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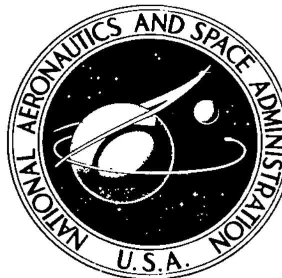

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# A PROPOSED METHOD FOR REGENERATION OF NEUTRON PRODUCING TARGETS, WITHIN AN ACCELERATOR, BY ION SPUTTERING TECHNIQUES

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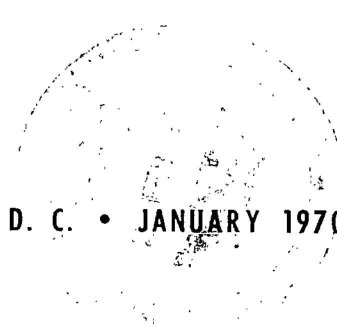

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16. Abstract

A proposed sputtering electrode system design is presented that makes possible regeneration of targets within an accelerator. The formation of a tritide target from an applied titanium film is also discussed. The design of an X-ray fluorescence detector for measuring the thickness of material deposited or removed from the target is suggested. Finally, titanium and titanium deuteride etch rates are presented as obtained by a sputter electrode system mockup for a rotating target assembly under development.

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# A PROPOSED METHOD FOR REGENERATION OF NEUTRON PRODUCING TARGETS, WITHIN AN ACCELERATOR, BY ION SPUTTERING TECHNIQUES

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## SUMMARY

A device is proposed for the regeneration of titanium tritide targets within an accelerator. Regeneration of a target is possible by using a sputter etch method to remove a tritium depleted film and/or other contamination from the target surface. A logical extension of the sputter etch method to target fabrication within the accelerator is also discussed, and a compact sputtering electrode design is presented that is applicable to a rotating target system. The design permits the use of existing, low pressure, sputtering techniques for the application of high purity target films. Both plating and etching operations can be performed. A rotating target system using this technique is presently under development.

Experimental sputter etch rates for titanium and titanium deuteride were measured at an argon pressure of 50 millitorr and an anode-to-cathode distance of 2.54 centimeters. For a variation in cathode potential from 1200 to 2500 volts, the measured titanium deuteride controlled etch rates varied from 0.016 to 0.164 and from 0.026 to 0.347 milligrams per square centimeter per hour, respectively.

The design of an X-ray fluorescence detector for measuring target film thickness is suggested. The measurement depends on the attenuation of the characteristic 8-keV X-rays of the accelerator target copper substrate. The 8-keV X-rays are excited by a radioisotope source that emits higher energy bremsstrahlung X-radiation.

## INTRODUCTION

Ion accelerators of 500 keV or less use the $T(d,n)He^4$ reaction for neutron production to obtain large neutron yields. Tritides of metals like titanium, erbium, or zirconium are commonly used as target materials. These tritide targets have had notably short lifetimes, and considerable effort has been expended in attempts to improve the target

lifetimes (refs. 1 to 3). It has been suggested that the primary cause of tritide target deterioration is the dilution and replacement of the surface tritium atoms by the bombarding deuterium ions with the release of the tritium gas to the accelerator system (refs. 4 and 5). Designers of small, sealed tube, neutron generating accelerators have been able to extend target life substantially by using a mixed deuterium and tritium gas system whereby the target is continuously replenished. However, the neutron yield has been low (refs. 4 and 6). Because of the tritium hazard associated with mixed beam operation for larger, high current, ion accelerators, other less effective techniques have been used to extend target lifetimes. For example, rotating targets appear to offer an advantage in lifetime over a disk target (refs. 7 and 8), on a milliampere-hour basis. The rotating target also offers less accelerator down time for target changing and a lower fabrication cost per unit target area (refs. 4 and 9). However, it is more complex and costly than a simple disk target.

An additional problem associated with the fabrication of accelerator targets, whether rotating or stationary, has been the poor adhesive quality of the vacuum evaporated films (refs. 5 and 10). This has led to inadvertent target peeling and flaking.

While no one has yet been able to produce a long lived, high current, tritide target, a search of the available literature does indicate a possible direction in which to proceed. Within the framework of current technology it is possible that substantial improvement could be made in the following areas:

(1) Adhesive strength of the bond between applied film and copper substrate
(2) Elimination of absorbed surface impurities because of handling problems during fabrication
(3) Improved quality control

We are therefore proposing to extend target life by utilizing present sputtering technology in the design of an accelerator target which can be fabricated and regenerated in place. Existing sputtering techniques can provide a superior bond between the titanium and copper substrate (target backing). Fabrication in place under good vacuum conditions will substantially reduce surface contamination and provide a level of quality control which has been lacking.

A detector design is suggested that determines the thickness of target films by an X-ray fluorescent method. The attenuation of characteristic 8-keV X-rays from the copper substrate through a titanium coating is used to measure the coating thickness. These X-rays are excited by a radioisotope that emits higher energy bremsstrahlung x-radiation.

## ION SPUTTERING

Target films have customarily been fabricated by the vacuum evaporation method (refs. 5 and 10 to 14). A factor partly responsible for the poor bonding property of these

films is the low thermal energy of the evaporated atoms (ref. 15). Atoms sputtered from a material, on the other hand, have an average energy of approximately 10 eV (ref. 16). An improvement in the adhesive strength of the bond between the applied film and the substrate is found to occur (refs. 15 and 17) with sputtering techniques.

The ions used for low energy sputtering are usually formed in a glow discharge. In the discharge, positive ions are accelerated across the Crookes' dark space (cathode dark space) and bombard the cathode (ref. 18). The impinging ions lose energy by a momentum transfer to atoms of the cathode. An atom is sputtered from the cathode surface when the energy transferred to it exceeds the binding energy of the atom to the surface (ref. 19).

In a diode configuration, a glow discharge can be sustained by field emission of electrons from the cathode over an argon pressure range of from 10 to 100 millitorr. By increasing the supply of electrons to a glow discharge by a thermionic filament, the discharge will occur at pressures as low as 0.1 millitorr. Excitation of the plasma by a radiofrequency field will cause the glow discharge to occur at argon pressures as low as 1 millitorr.

Anode-to-cathode spacings must be greater than the width of the Crookes' dark space for a stable glow discharge to form and sputtering to occur (ref. 18). In all other parts of the sputtering system, where sputtering is not desired, the gap distance between a cathode and any surface of the system must be narrower than the width of Crookes' dark space. We have found experimentally that a gap of approximately 0.3 to 0.6 centimeter is satisfactory for a system that is designed to sputter over an argon pressure range from 0.1 to 100 millitorr. The larger gap is preferable if sputtering in the pressure range of from 50 to 100 millitorr is not contemplated.

## APPLICATION OF SPUTTERING TECHNIQUES TO ACCELERATORS

We propose the use of sputtering techniques, combined with a high speed rotating target, to extend accelerator target lifetimes. The incorporation of a sputtering electrode within the accelerator target assembly will allow removal of a depleted layer of target material from the surface, as well as the application of titanium films for the formation of new targets. Oxide films, carbon deposits, and other contaminants can also be removed by the sputter electrode.

### Sputtering Methods

A sputtering electrode design is presented which is compatible with four different methods of applying voltage to the electrodes. The design provides an ability to sputter

TABLE I. - APPLICABLE SPUTTERING METHODS FOR ACCELERATOR TARGET PREPARATION

| Sputtering method | Argon gas pressure,[a] millitorr | Method may be used for - | | Remarks |
|---|---|---|---|---|
| | | Target cleaning | Target fabrication | |
| Cold cathode direct current sputtering | 10 to 100 | Yes | Not recommended | Good for target cleaning only. Simple two-electrode design. Requires only one dc high voltage power supply. Sputter electrode material may be dissimilar to target material. |
| Asymmetric alternating current sputtering, two electrodes | 10 to 100 | Yes | Yes[b] | Can be used for both target cleaning and fabrication, but sputter electrode and target must be of same material. Requires only one ac high voltage power supply. |
| Asymmetric alternating current sputtering with two electrodes, and anode and thermionic cathode | 0.1 to 1.0 | Yes | Yes[b] | Requires addition of an ac filament power supply and a dc high voltage power supply. |
| Direct current bias sputtering with three electrodes and thermionic cathode | 0.1 to 1.0 | Yes | Yes | Sputter electrode and target must be of same material. Requires one ac filament power supply and three dc high voltage power supplies. |
| Radiofrequency sputtering with two electrodes | 1 to 10 | Yes | Not recommended | Most important use is in sputtering of dielectric materials. |
| Radiofrequency sputtering with two electrodes, and anode and thermionic cathode | 0.1 to 1.0 | Yes | Yes[b] | Also can be used for target fabrication with use of gettering films. One ac filament power supply and one dc high voltage power supply. |

[a]Use of high purity argon gas is suggested for all sputtering methods.
[b]Use of gettering film on shutter (e.g., titanium) is necessary to minimize film comtamination.

over an argon gas pressure range from 0.1 to 100 millitorr. The four sputtering methods are (1) cold cathode dc sputtering (ref. 20), (2) asymmetric ac sputtering (ref. 21), (3) hot cathode dc bias sputtering (ref. 22), and (4) radiofrequency (rf) sputtering (ref. 23). All four sputtering methods are presented to show the specific advantages, as well as the limitations, of each technique. The methods and associated comments are listed in table I. A sketch of the electrode is shown as applied to a rotating target in figure 1.

The cold cathode dc sputtering method requires argon gas pressures from 10 to 100 millitorr. Only two electrodes (sputter electrode and accelerator target) and one high

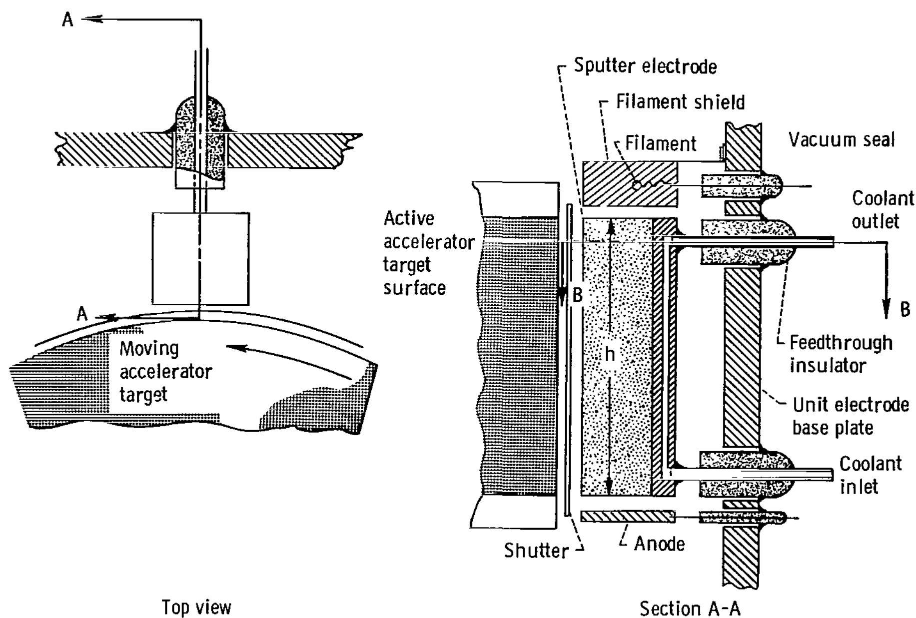


Figure 1. - Sputtering electrode assembly.

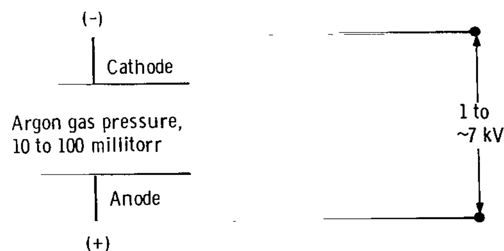


Figure 2. - Electrical schematic for cold cathode direct current sputtering method.

voltage power supply are required. An electrical schematic for the method is shown in figure 2. Theuerer and Hauser (ref. 24) have deposited superconducting niobium and tantalum films by this method, but their design included the entire enclosure of the substrate by gettering surfaces. In addition, the substrate was thoroughly outgassed by baking at $1000^{\circ}$ C. Without extensive use of gettering surfaces, this sputtering method is limited to etching operations. For example, the method is useful for removal of tritium depleted surface layers from a thick metal tritide target fabricated outside of the accelerator.

The asymmetric ac sputtering method requires the same electrode configuration and argon gas pressures as the cold cathode dc method. An electrical schematic of the electrode configuration and ac power supply are shown in figure 3. During sputter deposition of a target film by the asymmetric ac method, the accelerator target is under continual ion bombardment, which provides self-cleaning and degassing of the surface for improved

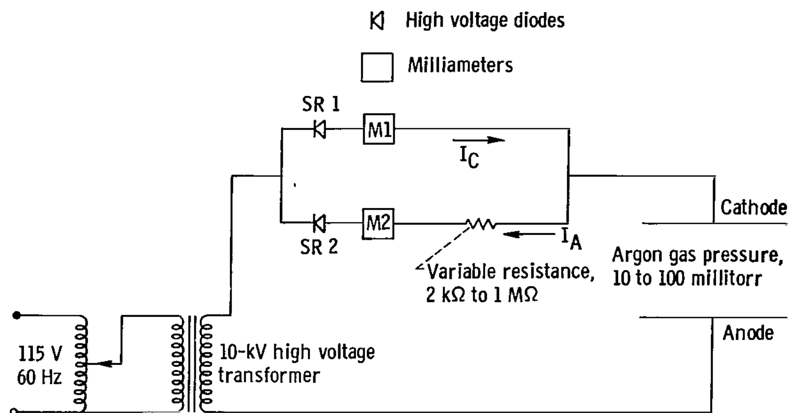


Figure 3. - Electrical schematic for asymmetric alternating current sputtering method.

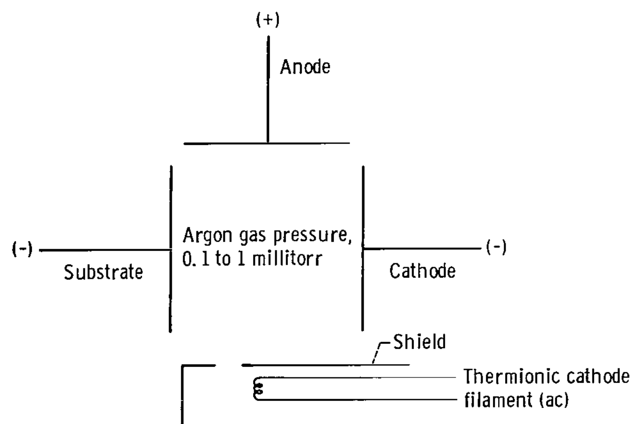


Figure 4. - Electrical schematic for hot cathode direct current bias sputtering method. Negative bias applied to cathode is larger than that applied to substrate.

film purity. Vratny and Harrington deposited tantalum films of improved purity by this method (ref. 21).

The electrical schematic for the hot cathode dc bias sputtering method is shown in figure 4. Three high voltage power supplies and an ac low voltage filament supply are required. The thermionic cathode and anode maintain the glow discharge over an argon pressure range from 0.1 to 1 millitorr. A negative bias is applied to both the accelerator target and the sputter electrode. A net deposition of material occurs at the accelerator target because its negative bias is much lower than that applied to the sputtering electrode. The ion bombardment of the accelerator target during deposition prevents gettering and burial of active gas contaminants by the target. Seeman has deposited superconducting niobium films of high chemical purity by this method (ref. 22). Maissel and Schaible have

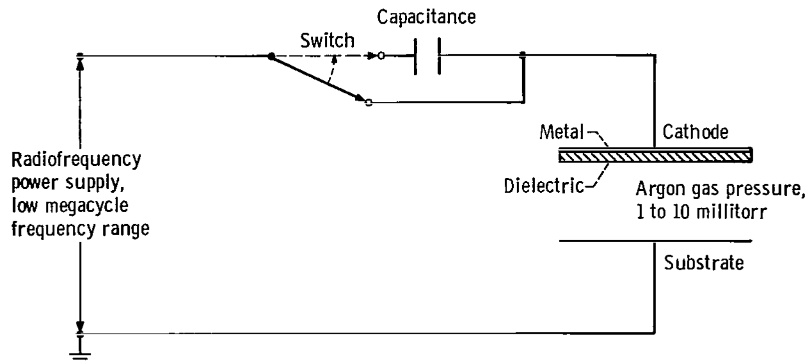


Figure 5. - Electrical schematic for radiofrequency sputtering method. Power supply size is dependent on electrode area and sputtering rate requirements. Capacitance is sized for a particular system to produce negative direct current bias on target suitable for sputtering to occur. The capacitor is necessary when dielectric material is absent.

shown that purer films are produced by bias sputtering than by ac sputtering techniques (ref. 25).

Radiofrequency sputtering uses the same two-electrode configuration as cold cathode dc sputtering and requires a single rf power supply. The electrical schematic of this method is shown in figure 5. Sputtering occurs over an argon pressure range of from 1 to 10 millitorr. An advantage of this method of sputtering over all others is its ability to sputter a dielectric material. This ability is important for maintenance of a sputter electrode system in an accelerator. For example, during etching experiments of titanium and titanium deuteride targets, the electrode receiving the titanium deposit formed a titanium film with insulating properties. This occurred after the film had been exposed to the atmosphere a number of times. Removal of the insulating film was accomplished by sputtering the film onto a shutter by an rf sputtering technique.

## Electrode Design for Rotating Target

The following description of the sputtering electrode assembly applies to an accelerator having a rotating target surface. Since the target surface is continually moving past the sputter electrode, the electrode dimensions can be minimized. This makes possible a small electrode unit and permits the placing of more than one electrode unit in the same accelerator system.

The sputtering electrode assembly consists of one or more sputter electrode units with electrical and coolant feedthroughs, a rotating accelerator target surface, anode and filament sections where appropriate, and a movable shutter between the sputter electrode units and the accelerator target. The sputter electrode unit (fig. 6) consists of a "sputter material" section attached to a heat sink section. The sputter material section

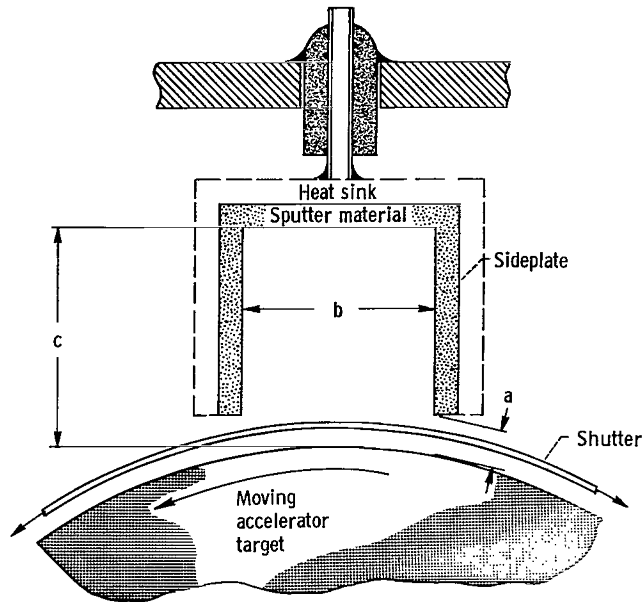


Figure 6. - Cross-sectional view of typical sputter electrode unit (section B-B of fig. 1). Dimensions: a, 0.32 to 0.64 centimeter; b, from ~1.2 centimeter to available space on accelerator target surface; c, ~2.5 to several centimeters.

faces the accelerator target and supplies the material to be sputtered onto the accelerator target, or receives a deposit that has been etched from the accelerator target surface. The heat sink section is supported by ceramic insulators from the base plate. The insulators function as electrical and coolant feedthroughs as well as support pieces. Two such feedthroughs are used per unit electrode. The sputter electrode units can be grouped side by side on a common base plate.

The shutter is remotely operated, and is usually kept at the same electrical potential as the accelerator target. The shutter area is made as large as possible, providing a large surface area that can be used as a gettering surface for absorption of active gaseous contaminants. A section of the shutter should be of a pure, nonreactive material, like platinum, that can be temporarily sputtered onto a particular sputter electrode unit to provide a protective layer. This layer will prevent sputter material absorption of tritium that has been introduced to react with an accelerator target film for target formation purposes. After the tritium has been absorbed, the protective layer is sputterd to a contaminated area of the shutter.

The anode and filament are supported by their own independent feedthroughs. If limited space requires placing of the filament shield close to the filament, the shield should be cooled.

In order to minimize active gas contamination of applied sputtered films, all surfaces of the sputter electrode system should be thoroughly outgassed. In a rotating target de-

sign, some components (e.g., seals) do not permit system baking at elevated temperatures. An alternative method of degassing the surface is by ion bombardment. A glow discharge is permitted to occur between the components of the sputter electrode system, including the target housing.

As discussed in the section ION SPUTTERING, a gap of from 0.3 to 0.6 centimeter is usually maintained between a cathode and any other part of the system to prevent a glow discharge. The target housing should have movable shields that permit temporary widening of the gap so that a discharge can occur. In our sputtering electrode system design, the sputtering electrode, accelerator target, shutter, and target housing are all insulated from each other. Each can temporarily act as a cathode relative to another component of the system. The sputtering method used for the ion bombardment cleaning of these components is the cold cathode dc method. Sputtering voltages are kept very low (a few hundred volts). The sputtering of metal from any surface at these voltages is negligible.

If only the hot cathode bias sputtering method is used, the target housing does not need movable gap shields. A fixed gap width of from 1 to 2 centimeters is used. (Actual gap width should be determined for a specific target housing design.) At the 0.1- to 1-millitorr argon pressures, sputtering will not occur in the gap. For outgassing of component surfaces, argon pressure is raised to approximately 100 millitorr. A glow discharge will occur in the gap regions, and surface cleaning can be accomplished as described in the proceding paragraph.

## Sputter Etch Removal of Tritium Depleted Layers from Targets Formed Outside Accelerator

A preliminary design study of a rotating accelerator target system with a sputter electrode unit incorporated in the system was made. The sputter electrode unit is designed to etch the tritium depleted titanium surface from thick accelerator targets that have been fabricated outside of the accelerator. The sputter etch removal rates for this design were obtained by building a mockup of the actual rotating target and sputter electrode assembly, and performing the etch removal rate experiments for titanium and titanium deuteride in a vacuum belljar.

Rotating target design. - The rotating accelerator target cylinder, constructed mostly of aluminum, measures approximately 15 centimeters in diameter and 12.5 centimeters in length. The target substrate is approximately 0.125-centimeter-thick copper. A turbulent flow of $5^{\circ}$ C water flows along the inner surface of the substrate. A 9-centimeter-wide titanium tritide layer is coated on the outer surface of the cylinder (h in fig. 1). The speed of rotation of the cylinder is 1100 rpm. The accelerator beam impinges on the side of the cylinder. A heat-transfer calculation for this target indicates that for a beam

power input of 400 watts per square centimeter, a titanium tritide thickness of 100 microns can be cooled without the surface temperature exceeding 65° C. Zero thermal resistance at the tritide-copper boundary was assumed. A thermal conductivity factor of 0.03 calories per centimeter per °C per second was used (ref. 7).

Target mockup for etch rate experiments. - A mockup of the accelerator target consisted of a fixed sector of a water cooled copper cylinder with an arc length of 10 centimeters. A 0.050-centimeter-thick sheet of commercial grade titanium was brazed to the surface. The sputter electrode unit was constructed of aluminum in the configuration of figure 7. The length of the electrode unit was 10 centimeters with a 9.5-centimeter-long opening.

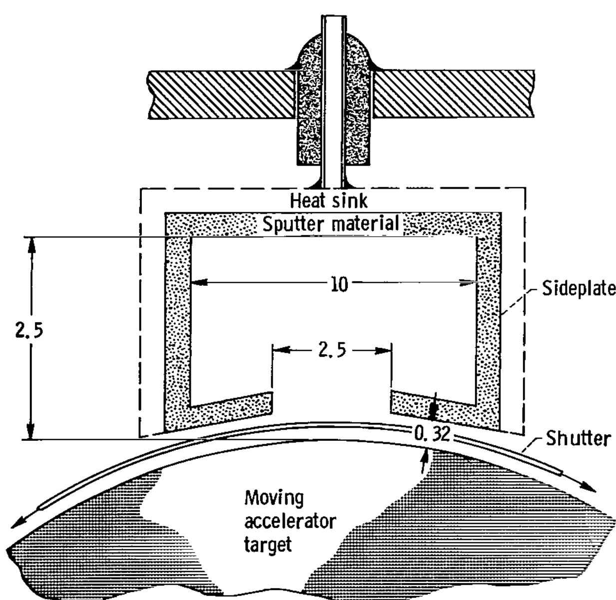


Figure 7. - Cross-sectional view of sputter electrode unit used for sputter etch measurements (section B-B of fig. 1). Dimensions are in centimeters.

Sputter etch rates of titanium and titanium deuteride. - Several pieces of 0.025-centimeter-thick commercially pure titanium were cut in pieces measuring approximately 0.5 by 2 centimeters. Pieces of similar size were cut from a commercially prepared titanium deuteride target with a 0.025-centimeter-thick copper backing and a deuterium loading of 0.3 cubic centimeters of deuterium per square centimeter of target area. All pieces were washed with a household detergent, rinsed thoroughly with methyl alcohol, then vacuum dried for several hours prior to weighing. The samples were weighed on a Cahn microbalance, then stored in a desiccator until used.

The sputtering method used was cold cathode dc sputtering with the accelerator target becoming the cathode and the sputter electrode unit the anode. Initial sputtering of the

cathode was done to clean the surface. An etched strip about 2 centimeters wide appeared on the cathode. Samples of titanium and titanium deuteride were placed on the clean etched surface. The vacuum system was evacuated to about $10^{-6}$ torr, then back filled with argon to a pressure of 50 millitorr. Gas pressure was measured by a thermocouple gage calibrated in argon. A voltage was applied to the cathode, and sputtering was allowed to take place for a timed interval. The samples were removed from the belljar and quickly weighed on the microbalance. The results of several such measurements are shown in figure 8. The mass removal rate of titanium and titanium deuteride, in milligrams per square centimeter per hour, are plotted as a function of cathode potential.

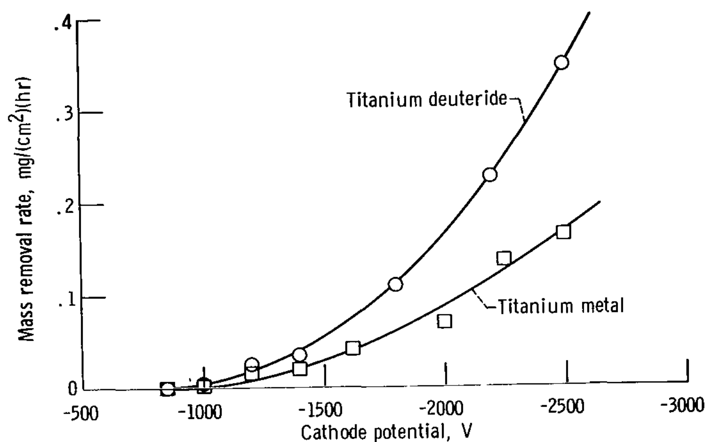


Figure 8. - Mass removal rate from titanium and titanium deuteride cathode by sputtering in argon. Argon gas pressure, 50 millitorr; anode-to-cathode separation distance, 2.54 centimeters.

Assumptions concerning measurement. - Target material thicknesses of 0.1 micron or more were removed during each etching. It was assumed that the sputtering of the titanium atoms and deuterium atoms of the titanium deuteride target occurred at a uniform rate. For the material thicknesses concerned, this is a valid assumption that is based on published data of many investigators who have sputtered material from cathodes composed of more than one element. Their experiments have shown that the chemical composition of a sputtered film will usually be the same as the parent cathode, providing an inert gas environment is used. Gillam (ref. 26) has made a detailed study of the system $AuCu_3$. He found that when the first material was sputtered from a cathode, approximately the first 40 angstroms of the surface is relatively depleted in the higher sputtering rate component. Subsequent material sputtered from the cathode maintained the same composition as the cathode. Films of ZO (ref. 27), InSb and CdS (ref. 28), and $Gd_3F_5O_{12}$ (ref. 29), to mention a few, have been deposited with the same composition as the parent cathode.

## Formation of Metal Tritide Targets Within Accelerator

Prior to application of a titanium film by sputtering, the target surface and housing must be thoroughly outgassed. Before introduction of the ultra-pure argon gas, a vacuum of $1\times10^{-6}$ torr or better is required. The argon gas may then be introduced, and the sputtering process begun.

A metallic film of titanium is first applied to the accelerator target substrate by the sputtering assembly described in a previous section. Tritide target formation within the accelerator is then accomplished at room temperature. The titanium film sputtered within an accelerator is free of an oxide coating and thus without the inhibiting oxide film; tritium gas chemically reacts with the titanium surface (ref. 30). The exothermic tritide reaction takes place readily, and speed of target formation is limited only by the rate of diffusion of tritium into titanium. In practice, a gate valve is used to isolate the accelerator target assembly during target formation.

Upon depletion of the formed target by the accelerator beam, the tritium depleted surface is removed by another sputter electrode etching unit. The target is regenerated by repeating this process.

# FILM THICKNESS MEASUREMENT

The thickness of applied or etched films must be known for proper fabrication of target films within the accelerator. Previously measured sputter etching and material application rates can be used to estimate material thicknesses over a timed interval, providing the sputtering parameters can be held constant. This is difficult to do in practice, and a more direct method of measuring coating thickness is desired.

A thickness detector design that uses an X-ray fluorescent technique is proposed. Several investigators have reported the measurement of coating thicknesses by this technique (refs. 31 and 32). An X-ray tube or radioisotope source is used to excite characteristic X-rays of a substrate. The attenuation of these X-rays through the coating is a measure of the coating thickness. We have used data presented by Cameron and Rhodes (ref. 32) in the design of a thickness detector adapted to our rotating target design. A cutaway cross section of the mounted detector is shown in figure 9.

The detector uses a proportional counting tube containing 90 percent xenon and 10 percent methane at a pressure of 1 atmosphere. The tube has a 2.54-centimeter-diameter by 0.0254-centimeter-thick beryllium side window. The source is a $Pm^{147}/Al$ bremsstraulung source. The $Pm^{147}$ (half-life, 2.6 yr; maximum beta energy, 223 keV) is in an aluminum matrix compressed into a 1-centimeter-diameter by 0.2-millimeter-thick disk. The front is covered with a 0.005-centimeter-thick aluminum foil and the back with 2 millimeters of gold. An activity of up to 100 millicuries of $Pm^{147}$ is used

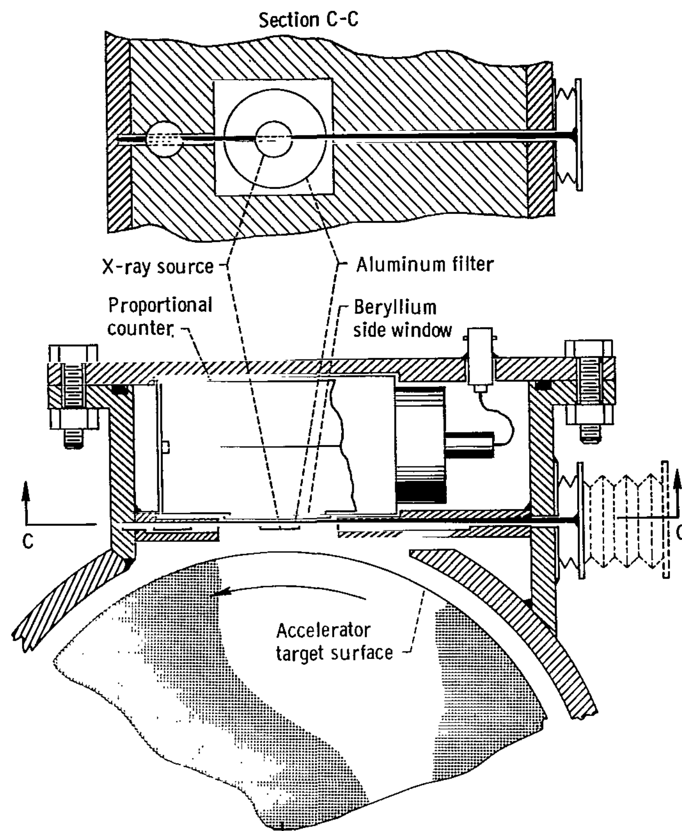


Figure 9. - X-ray fluorescence thickness detector for rotating accelerator target.

(ref. 32). The source back is bonded to a thin rod that is attached at one end to a linear motion vacuum feedthrough. This permits traversing the source from a position centered beneath the counting tube window to a shielded storage container. An aluminum and gold blank of the same dimensions as the source is also attached to the rod. The blank is centered beneath the counter window when the source is in its container. Correct counter geometry is thus preserved for background measurements.

Thickness measurements depend on the transmission of the 8-keV copper substrate X-rays, excited by the $Pm^{147}/Al$ source, through a titanium overlayer. These measurements must be made in the presence of an intense 4.5-keV titanium X-ray background caused by the tritium beta radiation. The mass absorption coefficients for the 4.5- and 8-keV X-rays in aluminum are approximately 250 and 50 square centimeters per gram, respectively. This difference in mass absorption coefficients permits the preferential absorption of the 4.5-keV X-rays. A thin, approximately 0.01-centimeter-thick aluminum filter foil that covers the counter window is sufficient to reduce the 4.5-keV X-ray intensity to an acceptable level.

The intensity of 8-keV copper X-rays excited by the $Pm^{147}/Al$ source is about $1.25\times10^4$ counts per second (ref. 32). The fraction of these X-rays reaching the counter window through the annular solid angle between source and window periphery is 0.138. The mass absorption coefficient $\mu$ at 8 keV for beryllium and aluminum are 2 and 50 square centimeters per gram, respectively (ref. 31). The attenuation of X-rays through the beryllium window and aluminum filter is estimated by an exponential of the form $e^{-\mu\rho X}$, where $\rho$ is the density and X is the thickness of material. The calculated fraction of 8-keV X-rays transmitted through the beryllium window is 0.91, and the fraction through the aluminum filter is 0.254. The X-ray path length in the counter is about 3 centimeters. Nearly total absorption of 8-keV X-rays occurs in this distance. The resulting proportional counter count rate $C_o$ is $(1.25\times10^4)(0.138)(0.91)(0.254)$, or 400 counts per second.

A preamplifier, a linear amplifier with discriminator window, and a scaler are all that is required to process the proportional counter pulse signals for integral counting.

Titanium coating thicknesses on the accelerator target substrate of between 1 and 50 microns can be accurately measured. This range of titanium thicknesses corresponds to an attenuation of 8-keV X-rays by approximately 9 and 99 percent, respectively. A calibration curve for the thickness detector is obtained by measuring the 8-keV X-ray attenuation through several known thickness samples of titanium plated on the copper substrate and plotting this thickness variation as a function of the X-ray attenuation.

After a titanium tritide target has been formed, a continuous background of characteristic 4.5-keV titanium X-rays and bremsstrahlung X-radiation is caused by the 18-keV tritium beta. For very thick targets ($\sim$5 $Ci/cm^2$), a target surface X-ray activity of approximately $5\times10^6$ counts per second can be expected (ref. 33). With our counter geometry and no aluminum filter, a count rate of approximately 400 000 counts per second would occur. In comparison, the 8-keV X-ray count rate caused by the $Pm^{147}/Al$ source would be less than 1600 counts per second. Some filtering is necessary to reduce the background count rate to a level where no pulse pileup will occur in the preamplifier. If the pulses are then processed through a pulse height analyzer, much of the background can be eliminated. We chose the simpler integral counting method and a thick 0.01-centimeter aluminum filter to reduce the background to about 100 counts per second for a thick tritide target. Radiation from neutron activated components of the target system does not contribute much to the background activity since our target system is constructed mostly of aluminum. Thickness measurements are not attempted until the short-lived neutron induced activities have decayed to acceptable levels.

The time required to measure a coating thickness is reasonable. To illustrate, assume that a 3-micron tritium depleted layer is etched from a 15-micron-thick tritide target. The background before and after the etching is about 100 counts per second. An accumulation of 100 000 counts during each counting is required for a statistical accuracy of $\pm0.3$ percent per count. The time required to measure each background count is

about 17 minutes. The mass absorption coefficient for the 8-keV X-ray in titanium is about 200 square centimeters per gram (ref. 31). The fraction of X-rays transmitted through 15 and 12 microns of titanium is 0.259 and 0.339, respectively. The 8-keV X-ray count rates for the 15- and 12-micron thicknesses are 104 and 163 counts per second, respectively. The times required to accumulate a 100 000 count for each thickness are 16 and 12 minutes, respectively. Therefore, the total counting time required for the measurement is only 62 minutes.

## CONCLUSIONS

The design of a sputtering electrode has been proposed for fabrication of titanium tritide targets within an accelerator. The electrode is incorporated into a rotating target assembly. Use of existing sputtering techniques permits the application of high purity titanium films on the accelerator target substrate. The tritide target is formed by diffusion of the tritium into the titanium film at room temperature. Regeneration of the tritide target is done by sputter etch removal of the tritium depleted film from the copper substrate, and by then repeating the fabrication process.

Use of a simple sputter etching electrode has been described that is used to extend the life of a thick tritide target that has been fabricated outside of the accelerator. A tritide target that is much thicker than the range of the accelerated ion is used. After the target surface layer has been depleted of tritium, the layer is etched away to expose fresh tritium material.

Sputtering etch rates for titanium and titanium deuteride were measured. A simple dc, two-electrode, sputtering method was used with an anode-to-cathode distance of 2.54 centimeters and an argon gas pressure of 50 millitorr. For a variation in cathode potential from 1200 to 2500 volts, the titanium and titanium deuteride etch rates varied from 0.016 to 0.164 and from 0.026 to 0.347 milligram per square centimeter per hour, respectively. This range of etch rates for titanium deuteride corresponds to a rate of decrease in titanium deuteride target thickness of approximately 0.1 to 1 micron per hour. This rate permits the controlled removal of very thin target surface layers. Etch rates can be increased by increasing applied voltage.

The design of a detector for measuring the thickness of material deposited or removed from a target has been suggested. The detector uses a radioisotope bremsstrahlung X-ray source to excite 8-keV X-rays from the copper target substrate. The atten-

uation of the X-rays through the titanium film is used to measure its thickness. Target films from 1 to 50 microns thick can be measured in a reasonable time.

Lewis Research Center,
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