COO-3158-64

CONF-780776--1

A SPECIMEN-EXCHANGE DEVICE FOR AN ULTRA-HIGH VACUUM ATOM-PROBE FIELD-ION MICROSCOPE

by

Alfred Wagner, Thomas M. Hall and David N. Seidman

Cornell University
Ithaca, New York 14853

July 1978

Report #3062

Issued by
The Materials Science Center

Prepared for

THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT
NO. EY-76-S-02-3158.*000

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A Specimen-Exchange Device for an Ultra-High Vacuum Atom-Probe Field-Ion Microscope[#]

Alfred Wagner*, Thomas M. Hall** and David N. Seidman***

Cornell University, Department of Materials Science and Engineering and the Materials Science Center, Ithaca, New York 14853, USA

ABSTRACT

A specimen-exchange device is described for an ultra-high vacuum field-ion microscope (FIM). This device completely eliminates the long pump-down period that is required if the FIM chamber is brought back to atmospheric pressure. The pressure in an air-lock is reduced to 10^{-6} Torr before the exchange takes place and the pressure in the FIM chamber remains below 10^{-7} Torr during the exchange and it drops to less than $3x10^{-9}$ Torr within 15 minutes after the exchange.

Paper presented at the Fifth Israel Vacuum Congress, Hebrew University of Jerusalem, Jerusalem, Israel.

* Now at Bell Laboratories, 600 Mountain Avenue, Murray Hill, New Jersey 07974, USA

** Now at Bell Laboratories, 555 Union Boulevard, Allentown, Pennsylvania 18103, USA

*** Lady Davis Visiting Professor (1978), Hebrew University of Jerusalem, School of Applied Science and Technology, Materials Science Division, Jerusalem, Israel.

1. INTRODUCTION

The time-of-flight (TOF) atom-probe field-ion microscope (FIM) makes it possible to measure the mass-to-charge (m/n) ratio of an individual atom which has been pulse field-evaporated from the surface of an FIM specimen. This instrument was first described by Müller, Panitz and McLane[1] and subsequently developed by Müller and co-workers[2] as an extension of the FIM. Thus it is now possible to combine the microstructural information, obtainable by the pulse field-evaporation of successive atomic layers, with a simultaneous chemical analysis, on an atom-by-atom basis, of the bulk of an FIM specimen. The major applications of the atom-probe, to date, have been to metallurgical problems involving precipitation by Brenner and co-workers[3-6] and Turner and co-workers[7-9].

We have recently developed an ultra-high vacuum atom-probe FIM for the specific purpose of studying the interaction of impurity atoms (or alloying elements) with lattice defects such as vacancies, self-interstitial atoms, dislocations, grain boundaries and voids; this work has been reported in detail elsewhere[10-13]. Our atom-probe has a number of unique features which makes it ideally suited for the study of defects and defect interactions. These features are: (1) a variable magnification internal image-intensification system; (2) a liquid-helium cooled goniometer stage; (3) a differentially-pumped low-energy ($\lesssim$3 keV) gas-ion gun for the <u>in-situ</u> irradiation of specimens; (4) an ultra-high vacuum (3×10^{-10} Torr) FIM chamber; (5) a high vacuum ($\approx10^{-6}$ Torr) specimen-exchange device; (6) a Chevron ion-detector; and (7) an eight-channel digital timer with a ±10 nsec resolution for measuring the TOFs.[14]

One of the major limitations of the atom probe, in the past, has been the problem of changing specimens. Prior to the construction of the specimen-exchange device it was necessary to bring the entire atom-probe FIM up to atmospheric pressure to insert a new specimen in the goniometer stage. This cumbersome process meant that typically only one specimen per day could be examined. To correct this situation we have designed and constructed a simple specimen-exchange device; this device is described in the present paper.

2. THE SPECIMEN-EXCHANGE DEVICE

Figure 1 shows an FIM specimen mounted in its copper specimen holder; the latter, in turn, is mounted in a goniometer stage which is attached to a liquid-helium cryostat via a gold braid. A detailed schematic diagram of the goniometer stage can be seen in Figure 2 of Hall et al.[12] The purpose of the specimen-exchange device is to remove (or insert) the copper specimen holder from (into) the goniometer stage while maintaining the ultra-high vacuum FIM chamber at as low a pressure as is possible and while keeping the liquid-helium cooled goniometer at cryogenic temperatures.

The exchange of a specimen is accomplished with the aid of the specimen-exchange device shown in Figure 2. The copper specimen holder is attached to the end of a 1 meter long, 9.5 mm diameter, stainless steel specimen-exchange rod with the aid of a bayonet-type clip. The copper specimen holder is surrounded by a retractable protection shield which also serves as an alignment guide when screwing the specimen holder into the goniometer stage. The specimen-exchange rod passes through a Vacuum Research Corporation S102 Wilson-type sliding motion feed-through that is sealed with two viton gaskets which are lubricated with Apeizon L vacuum grease. The specimen-exchange rod passes through a Varian 1.5 inch diameter ultra-high vacuum straight-through valve which seals the FIM chamber until the air lock has been evacuated. To insert a new FIM specimen into the FIM chamber the specimen exchange rod is first placed inside the air lock with the straight-through valve closed; this is accomplished with the aid of the Wilson-type feed-through which is mounted on a 1.5 inch diameter stainless steel tube by means of a standard quick coupling. Next the air lock is rough pumped using two Varian Vacsorb pumps in series; these are the same sorbtion pumps which one also used to evacuate the FIM chamber. When the pressure in the airlock has fallen into the 10^{-3} to $^{-4}$ Torr range it is then evacuated to 10^{-6} Torr employing a specially-constructed titanium sublimation pump (TSP) and a Varian 20 liter sec^{-1} triode-ion pump. When $\approx 10^{-6}$ Torr has been achieved the straight-through valve is opened and the specimen-exchange rod is pushed into the FIM chamber until the retractable protection and guide shield makes contact with the outer radiation shield. A small additional linear displacement of the specimen-exchange rod brings it into contact with the threaded copper plate that is attached to the goniometer stage through the copper sapphire block (see Figures 1 & 2). The copper specimen holder is then screwed into the threaded copper plate; an additional counterturn then separates the specimen exchange rod from the copper specimen holder. The specimen-exchange rod is then pulled out of the ultra-high vacuum FIM chamber and the straight-through valve is closed. The entire operation, described above, is observed through a glass-window (not shown) which is mounted on a 1.5 inch diameter port that is at 45° to the line-of-motion of the specimen-exchange rod. During the exchange operation the pressure in the FIM chamber remains below 10^{-7} Torr and the pressure in it drops to $<3x10^{-9}$ Torr within 15 minutes after exchange. With this specimen-exchange device the time required for specimen replacement has been reduced to less than 1 hour. Thus changing FIM specimens is now almost as simple as changing specimens in a conventional transmission electron-microscope.

ACKNOWLEDGEMENTS

This research was supported by the U.S. Department of Energy. Additional support was received from the National Science Foundation through the use of the technical facilities of the Materials Science Center at Cornell University. We wish to thank Mr. Anthony Babbaro for performing the machine-shop work and Mr. Robert Whitmarsh for technical assistance. D.N. Seidman wishes to thank the Lady Davis Foundation for a Visiting Professorship which made his attendance at the Fifth

Israel Vacuum Congress possible, and Dr. Yohanan Margoninski of the Physics Department of the Hebrew University for suggesting that we present and publish our work on the specimen-exchange device.

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FIGURE CAPTIONS

Figure 1: A cross-sectional view of the liquid-helium cryostat showing the relationship of the FIM specimen, in the goniometer stage, to the cryostat. The FIM specimen is cooled by conduction through a gold braid. The radiation shields are cooled by conduction employing the enthalpy of the helium exhaust gas.

Figure 2: The high-vacuum specimen-exchange device which allows a specimen to be changed without breaking the vacuum in the ultra-high vacuum (UHV) FIM.

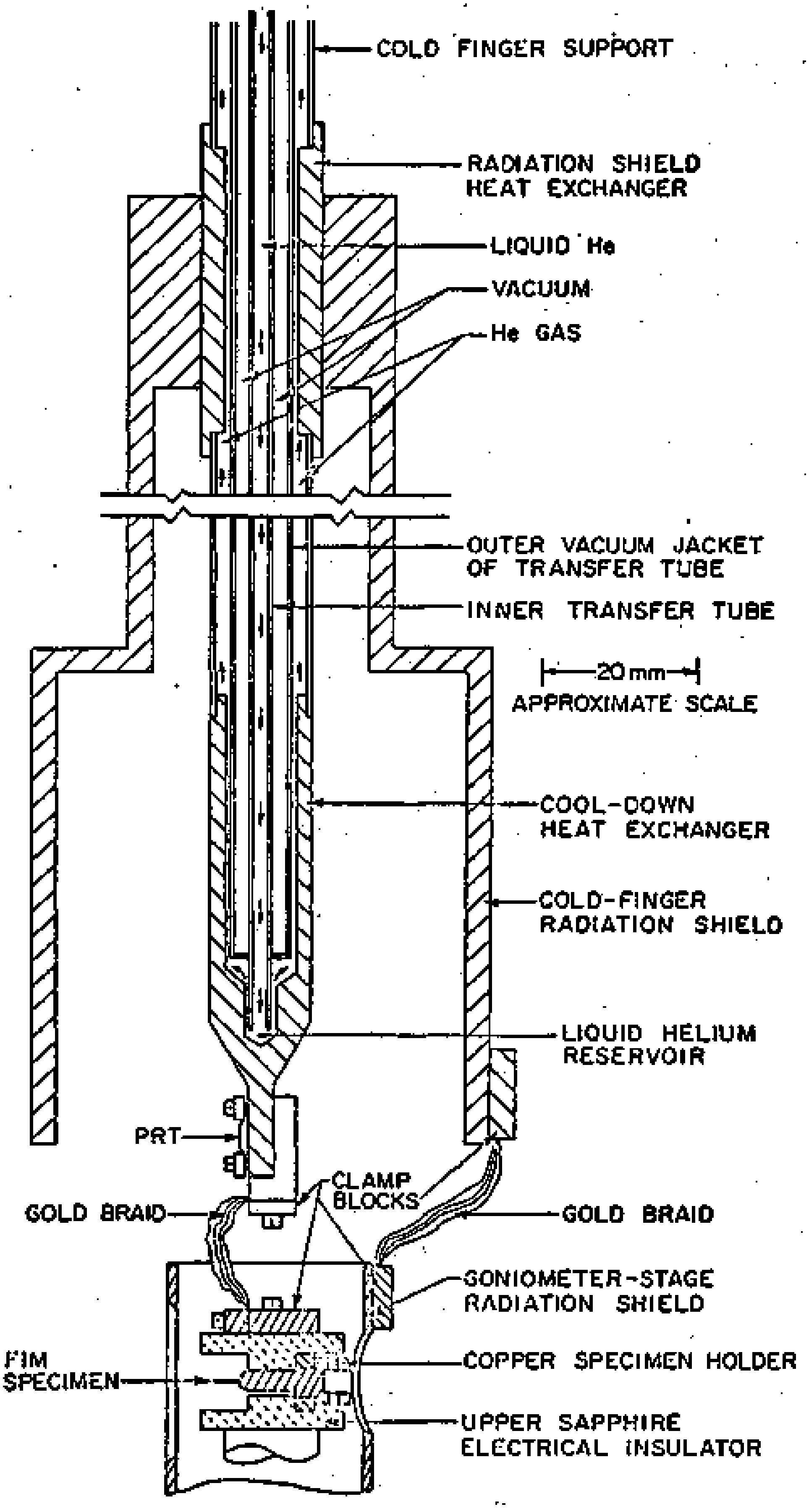
COLD FINGER SUPPORT
RADIATION SHIELD HEAT EXCHANGER
LIQUID He
VACUUM
He GAS
OUTER VACUUM JACKET OF TRANSFER TUBE
INNER TRANSFER TUBE
20 mm
APPROXIMATE SCALE
COOL-DOWN HEAT EXCHANGER
COLD-FINGER RADIATION SHIELD
LIQUID HELIUM RESERVOIR
PRT
CLAMP BLOCKS
GOLD BRAID
GOLD BRAID
GONIOMETER-STAGE RADIATION SHIELD
FIM SPECIMEN
COPPER SPECIMEN HOLDER
UPPER SAPPHIRE ELECTRICAL INSULATOR

Figure 1

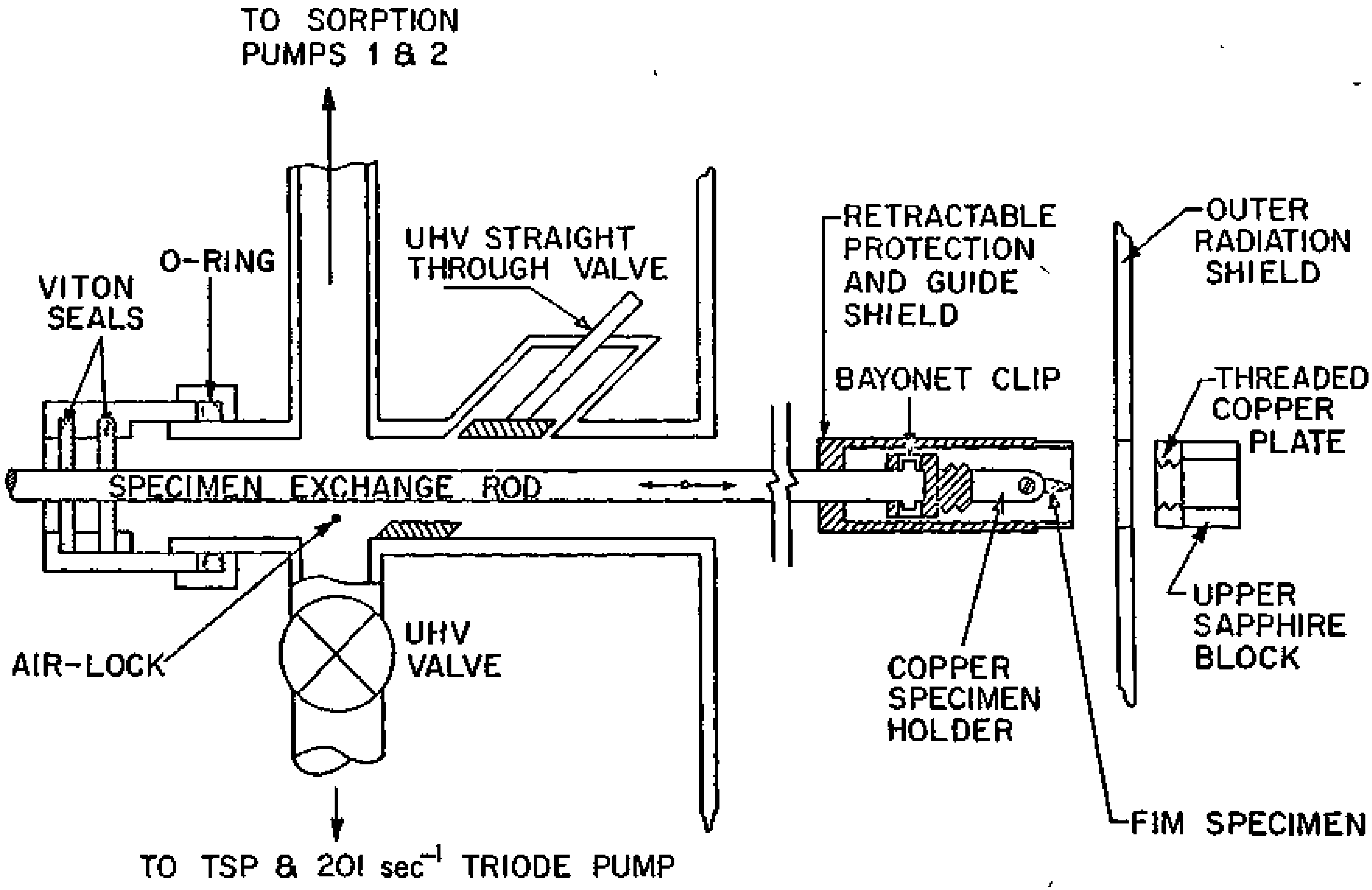
TO SORPTION PUMPS 1 & 2
VITON SEALS
O-RING
UHV STRAIGHT THROUGH VALVE
RETRACTABLE PROTECTION AND GUIDE SHIELD
OUTER RADIATION SHIELD
BAYONET CLIP
THREADED COPPER PLATE
SPECIMEN EXCHANGE ROD
UPPER SAPPHIRE BLOCK
AIR-LOCK
UHV VALVE
COPPER SPECIMEN HOLDER
FIM SPECIMEN
TO TSP & 20l sec^{-1} TRIODE PUMP

Figure 2