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IMPLANTATION AT -192 TO +500°C

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A VARIABLE-TEMPERATURE SAMPLE SYSTEM FOR ION IMPLANTATION AT -192 TO +500°C

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ABSTRACT

A variable temperature sample system based on exchange-gas coupling has been developed for ion-implantation use. The sample temperature can be controlled from -192°C to +500°C with rapid cooling. The system also has provisions for focusing and alignment of the ion beam, electron suppression, temperature monitoring, sample current measuring and cryo-shielding. Design considerations and operating characteristics will be discussed.

I. INTRODUCTION

Sample temperature-control systems for ion implantation present some unique problems. If the sample system has to be isolated from ground as in a post acceleration system, there are normally space and power limitations. These limitations restrict the amount of equipment that can be used, which makes in situ wide temperature ranges difficult to obtain. It is also necessary to manipulate the equipment controls from outside a high voltage enclosure. Furthermore, at times one may want to rapidly cool a sample after implantation, or to counteract large power inputs on the sample from an ion beam.

For these reasons a controlled-temperature sample system based on a variable, nonmechanical heat leak to a liquid nitrogen bath via exchange gas was developed. This system is described along with design considerations and test data.

II. THEORY OF HEAT LOSS

There are four sources of heat loss from a sample block:¹ conduction through the exchange gas, conduction along mechanical support elements, radiation, and condensation and vaporization of the exchange gas.

Conduction through the exchange gas can be estimated by using the equation

$$\frac{dT}{dt} = \frac{-KA}{C_p D} (T - T_{\text{bath}}) \quad (1)$$

where K = thermal conductivity of the gas

C_p = heat capacity at constant pressure of the sample block

A = effective area of the plates within the exchange gas

D = distance between these plates.

The solution of Eq. (1) is then:

$$T = T_{\text{bath}} + (T_{\text{initial}} - T_{\text{bath}}) \exp\left(\frac{-KA t}{C_p D}\right) \quad (2)$$

Conduction along the mechanical support, a thin-wall stainless steel cylinder in this case, is again given by Eq. (1), but now with

K = thermal conductivity of the support

A = cross-sectional area of the support

D = length of the support.

Radiation losses are estimated by using the formula

$$\frac{dT}{dt} = \frac{-\epsilon \sigma A}{C_p} (T^4 - T_{\text{bath}}^4) \quad (3)$$

where C_p = heat capacity at a constant pressure of the sample block

A = area of the plates within the exchange gas

ϵ = emissivity of the plate surfaces

σ = Stefan-Boltzmann constant.

In the case of radiation, the losses become significant at elevated temperatures because of the T^4 term.

Condensation and vaporization of the exchange gas becomes an important mode of heat transfer in cases where the exchange gas is the same as the cryogenic-bath fluid and the exchange gas pressure is greater than one atmosphere. This effect can produce extremely rapid cooling, as will be seen.

III. DESIGN AND OPERATION

The sample holder is shown schematically in Fig. 1. A cylindrical exchange-gas chamber with 0.05 cm thick 304 stainless steel walls connects the liquid nitrogen bath to the sample block, which is OFHC copper. Cylindrical Copper fins extend from the nitrogen bath and from the sample block into the exchange gas chamber, thereby providing a variable heat leak. The sample block is surrounded by a copper faraday cup with pumping apertures and this cup is coupled to the liquid nitrogen bath to provide trapping. The sample area is pictured in Fig. 2 with the lower section of the faraday cup removed.

A 100 cm length of amperex thermocoax², 0.18 cm in diameter, was wound in a four layer pancake configuration. This is attached to the sample block as shown in Fig. 2, and used as the heater. The temperature is measured with a chromel-alumel thermocouple. This system is being used on an implantation machine with a vertical line source.

Therefore, a vertical tantalum strip mounted on the beam stop is used for horizontal alignment and focusing of the ion beam. The strip is electrically isolated from the beam stop. The beam stop can be rotated out of the ion beam path with a rod which extends outside the vacuum system.

Electrical connections are made through two Varian mini-coaxials,³ and there are two leads for the heater, one for -150 volt electron suppression, another lead for ion beam alignment,

and a thermocouple feedthrough. The electron suppression plate is mounted in front of the sample and the whole sample system is isolated from chassis ground for charge counting. Electrical power for all of the power supplies originates at the isolation transformer of the post acceleration supply.

The exchange gas sample system is mounted in an ion beam implant line (Fig. 3) using standard four inch Aero Vac⁴ couplings. If desired, the implant angle can be varied from zero to ten degrees by aligning degree marks which are inscribed on the sample holder mounting flange.

In Fig. 3 the feedthroughs on flange 1 are used to control the ion beam stop and to evacuate or pressurize the exchange gas chamber of the sample system. By opening valve No. 1 and valve No. 2 the exchange gas chamber can be evacuated. Valve No. 2 opens the exchange gas chamber to the ion beam line. In this way the main pumping system can evacuate the exchange gas chamber and the accelerator tube at the same time. All of the controls can be manipulated with a lucite rod which extends outside the high voltage enclosure (see Fig. 3). After the vacuum reaches 10^{-5} Torr the liquid nitrogen traps can be filled to help pump condensable vapors. When the vacuum has reached approximately 10^{-6} Torr the heater can be turned on. By applying 5.5 amperes direct current at 23 volts the sample block will reach 400°C in fifteen minutes with an ultimate temperature of 500°C.

At the conclusion of the experiment valve No. 2 is closed and the exchange gas from a lecture bottle is admitted by opening valve No. 3. This greatly increases the thermal coupling to

the liquid nitrogen trap, since the effective area of the cylindrical fins is $\approx 40.7 \text{ cm}^2$ with a separation of 0.32 cm, and the temperature of the sample block falls rapidly as the heat is dissipated in the liquid nitrogen bath. The losses due to the thin wall stainless steels walls are small when compared to the losses of heat to the closely coupled copper tubes.

Although the construction of this sample system is straightforward, there are a few details worthy of mention. The sample system is of welded and soldered construction, depending on design criteria. The 304 stainless steel cup and sample block junction were hydrogen brazed at 950°C, since this is the hottest area. The stainless steel cup is soft soldered to the liquid nitrogen trap to facilitate replacement of any of the components.

IV. RESULTS

The sample block was heated to 500°C, then cooled under three different exchange gas conditions. The results are shown in Figs. 4 and 5. The format of Fig. 5 is chosen to produce a straight line when Eq. 2 is applicable by choosing for the ordinate $y = \ln \left(\frac{T - t_0}{t_i - t_0} \right)$.

In the vacuum run, the sample block was allowed to cool while the exchange gas chamber was under a vacuum of 10^{-6} Torr. The heat loss was relatively slow as can be seen in the figures. The principle sources of heat loss under this condition are conduction along the mechanical support elements and radiation.

Losses due to conduction along the mechanical support elements can be estimated by using Eq. 2 where^{1,5}

$$K = 0.0358 \frac{\text{cal}}{\text{sec cm deg}} \quad D = 5.08 \text{ cm}$$

$$C_p = 21.18 \frac{\text{cal}}{\text{deg}} \quad A = 0.803 \text{ cm}^2$$

The resulting value of $-\frac{KA}{C_p D}$ is $-(62 \text{ min.})^{-1}$.

Referring to Fig. 5, the slope of the vacuum run line varies, especially in the higher temperature range, corresponding to $Y \approx 0$ to -5 . This is due to the fact that radiation is present in addition to conduction along the mechanical support elements. The observed slope of this line at lower temperatures ($Y \approx -0.5$ to -1.0), where the radiation contribution is much less, corresponds to about $-(50 \text{ min.})^{-1}$. This is in good agreement with the above theoretical slope $-(62 \text{ min.})^{-1}$.

Helium was used as the exchange gas in the next run. The rate of heat loss due to conduction through the exchange gas was estimated by using Eq. 2 where¹

$$K = 3.64 \times 10^{-4} \frac{\text{cal}}{\text{sec cm deg}} \quad D = 0.318 \text{ cm}$$

$$C_p = 21.18 \frac{\text{cal}}{\text{deg}} \quad A = 40.7 \text{ cm}^2$$

Using these values, $-\frac{KA}{C_p D}$ is $-(7.57 \text{ min.})^{-1}$. This compares well with the slope of the helium run on Fig. 5, which is $-(10 \text{ min.})^{-1}$.

In the nitrogen run, dry nitrogen from a liquid nitrogen container was used as the exchange gas at a pressure of 10 psi above atmosphere. The above atmospheric pressure dry nitrogen

strikes the cold liquid nitrogen temperature surface. This produced condensation of the dry nitrogen and the liquid nitrogen drops to the bottom of the stainless steel cup. When a sufficient amount collects the sample block is rapidly cooled. This effect is especially effective in the temperature range from 0 to -192°C . Thus the cooling is seen to be very rapid, particularly at longer times, and the slope in Fig. 5 is observed to increase with time.

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REFERENCES

1. M. W. Zemansky, Heat and Thermodynamics, McGraw-Hill Book Company, Inc., New York, 1957.
2. Amperex Electronic Corp., 250 Duffy Ave., Hicksville, L.I., New York 11802 (516) 931-6200.
3. Aero Vac Products, High Voltage Engineering Corp., South Bedford St., Burlington, Mass. 01803 (617) 272-0765.
4. Varian Associates, 611 Hansen Way, Palo Alto, CA 94303.
5. Victor J. Johnson, "A Compendium of the Properties of Materials at Low Temperatures (Phase 1) Part II," Wadd Technical Report 60-56, January 1961.

FIGURE CAPTIONS

- Fig. 1 Schematic of sample holder.
- Fig. 2 Photograph of sample area of the sample holder.
- Fig. 3 Photograph of the sample system installed in the
 implantation beam line.
- Fig. 4 Graph of temperature vs time for various exchange
 gas conditions.
- Fig. 5 Graph of Y vs time.

FIGURE #1

FIGURE #3

FIGURE #14