A simple continuous transfer liquid helium cryostat for an ultra-high vacuum field ion microscope

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Abstract A simple continuous transfer liquid helium cryostat for an ultra-high vacuum field ion microscope has been constructed. In this cryostat the liquid helium is continuously transferred from a conventional 30 or 50 l storage vessel, employing a standard transfer line, to a small reservoir which is in direct contact with the field ion microscope specimen holder. The enthalpy of the outgoing cold helium gas is used to cool a copper radiation shield which surrounds both the specimen holder and the reservoir section of the cryostat. The economical operating temperature range of this cryostat is 8°K to room temperature with a temperature stability of better than ±0·1 degK over most of this temperature range. Details of construction and the operational characteristics are presented.

1 Introduction

A number of field ion microscopists (e.g. Müller 1960, Brenner 1962, Attardo *et al.* 1966, Bowkett and Ralph 1966, Southon and Forbes 1966 unpublished) have used standard cryostats on their field ion microscopes, employing liquid nitrogen, neon, hydrogen and helium as refrigerants to cool their specimens. However, relatively few microscopists (e.g. Speicher, Wolff and Pimbley 1967, Klipping and Vanselow 1967, Seidman and Scanlan 1968 unpublished, Brandon 1968, Brenner 1968 private communication) have employed the dynamic or continuous flow method which is used in a number of other areas of scientific research (e.g. Swenson and Stahl 1954, Shull 1962, Klipping 1964, Barrett and Grodzins 1965). The continuous flow or dynamic method has the following important advantages.

(i) The specimen temperature can be readily and continuously varied from 4·2°K to room temperature using only liquid helium as the refrigerant.

(ii) The consumption of liquid helium decreases as the operating temperature increases. This must be compared with a conventional static cryostat which raises the specimen temperature by the input of an additional heat source to the specimen. The latter method *always* results in an *increase* in liquid helium consumption.

(iii) The radiation shields are cooled by the outgoing cold helium gas, which therefore avoids the use of liquid nitrogen for this purpose.

(iv) If the experiment is terminated due to a poor or 'popped' specimen there is almost no liquid helium wasted. This may be compared with a static cryostat where all the liquid helium in the reservoir is lost in such a situation.

In the present paper the details are presented of a relatively

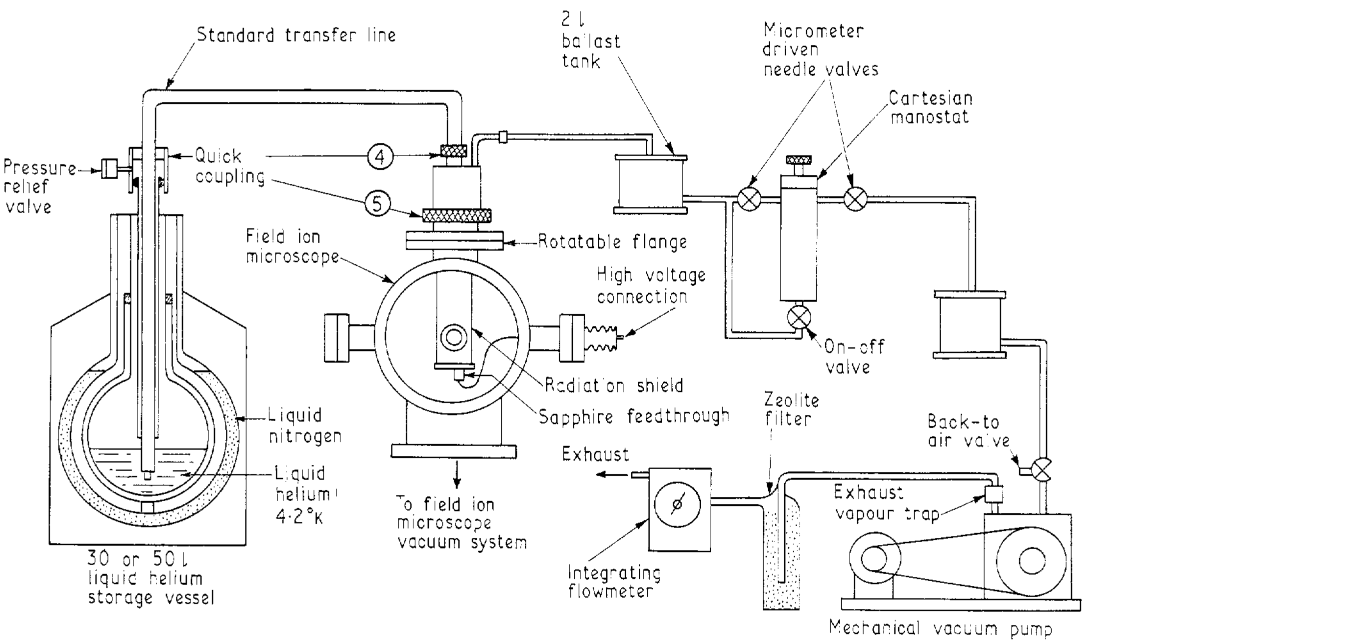

Figure 1 A schematic diagram of the pumping station used to control the flow of liquid helium to the cryostat. The pumping station is shown in its relationship to the helium storage vessel and field ion microscope

simple cryostat which has been used in conjunction with a bakeable ultra-high vacuum field ion microscope (Seidman and Bohlen 1968 unpublished). This cryostat design employs the dynamic method to cool the field ion microscope specimen. The refrigerant (liquid helium) is continuously transferred from a conventional 30 or 50 l storage vessel, with the aid of a standard transfer line, to a small reservoir which is in direct contact with the specimen holder. The cold outgoing gas is then used to cool a copper radiation shield which surrounds both the specimen holder and the reservoir section of the cryostat. The principle of operation, details of construction of the cryostat, and the operating characteristics are presented.

2 Principle of operation

The liquid helium is forced to flow from the storage vessel to the cryostat, via a transfer line, by creating an underpressure on the cryostat (see figure 1) side of the transfer line. The magnitude of the underpressure determines the flow rate of the liquid helium to the cryostat. Hence, a *low* flow rate of liquid helium yields a high temperature, and conversely a *high* flow rate produces a low temperature. Therefore, the temperature is changed by simply varying the flow rate of the refrigerant.

The underpressure is achieved by means of a Welch Duo-Seal mechanical pump which is part of a pumping station (Peterson and Simmons 1965) shown schematically in figure 1. This pumping station consists of a mechanical pump, a cartesian manostat, two micrometer driven needle valves, two ballast tanks, and an integrating flowmeter. The cartesian manostat (Manostat Corp., New York) is used to maintain a constant pressure drop across a micrometer driven needle valve which results in a constant flow rate of gas. The integrating flowmeter (manufactured by Precision Scientific Co, Chicago, Illinois) is used to monitor the consumption rate of liquid helium during the course of an experiment.

It is essential that *no* overpressure of helium gas be allowed to build up in the storage vessel. This is achieved by means of a 0·5 lb in^{-2} pressure relief valve (Circle Seal) which is part of the quick coupling used to attach the transfer tube (see figure 1) to the storage vessel. This pressure relief valve must be carefully adjusted to avoid a build-up of helium gas in the storage vessel which will also cause liquid helium to be transferred through the line.

3 Description of the cryostat

The entire cryostat is built on a rotatable Varian Conflat flange 1 (see figure 2) which mounts on an essentially all stainless-steel ultra-high vacuum field ion microscope (Seidman and Bohlen 1968 unpublished). The central 1·27 cm diameter tube 2 (304 stainless-steel, 0·0254 cm wall thickness) receives a standard transfer line 3† with a 0·953 cm diameter outer vacuum jacket. The transfer line is connected to the cryostat by means of two vacuum-tight quick couplings 4 and 5 which employ viton O-rings.

The liquid helium flow tube 6 (304 stainless steel, 0·318 cm diameter, 0·0254 cm wall thickness) is positioned so that the liquid exits into a small reservoir located near the OFHC copper piece 7 which is used to secure the electrical insulating sapphire specimen holders to the cryostat. Sapphire was chosen for this purpose because of its high dielectric strength and good low temperature thermal conductivity (White 1959). In order to ensure a high thermal conductivity connection between the sapphire‡ specimen holder and the copper piece,

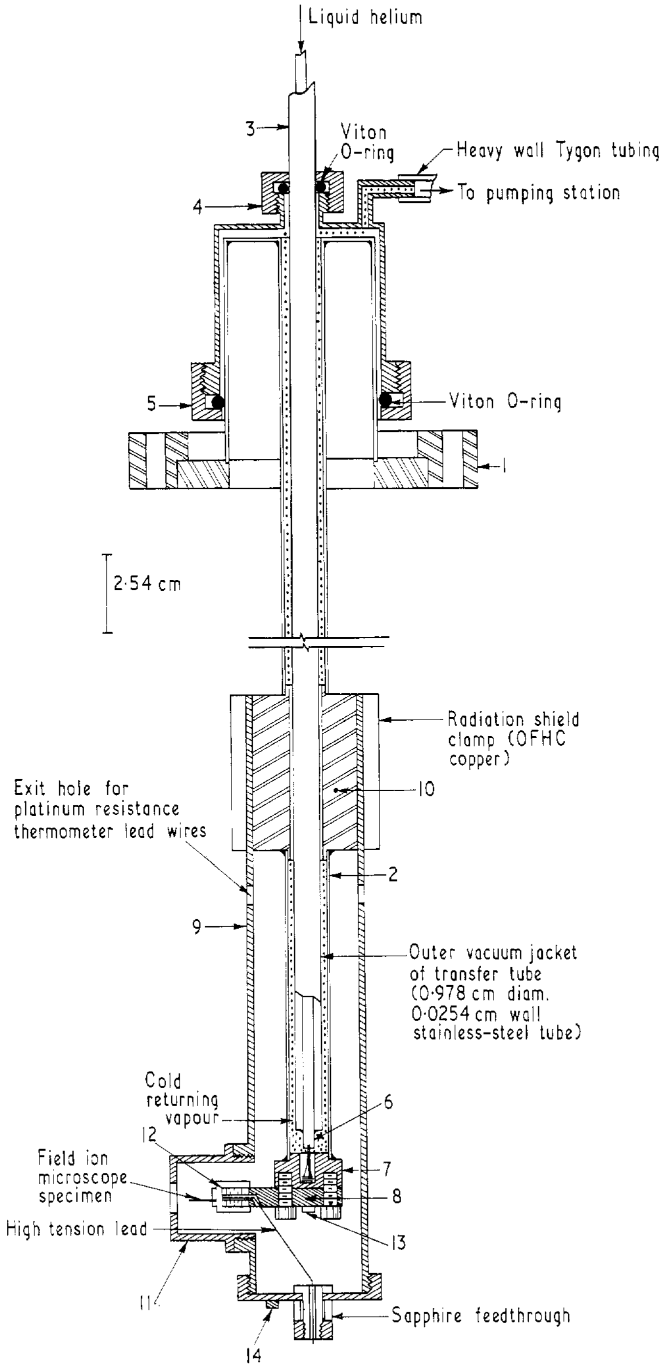

Figure 2 A cross-sectional side view of the cryostat with the transfer tube inserted in its operational position. During bakeout the brass quick couplings 4 and 5 and their associated viton O-rings are removed from the cryostat. The numbered components are referred to in the text

a 0·003 cm thick well-annealed gold foil was placed between these two sections.

The reservoir and the specimen holder are surrounded by a highly polished OFHC copper radiation shield 9 which is cooled by the outgoing cold helium gas. The radiation shield is clamped to a cylindrical copper piece 10 which acts as a heat exchanger. The clearance between the vacuum jacket of the transfer tube and the heat exchanger is less than 1 mm in order to ensure turbulent flow and therefore a high

† This transfer line was fabricated in the machine shop of the Laboratory of Atomic and Solid State Physics at Cornell University from their standard set of plans

‡ The sapphire piece was machined and polished to specifications by Insaco Inc, Quakertown, Pennsylvania, USA.

Reynold's number in this region. A high Reynold's number at this point is essential for achieving good convective heat transfer (see White 1959) between the outgoing cold gas, and the copper heat exchanger.

The main heat input into the present system is a result of the fact that an appreciable length (~140 cm) of the central 0·318 cm diameter stainless-steel liquid helium flow tube, of the transfer line, is exposed to a surface which is at room temperature. This radiation loss has been eliminated in the design employed by Seidman and Scanlan (1968 unpublished) with a resultant decrease in liquid helium consumption.

The field ion microscope specimen is inserted by removing the front radiation shield 11 from the cryostat and screwing the OFHC copper specimen holder 12 on to the threaded section of the sapphire block. This is a relatively easy operation which is accomplished in a few minutes.

The temperature of the specimen is continuously monitored with a miniature platinum resistance thermometer 13 (model 118G Rosemount Engineering Corp., Minneapolis, Minnesota, USA) which is mounted very close to the specimen. The resistance of the thermometer is measured by standard potentiometric methods. The validity of the temperature measured by this thermometer was verified by using a second platinum resistance thermometer which was placed at the position where the specimen normally resided. The two thermometers agreed with one another to better than ±0·5 degK over the entire temperature range from 8°K to room temperature. The temperature of the radiation shield is measured with a Chromel–constantan thermocouple 14 which is placed at a position which is as far as possible from the heat exchanger section.

4 Operational characteristics

The operational characteristics between 12 and 34°K are shown in figure 3. It is seen that between these two temperatures the consumption of liquid helium varies from 1·1 l h^{-1} to 0·3 l h^{-1} with a pressure of helium gas of 2 mtorr inside the field ion microscope. The relaxation time of the specimen from room temperature to 12°K is less than 20 min with an associated liquid helium consumption of less than 0·3 l. The time for the radiation shield to reach its steady-state temperature of 34°K is about a factor of two longer than the time for the specimen to reach 12°K. At a specimen temperature of 34°K the radiation shield achieved a temperature of 45°K. The lowest temperature at which this particular cryostat has been operated is 8°K with a liquid helium consumption of 1·5 l h^{-1}. Hence, the economical usable temperature range of this cryostat is 8°K to room temperature. Throughout this entire temperature range the specimen temperature stability is *better* than ±0·1 degK (with the exception of the small temperature range from 8 to 11°K where there are temperature oscillations of up to ±0·5 degK).

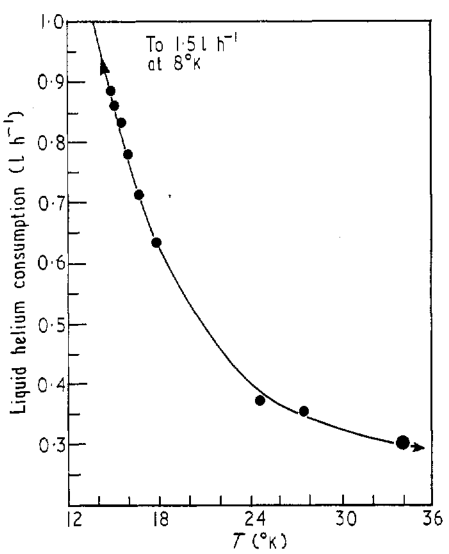

Figure 3 The liquid helium consumption as a function of the specimen temperature. The pressure of helium gas inside the microscope was 2 mtorr. The temperature of the radiation shield varied from 34 to 45°K as the specimen temperature varied from 12 to 34°K. The integrating flowmeter used for the measurements reported in this figure is a Precision Scientific Company (Chicago, Illinois) wet test gas meter (catalogue number 63126)

The present cryostat design compares favourably with the continuous flow system of Klipping and Vanselow (1967) who reported a liquid helium consumption of 0·5 l h^{-1} at 20°K in a pressure of 4 mtorr of helium. It is also interesting to note that the cost to operate the cryostat at 27·2°K is $1.06 per h.† This is considerably less expensive than the cost of $5.60 per h (assuming a consumption rate of 50 cm^3 h^{-1}, no rebate for recovered gas, and a price of $112 per litre for liquid neon) reported by Bowkett and Ralph (1966) for a static cryostat employing liquid neon as the refrigerant (boiling point of liquid neon, 27·2°K).

5 Conclusions

A simple cryostat has been constructed for a bakeable ultra-high vacuum field ion microscope. This cryostat has the following features and characteristics.

1 The specimen temperature is controlled by varying the flow rate of liquid helium to the reservoir of the cryostat.

2 The radiation shield of the cryostat is cooled by employing the enthalpy of the outgoing cold helium gas; this avoids the use of liquid nitrogen for this purpose.

3 The relaxation time of the specimen from room temperature to 12°K is less than 20 min.

4 The liquid helium consumption varies from 1·5 l h^{-1} at 8°K to 0·3 l h^{-1} at 34°K with a pressure of 2 mtorr helium gas inside the field ion microscope.

5 Temperature stability is better than ±0·1 degK over most of the temperature range from room temperature to 8°K. This stability is achieved via a pumping station which employs a cartesian diver to maintain a constant pressure drop across a micrometer driven needle valve which regulates the flow.

6 The temperature of the radiation shield varies from 34 to 45°K as the specimen temperature is varied from 12 to 34°K respectively.

The continuous transfer liquid helium cryostat described above has been used for a study of the temperature and crystallographic dependence of the field ionization characteristics of tungsten surfaces (Bohlen 1969 M.Sc. Thesis, Cornell University, New York). The design has proved to be very satisfactory for the study of this temperature dependent phenomenon. A slightly modified version of the same basic design has also been employed for a field ion microscope study of the annealing behaviour of tungsten specimens irradiated with tungsten ions (W^-) and has proved to be quite successful for this purpose (Scanlan, Styris and Seidman, unpublished).

† This is based on the price of $3.05 per litre for liquid helium when purchased in 50 l storage vessels and without a rebate for recovered helium gas. The price is 25% lower if the helium gas is recovered

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References

Attardo J J Galligan J M and Sadofsky J 1966 *J. Sci. Instrum.* **43** 607–8

Barrett P H and Grodzins L 1965 *Rev. Sci. Instrum.* **36** 1607–10

Bowkett K M and Ralph B 1966 *J. Sci. Instrum.* **43** 703–7

Brandon D G 1968 *Adv. in Optical and Electron Microscopy* **2** 343–404

Brenner S S 1962 *Metal Surfaces: Structure, Energetics and Kinetics* (Ohio: American Society for Metals) pp. 305–88

Klipping G 1964 *Chem. Ing. Tech.* **36** 430–41

Klipping G and Vanselow R 1967 *Z. Physik. Chem.* **52** 196–203

Müller E W 1960 *Advances in Electronics and Electron Physics* (New York: Academic Press) pp. 83–179

Peterson O G and Simmons R O 1965 *Rev. Sci. Instrum.* 1316–8

Shull C G 1962 *Massachusetts Institute of Technology Laboratory Report*

Speicher C A Wolff C W and Pimbley W T 1967 *J. Sci. Instrum.* **44** 167–8

Swenson C A and Stahl R H 1954 *Rev. Sci. Instrum.* **25** 608–11

White G K 1959 *Experimental Techniques in Low Temperature Physics* (London: Oxford University Press) Chap. 3 p. 311