MODIFICATIONS OF A TRIODE SPUTTERING
UNIT TO PROVIDE MAXIMUM FLEXIBILITY FOR
USE IN A RADIOACTIVE GLOVE BOX ENVIRONMENT*

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ABSTRACT

In the development of a method to contain polonium as an isotopic heat source the hollow microsphere concept was originated. To make this concept work many new fabrication processes were required. One of these involved sputtering and vacuum evaporation techniques to deposit a protective coating over the 150 μ diameter fueled microsphere in preparation for chemical vapor deposition of the structural cladding. The substrate area consisted of microsphere container dishes which were located in a circular pattern and were connected to a shaker powered by two electromagnets. The power supply was designed to provide amplitude, frequency and phase control to give proper movement of the microsphere and thus assure a uniform spherical coating.

A vacuum system of maximum flexibility was desired so that several evaporation methods could be evaluated in developing the process to deposit over the fueled microspheres.

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While sputtering appeared to be the most desirable process, the sputtering system used had to have the proper geometry so that vacuum deposition could be used in conjunction with the sputtering process if necessary. With these requirements in mind, sputtering equipment was evaluated. A triode sputtering system was chosen and modified for upside down installation to its designed geometry. The electromagnet used to focus the ion beam onto the target originally located on the outside of the bell jar system was relocated inside the vacuum system due to glove box space limitations. The relocation of the magnet helped the deposition rates but created a heat problem which required water cooling of the magnet.

One of the overriding requirements was to assure safe operating conditions. Basically this meant that no radioactivity was to be released. Thus, a major consideration in the installation of the vacuum system and sputtering unit in the radiation glove box was to provide a mechanism so that any malfunction (e.g., power or component failure) would result in immediate shutdown in such a way that no release of radioactive material would occur. The box in normal operation is maintained at a pressure of -1 in. of water. All mechanisms were interlocked to fail in such a manner as to maintain this negative pressure in the box.

The sputtering unit and vacuum system have performed to original design specifications. The safety interlock system has maintained safe operating conditions, periodically shutting down the sputtering and vacuum system.

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INTRODUCTION

In the development of a method to contain polonium as an isotopic heat source the hollow microsphere concept was originated. This paper describes the process to modify and install a sputtering system for deposition of a protective coating onto microsphere heat sources. The work described here shows what can be done by adapting commercially available equipment to specific problems. To make this concept work many new fabrication processes were required (see Fig. 1). Deposition of activation layer (or direct deposition of alpha emitter) and deposition of compatibility cladding in this process involves sputtering and vacuum evaporation techniques. The process described here is the modification and installation of a sputtering system to deposit a protective coating over the 150 μ diameter fueled microsphere in preparation for chemical vapor deposition of the containment shell.

The substrate area consisted of microsphere container dishes located in a circular pattern, connected to two electromagnets. The power supply was designed to provide amplitude, frequency and phase control to provide random movement of the microspheres to deposit a uniform spherical coating.

SPUTTERING SYSTEM REQUIREMENTS

A vacuum system of maximum flexibility was desired so that several evaporation methods could be evaluated in developing a process to deposit a protective coating over the fueled microspheres. The initial idea was to use vacuum vapor deposition with electron beam heating to evaporate the polonide fuel and then seal the fuel layer with a refractive metal film. This was to be done during a single pump down. As the program progressed it became obvious that the fueling process would involve reacting the polonium with a rare earth in a special chamber at elevated temperatures. This changed the requirement of the chamber in that the rare earth fueling could be done outside the hood line in a cold facility (nonradioactive). Further study indicated that a protective seal layer would be required to protect the fueled layer from the chemical vapor deposition process and that a sputtered film would be more desirable due to its adherence, higher energy molecules deposition and film properties. With these requirements in mind, sputtering equipment was evaluated.

SPUTTERING SYSTEM

Balzer's Sputron II appeared to provide the necessary flexibility. The Balzer's unit as depicted in Fig. 2 is a triode sputtering system. The sputtering unit can be broken down into three basic areas consisting of the ion generating chamber, the target area, and the substrate region. The ionization chamber contains the hot cathode, the auxiliary anode, the gas inlet valve, and the front diaphragm used to achieve a pressure drop between the ionization chamber and the vacuum chamber during operation. The front diaphragm in the ionization chamber increases the pressure of the ionization chamber by a factor of approximately 10 in comparison to the vacuum chamber. This increase serves to maintain a higher vacuum in the substrate region and to provide a chamber where the argon is more efficiently ionized. The auxiliary anode facilitates striking the arc at low pressures. The sputtering power level, target voltage and ion beam amperage can be controlled only indirectly by the chamber pressure voltage of the hot cathode and magnetic field to focus the ions onto the target. The parameters controlled by the operator are arc current, hot cathode amperage, chamber pressure, and magnetic field for ion plasma focusing. By increasing the hot cathode amperage

and arc current the sputtering amperage is increased or decreased for constant pressure. The sputtering amperage is also affected and can be maximized by adjusting the magnetic field. Increasing the pressure increases the number of ions generated for a given arc voltage and hot cathode amperage. The net result is an increase in the ion current. The normal operation parameters used were: ion amperage, 1.3 A; target voltage, 2 kV; filament amperage, 80 A; arc amperage, 40 A; arc voltage, 35 V; and pressure, 9×10^{-4} Torr. With these parameters, rates depending on material vary from 100 to 300 Å/min.

SPUTTERING SYSTEM INSTALLATION

Installation of this particular system upside down to its designed geometry was required, as illustrated in Fig. 3. Basically, then, the ionization chamber was mounted onto the base plate with the target assembly mounted at the top of the chamber. The electromagnet used to focus the ion beam onto the target was originally located on the outside of the bell jar system, but due to glove box space limitations, it was impossible to locate the magnet on the outside of the chamber, and it had to be moved inside the chamber. The relocation of the magnet helped increase the deposition rates,

with maximum on the order of 600 Å/min, and reduced the power input into the magnet to focus the ion beam onto the target. One of the requirements created by locating the magnet inside the vacuum chamber was water cooling to prevent overheating and consequent shorting out of the magnet. The substrate areas consisted of microsphere container dishes located in a circular pattern as seen in Fig. 4. Note the hole in the center of the substrate holder, which allows the ion beam to pass through for bombardment onto the sputtering target. The location and hole size in the substrate holder was critical. The position was experimentally located by measuring the rate for a set power level versus substrate position, holes size and the temperature rise of the substrate. As might be expected, temperature rise results were as reliable as the deposition rate.

Figures 4 and 5 depict the internal installation of the sputtering unit. Figures 6 and 7 show the glove box during fabrication giving some idea of the complexity and compactness resulting from a glove box installation. The diffusion pump can be recognized in Fig. 6. The control panel for the sputtering and safety interlock system is seen on the right in Fig. 7. The sputtering system is protected by the water interlock, vacuum interlock, and rear door interlock. The water interlock consists

of two flow devices for monitoring the water flow into the target and ion chamber cooling housing. In the event insufficient flow is sensed, the unit is automatically shut down and cannot be started until flow has been restored. A vacuum interlock connected to a relay controlled by a thermocouple senses the pressure in the vacuum chamber. The sputtering unit is inoperable until a vacuum of 50 μ or less has been achieved. The vacuum interlock is provided more as an operator safety measure rather than a device for protecting the sputtering unit. The rear door interlock is a safety mechanism for personnel protection and can be overridden in the event the unit must be serviced or inspected while in an operating condition. In addition to this, the sputtering system is interlocked to the overall vacuum system operation through the thermocouple interlock mechanism. A malfunction of the pumping system during disengagement of the mechanical pump will result in automatic shutdown of the sputtering unit through the thermocouple relay interlock.

MICROSPHERE SUBSTRATE HOLDER

A special microsphere shaker table designed and fabricated for the sputtering system can be seen in Fig. 4. This view was taken through the viewing port of the sputtering unit. This

particular shaker system is an outgrowth of a system previously developed for the vacuum vapor deposition of the rare earth metals onto the hollow microspheres. Our experience indicated that the microspheres were sticking under the sputtering operation, due to better adherence of the film. Two electromagnetic vibrators were fabricated and installed to prevent this occurrence. A special power supply allowing operation of either or both of the electromagnetic vibrating units was fabricated. This unit has three basic controls consisting of phase control, amplitude control, and frequency control. The provision for a special phase circuit allows adjustment of the phase between the two units from 0 to 90 deg. By adjusting the phase relationship between the two electromagnets, it is possible to get a rotary vibratory motion. The amplitude control simply controls the amount of power into each electromagnetic vibrator which, in turn, affects the accelerating force with which the unit moves. The frequency control consists of manual frequency control and ramp frequency control. The frequency is controllable from 3 to 30 Hz, and is automatically controlled with the ramp control over a frequency range of +5 Hz centered about a selected reference frequency. With these controls, we found that the spheres could be moved in a rotary or back and forth fashion at the choice of the operator.

When sticking occurs, the operator can increase the amplitude control to produce a more violent shaking action. Quite often we found that the spheres had been shaken loose allowing the deposit to continue.

Figure 8 is the metallograph photo of a sputtering run onto fueled polonide microspheres. The fuel is sandwiched between the microsphere and the sputtered film. The deposition time was about 10 hours. Considering the many heat treatments and the other various handling procedures, the film looked acceptable in that the polonide fuel was protected by the sputtered film.

CONCLUSIONS

As seen in Fig. 8, the unit has sputtered a protective film over fueled microspheres. Attempts were made to deposit the strength cladding using chemical vapor deposition and omitting the protective sputtered film. This resulted in losing the fueled layer. Thus, this process has provided the protective film it was designed to deposit.

LIST OF FIGURE CAPTIONS

- 1 The Hollow Microsphere Concept for Radioisotope Heat Sources
- 2 Schematic of the Sputron II
- 3 Schematic of Modified Sputron II
- 4 Substrate Holder as Viewed Through Vacuum Chamber Viewing Window
- 5 Electromagnet and Anode-Cathode Assembly Inside Vacuum Chamber
- 6 Radiation Glove Box and Diffusion Pump (lower right of photo)
- 7 Hood Line System Showing the Control Panel for the Sputtering and Safety Interlock System (right of photo)
- 8 Metallography of Fueled Microsphere with Sputtered Protective Film Overlay

RADIOISOTOPE HEAT SOURCES

ALPHA EMITTERS

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