Total analysis of surface structure and properties by UHV transfer system

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Abstract

We have developed an ultrahigh-vacuum (UHV) complex sample preparation and analysis system, which realizes a reliable surface science analyzing various characters on an identical surface. The system contains three sample-preparation-and-characterization chambers and five analysis chambers. They are (1) an electronic-properties-characterization chamber, (2) a magnetic-properties-characterization chamber, (3) an organic-molecule chamber, (4) UHV SEM, (5) a high-energy-resolution angle-resolved photoelectron spectrometer, (6) a high-energy-resolution display-type spherical mirror analyzer, (7) a room-temperature (RT) STM, and (8) an optical-properties characterization chamber. A special sample holder is used with six electrodes on it, which enables accurate temperature measurement of a sample by connecting a thermocouple directly to the sample even if it is transferred. Four other electrodes can be used for construction of various circuits including evaporators. Some examples are shown.

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1. Introduction

One of the difficulties in surface science is the total (structure and other properties) analysis on an identical surface. In solid state physics, one sample can be used by different researchers in different laboratories, but in surface science, we cannot carry one surface to other laboratories in air. Although structure and some properties have been studied for many surfaces, the surfaces in different studies are not guaranteed to be identical, and the coverage or the surface conditions may be different. Sometimes only one percent difference of the coverage produces much differences of surface conductivity. Hence we constructed a UHV total analysis system to prepare well-defined surfaces and to carry them to various analysis systems through UHV transfer system.

2. Experimental apparatus

Fig. 1 shows the ultrahigh-vacuum (UHV) complex sample preparation and analysis system. This system connects three sample-preparation-and-characterization chambers and five analysis chambers through a UHV sample transfer system 10 m long.
The three sample-preparation-and-characterization chambers are (1) an electronic-properties-characterization chamber, (2) a magnetic-properties-characterization chamber, and (3) an organic-molecule chamber. All three chambers are equipped with several evaporation sources and reflection high-energy electron diffraction (RHEED) apparatus, which characterize surface atomic structure. The organic-molecule chamber has special evaporation sources for organic molecules. The electronic-properties-characterization chamber is equipped with a surface electric conductance measurement system. The magnetic-properties-characterization chamber is equipped with magnets and a SMOKE (surface magneto-optical Kerr effect) system which can measure surface magnetization. The sample temperature can be changed between several tens K to 1000 or 2000 K depending on the heating setup.

The five analysis chambers are (4) a UHV SEM (scanning electron microscope and Auger electron micro-probe (JEOL JAMP-7810)), (5) a high-energy-resolution angle-resolved photoelectron spectrometer (Gammadata-Scienta SES2002), (6) a high-energy-resolution display-type spherical mirror analyzer (DIANA), (7) a room-temperature (RT) STM (scanning tunneling microscope), and (8) an optical-properties characterization chamber. The base pressure of the three sample-preparation chambers and the five analysis chambers is around or below $1 \times 10^{-8}$ Pa.

A low-temperature STM (LT-STM) in Fig. 1 is separated from the UHV complex sample preparation and analysis system because the vibration from the system may disturb the fine STM analysis. The excimer laser and the femto-second laser are used for the excitation of surface for the measurement of optical properties in the optical-properties-characterization chamber. The excimer laser is also used for the evaporation of high-melting-temperature materials by laser ablation in the electronic-properties-characterization chamber.

A special sample holder is used in all apparatus connected to this system. In other words all apparatus have a common sample holder receptor. Fig. 2a shows a schematic drawing of the sample holder and transfer system. Fig. 2b shows a photograph of the sample holder. The base box is usually made of Cu as shown in Fig. 2b so that the
sample can be cooled efficiently. Sometimes it is made of Ta so that it allows electron bombardment heating of the sample up to more than 2000 °C. The size of the base box is 11 mm × 20 mm × 27 mm. The weight is around 40 g. The sample is usually set at 9 mm above from the lid of the base box. The space between the lid and the sample is used for a heater or a filament for electron bombardment. The base pressure of the chamber is kept in the 10⁻⁸ Pa range during the heating with a 10 A direct current through the sample.

The holder has six electrodes (two sockets and four screw heads) as seen in Fig. 2b. Two of which (the two sockets) can pass a high-current of about 10 A, and the other four electrodes are used for thermocouples or a four-point probe for the electric conductance measurement. In the construction of Fig. 2b, direct current heating is done between one of the high-current electrodes and ground, evaporation of alkali atoms is done between another high-current electrode and ground, a two-point probe for four-point probe measurement is connected to two of the thermocouple electrodes, and the other two thermocouple electrodes can be used for the measurement of the temperature of the sample.

The important point is that the electrodes of the sample holder receptor for the thermocouple are made from the materials of the thermocouple. Usually the thermocouple is connected to the sample receptor in all commercial sample transfer systems. In this system we can transfer the thermocouple connecting directly to the sample, hence accurate temperature of the sample can be measured even when the sample is transferred. These electrodes are fixed firmly by screws, which are rotated by the transfer rod.

The transfer of the sample holder is made by inserting the head of the transfer rod to the sample holder axis. Short bars sprouting perpendicular to the axis enter the slit of the transfer rod and connect the sample holder and the transfer rod. The axis can rotate inside the sample holder, but the rotation is hindered by a spring which holds the hexagonal plate of the axis as shown in Fig. 2a. The sample holder is pushed into the receptor and the other short bars (seen in Fig. 2b) connect the screw of the receptor and the sample holder. The sample holder is fixed tightly by rotating the receptor screw through the axis by the transfer rod.

The typical time required to put in or remove the sample is about 2 min.

In the same way, the evaporation sources can be put into the chamber from outside using the same transfer system. Fig. 3 shows an evaporation source for Mg, which can be transferred into the chamber. Eleven evaporation sources can be set in the chamber. The size of the base box is 7 mm × 12 mm × 18 mm. The weight is around 15 g. The crucible is usually made of alumina and the box is made of stainless steel or Ta. The base box is separated into two parts which are connected through insulators. The heater filament is usually Ta wire of 0.3 mm diameter and connected to these two parts. When this evaporation source is inserted to a receptor the front part touches an electrode made of a plate spring and the rear part touches the ground. The base pressure of the chamber is kept in the 10⁻⁸ Pa range during the evaporation with about 2 A current through the filament around the crucible. Typical time required to put in or remove the evaporation source is about 1 min. It is necessary to wait about 3 h for the vacuum in the preparation chamber to become high enough to open the valve between the preparation chamber and the main chamber. Hence the total time to exchange the sample or evaporation source is about 3 or 4 h.

3. Example of experiment

Here we show one example of total analysis of Mg on Si(111) surface [1]. The preparation and the characterization of the surface and the surface electric conductance were measured in (1) the electronic properties characterization chamber. Fig. 4 shows a RHEED pattern of...
Si(111)3·1-Mg surface, which was observed at one-third ML (monolayer) Mg on Si(111) surface.

The surface electric conductance during the evaporation of Mg on Si(111) surface is shown in Fig. 5. The conductance increased rapidly after some period after the start of evaporation and monotonically increased until the evaporation stopped. This behavior of increase of conductance after some period can be understood by the percolation of metallic islands as discussed by Heun et al. [2]. After the percolation, these metallic islands form continuous metallic film.

The electronic structure of this metallic film was measured using (5) the high-energy-resolution angle-resolved photoelectron spectrometer after transportation using the transfer system. Fig. 6 shows the electronic state at high coverages of metallic film. Free-electron-like quantum well states are clearly seen. These quantum well states correspond to those observed by Aballe et al. [3]. These states are considered to be the origin of the high conductance of this surface.

Another example is the optical properties of organic thin-film on several kinds of substrates. The measurements were made using (8) the optical-properties characterization chamber after preparation using (3) the organic-molecule chamber. Fig. 7 shows the RHEED patterns of Ag(111) structure after deposition of Ag on Si(111)7×7 clean surface. A textured pattern is seen from the Ag(111) islands randomly oriented parallel to the surface.

Fig. 8 shows the photo-luminescence (PL) spectral intensities from an aluminum tris(8-hydroxyquinoline) (Alq3) molecule film on a slide glass (insulator), Si(111)7×7 clean surface (surface metallic and bulk semiconducting) and Ag(111)/Si(111) (bulk metallic) surfaces. PL intensity from Alq3 increases on all substrates at the first stage, as shown in Fig. 8. The PL intensity difference increases as the thickness of Alq3 increases until about 100 nm. The PL intensity on an Ag surface is about half of that on a glass. Similar behavior was reported by Choong et al. [4].
although the thickness of the turning point is different. Various interactions between the Alq$_3$ molecule and the substrate or the difference of relaxation process by impurities are considered to be the origin of the difference of PL intensities. At thin (below about 10 nm) thickness, the PL intensity on a metal substrate is considered to be smaller than those on a semiconductor or an insulator surface by the effect of image charge described by classical electro-magnetic theory [5], and this idea is supported by theoretical works. At greater thickness, however, the re-absorption or interference effects have to be considered. Although we cannot explain the present results sufficiently we could observe an interesting phenomena.


4. Conclusion

We realized a UHV complex sample preparation and analysis system for accurate surface science. The system contains three sample-preparation-and-characterization chambers and five analysis chambers. This kind of total analysis system for an identical surface is necessary for the precise understanding of surface properties and for the development of quantitative theoretical work on surfaces. Examples of total analysis for Mg on Si(111) and Alq$_3$ on various substrates were described.

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