A bakeable, demountable field ion microscope with a continuous transfer liquid helium cryostat

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Abstract A stainless field ion microscope head which is bakeable to 250°C and completely demountable is described, and the operational characteristics are presented. The microscope employs the continuous flow technique to cool the specimen cryogenically between 4·2 and 78·K. The liquid helium consumption varies from 0·85 l h⁻¹ to 0·133 l h⁻¹ between 11·7 and 34·8·K, with a pressure of 2·5 mtorr helium inside the microscope. The field ion microscope head is particularly well suited for hydrogen promotion experiments. An example of a gold specimen imaged by the latter technique in the present microscope is given.

1 Introduction
The present paper describes an all stainless-steel field ion microscope head which is bakeable to 250°C and completely demountable. (The head is inherently bakeable to 400°C although in practice it is only baked to a maximum temperature of 250°C for 3 h.) The microscope employs the continuous flow method (Seidman and Scanlan 1968 unpublished, Seidman et al. 1969) to cool the specimen cryogenically. The continuous flow method has the advantage that the temperature of the specimen can be readily and continuously varied from 4·2·K to room temperature using liquid helium as the refrigerant. This method of cooling also has the advantage that the liquid helium consumption decreases as the operating temperature of the field ion microscope increases. The microscope head is dynamically pumped with an oil-diffusion pump type system which is isolated from the head by a liquid nitrogen cold trap (Granville-Phillips Co.). This system routinely and quickly yields a background pressure in the range 5–10 mtorr nitrogen equivalent.

In the following sections the details of construction of the microscope head are described, the liquid helium consumption as a function of temperature is given, and a sample field ion micrograph of a gold specimen imaged in this microscope is presented.

2 Description of the field ion microscope head
A cross-sectional side view of the head is shown in figure 1. The general L-shaped geometry of the field ion microscope is similar to the geometry which was employed by Southon and Brandon (1963), Monatgu-Pollock and Rhodin (1966) and Plummer (1968 PhD Thesis, Cornell University). The three essential sections of the head are as follows: (i) liquid nitrogen and liquid helium cryostats; (ii) radiation shields and conduction rod; (iii) specimen mount.

2.1 Liquid nitrogen and liquid helium cryostats
The cryostat section of the field ion microscope consists of an outer liquid nitrogen cryostat and an inner liquid helium cryostat. The liquid nitrogen cryostat is constructed from a 5·08 cm outside diameter stainless-steel tube (see figure 1)† and a 2·54 cm outside diameter tube 2 which are concentrically brazed to a fairly massive copper block 3. The central 1·27 cm outside diameter tube 4 with 0·0254 cm wall thickness serves as the liquid helium cryostat and is attached to the microscope via a Varian Mini-Conflat flange 5. This connection is submerged in liquid nitrogen during operation to minimize the heat flux to the lower end of the tube. Note that the liquid helium exits from a standard transfer tube at the lower end of the central 1·27 cm outside diameter tube 6. This central tube was brazed to an OFHC copper fitting 7 which receives the OFHC copper conduction rod 8. The liquid helium transfer line is connected to the 1·27 cm outside diameter tube by means of a modified quick coupling (not shown in figure 1).

2.2 Radiation shields and conduction rod
The outermost copper radiation shield 9 (see figure 1) is attached to the liquid nitrogen cryostat via a threaded connection. This radiation shield is maintained at a temperature of 78·K over its entire length (verified by a thermocouple measurement of temperature) during the operation of the field ion microscope. A 0·635 cm outside diameter OFHC copper conduction rod 8 is used to cool the specimen. This conduction rod is screwed into its fitting until a pressure of approximately 200–300 kg mm⁻² is achieved. Surrounding this rod is an intermediate OFHC copper radiation shield 10 which is essential in order to reach the low temperatures reported in this paper.

2.3 Specimen mount
The specimen holder is a cylindrical OFHC copper cap 11 with a nickel bridge (not shown) brazed on to the cap. The specimen is spot-welded to this nickel bridge. The copper specimen holder is screwed on to an intermediate sapphire block 12 (machined to specifications by INSACO Inc, Quakertown, Pennsylvania) which electrically insulates the specimen from the microscope and simultaneously provides a good thermal connection (White 1959) to the 0·635 cm outside diameter conduction rod. This specimen holder is inserted into the microscope by removing the 10·16 cm diameter viewing screen 13 which is mounted on a 7 cm outside diameter Varian Conflat flange. The entire procedure of specimen exchange is accomplished in a few minutes. A platinum resistance thermometer 14 (model 118G, Rosemount Engineering Corp., Minneapolis) was inserted between the

† All tubulation is 304 stainless steel unless noted to the contrary.
Figure 1 A cross-sectional side view of the field ion microscope head. The numbered components are referred to in the text. The figure is not to scale, hence the actual angle subtended by the screen at the specimen is larger than the angle indicated in this figure.

sapphire–copper conduction rod interface in order to monitor the specimen temperature. The validity of the temperature measured by this thermometer was verified by a series of experiments using a second platinum resistor thermometer which was placed at the point where the specimen normally resided. The maximum temperature difference observed between the two thermometers is 1 degC. In order to ensure good thermal contact between interfaces 0.003 cm thick well-annealed gold foils are placed at all junctions. We at present measure temperature with a special platinum resistance thermometer which was constructed by wrapping 0.0051 cm diameter platinum wire around the 0.635 cm outside diameter copper conduction rod 8 using fibreglass as an insulator, as near as possible to the specimen holder. This thermometer was also calibrated with a second platinum resistance thermometer.

2.4 Other parts

In addition, the field ion microscope head has a total of four access ports (only one port is shown in figure 1) not counting the port which receives the viewing screen 13. One of these ports is used for an ionization gauge tube (not shown in figure 1), while the remainder are used for variable leak valves (Varian model No 951-5100) which admit the various imaging gases. For example, a partial pressure of hydrogen is obtained inside the microscope by first diffusing this gas through a heated Pd–25% Ag thimble (K-B Glass Co, New York) and then admitting the diffused hydrogen gas to the microscope via a Varian variable leak valve. The present head is particularly useful for hydrogen promotion experiments (see Müller et al. 1965), because of the fact that the head is dynamically pumped with an oil diffusion pump system. This dynamic system is useful because it provides rapid removal of the
A bakeable field ion microscope with liquid helium cryostat

hydrogen gas inside the head and avoids the problem of hydrogen saturation which is common for ion pumps (see Whitnell 1967).

3 Operational characteristics

The microscope is normally operated in the temperature range 4.2-78°K using liquid helium as the refrigerant. In practice the field ion microscope is first pre-chilled to 78°K using liquid nitrogen and then cooled from 78°K to the desired imaging temperature with liquid helium. The liquid helium consumption as a function of temperature between 11.7 and 34.8°K is shown in figure 2. It is seen that between these two temperatures the consumption of liquid helium varies from 0.85 l h⁻¹ to 0.133 l h⁻¹ with a partial pressure of 2.5 mtorr helium gas inside the microscope. The pumping station used to maintain a given flow rate of liquid helium, and hence a given temperature, is described by Seidman et al. (1969).

Figure 2  The liquid helium consumption as a function of temperature between 11.7 and 34.8°K. The flow rate was measured with a Precision Scientific (model 63126) wet test gas meter. Helium pressure inside the microscope was 2.5 mtorr

The cost‡ to operate this microscope at 20.4°K (boiling point of liquid hydrogen) is $0.95 per h, and at 27.2°K (boiling point of liquid neon) the cost is $0.63 per h. Thus, the operating expense of the present microscope at 27.2°K is $0.43 per h lower than for the cryostat previously reported on by Seidman et al. (1969). Once again, it is interesting to note that the operating cost at 27.2°K is considerably lower than the $5.60 per h reported by Bowkett and Ralph (1966) for a field ion microscope with a static cryostat employing liquid neon as the refrigerant. The general point that we wish to make is that it is far more economical and simple to use liquid helium as a refrigerant for the entire temperature range between 4-2 and 78°K, than it is to use a combination of refrigerants (liquid helium, hydrogen, neon and nitrogen) to achieve various temperatures in this range.

Figure 3  A field ion microscope pattern of a gold surface obtained in the present microscope. The initial surface was field evaporated in a 0.5% H₂–He gas mixture. The imaging gas was a 25% Ne–He gas mixture. The numbers between the indexed poles are in angstroms and indicate the local radii of curvature. The image was recorded at an aperture opening of f/1.4 on a Super Farron lens. Exposure time was 4 s with the aid of a two-stage image intensifier tube (Radio Corporation of America model C33011). The imaging temperature was 16°K

The specimen may readily be heated above room temperature by radiant heating from a tungsten filament. The present microscope does not incorporate this design feature, but it is intended to install this feature in the near future.

A pattern of a gold surface obtained by Ast and Seidman (1968) in the present microscope is shown in figure 3. This pattern was obtained by first field evaporating the gold specimen in a 0.5% H₂–He gas mixture (3 mtorr total pressure), removing the hydrogen gas, and then imaging the surface in a 25% Ne–He gas mixture (background pressure ~7 mtorr nitrogen equivalent). The gold specimen was at a best image voltage of 7.2 kv and a temperature of the order of 16°K.

4 Summary

A stainless-steel field ion microscope head has been constructed with the following characteristics. (i) The field ion microscope head is bakeable to 250°C and completely demountable (figure 1). (ii) The continuous flow method is used to cool the specimen cryogenically between 4.2 and 78°K. (iii) The liquid helium consumption varies from 0.85 l h⁻¹ to 0.133 l h⁻¹ between 11.7 and 34.8°K (figure 2). (iv) The present microscope is well suited for using the hydrogen promotion technique to image relatively soft non-refractory metals. An example of a gold surface (figure 3) imaged by this technique with the present field ion microscope is shown.

† This is based on the price of $3.05 per litre of liquid helium.

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References


Müller E W Nakamura S Nishikawa O and McLane S B 1965 J. Appl. Phys. 36 2496–503


Southon M J and Brandon D G 1963 Phil. Mag. 8 579–91
