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ULTRA-HIGH VACUUM (UHV) ATOM-PROBE FIELD-ION MICROSCOPE

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ABSTRACT

A differentially-pumped low-energy (50 to 3000 eV) ion-beam system for the in-situ irradiation of specimens in an UHV atom-probe field-ion microscope (FIM) was designed and constructed. The ion-beam system consists of a Finkelstein-type ion-source, an Einzel lens and a magnetic mass-analyzer. The ion source is connected to the analyzer chamber by small apertures which results in differential pumping between the ion source and the analyzer chamber; during a typical in-situ irradiation of a specimen the pressure in the atom-probe is $\sim 10^{-7}$ Torr. The ion-current density is $\sim 0.5 \mu\text{A cm}^{-2}$ for 300 eV $(\text{He}^4)^+$ ions at the atom-probe FIM specimen; ion current-densities as high as (2 to 3) mA cm^{-2} have been achieved for an ion-beam energy ≥ 1 keV. Therefore the irradiation of a specimen with a single-mass, mono-energetic ion-beam is accomplished with the complete elimination of a long pump-down time after the irradiation.

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I. INTRODUCTION

The fundamental properties of light gases (H and He) in metals is of current interest because of important practical materials problems associated with the liquid-metal fast-breeder reactor,⁽¹⁾ the controlled thermonuclear-reactor,⁽²⁾ and the hydrogen-embrittlement phenomenon.⁽³⁾ We are presently investigating the low-temperature diffusive properties of both hydrogen and helium⁽⁴⁾ and the range-profiles of low-energy (<3 keV) ions of these gases in metals employing the time-of-flight atom-probe field-ion microscope (FIM).⁽⁵⁾ Our atom-probe FIM was specifically designed for the investigation of the interaction of impurity atoms with lattice defects such as vacancies, self-interstitial atoms, dislocations, grain boundaries and voids; a detailed description of our atom-probe FIM has been reported elsewhere.⁽⁶⁻⁹⁾ Unique features of our atom-probe FIM which make it ideally suited for the study of defects and defect interactions include: (1) a variable magnification internal image-intensification system; (2) a liquid-helium cooled goniometer-stage; (3) a differentially-pumped low-energy (≤ 3 keV) gas ion-beam system for the in-situ irradiation of specimens; (4) an ultra-high vacuum (3×10^{-10} Torr) FIM chamber; (5) a high vacuum ($\approx 10^{-6}$ Torr) specimen-exchange device;⁽¹⁰⁾ (6) a Chevron ion-detector; and (7) an eight-channel digital-timer with a ± 10 nsec resolution for measuring the TOFs.⁽¹¹⁾

This article describes a specially designed and constructed differentially-pumped low-energy (50 to 3000 eV) ion-beam system for an atom-probe FIM. This system allows the atom-probe FIM to be maintained at high vacuum (10^{-6} to 10^{-7} Torr) during the in-situ irradiation of FIM specimens. The ion-beam system produces a mass-analyzed high current-density ($> 0.5 \mu\text{A cm}^{-2}$) beam of gas ions (H_2 , He, Ne, Ar or Xe) and consequently eliminates the long pump-down time after an irradiation has been completed; ion current-densities of (2 to 3) mA cm^{-2} have been achieved for ion beams with energies ≥ 1 keV.

II. DESIGN AND PERFORMANCE OF THE ION-BEAM SYSTEM

A. Ion gun and mass analyzer

The design criteria for the ion gun and mass analyzer were as follows:

(1) the production of a high current-density ($0.5 \mu\text{A cm}^{-2}$ or greater) low-energy (50 to 3000 eV) ion-beam of gases such as H, H_2 , He, Ne, Ar or Xe); (2) a mass-analyzed ion-beam of the above-mentioned gases in order to eliminate both neutral species and trace quantities of impurity gases. The relatively high current-density ion-beam is needed in order to minimize the irradiation time; a short irradiation time prevents the contamination of the clean and atomically perfect FIM specimen, during the course of an irradiation, when the imaging electric field is removed from the specimen. This goal is achieved if the irradiation time is short compared to the time required to form a monolayer of adsorbed atoms or molecules at the background pressure in the atom-probe FIM chamber. The mass-analyzed beam is required to implant a particular gas species in the FIM specimen without any interference effects from the other species which are commonly found even in high-purity gases.

Figure 1 shows a schematic top-view of the entire ion-beam system. A Finkelstein⁽¹²⁾ type ion-source (see Fig. 2) is used to obtain a plasma of the desired ion. The Finkelstein ion-source is a hot-cathode electron-bombardment type source and it is expected that the ion-energy spread of this source is 1 to 10 eV (see Table 2-5 in Wilson and Brewer⁽¹²⁾). The low-energy ion-beam is extracted from the plasma chamber of the ion source and is analyzed employing a CENCO Model G electromagnet with 7.5 cm diameter cylindrical pole-pieces; the electromagnet is removed when the atom-probe FIM and ion-beam system are baked out. The analyzing magnet bends the ion beam, of the desired mass to charge ratio, through an angle of 15° so that it travels down a flight tube, through a second Einzel lens, towards the FIM specimen. The FIM specimen is attached to a

liquid-helium goniometer stage; (8,10) the goniometer is used to rotate the specimen so that its axis is parallel, within $\pm 5^\circ$, to the ion beam during the irradiation.

We now consider the different elements of the ion-beam system in somewhat more detail. Figure 2 exhibits a cross-sectional view of the Finkelstein-type ion-source; the entire ion-gun (ion source plus the first Einzel lens) is mounted on a $4\frac{1}{2}$ inch diameter double-sided Varian Con-flat flange for easy assembly and removal; this figure has cylindrical symmetry about the long horizontal-axis of the ion source. The major components of the ion gun were fabricated from 304 stainless steel and Corning machinable glass-ceramic No. 2598; the latter is an excellent electrical insulator and is also compatible with UHV requirements. A gas is admitted into the plasma chamber of the ion source, from a gas train to the desired pressure, with the aid of a Varian leak-valve. The gas molecules or atoms are ionized by thermionically-emitted electrons from a resistively-heated tungsten-filament which is maintained at a positive bias-potential. The cylindrically-shaped Ta anode, inside the plasma chamber, as well as the plasma chamber itself are also held at a positive potential; the filament and the plasma chamber are maintained at the same positive potential while the Ta anode is held at a potential of ≈ 100 to 125 Vdc above these two elements. The solenoid is used to produce a magnetic field that aids in maintaining a high-density plasma in the plasma chamber. An Einzel lens is employed to extract and focus an ion-beam from the plasma chamber; this is the first of two Einzel lenses employed. The extraction lens and final lens of this first Einzel lens are maintained at ground potential, while the focusing lens is maintained at a positive potential; this first Einzel lens is used to produce a maximum ion current-density beam. The ion source and the first Einzel lens both sit inside the outer cylinder which contains a 10 mm diameter exit aperture, through which the ion beam travels on its way towards the analyzing magnet. Figure 3 exhibits the electrical circuit employed to supply the required potentials for the ion gun.

For evaluation purposes only a simple stainless-steel plate with a specially-constructed aperture system in front of it was placed at about 2 cm from the 10 mm diameter exit orifice of the ion source. The total ion-current was measured on this stainless-steel plate, employing different gases at different pressures, as a function of the ion-beam energy; the maximum area on which the ion current was measured was $0,8 \text{ cm}^2$; therefore the current densities we report are lower limits. The results of this experiment are shown in Fig. 4 for the gases H_2 , He and Ne. We have also obtained a current density of $(2 \text{ to } 3) \text{ mA cm}^{-2}$ at $\sim 5 \times 10^{-3}$ Torr He for ion-beams with energies $\geq 1 \text{ keV}$; however, in order to maintain high-vacuum conditions in the atom-probe FIM chamber and a long filament life-time, the gas pressure in the ion source was usually kept at a value of $< 10^{-3}$ Torr. The following values represent typical ones during the operation of the ion source: filament current ~ 8 to 9 Adc for a 0.34 mm diameter tungsten wire; tungsten emission current ~ 1 to 5 mA ; Ta anode voltage ~ 100 to 125 Vdc above the filament voltage; magnetic solenoid current $\sim 3 \text{ Adc}$; and focusing voltage for the first Einzel lens ~ 60 to 80% of the filament bias-voltage. The measured ion current-densities at an unbiased atom-probe FIM specimen, after mass analysis, are $\sim 5\%$ less than the values shown in Fig. 3. During an in-situ irradiation the FIM specimen is biased at a positive 15 Vdc , with respect to the ground-potential surroundings, in order to suppress secondary-electron emission. As a result the measured ion current-densities at the biased FIM specimen are $\sim 10\%$ less than the values shown in Fig. 4. The final potential-energy of the ion beam is simply the difference between the bias potential on the tungsten filament and the secondary-electron suppression potential on the FIM tip. The present system is capable of accelerating ions to a final energy of 3000 eV and a simple exchange of power supplies will enable us to go to 5000 eV , as all components are rated for this higher voltage.

The magnetically-analyzed ion-beam passes through a second 7.5 mm diameter aperture (see Fig. 1) on its way to the FIM specimen. Just after this aperture there is a beam-stop and current monitor, which is moved perpendicular to the path

of the ion beam employing a Varian linear-motion feedthrough. The beam-stop and current-monitor is used to find the desired ion-species for an irradiation; once the desired ion-species is found the current density on the beam stop is maximized by adjusting the potentials on the ion gun. Next the beam stop is removed and the second Einzel lens is adjusted to give a current density which is $\approx 10\%$ less than the maximum value: this last step insures a uniform current-density on the FIM specimen.

Figures 5(a) and 5(b) show the profiles of 500 eV He^+ and Ne^+ ion beams, respectively, measured at the FIM specimen. The profiles were obtained by linearly ramping the analyzing magnet's current, so that the ion beam was scanned across the 6.4 mm diameter beam-defining aperture (see Fig. 1). For the collector geometry employed, the mass resolution ($m/\delta m$) is ≈ 5 to 6 at the beam-defining aperture; the value of δm corresponds to the full peak-width at half-maximum intensity (FWHM). In the Appendix we show that an ($m/\delta m$) of at least 100 is expected at the $< 250 \mu\text{m}$ diameter FIM specimen; this mass resolution is more than sufficient for this ion-beam system.

B. Differential pumping-system

A differential pumping-system was employed in order to maintain the pressure in the atom-probe FIM chamber at as small a value as possible during the course of an irradiation. This was accomplished by: (1) pumping on the ion source (see Fig. 1) with a 2-inch diameter CVC diffusion pump, which was trapped by a Granville-Phillips liquid-nitrogen cold-trap, for an effective pumping speed of $\approx 30 \text{ liter sec}^{-1}$; (2) pumping on the atom-probe FIM chamber with a 2-inch diameter CVC diffusion pump, which was trapped by a Granville-Phillips liquid-nitrogen cold-trap, for an effective pumping speed of $\approx 10 \text{ liter sec}^{-1}$ and also pumping with a titanium sublimation-pump (TSP); and (3) the use of two 7.5 mm diameter apertures in between the ion source and the atom-probe FIM chamber respectively (see Fig. 1 to locate the position of

these apertures). In addition the 2 mm diameter exit orifice of the plasma chamber (see Fig. 2) also provided a differential pressure between the plasma chamber and outer cylinder which contained the ion source.

The differential-pumping efficiency (η_p), i.e., the ratio between the pressure in the ion source to the pressure in the atom-probe FIM chamber, was measured for four different gases. The values of η_p are as follows:

Gas	H ₂	He	Ne	Ar
η_p	~ 1000	100	150	150 .

The measured values of η_p for He, Ne and Ar are in agreement with a straight-forward kinetic-gas calculation of η_p for each of these gases, while the calculated value of η_p of ~ 100 for H₂, without the TSP, differs from the measured value by a factor of ~ 10 . We attribute this discrepancy to the use of the TSP, in the atom-probe FIM chamber, which has an extremely high pumping-speed for H₂.

To achieve a dose of 5×10^{15} He⁺ ion cm⁻² typically requires ~ 20 to 30 min. For a He⁺ ion irradiation in this time period the pressure in the atom-probe FIM chamber rises to $\sim 5 \times 10^{-7}$ Torr; this pressure is due mainly to He gas because we employ a mass-analyzed ion beam. Thus the residual pressure in the atom-probe FIM chamber remains almost constant at $\sim 3 \times 10^{-10}$ Torr during the irradiation; therefore probably less than one monolayer of adsorbed gases form on the specimen during the course of a typical irradiation. The pump-down time to 3×10^{-10} Torr, in the atom-probe FIM chamber, after an in-situ He⁺ ion irradiation has been reduced to 0.5 hours; in earlier work without appreciable differential-pumping the pump-down time was 3.0 hours. (14)

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APPENDIX

In this appendix we calculate the mass resolution ($m/\delta m$) at the beam-defining aperture and at the FIM tip. Under the assumptions of no energy spread and no space-charge expansion in the ion beam the spatial separation (δx) of an ion beam with mass m from an ion beam with mass $m + \delta m$ is given by: ⁽¹⁵⁾

$$\delta x = - \frac{1}{2} \frac{\delta m}{m} [R(1 - \cos \theta) + L_d \sin \theta], \quad (A1)$$

where R is the radius of the circle which the ion beam follows in the analyzing magnet, θ is the angle through which the ion beam is bent in the analyzing magnet and L_d the distance through which the ion beam drifts before reaching the beam-defining aperture (or the FIM tip); Fig. 6 is a schematic drawing which illustrates all the important quantities. In our design the critical values are: (1) $\theta = 15^\circ$; (2) $R = 25$ cm; and (3) $L_d = 27$ cm. These values yield an ($m/\delta m$) of 6 for a beam-defining aperture with a 0.635 cm radius (δx); this theoretical value agrees well with our experimental value. The FIM specimen has a diameter of $<250 \mu\text{m}$ (typically $125 \mu\text{m}$), therefore an ($m/\delta m$) of >100 is expected at the FIM tip on the basis of Eq. (A1).

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FIGURE CAPTIONS

Figure 1. A schematic diagram showing the differentially pumped ion-gun and its relationship to the mass-analyzer chamber and the atom-probe FIM specimen.

Figure 2. A schematic diagram of the low-energy ion-gun showing its physical configuration.

Figure 3. A schematic diagram of the low-energy ion-gun showing the electrical circuit.

Figure 4. The ion-current (or current density) versus ion-energy plot for different low-energy ion beams; the curves are for three different gases (H_2 , He and Ne) at the indicated gauge pressures; all pressures were measured employing a Veeco RG-75 ionization gauge.

Figure 5. The ion beam profiles of: (a) 500 eV He^+ ions; and (b) 500 eV Ne^+ ions. The linearity of the analyzing magnet current is shown in 5(b).

Figure 6. A schematic diagram illustrating the mass separation of ions of mass (m) and mass (m + δm).

Fig. 1

Fig. 2

P.S. $\equiv$ POWER SUPPLY

Fig. 3

Fig. 4

Fig. 5

Fig. 6