*We supply photographs of the equipment in the file ‘JoVE-NRA-orientation.pptx’. In ‘Filmed Steps.docx’ we highlighted all the actions that can be visualized by videographer footage of actions performed by a lab member in different colors, depending on which equipment specified in ‘JoVE-NRA-orientation.pptx’ is used to change an experimental parameter or to display this change. Thus a change of text highlighting color suggests a cut in the video footage to a different camera angle. ‘Filmed Steps.docx’ provides the chronological order of the Steps/actions. The highlighting code in following table indicates which action (or parameter change indication) takes place where and thus makes suggestions for scene sets to be filmed. The combined information in ‘Filmed Steps.docx’, ‘JoVE-NRA-orientation.pptx’, and in the table below is equivalent to the ‘Screenwriter\_Guide’.*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **#** | **Scene/Shot** | **Location** | **Steps** | **Note** |
| **1** | UHV chamber, outside | BL-1E | 2.2.1) - 2.3.2), 2.3.5), 4.2) | Fig. 1 (A), Photo |
| **2** | UHV chamber, inside | BL-1E | 2.2.1), 2.2.5), 2.3.1), 4.1) | Fig. 1 (B), Photo |
| **3** | UHV Pressure gauge | BL-1E | 2.2.2), 2.2.5), 4.2) | See Photo |
| **4** | Ion gun controller | BL-1E | 2.2.1), 2.2.2) | See Photo |
| **5** | Digital tester | BL-1E | 2.2.2), 2.2.4), 2.2.5) | See Photo |
| **6** | High voltage power supply | BL-1E | 2.2.4) | Sample bias,  See Photo |
| **7** | Filament heater supply | BL-1E | 2.2.4), 4.1) | See Photo |
| **8** | LEED, screen & controller | BL-1E | 2.2.6) | Darkened room, Fig. 3 |
| **9** | High voltage power supply | BL-1E | 2.3.2) | Ion Beam Deflector, Photo |
| **10** | Control Panel | Control Room | 2.3.4), 2.3.5), 3.9), 3.10), 4.3), 5.1) | See Photo |
| **11** | Control System | Control Room | 2.3.5), 4.5) | PC screen |
| **12** | Beam Profile Montitor | Control Room | 3.10) | Oscilloscope |
| **13** | Current Integrator | Control Room | 2.3.4) | See Photo |
| **14** | TV monitor | Control Room | 2.3.5), 3.10) | TV monitor |
| **15** | BL-2C chamber | BL-2C | 3.1), 3.2), 3.5), 3.7), | Fig. 2, Photo |
| **16** | Preparation table | BL-2C | 3.3), 3.4) | See Photo |
| **17** | Measurement PC | BL-2C | 4.6), 4.7), 5.2), 5.4) | PC screen |

2.2.1) Position sample in chamber center with the manipulator x, y, z-stage and rotate to align surface between viewport and ion gun (facing the gas doser). Switch on ion gun power supply and adjust ‘Emission’ control to 20 mA. Look at the sample through the viewport and fine-adjust sample rotation angle so that the mirror image of the glowing ion gun filament is visible on the sample surface.

2.2.2) Set ‘Beam energy’ on ion gun power supply to 800 eV. Close NEG pump gate valve at the chamber bottom and introduce 6x10-3 Pa Ar gas into UHV chamber through variable leak valve. Confirm a sputter ion current (digital tester, from sample to ground) around 2 A and sputter surface for 10 min at room temperature.

2.2.3) Add liquid nitrogen to the manipulator cryostat. At manipulator head, connect filament heater leads to power supply and digital tester (20 mV range) to thermocouple feedthrough. Ground the filament.

2.2.4) Connect sample contact to bias voltage power supply. Apply sample bias of 1 kV. Use filament heater currents up to 6.6 A for annealing, oxidation, and flash-heating in the next Step (2.2.5) while monitoring the thermocouple voltage (sample temperature) with the digital tester.

2.2.5) Anneal sample in UHV to 1000 K for 10 min ensuring the pressure remains below 2x10-7 Pa. Oxidize at 750 K in 5.0×10-5 Pa O2 for 5 min, then reduce at room temperature (RT) in 5.0×10-5 Pa H2. Perform a final flashing to 600 K in UHV.

2.2.6) Observe LEED pattern and repeat steps 2.2.1) to 2.2.2) (sputtering) and 2.2.3.) to 2.2.5) (annealing/oxidation/H2-reduction) until a clear (1×1) structure with bright spots on low background results (Figure 3) and no impurities remain in Auger electron spectroscopy. Sputter for only 2-3 minutes in the repeated sputter/annealing cycles.

2.3.1.) At the 1E UHV chamber, position sample in chamber center (x = 25, y = 26, adjust z by eye to height of QMS front aperture) and rotate to face 15N ion beam line. Bring quartz plate beam profile monitor (Figure 1 (B)) into NRA measurement position by lowering sample holder by sample-monitor distance measured in Step 2.1.5). Set digital camera on window flange below the manipulator to transmit beam profile image on the quartz plate to the TV monitor in the accelerator control room.

2.3.2) Remove all other electrical contacts to sample at manipulator head and connect signal line to digital current integrator in control room. Set electrostatic deflector voltage on BL-1E to 8500 V. Open the three manual gate valves on BL-1E between the UHV chamber and the bending magnet BM04.

2.3.4) In the control room, switch current integrator from ‘Stand by’ mode to ‘Operate’. Connect integrator analog output to current indicator. At the accelerator control panel, adjust 15N ion beam energy with accelerator in slit feedback mode to an energy analyzer magnet field of 5540 Gauss (Parameter: NMR03) and match bending magnet field (Parameter HPB04) to ~-6033.4 Gauss to direct ion beam onto target in 1E UHV chamber. Set magnetic quadrupole lens parameters (MQ04) to XCC=4.64 A and YCC=5.15 A to focus beam approximately.

2.3.5) In the control room, open two gate valves between accelerator and beam line 1E. Open Faraday cup (FC) FC04 and observe ion beam profile on quartz plate in target chamber on the TV monitor. Fine tune BM04 and MQ04 parameter settings to obtain well-focused ion beam in center of profile monitor plate. Adjust z-position of quartz monitor with sample manipulator if necessary.

3.1) Lift up any previously used sample from the beam line position into to the manipulator transfer rod, secure height with fixation screw, and close gate valve to the beam line.

3.2) Detach sample current line at electrical feedthrough and rotary pump line at KF flange coupling of the manipulator. Detach manipulator from the gate valve flange.

3.3) Place manipulator onto preparation table and slide sample holder out of the transfer tube. Rotate manipulator axis to place sample horizontally.

3.4) Loosen two M2 cap screws of sample clamp (Figure 2 (B)) and remove old target. Set new sample, align parallel to manipulator axis, and tighten clamp screws. Retract sample into transfer tube and secure position with fixation screw.

3.5) Replace copper gasket on gate valve and reinstall manipulator on the beam line. Attach rotary pump line to manipulator. Close valve in the rotary pump line to the turbo-molecular pump (TMP).

3.7) Lower sample to beam line position and align surface normal of beam profile monitor (glass plate) to incident beam direction with aid of BL-2C camera and nearby TV monitor. Then connect BL-2C camera signal line to TV monitor in control room.

3.9) Roughly align 15N ion beam to target in BL-2C by setting bending magnet field (Parameter HPB04) to ~0.6 Gauss (Polarity: positive), magnetic quadrupole lens MQ04 parameters to XCC=4.64 A and YCC=5.15 A, and quadrupole lens MQ-2C parameters A=3.6 A and B=3.9 A to focus beam approximately.

3.10) Fine-tune HPB04/MQ04(XCC, YCC)/MQ-2C(A, B) parameters to optimize beam transmission (unobstructed passage to target) and beam profile on target (use beam profile monitors BPM-1C and BPM-2C and BL-2C camera image) and take note of the best settings.

4.1) Flash-heat Pd sample to 600 K to free surface from any adsorbed contaminants. Stabilize sample temperature at 145 K with filament heater (~3.6 A) and running He compression cryostat (or liquid nitrogen cooling).

4.2) Close valves to accelerator and to NEG pump and expose sample to 2000 L H2 (2.66x10-3 Pa x 100 s) at 145 K. Let sample cool to 80 K and adjust a H2 background pressure of 1x10-6 Pa.

4.3) In the control room, set 15N ion beam energy at BM03 to desired start value for the energy scan (typically NMR03 = 5530 Gauss) and adjust BM04 according to the MagparNNN.xls table.

4.5) Verify again that all valves on BL-1E are open, that the sample current signal line is connected, that the current digitizer is set to ‘Operate’, and that a 15N beam of ideally 15±5 nA is available on FC04.

4.6) Set the stat START, STOP and STEP values of the BM03 parameter for the energy scan (typically 5530 Gauss, 5600 Gauss, and 1 Gauss, respectively) and turn the option ‘Force TVC to gvm’ on. For a ~15 nA beam of 15N2+, set the ‘Acquisition time’ parameter to 50 s.

4.7) Click the ‘Execution’ arrow in AutoScanLinuxUHVfb3.vi console to start automated acquisition of a depth profile (up to ~35 nm depth in Pd for STOP = 5600 Gauss). At the end of the scan (or for an earlier termination), click ‘Stop measurement’ to close the data file.

5.1) In the control room, set 15N ion beam energy at BM03 to desired START value for the energy scan (typically NMR03 = 5530 Gauss to start profiling at the surface).

5.2) Load the NRA data acquisition software (NRAmain.vi) on the accelerator control PC at BL-2C. Select depth profiling routine ‘AutoScanLinux11.vi’. Enter the desired BM03 parameters for the automated energy scan (START, STOP, STEP), matching START to the BM03 value set in 5.1).

5.4) Click ‘Execution’ arrow in ‘AutoScanLinux11.vi’ to acquire a depth profile that terminates automatically at the STOP value of the BM03 parameter. At the end of the scan (or for earlier termination), click ‘Stop measurement’ to close the data file.