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ABSTRACT

A field ion microscope is described which allows "in situ" irradiation of a metal specimen at liquid helium temperature. 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. Radiation damage in iridium and the migration and clustering of defects that accompany subsequent heating of the specimen have been studied.

I. INTRODUCTION

Because of its high resolution, the field ion microscope[1] enables direct observation of atomic structure at the surface of a metal. Experiments by Brandon and Wald[2], Sinha and Müller[3] 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 cryotip (B). The stainless steel cryotip is surrounded by a copper heat shield (C) to minimize the rate of evaporation of liquid helium. The cryotip 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 was used to insure proper cooling of the specimen as well as electric insulation of the microscope chamber. The high voltage lead and the thermocouple leads were led into the microscope chamber through a 1 1/2" flange seen in Fig. 2a. The ultra-high vacuum was obtained through an oil diffusion pump and a vac ion pump. The entire system is bakeable to 400°C which allows a vacuum of 10^{-9} Torr to be attained in 4 to 6 hours pumping time with the liquid nitrogen and liquid helium filled. For a precooled cryotip, 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	1×10^{-4} Torr
800 cc/hour	1×10^{-3} Torr
1000 cc/hour	3×10^{-3} Torr
1500 cc/hour	5×10^{-3} Torr

At the attachment of the field ion microscope system to the cyclotron beam pipe, a 3 mil 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 field ion microscope assembly coupled to the cyclotron beam pipe is shown in Fig. 2a, b. With a precooled cryotip filled with liquid helium, the specimen temperature, as measured with a resistance thermometer was 5°K. Cooling of the specimen to liquid helium temperature[4] led to the expected improvement in image resolution and quality as shown in Fig. 3(a), (b).

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,[2] Sinha and Muller[3] in their investigations of radiation damage. However, the imaging electrostatic field introduces up to 10% dilatation in the specimen lattice and this may affect considerably the nature of the damage at the surface and even in the bulk.

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} 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^{15} 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.[1] 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^{15} 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.

ACKNOWLEDGEMENTS

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2. D. G. Brandon and M. Wald, Phil. Mag. 6, 1035 (1961).

3. M. K. Sinha and E. W. Müller, J.A.P. 35, 4, 1256 (1964).

4. M. J. Attardo, J. M. Galligan and J. Sadofski, J. Sci. Instrum. 43, 607 (1966).

FIGURE CAPTIONS

Fig. 1 Microscope chamber.

Fig. 2a Field ion microscope coupled to the 88" Cyclotron of the Lawrence Radiation Laboratory, Berkeley.

Fig. 2b Schematic diagram of experimental set up.

Fig. 3 Iridium single crystal (a) at 77°K; (b) same surface at 5°K.

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

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. 3.

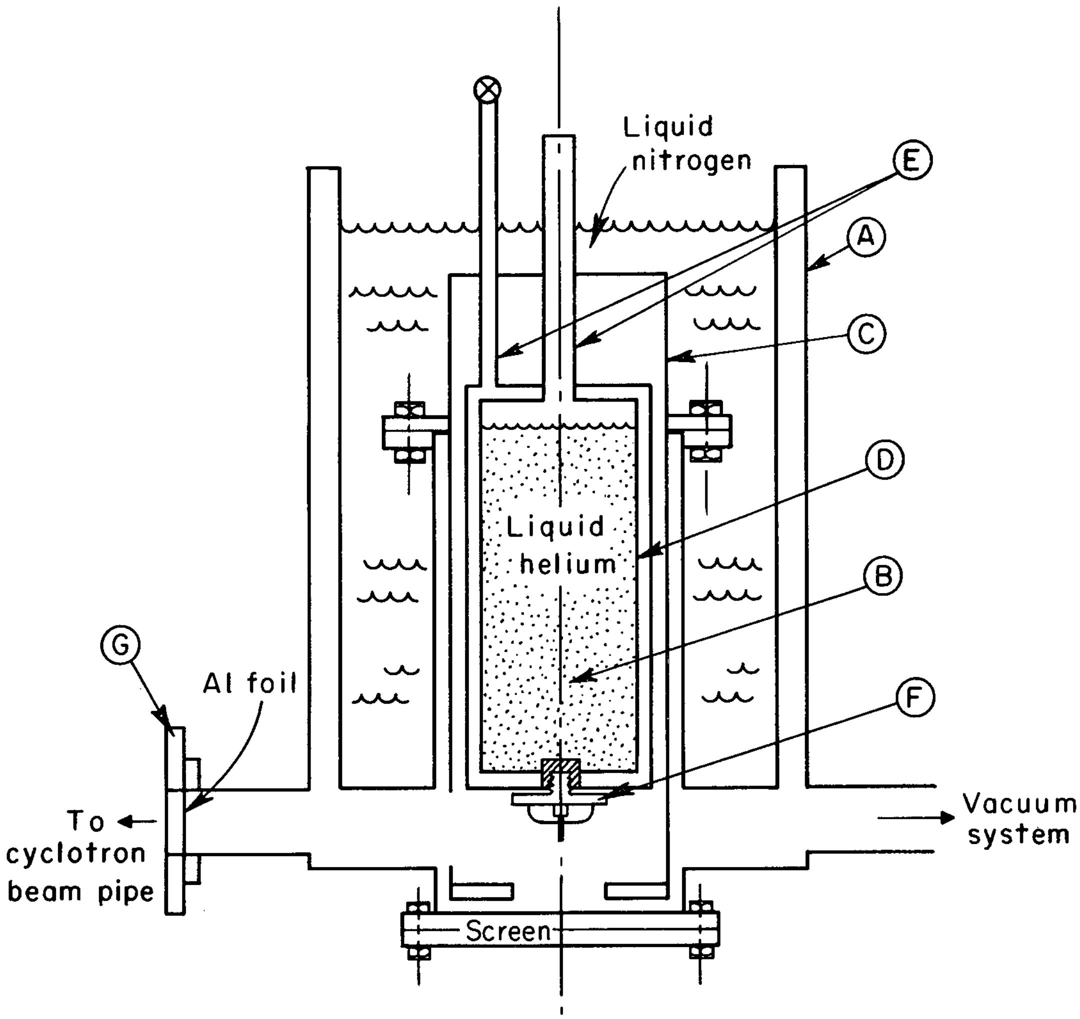
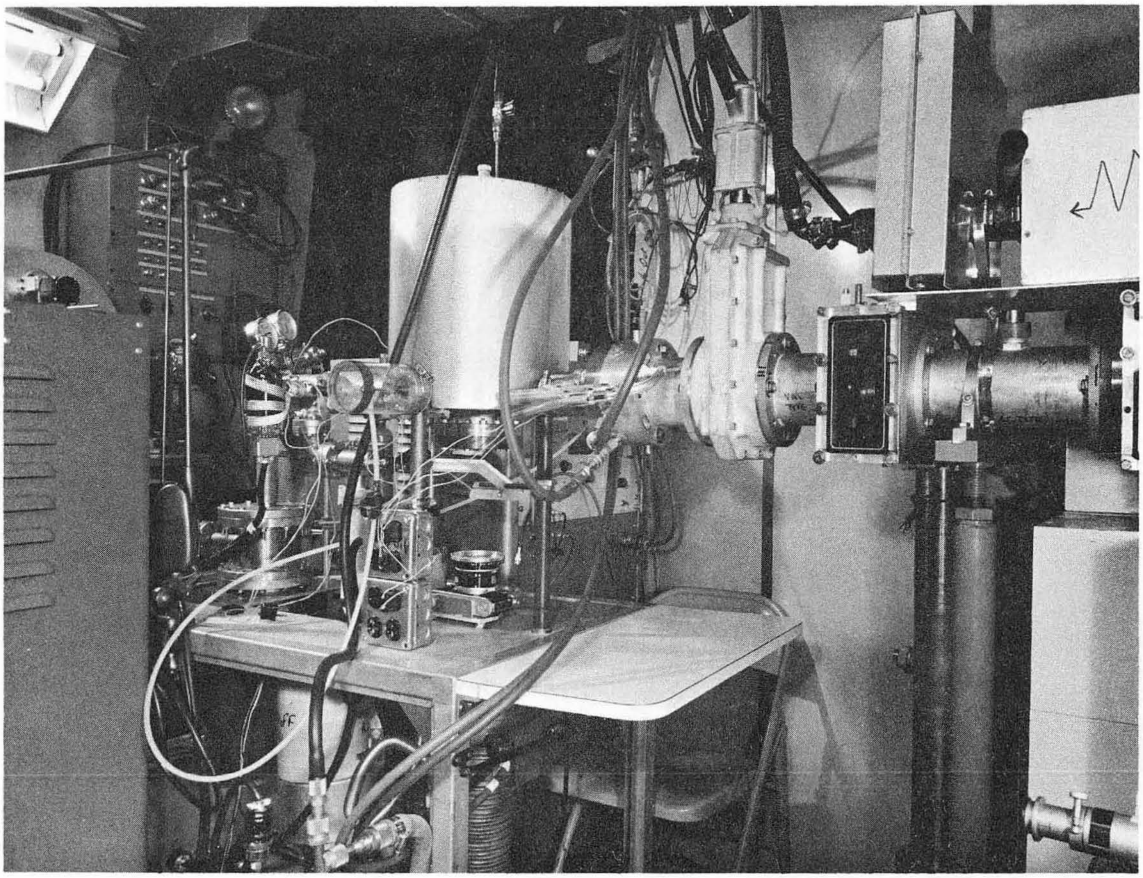

Liquid
nitrogen
E
A
C
D
Liquid
helium
B
F
G
Al foil
To
cyclotron
beam pipe
Vacuum
system
Screen
XBL671-393

Fig. 1



XBB 671-184

Fig. 2a

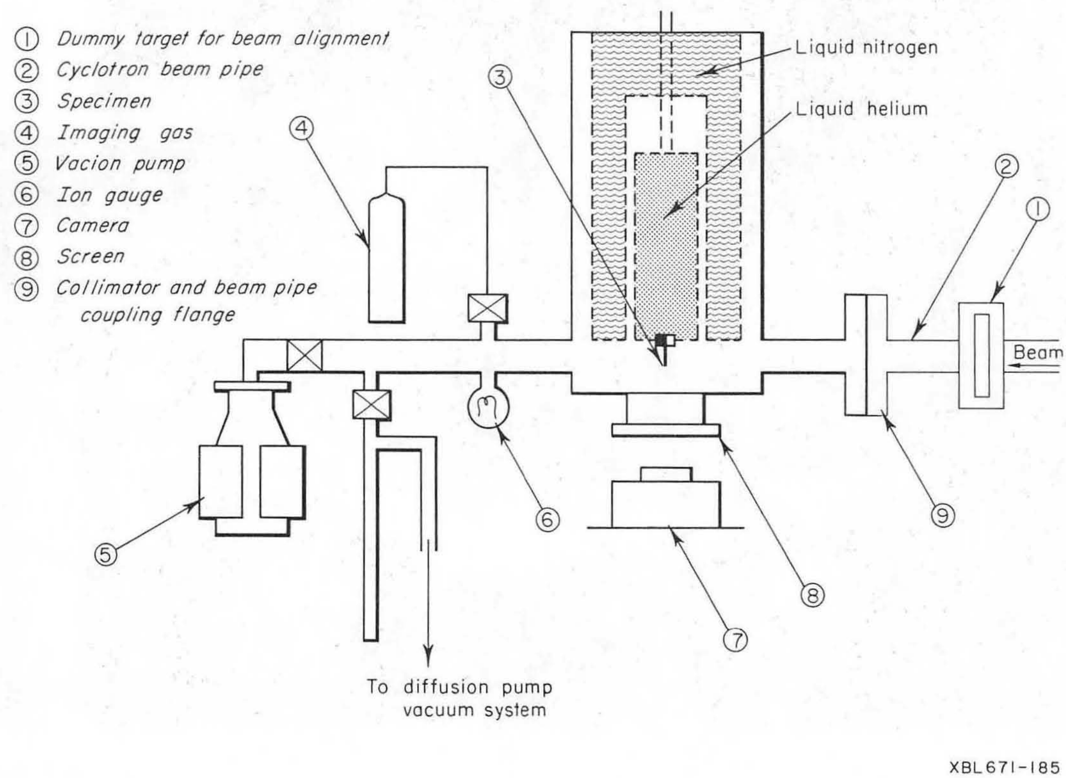
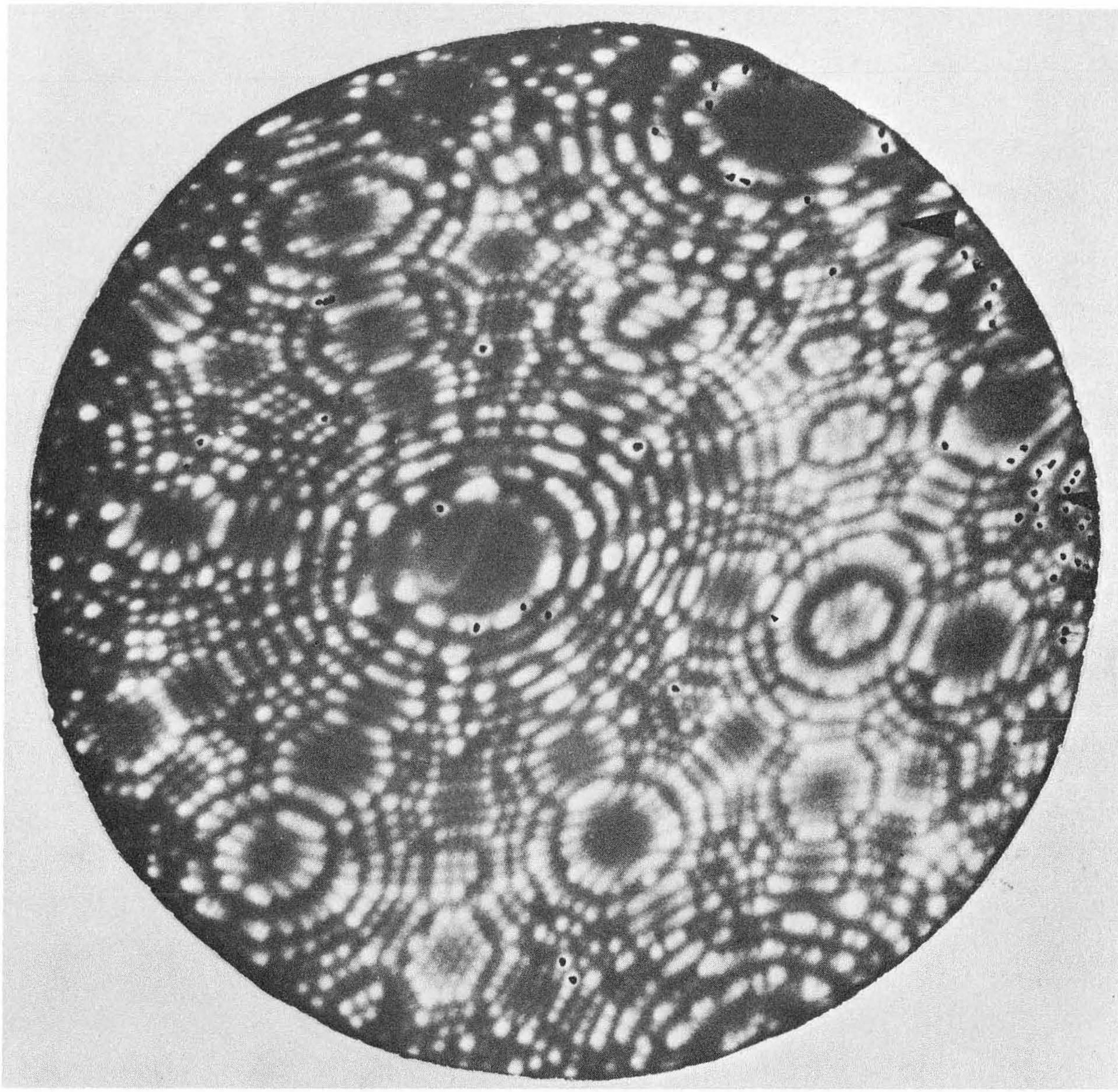
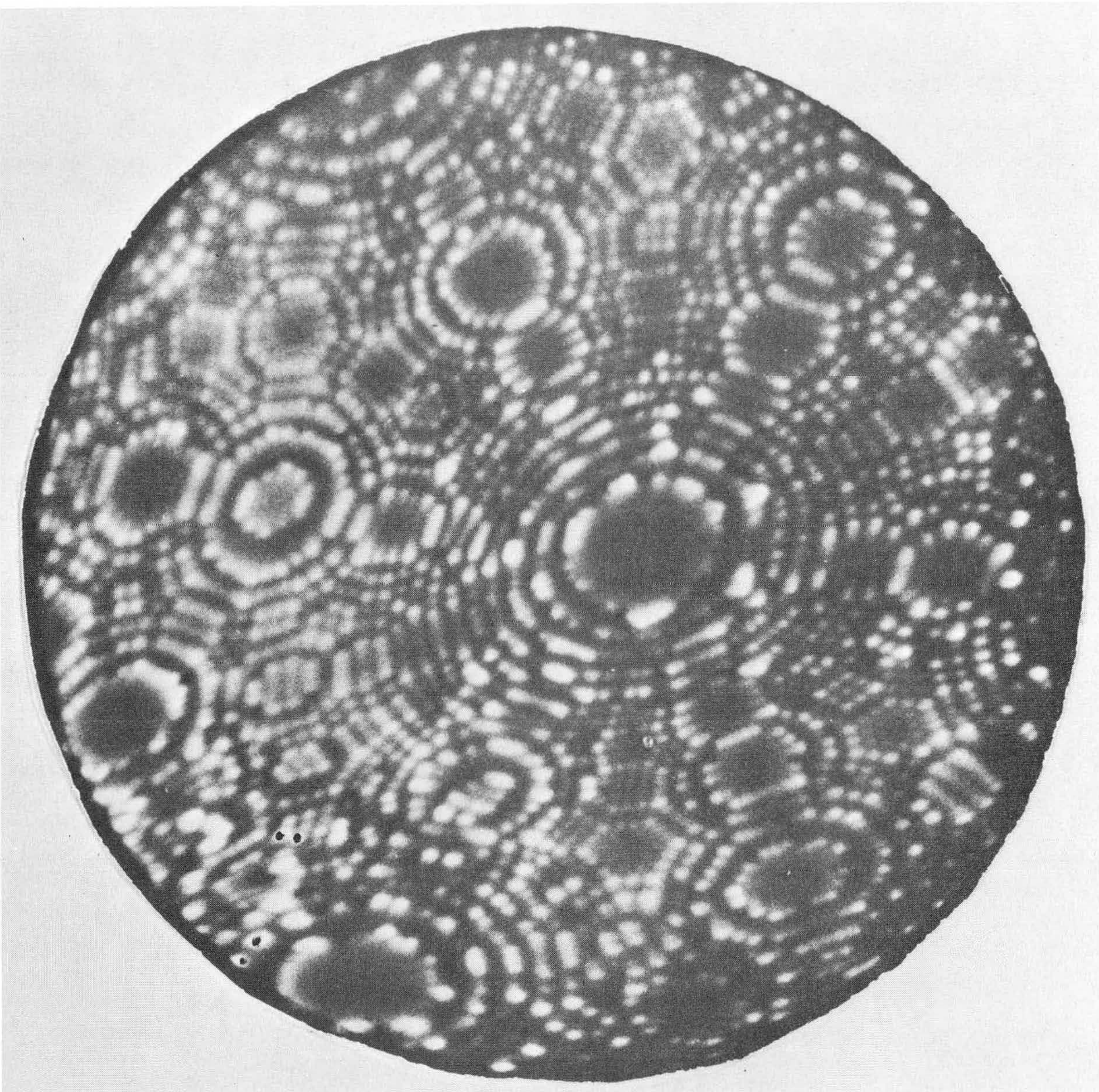

① Dummy target for beam alignment
② Cyclotron beam pipe
③ Specimen
④ Imaging gas
⑤ Vacion pump
⑥ Ion gauge
⑦ Camera
⑧ Screen
⑨ Collimator and beam pipe coupling flange
Liquid nitrogen
Liquid helium
Beam
To diffusion pump vacuum system
XBL671-185

Fig. 2b



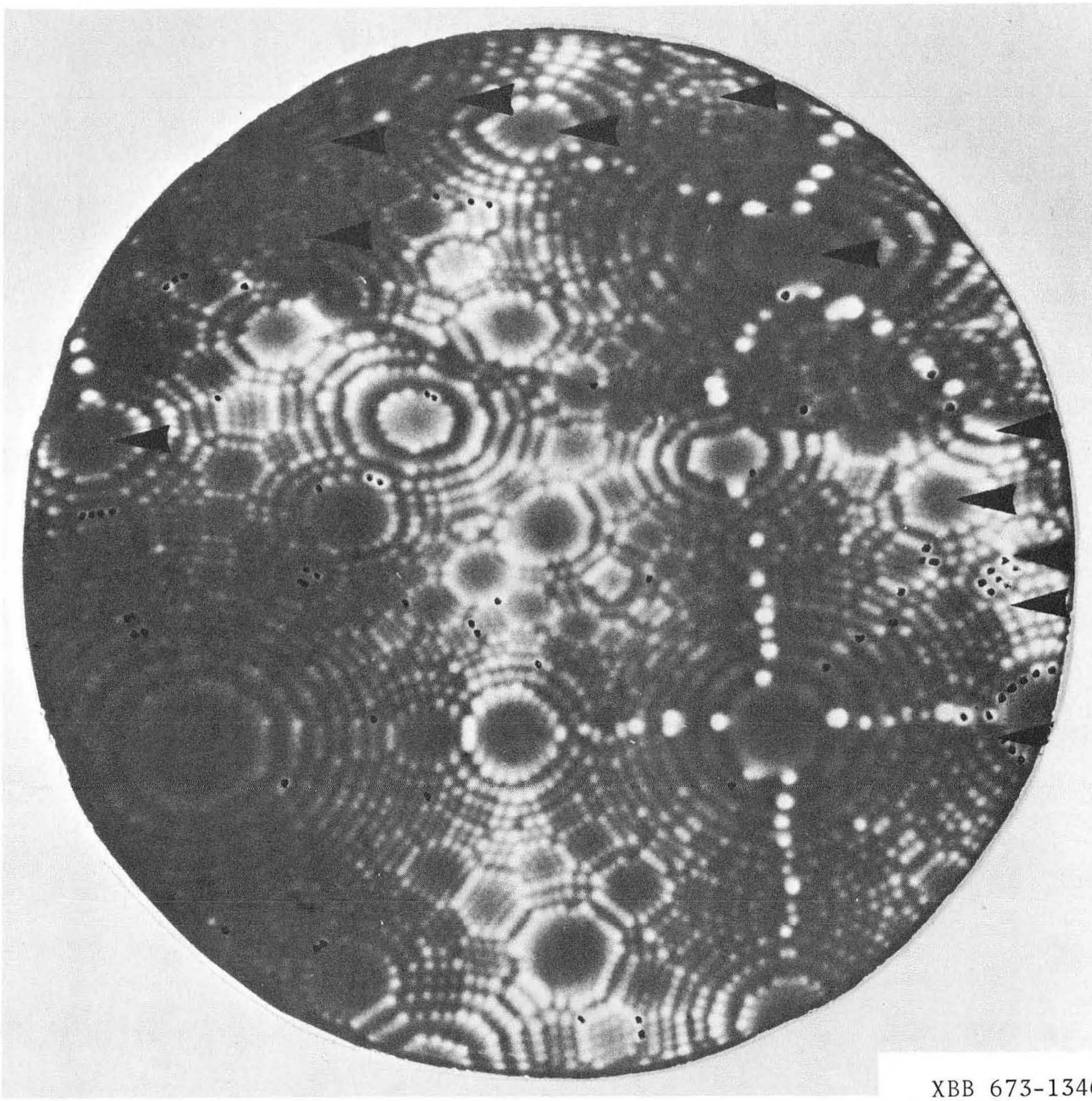
XBB 673-1344

Fig. 3a



XBB 673-1348

Fig. 3b



XBB 673-1346

Fig. 4

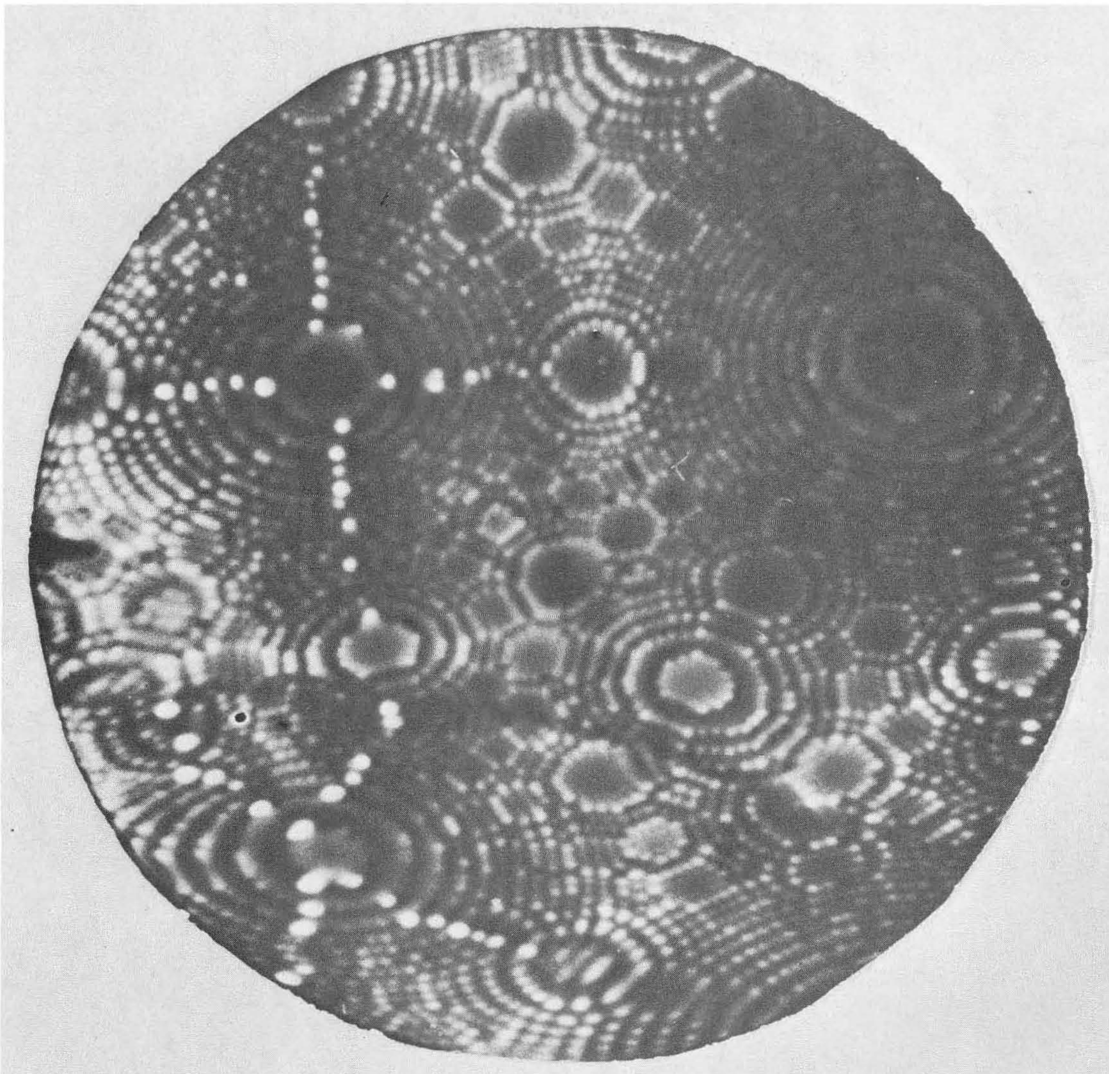


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Fig. 5