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MASTER

OPERATING PROCEDURE FOR COMBINED AUGER -
AND ELLIPSOMETER UHV-SYSTEM

Felix Schwager

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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.

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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	→	ground
d. Ground retarding grid 23 (2nd grid with Teflon). Connect through hole of Teflon, tighten with Allen wrench	→	oscillator
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 counter-clockwise 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 "vacuon" 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 N_2 -cylinders
 3. purge manifold with N_2 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 N_2 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 N_2 - 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 N_2 - flow.
- j. Take heaters off sorption pumps 13 and attach styrofoam dewars. Cool one sorption pump down with liquid N_2 until boiling of N_2 calms down.

- k. Open valve between sorption pump and manifold (15 or 16), first with wrench until click is heard, then manually.
- l. 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 $\sim 5 \times 10^{-4}$ torr, close valve 11 to manifold and open gate valve to small "vacuon" 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 "vacuon" 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 $\sim 1.5\text{keV}$
 - low energy range high voltage $\sim 2.5\text{keV}$
 - high energy range $\rightarrow$ low voltage $\sim 1.5\text{keV}$
 - 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 C_2 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, C_2 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!!!

D. Measurement and Recording

1. Recording of measurements on an HP7044A recorder.

- i. Oscilloscope on Channel 2 (CH2) $\rightarrow$ horizontal line on screen
- ii. Recorder: LINE, SERVO ON
- iii. "Auger Ramp Generator" (F, 55), ON
(Scan rate "Slope" fast for scaling test)
- iv. X-position adjustment on recorder or "Recorder Drive" (F),
"Recorder Offset Voltage" (65)
- v. Y-position adjustment on oscilloscope H with 64 and
scaling with 66.

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" 38 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 4mA

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; 10V/sec

ii. Medium energies (-70 to -500 volts):

current; $I_p = 2$ to 3mA

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 ($p \sim 10$ 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 $\sim 1 \times 10^{-8}$ 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 $\sim 10^{-5}$ Torr, close leak valve.¹
- 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)

¹ 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.

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

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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)

XBL 819-1367

Fig. 1

XBL 819-1364

Fig. 2

XBL 819-1299

Fig. 3

XBL 819-1298

Fig. 4

Fig. 5

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