The Rotating Heat-Pipe Oven; A Universal Device for the Containment of Atomic and Molecular Vapors

Merrill M. Hessel*

Quantum Electronics Division National Bureau of Standards, Boulder, Colorado 80302
Physics Department, Fordham University, Bronx, New York

Thomas B. Lucatorto

Optical Physics Division National Bureau of Standards, Washington, D. C. 20234

A new type of heat-pipe oven has been developed that uses centrifugal force as a return mechanism for the condensed vapors in contrast to capillary return forces for the conventional heat-pipe oven. Since this new oven is no longer limited to materials that wet wicks, it can be used to contain *any* material that does not react with the walls of the containing vessel. We describe the operation of this oven with sodium and InI even when the InI is solid. Spectroscopic and laser applications of the "rotating" heat-pipe oven are discussed.

The heat-pipe oven is a unique spectroscopic furnace that was developed by Vidal and Cooper.[1] This oven produces atomic and molecular vapors of well-defined temperature, pressure, and optical pathlength, and it is able to contain vapors that react with the windows of conventional cells. The term "heat pipe" generally refers to a class of devices which utilize the evaporation and condensation of a working fluid to transfer large amounts of heat[2] under nearly isothermal conditions. Typically the heat pipe is in the form of a long cylinder in which the working fluid is returned from the region of condensation by the capillary action of a wick. The essential modification in order to adapt the heat pipe to spectroscopy was the addition of a buffer gas to the tube; the modified version is called a "heat-pipe oven."

The heat-pipe oven has typically been used to study the spectra[1,3–5] of alkali vapors. In the alkali ovens a long stainless steel tube was fitted with a double layer of fine stainless steel mesh that is in contact with the i.d. of the tube. Each end was capped off with a window and connected through side ports to a gas handling system. The alkali is placed in the middle of the tube, the tube is evacuated and refilled with an inert buffer gas at a few Torr (1 Torr= 133 N/m²), and heat is applied to a short region about the middle. As the metal evaporates from the heated region it condenses in the slightly cooler adjacent regions. If enough heat is applied, the pressure of the alkali vapor becomes equal to that of the buffer gas with the result that the evaporation and condensation of the metal creates an almost isothermal zone. The length of the zone is proportional to the power input. The resultant mass motion of the alkali vapor from the middle pumps the buffer gas into the ends of the pipe creating a situation which is characterized by:

1. an almost isothermal, isobaric region of pure alkali vapor centered around the middle of the pipe;
2. a relatively short transition zone of mixed composition and sharp temperature falloff at the interface of the buffer gas and the alkali vapor;
3. a plug of pure buffer gas at each end that is at the same pressure as the metal vapor and which protects the end windows from chemical attack.

The advantages of the heat-pipe oven are the ease and accuracy with which the pressure, temperature and optical pathlength of the vapor can be controlled and the isolation of the windows from the contaminating effects of the vapor. Up to now there have been two variations of the original design:

1. The oven has been adapted for use in vacuum uv spectroscopy by the replacement of the conventional windows with windows made of thin metallic or plastic films.[3]
2. A double concentric heat-pipe oven has been designed which permits operation with a variable mixture of vapors.[5]

The conventional heat-pipe ovens described above are limited to materials that wet wicks. Even for certain inorganic salts for which a wick could be found, the very low thermal conductivity caused a large temperature gradient across the wick creating a large pressure gradient that was sufficient to blow the liquid salt out of the wick.[6]

To eliminate the need for a wick we have devised the "rotating heat-pipe oven," a device that can be used with *any* material that does not react with the walls of the pipe. The return mechanism for the condensed vapor is provided by centrifugal force.[7,8]

Figure 1 is a schematic diagram of the stainless steel rotating heat-pipe oven that has been constructed for spectrographic studies of materials that have sufficient vapor pressure at temperatures less than 1100°C. This heat pipe has a double conical taper with the bases of the cones meeting at the evaporator region in the middle of the pipe. When the pipe rotates at a sufficient speed, the sample is thrown against the inner wall of the tube and a component of force is generated toward the evaporator region. In our version the angle of the taper was 2° and the average internal diameter 2 cm; the return acceleration of the working fluid at 2000 rpm was approximately 2g. By increasing the diameter, angle of the taper, or the angular speed one could easily increase the return force by an order of magnitude or more. Such higher forces would be useful for dealing with materials which are very viscous at the desired operating temperature.

Because of the rotation, a special high-vacuum rotating seal and water cooling collars had to be used. The rotating vacuum seal was a spring-loaded seal that is commercially available.[9] On the atmospheric pressure side of this seal was a small reservoir filled with a lubricating oil having very low vapor pressure. The cooling collars were added to enhance the temperature falloff in the transition zones and to protect
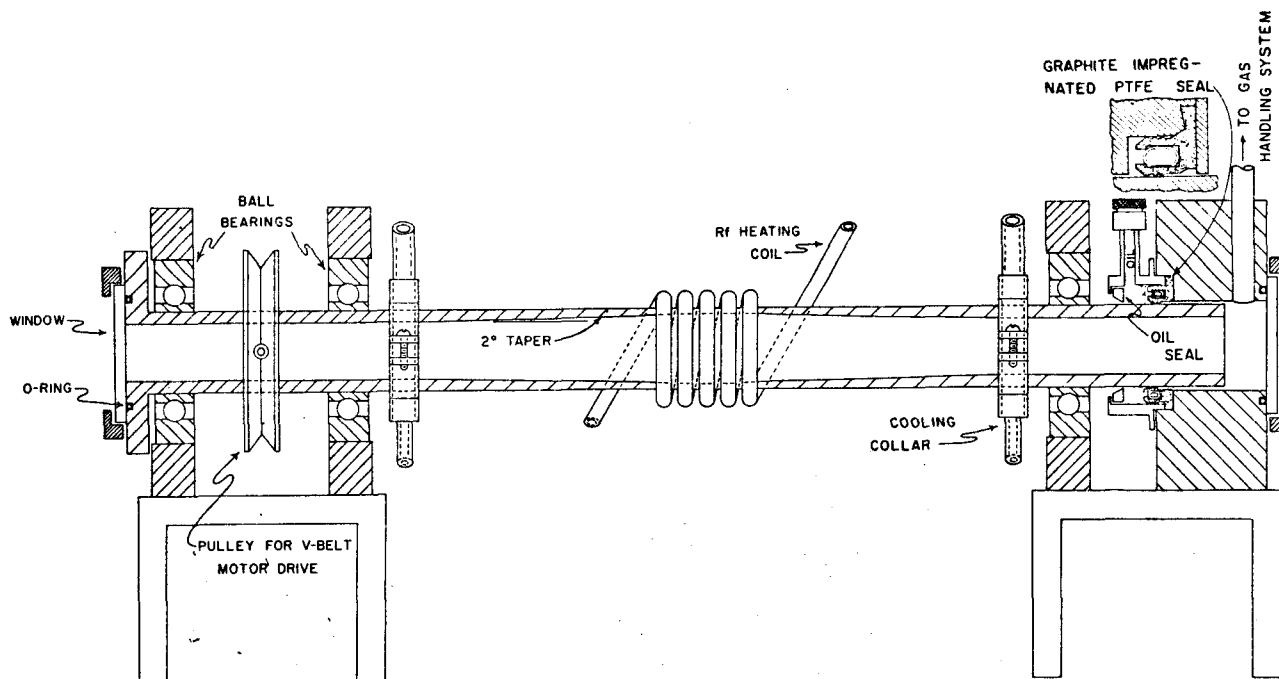
       561

FIG. 1. Schematic diagram of the rotating heat-pipe oven.

the ball bearings and the seal from excessive heat. The water chamber was sealed to the rotating pipe by means of two ordinary leather oil seals impregnated with silicone grease. The length between collars was about 30 cm; this was the maximum attainable length of the central hot zone of pure vapor.

The oven was intended mainly for use in spectroscopic studies of the visible and uv absorption in various salts. However, as a preliminary test, liquid sodium, a substance with well-known vapor pressure and low viscosity, was used as the working fluid. Conventional stainless steel heat-pipe ovens with stainless steel wicks are known to work well with sodium, and it was desired to compare the operation of the new pipe with the capillary-return version. In addition to the lack of a wick the new pipe differed from the older versions in that, for mechanical strength, the wall thickness was about 0.25 cm greater than the 0.15 cm for conventional heat pipes and therefore had greater heat conductivity.

A rotational speed of only 600 rpm ($\frac{1}{6}g$) was sufficient to provide adequate return for the sodium at all operating pathlengths up to the maximum length of the tapered

section. Although an infrared sensor used to measure the temperature gave poor absolute temperature values because of variations in the emissivity of the stainless steel, we could still detect a long, nearly isothermal region bounded by relatively sharp temperature gradients. Even though these transition zones were probably not as short as with the thinner conventional pipe, it would still be possible to use the differential length method to make an accurate measure of the optical depth of the vapor. Thus sodium in the rotating heat pipe can be operated in the same manner as in a conventional heat-pipe oven.

InI was heated in the rotating heat pipe with argon as a buffer gas. A high pressure xenon arc was placed at one end of the rotating heat-pipe oven and the molecular absorption spectrum of the $A\ ^3\Pi_0 - X^1\Sigma^+$ electronic transitions of InI was photographed on a 10 m air Eagle spectrograph. Figure 2 shows a portion of these spectra recorded at five different buffer gas pressures along with the emission lines of the thorium reference spectra. At 14 Torr [Fig. 2(a)] there was almost total absorption. As the Ar pressure was lowered, the absorption spectra became weaker. At about 3 Torr [Fig. 2(d)] the InI was no longer molten and looked like a powder. Reducing the buffer gas pressure to 1 Torr produced no significant change in the appearance of the absorption spectra, indicating a departure from true heat pipe action. It is possible that the low thermal conductivity of the solid created temperature gradients and, therefore, a nonhomogeneous vapor distribution. The centrifugal action of the pipe still contained the solid material and the buffer gas prevented contamination of the windows. It is therefore possible to use the rotating heat pipe to contain and study vapors of many materials that sublime.

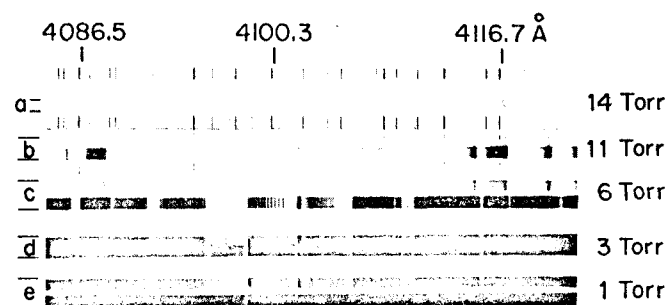Several modifications to the rotating oven can be made:

FIG. 2. Absorption spectra of InI. The thin emission lines are thorium reference spectra. The argon buffer gas pressure is (a) 14 Torr; (b) 11 Torr; (c) 6 Torr; (d) 3 Torr; (e) 1 Torr. Two different exposures times are shown at each buffer gas pressure.

(a) A vacuum seal can be added to each end and thin film windows mounted for work in the vacuum ultraviolet.

(b) A double concentric design[5] can be made for containment of mixed vapors and for plasma and laser studies.

(c) A molded ceramic tube can be used for high temperatures and high resistance to chemical attack.

ACKNOWLEDGMENTS

*Visiting scientist, Institute for Basic Standards, 1972–1973.

[1]C. R. Vidal and J. Cooper, J. Appl. Phys. **40**, 3370 (1969).

[2]For a general review of heat pipes as heat transfer devices, see Winter and Barsch, Ann. Rev. Heat Transfer **6**, 219 (1971).

[3]D. Ederer, T. Lucatorto, and R. Madden, Phys. Rev. Lett. **22**, 1537 (1970).

[4]M. M. Hessel and P. Jankowski, J. Appl. Phys. **43**, 209 (1972).

[5]C. R. Vidal and M. M. Hessel, J. Appl. Phys. **43**, 2776 (1972).

[6]W. B. Hall and S. W. Kessler, Tech. Rep. E. Com, 01507-4, September 1966, p. 46, United States Army Electronics Command Fort Monmouth, N.J. (unpublished).

[7]P. J. Marto, T. J. Daley, L. J. Ballback, Naval Postgraduate School Rept. NPS-59Mx70061A(1970) (unpublished)

[8]S. H. Chan, Z. Kanai, and W. T. Yang, J. Nucl. Energy **25**, 479 (1971).

[9]The rotating seal was obtained from a supplier of hydraulic seals for special applications and was made from graphite-impregnated PTFE (polytetrafluoroethylene, a commonly used, high-temperature, low vapor pressure plastic). Subsequent experience with neoprene seals and with magnetically trapped fluid seals has shown these types also satisfactory.