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LIQUID HELIUM FIELD-ION MICROSCOPE FOR RADIATION DAMAGE INVESTIGATIONS

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ABSTRACT

A field ion microscope is described which allows "in situ" irradiation of a metal specimen at a temperature close to  $5^{\circ}\text{K}$ . It has been used with the 88" Cyclotron at Berkeley which provides 10 MeV protons. The design permits "field off" irradiation of the specimen, as well as "in situ" temperature cycling. Qualitative observations on the surface damage of iridium upon "field off" and "field on" irradiation are given.

## I. INTRODUCTION

Because of its high resolution, the field ion microscope<sup>1</sup> enables direct observation of atomic structure at the surface of a metal. Experiments by Müller,<sup>2</sup> Brandon and Wald,<sup>3</sup> and Sinha and Müller<sup>4</sup> have proved its usefulness in analyzing the effects of radiation damage in metals. The liquid helium field ion microscope described in this paper was built primarily for direct observation of the defects produced by irradiation and the changes that accompany subsequent heating. It allows irradiation of the specimen inside the field ion microscope at 5°K and can be used with a variety of radiation sources. For the present experiments the instrument was set at the end of the beam pipe of the 88" cyclotron of the Berkeley Lawrence Radiation Laboratory. 10 MeV protons were used as the bombarding particles.

## II. DESCRIPTION OF THE INSTRUMENT

The microscope chamber (Fig. 1) is composed of a large stainless steel dewar (A) holding liquid nitrogen and surrounding the liquid helium cold finger (B). The stainless steel cold finger is surrounded by a silver-plated copper heat shield (C) to minimize the evaporation rate of liquid helium. The cold finger is composed of a liquid helium tank encased by a vacuum jacket (D) which is attached to the thermal shield through two thin wall stainless steel tubes (E). An alumina specimen holder (F) in which the specimen is clamped with a copper block was used to insure proper cooling of the specimen as well as electric insulation of the microscope chamber. Figure 2 shows a detailed view of the specimen holder and of the high voltage lead which is fed into the microscope chamber through a glass-Kovar tube mounted on a 1-1/2"

flange. Good thermal contact between the copper block at  $4.3^{\circ}\text{K}$  and the alumina specimen holder is insured through an indium-gallium alloy coating on the copper threads and a deposit of gallium vaporized on the threaded part of the specimen holder.

The high voltage line is made of a 0.01 cm. diameter inconel lead which is effectively thermally anchored on an alumina block at  $77^{\circ}\text{K}$  and then connected to the specimen holder.

The ultra-high vacuum was obtained through an oil diffusion pumping system and a vac ion pump. Two bakeable valves allowing to switch from the vac ion pump which was used during ultra-high vacuum experiments to the oil diffusion pumping system which was used for pumping the imaging gas.

The entire system is bakeable to  $300^{\circ}\text{C}$  which allows a vacuum of  $10^{-8}$  Torr to be attained in 4 to 6 hours pumping time, and a vacuum of  $10^{-9}$  Torr with the liquid nitrogen and the liquid helium filled in the microscope.

For a precooled cold finger, the liquid helium consumption was found to be strongly dependent on the gas pressure in the microscope chamber as shown in Table I.

Table I.

Liquid Helium Consumption	Helium Gas Pressure
500 cc/hour	$1 \times 10^{-4}$ Torr
800 cc/hour	$1 \times 10^{-3}$ Torr
1000 cc/hour	$3 \times 10^{-3}$ Torr
1500 cc/hour	$5 \times 10^{-3}$ Torr

At the attachment of the field ion microscope to the cyclotron beam pipe, a 0.01 cm. thick aluminum foil was inserted in the connecting flange (G), thus providing isolation of the vacuum system of the microscope from the cyclotron vacuum system.

The temperature of the specimen was measured with a resistance thermometer made of a 4 cm. long loop of annealed platinum wire. This wire (99.999% pure) was mounted between two copper attachment points which were screwed and glued into the specimen holder (Fig. 1). The wire was then spot welded in its middle to the specimen. The current and potential leads were fed into the microscope chamber in the same way as the high voltage lead. They were also thermally anchored to alumina blocks at 77°K before being spot welded to the platinum loop.

Prior to use inside the microscope the resistance thermometer mounted on the specimen holder was calibrated between 4°K and 273°K using standard resistivity measurement methods. With a well pre-cooled cold finger filled with liquid helium the specimen temperature, as measured with this method, was 5°K.

Cooling of the specimen from liquid nitrogen to liquid helium temperature<sup>5,6</sup> led to the expected improvement in image resolution and quality. Figures 3(a) and (b) show the same iridium surface at 77°K and 5°K. Both pictures were recorded under the same imaging voltage and imaging gas pressure in order to observe only the temperature effects on the image features.

"In situ" temperature cycling between 5°K and temperatures  $T \geq 14^\circ\text{K}$  was obtained by letting the liquid helium evaporate and the cold finger warm up to the desired temperature. The temperature increase



of the cold finger was measured with a Cu-Cu constantan thermocouple held in contact with the copper block at the bottom of the cold finger (Fig. 2). The specimen temperature was deduced from these measurements by using the results of a calibration run in which both the specimen and the copper block temperatures had been measured simultaneously.

All irradiation and temperature cycling experiments were performed with the vacuum inside the microscope chamber and with the specimen holder stripped of the resistance thermometer (Fig. 2).

### III. QUALITATIVE OBSERVATIONS

The radiation damage resulting from 10 MeV protons was observed by comparing the specimen surface before and after irradiation and after successive field evaporation. The direct comparison method requires that no changes other than those induced by the bombarding particles occur at the surface, i.e., there should not be any contamination or corrosion of the surface during the irradiation time. Such a condition is easily achieved so long as the high electrostatic imaging field is continuously applied to the specimen, since it ionizes all gas impurities before they reach the specimen surface. This technique has been successfully used by Brandon and Wald,<sup>2</sup> Sinha and Miller<sup>3</sup> in their investigations of radiation damage. However, the imaging electrostatic field introduces up to 10% dilatation in the specimen lattice<sup>7</sup> and this may affect considerably the nature of the damage at the surface and even in the bulk of the specimen.

The present system, because of the ultra high vacuum and because the cyclotron could provide a short pulse at a high flux density, allows "field off" irradiation, i.e., no imaging field is applied and the lattice is not distorted during the bombardment time.

Prior to irradiation, the contamination and corrosion rates of the specimen surface were observed as a function of time when the imaging field was not applied. For a vacuum of  $10^{-9}$  Torr in the microscope the first changes of the specimen surface appeared after 8 minutes under "field off" conditions.

Figure 4 (a,b) shows the surface damage produced on an iridium specimen by 10 MeV protons at liquid helium temperature, with the "field off". The total flux during the 1 minute pulse, as measured on the dummy target, was about  $10^{14}$  protons spread over a 1" diameter circular area. Changes in the surface layer of the specimen due to the proton bombardment were identified by using an image comparator.<sup>1</sup> The changes consisted mostly of surface vacancies, the partial evaporation of atomic layers on several planes, and the appearance of a few interstitials. Figure 5 (a) and (b) shows the surface damage produced on an iridium bicrystal at liquid nitrogen temperature with 10 MeV protons. This specimen was irradiated "field on" with a flux of about  $10^{14}$  protons spread over a 1" diameter circle. In this case the surface damage was more extensive than in the "field off" irradiated specimen.

Further experiments are required before it can be definitely concluded that the lattice distortion due to the imaging field is the cause of this more extensive damage.

The annealing characteristics of radiation induced defects have been studied for the annealing stages 1, 2, 3, and the results will be presented in a later paper.

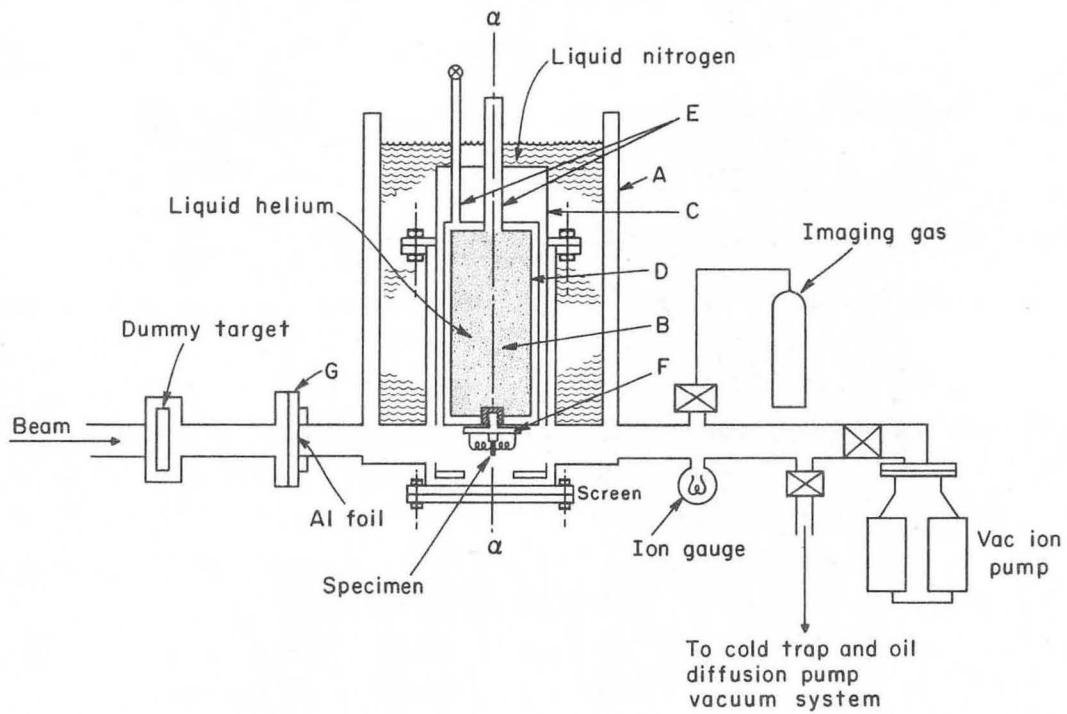
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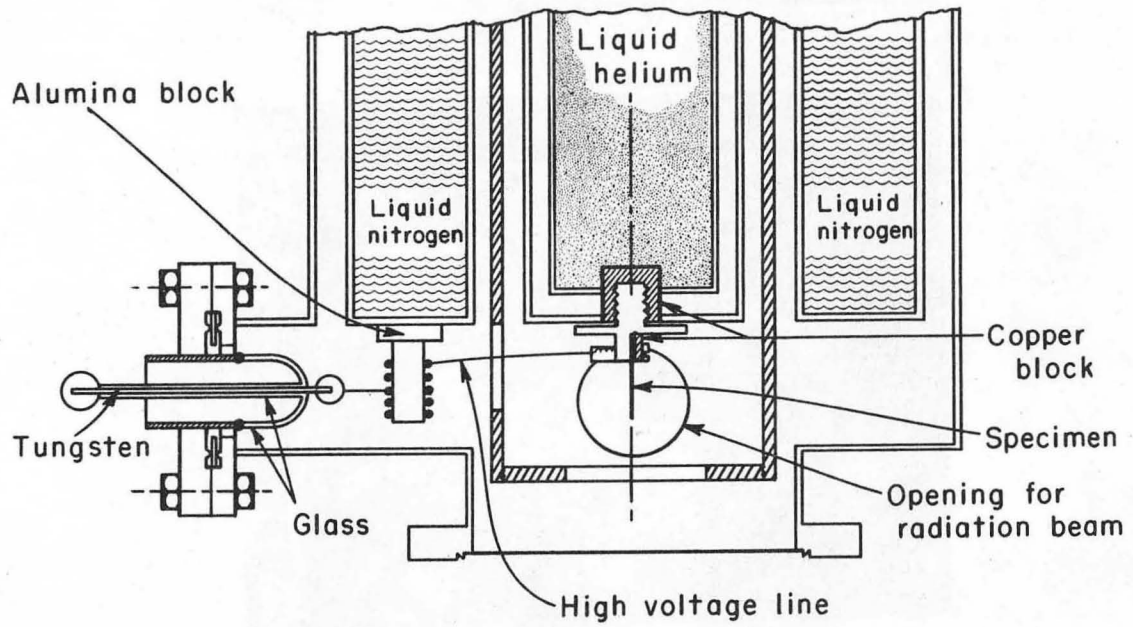
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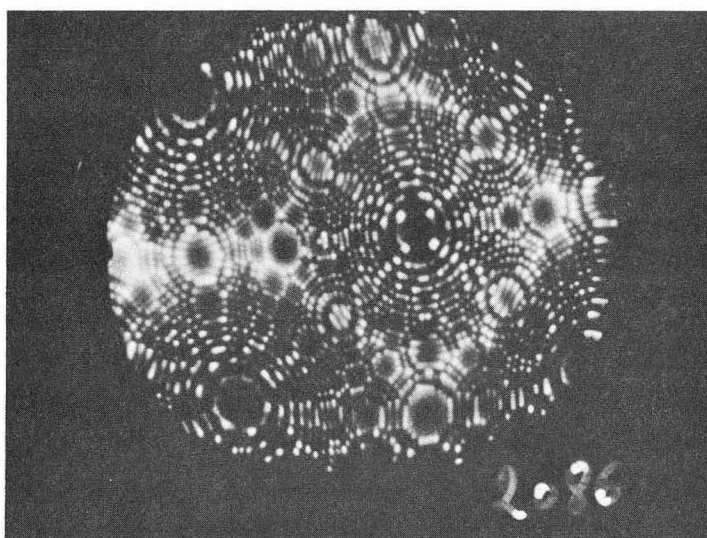
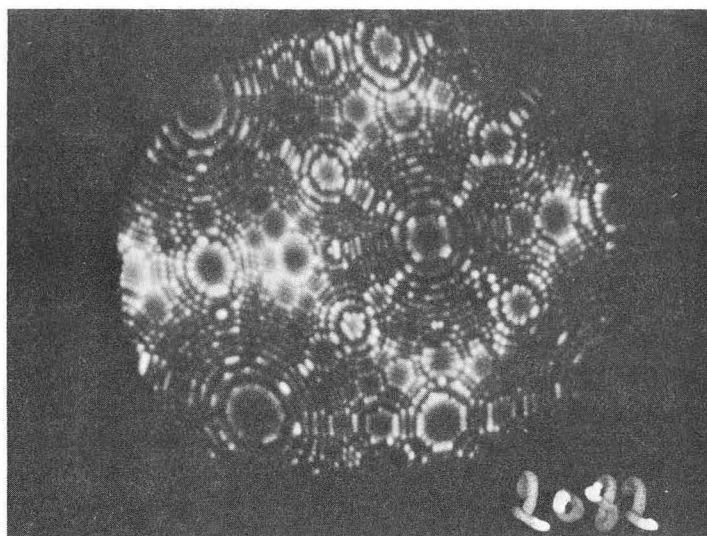
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Fig. 1 Schematic diagram of microscope chamber and experimental set up.



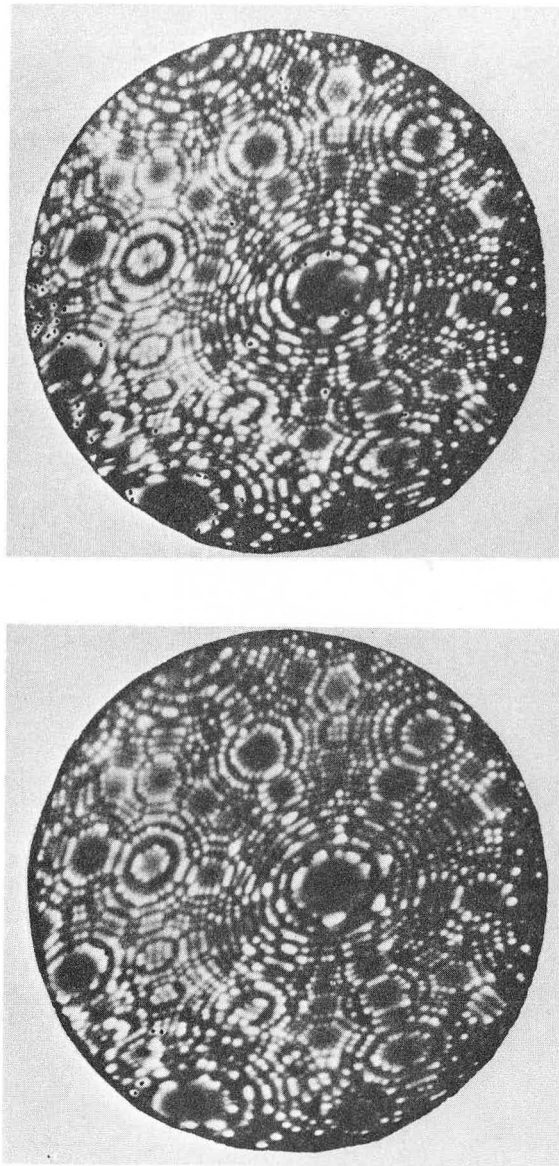
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Fig. 2 Detailed view of specimen holder and high voltage lead. (Cross section through plane  $\alpha$  shown in Fig. 1).



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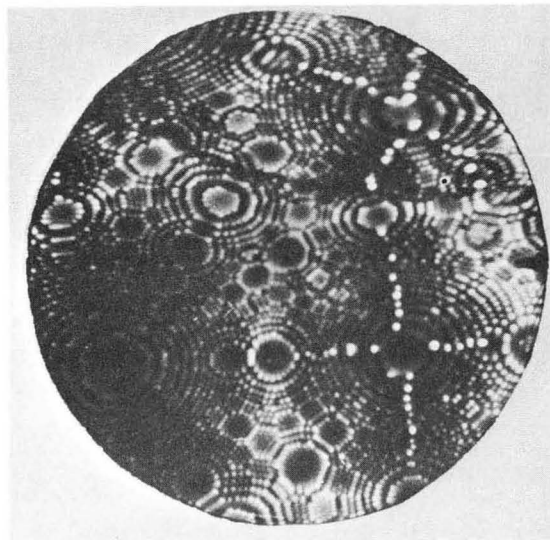
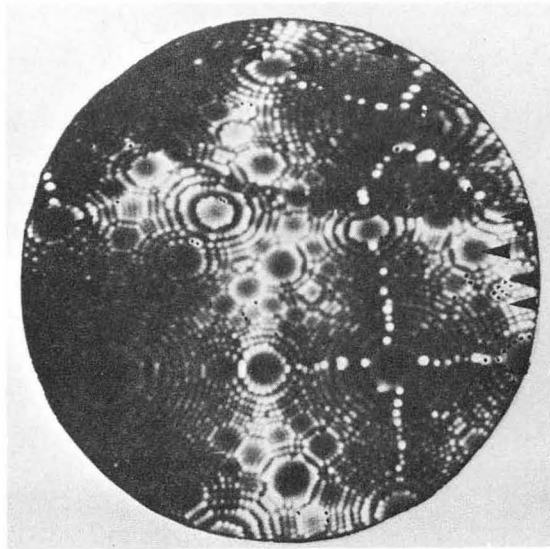
Fig. 3 Iridium single crystal (a) at 77°K, (b) same surface at 5°K. (Picture taken with the same imaging voltage and imaging gas pressure as Fig. 3a).



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Fig. 4 Iridium single crystal before and after "field off" irradiation with 10 MeV protons at a temperature below 10°K. (a) before, (b) after irradiation. Dotted atoms in (a) have been ejected from the surface during irradiation and are missing in (b). Extensively damaged regions are indicated by arrows. Dotted atoms in (b) are surface interstitials and are missing in (a).





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Fig. 5 Iridium bicrystal before (a) and after (b) "field on" irradiation at liquid nitrogen temperature. Missing atoms, damaged regions and interstitials identified as in Fig. 4.

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