OPERATING PROCEDURE FOR COMBINED AUGER -
AND ELLIPSOMETER UHV-SYSTEM

Felix Schwager

November 1981

TWO-WEEK LOAN COPY

This is a Library Circulating Copy
which may be borrowed for two weeks.
For a personal retention copy, call
Tech. Info. Division, Ext. 6782

Prepared for the U.S. Department of Energy under Contract W-7405-ENG-48
DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.
OPERATING PROCEDURE FOR COMBINED AUGER - AND ELLIPSOMETER UHV-SYSTEM

FELIX SCHWAGER

Materials and Molecular Research Division
Lawrence Berkeley Laboratory
University of California
Berkeley, CA 94720

ABSTRACT

Operating instructions are given for conducting ion etching and Auger analysis of surface layers with laboratory-built equipment which provides for the simultaneous use of ellipsometry.

This work was supported by the Assistant Secretary for Conservation and Renewable Energy, Office of Energy Systems Research, Division of Energy Storage of the U.S. Department of Energy under Contract No. W-7405-ENG-48.
I. Electrical Connections (Figure 1)

a. Faraday cup 18 − microammeter

b. Ion gun 19 − small Burndy (military) connector to Ion gun power supply

c. Connect 1st & 3rd grid to bolt near first grid (on the back side) 20-21-22

d. Ground retarding grid 23 (2nd grid with Teflon). Connect through hole of Teflon, tighten with Allen wrench

e. 280 /s (big) "vacion" pump 24 − metal shielded cable from pump power supply

f. Ground (screws on top of pump magnets) 25 − braided cable to power supply

g. Electron gun 26 − large Burndy connector to 3-gun power supply

h. Small "vacion" pump 27 (ceramic plug on front) − metal shielded cable from small pump power supply

i. Ground, Phillips screws 28 on top of pump magnet − braided ground cable to power supply

j. Gauge on manifold 29 − "m torr" meter, separate on table (Varian 801)

k. Pins of vacuum ion gauge (see drawing) 30 − 1 long black cable with alligator (center pin) 31

2 short black cables to either two of the three top pins 32, 33, 35

1 red cable to pin counterclockwise from broken pin 34

l. Phos. screen (back of chamber) − "Differential Preamplifier" box ("gain" should be 10)
II. SAMPLE TRANSFER (Figure 2)

a. Put specimen on holder in low position with 1, 2, 3, 4; bellows 5 should be straight.

b. Turn off ion gauge and other filaments (ULTEK digital gauge control OFF).

c. Close the gate valves of the 2 "vacion" pumps. If the chamber is only to be opened for sample transfer, leave the pump switch on.

d. Purge with nitrogen:
   1. close valves 6 and 9
   2. open valve 7 to N₂-cylinders
   3. purge manifold with N₂ by means of valves 7 and 8; pressure in manifold <5 psi (keep cylinder connected) (turn counter clockwise)
   4. open valve 11 completely to chamber, first with wrench then with handle, let N₂ in.

e. Open chamber on top by loosening the screws slowly, alternating from one screw to the other around flange.

f. Take sample holder out; take old copper gasket off (with special screw driver in side groove).

g. Cover opening with aluminum foil, leave N₂ - flow on.

h. After sample transfer to holder, put new holder back in position using new copper gasket. Tighten screws alternating (~1/4 turns).

i. Stop N₂ - flow.

j. Take heaters off sorption pumps 13 and attach styrofoam dewars. Cool one sorption pump down with liquid N₂ until boiling of N₂ calms down.
k. Open valve between sorption pump and manifold (15 or 16), first with wrench until click is heard, then manually.

1. Check pressure on "m-torr" gauge at manifold. When it reads <10 m-torr, check pressure in chamber by turning the "ULTEK digital gauge control" on for a short time.

m. If pressure reaches \(~5\times10^{-4}\) torr, close valve 11 to manifold and open gate valve to small "vacion" pump (10). (If pump is turned off, start it in "Start" mode before opening valve). During opening of the valve, the pump power supply should be in "Start" mode and the control panel selection switch in 200 mA position. The current should not exceed \(~100\) mA.

n. If pressure in chamber reaches about \(10^{-6}\) torr, open the gate valve to the big "vacion" pump slowly.

o. Switch both pumps into "Protect" mode.

p. Close valve to sorption pump (15 or 16) and tighten with wrench, replace the dewar by the heater and bake the sorption pump out (lift rubber sleeve on neck 17 of pump above hole for degassing).

III. AUGER MEASUREMENTS (Figures 3 and 4)

A. Preparations

1. Bring sample with specimen holder in position for ellipsometer measurement with 1, 2, 3, 4, 5 and screws on base plate (Fig. 2).

2. Take four zone reading with ellipsometer.

3. Faraday cells of ellipsometer OFF.

4. Check connection between racks and UHV-chamber.

   Check connection between REF INPUT, 48 panel G and REF SIGNAL on "Oscillator Modulator," 78 panel F.
Check connection between output of the high voltage ramp (rear of ORTEC on the right) and the Digital Voltmeter.

5. Check pressure.
   a. at "vacion" pump central unit A
   b. if pressure $<10^{-4}$ Torr with ion gauge
      ("ULTEK" digital gauge control E)
      i. Mode: Automode
      ii. Function: 0.01
      iii. Push red button+Red Light
      iv. Switch slowly to DEGAS; filament in
          chamber shines bright (burns off dirt on filament)
      v. After ~5 min. switch slowly back to 0.01
         (push red button in case red light goes off)
      vi. Wait until panel readings are steady

B. Electron Beam

1. Auger electron beam power supply C on (36)
   i. Current limit 37 set to 10
   ii. Voltage 80V (~full scale) on knob 38, panel C

2. Switch Auger control panels F on, 52, 39, 40
   i. Modulation Amplitude 42 on Oscillator F,
      between position 4 and 6 (see Fig. 5)
      - low energy range, -10 to -60V, pos. 1.0
      - medium energy range, -60 to -150V, pos. 3.0
      - higher energy range, -150 to -540V, pos. 4.0-7.0
   ii. Frequency (43) ~880
iii. "Ortec" power supply (left on F; 44, 45) for beam current normal ≈ 1.5 keV  
- low energy range - high voltage ≈ 2.5 keV  
- high energy range - low voltage ≈ 1.5 keV  
iv. "Ortec" power supply (right on F):  
"scan voltage" in pos. 0 (46), 300 (47)  
on the back: "negative", "external"  
v. Set "Auger Ramp Voltage" (54) and scan rate (54');  
ramp switch (55) OFF.

3. Increase beam current on "Auger electron beam power supply" C with  
41 to desired setting, usually 2 to 3 mA on panel C₂ on C.

C. Optimizing Electron Beam  
1. Lock-in amplifier G, power ON  
i. "Offset" (55): ON  
ii. "Post Filter" (56): ON, 1s  
iii. "Time-Const" (57): 0.3s  
iv. "Mode" (58): 2 f (compare mode on oscillator F: f)  
v. "Sensitivity" (62): 10 or 2.5 (do not overload)  

2. Oscilloscope H  
i. Press CH1 (Channel 1)  
ii. Adjust intensity (40), Focus (50)  
iii. Adjust signal size with 51
(If no such signal at any sensitivity on knob 51, check beam voltage (44) panel F, beam current (41, C2 panel C) or see D, 2.

iv. Smooth out high frequency with phase shift (59), vernier (60) and amplitude (61) on "Neutralizer" (F).

6. Lock-in amplifier G.

i. Adjust with phase knobs 63, 67, so that needle on panel goes to 0.

ii. Add 90° to zero-setting of i with 62

READY FOR AUGER-MEASUREMENT!!!
2. **Elastic Beam**

The elastic beam is a good test for the quality of the beam. The beam orientation is most important. It can be adjusted with the "focus" and the "deflection" (68, 68'). The elastic beam is found at the incident beam energy (see "Ortec" power supply on the left of F). The peak of the elastic beam should be intense and symmetric.

3. **Optimal Signals**

   i. Low energies (-10 to -70 volts):
      
      - high current: \( I_p = 3 \) to 4 mA
      - high voltage: \( E_p = 2.0 \) to 2.5 keV
      - low modulation amplitude; \(~1.0\)
      - high sensitivity; 2.5 on phase sensitive detector
      - low scan rate; 10 V/sec

   ii. Medium energies (-70 to -500 volts):
      
      - current; \( I_p = 2 \) to 3 mA
      - voltage; \( E_p = 1.5 \) keV
      - mod. amplitude; 4 to 6
      - sensitivity; 10 on phase sensitive detector
      - scan rate: 100

**IV. ION-ETCHING**

a. **Preparations**

   1. Fill copper line between valve 9 and leak-valve 70 with Argon (Fig. 2).
      
      i. purge manifold with Ar (p ~10 psi)
      
      ii. open valve 9 to Copper-tube and purge
2. Fill chamber with Argon
   i. switch ion gauge E on (See III.2)
   ii. close gate valve of big "vacion" pump until you hear a click
       (pressure increases ~1x10^{-8} Torr)
   iii. close gate valve of small "vacion" pump (pressure increases to
        10^{-7} - 10^{-6} Torr)
   iv. open leak valve 70 (~2 turns counter-clockwise, check pressure
       on ion-gauge and remember closed position of leak valve. Damage
       can result when valve is overtightened!!!). If Ar-pressure in
       chamber ~10^{-5} Torr, close leak valve.\footnote{A small continuous Ar-flow can be maintained by opening leak valve and gate valve of small (noble-gas) "vacion" pump a little. This procedure helps to clean the system of reactive sputter-material.}
   v. after sputter-experiments, open small gate valve slowly and pump
      for awhile with the small pump only to keep Ar out of the big
      pump.

b. Ion-Bombardment

1. Turn electron beam off.
   i. turn 41 to 0 (meter C_{2})
   ii. power of left power supply (39) OFF (Panel F)

2. Apply positive voltage to screen to protect it from sputter debris.
   i. switch right "Ortec" power supply (40) OFF
   ii. turn switches on the rear of this power supply to "internal" and
       "positive" (with screw driver)
   iii. turn power supply on again (40)
   iv. set it to +300 volts with 47

3. Switch ion-gauge filament OFF (69)
4. Ion-beam (Panel D)
   i. "Operate" mode, 71
   ii. power on, 72
   iii. "beam energy" on, 73
   iv. "emission" current to 30 mA, 74
5. Control focus and beam direction visually in completely dark room
6. Positioning of beam (requires 2 people)
   i. "scan" voltage OFF, 75
   ii. "focus" (focal point on sample), 76
   iii. "deflection" 77  x: direction ellipsometer arms
       y: direction front window
V. INSTRUMENTS
A. "VacIon" pump control unit, Varian Mod. 921-0034 (for big pump)
B. Microvolt-Ammeter, "Keithly" 150AR
C. Auger incident electron beam gun control power supply, LLL 7S 1083
D. Ion bombardment gun power supply, LLL
E. "Ultek" digital ion gauge control
   Perkin Elmer Mod. 605-0002
F. - Two "Ortec" high voltage (0-3kV) power supplies, Mod. 456,
   - Neutralizer, LLL 7S 1143
   - Oscillator modulator, LLL 7S 1103
   - Recorder drive, LLL 7S 1122
   - Auger ramp generator, LLL 7S 1113
G. Phase sensitive detector (Lock-in amplifier), Princeton Applied Res.
   Mod. 5101, 5Hz-100 kHz
H. Oscilloscope, Tektronix T92212
I. "VacIon" pump control unit, Varian Mod. 921-0062 (for small noble gas pump)
J. Titanium sublimation pump control unit, Varian Mini Ti-Ball/TSP

ACKNOWLEDGMENT

This work was supported by the Assistant Secretary for Conservation and Renewable Energy, Office of Energy Systems Research, Division of Energy Storage of the U.S. Department of Energy under Contract No. W-7405-ENG-48.
FIGURE CAPTIONS

Figures 1, 2. Elements of the ultrahigh vacuum chamber

1 - vertical specimen movement
2,3 - horizontal specimen movement
4 - specimen rotation
5 - bellows
6-9 - manifold connectors
10 - gate valve to small (noble gas) ion pump
11 - manifold main valve
13 - sorption pumps
15,16 - sorption pump valves
17 - vents for sorption pumps
18 - feed-through for Faraday cup
19 - ion gun
20-23 - connections to screen and grids for
AUGER and LEED
24 - high voltage connector for large ion pump
25 - ground connection for large ion pump
26 - electron gun
27 - high voltage connector to small ion pump
28 - ground connection for small ion pump
29 - thermal conductivity pressure gauge on manifold
(Varian 0531)
30 - ion vacuum gauge
31-35 - connecting pins for ion gauge
70 - leak valve
Figure 3. Power supplies

A - Power supply for large ion pump
B - Microammeter for Faraday cup
C - Power supply for Auger electron gun
D - Power supply for Ion gun

Figure 4. Electronics for Auger analysis

E - Ion gauge control
F - Auger control panels
G - Phase sensitive detector
H - Oscilloscope

Figure 5. Calibration of modulation oscillator amplitude control (Fig. 4, panel F, knob 42)
Cu-tubing for Ar

Fig. 2
Fig. 3
MODULATION AMPLITUDE CALIBRATION

Fig. 5
This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.