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NUCLEAR TARGETS

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ABSTRACT

Of all the diverse physical, chemical and mechanical techniques used to make thin film nuclear targets, material deposition by vacuum evaporation is the most widely employed. This review commences with a brief description of the basic principles that regulate vacuum evaporation and the physical processes involved in thin film formation, followed by a description of the experimental methods used. The principle methods of heating the evaporant are detailed and the means of measuring and controlling the film thickness are elucidated. Types of thin film nuclear targets are considered and various film release agents are listed. Thin film nuclear target behaviour under ion-bombardment is described and the dependence of nuclear experimental results upon target thickness and uniformity is outlined. Parameters such as thermal effects, radiation damage and sputtering of target material all influence the useful lifetime of thin nuclear targets. Target impurities can also have a serious effect upon experimental results; these effects are

briefly considered. Special problems associated with preparing suitable targets for lifetime measurements are discussed.

Carbon is used extensively as a target material and also for the manufacture of stripper-foils to change the polarity of the accelerated ions. The causes of stripper-foil thickening and breaking under heavy-ion bombardment are considered. A comparison is made between foils manufactured by a glow discharge process and those produced by vacuum sublimation. Consideration is given to the methods of carbon stripper-foil manufacture and to the characteristics of stripper-foils made by different techniques. Various methods to increase stripper-foil lifetimes are considered.

Finally, techniques are described that have been developed for the fabrication of special targets, both from natural and isotopically enriched material, and also of elements that are either chemically unstable, or thermally unstable under irradiation. The reduction of metal oxides by the use of hydrogen or by utilising a metallothermic technique, and the simultaneous evaporation of reduced rare earth elements is described.

A comprehensive list of the common targets is presented.

1. Introduction

The practical applications of vacuum deposited thin films have developed continuously during the past 40 years. Wartime requirements, for example, of high efficiency optical systems, led to studies of the basic physics involved in vacuum deposition techniques. The knowledge gained has served well in the more recent developments of the solid state electronics industry. The technology and understanding of films less than 1 μM thick have made considerable advances, primarily because of the demand for reliable, thin-film microelectronic devices to satisfy the requirements of the satellite and computer era. In addition to major contributions to a variety of new and future scientifically based technologies, thin film studies have provided impetus to many new areas of research in solid-state physics, chemistry and surface physics, which are based on phenomena uniquely characteristic of the geometry, thickness and structure of thin films.

Thin films vary in structure from amorphous films such as anodic oxide dielectrics to single crystal films such as epitaxially grown silicon. Most films used for nuclear targets however are located between these extremes and are polycrystalline; being composed of many small crystals, fitted together with more or less random orientation. Crystallite size depends on deposition parameters such as substrate temperature, deposition rate, substrate topography, material, etc., and after deposition may be modified by annealing, causing the larger crystallites to grow at the expense of the smaller ones. This does not matter except in a small range of specialist experiments which determine ion-solid forces (not to be discussed in this review).

Low energy nuclear physics research is a field of study that places stringent technical needs on the thin films used as irradiation specimens. Thin films, sources and targets, are necessary for many applications in

nuclear physics and nuclear chemistry experiments; consequently target preparation is crucial to the success of investigations being undertaken.

Although many excellent publications exist on general thin film technology¹⁻¹² few comprehensive articles have been published on general nuclear target preparation; except for the reviews of Yaffe¹³, Parker and Slatis¹⁴ and Muggleton¹⁵. However, in 1963 a symposium on research materials for nuclear measurements was held at the Central Bureau of Nuclear Measurements (BCM), Geel, Belgium, which was directed to identifying the availability of these valuable materials and their transformation into thin films and other forms of well defined character. Similar symposia were held at AERE, Harwell, UK¹⁶ and at Gatlinburg, Tennessee, USA¹⁷.

It was considered that there was sufficient interest in nuclear target preparation that an International Nuclear Target Development Society should be formed; this was established in 1974. Since then, annual conferences of the society have taken place at major research establishments and universities throughout the world.¹⁸⁻²⁸

Apart from individual papers on nuclear target techniques published periodically in Nuclear Instruments and Methods in Physics Research, and the conference proceedings referred to above, often the information exists only as laboratory notes compiled by individuals in various laboratories who prepare the targets but seldom published the results.

The studies to be described refer to low-energy nuclear physics research using targets with thicknesses from $\sim 1 \mu\text{g}/\text{cm}^2$ to $\sim 10 \text{ mg}/\text{cm}^2$ surface density. The majority of targets used are in the form of thin solid films and consequently gas or liquid targets will not be considered. Thin film targets have been developed using a multitude of deposition techniques, which can be roughly classified as chemical, mechanical or physical preparations. However, the object of this review is to discuss the techniques involved in producing nuclear targets by vacuum deposition methods.

Target preparation is crucial to the success of an experiment. It is therefore necessary that the target conforms to what is required regarding purity, composition, thickness, etc. In general, target properties required can be summarised as follows:

- a) A constant and adequate yield during the experiment.
- b) Uniform thickness.
- c) Ease of preparation.
- d) Good tensile strength.
- e) Chemical and isotopic purity.
- f) Conservation of rare and expensive isotopic materials.
- g) Stability under beam bombardment.

Some applications will now be given of physical aspects of the above requirements.

a),b) Achievement of satisfactory yield depends on the target having a uniform and acceptable thickness. For many purposes a constant yield is obtained with a target which is either thin or thick in the sense of whether it introduces negligible or full energy loss in the exciting beam. However, in the measurement of resonance phenomena, it is more appropriate to define a target as thin if its thickness is small compared to the half width of a resonance. A target which is thin for a broad resonance may not necessarily be thin for a narrow resonance. Likewise, a thin target for protons may be thick for heavy ions.

Target properties c) and d) are obvious practical considerations, in particular d) as the target must be strong enough not to break under beam irradiation throughout the length of the experiment and to withstand mounting in the target chamber.

e) Chemical purity is necessary to prevent background interference from unwanted elements within the target, it is therefore necessary to use chemicals of the highest purity obtainable. Great care is also necessary to reduce chemical contamination introduced from the vacuum equipment or transferred from the source used to evaporate the target material.

Isotopic purity, in the context used here, refers to the ratio between the isotope of interest and other isotopes of the same element. The availability of isotopes is severely limited and only a handful of manufacturers exist throughout the world, consequently it is often necessary to make do with what is obtainable. In general the experimenter requires an isotope with the highest enrichment obtainable.

f) Because of the difficulties involved in manufacturing highly enriched isotopes and their limited availability, they are very expensive (e.g. prices vary from 25 cents per milligram for the more abundant isotopes such as 97% enriched ^{98}Mo to \$10,700 per milligram for 40% enriched ^{46}Ca). The utmost care is necessary to conserve the small amount of such rare material that is usually available. A compromise is invariably made between the thickness or mass of the target required and the uniformity of the deposited film.

g) Finally, it is necessary that the target remains stable under beam bombardment and gives a constant yield throughout the length of the experiment. Target stability is a function of target material, target thickness and the temperature generated in the target by the beam. Various techniques are employed to keep the target from deteriorating, i.e. cooling the target with gas, water or liquid N_2 , or He, moving the target relative to the beam spot, using a high melting point compound of the isotope under investigation; sandwiching the target between thin carbon films, using low beam currents, etc.

2. Deposition Techniques

Material deposition techniques can be roughly classified as chemical, mechanical and physical preparations.

2.1 Chemical Techniques.

2.1.1. Electrodeposition.

Electroplating has been known for a considerable time and many standard textbooks now exist on the subject²⁹⁻³⁶. However, of the 70 metallic elements, it has been found possible to plate only 33 of these successfully. Natural isotopic targets have been prepared by electrodeposition³⁷⁻⁴⁰ but the main application of this technique is in the production of radioactive targets^{13,14,42}.

Although this technique has the advantages of economy and the ability to conserve the small amounts of expensive isotope used, good electroplated deposits depend largely on the use of additives, all of which are potentially capable of introducing impurities.

2.1.2. Molecular Plating⁴³⁻⁵⁰

The most significant difference between molecular plating and conventional electrodeposition is that electrolyte dissociation does not occur to any significant degree during the passage of current; the material in solution is not deposited as the metal but as a compound. Parker and Falk⁵¹ named this method "molecular plating". Other differences compared to electrolytic plating, are the use of high voltages (50-2000V) and of an organic solution.

As with electrodeposition the yield of self-supporting foils is small.

2.1.3. Electrophoresis⁵²⁻⁵⁸

Finely divided metals or oxides are mixed in a polar organic liquid such as alcohol so as to form a stable suspension. A d.c. potential, between 300 and 800 volts is applied between a suitable electrode immersed in the solution and a conducting backing. The finely suspended particles become ionised and are deposited upon the appropriately

polarised backing. The same disadvantages apply with the plating techniques previously described.

2.1.4 Vapour Deposition⁵⁰⁻⁶⁰

In vapour plating a volatile compound of the element to be deposited is vapourised, then thermally decomposed on to a heated substrate. Several excellent surveys and reviews describing general and specialised aspects of chemical vapour deposition have been published. The most comprehensive work covering many aspects of chemical vapour deposition is the monograph edited by Powell et al⁵⁰. Thermal decomposition has been used by Holmgren et al⁶², Kashy et al⁶³ and Muggleton³⁴ to produce isotopic carbon targets from enriched methyl iodide.

2.1.5 Absorption Gettering⁶⁵⁻⁷³

The relatively ready absorption of hydrogen by some metals has led to the use of this property as a means of target preparation for hydrogen, deuterium and tritium, Zirconium, titanium and erbium are useful metals for this purpose.

2.1.6 Electrospaying

The preparation of nuclear targets by electrospaying has been reported extensively⁷⁴⁻⁸⁶. A suitable compound of the target material is dissolved in a normally non-conducting, volatile liquid such as acetone or methanol. A glass capillary tube is drawn down to form a fine jet of such dimensions that under normal conditions no liquid can escape. A thin wire is inserted into the jet within a few millimeters from the tip; the other end being connected to the positive terminal of an H.T. supply capable of providing 3-15 kV d.c. The receiving backing is placed approximately 10mm below the jet and connected to the negative side of the supply. When a

voltage is applied, liquid is ejected under the influence of the electric field, together with any charged particles of target material as a fine mist, which evaporates, leaving the target material on the backing plate. Large-area targets with thicknesses up to 5mg/cm^2 and areas up to 1000 cm^2 have been prepared by this technique. However, spraying times are excessive (8-10 hrs in some instances) and it is difficult to keep the spraying rate constant.

2.1.7. Mechanical and Miscellaneous Techniques.

Under this heading are grouped adaptations of common mechanical processes such as rolling⁸⁷⁻⁸⁹, compacting^{83,84}, settling powders from a suspension^{83,84,84-101}, use of slurries, painting^{84,102-104} casting¹⁰⁵, polymerising^{106,107} etc.

3. Preparation of Targets by High Vacuum Techniques

As high vacuum evaporation is the most generally used technique to prepare these nuclear targets it will be discussed in greater detail. In its simplest form vacuum evaporation involves heating the material to be deposited to a high temperature in an evacuated chamber and condensing the vapourised material on a suitable substrate.

3.1 Factors Influencing Quality of Evaporated Film

The evaporation and transit of evaporant atoms in vacuum, film nucleation and growth have been documented in the literature¹⁰⁸⁻¹¹⁸ and consequently will not be considered in detail. However, the growth and structure of evaporated films are affected by factors, such as the nature of the substrate and the amount of surface contamination present, as well as the parameters of the deposition process. It is proposed to briefly examine some of these factors.

3.1.1. Role of the Substrate

An increase in the binding force between the substrate and the evaporant atoms usually decreases the surface mobility of these evaporated atoms; this increases the number of initial nuclei and consequently increases the film adhesion¹¹⁹. These factors affect coalescence and the size and structure of the islands during initial growth. Different coefficients of expansion between the substrate and the evaporated film produce stresses in the film when the temperature differs from that during evaporation.

3.1.1.1. Substrate Temperature

The most important effect of heating the substrate in vacuum is to clean the surface, resulting in desorption of contaminants, also an increase in the substrate temperature increases the surface mobility of the condensed atoms and may change the crystalline structure of the film. Interdiffusion between adjacent atoms increases with increasing temperature and may cause films to become more homogeneous. There is also the probability of desorption of evaporant atoms which increases with increasing substrate temperature.

3.1.1.2. Contamination.

The main sources of contamination in evaporated films are contaminants originally on the substrate, residual contaminant gases or vapours in the vacuum system, or gases released from the evaporant source heater material. One result of contamination is the loss of adequate adherence of the evaporated film; substrate contamination is particularly troublesome in this respect¹²⁰.

3.1.1.3. Evaporation Rate.

Since the rate of bombardment by residual gases is constant (for constant residual pressure), higher deposition rates produce films containing smaller amounts of residual gas. In the deposition of a chemically active metal the gas content of the film may be thickness dependent, since the film will be gathering the gas during deposition. The first layers deposited will contain more gas than the final layers. Residual gas content can be minimised by using a shutter prior to evaporating the bulk of the film^{121,122}.

Stresses arising from the growth process are often a function of the thickness and deposition rate. Films that generally curl when detached from the substrate are a qualitative indication of the non-uniformity of stresses with thickness.

During condensation of the film onto a solid substrate the properties of the initial surface are gradually lost as the film thickness increases. Various growth modes are possible^{116,123-125} depending upon the method of formation of the film, the substrate temperature and the physico-chemical properties of both the substrate and the film.

3.1.1.4. Film Growth.

Various growth modes are comprehensively described in the literature¹¹⁶⁻¹²⁵ and can be characterised into four distinct forms, a) island growth, b) layer growth, c) island growth following layer growth, and d) vertical stack growth. Fig. 1. At different temperatures various kinds of growth can occur. For example a given material deposited from the vapour phase onto a substrate may have vertical stack growth at low temperatures, island growth following layer growth at relatively high temperatures and island growth at higher temperatures. Moreover, with suitable annealing

treatment a film can undergo marked structural changes.

The foregoing observations indicate some of the problems encountered in thin film deposition by vacuum evaporation.

3.2 High Vacuum Deposition Apparatus

Rotary pump/oil diffusion pump combinations are the most generally used high vacuum systems for the vacuum evaporation of thin films. They combine the advantages of high speed pumping and quick cycling between individual evaporations, making them suitable for systems using either electron bombardment heating or induction heating of the evaporant. This is useful where high pumping speeds are necessary to minimise electrical breakdowns, caused by a build-up of gases evolved from either the source or adjacent equipment inside the vacuum chamber.

One of the limitations with oil diffusion pumped systems is the relatively poor ultimate vacuum obtainable ($\sim 1 \times 10^{-4}$ Pa) without resorting to the use of metal gaskets and lengthy outgassing procedures. Modern diffusion pumps with appropriate trapping are designed to minimise backstreaming of the liquids used in the pump boiler. Nevertheless, there is always the possibility of hydrocarbons in the system contaminating the target. Systems pumped by vac-ion/sublimar pump combinations do not have the high pumping speeds necessary to prevent the type of electrical breakdown in the region of 1 Pa, as previously mentioned; however, the ultimate vacuum obtainable ($< 1 \times 10^{-5}$ Pa) is a clean one and effectively hydrocarbon free, a requirement for certain experiments. Recently the use of turbomolecular pumped and cryopumped systems have increased in popularity for ultra-clean vacuum systems, however the capital cost of such pumps has limited their use for nuclear target preparation.

Fig. 2 depicts a typical oil-diffusion pumped high vacuum evaporator

used for nuclear targets preparation. The assembly consists of a vacuum chamber, baffle valve and trapped oil-diffusion pump, backed by a rotary pump. Thermocouple gauges are used to measure the backing pressure obtained by the rotary pump, while the high vacuum pressure is measured by a Penning ionisation gauge attached to the high vacuum chamber. With a suitably backed 100ml oil-diffusion pump a pumping speed greater than 400 litres/second and an ultimate pressure of less than 1×10^{-3} Pa can be achieved in the work chamber. Provision is made in the work chamber for ancillary attachments such as rotary seals, shutters, thickness monitoring heads, auxiliary substrate heaters, and water-cooling coils. A typical chamber is 300mm dia x 300mm high, made of stainless steel and surmounted by a borosilicate bell jar. It is not advisable to use brass in the construction as it is liable to become overheated during the evaporation process and release zinc from the brass alloy. For the evaporation of refractory metals such as molybdenum, tantalum, tungsten, uranium, etc. which require temperatures in the region of 3000°C , a water-cooled stainless steel bell jar is used to minimise the danger associated with a glass bell jar imploding.

The availability of higher-energy Van de Graaff and heavy-ion cyclotron accelerators has enabled experiments with heavy ions to be undertaken that were previously impracticable. There is therefore an increasing demand for targets suitable for heavy-ion investigations. To minimise unwanted reactions from contaminating elements it is imperative that the target be of adequate chemical purity. Over twenty years ago it was found that despite continuously evaporating an aluminium target in situ, oxygen contamination of the surface was readily detectable, sufficient to invalidate the experiment being undertaken¹²⁶. It was suggested that in such instances an ambient vacuum of less than 1×10^{-7} Pa may be necessary. More recently Thomas et al¹²⁷ have reported the difficulties experimenters

in their laboratory were having in analysing data, due to the oxygen, carbon and nitrogen impurities in the aluminium and vanadium targets they were using.

The ultra-clean evaporator the author has used to minimise target contamination is shown schematically in Fig. 3. This vacuum system, which is capable of achieving a base pressure $<1 \times 10^{-5}$ Pa was designed to be free of organic contamination, it is evacuated with a 1000 litre/sec sublimator pump and 20 litre/sec sputter pump combination. The apparatus is initially evacuated with a carbon vane/sorption pump combination. Chemically reactive targets such as Li, Ca, Ba, Na, K, etc. may be prepared in such a system. Their subsequent deterioration can be prevented by transferring the target, still under vacuum, from the evaporator either to the accelerator target chamber or to an evacuated storage chamber until required.

A typical transfer lock and evacuated chamber of the type described above has been designed by Worthington et al.¹²⁹. Their chamber accommodates ninety targets at a pressure less than 1×10^{-4} Pa. A similar system used in the author's laboratory operates below 1×10^{-6} Pa and will store targets at this pressure indefinitely. Other vacuum transfer and storage systems are reported in the literature.¹³⁰⁻¹³²

3.2.1. Methods of Heating the Evaporant

Both metals and thermally stable compounds evaporate rapidly in vacuum at temperatures sufficient for their vapour pressures to exceed 1 Pa. The three basic means used in the laboratory for achieving the high temperatures required are: 1) resistance heating; 2) induction heating; and 3) heating by means of electron bombardment. Each method possesses merits and limitations. Secondary means of heating, e.g. by laser beams, imploding wires etc. are not practicable for nuclear target manufacture. In the case

of laser beam heating the apparatus is expensive, of limited application and too specialised for the general nuclear target laboratory. Whereas imploding wire deposition is inexpensive and simple in concept, it is limited to materials that are available in wire form and the deposition is difficult to control.

3.2.1.1. Resistance Heating.

The simplest type of vapour source for evaporation is one in which the material to be volatilised is raised in temperature by electrical resistance heating. The most widely used vapour sources are those with heating elements of refractory metals such as W, Ta or Mo made into filament, or foil boats for supporting the evaporant. Modified forms of these sources are ones in which the evaporant is contained in a crucible made of refractory oxides or graphite and heated by means of a separate element. Source design is widely discussed in standard works on vacuum deposition techniques.¹⁻¹¹

3.2.1.2 Induction Heating

An alternative heating method is by means of eddy current or induction heating whereby the crucible containing the target material is raised to evaporation temperature by inserting it in an R.F. field. The method is useful for evaporating large quantities of material but the evaporation rate is difficult to control, it requires relatively expensive and bulky, R.F. heating apparatus and the radiated R.F. is liable to cause electrical interference with adjacent electronic equipment. However, we have employed this technique for special applications, such as to reduce oxides to the elemental form by heating the oxide in a hydrogen atmosphere.

3.2.1.3. Electron Bombardment Heating.

Some materials required for nuclear targets cannot be evaporated from conventional sources without alloying or reacting chemically with the source, producing film contamination and source destruction. These problems are encountered with boron and silicon, which are both highly reactive at elevated temperatures, and with molten aluminium, nickel, iron and cobalt, all of which readily alloy with refractory metals. One group of vapour sources which has been investigated in order to overcome the foregoing problems and which has found widespread use, particularly in the electronics component industry, are electron bombardment sources. Muggleton¹³³, Arnison¹³⁴, Kobisk¹³⁵, Maier-Komor¹³⁷, Nickel¹³⁸ and Heagney¹³⁹ have all reported preparing nuclear targets by electron bombardment evaporation.

Perhaps the most widely used electron gun design is the magnetically focused gun shown in Fig 4, where the electron source can be placed out of the path of the emitted gas and vapour molecules by using a magnetic field to bend the electron beam through 270°. With this assembly the filament can be protected against sputtering and emission poisoning caused by bombarding positive ions. The main disadvantage of this gun is that the electron beam has a fixed focus and is not readily adjustable; although in more elaborate designs, it is possible to scan the beam across the sample in the form of a raster.

Although electron-bombardment heating is very versatile and almost the universal evaporation technique it is technically complicated and its use is not justified if the more easily controlled alternative of resistance heating is available. The method is of practical importance in cases where very high evaporation temperatures are involved and where the greatest film purity is desired and no suitable support material exists.

3.2.1.4. Deposition by Electrical Discharge.

An electrical discharge has been used to fabricate targets by decomposing a vapourised compound of the element of interest. The main use has been to produce isotopic carbon targets by cracking either enriched acetylene¹⁴⁰, or methane¹⁴¹⁻¹⁴³ onto metallic backings. Both ^{13}C and ^{14}C have been fabricated this way. This technique has been used extensively in the last few years for making carbon stripper foils with extended lifetimes and will be discussed more fully in a following section on carbon stripper foils and target backings.

3.2.1.5 Sputtering

Deposition of thin films by the sputtering process has been known and practised for well over a century, but it is only relatively recently that this method has become a serious competitor to vacuum evaporation. The unpopularity of this method for depositing films has been mainly the tendency of sputtered films to be contaminated. One advantage sputtering offers over most other deposition techniques is the ability to deposit refractory materials relatively simply.

In the basic sputtering process a gaseous discharge is formed between the material to be deposited (the cathode) and a suitable electrode (the anode) placed in the vacuum chamber. Under the influence of an applied electric field, positively charged ions bombard the cathode material causing particles of the material to be ejected and collected on a suitable backing. The reader who is interested in additional information on the broad structure of sputtered films is referred to the many excellent works that exist on the subject.^{1-11,144-146} Eckroad et.al.¹⁴⁷ have used off-the-shelf components to construct a relatively simple sputtering apparatus with which they have produced sputtered films and have also chinned metallic foil

targets to the required thickness. The most serious drawback of self-sustained glow discharge sputtering is the contamination of the deposited film by the gas used to produce the discharge. This phenomenon has been used in the fabrication of nitrogen, hydrogen and oxygen targets by reactive sputtering in one or other of the gases. ^{15}N -enriched targets of Ta^{15}N have been produced by sputtering tantalum in a ^{15}N atmosphere.^{148,149} Similarly titanium nitride targets have been made by this technique.¹⁵⁰

The use of ion-beam sputtering has attained considerable significance in the manufacture of microelectronic devices over the past ten years, and is being used to a progressively greater extent in nuclear target preparation. Films made by this technique can be very pure compared with films made by other techniques.^{151,152}

Focussed ion-beam sputtering differs from thermal deposition evaporation methods in several fundamental ways: the deposition of the target atoms is independent of the vapour pressure of the target material, it is a high vacuum process, is very economical of target material, and substrate heating during deposition is negligible. Targets having high melting points, such as refractory metals, can be produced in a very pure form. Targets can be fabricated from milligram quantities of rare materials.

Von-Ardenne type duoplasmatron ion sources have been adopted for use in nuclear target preparation.¹⁵³⁻¹⁵⁵ Fig 5 shows a diagram of this type of ion source which has three main elements: a heated cathode, an anode and an intermediate electrode negatively biased with respect to the anode. The negative potential difference is established from anode to cathode by a current-regulated power supply. Gas to be ionised is fed to the discharge chamber via a precision leak valve. At a partial pressure of approximately 10Pa, a low-voltage arc discharge strikes in the region between cathode and

anode. This arc ionises the noble gas (argon, krypton or xenon) in the discharge chamber. This plasma is constricted by the axial magnetic field. A high ion density is achieved in the region between the intermediate electrode and anode. The name "duoplasmatron" is derived from this dual plasma construction.

A small anode aperture is centred on the axis of the discharge, the noble-gas ions being extracted from the plasma by an electrode mounted at a high negative potential with respect to the anode. The accelerated ions, with an energy corresponding to the extraction potential, form a well defined diverging beam. By converging the ion beam to a diameter of a few millimetres on to a quantity of about 10 mg of the target material, economy of target material is optimised. Typically, an argon ion beam of 20 keV at 2mA produces a sputtering rate of $20\mu\text{g}\cdot\text{cm}^{-2}\text{ min}^{-1}$ of titanium on to a substrate located 25mm from the sputtering source.

A simple glow discharge ion source has been developed by Nolen et. al.¹⁵⁶ to prepare thin targets of W, Pt, Mo, Zn, Ti and Si by ion beam sputtering with good efficiency for separated isotopes.

To overcome some of the disadvantages associated with duoplasmatron ion sources, i.e. difficult ignition, short filament lifetime and the impossibility of using duoplasmatron sources for reactive sputtering with gases such as O_2 , N_2 , $\text{C}_{1.1}$, etc. a Penning ion source has been developed and used successfully by Wirth et.al^{157,158} in nuclear target preparation.

An improved fine beam ion source has recently been introduced for thin film deposition which utilizes the saddle-field principle first proposed by McIlraith.¹⁵⁹ Some of the unique properties of this source make it quite useful for producing particular types of thin films. The advantages of this type of source over the others previously described include:

- 1) The beam contains a high percentage of neutral particles, thus insulating materials can be sputtered as well as metals.
- 2) The beam produced by the source is approximately 2mm diameter, therefore only small quantities of separated rare isotopes can be used.
- 3) No heated filament is used, consequently the temperature rise of the evaporant is only approximately 10°C , insuring no thermal damage to substrate or release agent, therefore the substrate can be placed close to the source resulting in a high sputtering efficiency.
- 4) The device operates electrostatically; no magnetic field is used.
- 5) Compared with duoplasmatron sources the electrical supplies are relatively simple and inexpensive.
- 6) The source can be made small (approximately 4cm diameter x 4cm long), is simple in construction and is inexpensive to manufacture.
- 7) Although the sputtering rates are low (e.g. typically $40\mu\text{g}/\text{cm}^2/\text{hr}$ for Au and $5.5\mu\text{g}/\text{cm}^2/\text{hr}$ for Ni), once the source has stabilised it can be left virtually unattended for days with only minor adjustments.

A typical Fine Beam Saddle Field Ion Source is shown in Fig 6, while Fig 7 shows the general operation principles of the source. Secondary electrons, liberated from the cathodes by the ubiquitous presence of cosmic rays and stray radioactivity, oscillate between the two cathodes through a central anode under the influence of the applied d.c. field. Argon introduced into the source readily ionises and the positive ions produced are attracted to the cathodes. The emerging beam contains a percentage of neutrals, together with charged ions.

This type of ion source has proved to be ideal for the deposition of small amounts of very expensive isotopes.¹⁶⁰⁻¹⁶²

Table 1 shows typical sputter rates for various materials deposited.

TABLE 1

MATERIAL	Ag	Au	Co	Fe	Mn	Nd	Ni	Os	Pb	Pt	Si	Sn	W	Zn
DEPOSITION RATE ($\mu\text{g}/\text{cm}^2/\text{h}$)	24	44	12	10	9	29	13	8	25	45	4	14	12	10

Deposition parameters: Voltage 4.5kV, Ion current 3mA, Ion Source at 30° to material source, Ion Source to material distance 5cm, substrate immediately above material at a distance of 3 cm.

3.3 Film Thickness Monitoring and Measurement

For obvious reasons it is desirable to be able to monitor film thickness and rate of film growth during the deposition process. Various thin-film monitoring techniques have been described in detail,^{2-6,11,15,163-192} but for the purpose of this paper only those that have been used in nuclear target preparation will be discussed.

3.3.1 Gravimetric Devices.

Gravimetric methods are among the earliest and most convenient to use for determining film thickness. Various balances, such as pivotal, torsion fibre, quartz or tungsten helical spiral and magnetic suspension have found applications as monitors. For more recent developments, especially vacuum applications the reader is referred to Vacuum Microbalance Techniques (Plenum: New York), the proceedings of annual symposia which have appeared

regularly since 1961.

Perhaps the simplest technique for determining evaporated film mass is to weigh a small specimen foil, (for example a thin glass microscope cover slide or a thin foil of nickel or Havar), mounted adjacent to the target substrate, before and after deposition, and thus determine directly the areal density of the deposited film. However, with films of a few $\mu\text{g}/\text{cm}^2$ it may be difficult to obtain adequate accuracy, because of gas desorption by the monitor foil. The problem is further aggravated when handling materials that rapidly oxidise, or are deliquescent. To minimise the weight loss problems encountered in the single technique referred to, various workers have used sensitive micro-balances within the vacuum system itself and weighed the target material as it was deposited.¹⁷²⁻¹⁷⁶

3.3.2. Optical Methods.

A simple rule-of-thumb method to determine film thickness is to view the incandescent evaporation source directly through a transparent glass slide mounted inside the vacuum chamber; when the evaporated film obscures the filament, as observed by the eye, the film will be a predetermined thickness. Though various materials will have different thicknesses at which they become opaque, these thicknesses can be accurately determined. For a uniform source a range of thicknesses can be measured, by using the formulae published in standard references on thin film deposition.¹⁻¹¹

This optical method is only applicable to materials that are transparent in the range of thicknesses required. As different materials have different evaporation temperatures, it is difficult to determine accurately the exact moment a film becomes truly opaque; considerable experience is necessary to make this technique produce meaningful results.

More precise optical methods have been reported¹⁶⁶⁻¹⁶⁹ but their

application to nuclear target fabrication is limited by the small useful range of thicknesses that can be measured. It is also necessary to calibrate the device for each target material used.

Maier-Komor¹⁸³ has used a quartz-spectrophotometer to measure the transmission characteristics of carbon foils in the wavelength range of 20000-25000 Å. The transmittance, as a function of carbon foil thickness was measured at different wavelengths, and the absorption coefficients and reflectivities of the foils calculated. Knowing these optical constants for one wavelength it was relatively easy to calculate the absolute thickness of a carbon foil by measuring its transmittance for this monochromatic light. It is claimed that it is possible to distinguish between a 5 $\mu\text{g}/\text{cm}^2$ carbon foil and one of 4.98 $\mu\text{g}/\text{cm}^2$.

A simple densitometer has been developed by the author for measuring the thickness of thin carbon backings and stripper foils. The apparatus consists of an inexpensive photovoltaic cell, a light source and microammeter, mounted in a light-tight box. A carbon foil to be measured is inserted between the light source and the photocell, the percentage reduction in light transmission being noted. This reduction in transmitted light will be proportional to the foil thickness. The instrument was calibrated against known samples whose thicknesses had been accurately determined by Rutherford scattering. Although not as accurate as the design previously mentioned,¹⁸³ this instrument was inexpensive to construct, is easy to use, readings can be rapidly taken, and the results are quite adequate for all practical purposes.

3.3.3 Moving Coil Microbalances

The use of a moving-coil meter movement has been reported by various workers in the field of thin film technology.¹⁷⁰⁻¹⁷⁶ This type of

instrument is capable of measuring both rate of deposition and the final weight of the deposit by the displacement of a vane attached to the meter movement. The usual method of unbalanced position detection, used with meter-type microbalances, employs an optical system with photocells (or photosensitive semiconductor diodes). An imbalance is detected by photodetectors mounted each side of the centre-pivoted pointer, amplified, and the resulting d.c. signal applied to the meter coil to restore balance. The voltage applied to the coil is a measure of the torque causing imbalance, which in turn can be used to calibrate the instrument in terms of mass of material condensed on the vane. After evaluating a moving coil microbalance of the type described, the author concluded that the instrument's usefulness was inadequate for routine thin-film nuclear target work. The device was far too sensitive to vibration and not robust enough for practical use.

3.3.4. Quartz-Crystal Monitors

Perhaps the most widely used device for measuring film thickness is the quartz-crystal thickness monitor which combines robustness with sensitivity and ease of operation. The sensitive element of this instrument is a quartz crystal, oscillating in the thickness-shear mode. The characteristic frequency of the crystal is a function of the thickness of the quartz plate and on the frequency constant of the quartz. If the effective plate thickness is increased by depositing a film upon it a frequency shift results. Sauerbrey¹⁰⁴ showed quantitatively that the shift in resonant frequency of the crystal is linearly proportional to the mass of the material deposited (at least for thin films).

The choice of resonant frequency depends upon the characteristics of the particular installation and on the purpose of the investigation: the

thinner the films under investigations the higher the desired sensitivity and therefore the higher the change in frequency should be. For a large thickness range the deviation from the linear region should occur as late as possible and consequently thicker crystals with a low frequency change should be selected. In practice the best compromise crystal resonant frequency is about 5 MHz

Beside mass changes other factors can also lead to frequency drift. Damping of the crystal varies with changes in its surrounding medium, i.e. initial evacuation of the evaporator containing the crystal monitor. Small pressure variations have no influence, so if all measurements are undertaken in vacuum the damping will be negligibly small. By suitable construction of the crystal holder frequency shifts due to mechanical shocks (i.e. activating a shutter, etc.) can be eliminated. The most difficult erroneous frequency shifts to eliminate are those due to variation in crystal temperature. It is therefore important to select crystals with a low frequency-temperature coefficient. AT-cut crystals, sliced at an angle of $35^{\circ} 20'$ relative to the natural crystal axis and having a near zero temperature coefficient from -5°C to $+55^{\circ}\text{C}$ are normally used. The heat from evaporation sources is often appreciable, resulting in a considerable increase in crystal temperature during deposition and can exceed the range of the temperature coefficient, therefore cooling of the crystal is necessary¹⁷⁵. The temperature drift can be further minimised by mounting a second, shielded, compensating crystal in close proximity and water cooling the combined crystal housing¹⁷⁷.

Quartz crystals are possibly the most versatile thin film monitoring devices and are marketed by the majority of high vacuum equipment manufacturers. Many excellent articles have been published on the design and uses of this type of instrument.^{183-185, 193-200} Their use for thin film

nuclear target fabrication has been extensively reported.²⁰¹⁻²⁰³

3.3.5 Thickness Measurements Using Charged Particles.

Apart from gravimetric techniques, all the monitors described are secondary devices and require calibration against known standards. From previous observations it will be realised that the condensation or sticking coefficient of vacuum-deposited films depends upon many parameters, such as substrate material, substrate temperature, surface cleanliness, surface texture, surface electronic states, crystallographic form of the substrate, etc. Therefore the characteristics of a thin film deposited on to a monitoring substrate can differ greatly from the film deposited onto the substrate material used as the target. Few of the methods described are easily adapted to both thickness and uniformity measurements.

The loss of energy of alpha particles as they pass through foils has been used by many investigators to determine target thickness and uniformity.^{184-192,204,205} One method that has been used by the author is the loss of energy of charged particles as they traverse the target, as described by Thompson.¹⁸² Alpha particles of a known energy pass through the target to be measured; their mean energy is then measured upon emergence from the target. This permits the determination of the energy lost in the target, from which the target thickness can be calculated using published stopping-power tables.²⁰⁶⁻²⁰⁸ The following limitation should be observed:

- (a) The lower limit to the thickness measurable is determined by the accuracy to which the energy of the alpha peak can be measured;
- (b) The upper thickness limit is $\sim 5 \text{ mg/cm}^2$ for most materials;
- (c) The final limitation is that the method only works for targets of known composition and preferably for targets of pure elements.

However, the technique described is in general quite accurate and useful for target thickness measurements.

4. Thin Target Preparation

The types of targets used in low energy nuclear physics research fall naturally into two groups, a) Self-supporting targets, which as the name implies requires no backing membrane to give mechanical support; b) backed targets, where the element or compound to be investigated is deposited on to, and supported by, a thin membrane of a stronger material. The most commonly used backing materials are listed in TABLE 2 where their individual merits are discussed.

TABLE 2 Backing Materials

Material	Method of preparation	Chemical inertness
Aluminium	Commercially available with thickness down to $180 \mu\text{g cm}^{-2}$. Evaporated Al, practical unsupported thickness $20 \mu\text{g cm}^{-2}$.	Soluble in alkalis HCl , H_2SO_4 . Insoluble in HNO_3 , acetic acid.
Aluminium oxide	Prepared anodically by immersion of Al foil in an electrolytic solution, e.g. ammonium citrate + citric acid in double-distilled water. $\text{PH} \sim 5.0$. Exposed Al foil removed with dilute HCl leaving self-supporting Al_2O_3 film. Minimum self-supporting thickness about $25 \mu\text{g cm}^{-2}$. 208-211	Very slight solubility in acids and alkalis.
Carbon	(a) Evaporated from carbon arc on to a glass microscope slide coated with a water-soluble release agent. Carbon film removed by immersing slide in water. (b) Evaporated, using electron gun, on to glass-coated slide as described later. Minimum self-supporting thickness $2 \mu\text{g cm}^{-2}$.	Isoluble in acids and alkalis.

Collodion ($C_{12}H_{17}N_3O_{18}$)x	Solution of collodion in amyl acetate. Film prepared by dipping glass slide into solution and slowly withdrawing it. When solvent has evaporated, film floats on water as for carbon. Temperature-sensitive.	Insoluble in acids and alkalis.
Copper	Commercially available with thickness down to 3.0 mg cm^{-2}. Useful as a backing for electrolytically deposited films. Used as a backing for the preparation of self-supporting Fe, Ni and Cr targets. Target material deposited on to Cu foil which is then dissolved in trichloroacetic acid+ammonia solution ($30\text{g CCl}_3\text{COOH}+50 \text{ ml NH}_4\text{OH}+500 \text{ ml distilled water}$).	Soluble in HNO_3, hot H_2SO_4, very slightly soluble in HCl.
Formvar	Polyvinyl formal dissolved in ethylene dichloride or chloroform. (a) Film can be prepared by placing drop of solution on to surface of water. Surface tension forces cause droplets to spread and the solvent evaporates, leaving thin film on water surface. Film may be picked up with metal frame. (b) Prepare film as for collodion.	Insoluble in acids and alkalis.
Gold	(a) Commercially available with thicknesses down to $200 \mu\text{g cm}^{-2}$ (bookbinders' gold leaf). Fragile, fibrous and difficult to handle. (b) Evaporated on to glass microscope slide previously coated with a water-soluble release agent. Slide slowly immersed in water bath and floating gold film picked up on suitable target frame. Minimum thickness about $100 \mu\text{g cm}^{-2}$.	Soluble in KCN, aqua regia.

Mylar (polyethylene terephthalate) (C₁₀H₈O₄)	Commercially available polyester compound. Temperature-sensitive.	Insoluble in acids and alkalis.
Nickel	Commercially available with thicknesses down to 4 mg cm⁻². Useful as backing for electrodeposited films. Evaporated backings thicker than 50 μg cm⁻².	Soluble in HNO₃, slightly soluble in HCl, H₂SO₄.
Nylon	Nylon chips are dissolved in isobutyl alcohol by careful boiling in a double boiler. Films are prepared as for Formvar. Water bath is made neutral by tri-sodium phosphate, allowing better spreading of floating nylon film. Minimum thickness 10 μg.cm⁻².²¹²	Insoluble in acids.
VYNS	VYNS dissolved in cyclohexanane to form a saturated solution at 50-60° C and then diluted with cyclohexanone to give a one-third saturated stock solution. A few drops of the solution are pipetted into the corner of a rectangular water bath. On pulling the VYNS drop across the surface of the bath the thickness of the spreading film is readily controlled. When the solvent dries off, the VYNS film is lifted from the water surface with a wire loop or target frame. Minimum thick- ness 1 μg cm⁻². Temperature-sensitive.	Insoluble in acids and alkalis.
$\left[\begin{array}{c} -\text{CH}_2-\text{CH}- \\ \\ \text{Cl} \end{array} \right]_n \text{O.CO.CH}_3$		
Zapon	Zapon lacquer is diluted in amyl acetate and films prepared as for VYNS. Characteristics similar to collodion. Temperature-sensitive.	Insoluble in acids.

Thin film targets are vital to the majority of accelerator experiments in nuclear structure investigations. In fact, in many instances the preparation of such targets is crucial to the success of the experiment. Backed targets have thicker, usually metallic backings on to which the target materials are deposited. However, the majority of those targets used in low energy nuclear physics experiments are either self-supporting or condensed on to a thin membrane such as metal, plastic or thin carbon. The choice of backing material is determined by the experiment being undertaken.

4.1 Polycrystalline, Self-Supporting Targets, and Targets on Thin Film Backings.

Depending upon the material, the thickness required and the chemical stability of the material, the target can be deposited either on to a suitable substrate and subsequently removed, or on to a thin carbon backing. The requirements for these backings are that they should be strong mechanically, thermally stable under beam bombardment, and should not interfere with the experiment under investigation. Carbon frequently fulfils these requirements and has the additional advantage that it is structure-free ^{213,214} is relatively simple to produce and has good vacuum properties.

4.1.1 A General Technique for Producing Self-Supporting Targets.

The steps used by the author for preparing self-supporting films are illustrated in Fig 8 and Fig 9. A polished substrate (usually a borosilicate glass microscope slide) is thoroughly cleaned by boiling in a suitable detergent such as Haemo-Sol (Haemo-Sol Inc., Baltimore, USA), followed by successive rinsings in de-ionised water. The cleaned substrate is then coated with a water-soluble film, by dipping the cleaned substrate

into a concentrated detergent solution such as RBS25 (Chemical Products, R. Borhghgraef, Belgium), or Teepol (Shell Ltd.), or into a glucose solution. Alternatively, a water-soluble compound may be vacuum deposited on to the substrate. Target material is now deposited on to the substrate by vacuum deposition. On removal from the vacuum evaporator the slide is lowered at an angle of approximately 45° , with target film upmost, into a bath of de-ionised water. The water dissolves the soluble interface between film and substrate allowing the film to float on the surface of the water. A suitable target frame is lowered beneath the floating film and carefully raised vertically with the film adhering to the frame. The target is allowed to dry and stored in a desiccator until ready for use.

4.1.2 Release Agents Used in the Preparation of Self-Supporting Targets

The choice of a water-soluble release agent as the interface between substrate and deposited target material will be dictated by: the target material required, the temperature stability of the release agent, the experiment being undertaken (e.g. the soluble release agent may leave elemental traces that can interfere with the experiment in progress), and the crystal structure and lattice constant of the material being deposited. In vacuum deposition, the choice of surface can make the difference between depositing a uniform, cohesive film and condensing no film at all. By selecting a suitable release agent, stresses introduced into a condensing film, with subsequent shattering of the film when removed from the substrate, can be minimised. Ramsay²¹⁵ suggests that the crystal structure and lattice constant of the material which is to be deposited must be matched with a chosen release agent. The most commonly used release agents are listed in Table 3.

TABLE 3. RELEASE AGENTS

RELEASE AGENT	FORMULAE	CRYSTALLINE FORM	MOL. WT.	DENSITY	MP(°C)	BP(°C)	SOLUBILITY IN GMS PER 100cc.		
							COLD WATER	HOT WATER	OTHER SOLVENTS
BARIUM CHLORIDE	BaCl ₂	CUB	208.25	3.856	925	1560	37.5	- 59	ss HCl, HNO ₃ vs ls al.
BARIUM IODIDE	BaI ₂	RHOMB	391.15	5.15	740	-	170	-	al 77 ²⁰
BORIC ACID	H ₃ BO ₃	TRIC	61.83	1.435	169	300	6.35	27.6	glc, eth, al.
BORON OXIDE	B ₂ O ₃	RHOMB	69.62	2.46	460	cal 860	sl.s	s	
CAESIUM BROMIDE	CsBr	CUB	212.81	4.44	636	1300	124	vs.	s.a.
CAESIUM IODIDE	CsI	RHOMB	259.81	4.51	621	1280	44	160	s.al.
CALCIUM IODIDE	CaI ₂	HEX	293.89	3.956	740	1100	209	426	s.al, acet.
COPPER	Cu	CUB	63.546	8.92	1083	2595	-	-	a. trichloro-acetic + ammonia
MAGNESIUM CHLORIDE	MgCl ₂	HEX	95.22	2.3	708	1412	54.25	72.7	al.
NICKEL CHLORIDE	NiCl ₂	HEX	129.62	3.55	1001	973 _{1, 2b}	64.2	87.6	al, NH ₄ OH
POTASSIUM CHLORIDE	KCl	CUB	74.56	1.984	776	1500	34.7	56.7	s.sl al, s eth, glyc, alk.
POTASSIUM IODIDE	KI	CUB	166.01	3.13	686	1330	127.5	208	al, acet, sl eth & NH ₃
SODIUM CHLORIDE	NaCl	CUB	58.44	2.165	801	1413	35.7	39.12	sl s al, liq NH ₃ s glyc.
SODIUM IODIDE	NaI	CUB	149.88	3.667	651	1304	184	302	al, acet, s. glyc.

TABLE 3 (cont'd)

RELEASE AGENT	FORMULAE	CRYSTALLINE FORM	MOL. WT.	DENSITY	MP(°C)	BP(°C)	SOLUBILITY IN GMS PER 100cc.		
							COLD WATER	HOT WATER	OTHER SOLVENTS
ZINC CHLORIDE	ZnCl_2	HEX	138.28	2.91	283	732	432	615	al. v.s. eth.
ALANINE ^(a)	$\text{C}_3\text{H}_7\text{NO}_2$	ORH	89.10	-	295	258 _{sub}	v	-	al.
BEDACRYL	-	-	-	-	-	-	-	-	xyl.
BETAINE ^(b) MONOHYDRATE	$\text{C}_5\text{H}_{11}\text{NO}_2 \cdot \text{H}_2\text{O}$	-	117.15	-	293 _d	-	v	-	al.
FORMVAR ^(c) 15/95E	$\text{C}_5\text{H}_8\text{O}_2$	-	-	1.2	110	-	i	i	chloro, toluene,
GLUCOSE-D	$\text{CH}_2\text{OHCHCHOH}_4\text{O}$	-	180.16	-	150	-	s	vs	al ^h , ace.
HEXADECYLAMINE ^(d) (ACETYLAMINE)	$[(\text{CH}_3\text{CH}_2)_{15}\text{NH}_2]$	-	241.46	-	-	-	i	i	hexane
RBS 25 ^(e)	-	-	-	-	-	-	v	v	-
SUCROSE	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	-	342.3	1.58	185	-	s	v	s. al.
TEEPOL ^(f) 610	SODIUM SEC ALKYL SULPHATE	-	-	-	-	-	v	-	-

(a) ALANINE Koch-Light Laboratories Ltd, England.

(b) BETAINE Koch-Light Laboratories Ltd., England

(c) FORMVAR 15/95E Shawinigan Resins Corp., U.S.A.

(d) HEXADECYLAMINE Maxman³⁸³

(e) RBS 25 R. Borhghraef, Belgium

(f) TEEPOL 610 Shell Chemical Co.

It is useful here to recapitulate the formation process of thin films, as detailed previously. The process commences with the arrival of a single vapour molecule at the substrate surface. It may condense, migrate across the surface, or re-evaporate. In migration both collision and recombination can occur. With subsequent loss of energy, stable islands are formed at preferred growth points or adsorption sites, in the process of nucleation. With the impinging of more molecules, growth occurs until the areas start to coalesce. This somewhat oversimplified summary serves to illustrate how the substrate directly affects the formation of a film. The condensing molecules must have an adequate number of adsorption sites and these sites must be spaced at an interval which will allow coalescence and inhibit re-evaporation. For epitaxial film growth, in which atoms deposited from the vapour on to a solid surface take up and continue the original crystal structure, the matching of the lattice spacings is critical. In polycrystalline films the lattice constants may not be so critical but it is the determining factor for both the number of adsorption sites and the distance between discrete sites. The critical area for causing stress and creating crystal defects is at the substrate film interface. To take an example, cobalt: in the solid form this is normally hexagonal but can be induced to grow at room temperature in a cubic arrangement on a substrate, such as copper, which has a cubic crystal structure. Below 20 angstroms thick the lattice is strained to exactly match the substrate. Above 20 angstroms dislocations are generated to accommodate part of the difference between the cobalt and copper lattices. If such a cobalt film were removed from its copper substrate it would disintegrate from internal stress⁸. Ideally, in order to obtain a uniform stress free film, substrates with the same crystalline structure as the depositing material should be used; this is particularly true for a single crystal film. Fortunately this is not

always a stringent requirement, for reasons which are not fully understood²¹⁵. Many elements can be satisfactorily formed on an amorphous release agent such as Teepol, RBS25, Betaine, etc. A comprehensive study of various parting agents has been undertaken by Braski²¹⁶.

Abele et al.²¹⁷ made a detailed investigation of the effects of release agents on the uniformity of thin, self-supporting targets and concluded that a release agent, such as Teepol, should be used in preference to agents with a more crystalline structure. Their interpretation was that, with a crystalline release agent, a number of evaporated atoms strike the release agent at an angle in a region off-centre of the evaporation source; therefore some crystallite replicas partially shade any "valleys" on the foil surfaces and there is less thickness of foil in these valleys. This observation was confirmed independently by Muggleton and Under²¹⁸ when investigating how the method of target preparation modified the charge state distribution of bromine ions as they passed through a thin carbon foil.

During an investigation into the pre-equilibrium charge states of heavy-ions in thin carbon targets²¹⁹ it was observed that there were irregularities in their initial charge state measurements, due presumably to the uneven topography of the carbon film surface. Further investigations indicated that this unevenness clearly related to the release agent used during the preparation of the targets. Sodium chloride was chosen as a release agent because, from previous experience, an evaporated chloride contained fewer contaminants and gave a much smoother, more uniform surface than did detergent type release agents (e.g. RBS25, Teepol, Betaine, etc.)

4.2 Targets for Heavy-Ion Bombardment

With the introduction of Van de Graaff accelerators capable of

producing ion beams heavier than mass-16 it is now possible to undertake the type of heavy-ion experiments that were not feasible a few years ago. Many of the same target preparation techniques used to fabricate samples to be bombarded by light-ions (protons, deuterons, alphas, etc.) are used in making targets for heavy-ion interaction studies. However, the large energy losses and straggling that take place when high energy heavy-ion beams penetrate targets demand more stringent control over target parameters. Factors such as target thickness (atoms per square centimetre), thickness uniformity, chemical purity, compound form, substrate material and target flatness are all of concern in light-ion experiments, but they assume far more importance in heavy-ion interactions studies. The unique characteristics of heavy-ion targets have been discussed in detail.²²⁰⁻²²³

4.2.1. Target Thickness and Uniformity

Both thick and thin targets are used in heavy-ion research. Thin targets ($\leq 1 \text{ mg/cm}^2$) are frequently used to minimise energy losses in the target by the incident beam as well as by the resultant reaction products. As the ion beam passes through the target, particle energy is lost by direct collision mechanisms and straggling occurs.

The amount of energy lost increases as the square of the projectile atomic number.

For heavy-ions the direct collision effects tend to become more important than straggling effects. The energy loss effects dominate since they are proportional to Z_{Proj}^2 , whereas the straggling effects are proportional to the first power of Z_{Proj} ²²⁰. For heavy-ion research, target non-uniformity becomes an important consideration if satisfactory resolution of the energy states of reaction products is to be achieved. Energy losses will vary with the non-linearities in the target, heavier ions losing more

energy per unit thickness of target than lighter ones.

When using very heavy ion beams, excellent target uniformity is necessary if accurate measurements are to be achieved. A few typical examples will indicate the problems associated with target non-uniformities. In Fig. 10 is shown a grossly non-uniform target with a step in the centre of it. This example illustrates how target non-uniformity increases the energy spread of the transmitted beam. The beam, passing through the thinner section (shown as dashed lines), has a smaller energy loss than has that portion of the beam passing through the thicker section (dotted lines). The effect of combining the two smaller peaks into the larger one (solid curve) is to increase the energy spread of the beam.

Three other forms of target non-uniformity encountered in practice are illustrated in Fig. 11. The target depicted in a) is a wedge-shape and can occur if the substrate is situated too near the evaporation source during target preparation. In b) the non-uniformity takes the form of microscopic roughness on both surfaces and is encountered, for example, when a unsuitable release agent is used.

A third type is a void in the target c) or a cavity caused by a material that has been deposited onto a backing having an isolated cavity between the deposited film and the backing, or where a portion of the target has become detached from the backing.

4.3 Target Lifetime

Since the cross-section for a particular reaction of interest may be very small, the target may be required to endure long-term beam bombardment to obtain statistically significant data. Several effects occur within the solid target under heavy-ion bombardment which tend to shorten target lifetimes: these effects will now be briefly discussed.

4.3.1. Thermal Effects

The energy per second, or power, deposited in a solid target by a penetrating beam of heavy-ions can cause intense heating of the target foil resulting in a limited lifetime due to the evaporation of target atoms in the vacuum, even at target temperatures below the melting point.²²¹⁻²²³ It has been assumed that the target foil will be destroyed if one third of the target atoms are evaporated. Therefore in designing targets, careful consideration must be given to the required beam intensity, target thickness and the power dissipation limit of the target. A careful balance must be maintained between the parameters noted and the time required to achieve significant results without thermally destroying the target.

Several investigators have endeavoured to maintain target temperatures by utilising the following techniques:

- 1) Design of target holders which provide gas cooling^{224,225}
- 2) Use of targets coated with a good thermal conductor²²⁶
- 3) Use of a rotating target mount to limit beam dwell time^{225,226}
- 4) Ion beam scanning on a stationary target²²²

However, all these approaches to minimise thermal effects place additional constraints on the targets, e.g. target size may increase with gas cooling, and increased mechanical strength is frequently required, all of which influence the choice of methods used to prepare such samples.

4.3.2. Target Damage Caused by Heavy-Ion Bombardment

The process that describes the damage caused by the bombardment of targets with heavy-ions has been comprehensively dealt with by Schilling²²⁷, Yntema and Nickel²²¹ and Schulson²²⁸, it will be therefore sufficient to summarise the process in the following two steps:

- 1) Heavy-ions colliding with a target cause a transfer of kinetic energy in approximately 10^{-17} second to one target atom. The target atom receiving the input energy is called a "primary-knock-on-atom" (PKA). If the transfer of energy is sufficiently large ($\sim 25\text{eV}$), the primary-knock-on-atom will be dislodged from the lattice and a vacancy-interstitial pair (Frenkel Defect) will be formed.²²⁹ The number of primary-knock-on-atoms formed is dependent on the heavy-ion flux and the differential cross section.
- 2) Primary-knock-on-atoms subsequently collide with other atoms and distribute their energy throughout the target lattice until their residual energy is too low to cause further lattice displacements.

The second process is called a displacement or collision cascade which proceeds until the energy of the primary-knock-on-atom decreases to $\sim 25\text{ eV}$. The collision-cascade process lasts ~ 1 to 3×10^{-12} sec, with the remaining energy dissipated in about 10^{-11} sec. The cascade region consists of undisplaced atoms, lattice vacancies and interstitial atoms arranged such that the vacancies are concentrated in a "depleted zone" and the interstitial nuclides are in the periphery of that zone.

A single primary-knock-on-atom generally leaves a vacancy in the target lattice and, if the moving interstitial nuclide has sufficient energy to escape the vacancy, direct displacement occurs. However, if the interstitial atom has insufficient energy, a replacement occurs. For heavy-ion collisions, replacement collisions outnumber displacement collisions.

To determine the effect of radiation damage on the useful target lifetime, a partial lifetime resulting from nucleus-nucleus interactions must be established, i.e. the time required to displace one-half the target

atoms from the original lattice²²¹.

It has been estimated that if the target temperature is maintained at a few hundred degrees (as would be expected), a large fraction of the damage sites would be removed by annealing and the partial lifetime increased by one or two orders of magnitude²²⁹. It appears that the effect of radiation on target lifetime is minimal compared with thermal effects; however radiation damage might become the primary consideration for targets where the thermal effects are minimised through cooling.²³⁰

4.3.3. Sputtering of Target Material

A special kind of radiation damage is sputtering of target atoms from the surface (spallation). Sputtering is assumed to result from cascades of atomic collisions. When an impinging ion collides with a target atom, the recoiling atom, or primary-knock-on-atom, has sufficient energy to produce other recoil atoms in subsequent collisions. This series of collisions can result in ejection of atoms from the target surface into the surrounding vacuum. The major portion of atom sputtering, caused by recoil atoms, occurs within only a few atomic layers of the target surface because of the relatively low energy of the recoil atoms. Maor²²⁹ concluded from his studies of the effects of heavy-ion radiation damage on target lifetimes, that the damage caused by sputtering is negligible compared to radiation damage. Most of the energy loss is consumed in generating thermal effects and radiation damage, with only small amounts being dissipated in the sputtering process. Sputtering of target material can be minimised by coating the target layer with non-target material. This technique was reported by Folger and Nickel²³¹ who used 5.5 mg/cm² gold targets coated with 60 µg/cm² carbon layers which reduced the gold sputtering rates by a factor of approximately 100 during bombardment with 80 MeV iodine ions.

Sputtering yield data for several materials under heavy-ion bombardment is reported by Anderson and Bay²³².

4.4 Target Purity

In determining the appropriate target design and composition, one of the major considerations in both light-ion or heavy-ion experiments is the unwanted reactions which can either mask or reduce detection of the desired events. For heavy-ion experiments, low-Z contaminants are particularly troublesome since they generate significant yields that are not easily subtracted from the total target yields. On the other hand, high-Z materials produce a "smooth" background that can be more easily subtracted. It has been known since 1928^{233,234} that as the atomic number of target nuclei decreases there is a decrease in the coulomb barrier energy and a corresponding larger cross-section for nuclear reactions with the incident beam. The energy loss of the heavy-ion in the target also increases with decreasing atomic number of the target nuclide. For all these reasons it is clear that low-Z impurities in the target should be eliminated. Target materials are used in their elemental form whenever possible to avoid the presence of extraneous low-Z nuclides. Usually a compromise must be made because other considerations such as chemical reactivity, material availability, and material cost may limit the target material to some compound form.

4.5. Doppler Shift Effects

Lifetime measurements of excited states of nuclei formed by heavy-ion collisions create special problems in the preparation of suitable targets. Two basic measurement techniques are employed utilising the Doppler shift phenomenon to determine nuclear events having periods of 10^{-15} to 10^{-9}

seconds. They are the Recoil-Distance Plunger Method (RDM) and the Doppler Shift Attenuation Method (DSAM). Both of these methods are described in detail.²³⁵⁻²³⁷

Targets used in each of these experimental techniques have specific characteristics which will be briefly described below.

4.5.1 Targets for the Recoil-Distance Plunger Method (RDM).

In experiments using the recoil-distance plunger method, excited nuclei recoil from a thin target into a vacuum and are stopped by a plunger foil situated a small distance from the target. The targets used for the recoil-distance plunger method must be thin, flat and plane parallel to the plunger foil. Recoiling nuclei must escape the target, and the distance between the target and plunger foil must be determined to a high degree of accuracy. Deviation in flatness of the target will cause errors in distance determination and may prevent some nuclei escaping from the target. Also, consideration must be given to beam heating effects in the plunger foil. It has been observed²³³ that thin Gd and Cm targets deposited on Mo backings failed mechanically. It was claimed that the failure was due to tensile stresses set-up between the target and backing by the temperature induced from the irradiating beam. It is sometimes necessary to minimise plunger foil thickness to allow maximum transmission of the incident beam and yet allow enough thickness to stop recoil target nuclei. The plunger foil is normally the determining factor governing the maximum beam intensity that may be used.

4.5.2 Targets for the Doppler Shift

Attenuation Method (DSAM)

Targets for Doppler Shift Attenuation Method measurements frequently

consist of thin deposits of the target atoms on relatively thick substrates made from heavy elements. The thin target deposits must be uniform and void free, adherently bonded to the substrate material. Voids in the interface between target film and substrate material can result in lifetime measurement inaccuracies. The recoiling ions that pass through voids (microcavities) during part of the slowing down process give rise to lower attenuation of the Doppler broadened lineshape. Evidence for void formation has been presented by Samoilov et al. for oxides of vanadium and niobium as well as many other compounds.²³⁸ As bulk density values are used in stopping power calculations, voids will cause errors in measurements. Generally, the targets must be free from contaminants because of inherent changes in density and stopping power which these impurity nuclides may cause. Other areas of concern include losses of material due to sputtering or vaporisation and the possibility of separation between the target layer and the substrate, known as blistering²³⁹. In the author's laboratory, lead used as a target stopper material melted on exposure to the heavy-ion beam and had to be replaced with tantalum. A similar Doppler Shift Attenuation experiment undertaken in the same laboratory, indicated either blister or void formation at the interface between the Fe and Cu layers in the Os-Fe-Cu sandwich target being used.

5. Carbon Backings and Stripper Foils

5.1 Introduction

Extensive use of carbon films is made in nuclear structure research for thin carbon backings and for stripper foils. The contents of this section will be concerned with this aspect of thin-film nuclear target development.

5.2 Carbon Backings and Stripper Foils in General

Depending upon the material, the thickness required and the chemical stability of the material, the target can be deposited either on to a suitable substrate and subsequently removed, or on to a thin backing. The requirements for these backings are that they should be strong mechanically, thermally stable under beam bombardment, and should not interfere with the experiment under investigation. Carbon frequently fulfils these requirements and has the additional advantages that it is structure-free and has good vacuum properties. It is also relatively simple to produce.

Thin ($< 5 \mu\text{g}/\text{cm}^2$) self-supporting carbon foils are used extensively as electron strippers in heavy-ion accelerators, especially in tandems where the negative ion beam is converted in the high voltage terminal to a multiply-charged positive beam, by means of a charge-exchange process. Both gaseous and solid strippers have been used. However, for heavy ions the mean charge state obtainable with gas strippers is considerably lower than that obtained with foil strippers. Consequently the energy of the beam from the tandem is much higher if a foil stripper is used. The energy for very heavy ions is approximately doubled. For example, in an electrostatic accelerator with a terminal voltage of 15 MV, the average charge state in a uranium beam stripped by N_2 gas is +5 and that in one stripped by carbon foils is +11. That is, the energy to which the stripped ion will be accelerated changes from 90 MeV to 180 MeV if a carbon foil can be used instead of a gas stripper²⁴⁰.

Compared with other possible foil materials, carbon has the advantage of being mechanically stable at very small thicknesses and has a low atomic number which reduces multiple scattering of the ions passing through the foil.

As the use of thin carbon foils is so extensive their preparation has been widely documented.²⁴¹⁻²⁴⁵

5.3 Stripper Foils

The main interest of the experimentalist is in the quantity of monoenergetic heavy ions per stripper foil he has available for his experiment. Therefore, he is primarily interested in the quality of the stripper foil and not so much in its lifetime. The main criterion of foil quality is given by its thickness: the stripper foil should not be thicker than necessary to achieve the equilibrium charge state distribution, otherwise the quality of the beam is unnecessarily worsened. It has been demonstrated that for tandem accelerators with a terminal voltage below 15 MV the foil thickness should be less than $5 \mu\text{g}/\text{cm}^2$. The intensity of the most probable charge state decreases very slowly for thinner stripper foils, therefore the lower straggling in a thinner foil results in higher transmission.²⁶⁶ It was shown that with heavy-ions up to nickel a $3 \mu\text{g}/\text{cm}^2$ stripper foil increased the emittance by 50% compared to a $5 \mu\text{g}/\text{cm}^2$ foil. Obviously there must be a practical limit to the foil thickness and consequently a compromise is made between minimum thickness and mechanical strength. Carbon stripper foils, with thicknesses of $2-3 \mu\text{g}/\text{cm}^2$, are routinely produced in the author's laboratory by the technique described in the following section.

5.4 Techniques for Producing Carbon Stripper Foils

There are two main techniques currently used for making carbon stripper foils:

- 1) evaporation/condensation processes in high vacuum, whereby either an electric arc is struck between two carbon electrodes, or conduction heating of the electrodes takes place. Alternatively, an electron gun is used to sublime a pellet of pressed carbon powder.
- 2) decomposition of organic compounds such as methyl iodide or ethylene by means of a d.c. glow discharge.

5.4.1 Evaporation Condensation Process in High Vacuum.

The techniques employed for the evaporation, or more correctly sublimation, of carbon are described in the following sections.

5.4.1.1. Conduction Heating.

The conduction method, first described by Bradley²⁴¹, is the most widely used technique although many experimenters have modified the original concept, all report using similar apparatus.^{242,244,287,288}. The technique used in the author's laboratory to produce carbon stripper foils is a modification of the technique described in Ref. 244. Briefly, two 1/4" diameter, high purity graphite rods, having pointed ends and clamped in water-cooled supports, are held in contact while a current of approximately 200 amperes is passed through them. Sufficient temperature is generated at the junction to cause sublimation of the carbon. The carbon vapour is condensed on 75mm x 25mm borosilicate glass microscope slides which have been coated with a water-soluble release agent; in our case we use RBS25. Carbon deposition takes place in a vacuum of $\sim 1 \times 10^{-2}$ Pa. The carbon-coated slides are removed from the vacuum evaporator and dipped into a dilute solution of cellulose nitrate (70 ml of collodion diluted to 1 litre in amyl acetate). This cellulose nitrate film thickness is controlled by the rate of

withdrawal of the slide from the solution¹³.

When dry the cellulose coated carbon film is floated off on a distilled water bath (Fig. 9 and picked up on an appropriate stripper foil frame. By using a thin plastic coating, the extremely fragile carbon films ($2-5 \mu\text{g}/\text{cm}^2$) can be mounted on the stripper frames without breaking. With the majority of self-supporting foils the most critical part of the operation seems to be when the mounted foil is drying; most breakages occur at this stage. A thin plastic supporting film minimises these losses. By placing the dried foils in an oven at 400°C for 10 minutes the cellulose layer is decomposed leaving only the carbon foil. Previous attempts at removing this plastic layer with the accelerator beam or by focusing a high intensity lamp onto the centre of the foil were only partly successful in removing the cellulose, as a residue of the decomposed layer remained on the carbon foil for a considerable period of time. All stripping and mounting steps are undertaken in a laminar flow cabinet to minimise dust accumulation on the carbon surface.

5.4.1.2. Arc Evaporation

Bradley²⁴¹ first published the technique of producing thin carbon foils by carbon-arc evaporation and he was soon followed by Pfeiffer²⁶⁹ and Blue.²⁷⁰ More recently Takeuchi et al.²⁵⁷ have reported fabricating carbon stripper foils by carbon-arc evaporation. They used 3 mm diameter spectroscopic grade graphite electrodes mounted horizontally in a vacuum evaporator. One electrode was fixed while the other one could be adjusted from outside the vacuum chamber to maintain a working gap of 1-3mm. The power supply used was that of an argon-arc welder with a no-load voltage of 80 volts; when a gaseous discharge was initiated this voltage dropped to 20 volts. In the plasma of this carbon-arc process the carbon atoms gain much

more energy than in the conduction heating method. Thus the condensation mechanism can occur differently, which may result in a different structure for the carbon film.²⁶⁵

5.4.1.3. Carbon Evaporation by Electron Bombardment

The technique of carbon deposition by electron bombardment has increased in interest since the publications by Maxman²⁷¹ and Morgan.²⁷² With this method the control of evaporation rate is relatively simple and the carbon source is nearly unlimited. Either a thick, long, carbon rod can be fed vertically into the electron gun crucible, or alternatively powdered carbon can be pressed into a suitable sized pellet for insertion in the crucible. Although control of evaporation rate is simple with this technique, the deposition rate is slower and capital outlay for equipment is much higher than with the conduction method described in 5.4.1.1. above.

5.4.2 Carbon Stripper Foils Produced by a d.c. Glow Discharge

The use of a glow discharge to crack hydrocarbon gases was first reported by Konig and Helwig.²⁷³ Since then thin isotopic carbon targets have been made by using a d.c. glow discharge in isotopically enriched acetylene gas.^{274,275} More recently hard, high-resistivity carbon layers have been produced on metal substrates from ethylene by the same technique.²⁷⁶ This led later to the production of thin self-supporting carbon films suitable for use as electron strippers.²⁵⁸

A high proportion of laboratories engaged in low-energy nuclear research use this glow discharge technique for making carbon stripper foils; it is claimed that the lifetimes of these foils far exceed those of foils made by other methods.²⁵⁸

5.4.2.1. Glow Discharge Technique for Making Carbon Stripper Foils as developed by the Author

The apparatus and technique used by the author to produce foils by a d.c. glow discharge is similar to that described by Tait et al.²⁵⁸ and Gallant and Dmytrenko.²⁷⁷ The assembly shown in Fig. 12 and Fig. 13 comprises two insulated planar electrodes spaced approximately 60 mm apart and housed in a vacuum chamber. This chamber consists of two vacuum flanges separated by a glass tube. The base flange, which supports the cathode, is mounted on a standard vacuum evaporator. The top plate supports the anode, a thermocouple pressure gauge and a needle valve. An earthed electrode plate is placed between the baseplate and the cathode so as to enable the confinement of a glow discharge to the volume between the two electrodes. Without this plate the discharge spreads throughout the chamber making control and reproducibility impossible.

The procedure we used to produce thin carbon foils by the d.c. glow discharge method was as follows: a 75 mm x 75 mm, highly polished stainless steel plate was used as the substrate. This plate was thoroughly cleaned in a detergent solution, followed by an alcohol rinse and successive distilled water rinses to remove any surface contamination. A thin layer ($\sim 10 \mu\text{g}/\text{cm}^2$) of sodium chloride was vacuum evaporated onto the polished surface of the plate; care was taken to ensure that the plate was free of surface dust to prevent pinholes in the final film. The plate was stored in a vacuum desiccator until required. Prior to carbon deposition the stainless steel plate was placed, sodium chloride coated surface upmost, on the cathode and the bell jar evacuated to less than 5×10^{-3} Pa. The chamber was back-filled with a 90% ethylene: 10% argon mixture to atmospheric pressure and after a few minutes was evacuated once more. The ethylene/argon mixture was again introduced and a pressure of 4 Pa (as

measured by a thermocouple gauge) was maintained. To safeguard the gauge it was disconnected before applying a potential to the cathode. As stripper foils thicker than $5 \mu\text{g}/\text{cm}^2$ are unacceptable deposition parameters were chosen to produce foils less than this value. Typical values of voltage, pressure and time were: 3 kV, 20 mA, 25 Pa and 10 seconds to produce a $4.5 \mu\text{g}/\text{cm}^2$ foil. Such plasma-made foils were found to be mechanically weaker than those made by evaporation; therefore to minimise breakages when attempting to mount the foils, the stainless steel substrate was dipped into a weak collodion solution as described in section 5.4.1.1.

5.5. Techniques to extend Carbon Stripper Foil Lifetimes

The literature on carbon stripper foils is voluminous and to some extent controversial. No attempt will be made to treat the subject comprehensively. For such detailed reviews the reader is referred to several other publications.^{214,258,261,266,270,278-280} It is apparent that short stripper-foil lifetimes are partially caused by a change in the mostly amorphous structure of the carbon films to a more crystalline form in the beam spot area.

To date, efforts to improve foil lifetime have followed two general lines of investigation: 1) modifying the crystalline structure of the foil, and 2) developing techniques to relieve the stress in the foil and/or to present a slackened foil to the irradiating beam. Currently there exists considerable difference of opinion as to the most effective fabrication technique to use.

5.5.1 Modifications to the Crystalline Structure

Two fundamental approaches to extending carbon stripper foil lifetime by modifying the foil's crystalline structure have been investigated. One

technique is to prepare foils of an amorphous structure by a d.c. glow discharge in a hydrocarbon gas. These foils resist the graphitisation process for a longer time. The graphitisation process which takes place during heavy ion bombardment, is delayed relative to the standard carbon foil, due to the high hydrogen content which stabilises the amorphous composition.^{281,277,290} The other method is to induce a graphitised structure by either heat treatment, by electron bombardment, or by laser-beam irradiation.^{286,288}

5.5.2 Reducing Foil Breakages Due to Shrinkage

Considerable ingenuity has been shown by various groups investigating means of reducing the incidence of foil breakages due to shrinkage. One technique reported is to slacken the foil once it is mounted on the retaining ring.²⁹¹ The carbon foils are floated onto softened aluminium rings of 16 mm and 17 mm outside diameter and allowed to dry. These rings are then compressed in two stages under a hydraulic press to an outside diameter of 15 mm. This reduction in diameter causes the attached carbon foil to adopt a dome shape, thus allowing the surface area of the foil to shrink by more than 20% during irradiation before destructive stresses develop. Using this technique, the yield of useful 2-3 $\mu\text{g}/\text{cm}^2$ foils, suitable for stripping is very low due to their extreme fragility. The typical capacity of a foil changer is approximately 250 stripper foils, and consequently making a full loading of foils is both tedious and time-consuming using the above method.

A simpler technique to produce "slackened" foils was to use a highly polished "dimpled" stainless steel sheet as the substrate. The dimpled plate was coated with a water soluble release agent and used as the substrate for carbon foils produced either by vacuum sublimation or by gas-

discharge. When floated off on a standard water bath and picked up on a suitable frame the mounted foils were slack in the centre. However, considerable care was necessary to locate the slack portion centrally across the frame aperture.²⁸²

Another technique was to seal the target frame, supporting the still wet carbon foil, across a circular aperture. An air-jet vacuum pump was used to evacuate the space behind the aperture to obtain a vacuum of approximately 5×10^{-4} Pa. This vacuum was controlled by means of a small needle-valve and maintained until the carbon foil, still floating on its frame, depressed into a domed area 2-3 mm deep. The foil was dried with a heat lamp before the pressure across the foil was reduced.²⁸³

An entirely different approach was to deposit carbon from an arc source onto heated soda glass substrates. The glass surface had been given a prolonged chemical treatment to form a "fogged", roughened surface.²⁸⁴

This roughened surface increased the effective area of the deposited carbon, thus prolonging the time before the foil stressed and ultimately ruptured under ion bombardment.

The technique used by the author for manufacturing stripper foils has been described in section 5.4.1.1. It is thought that two factors have contributed to increasing the lifetime of these stripper foils compared with the standard carbon foils used for target backings. Firstly, all floatation and mounting operations are undertaken in a laminar-flow cabinet to minimise the possibility of airborne dust particles settling on the carbon surface. The foils, still wet, are allowed to dry in the cabinet with the low pressure airflow blowing perpendicular to the foil surface. The tendency is for the foil to form a "dome" centre. Secondly, the foils are transferred, when dry, to an oven and baked for 20-30 minutes at 400°C to remove the supporting collodion layer. Simultaneously, the temperature is sufficient

to partially graphitise the carbon.

To date, the stripper foils produced at ANU by the above method have been satisfactory for the experiments being undertaken, using heavy-ion beams with masses up to and including that of bromine. Consequently there has been no necessity to undertake more elaborate fabrication techniques.

5.6. Comparison of Carbon Stripper-Foil Lifetimes

It is difficult to obtain a realistic comparison of stripper-foil lifetimes between the various laboratories engaged in this work. Foil lifetimes are compared between foils produced by the standard sublimation technique and those produced by the large number of techniques detailed above. Extended lifetime claims vary from a factor of 55^{257} to a factor of 6^{285} . However, many results quoted have been obtained with test machines, and the results extrapolated. Performances have been quoted for stripper-foils as thick as $10 \mu\text{g}/\text{cm}^2$ in many cases which bear little relationship to how a realistic stripper foil, $2\text{-}3 \mu\text{g}/\text{cm}^2$ thick, will perform in practice.

Qualitative statistics for carbon stripper foils, made by the author and used in the terminal of the ANU 14UD Van de Graaff accelerator over a period of ten years show that the main reason for foil rejection has been thickening of the foil, not breakage as has been reported by other laboratories. The technique used by the author only differs slightly from those techniques used in similar nuclear target laboratories, it is therefore difficult to explain why our foils have a superior lifetime to those reported elsewhere.

6. Special Target Making Techniques

This final section is primarily concerned with special nuclear target preparation techniques.

Section 6.1 discusses targets that are made by first reducing the oxide of the element being investigated, followed by evaporation of the resulting metal, to produce the required target. The two major methods of oxide reduction, i.e. reduction with hydrogen or the simultaneous reduction-distillation by the metallothermic method, are described. A technique for producing thin, self-supporting targets of calcium, magnesium and zinc is outlined. The increasing importance of rare-earth isotopic targets is emphasised and the use of lanthanum and thorium in the reduction-distillation of these targets is discussed. The evaporation process is treated in detail.

Techniques that have been developed for fabricating various isotopic oxygen targets are discussed in section 6.2 and comparison is made between the qualities of the methods.

Section 6.3 deals with miscellaneous targets and target-making techniques. These targets are either chemically or thermally unstable in their elemental form and chemical conversion is necessary to produce a satisfactory target. The development of a technique to fabricate self-supporting, stress-free targets of some refractory elements such as B, Be, Co, Cr, Fe, Ni and Ta, is described. This section concludes with a description of the technique developed to make self-supporting targets of Ca, Mg and Zn.

6.1 Targets Produced by First Reducing the Oxide to the Metal

The majority of solid, natural isotopes are only supplied in the form of simple compounds; usually oxides. This is frequently because the metal is not the most stable form of the element. It is therefore advantageous,

in the case of the more reactive elements, to perform the reduction to the metal simultaneous with, or immediately prior to, fabricating the target.

Three general reduction methods are used in nuclear target manufacture; reduction using hydrogen gas, reduction/distillation with active metals or with carbon, and electrolytic reduction.

6.1.1 Hydrogen Reductions

Hydrogen reductions are by far the simplest and most straight-forward of all reduction techniques. This method of oxide removal has been utilised for many years in the electronic valve industry for cleaning metal electrodes and components. When metal parts are heated in an hydrogen atmosphere, not only are foreign (impurity) gases driven off but also metal oxides are reduced by the reaction $2M_xO \rightarrow 2M_x + O_2$. For this reaction to be effective, the partial pressure of the oxide (O_2 dissociation pressure) must be larger at the chosen temperature than that of water vapour formed from O_2 and H_2 in the reaction vessel. If this is not the case the reverse reaction occurs, $2M_x + O_2 \rightarrow 2M_xO$: not only is there no reduction, but previously unoxidised metal surfaces become oxidised.²⁹⁸ It is therefore necessary to keep the partial pressure of O_2 in the reaction vessel as low as possible; also the hydrogen used must be pure and free from water vapour. Cu, Ni, Fe, Co, Mo and W are reduced at all temperatures between 200 and 2000°C and remain oxide free. This is because the dissociation pressures of their oxides are higher than that of water vapour. However, other oxides having lower dissociation pressure, i.e. BeO, MgO, Al_2O_3 , etc., are not reduced.

Friebel et al.^{297,298} and Heagney and Heagney²⁹⁹ report the reduction of Cr_2O_3 , CuO, Fe_2O_3 , GeO_2 , In_2O_3 , $Pb(NO_3)_2$, Sn_2O_3 , Tl_2O_3 and WO_3 by heating these compounds to a high temperature in a hydrogen atmosphere. In all cases they have carried out the reductions in hydrogen-filled tube furnaces,

taking precautions to ensure the hydrogen gas used was ultra pure and dry, with a water and oxygen content less than 1 part per million.

The author has carried out routine hydrogen reductions of CuO , Fe_2O_3 , Sn_2O_3 , $\text{Pb}(\text{NO}_3)_2$ and WO_3 in a standard vacuum evaporator chamber, which has the advantages that, once reduced, the metal can be evaporated without exposing it to atmosphere and the reduction process can be observed continuously. It has been found that reductions are satisfactorily achieved using high-purity cylinder H_2 , without resorting to purification, such as drying the gas or passing it through a palladium barrier to eliminate residual oxygen. The oxide to be reduced is contained in an evaporation boat, which is mounted in the vacuum chamber. The chamber is initially evacuated to $\sim 1 \times 10^{-2}$ Pa, isolated from the vacuum system and filled with hydrogen. This step is repeated to ensure that the residual oxygen within the chamber is negligible. The chamber is once more filled with hydrogen to a pressure of approximately 5×10^4 F. and maintained at this pressure while hydrogen is leaked into the chamber at a flow rate of 2 litres/minute. The evaporation boat is heated by either R.F. or resistive heating to approximately 1000°C and the reaction allowed to proceed until complete conversion has taken place. When cool either the chamber is evacuated to a good vacuum for subsequent evaporation of the reduced metal or alternatively the metal is removed and stored for future use.

Hydrogen reduction of oxides has the advantages of:

- 1) it is a clean procedure, because there are no impurities to contaminate the prepared element as is the case with metallothermic reductions to be described in the following section.
 - 2) efficiency of hydrogen reduction is in most cases nearly 100%.
- However, as has been explained previously this technique is limited to the reduction of the oxides of those elements with a lower oxygen affinity than hydrogen.²⁹⁶

6.1.2 Metallothermic Reductions

The application of pyrochemistry to the preparation of pure metals has been used for many years³⁰⁰⁻³⁰² and presents itself as a useful technique for reducing isotopic oxides of those elements that are not readily reduced by hydrogen. This technique involves the reaction of a reducing metal (referred to as the reductant), with the oxide of the element to be reduced. The temperature at which this reaction occurs must be such that the vapour pressure of the reduced metal is at least 10 Pa, while the reductant vapour pressure is at least two orders of magnitude less than this value. Under these conditions the reduced metal is separated from the oxide by vaporisation according to the chemical equation $M_xO_y + zR \rightarrow R_zO_y + xM^\dagger$, where M is the pure metal distillate and R is the reductant metal.³⁰³

The technique, in general utilises an active reductant such as Al, C, Ca, La, Ta, Th, Ti, Y or Zr, whose oxides are non-volatile. The process usually takes place in a vacuum evaporator, using any one of the many heating methods described previously to achieve the reaction temperature. In the majority of cases the reduced metal evaporates near the reaction temperature, whereas the reductant and its oxides have a relatively low vapour pressure at this temperature.

One can often simultaneously reduce the target material and evaporate it onto the substrate. However, in many instances it is necessary to evaporate onto a thin backing or membrane (usually carbon), particularly with materials such as Ba, Ca or Mg which react with the atmosphere and have to be conveyed to the accelerator target chamber in a sealed container. Unfortunately the sticking coefficient for Mg and Ca evaporated films on carbon is low and the resulting deposits are patchy or uneven. To improve condensation, the author mounts an auxiliary heater of coiled tungsten wire adjacent to the evaporation boat. Using the radiation generated by this

heater to heat and outgas the carbon foils before reduction and evaporation take place, film adhesion is considerably improved. The backings are also shielded from the evaporation source, apart from a small central hole for the stream of evaporant. The two precautionary steps of outgassing the foils and shielding most of the heat from the evaporation source greatly improve condensation of the evaporant. The powdered oxide of the material to be reduced is heated in air for 2-3 hours to drive off absorbed compounds such as H_2O or CO_2 . The oxide is then intimately mixed with the finely divided reductant and the mixture is pressed into a pellet to assure good physical contact and to exclude as much gas as possible. Generally, one and a half to two times the stoichiometric ratio of reductant to oxide is used to allow for oxidised reductant and non-uniform mixing. It is important to use finely-divided reductant, but since active metal powders oxidise rapidly, they are most effective if freshly prepared from the bulk material just prior to each reduction. Fresh filings from a lump of the reductant are generally used. The compressed pellet is designed to make good thermal contact in the tantalum crucible. Most reductions take place at 1000-2500°K.

A list of the materials the author has reduced by H_2 reduction and by reduction-distillation methods using reductant metals is given in Table 4.

6.1.3. Rare-Earth Targets.

With the introduction of higher energy Van de Graaff accelerators it is now possible to undertake many more experiments involving the rare-earth group of isotopes that previously were impracticable. Consequently there is an increasing demand for nuclear targets fabricated from this group of elements.

Spedding and Doane³⁰⁴ developed a metallothermic method for the

TABLE 4

Metals Reduced from their Oxides using H₂

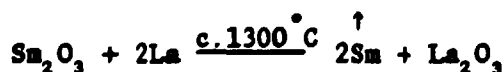
Cr. W. Fe. Sn. Cu. Pb

Metals Reduced by the Metallothermic Method

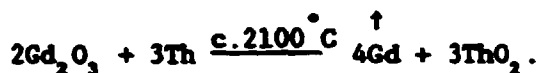
<u>Material</u>	<u>Reductant</u>	<u>Chemical Reaction</u>
		↑
Ba	Ti	$2\text{Ba}(\text{Na}_3)_2 + \text{Ti} - 2\text{Ba} + \text{TiO}_2 + 4\text{NO}_2 + \text{O}_2$
		↑
Ca	Y	$3\text{CaO} + 2\text{Y} - 3\text{Ca} + \text{Y}_2\text{O}_3$
		↑
Mg	Ti	$2\text{MgO} + \text{Ti} - 2\text{Mg} + \text{TiO}_2$
		↑
Zn	Ti	$2\text{ZnO} + \text{Ti} - 2\text{Zn} + \text{TiO}_2$

production of Eu, Sm and Yb metals by the reduction of their oxides with lanthanum in vacuo. This technique was used by Westgaard and Bjornholm who fabricated self-supporting foils of Dy, Ef, Eu, Gd, Nd, Sm, and Yb for use as nuclear targets.³⁰⁵

Although lanthanum has a melting point of 920°C its vapour pressure at this temperature (1×10^{-4} Pa) is low enough to ensure its use as a reductant for clean separation from the relatively volatile Eu, Sm and Yb metals. For example the reaction for Sm is:



The use of lanthanum as a reductant is difficult for the less volatile rare earth elements, e.g. Dy, Er, Gd, Lu and Nd as the distillate tends to become contaminated by lanthanum. To overcome this difficulty less volatile reducing agents such as metallic thorium or zirconium (both having practically non-volatile oxides) are used. For example, gadolinium oxide can be reduced with thorium metal according to the chemical reaction:



6.1.3.1. Lanthanum Reduction

Lanthanum metal has been used by the author as a reductant for the preparation of Dy, Eu, Sm and Yb metals from their oxides.

Approximately 50-60 mg of the powdered oxide is carefully dried by heating in a porcelain crucible over a bunsen burner to drive off absorbed gases. The oxide is then intimately mixed with approximately 1.2 times the stoichiometric amount of freshly prepared lanthanum metal filings, using an agate pestle and mortar. The mixture is then transferred to a 5 mm i/d x 20 mm long tantalum crucible and compacted with a glass rod. A conical tantalum stopper, with a 1 mm central hole, is hammered into the entrance of the crucible. Both crucible and stopper had been vacuum stoved previously to remove occluded gas and surface oxides. Tantalum was chosen as the crucible material because it has a negligible tendency to alloy with the rare earth metals; it also has a high melting point and low vapour pressure, and is easily machined.

The loaded crucible is mounted in the electron bombardment apparatus depicted in Fig. 14 and the system evacuated to a pressure $< 1 \times 10^{-3}$ Pa. After degassing the crucible, by preheating with the electron gun filament, the high voltage is switched on and slowly increased to approximately 3 kV. The filament current is adjusted until the power dissipated in the crucible raises it to operating temperature. The temperature is measured with a disappearing-filament pyrometer. Crucible temperature is maintained until evaporation and distillation is completed (approximately 5 minutes). Reduced metal condensation takes place on a 0.025 mm thick molybdenum foil, placed 2-3 mm above the crucible exit. Other workers report using a 0.5 mm thick, polished tungsten plate onto which the evaporant condensed.³⁰⁵⁻³⁰⁸

After cooling, this condensate was removed by tapping the plate with a few hammer strokes, whereupon the main portion of the deposit could be peeled off in one piece; metal residue was scraped from the plate with a sharp knife and stored for redistillation. Our experience was that either the small lump of condensate broke into small pieces or else was so stressed that the subsequent rolling operation to produce thin foils caused the foils to crack and shred. To minimise the stresses introduced during condensation it was decided to evaporate onto a thin (0.025 mm thick) molybdenum foil which could be dissolved in a HF/HNO₃ acid solution [20 ml HF to 15 drops of HNO₃]. This acid etch rapidly dissolves molybdenum without appreciably attacking the rare earth metals. The assembly, which supports the molybdenum foil, was designed so that the rare earth deposit becomes radiantly heated by the evaporation source during deposition; this produced a far more malleable and stress free deposit.

Target foils were produced by rolling the condensate between polished stainless steel sheets until the required thickness was achieved. In some instances foils as thin as 300 µg/cm were made this way. As most rare-earth metals exhibit pyrophoric behaviour and are sensitive to atmospheric decomposition, the greatest care has to be taken when handling thin foils made from this range of materials. As quite often foils will spontaneously ignite during handling, it is advisable to handle them with plastic coated tweezers and to ensure that metal surfaces likely to come in contact with the foil (e.g. stainless steel rolling plates, balance pans, etc.) are adequately earthed. The finished foils are generally stored between cleaned degassed microscope glass slides and housed in an evacuated vacuum desiccator until required.

Temperatures necessary to perform the chemical reaction and evaporation of the reduced metal are:

Yb : 1100-1200°C, Sm : 1300-1400°C, Dy : 1650-1750°C.

6.1.3.2. Thorium Reduction

Metallic samples of the separated isotopes of Dy, Er, Nd and Gd have been prepared by the reduction of their oxides using thorium metal as a reductant. The technique used is identical to that described for lanthanum reduction described in 6.1.3.1. the only exception being that twice the stoichiometric amount of fresh thorium filings are used.

Thorium is used as a reductant in preference to lanthanum for the higher melting point rare earth oxides to minimise contamination by the reductant.

Crucible temperatures required to perform the combined reduction/distillation process are:

Dy: 1650-1750 °C, Er : 1750-1850 °C,

Nd: 1750-1850 °C, Gd : 2100-2200 °C.

The reduction/distillation temperatures for both lanthanum and thorium are the temperatures quoted by Westgaard and Bjornholm.³⁰⁵ These temperatures have been independently confirmed by the author.

6.2 Thin Film Isotopic Oxygen Targets

Considerable interest continues to be shown in the nuclear properties of oxygen and its isotopes. Jaklovsky³¹⁸ lists over 340 references to the preparation and use of isotopic oxygen targets. These targets range from gas targets, and targets using enriched water, to plastics such as Mylar ($C_{10}H_8O_4$), boric acid (H_3BO_3) and various inorganic oxides. The form each target takes is dictated by the experiment to be undertaken and the quantity of target material available. In the majority of nuclear-structure investigations the main requirements are a thin self-supporting target for optimum resolution, containing the minimum amount of contaminating elements and having the maximum concentration of the isotope under investigation.

For isotopic oxygen targets the use of gas cells or frozen water is often impracticable and recourse is made to thin films containing a compound of the isotope in question.

Those isotopic oxygen targets developed by the author are described in the following sections.

6.2.1. Tungsten Oxide

The preparation of tungsten oxide targets have been reported by the author.³⁰⁹

Tungsten was chosen as a suitable compound since it is easily oxidised and the oxide formed can be vacuum evaporated without difficulty. Furthermore it is often important that in nuclear spectroscopy a wide separation of mass numbers exists between the element being investigated and any other elements present. The separation between oxygen and tungsten adequately fulfils this requirement. Finally, tungsten oxide is a stable compound which can be stored without deterioration.

Isotopically enriched tungsten oxide was produced by heating cleaned tungsten filaments to incandescence in an enriched oxygen gas atmosphere and collecting the tungsten oxide formed on the walls of the glass vacuum vessel. The tungsten oxide was evaporated from a platinum boat and collected on suitable backings. Platinum was chosen to prevent the oxide being reduced, a factor which makes the usual source materials such as tungsten, molybdenum or tantalum unsuitable.

6.2.2. Self-Supporting Tantalum Pentoxide Films (Ta_2O_5)

The technique adopted to produce targets of tantalum pentoxide is similar to that described for niobium.³¹⁰ Tantalum was chosen as both tantalum and niobium appear to be the only rare metals which can be anodised

using water as an electrolyte without adding another oxidising agent; as is the case when anodising a metal such as aluminium which requires the addition of ammonium citrate $(\text{NH}_4)_3 \cdot \text{HC}_6\text{H}_5\text{O}_7$.

Tantalum films, approximately $100 \mu\text{g}/\text{cm}^2$, were vacuum deposited, using an electron gun, onto glass microscope slides that had been previously vacuum coated with a thin layer of barium chloride. The microscope slides were maintained at a temperature of 350°C prior to and during tantalum deposition and allowed to cool naturally to room temperature before venting the system. After clamping the microscope slides, tantalum film downwards, they were lowered, with a suitable d.c. voltage applied to the tantalum side, so that the tantalum film made contact with the small amount of oxygen enriched water [Monsanto Research Corp., Miamisburg, Ohio, USA] that had been introduced into the electrolysing cell. Tantalum films of $100 \mu\text{g}/\text{cm}^2$ were totally anodised over an area of 1.3 cm^2 with an applied voltage of 100 volts. Anodising was assumed to have been completed when the cell current had fallen to zero and the anodised area was transparent; this occurred in a few minutes. The resulting films were floated off on a water bath and mounted on suitable target frames.

6.2.3. Self-Supporting Silicon Dioxide Targets $(\text{Si}^{18}\text{O}_2)^{309}$

Silicon dioxide, enriched to 93% ^{18}O [Isotope Labelling Corp, Whippery, N.J., USA] was vacuum deposited from a directional tantalum boat onto glass slides that had been coated with the water soluble release agent RSB25. The evaporated film of Si^{18}O_2 was readily floated off on a water bath and picked up on target frames. Alternatively the Si^{18}O_2 was evaporated onto self-supporting carbon films.

6.2.4 Self-Supporting Nickel Oxide Targets ($Ni^{100}O$)³⁰⁸

Commercially available nickel foils [Chromium Corp of America, Cleveland, Ohio, USA], approximately $90 \mu\text{g}/\text{cm}^2$ thick, were mounted on target frames and supported in a reaction chamber. This chamber was connected to a vacuum system capable of achieving a base pressure of $<1 \times 10^{-4}$ Pa. A glass cylinder, containing 100 atmospheric litres of 99.3% enriched Oxygen-18 gas was joined to the system, together with a small cylinder of molecular sieve. After evacuation of the complete apparatus to $<1 \times 10^{-3}$ Pa, the reaction chamber was isolated from the main vacuum system and the glass seal on the Oxygen-18 gas bottle broken. Oxygen gas was admitted into the reaction chamber to a pressure of ~ 10 Pa, as measured by a capsule dial gauge; this pressure was not critical. For convenience the Sun was used as a radiant heat source, the solar rays being focused onto the centre of the nickel foils by a suitable lens. When the centre portion of the foil became transparent, as seen with a mirror, oxidation was assumed complete. The remaining enriched oxygen gas was absorbed by cooling the molecular sieve with liquid nitrogen. After absorption was completed the molecular sieve was isolated from the reaction chamber and the enriched oxygen gas stored for future use.

6.3 Preparation of Targets from Elements that are either Chemically or Thermally Unstable, and those Targets where a Carbon Backing is Unsuitable

A number of elements are either difficult to produce in elemental form, are chemically unstable, or will not withstand heavy-ion beam irradiation without re-evaporating. Typical elements that fall within this category are: barium, cadmium, caesium, calcium, lead, mercury, etc., plus the gaseous elements such as hydrogen, nitrogen, oxygen and the inert gases.

In this discussion only the solid elements will be considered. Here will be described nuclear targets of these elements made in chemically stable forms.

In some nuclear structure experiments carbon is an undesirable impurity which seriously interferes with the reaction under investigation. A technique for producing carbon-free targets will be described.

6.3.1 Mercury Targets

Techniques for preparing mercury targets by electro spraying,³¹¹ mass separator implantation,³¹¹⁻³¹³ forming an amalgam with gold,³¹³ or electroplating in metallic form and cooling,³¹⁴ are considered unsatisfactory due to the thermal instability of the targets under beam conditions.

A modification of the methods described by Doubt et al.,³¹⁵ Esat et al.,³¹⁷ and Friebe et al.,³¹⁸ involves the vacuum deposition and condensation of β -HgS (Metacinnabar), a stable mercury compound.

6.3.1.1. Formation of Mercury Sulphide from Mercury Oxide.

Enriched mercury isotopes are obtained as HgO from Oak Ridge National Laboratory, Separated Isotope Division. Small quantities of the mercury oxide are dissolved in 2N.HCl (it is important to use dilute HCl as mercury oxide is not soluble in the concentrated acid), and H₂S bubbled through the solution to form a black precipitate of β -HgS. The precipitate is thoroughly washed by a succession of washing and decantings with distilled water, followed by a final wash in absolute alcohol. The precipitate is dried under an infra-red lamp.

6.3.1.2. Deposition of Mercury Sulphide

Mercury sulphide was evaporated from a quartz crucible, mounted in a hole drilled in the end of a stainless steel rod. Heat to the crucible was

provided by a small 500 watt cartridge heater. For efficient condensation the target backing was mounted on a liquid nitrogen cooled copper plate. Both crucible and cooling plate temperatures were continuously monitored by means of thermocouples in contact with the crucible and foil respectively. Generally HgS sublimation was commenced when the target temperature reached -80°C . At temperatures less than -130°C large internal stresses were apparent, causing the thin deposited film to split when warmed up.

A predetermined amount of HgS was placed in the quartz boat, and the distance between boat and backing adjusted to approximately 2 mm, and not more than 5 mm. The system was evacuated to $<1 \times 10^{-3}$ Pa and the cooling plate allowed to reach -80°C before deposition was commenced. The crucible was slowly heated to 250°C - 280°C and maintained at that temperature for approximately 20 minutes, by which time typically 50% of the HgS charge had been deposited onto the cooled backing foil. Above 300°C the weakly-bound molecules of HgS disassociated, as indicated by a yellow deposit surrounding the target area. After the boat was cooled, and the backing had reached room temperature the target was removed and a satisfactory deposition was indicated if the target was a shiny grey/black in appearance. A further protective film of carbon ($10\text{-}15\text{ }\mu\text{g}/\text{cm}^2$ thick) was evaporated on to the face of the target to minimise re-evaporation under beam bombardment.

6.3.2 Compound Targets

6.3.2.1 Barium Targets

Barium is a chemically active metal which reacts violently when exposed to atmosphere and must therefore be handled in an inert gas atmosphere or under high vacuum. Metallic barium is used extensively in the electronic valve industry as a "getter" due to its high affinity for oxygen.

The affinity of barium for oxygen, and the metal's relatively low melting point, result in metallic barium targets deteriorating rapidly under irradiation. To improve target lifetime under beam conditions it was decided to use a stable compound of the element. The selected compound was barium chloride, which was made from barium nitrate by firstly converting the nitrate to barium oxide by gently heating in vacuum. The oxide was then dissolved in 2N hydrochloric acid and slowly evaporated to dryness under an infra-red lamp, leaving a white crystalline deposit of barium chloride. The barium chloride was gently heated to drive-off water of crystallisation immediately prior to vacuum evaporation of the compound; otherwise it was found that the hydrated form of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) decomposed in vacuum. Vacuum evaporation of barium chloride from a collimated carbon crucible on to thin carbon foils was successfully accomplished.

6.3.2.2. Cadmium Chloride

Metallic cadmium and zinc are the most difficult elements to deposit by vacuum evaporation due their very low "sticking coefficients"⁴. Consequently to produce isotopically pure cadmium targets it was decided to utilise the same technique as described in the previous section for barium chloride. Isotopic cadmium oxide was converted to the chloride by dissolving in 2N hydrochloric acid and drying the solution under an infra-red lamp. Vacuum-evaporated films from a collimated carbon crucible onto thin, self-supporting carbon backings was readily achieved.

6.3.2.3. Lead Sulphide

For some experiments, investigating certain of the lead isotopes, it was decided to use lead sulphide as the most suitable compound. The lead isotopes were supplied as lead nitrate $\text{Pb}(\text{NO}_3)_2$, which were firstly

dissolved in distilled water and then converted to lead sulphide by bubbling hydrogen sulphide through the liquid. Lead sulphide, which precipitated out as a black powder, was thoroughly washed in distilled water, rinsed in alcohol, and then dried in an oven. The compound was vacuum evaporated from a carbon crucible, onto thin, self-supporting carbon backings.

6.3.2.4 Selenium

Low melting point materials pose special target lifetime problems. Unless special precautions are taken the target material will be rapidly re-evaporated under heavy-ion bombardment. An obvious solution is to cool the target but for thin self-supporting targets this is often impracticable. In many cases it is possible to evaporate the target material on to a thin self-supporting carbon backing and deposit a thin "flash" of carbon over the top, thus forming a carbon-target material-carbon sandwich. The protective carbon layers considerably reduce re-evaporation.

A further technique that has been successfully developed for making selenium targets (melting point 217°C) is to evaporate the selenium on to a glass microscope slide that has been coated with a thin water-soluble release agent, such as RBS25. A protective carbon layer, $\sim 10\text{ }\mu\text{g}/\text{cm}^2$ thick, is evaporated on the selenium surface. The multiple foil is floated off, as described in section 4.1.1. and picked up from the surface of the water bath by allowing the film to fold across a standard target frame, thus trapping the double-thickness selenium film between two protective carbon layers. This technique is illustrated in Fig. 15.

6.4.3. Targets that Require Annealing to Remove Thermal Stresses.

Many high melting point metals, e.g. B, Be, Co, Cr, Fe, Ni, Ta, etc., can easily be vacuum evaporated, using the "e"-type electron gun described

in 3.2.3. However, for thin self-supporting targets of these materials, great difficulty is experienced obtaining films that do not shred or break when mounted on target frames. The reason for fragility in these instances appears to be due to thermal stresses introduced during condensation. Stress-free targets were obtained by vacuum depositing the target material onto thin copper foil [0.025 mm thick] which was mounted on an auxiliary pancake heater within the vacuum chamber. The chamber was evacuated to a vacuum of $<1 \times 10^{-3}$ Pa and the copper backing slowly heated to approximately 350°C and maintained at this temperature while the target material was evaporated. The heater was then switched off and the assembly allowed to slowly reach ambient temperature before the system was vented to atmosphere. When cool, the coated copper foil was removed from the system and cut into suitably sized pieces; these pieces were floated, copper surface downwards, on a bath of trichloroacetic solution, as described in table 1. After the copper backing had dissolved, the floating target material was thoroughly washed with distilled water and the foils mounted on target frames.

6.4.4 Thin Self-Supporting Targets of Magnesium, Calcium and Zinc

In some nuclear structure experiments, e.g. $^{28}\text{Si}(^{16}\text{O}, ^{12}\text{C})^{32}\text{S}$, and $^{26}\text{Mg}(^{18}\text{O}, ^{16}\text{O})^{28}\text{Mg}$, the presence of carbon can seriously interfere with the reaction being investigated. This is because carbon has a very large cross section for certain reactions, resulting in a large number of spurious background counts, sufficient to mask the required reaction products. Consequently, it is imperative for these experiments that the amount of carbon in the target is negligible. The simultaneous reduction/distillation of the elements Mg, Ca or Zn onto thin carbon backings is not possible and self-supporting targets are required. One method suggested was to evaporate the element onto a mirror-finish, chromium coated metal plate and carefully

peel away the deposited film.³¹⁹ Although an attractive technique it is only practicable for foils $\geq 500 \mu\text{g}/\text{cm}^2$. The foils required by experimentalists in the author's laboratory were $100 \mu\text{g}/\text{cm}^2$ or less. Unfortunately thin foils of magnesium, calcium or zinc are rapidly attacked by water and cannot therefore be floated from a water soluble substrate. Exhaustive investigations into floating films of these elements onto a suitable liquid other than water were unsuccessful. Either the soluble release agent was incompatible with the evaporant or the release agent was not soluble in a suitable liquid or if the film did float off it sank and could not be readily picked-up on the target frame. The most satisfactory technique was to coat a glass microscope slide with a thin layer of Formvar, as described in Table 2. The target material was then vacuum evaporated onto the Formvar surface and the evaporated film scored into pieces of a size suitable to be mounted on a standard target frame. The Formvar-supported films were carefully floated off on a water bath, care being taken to ensure no water came into contact with the target material. The films were picked up on target frames, the residual water removed with the corner of a piece of absorbent filter paper and the mounted films placed beneath a desk lamp to dry. When dry the thin formvar backings were removed by suspending the targets over a beaker of warm chloroform for approximately half an hour. Thin Mg, Ca and Zn films oxidise in air, so the completed targets were stored in a vacuum desiccator until required.

A comprehensive list of the common targets is presented in Table 5.

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TABLE 5

Material Element or Compound	Chemical Form of Target	Method of Fabrication	M.P. (°C)	B.P. (°C)	Evap.Temp. (°C) $V_0 = 1\text{Pa}$	Remarks	Refs.
ADENINE (PURINE-6AMINO)	$(C_5 H_5 N_5)$	Evaporation from Mo dimple boat	350	220 _{SUB}	-	Useful thin nitrogen target on suitable backing	320
ALUMINIUM*	Al	Vacuum evaporation from Al_2O_3 coated dimple boat, W spiral or C crucible	660	2467	~1200	Aluminium film floated from slide coated with release agent.	2,6
ALUMINIUM*	Al_2O_3	Vacuum evaporation (electron bombardment)	2045	2980	~1800		
	Al_2O_3	Anodising	-	-	-	Used as thin backing. Also source of isotopic O_2	209,321-326
AMMONIUM CHLORIDE	NH_4Cl	Vacuum evaporated from Mo dimple boat	340 _{SUB}	520	-	Sublimes. Useful thin nitrogen target on thin backing. Unstable at high beam currents.	135
ANTIMONY*	Sb	Vacuum evaporation	630	1380	~680	Antimony film floated from slide coated with release agent.	2,6,135
ANTIMONY CHLORIDE	Sb	Electrodeposition	-	-	-		327
ANTIMONY TRISULPHIDE	Sb_2S_3	Vacuum evaporation	550	1150	550	Can be used as a sulphur target	135
ARSENIC	As	Vacuum evaporation	817	613 (SUB)	~350	Toxic	2,6,328
BARIUM	Ba	Vacuum evaporation	717	1640	629	Oxidises rapidly when exposed to air.	2,8,49,329,330
BARIUM CHLORIDE	$BaCl_2$	Vacuum evaporation	963	1560	-	Barium or Chlorine target on thin backing. Useful as release agent.	135

BARIUM OXIDE	BaO	Vacuum evaporation from Pt. crucible	1925	> 2000	1540	Only slight decomposition from Pt. source	2
BERYLLIUM*	Be	Vacuum evaporation (Electron Bombardment)	1284	2970	1246	Highly toxic material Floated from slide coated with release agent.	2,6,135, 331,333- 335
BISMUTH*	Bi	Vacuum evaporation	271	1560	698	" "	2,6,135, 336
		Electrodeposition from $\text{Bi}(\text{NO}_3)_3$ [$\text{HNO}_3 + \text{NH}_4\text{OH}$, 5v, 70 _{mA}]	-	-	-	Deposited on Mylar + Ag backing	327
BORON*	B	Vacuum evaporation [Electron bombardment]	2300	2550	1350	Molten metal reacts with all source materials. Floated from slide coated with release agent.	2,8,337- 342
	BORIC ACID (H_3BO_3)	Vacuum evaporation	236	-	-	Unstable at high beam currents.	135
CADMIUM*	Cd	Vacuum evaporation	321	765	264 _{SUB}	Condensation difficult Floated from slide coated with release agent.	2,8,135, 343
		Electrodeposition of CdCl_2 (0.1N HCl)					14,37
	CdS	Vacuum evaporation.	1750	-	1380 _{SUB}	Cadmium or Sulphur target.	2,135
CALCIUM*	Ca	Vacuum evaporation	810	1485	605 _{SUB}	Possible to get self supporting targets by stripping in Hexane	2,6,14, 135,271, 344
						Film peeled from polished chromium plated metal plate.	

CALCIUM CARBONATE (CaCO ₃)	Ca	Reduction of oxide after decomposition of carbonate, with simultaneous evaporation of metallic Ca.	-	-	900-1200	Oxide reduced by using Ta boat, CaCO ₃ mixed with La.	131,135, 345,347, 348
CALCIUM CYANAMIDE (CaCN ₂)	CaCN ₂	Vacuum evaporation	1300 SUB	-	-	Can be used as a nitrogen target	135
CALCIUM FLUORIDE (CaF ₂)	CaF ₂	Vacuum evaporation	1360	2500	1400	Can be used as a fluorine target.	2,8.135

CARBON *	C	Vacuum evaporation	< 3550	4827	2680	Resistance heating or carbon arc using pointed carbon rods. Electron bombardment. Floated from slide coated with release agent.	241-248, 253, 256, 257, 265, 285-294
	C	Electrical Discharge	-	-	-	Glow discharge in either acetylene, methane or ethylene/5% argon	249, 252, 254, 258, 259, 261, 264, 274, 275, 277, 283, 349
						Electron-beam-heated carbon source used to introduce carbon vapour into an argon discharge	250
		Low pressure plasma				Deposition using an R.F. discharge in Butane gas	251, 351-354
	C	Thermal cracking	-	-	-	Thermal cracking from Methyl Iodide.	63, 64, 143, 355
	C	Settling	-	-	-	Air suspension of graphite powder settled then pressed.	100
CAESIUM	CsBr	Vacuum evaporation	636	1300	-	Deposit onto thin backings. Soluble release agent.	2, 356, 357
CERIUM	Ce	Vacuum reduction of CeO and simultaneous evaporation of Ce	800	3257	-		2, 356, 357

CHLORINE	NaCl, BaCl ₂ , KCl	Vacuum evaporation	786- 963	1413-1700	-	Deposit onto thin back- ings. All compounds useful as release agents.	135,346, 358
CHROMIUM*	Cr	Vacuum evaporation	1900	2672	1205 _{SUB}	Cr deposited onto heated copper foil to relieve stresses. Copper foil dissolved in Trichloroac- etic acid + ammonia.	2,6,135, 297,359
		Electrodeposition					360,361
	Cr ₂ O ₃	Electron bombardment from C crucible with simultaneous reduction and evaporation					131
COBALT*	Co	Vacuum evaporation	1478	2870	1649	Co deposited onto heated copper foil to relieve stresses. Copper dissol- ved in trichloroacetic acid + ammonia.	2,6,14, 135,271, 362,364
		Sputtering	-	-	-		152
COPPER*	Cu	Vacuum evaporation	1083	2567	1273	Floated from slide coated with release agent.	2,6,14, 135,152, 271
		Vacuum reduction of Cu ₂ O and simultaneous evaporation of Cu					271,348
		Electrodeposition from CuCl ₂					14

COPPER (cont).	Cu	Electrodeposition from CuSO_4					37,363
DEUTERATED * POLYETHYLENE	$[\text{C}_2\text{D}_4]_n$	Polymerisation				Deuterium or Hydrogen target	106,107, 365,366
		Film casting					367
FORMVAR *	$\text{C}_5\text{H}_8\text{O}_2$	Dissolve in chloroform. (3g Formvar to 100ml chloroform). Dip glass in solution and remove film when dry				Hydrogen target or backing foil	13,14, 135
GALLIUM	Ga	Vacuum evaporation	30	2403	1093		2,6,14, 368
	GaN	Vacuum evaporation			850 _{SUB}	Also useful as a nitrogen target	135
	Ga_2S_3	Electrodeposition					327
GERMANIUM *	Ge	Vacuum evaporation	959	2830	1251	Floated from slide coated with collodion as release agent.	2,6,14, 135,271, 369
	GeO	Electrodeposition 0.1N NH_4OH					327
GOLD *	Au	Vacuum evaporation	1063	2966	1465	Floated from slide coated with release agent.	2,6,135, 346
		Electrodeposition				Deposited from AuCl or AuCN	327

HAFNIUM	HfO ₂	Vacuum evaporation (electron bombardment)	2812	~5400			137,370
	Hf	Sputtering					152
HYDROGEN (H ₂ , D, T)	POLYETHYLENE [C ₂ H ₂] _n	Polymerisation					106,107, 135,366
		Absorption into Ti or Ta					70,71, 371,372, 374,375
INDIUM *	In	Vacuum evaporation	157	2000	952	Floated from slide coated with release agent.	2,6,14
	InCl	Vacuum evaporation	235	570			327
		Electrodeposition					327
IODINE	BaI ₂ , CaI ₂ , CsI, KI, NaI	Vacuum evaporation	521- 740	1100- 1330		Useful release agents.	
IRIDIUM	Ir	Vacuum evaporation (Electron bombardment)	2454	1130	2556		2,6,14, 135
		Isotope separation					376
IRON *	Fe	Vacuum evaporation	1535	2750	1447	Fe deposited onto heated Cu foil to relieve stresses. Cu foil dissolved in Trichloroacetic acid + ammonia.	2,6,14, 135,271

IRON (CONT.)	Fe	Vacuum reduction of Fe_2O_3 and simultaneous evaporation of Fe. Alternatively reduction of the oxide in H_2 prior to evaporation					131,377
		Electrodeposition					37,378
LEAD *	Pb	Vacuum evaporation	328	1725	718		2,6,327, 379-382
		Vacuum reduction of PbO and simultaneous evaporation of Pb				Self supporting Pb films obtained by depositing onto Formvar and dissolving Formvar with chloroform	131
	PbCl_2	Electrodeposition					14
	PbCl_2	Vacuum evaporation	501	954			2,6
	PbS	Vacuum evaporation	1114	1281			
LITHIUM	Li	Vacuum evaporation	179	1330	514	Highly reactive metal which requires depositing in a vacuum transfer system to prevent rapid oxidation	2,6,135, 346,383-387
	Li	Electrodeposition					388
	LiF	Vacuum evaporation	870	1681		Stable lithium compound	2,6,135

MAGNESIUM*	Mg	Vacuum evaporation	651	1107	443 SUB	Self-supporting films obtained by depositing onto Formvar and dissolving Formvar with chloroform.	2,6,14, 135
		Vacuum reduction of MgO and simultaneous evaporation of Mg				Oxide reduced by Ta evaporation source, or by mixing MgO with Ti powder and forming a pellet.	135,136, 299,307, 348,389, 392,394, 455,466
MANGANESE*	Mn	Vacuum evaporation (Electron bombardment)	980	2097	1244	Mn deposited onto heated copper foil to relieve stresses. Copper dissolved in trichloroacetic acid + ammonia.	135,136, 348,389-394
	MnSO ₄	Electrodeposition					327,363
MERCURY	Hg	Absorption of Hg into thin Au backing					14,313
		Electrodeposition from Hg ₂ Cl ₂					314
	HgO	Electrospraying					311
	Hg ₂ Cl ₂ HgCl ₂	Vacuum evaporation	276	302			
	HgS	Vacuum evaporation	584				315-317
		Settling					83

MOLYBDENUM*	Mo	Vacuum evaporation (electron bombardment)	2622	5560	2533		2,6
		Sputtering					156
		Rolling					395
	MoO ₃	Vacuum evaporation	795	1155		Useful O ₂ target	135,346
NICKEL*	Ni	Vacuum evaporation	1455	2732	1510	Ni deposited onto heated Cu foil to relieve stresses. Cu foil dissolved in Trichloroacetic acid + ammonia.	2,6,14, 135,346
	NiCl ₂	Vacuum evaporation	1001	973 SUB			14
	Ni	Electrodeposition				Electrodeposit from either NiSO ₄ or NiCl ₂ solution	14,37, 135,363, 392
NIOBIUM*	Nb	Vacuum evaporation	2500	3300			2,6,396
	NbCl ₅	Vacuum evaporation	204	254			14
	Nb	Sputtering					310,400
NITROGEN	GaN	Vacuum deposition	300 SUB				135
	ADENINE [C ₅ H ₅ N ₅ ·NH ₂] MELAMINE [C ₃ H ₆ N ₆] AMMONIA (NH ₃)	Vacuum evaporation					135,346, 397
		Freezing ammonia vapour onto a cooled copper backing					398,399

NITROGEN (CONT.)	TiN Ta ₂ N ₃	Sputtering				Ti or Ta sputtered in a N ₂ atmosphere	148,149, 401,405
	Si ₃ N ₄	Sputtering				Si sputtered in N ₂ atmosphere	402
OSMIUM	Os	Vacuum evaporation (electron bombardment)	3000	~ 5000			403
		Electrodeposition					404,406, 408-410
		Isotope separator					376
OXYGEN	MoO ₃ , NbO, SiO ₂ , V ₂ O ₅ NO ₃	Vacuum evaporation				Oxide evaporation from Pt boat to prevent reduction of oxide	309,346, 407,411
	SiO ₂ . V ₂ O ₅	Anodising					71
	Ta ₂ O ₅	Anodising					412
	Nb ₂ O ₅	Anodising					310
	Al ₂ O ₃	Anodising					209,321- 326
PALLADIUM *	Pd	Vacuum evaporation	1555	1556	2927	Pd film floated from slide coated with release agent.	2,6,14, 135
		Electrodeposition					14
		Isotope separation					376

PHOSPHORUS	P	Vacuum evaporation	590	280			413-415
	P ₂ O ₅	Vacuum evaporation	585	300 SUB			14
	Zn ₃ P ₂	Vacuum evaporation	420	1100			135
PLATINUM*	Pt	Vacuum evaporation	1774	3800	2090	Floated from slide coated with release agent.	2,6,14
		Sputtering					156
		Electrodeposition					14,416, 418
		Isotope separation					376
POTASSIUM	K	Vacuum evaporation	66	744		Possible to get self supporting targets by stripping in Hexane	346,383
	K ₂ CO ₃	Vacuum evaporation	891				14
	KCl		776	1500			135,418
RARE EARTHS*	Pr, Nd, Sm, Eu, Gd, Tb, Dy, Er, Yb, Lu, Ho, La	Vacuum reduction of the oxide with simultaneous evaporation of the metal				Oxide reduced with La or Th powder. Metal pellet rolled to produce self-supporting target.	305,306, 308,348, 419,420
		Electrodeposition					417
RHENIUM	ReCl ₄	Vacuum evaporation		500			14
		Electrodeposition					
RHODIUM	Rh	Vacuum evaporation	1967	3800	2149		2,6
		Electrodeposition					14

RUBIDIUM	RbCl	Vacuum evaporation	715	1390			
	RbC ₂ H ₃ O ₂	Vacuum evaporation	246				421
RUTHENIUM	Ru	Vacuum evaporation (electron bombardment)	2250	3900			
		Electrodeposition					423,422,40
SCANDIUM *	Sc	Vacuum evaporation (electron bombardment)	1539	2727		Floated from slide coated with release agent.	135,383
	ScCl ₃	Vacuum evaporation	939	850 _{SUB}			14
SELENIUM *	Se	Vacuum evaporation	217	685	234	Floated from slide coated with release agent.	2,6,14,135,424-426
SILICON *	Si	Vacuum evaporation (electron bombardment)	1410	2355	1343	Floated from slide coated with release agent.	2,6,135,383,427
		Sputtering					156,428
		Electrochemical etching					429
	SiO, SiO ₂	Vacuum evaporation	1700	> 2000	1725		135,346
	SiO	Anodizing					71,439
SILVER *	Ag	Vacuum evaporation	961	2212	1047	Floated from slide coated with release agent.	2,6,135,346

SILVER (CONT.)	Ag	Electrodeposition					37,363
SODIUM	Na	Vacuum evaporation	98	892	580		346
	NaCl, NaI, NaBr	Vacuum evaporation	651- 801	1304- 1413		All compounds useful as release agents.	135
STRONTIUM	Sr	Vacuum evaporation	709	1384	800		346
	SrCO ₂	Vacuum evaporation					135
SULPHUR	S	Vacuum evaporation	113	445			431,432, 434,435
	Ag ₂ S	Vacuum evaporation	825				430,433, 436,437
	Sb ₃ S ₂ , CdS, PbS	Vacuum evaporation				PbS is the most stable compound at high beam currents.	135,438
TANTALUM	Ta	Vacuum evaporation (electron bombardment)	2996	5425	3350		137
	TaN	Sputtering					148,149, 152,401, 441
TELLURIUM	Te	Vacuum evaporation	452	1390	650		2,8,440
	TeCl ₄	Vacuum evaporation	224	380			14
THALLIUM	Tl	Vacuum evaporation	305	1457	606		2,6,135
		Electrodeposition					14

TALLIUM (CONT.)	$TlCl_3$	Electrodeposition					14
	Tl_2O_3	Vacuum evaporation	717	875			135,348
THORIUM	Th	Vacuum evaporation (electron bombardment)	1825	~ 4000	2196	Radioactive, α emitter	2,6,135
		Sputtering					152
	$ThNO_3$	Electrodeposition					442
THULLIUM	Tm	Vacuum evaporation	1545	1727	1120		14
TIN *	Sn	Vacuum evaporation	232	2260	1187	Floated from slide coated with release agent.	2,6,135, 346
		Vacuum reduction of SnO_2 and simultaneous evaporation of Sn					348
	$SnCl_2$	Electrodeposition					14,363
	$SnSO_4$	Electrodeposition					17
TITANIUM *	Ti	Vacuum evaporation	1727	3260	1546	Floated from slide coated with release agent.	2,6,135, 346,443
		Sputtering					156
	TiN	Sputtering				Also useful as nitrogen target	148,401

TUNGSTEN	W	Vacuum evaporation (electron bombardment)	3410	5927	3500		137,444
	WO ₃	Vacuum evaporation					407,411
	W	Sputtering					152,156
URANIUM	U	Vacuum evaporation (electron bombardment)	1132	3138	2350	Radioactive α-emitter	2,6,135, 152,445- 449
	UO ₂	Electrospraying					55,57, 178,450
	U ₃ O ₈	Electrodeposition					451-453
	U	Isotope separation					454
	U ₃ O ₈	Painting					103
	U	Rolling					395
	UF ₄	Vacuum evaporation	960				135

URANIUM (CONT.)	U	Sputtering					152
VANADIUM*	V	Vacuum evaporation	1697	3450	1888	V deposited onto heated copper foil to relieve stresses. Copper dissolved in trichloroacetic acid + ammonia.	2,6,135, 346
	V ₂ O ₅	Vacuum evaporation (electron bombardment)	690				71
ZINC*	Zn	Vacuum reduction of ZnO and simultaneous evaporation of Zn				Condensation difficult. Self-supporting films obtained by depositing onto Formvar and dissolving Formvar with chloroform.	135,343, 345,348, 392
		Vacuum evaporation	419	907	343 _{SUB}		2,6
		Sputtering					156
		Rolling					348
		Electrodeposition					363,14, 17
ZIRCONIUM	Zr	Vacuum evaporation (electron bombardment)	1842	4377	2000		2,6,135, 346
		Sputtering					152
<u>NB.</u> * Denotes targets that can be readily made self-supporting.							

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FIGURE CAPTIONS

- Fig. 1 Various possible ways of film growth: a) island growth; b) layer growth; c) island growth following layer growth; d) vertical stack growth (Carofolini²⁴).
- Fig. 2 Schematic section of a typical oil diffusion pump evaporation system. (Muggleton¹⁵).
- Fig. 3 Clean, high vacuum pumping system using sublimar and sputter-ion pumps.
- Fig. 4 Bent beam electron bombardment source (Varian Associates).
- Fig. 5 Duoplasmatron ion source used for producing nuclear targets by focussed ion beam sputtering (General Ionex Corporation).
- Fig. 6 Fine beam saddle field ion source (Ion Tech Ltd).
- Fig. 7 Schematic section of fine beam saddle field ion source.
- Fig. 8 Steps in the preparation of self-supporting films (Muggleton¹⁵).
- Fig. 9 Self-supporting carbon foils; a) being floated from glass microscope slides; b) being picked-up on target frame. . (Muggleton¹⁵).
- Fig. 10 Illustration of a grossly non-uniform target and the effect on energy resolution ΔE . (Erskine²²⁰).
- Fig. 11 Illustration of various types of target non-uniformities. (Erskine²²⁰).
- Fig. 12 Photograph of ethylene cracking apparatus for producing carbon films using a d.c. glow discharge.
- Fig. 13 Schematic diagram of the ethylene cracking apparatus shown in Fig. 12.
- Fig. 14 Schematic diagram of the apparatus used in the simultaneous reduction and evaporation of rare earth elements.
- Fig. 15 Diagram demonstrating how a selenium film is picked-up on a large frame to give thermal protection to the target by sandwiching the selenium between two carbon films.

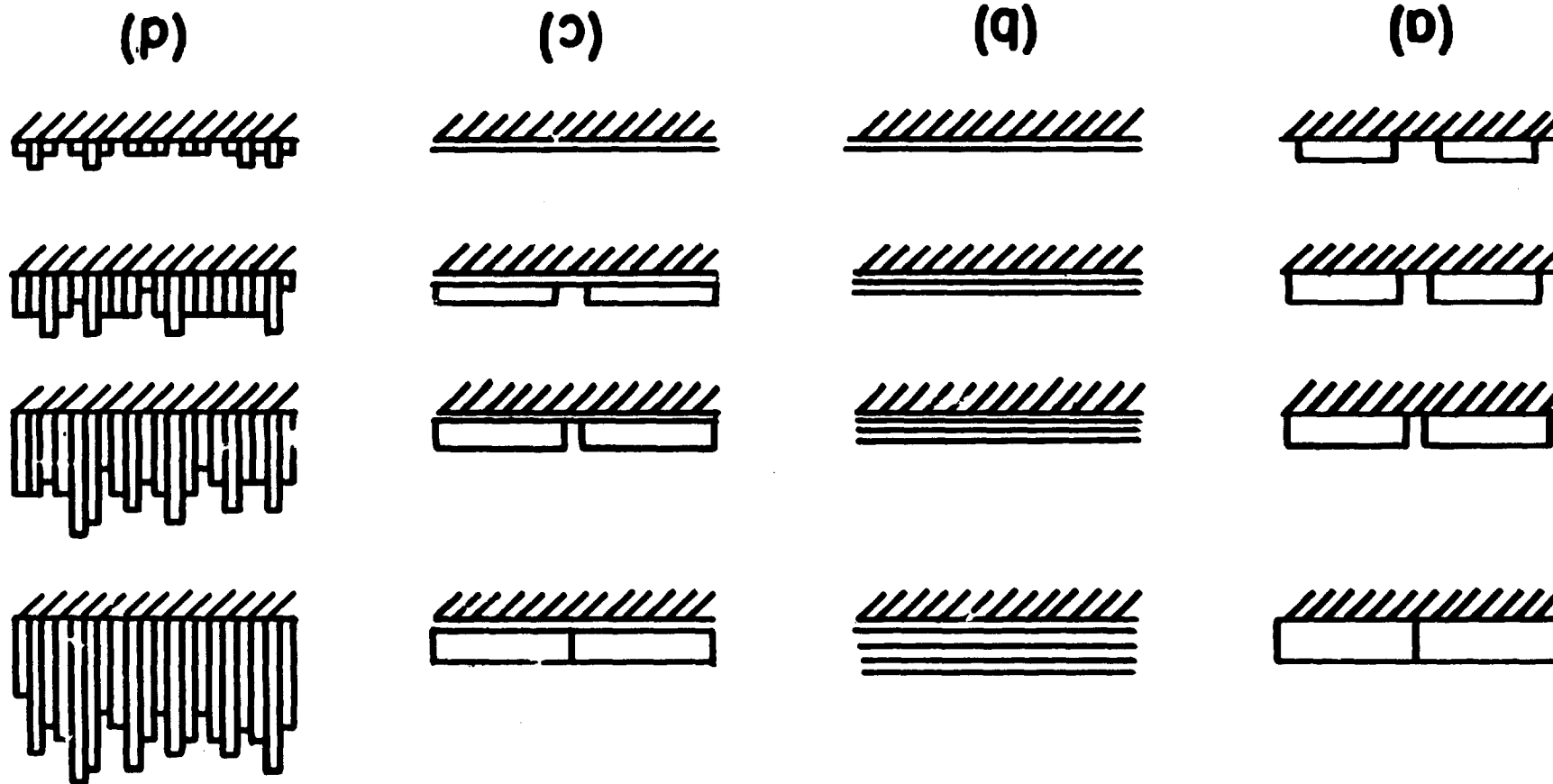


Figure 1

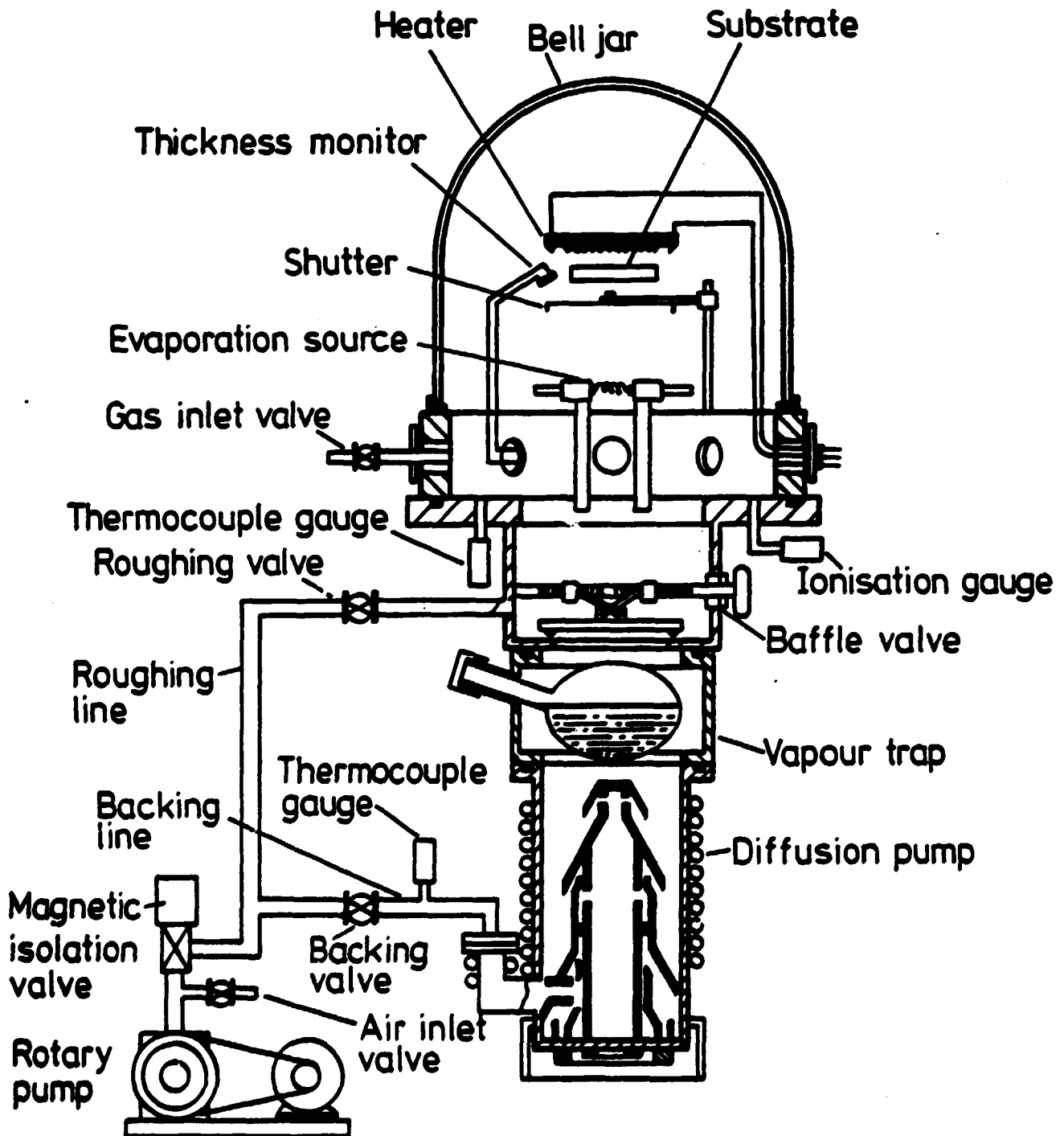


Figure 2

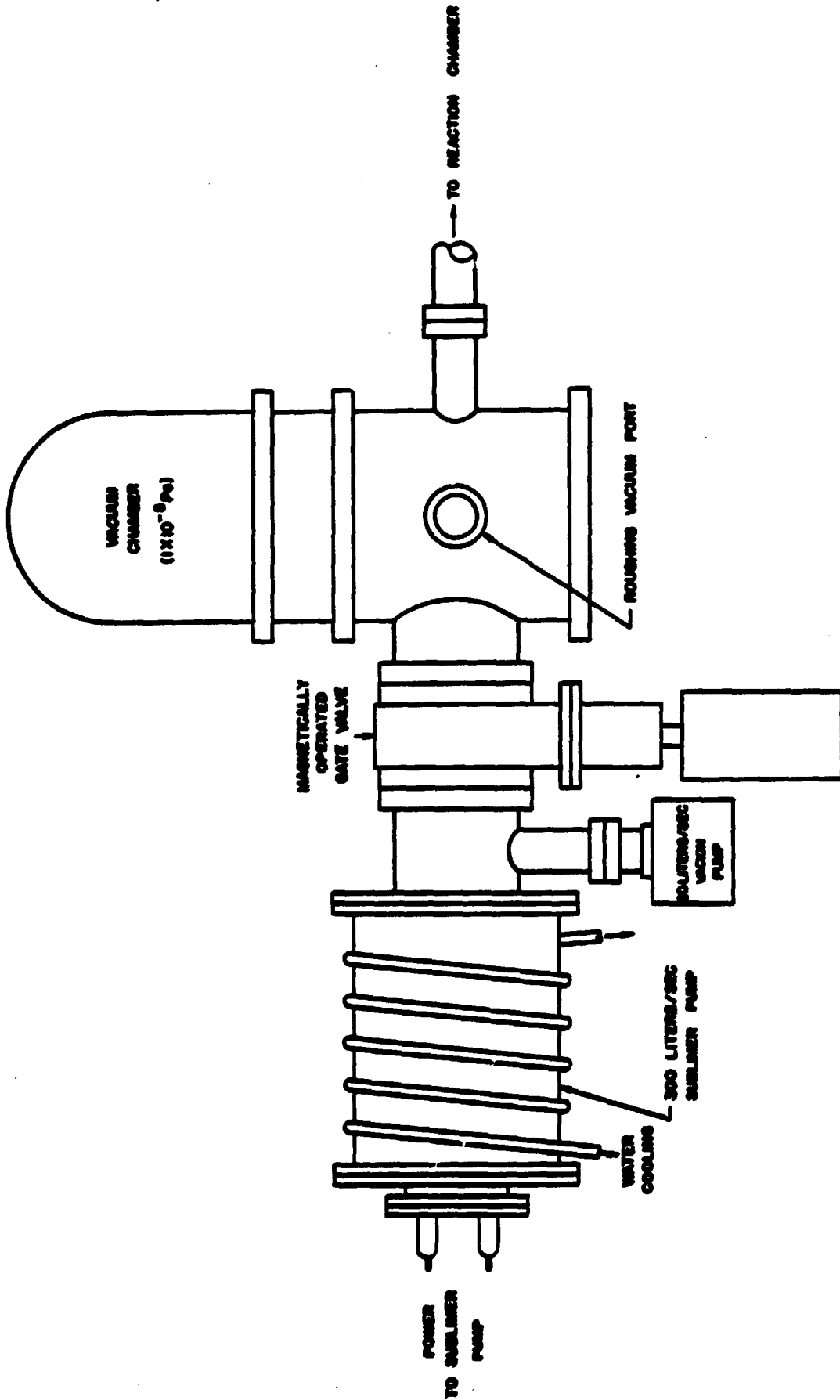


Figure 3

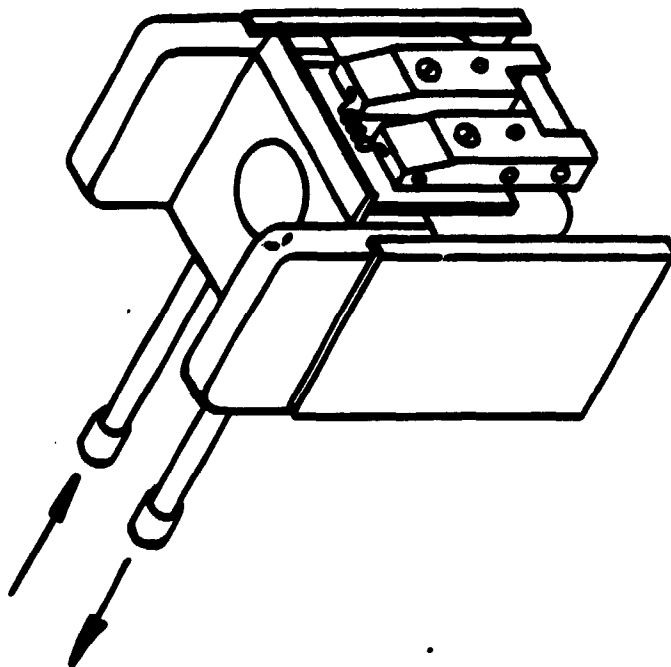
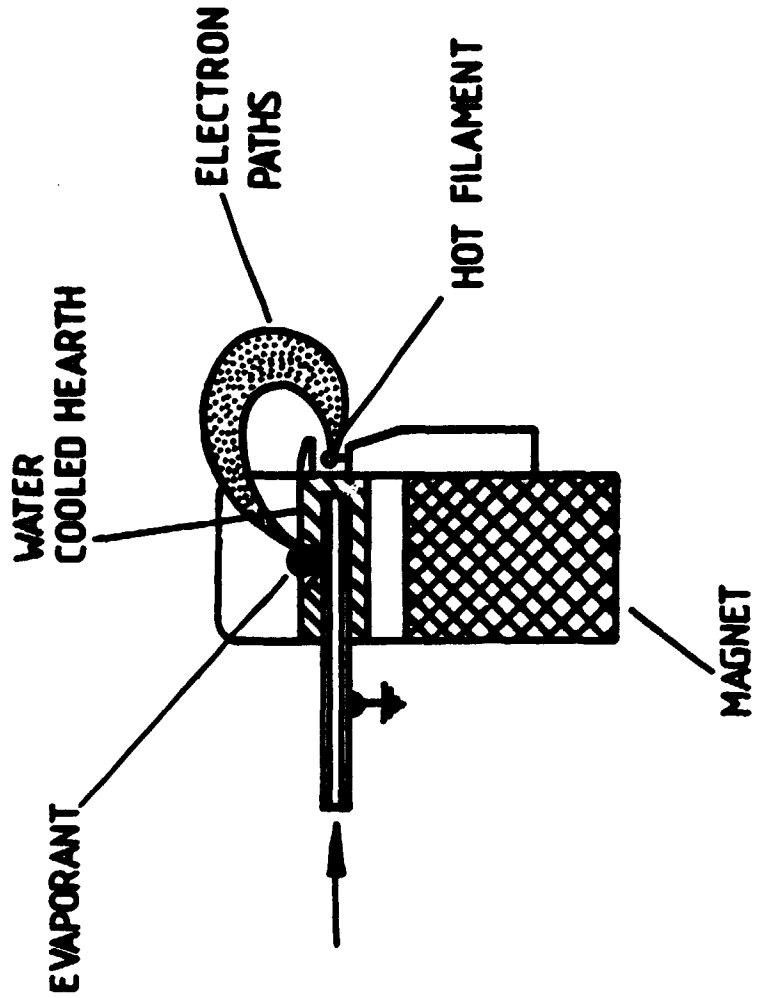


Figure 4

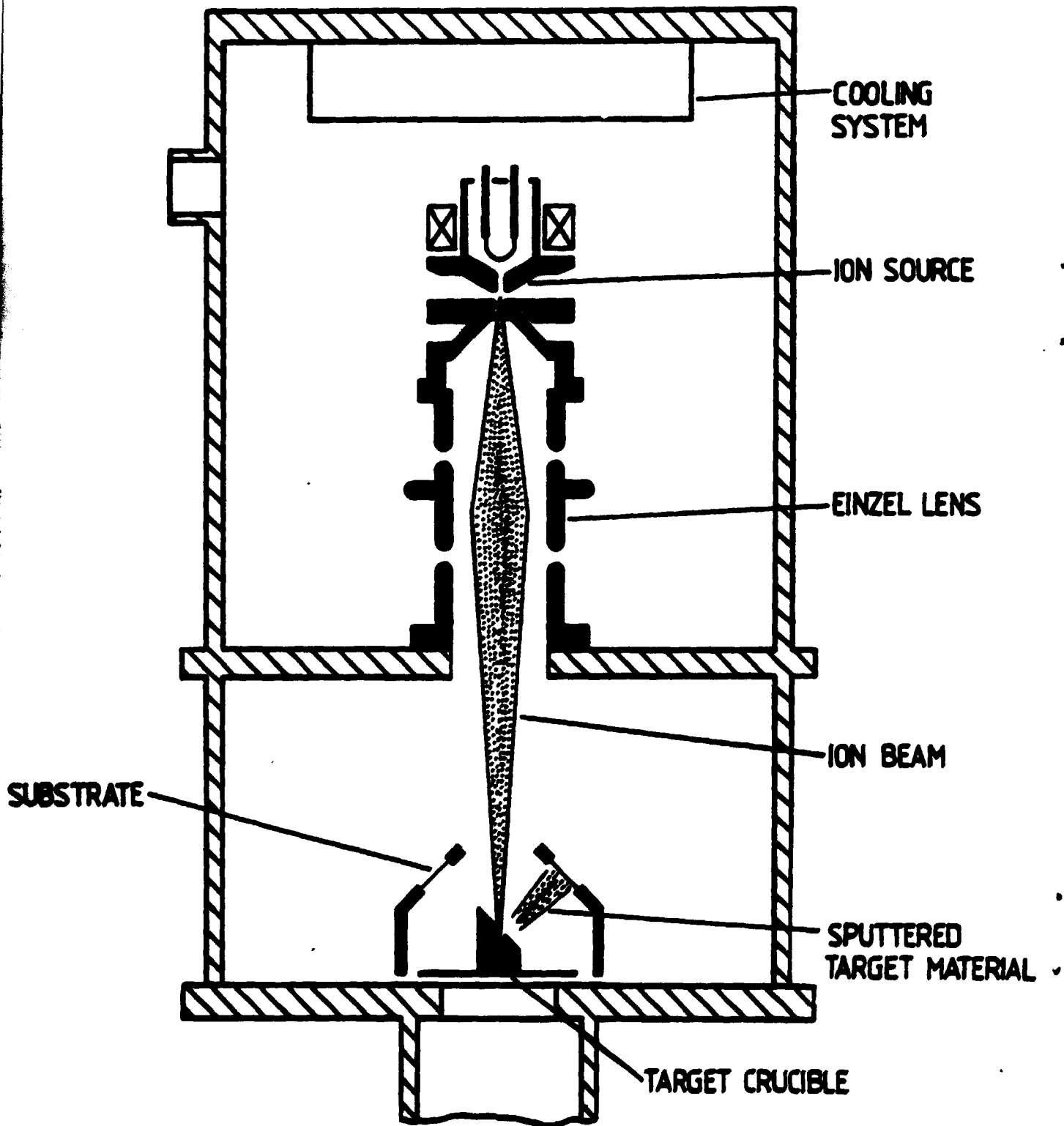


Figure 5

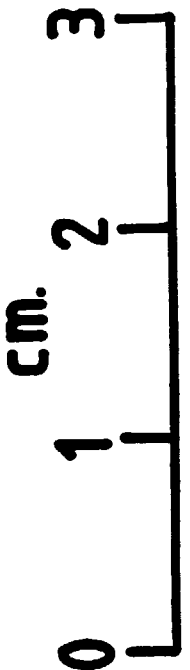
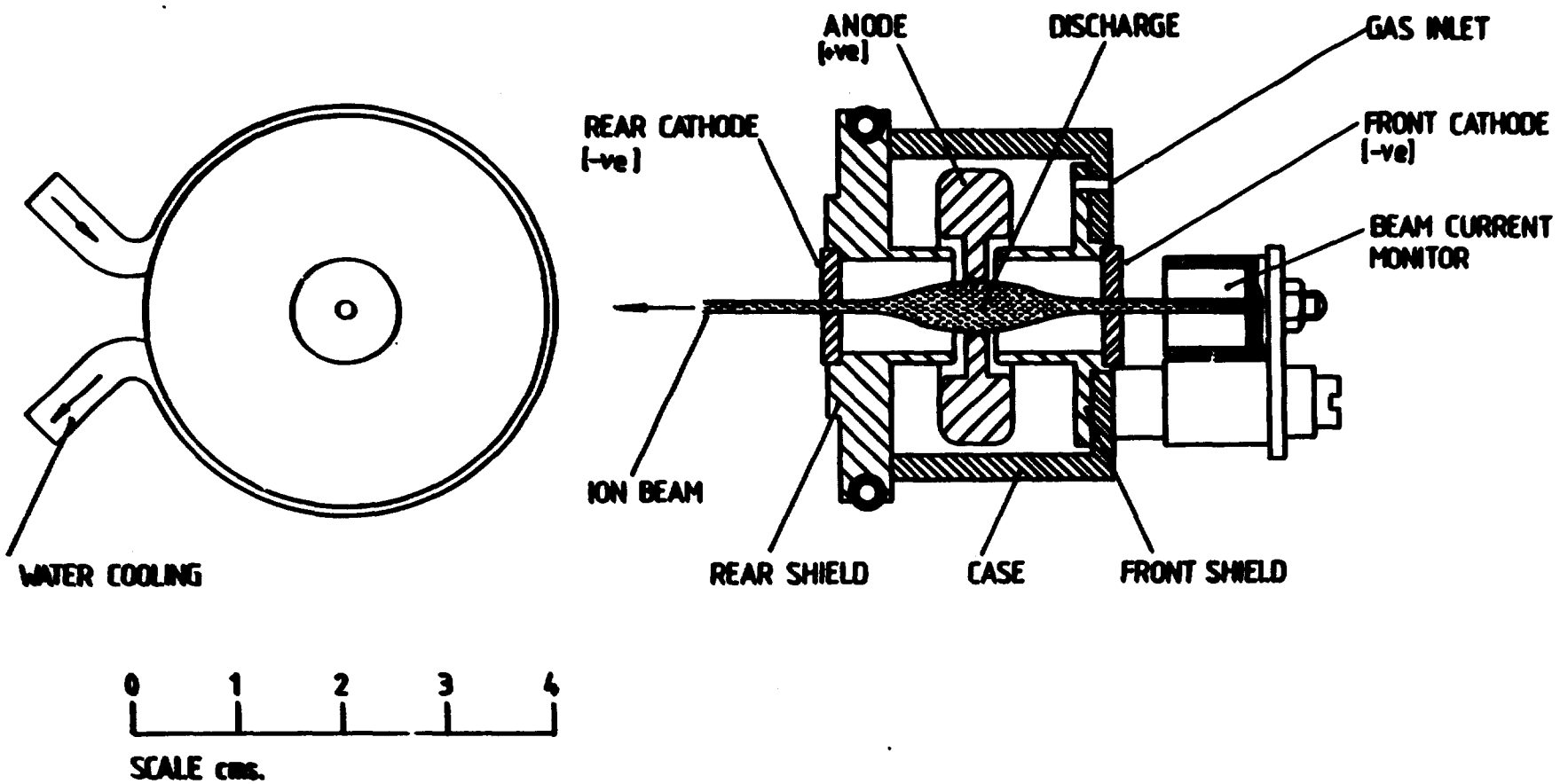


Figure 6

Figure 7





Clean microscope
slide



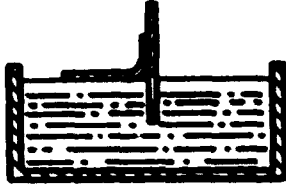
Slide coated with
soluble film



Slide+soluble film
+ evaporated film



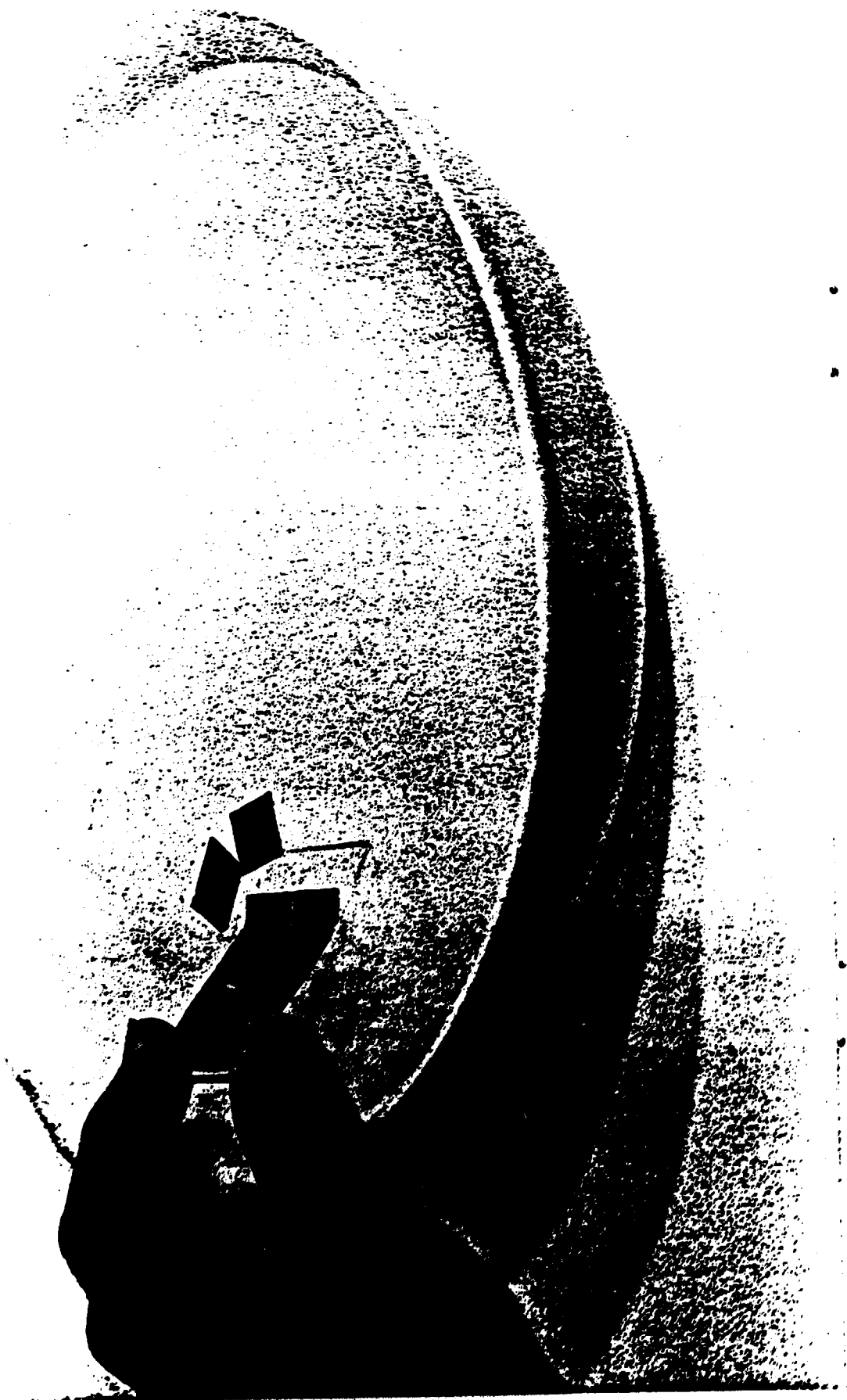
Evaporated film
floated on water
bath

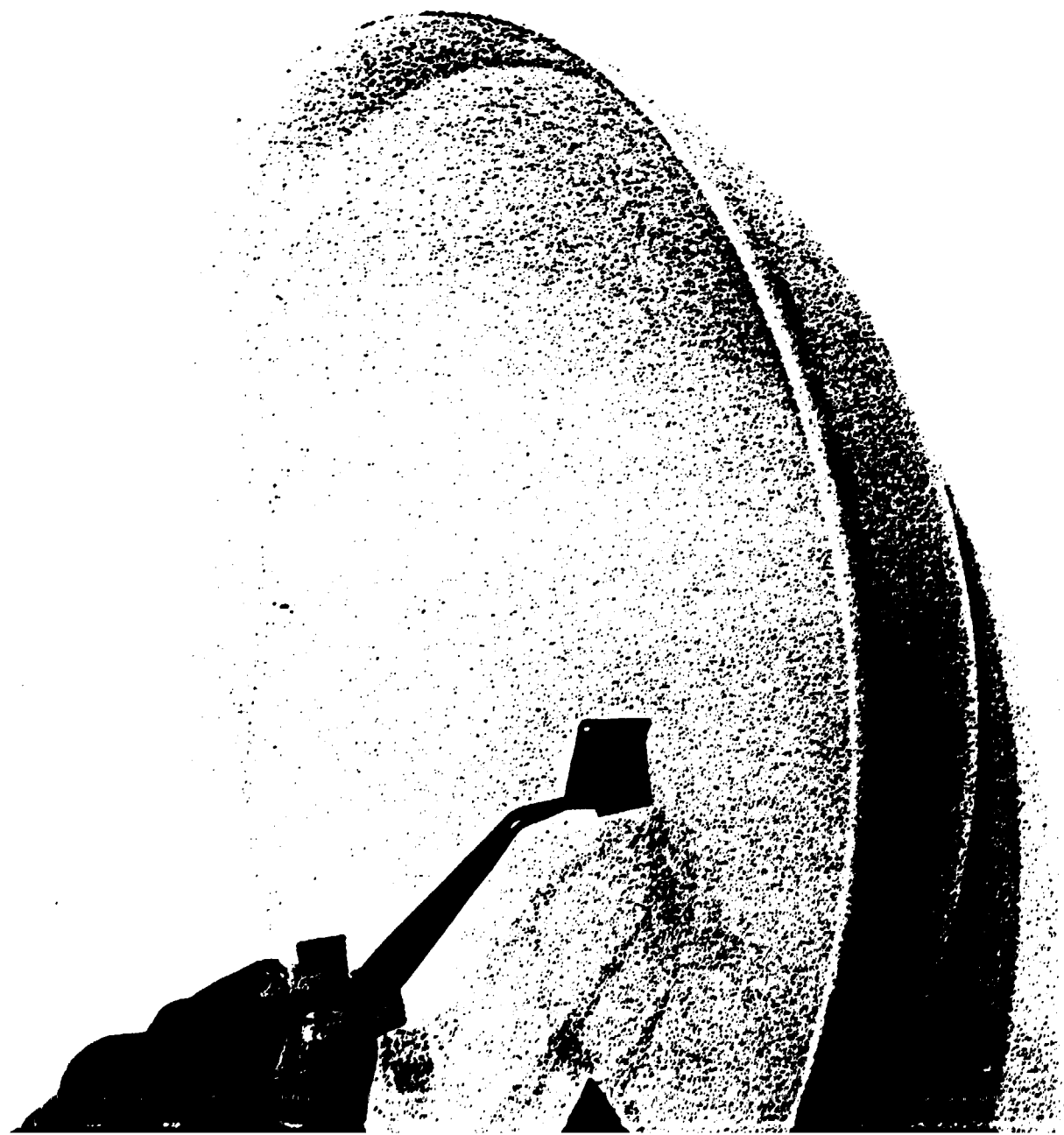


Evaporated film picked
up on target frame



Film mounted on
target frame





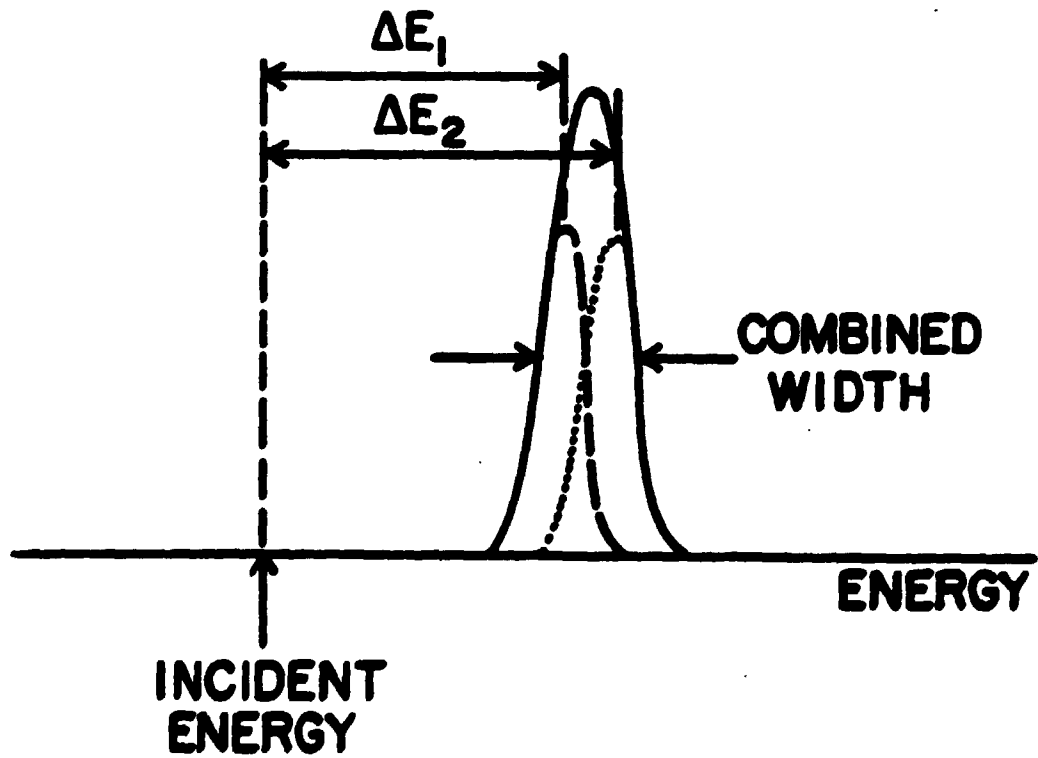
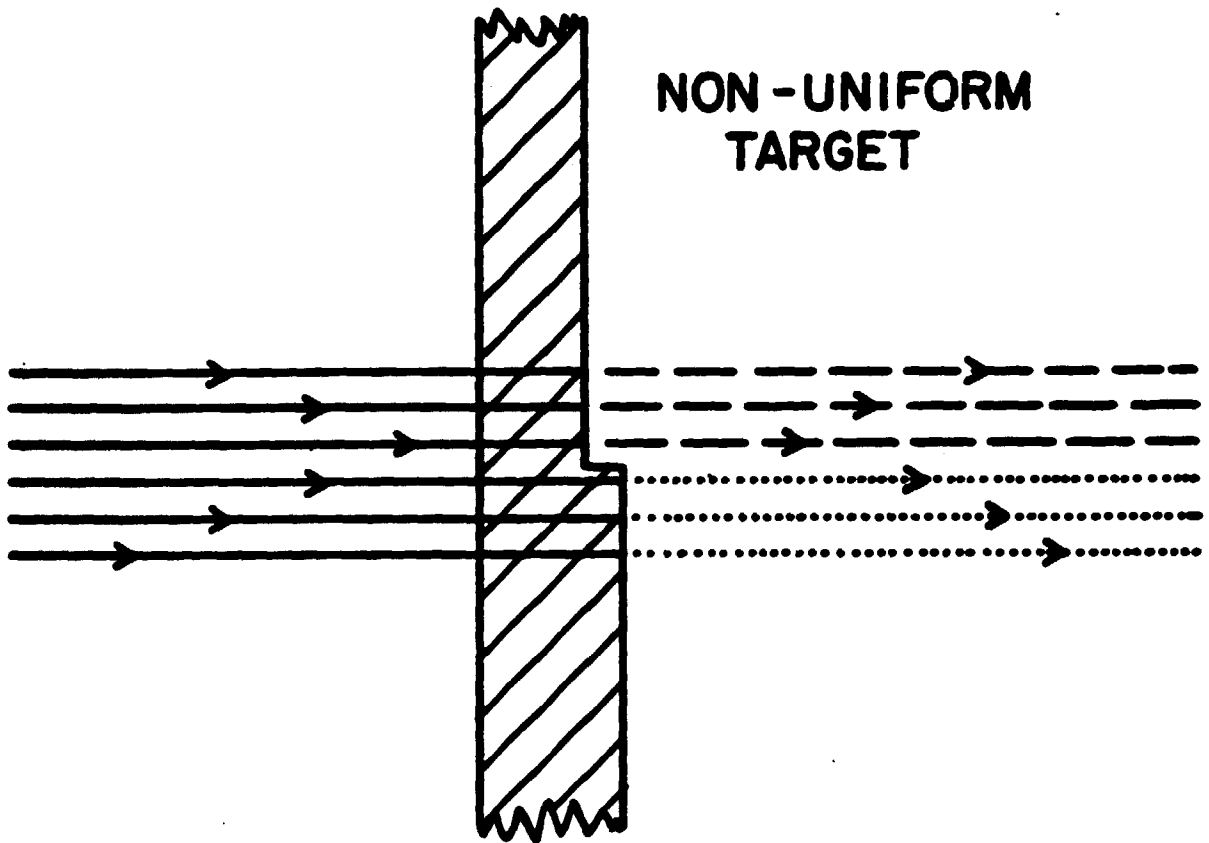
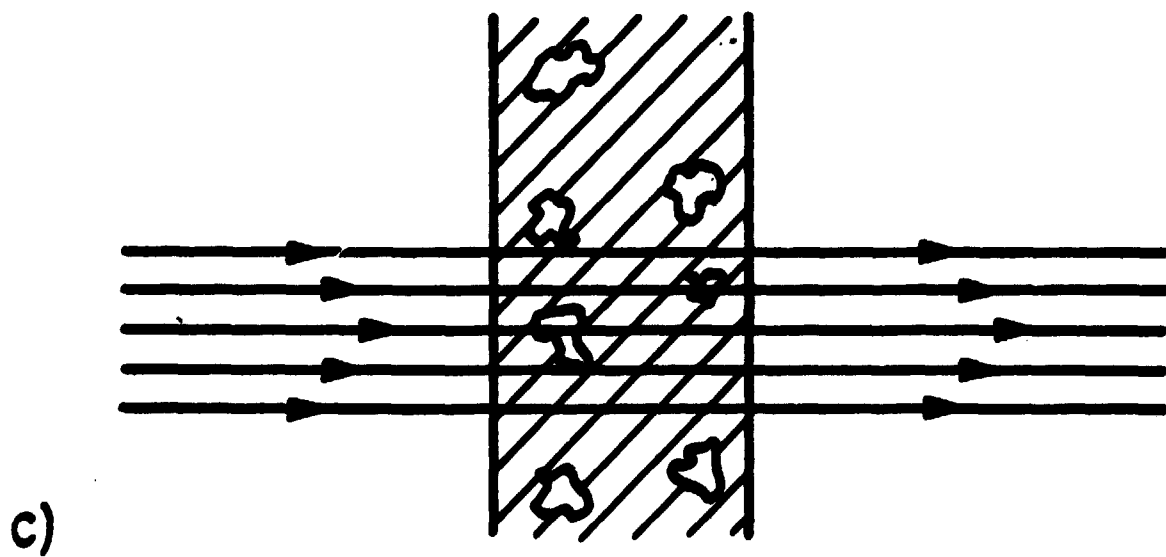
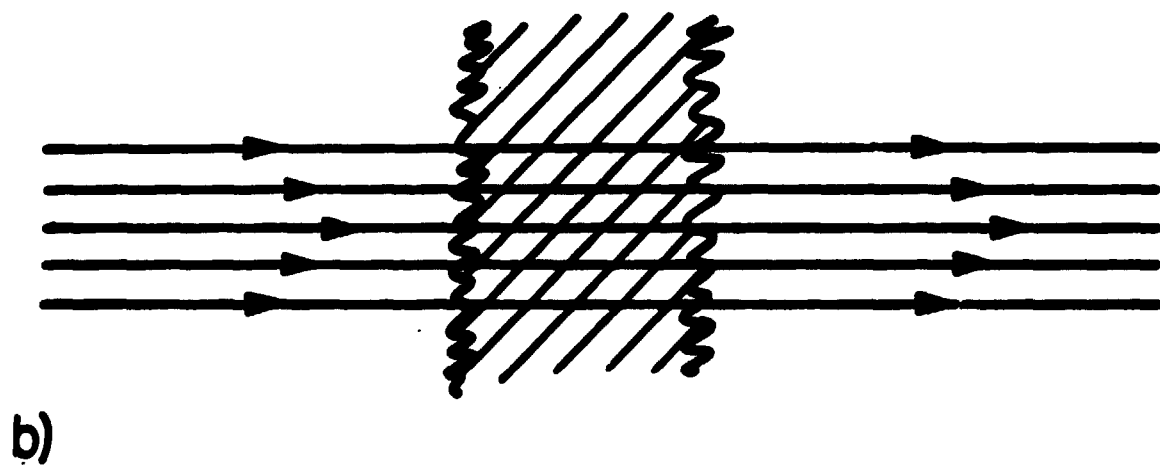
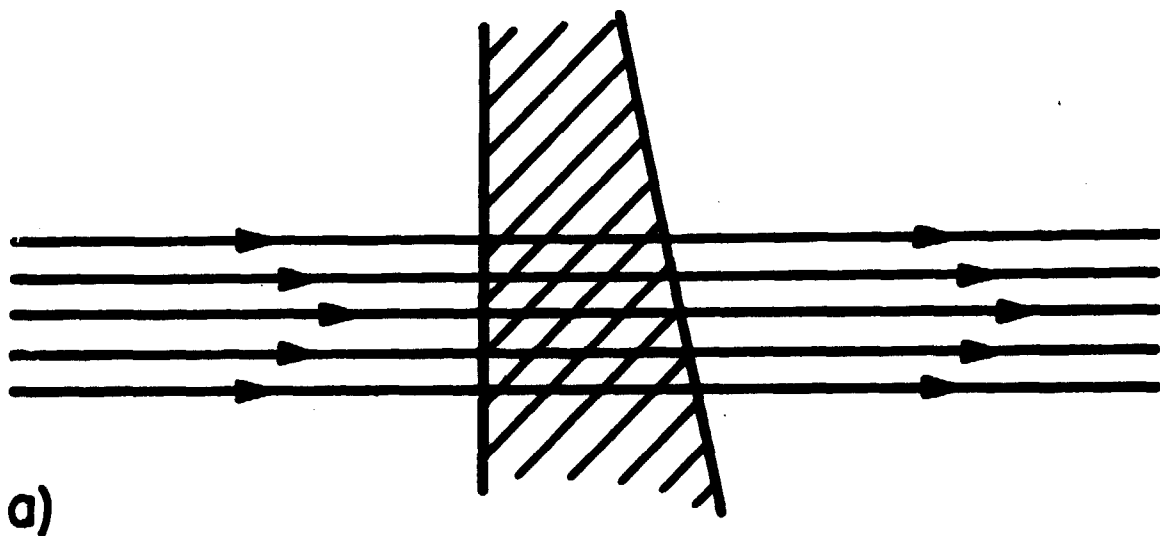
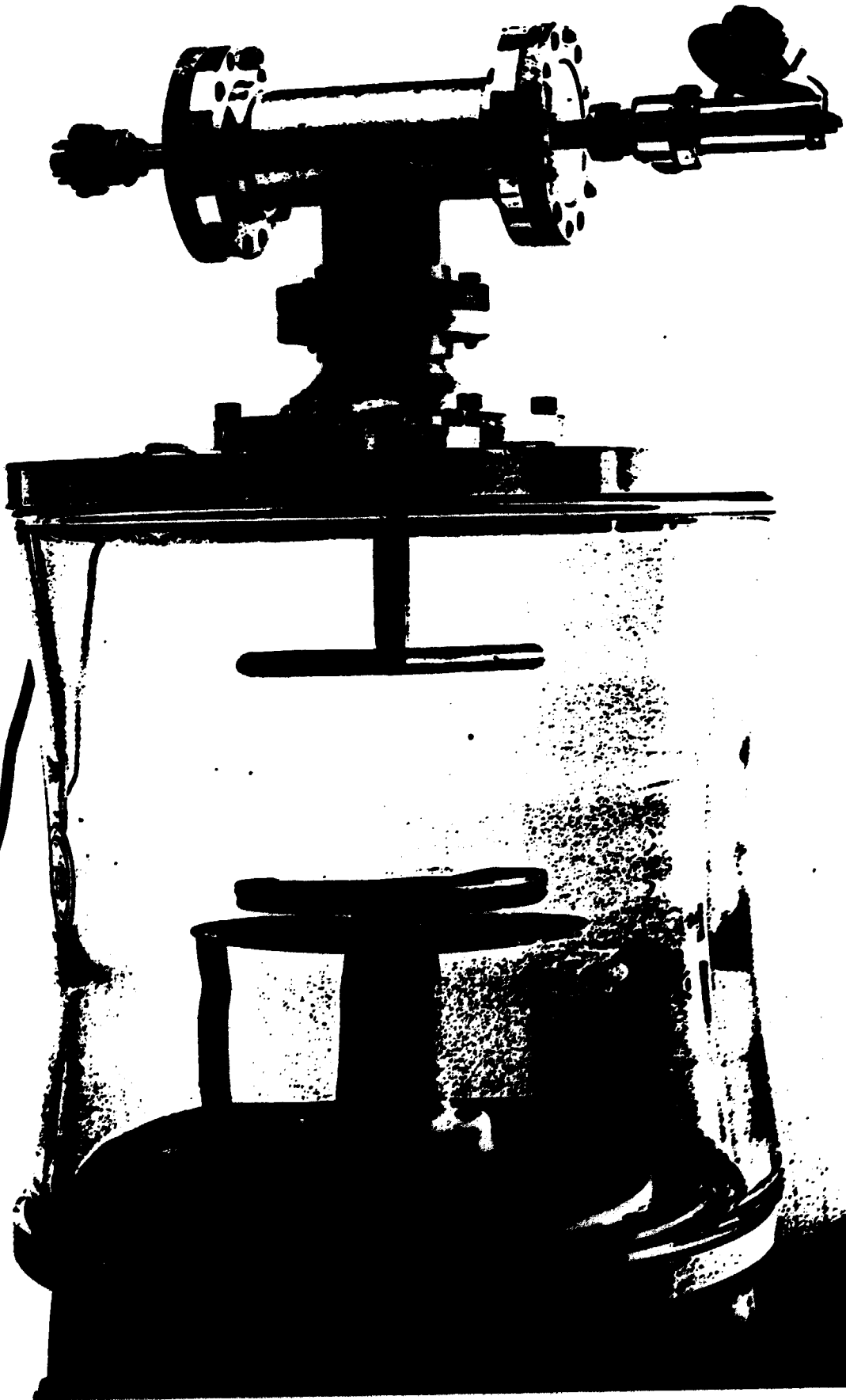
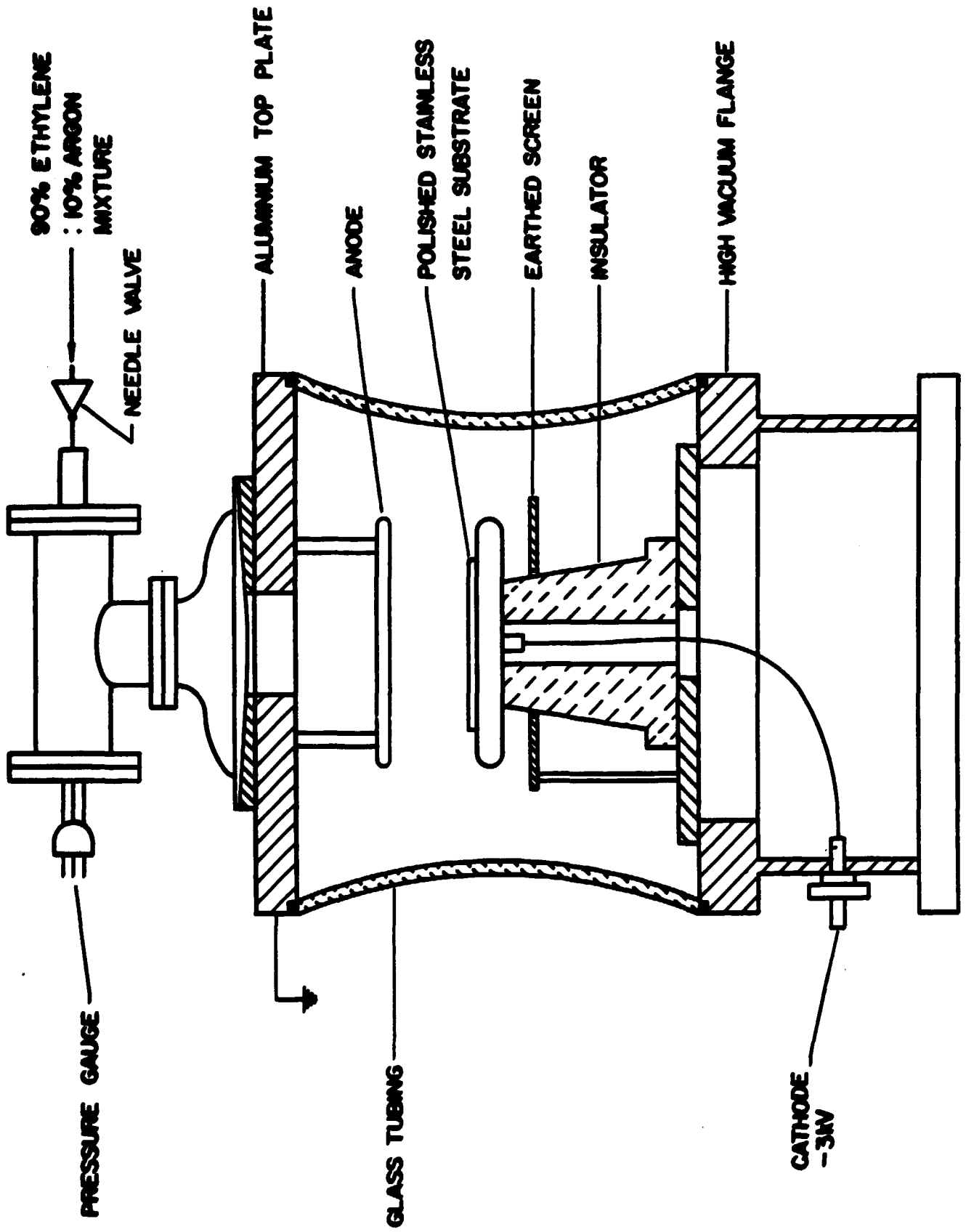
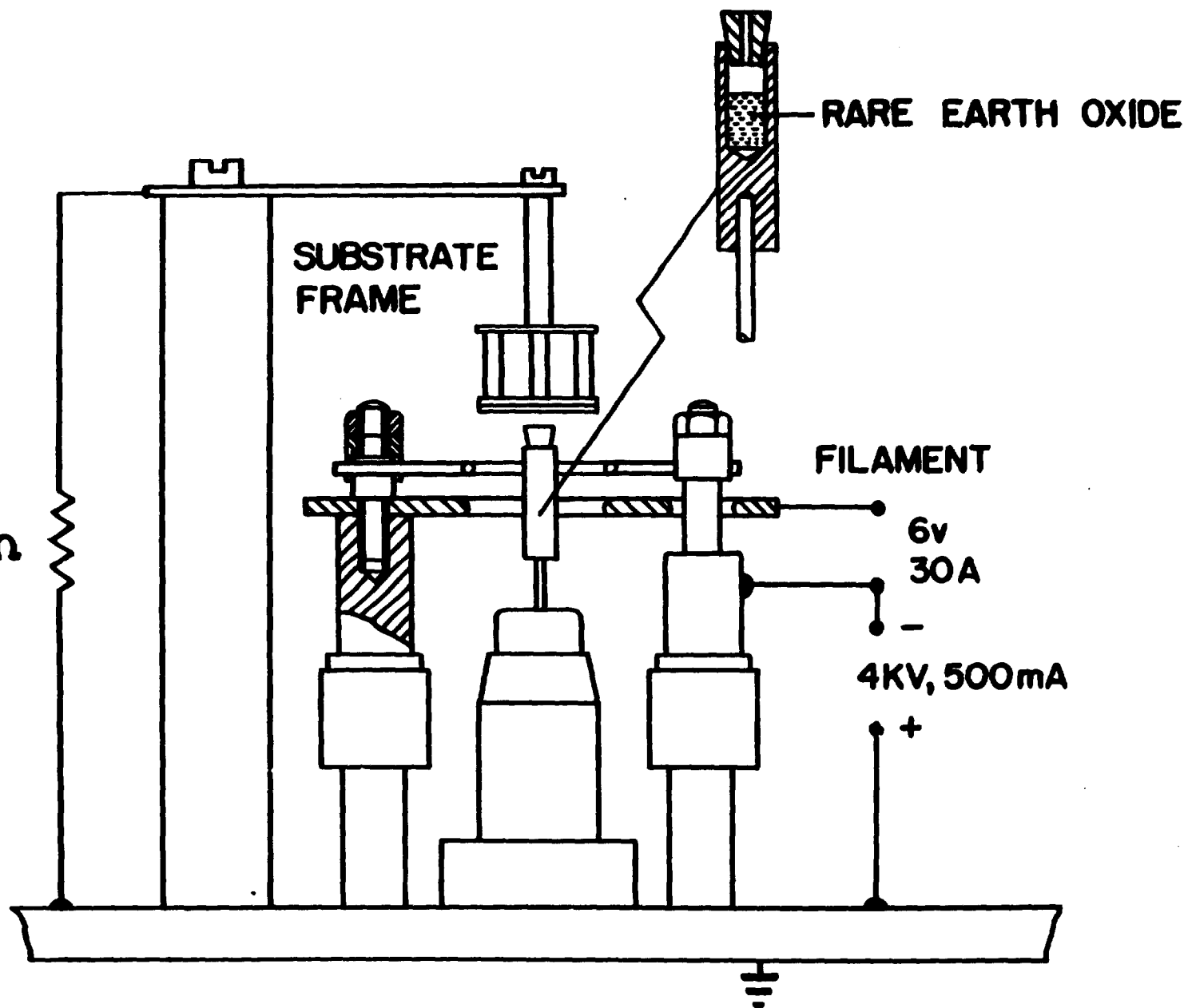


Figure 10









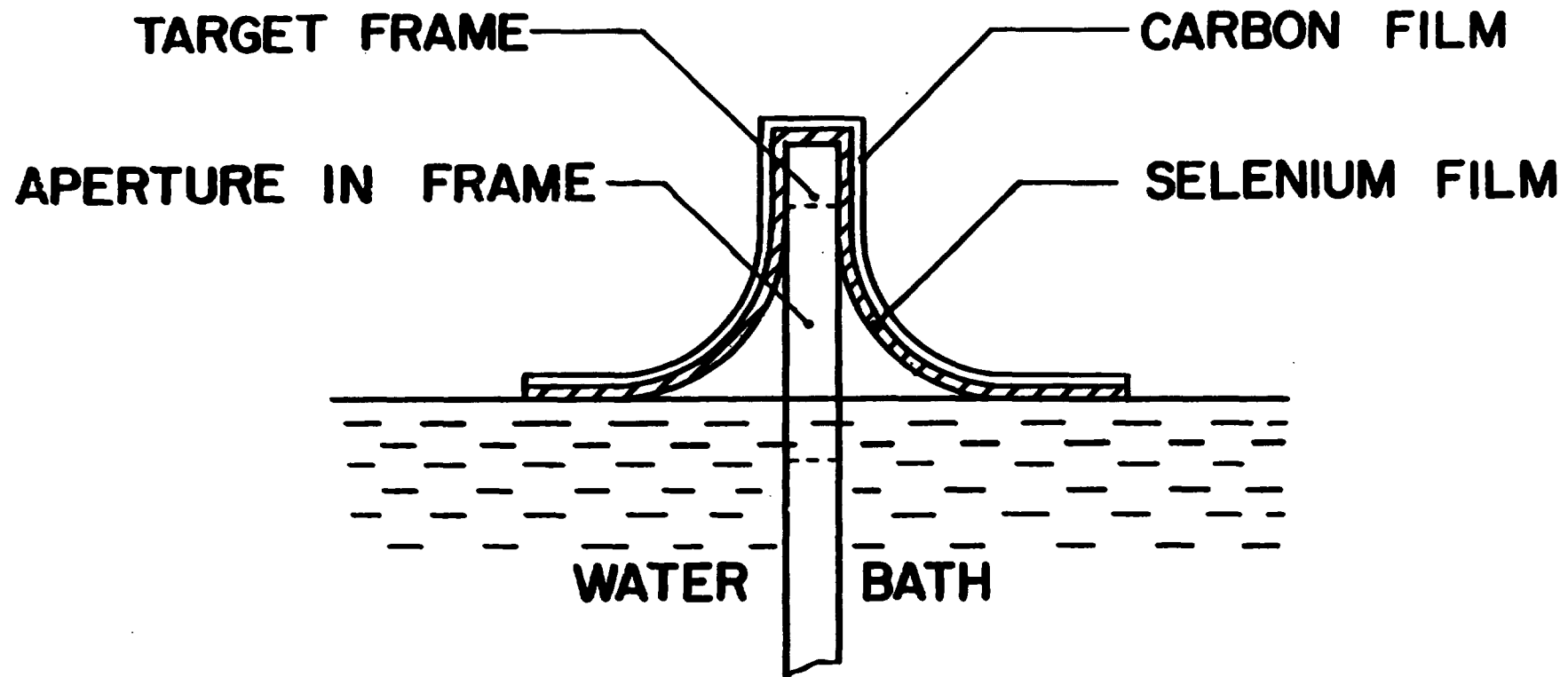


Figure 15