Ultrahigh vacuum and surface science in the Central Laser Facility (CLF)

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Introduction

Physical and chemical processes occurring at interfaces are of particular importance to both science and technology. Over recent decades numerous tools have been developed to probe the static structure and spectroscopy of solid surfaces in detail[1]. However, there is a need to move to more realistic experimental environments, shorter more dynamical timescales, and to be able to yield information at the atomic and molecular level. This challenge requires the development of new experimental tools based on coupling ultrahigh vacuum (UHV) surface science technology with advanced laser pump-probe techniques. Thus, a surface science facility has been installed and developed for the Lasers for Science Facility (LSF) at the CCLRC, Rutherford Appleton Laboratory (RAL).

In this article we describe the UHV chamber experimental design and the surface science instrumentation provided.

Overview

A schematic of the UHV chamber is shown in Figure 1 with a photograph of the chamber at RAL in Figure 2. The 30 cm diameter stainless-steel chamber is divided into three levels: (1) surface analysis, (2) sample cleaning and (3) pumping.

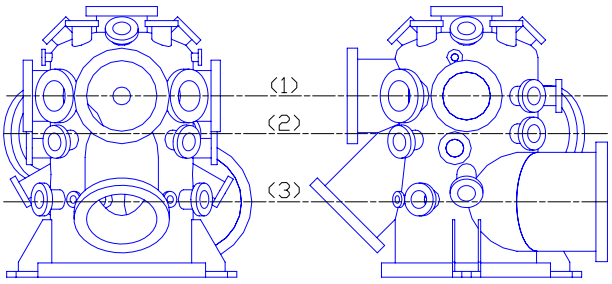

Figure 1. Schematic of the UHV chamber (side view).

The chamber is pumped by a liquid nitrogen trapped 6" oil diffusion pump (BOC Edwards) backed by a mechanical rotary pump (BOC Edwards). With the addition of further pumping, provided by a liquid nitrogen trapped titanium sublimation pump (Instrument Technology Ltd.), and baking at 120 °C for 48 hours a base pressure of better than 2×10^{-10} mbar is routinely achieved. An argon ion sputter gun is provided for surface cleaning purposes while the sample surface contamination and reconstruction is checked by Auger electron spectroscopy (AES) and low energy electron diffraction (LEED) spectroscopy (SPECS Scientific Instruments) respectively.

A differentially pumped precision XYZθ manipulator (Centiax, Thermo Vacuum Generator) and a closed-cycle helium cryostat (APD Cryogenics) capable of reaching temperatures around 10K is provided. The sample can also be heated up to 1500K with computerised control and monitoring. Two gas lines are provided, one gas handling line for gas purification and dosing into the chamber by fine control leak valves, and a second gas line (bakeable) for dosing high purity argon, oxygen and hydrogen for sputtering, oxidation and reduction processes.

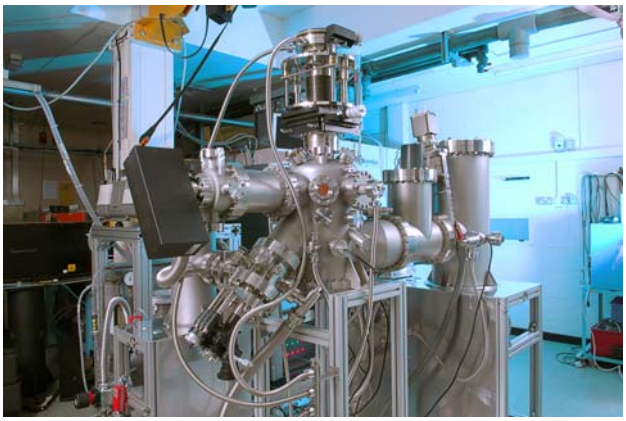

Figure 2. The UHV chamber in the Nanosecond science Laboratory (Lab B) at the Central Laser Facility.

Experimental Design

The chamber is equipped with a high quality mass spectrometer (Hiden Analytical Ltd.) for line-of-sight temperature programmed desorption (LoSTPD)[2] and pulsed laser desorption as shown in Figure 3. In addition, charged particle and optical detection tools are provided.

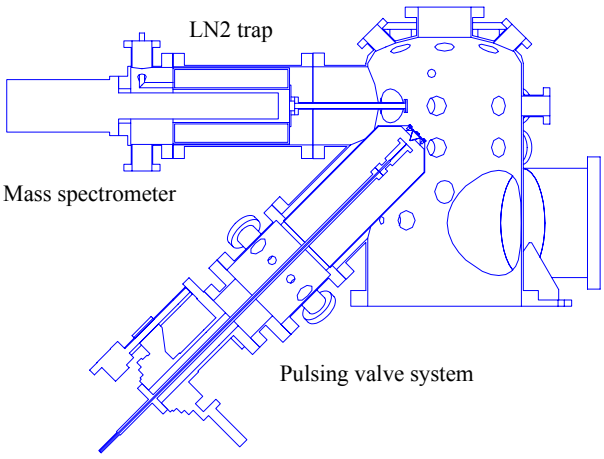

Figure 3. Schematic of the UHV chamber with mass spectrometer and pulsing valve system.

Surface infrared spectroscopy represents a particularly powerful method for studying molecular orientation and the nature of bonds formed/broken on adsorption. Two differentially pumped infrared windows are provided to carry out surface infrared spectroscopy in both grazing incidence reflection (FT-RAIRS) and transmission geometries for semiconductor[3] and insulator surfaces as shown in Figure 4.

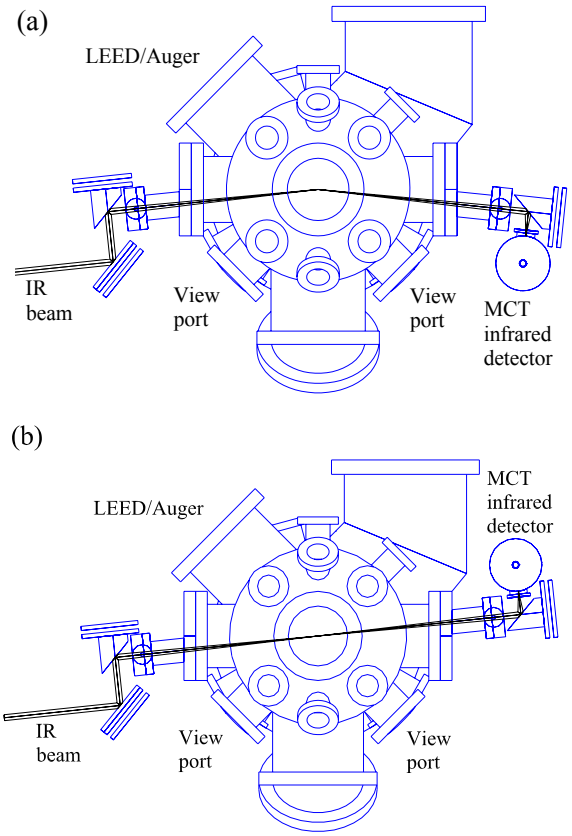

Figure 4. Schematic of the UHV chamber (top view) with infrared optics in (a) RAIRS and (b) transmission geometry.

First Ultrahigh Vacuum at the LSF

The UHV chamber was installed on the 7th February 2005 at the LSF in the Nanosecond Laboratory and is currently completing its commissioning.

After 48 hours baking followed by degassing of all filaments, a pressure of 2×10^{-10} mbar was achieved (measured by a hot cathode ion gauge). The main contaminant, hydrogen, was found by using a residual gas analyzer (RGA 200, Stanford Research System) quadrupole mass spectrometer as shown in the spectrum in Figure 5. Hydrogen contamination is expected for a new UHV system since stainless steels are commonly contaminated with dissolve H_2 which outgases over time. The TSP can be used to reduce the partial pressure of H_2 in the chamber.

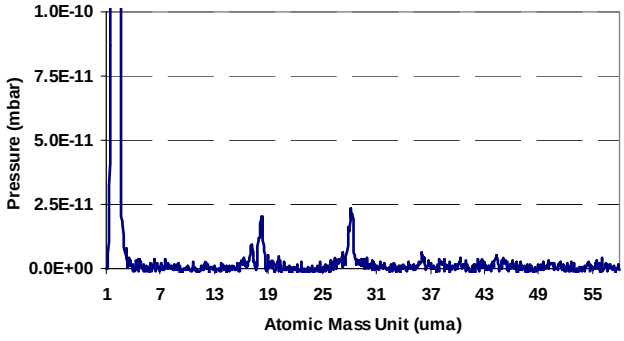

Figure 5. RGA 200 mass spectrum (2×10^{-10} mbar base pressure after 48 hours bake at 120 °C). The hydrogen intensity peak at 2 m_u is off scale ($P \approx 2\times10^{-9}$ mbar) due to the sensitivity of the mass spectrometer to that species.

Sample Mounting and Manipulation

For commissioning purpose, a platinum Pt(111) sample (10 mm × 1 mm) was provided. The crystal is aligned to 0.5 degrees of the (111) plane and polished to a 0.03 micron surface roughness (Surface Preparation Lab). The Pt(111) crystal is clamped by tantalum plates onto the surface of a substrate heater (HeatWaves Labs, Inc.) capable of 1500 K operation in UHV.

The entire sample assembly is mounted on a precision manipulator, which permits translation along X, Y and Z axes and unlimited rotation due the differentially pumped double o-ring rotary seal. The gold plated sample stud made of oxygen free copper (University of Nottingham) is shown in Figure 6.

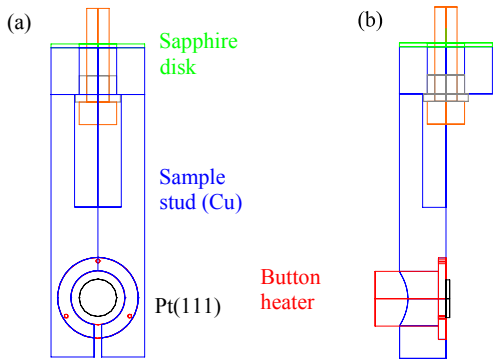

Figure 6. Sample holder front (a) and side view (b).

A K type thermocouple (Cr/Al) is attached to the sample to monitor the temperature during heating processes. Two additional thermocouples K and KP type (-272 to 330 K) are attached to the sample holder body for low temperature with computerised control and monitoring.

Initial cleaning of the Pt(111) crystal was achieved by argon ion bombardment followed by annealing at 1500 K for two minutes. Once the contamination had been reduced, an oxidation process and annealing was carried out, during which the crystal was held at 900 K in 5×10^{-7} mbar O_2 for 20 minutes and annealed for 1 minute at 1500 K.

Low energy electron diffraction (LEED) was then employed to determine the state of the platinum crystal surface. The LEED patterns were recorded with a digital camera at room temperature. Using a low energy beam, a 1×1 LEED hexagonal pattern was obtained, which confirmed a well-ordered surface, as shown in Figure 7.

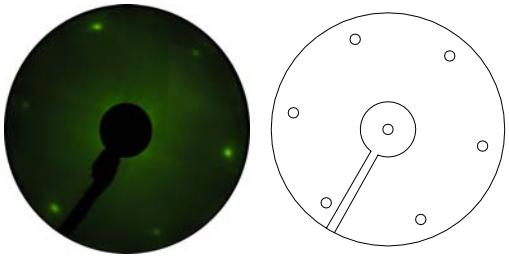

Figure 7. Hexagonal LEED pattern of Pt(111)–(1×1). Beam energy: 80 eV.

Acknowledgements

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