zinc evaporates, it does not condense on the backing unless it is evaporated with a high vapor pressure. Zinc is consequently evaporated from an enclosed crucible with a pinhole at the top through which the zinc can diffuse as a jet. The crucible is constructed from mild steel which alloys with zinc only very slowly if the temperature is kept at 400°C.

Platinum Foils

Platinum is best evaporated when it is in the form of a fine wire. The platinum wire is twisted together with several strands of 5-mil tungsten wire. The wires are clamped in the electrode holders and heated by resistance to 2100°C to evaporate the platinum.

If the platinum is in a powder or chip form, it can be evaporated from a carbon boat (Fig. 2). The boat is machined from a bulk piece of carbon with walls as thin as possible to increase the resistance across the boat.

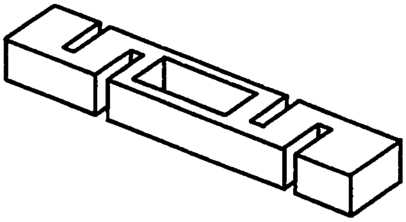


Fig. 2 Carbon Boat

Gold, Silver, and Aluminum Foils

Gold, silver, and aluminum foils are prepared by vacuum evaporation of the metal from a tungsten boat. Temperatures of 1500°C for gold, 1050°C for silver, and 1000°C for aluminum are required. These metals evaporate very smoothly since there is little problem of degassing.

Mercury Foils

Mercury foils are prepared by subliming HgI_2 from a tantalum boat at about 350°C. It is necessary to clean the apparatus immediately after subliming to prevent corrosion. No decomposition of the HgI_2 is noted during sublimation. The adhesion to the backing is not good, but the foils can be used if reasonable care is taken to prevent rubbing off the deposit.

Thallium, Lead, Indium, and Bismuth Foils

Thallium, lead, indium, and bismuth foils are prepared by vacuum evaporation from a tantalum boat. A temperature of about 700°C is used. The thin oxide coat often found on these metals can be removed by momentarily heating at 1000°C with a shield covering the backing. The temperature is then decreased to 700°C and the shield removed to expose the backing. Evaporation is continued in the usual manner.

Lanthanum, Cerium, and Samarium Foils

Lanthanum, cerium, and samarium foils are prepared by vacuum evaporation from tantalum boats. Temperatures of 1000 to 1500°C are required. Although these metals cannot usually be obtained in an oxide-free state, the oxides evaporate at higher temperatures and consequently remain in the boat. No oxides are noted in the deposit immediately after evaporation. The foils must be stored in a vacuum, as they oxidize in air.

The metal must be completely degassed before evaporation begins; about 1/2 hour of heating below the melting point of the metal is necessary. Sometimes it is necessary to wrap a grid of fine tungsten wire around the boat as a screen to prevent gassy pieces of the metal from splattering out of the boat. After the temperature has been increased to the melting point and the pressure has decreased to 10^{-5} mm Hg, the evaporation is straightforward.

Yttrium Foils

Yttrium is evaporated from a tantalum boat at 1600°C. Although yttrium is similar to the rare earth metals, it does not oxidize as readily at room temperatures and consequently does not require much degassing before evaporation.

SECTION 4

PREPARATION OF FOILS BY VAPOR DEPOSITION

This method consists of evaporating a compound of the desired element into a stream of a carrier gas. The resulting gaseous mixture is then thermally decomposed on the surface of the backing material. A metal or an oxide is usually obtained on the backing. A general discussion is given by Powell et al.[2] The apparatus varies somewhat, depending on the temperatures required.

Vapor Deposition of Titanium

The procedure is carried out in a quartz gas system as shown in Fig. 3. Hydrogen is bubbled through $TiCl_4$, a liquid at room temperature, and the resulting gas mixture is passed into a chamber containing the tungsten backing. After the mixture has swept through the chamber for several minutes, the backing is heated to 1200°C by an induction coil heater. The $TiCl_4$ decomposes to the metal on the heated backing. The gaseous decomposition products (HCl and unreacted $TiCl_4$) are collected in a liquid nitrogen cold trap. About 1 hour is required to deposit 1 mil of titanium.

Vapor Deposition of Zirconium

The procedure used is similar to that of Robb and Shipko.[7] The apparatus is shown in Fig. 4. The zirconium is several pieces of ribbon twisted together and formed into a "C" shape surrounding the tungsten backing. Both the zirconium and the backing are in the field of an induction coil heater, but since the zirconium does not form a complete circle it receives heat only by radiation from the backing.

1. Iodine crystals are placed in the lower chamber.
2. The iodine is prevented from subliming by cooling the lower chamber with liquid nitrogen.
3. The system is pumped down to a pressure of 0.1 micron.
4. The tungsten backing is heated to 1200°C. After several minutes, the liquid nitrogen is removed from the iodine.
5. As the iodine sublimes, it reacts with the warm zirconium to

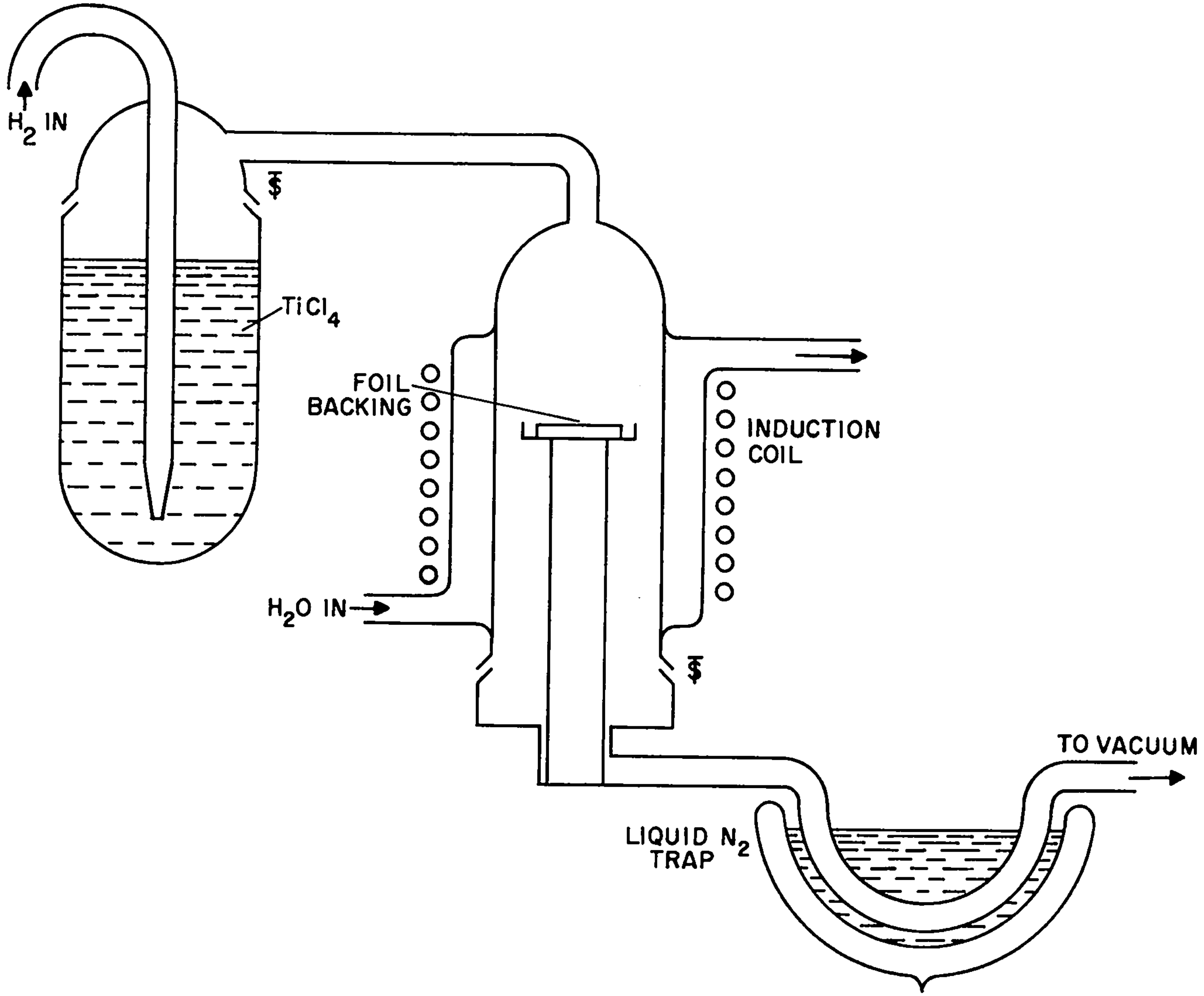


Fig. 3 Vapor Deposition of Titanium

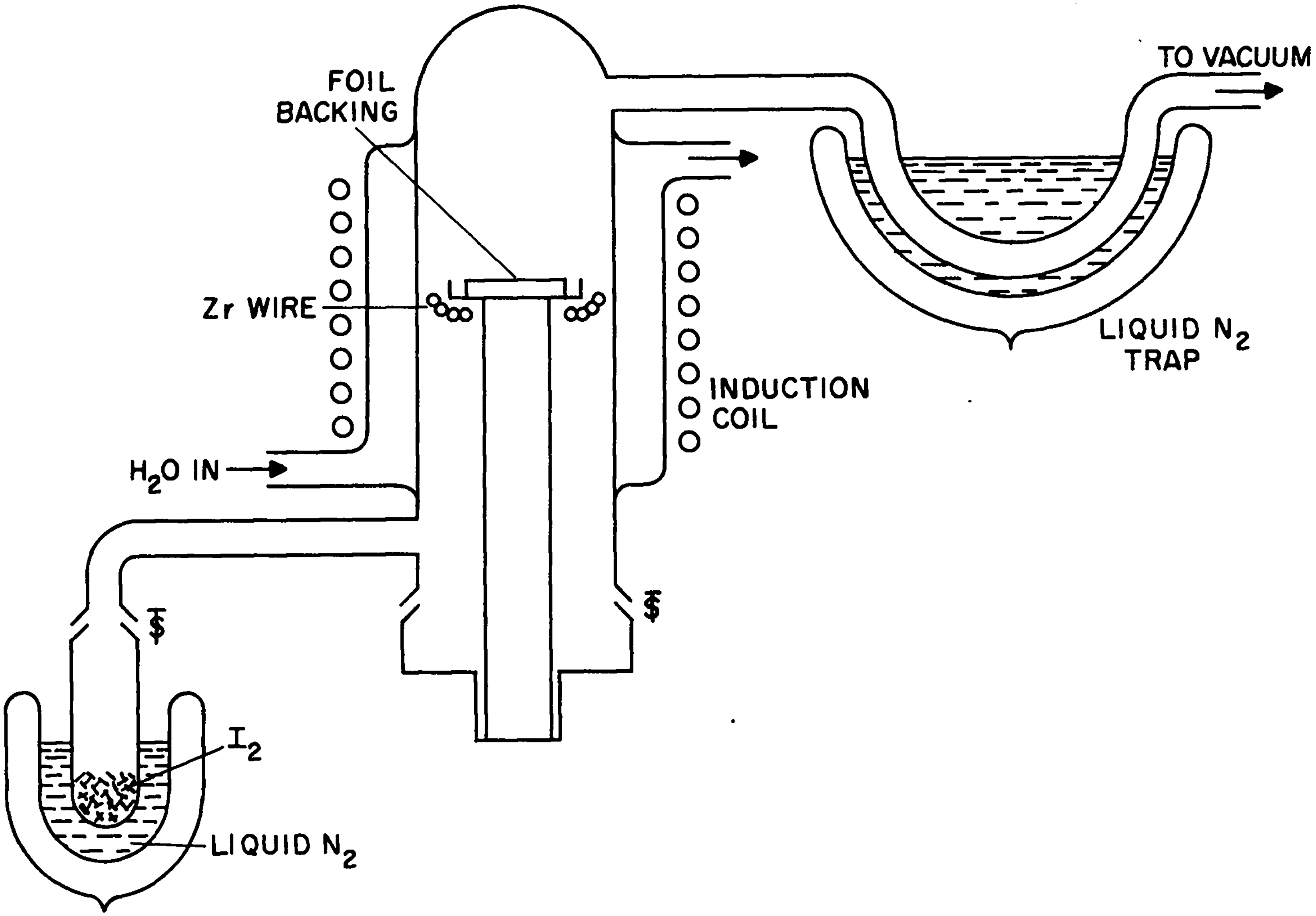


Fig. 4 Vapor Deposition of Zirconium

form ZrI_4. The ZrI_4, a gas at about 300°C, decomposes to the metal on the backing. Some ZrI_4 condenses on the walls of the chamber. One mil of zirconium is deposited in about 1/2 hour.

6. The iodine is then cooled with liquid nitrogen. After sublimation has stopped, the heater is shut off and the foil cooled to room temperature.

Vapor Deposition of Uranium

Uranium foils (as UO_2) can be prepared by decomposing uranium(IV) acetylacetonate to the oxide on a variety of metal backings. This method is advantageous in that only low temperatures (600°C or less) are required and the apparatus is consequently more simple than that necessary for vacuum evaporation of UO_2.

Uranium oxide (UO_3) is refluxed with hexachloropentane to form UCl_4. Excess hexachloropentane is used; UCl_4 is not soluble in the hexachloropentane and settles to the bottom of the refluxing flask. After cooling, the hexachloropentane is decanted and the UCl_4 is dissolved in ethanol. An amount of 2,4-pentanedione 20% in excess of the stoichiometric amount is added. The solution is made neutral to litmus paper by adding NaOH. Precipitation of uranium(IV) acetylacetonate begins immediately. The precipitate is filtered off and dried at 120°C.

Small foils, less than 1/2 inch in diameter, are made in the apparatus shown in Fig. 5a. The backing is heated by a resistance coil around the outside of the tube. Since larger foils are not heated uniformly in this way, an induction coil heater is used, Fig. 5b. The

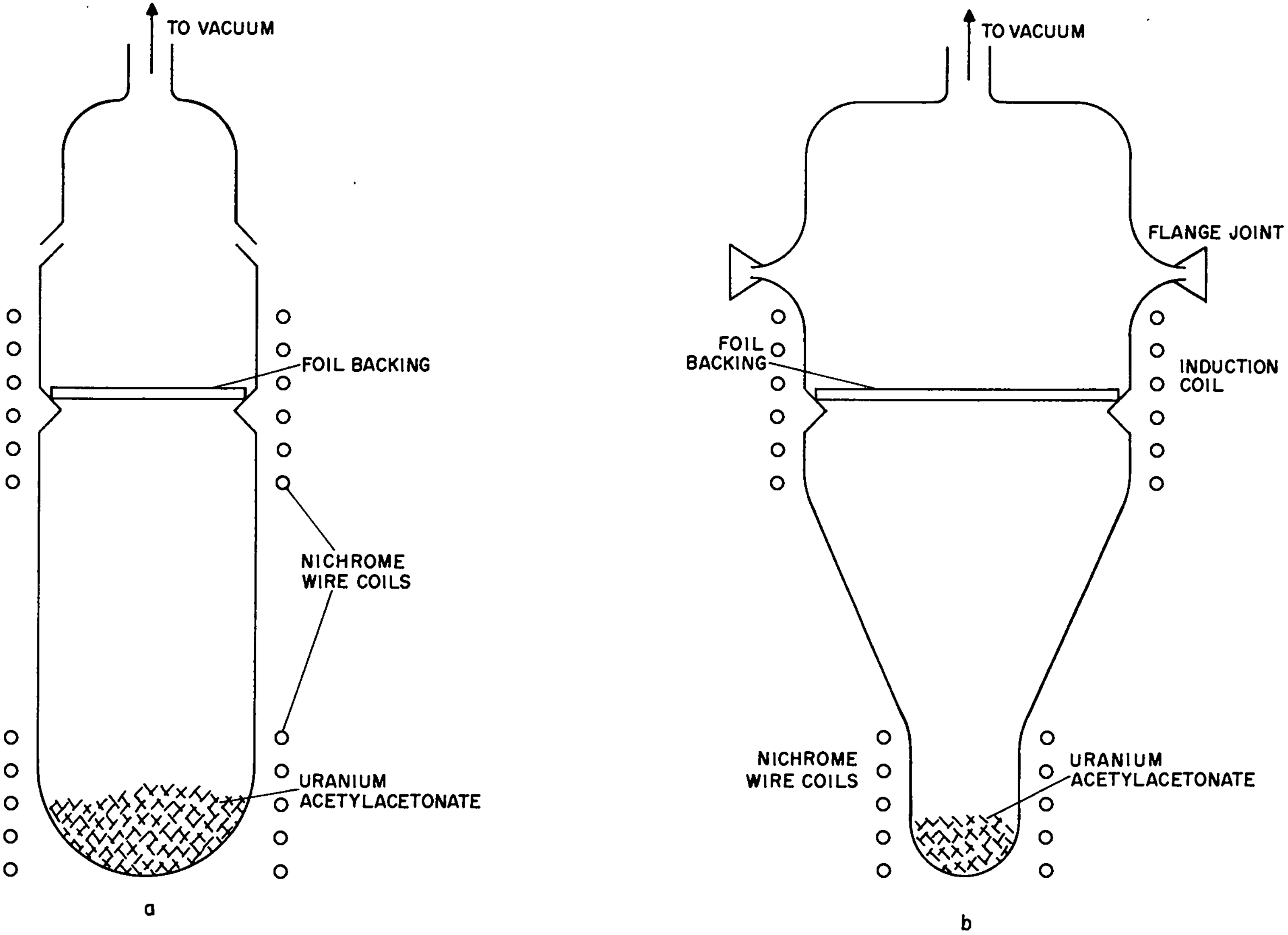


Fig. 5 Vapor Deposition of Uranium

uranium(IV) acetylacetonate is heated by resistance coils in both cases.

The dry uranium(IV) acetylacetonate is placed in the lower part of the vacuum chamber. The backing is placed above. The chamber is pumped down to 10^{-5} mm Hg and the backing heated to ~ 600°C. The uranium(IV) acetylacetonate is then heated to 315°C to vaporize it. The vapors decompose to UO_2 on the surface of the backing, and on the walls of the chamber, if resistance heating is used. The deposition rate at these temperatures is 100 μg/minute.

SECTION 5

MISCELLANEOUS METHODS FOR PREPARING FOILS

Machining

Occasionally a very thick (greater than 10 mils) self-supporting foil is desired. Foils of the alkali metals (15 mils thick) and of carbon (20 mils thick) have been prepared by machining a bulk piece of the material to the required thickness, using either a lathe or a microtome.

A piece of lithium of the desired diameter and several millimeters thick is mounted on a vacuum chuck in a lathe or microtome. A steady stream of mineral oil is pumped over the lithium if a lathe is used. The microtome is operated in a drybox. One side of the lithium is trimmed flat, taking off slices 10 microns thick at each pass of the cutting tool. The lithium is then turned over and the other side similarly trimmed until the remaining piece has the desired thickness.

Foils of sodium and potassium are prepared in a similar manner.

To protect the foil from oxidation when it is removed from the protective atmosphere, both sides are coated with silicon grease (applied with a tissue) and covered with a film of Mylar, 0.25 mil thick.

Thick carbon foils can also be prepared by machining in a microtome. The procedure is similar to that used for lithium foils, except that no precautions to prevent oxidation are required. A bulk piece of carbon is mounted in a microtome and passed across the cutting blade. The chuck is advanced about 10 microns at each pass. The carbon is taken off as a dust rather than in slices. Machining is continued until the remaining piece is the correct thickness.

Painting

Thick carbon foils, 15 mg/cm^2, are prepared by painting. Colloidal carbon ("Aquadag", Acheson Colloids Co.) is diluted with water until it flows readily. The diluted colloid is painted on the backing in thin coats and dried under an infrared heat lamp after each coat. Although this procedure is tedious, almost any thickness of carbon can be obtained. If the carbon is painted on a glass backing, the foil can be removed from the glass by sliding a knife blade between the carbon and the glass.

Absorption -- Hydrogen, Deuterium, and Tritium Foils

The method generally used in this Laboratory to prepare foils of the hydrogen isotopes is that of Graves et al.[8] In essence, a tungsten disk, 1 cm in diameter, is coated with zirconium. When the zirconium deposit is heated and cooled in the presence of hydrogen, hydrogen is collected by the zirconium by occlusion into the metal lattice.

The zirconium is coated on the tungsten by first spot welding a piece of 1-mil zirconium to the tungsten (to achieve thermal contact), and then heating the foil to the melting point of zirconium so that molten zirconium flows over the surface of the tungsten. To obtain uniform coats of zirconium, the following procedure must be followed.

1. The tungsten must be free of absorbed gases. This is achieved by heating the tungsten (with an induction coil heater) _in vacuo_ to 2000°C. The heating is continued for 1-minute periods until a Pirani gauge on the vacuum system fails to show an increase in pressure during the heating.

2. After the tungsten is cooled, it is removed from the vacuum system, the zirconium is spot welded onto it, and the foil is returned to the vacuum system.

3. The zirconium is then degassed by heating to 1600°C _in vacuo_. Heating periods of 15-30 seconds are used with sufficient time between heatings to let the system pump down. The foil is considered degassed when the pressure during heating does not rise above 2×10^{-5} mm Hg.

4. The foil is then heated to 1700°C (melting point of zirconium) several times for 15 seconds. The zirconium collects in a ball at the center of the tungsten disk.

5. The foil is next heated for 15 seconds at 1800°C and cooled for 10 seconds. This heating and cooling cycle is repeated 15 to 20 times;

during each cycle the zirconium flows slightly toward the edge of the tungsten. If the zirconium does not flow, the tungsten and/or the zirconium were not sufficiently degassed.

The coating procedure also activates the zirconium so that hydrogen is absorbed. The foil must not be exposed to the atmosphere before hydrogen is absorbed or the absorption will be blocked.

The following procedure is used to absorb the hydrogen.

1. Hydrogen is introduced into the heating chamber at a pressure of 10 mm Hg.

2. The foil is heated to 1200°C for 1 minute, then cooled to room temperature in the atmosphere of hydrogen.

3. The volume of hydrogen collected is calculated from the change in pressure. Excess hydrogen is pumped out of the chamber and the foil removed.

The average amount of hydrogen collected by zirconium corresponds to a 1:1 mole ratio (hydrogen to metal). Although the maximum collection rate occurs at 500 to 600°C, measurable absorption occurs at all temperatures between 200 and 1000°C.

When tritium foils are prepared, special precautions are necessary. Tritium is stored and handled in an all-glass vacuum system capable of a vacuum of 10^{-6} mm Hg. The vacuum system and all auxiliary equipment (pressure gauges, tools, extra glassware) are in a bench hood with a forced air draft of 100 linear feet/minute. Tritium-contaminated apparatus that is to be discarded is sealed in polyethylene bags which are sealed in cardboard boxes for ultimate disposal. Protective clothing and gloves are worn when handling contaminated apparatus.

A tritium sniffer monitors the air immediately outside the hood. A full scale deflection of the meter on the most sensitive range indicates a concentration of tritium in the air of 200 $\mu c/m^3$. At this concentration, only 3 working hours per week are tolerable. In practice, the sniffer seldom should indicate more than 30 $\mu c/m^3$. Urine samples are submitted routinely for tritium assay.

Amounts of tritium up to 12 liters are stored in metal cans, semi-permanently mounted in the hood and attached to the vacuum system. Smaller amounts of tritium (on the order of 100 ml) are taken from the can and stored in a charcoal trap at liquid nitrogen temperature. The tritium can be released from the charcoal trap by warming the trap to room temperature. In the event of breakage, the volume of tritium which could escape is only that amount in the system.

When tritium has been in the vacuum system, the system is pumped down to a pressure of 10^{-6} mm Hg, to insure that there are no small amounts of tritium present which were not absorbed into the charcoal trap.

The charcoal trap is also used to separate helium from the tritium. Since helium is not absorbed on charcoal, the helium can be pumped away while maintaining the trap at liquid nitrogen temperature.

Tritium foils are stored in cardboard boxes sealed with tape. Sealed containers of tritium foils which have been standing for some time should be opened in the hood, as the foils very slowly release tritium.

Pressing -- Iodine

Thick, self-supporting foils of iodine (50 μg/cm^2) can be made by pressing. Iodine crystals are crushed very fine and compacted into a solid disk several millimeters thick in a die on a hydraulic press. Since iodine sublimes at room temperature, the disk of iodine is simply left in a hood until a disk of the desired thickness remains. Sublimation occurs evenly over the surface of the disk.

SECTION 6

ELECTRODEPOSITION

A number of metals may be deposited by electroplating. This method is particularly useful for preparing foils of isotopes which are available only in limited quantities. Normally over 90% of the metal can be deposited on the backing, and the remainder can be recovered from solution by chemical means.

Electrodeposition is also useful in preparing thicker foils of the transition metal elements, many of which alloy with tungsten when molten and cannot be prepared by vacuum evaporation. Foils of precious metals are prepared by electrodeposition owing to the high intrinsic value of the metal. The electrodeposition cell[9] is shown in Fig. 6. A glass chimney is held down by springs onto a metal base. A gasket of rubber or "Tygon" (U.S. Stoneware Co.) between the chimney and base forms a

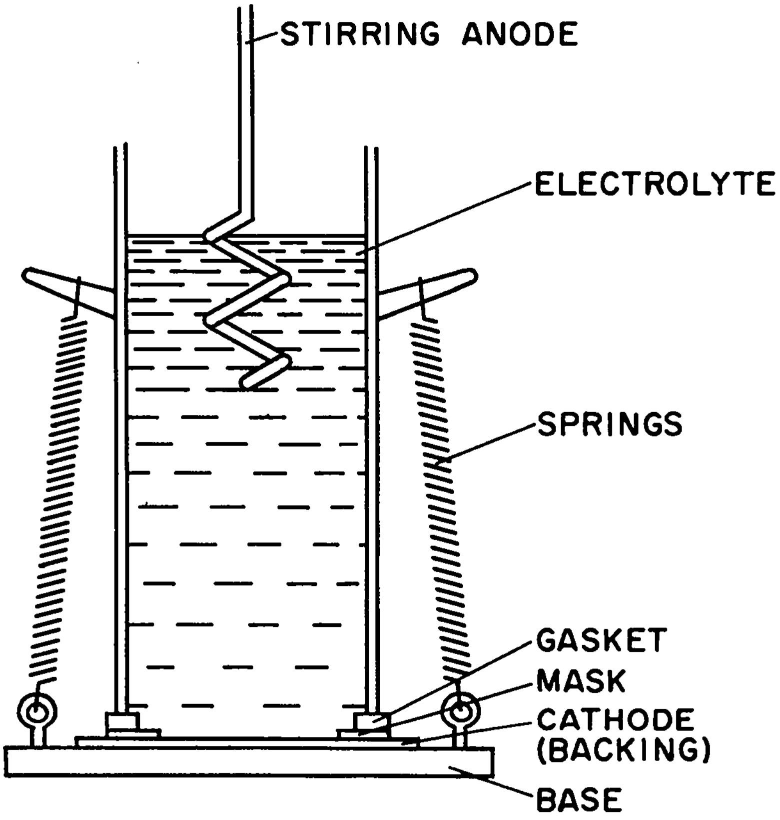


Fig. 6 Electrodeposition Cell

leak-proof seal. The backing material is slipped between the gasket and the base; the backing is in contact with the base. A platinum spiral is rotated inside the chimney at about 100 R.P.M. to stir the electrolyte. Electrical connections are made to the stirrer (anode) and the base (cathode).

The source of voltage can be any variable, low-power D.C. supply. For best results, the D.C. voltage should have the smallest possible A.C. ripple. From this standpoint, a storage battery would be preferable, but other considerations (need to recharge and difficulty of varying voltage) generally make a battery impractical. An all semiconductor power supply, illustrated in Fig. 7, utilizing a Zener diode filter, is used in this Laboratory.

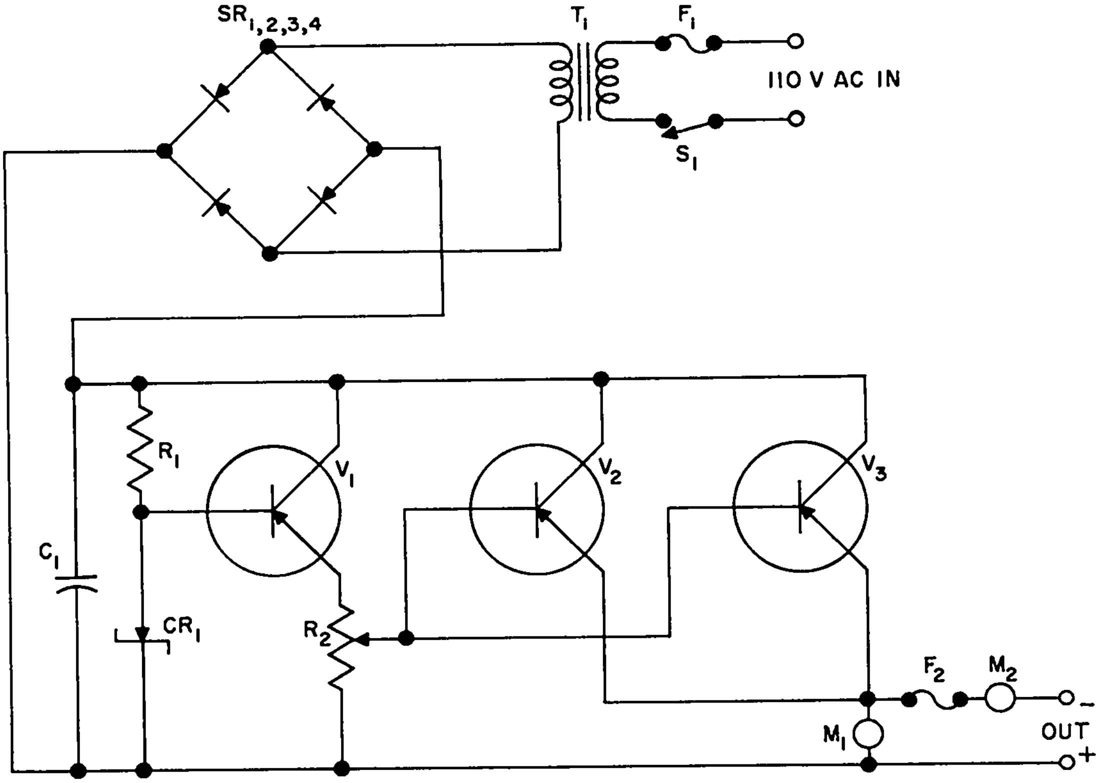
The most difficult part of electrodeposition is determining the correct electrolyte. A number of electrolytes found to be particularly useful in the preparation of foils are listed below. Temperatures and current densities are also given.

<u>Chromium</u>

CrO_3 -- 500 g/l
H_2SO_4 -- 5 g/l
60°C, 30 ma/cm^2

<u>Iron</u>

$FeSO_4 \cdot 7H_2O$ -- 120 g/l
$(NH_4)_2C_2O_4 \cdot H_2O$ -- saturated (~50 g/l)
H_3BO_3 -- 5 g/l
20°C, 15 ma/cm^2



T_1	110 v pri, 7.5 v sec. filament transformer
S_1	SPST switch
F_1	1 amp fuse
F_2	1 amp fuse
C_1	2000 µf, 25 v elec. capacitor
R_1	50 ohm, 1 watt resistor
R_2	100 ohm, 5 watt variable potentiometer
$SR_{1,2,3,4}$	1N536 rectifiers
CR_1	1N429 Zener diode (5.9 volts)
V_1, V_2, V_3	2N554 transistors
M_1	0-10 D.C. voltmeter
M_2	0-1 D.C. ammeter

Fig. 7 D.C. Power Supply

Manganese

$MnSO_4$ -- 150 g/l

$(NH_4)_2SO_4$ -- 75 g/l

Gum arabic -- 5 g/l

20°C, 15 ma/cm^2

Nickel

$NiCl_2$ -- 150 g/l

$(NH_4)_2SO_4$ -- 200 g/l

NH_4OH -- 100 ml/l

60°C, 10 ma/cm^2

Zinc

$ZnSO_4$ -- 150 g/l

3N NaOH is added until $Zn(OH)_2$ begins to precipitate. The precipitate is dissolved by adding small quantities of dilute H_2SO_4.

60°C, 10 ma/cm^2

Plating solutions for the precious metals are given by Hampel.[10]

SECTION 7

THICKNESS MEASUREMENT

The thickness of the deposit is most conveniently determined by knowing the amount of material deposited and the area covered. It is assumed that the deposit is uniform. If the deposit is radioactive, the foil may be counted to determine the amount. The deposit may be weighed, but it is necessary (1) that the chemical form of the deposit be known, e.g., the degree of oxidation; and (2) that the backing material maintain a constant weight. The chemical form is usually known or can be determined, but some types of backings, e.g., 0.005 mil nickel, do not maintain a constant weight. In some cases the only way to determine the amount of deposit is to dissolve the foil and analyze the solution. This is done after the foil has been used.

An optical method utilizing Fizeau fringes can sometimes be used for very thin foils.[3] The foil is prepared by depositing on both the backing and on a glass microscope slide at the same time. The deposition geometry must be arranged so that each receives a deposit of the same thickness. A groove is scratched in the deposit on the glass so that a channel with a depth equal to the thickness of the deposit is formed. A second microscope slide is brought near the deposit, producing Fizeau fringes. These fringes trace out points of equal air gap thickness between the second slide and the deposit when viewed under a monochromatic light. With a sodium lamp as the monochromatic light source, each fringe represents a change in air gap thickness of 29.5×10^{-6} cm (1/2 wavelength). The fringes are displaced as they pass over the channel since the air gap is greater along the channel. This displacement, expressed as a fraction of the fringe spacing, multiplied by 29.5×10^{-6} cm, gives the depth of the channel, i.e., the thickness of the deposit.

The set-up for measuring the fringes may be quite crude and still give accurate results. A half-silvered mirror is mounted at a 45° angle over the slides so that a horizontal light source is reflected down on them. The half-silvered mirror also allows perpendicular observation of the fringes, a necessary condition. A traveling microscope is used to measure the fringe spacing and displacement.

This method can be used to measure foils only when the thickness is between 10 and 3000 Å. By using a similar method with white light and a spectrograph, the maximum thickness measured can be extended to 10,000 Å.[11]

REFERENCES

1. L. N. Blumberg, P. Stein, and J. C. Gursky, unclassified report LA-2711, 1962.
2. C. F. Powell, I. E. Campbell, and B. W. Gonser, "Vapor-Plating", Wiley and Sons, New York, 1955.
3. L. Holland, "Vacuum Deposition of Thin Films", Wiley and Sons, New York, 1956.
4. S. Dushman, "Scientific Foundations of Vacuum Technique", Wiley and Sons, New York, 1949, Ch. 11.
5. J. Strong, et al., "Procedures in Experimental Physics", Prentice-Hall, Inc., New York, 1938, Ch. 4.
6. J. G. Povelites, "Preparation of Foils", paper in Second International Conference on Peaceful Uses of Atomic Energy, 14, pp. 432-436, United Nations, Geneva, 1958.
7. W. L. Robb and F. J. Shipko, "Iodine Decomposition Process for Coating Uranium with Zirconium", unclassified report KAPL-876, 1953.
8. E. R. Graves, A. A. Rodrigues, M. Goldblatt, and D. I. Meyer, Rev. Sci. Instr., 20, 579-582 (1949).
9. R. W. Dodson, A. C. Graves, L. Helmholz, D. L. Hufford, R. M. Potter, and J. G. Povelites, Chapter 1 in A. C. Graves and D. K. Froman, "Miscellaneous Physical and Chemical Techniques of the Los Alamos Project", McGraw-Hill, New York, 1952.
10. C. A. Hampel, "Rare Metals Handbook", Reinhold, New York, 1954.
11. G. D. Scott, "The Measurement of the Thickness of Evaporated Films by a Multiple Beam Interference Method", paper in E. S. Perry and J. H. Durant, "Nat. Symposium on Vacuum Technology Transactions", pp. 24-28, Pergamon, New York, 1956.