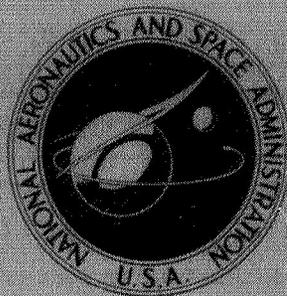


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VACUUM-PACKAGED CESIUM
FOR NUCLEAR THERMIONIC DIODES

by James F. Morris

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VACUUM-PACKAGED CESIUM FOR NUCLEAR THERMIONIC DIODES

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SUMMARY

Out-of-core nuclear thermionics places special demands on the physicochemistry of the converters. The emitters, collectors, and cesium require careful preparation to insure diode performance. For cesium, vacuum packaging allows excellent quality control. Providing this assurance, though, meant developing a new dispensing train and vacuum processing station. These designs and the resulting baked-out, brazed-shut molybdenum capsules of cesium are the subjects of the present report.

REQUIREMENTS FOR CESIUM PACKAGING

Without the space program, cesium diodes might still be interesting laboratory oddities. Now, however, they offer potentially efficient means for changing nuclear energy into electric power. Although this application greatly advanced the thermionic technology, it also restricted converter materials and operating ranges. For example, the recent upsurge in out-of-core thermionics (refs. 1 to 4) brought lower diode temperatures. These new thermal bounds result from the limitations of refractory alloys to contain alkali metals carrying heat out of the reactor and into the converters. Of course, both the out-of-core service and its reduced temperatures contribute to longer diode lives. But the temperature reductions mean decreases in converter performance - unless internal physicochemical gains offset these losses: New diode materials must yield greater currents and voltages at lower temperatures. Consequently, better emitters, better collectors, and better cesium quality control are more important today than ever before. In response to these needs this report presents one method for assuring cesium purity in thermionic diodes - encapsulating the cesium in degassed, nonreactive, vacuum-tight, breakable ampules.

Vacuum packaging cesium required the development of a dispensing train, a capsule and closure, and a special processing station. The system designed to dispense cesium utilizes the unique capabilities of the freeze, melt (FM) valve (ref. 5). With such a dis-

penser, heating or cooling across the melting point of cesium in a terminal orifice starts or stops injection into the ampule. But just providing this cesium container posed problems: The capsule must result from simple fabrication, permit bake-out before and after loading and closure, enable sealing in high vacuum, allow the release of its contents within a thermionic diode, and of course, resist cesium. Finally, a molybdenum tube with a brazed copper plug evolved as a workable cesium container. Ampules like this with the previously mentioned dispensing system then became parts of a vacuum-packaging device. And the result was a miniature evacuated bottling works for cesium.

THE CESIUM-DISPENSING TRAIN

The present apparatus combines three packless valves and a cesium-shipping container with the FM valve and two deformable-gasket seals to provide a removable dispensing system. Since reference 5 describes this assembly in detail, however, further discussion of it here seems unnecessary.

THE CESIUM AMPULE

In early experiments on thermionic energy conversion, breaking a glass ampule released cesium within the diode. Cesium, though, reacts with components of many glasses. So, although its fused seal was desirable, the glass capsule yielded to the copper tube closed with pinch-offs. Some feel these latter closures are satisfactory. Still others, including professional experts on copper pinch-offs, find them less than reliable. This uncertainty prompted a search for a new cesium container with a more positive seal for the present work.

As the introduction indicated, the material forming an evacuated package for cesium must be nonreactive, degassable, and vacuum-tight. One further requirement caused difficulty, however, in developing a suitable cesium capsule for use in thermionic diodes: The sealed ampule must withstand high-temperature bake-outs. Yet it must open opportunely within the closed converter. For this purpose, pinched-off tubes of clean niobium or tantalum showed some promise. But the pinch-offs often failed unpredictably. Nut-and-bolt knife-edge seals (refs. 5 and 6) made of refractory metals also seemed feasible. When these closures were small enough, though, the threaded bolt sections distended during sealing and relaxed with subsequent heat cycling. Again the statistical faults were unacceptable. Ultimately, a crushable molybdenum vial sealed with a copper braze became the cesium package.

The smallest such capsule is shorter than 2 centimeters and less than 1/2 centi-

meter in diameter; the vial and top that make up this ampule appear in figure 1. As these dimensions imply, brazing the copper plug to the molybdenum tube, so close to the relatively volatile cesium, was not a simple operation. The problem diminished, however, with cooling of the tube bottom - and with some other precautions: Use of a long, thin-walled ampule with a small diameter reduced the heat transfer to the cesium; in addition it made breaking the capsule in the thermionic diode easier. Then very high-temperature degassing of the molybdenum tube prior to filling accelerated and greatly improved the subsequent braze. And preheating the tube top to drive the injected cesium down the cold bottom before brazing also helped. With these preparations, brazed closures of capsules holding cesium were successful.

If larger cesium containers are necessary, they result simply from lengthening the ampule in figure 1. Replacing the cap on the tube bottom with a cylindrical bulb provides even greater volumes. And for all of these sizes the equipment and procedures used to encapsulate cesium are the same.

THE EVACUATED STATION FOR CESIUM PACKAGING

As previously indicated, vacuum encapsulation allows better quality control of cesium for thermionic diodes. But the requirements for such insurance are more restrictive than the preceding statement implies: Only vacuum-baked containers guarantee the cesium purity needed for thermionics. Without such degassing the high temperatures and cesium exposures in diodes release many contaminants that are tightly bound under normal conditions. To avoid this the inside of the capsule and the dispensing system must be free of impurities prior to cesium injection and sealing. Subsequently, within the converter, the contamination from handling the ampule must go before diode closure. Thus, a good cesium package undergoes high-temperature bake-outs preceding and following its fill and seal. These rigors demand a rugged, reliable capsule and a completely bakable station for vacuum processing.

The cesium packaging plant shown by figure 2 combines bakability with accessibility. This effectiveness results from utilizing high-temperature, high-vacuum deformed-gasket seals: A miniature vacuum flange allows the removal of the dispensing train from the 8-inch (20.32-cm) flange that serves as the chamber top. Then a nut-and-bolt seal enables separation of the FM valve from the rest of the cesium feed system. On the side of the station, four small deformed-gasket flanges provide openings for lines of water, electricity, and sight. The water circulates through a plate to cool the capsule holder, the electricity energizes the electron-bombardment ring to braze the ampules closed, and the view with two additional ports on top permits checks on the alignment indices for dispensing and sealing. A 6-inch (15.24-cm) vacuum flange joins

the bottom of the chamber to a 140-liter-per-second ($0.14 \text{ m}^3/\text{sec}$) ion pump. Another vacuum system connects through a small flange to the upper cavity, which is part of the seals for three rods that rotate and translate through water-cooled O-ring couplings. As they move, these rods actuate the capsule holder on the right, the copper-plug feeder on the left, and the inserter in the middle. A description of their movements indicates the steps necessary to package cesium.

ENCAPSULATING CESIUM

Before the actual packaging of cesium, several parts used in this sequence require preparations: Simple fabricating techniques produce the molybdenum vials and their copper tops. These copper pieces go directly into the plug feeder of the vacuum station. But the molybdenum tubes undergo degassing at temperatures above the melting point of copper before their installation in the capsule holder. Then, when the assembly and bake-out of the cesium-dispensing system are complete, coupling several vacuum flanges closes the chamber for evacuation. Subsequently the surrounding oven bakes the apparatus at 450°C until the pressure drops below 10^{-8} torr. With cooling the vacuum hardens even more, and the packaging plant is ready to process cesium.

As reference 5 states, heating the dispensing train maintains the cesium as a liquid. Cooling the FM valve with sufficient water flow assures a rapid shut-off by cesium solidification. And setting the orifice-heater voltage properly enables opening or closing the FM valve with a switch. Because the combination of these heat transfer rates dictates the orifice size and cesium viscosity, the present FM valve is very adaptable. It dispenses quantities ranging from separate 3-milligram drops to continuous streams about 6 mils (0.1524 mm) in diameter - depending on the balance of heating and cooling effects. Calibration prior to capsule loading establishes the conditions for gating the desired amounts of cesium. This procedure also provides a final rinse of the supply system. After running the calibrating and cleansing cesium into a throw-away ampule, the dispenser is prepared for the packaging operation.

Now the encapsulated cesium doses result from an uncomplicated repetitive procedure:

- (1) Rotate the capsule holder and raise it to place an empty molybdenum vial immediately below the FM valve.
- (2) Switch the orifice heater on then off, injecting an appropriate amount of cesium into the tube.
- (3) Revolve the newly loaded vial to the electron-bombardment station and heat the tube top to drive the cesium down to the cold bottom.
- (4) Lower the capsule holder to contact the cooling coil - removing the added heat

and storing enough cold to assure negligible cesium vaporization during brazing.

(5) Aline the open vial of cesium, a copper plug, and the inserter and push the stopper into the tube.

(6) Return the covered but unsealed vial to the electron-bombardment loop and heat the tube top until melted copper wets the lip entirely.

(7) Move the capsule holder with the brazed ampule down to rest on the cooling coil until the next cycle.

Repetition of this sequence produces up to 12 assorted capsules of cesium in addition to those for calibration and analyses. Three ampules, big enough to permit wet chemical determinations of cesium purity, allow sampling before, during, and after the encapsulating run. Of course, larger or less simple versions of the vacuum packaging plant could yield more ampules of cesium in each batch. But the present output is adequate for its intended use.

AFTER CESIUM

Although this report emphasizes cesium, adaptations of the process to vacuum-package a variety of materials are immediately obvious. But the gains must compensate the effort. So encapsulating other metals with great reactivities, low melting points, and requisite high purities seems a worthwhile application. Alkali metals used for high-temperature energy transport fall into this category. Here the investment for extreme cleanliness pays excellent dividends because trace impurities can ruin these heat transfer systems quickly. To avoid such failures in advanced out-of-core thermionics (ref. 4), a vacuum system like the present one might load heat pipes - as well as cesium capsules.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 13, 1970,
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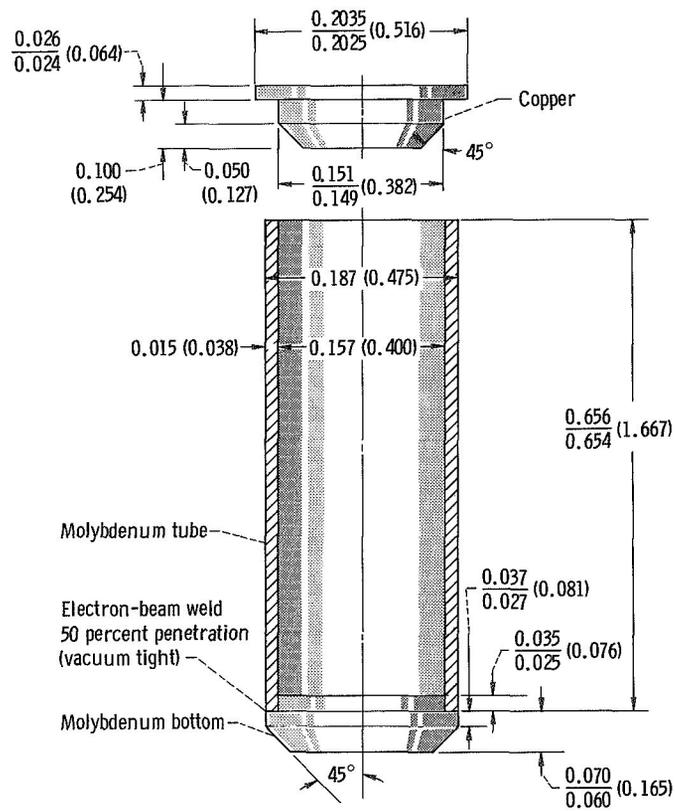
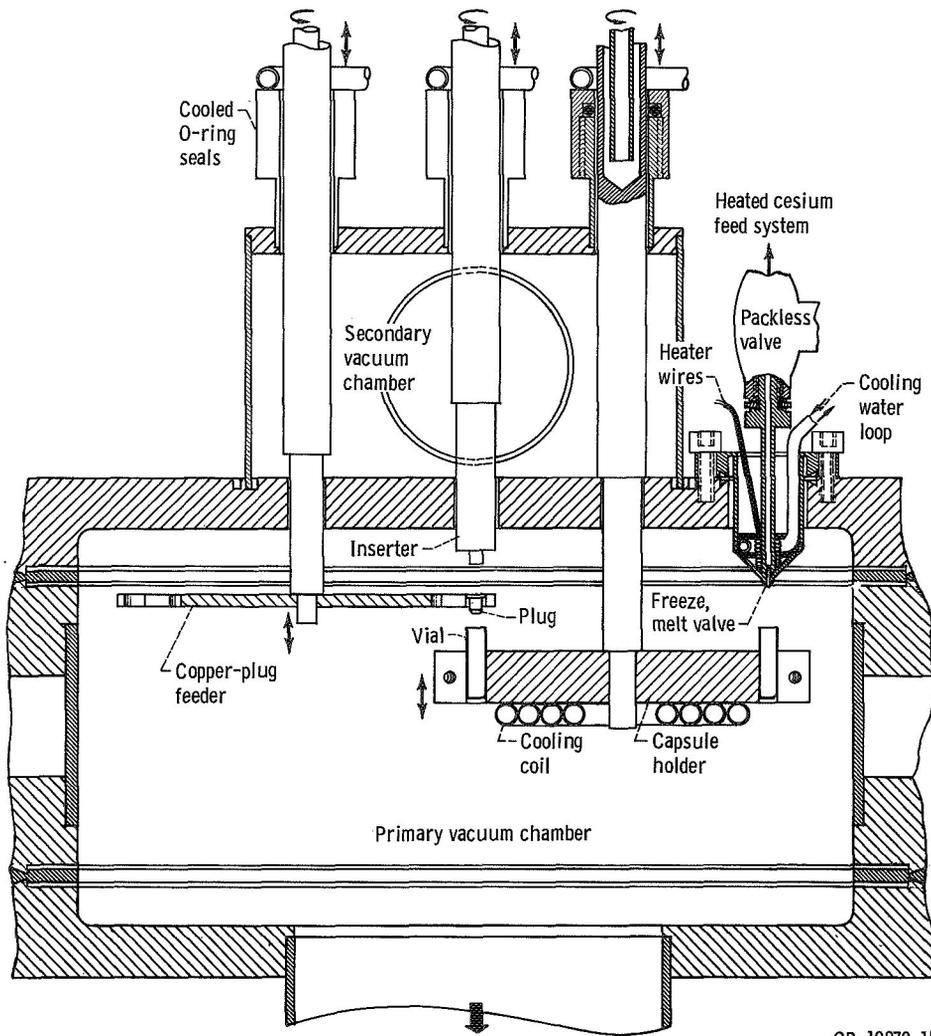


Figure 1. - Cesium capsule. (Dimensions are in inches (cm).)



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Figure 2. - Station for vacuum packaging cesium.

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