Journal of the Korean Vacuum Society
Vol. 2, No. 3, September 1993, pp. 288-293

〈연구논문〉

A Design of Transferable Sample Holder for Resistive Heating and Conductive Cooling

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(Received July 30, 1993)

통전가열과 접촉냉각이 가능한 교환시료 고정장치의 고안

서재명 · 김기정 · 임삼호 · 구세정 · 박 찬 · 김영기

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(1993년 7월 30일 접수)

Abstract — A simple and novel sample-holder which has multi-functions of resistive heating, conductive cooling, electrical biasing and sample exchanging has been designed in order to investigate gas-surface interactions for various surface directions under ultrahigh vacuum and low temperature. It has been proved that the wafer can be cooled from 1600 K to 20 K within two minutes. Therefore the controlled adsorption of most gases, except H, He and Ne, were possible on the clean surfaces. The design has substantial applications in areas like surface phase transitions and gas-surface chemical reactions.

요　약 — 통전가열, 접촉냉각, 전기적 bias 및 시료 교환을 자유로이 할 수 있는 다기능의 새롭고 간단한 시료 고정장치를 저온 및 초고진공 상태에서 기체와 여러 방향의 시료표면 사이의 상호작용을 연구하기 위하여 고안하였다. 이 장치로써 2분 이내에 기판의 온도를 1600 K에서 20 K로 2분 이내에 냉각시킬 수 있었다. 그러므로 청결한 표면위에 수소나 He이나 Ne을 제외하고는 대부분의 기체를 흡착시킬 수 있게 되었다. 이러한 장치는 표면에서의 상변화, 기체와 표면의 화학반응의 연구에 중요하게 활용될 수 있다.

1. Introduction

Studies on the clean surface held at low temperature open more chances to observe the dynamics of adsorbed species through conventional surface analytical techniques such as X-ray photoelectron spectroscopy (XPS), Ultraviolet photoelectron spectroscopy (UPS) and Low energy electron diffraction (LEED) combined with the closed-cycle He refrigerator. In addition to the gas-surface dynamics, the enhanced chemical reaction or the phase-transition can be observed at the low temperature.

In-situ preparation of an atomically clean surface is the starting point of these studies. Especially the direction-dependent surface studies usually require the resistive heating of wafers under ultrahigh vacuum (UHV). Furthermore, in order to condense exposed gases such as oxidizers or rare gases, the clean surface should be cooled down to lower temperature than the physisorption temperature of these gases[1]. One of the accompanying problems is the unwanted physisorption of residual gases on the prepared surface during the cooling period. Therefore the residual gas pressure should be kept minimum during and after the resistive heating, until the sample temperature reaches down to the required temperature.

Another problem is in the contradictory conditions for resistive heating and conductive cooling. For example, the efficient resistive heating requires that the sample is only heated while the rest of the section is not. However, such a thermal insulation to the sample introduces the poor conductive-cooling from the cold head of the refrigerator. Therefore, it is difficult to reach the required physisorption temperature. Conversely, if the conductive cooling is efficient, the whole system including the refrigerator goes quickly to thermal equilibrium with the annealing surface, which might cause the permanent damage to the cooling system. Therefore it is necessary to increase thermal isolation during the resistive heating and to increase thermal conductance during the cooling period.

In addition to the preparation of the clean and cold surface, sometimes it is necessary to bias the sample electrically. For example, in order to monitor the work-function it is necessary to make the secondary electron cut-off energy of the sample be separated from that of the analyzer by biasing the sample with appropriate potential. Sometimes, it is also necessary to retard the incident electron-beam. Since the cold head of the closed-cycle He-refrigerator is usually grounded the sample should be electrically isolated from the ground in order to be biased.

In this paper the novel design of a sample holder and stage which can solve the above problems is presented. In addition to solutions to the listed problems, the holder can be transferred by the load-lock in order to exchange the sample from the outside of the vacuum and the sample temperature can be monitored by the thermocouple during the cooling procedure.

2. Design of Multi-Functional Sample Holder

In Fig. 1(a) the design of the sample holder is presented. The sample holder consists of three parts. The main part is made of an oxygen-free-high-conductivity (OFHC) copper block. It consists of two 1″-diameter plates and the top 7/8″ diameter plate. The space between the top and the 2nd plate is used for the load-lock transfer. Three plates are connected by 5/8″ diameter concentric plates. Actually these 5 plates consist of one body with a 8 mm-screw-hole at the center. The bottom plate

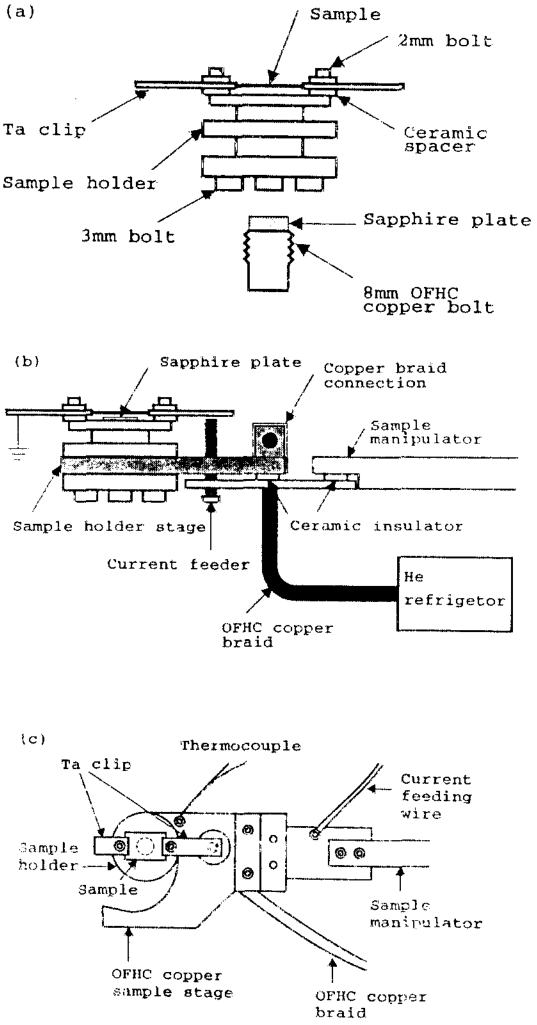

Fig. 1. Design of the sample holder and the sample holder stage. (a) Side view of the sample holder and the OFHC copper bolt. The copper bolt is inserted into the center of the holder and moved up by the vacuum screw driver until it is in contact with the sample. The sapphire plate can be optionally mounted to bias while cooling. (b) Side view of the sample holder stage when the holder is mounted. The vacuum screw driver from the bottom rotate 3 mm bolts, the current feeder and the 8 mm OFHC copper bolt. The manipulator has the freedoms of translation, rotation, and tilt. The length of the OFHC copper braid is 30 cm long. The sign of the earth-ground at the left Ta-clip indicates the current feed-through from the chamber wall. (c) Top view of the sample holder stage when the holder is mounted. The dotted circle inside the sample indicates the

cold 8 mm-OFHC copper bolt beneath the sample. The dashed circle at the right hand side of Ta clip indicates the current feeder beneath the clip.

has eight 3 mm-screw holes. The function of the eight 3 mm-screws is to make the mechanical contact between the sample holder and the sample stage which is fixed to the manipulator and connected to the cold-head by OFHC copper braid as shown in Fig. 1(b). Tightening these screws by the vacuum screw driver makes the top face of the sample holder stage (already inserted between the middle plate and the bottom plate) to be pushed to the bottom face of the second plate of the sample holder. Since both materials are made of soft OFHC copper blocks, this contact does not lose any thermal conductivity as long as both faces are well polished.

The second part of the sample holder consists of two Ta-plates which are electrically and thermally isolated from the copper part of the holder by the ceramic spacers. The wafer sample is clipped and held by these Ta-plates. The current can be fed through the 4 mm-diameter screw installed at the sample manipulator. The screw can be elevated and contacted with one of the Ta-plates as shown in Fig. 1(b). Another Ta-plate can be electrically connected with the outside current-probe coming from one of the flanges of the chamber. Disconnecting this outside current-probe, the sample can be biased through the sample-stage current feeder. Due to the restoring force of Ta-plate spring holding the sample, the sample can be evenly heated and any significant tension is not exerted on the clipped sample even for the rapid change of sample temperature.

The third and most functional part of the sample holder is the 8 mm-diameter OFHC bolt. The bolt can be raised in the 8 mm screw hole at the center of the sample holder by the vacuum screw driver. Even when the sample stage is at the lowest temperature, the sample can be resistively heated as long as this bolt is not in contact with the backside of the sample. For example when Si-wafer had been heated up to 1600 K for 30 sec, the maximum temperature-increase at the sample stage was less than 5 K. After preparation of the clean surface, the bolt is elevated to be in contact with the sample. At this point the Ta plate-springs softly push down from the top-side of the sample and the cold bolt pushes up from the back-side. As the sample stage is held near the minimum temperature (<20 K) during the annealing process, it takes less than 2 min. to reach the minimum equilibrium temperature.

In addition to the fast cooling efficiency, the pressure during the outgassing can be relatively low due to the thermal isolation of the sample and the cold part acting as a cryo-pump. Especially it is easy to decap the protective layer on the sample without re-contaminating the clean surface. By exposing the gases to the clean and cold surface, it has been found from the XPS spectra that Ar and O_2 had been physisorbed. This reflects that the sample temperature is lower than 20 K. In order to bias the cold sample, the sapphire plate can be mounted on the top of the 8 mm-bolt by In-welding. Even though the sapphire plate has good characteristics of electrical insulation and thermal conduction, it is found that O_2 can not be physisorbed but the multilayers of N_2O or Xe can be physisorbed. This implies that the sample holder can not be cooled to 20 K but cooled less than 50 K at the expense of biasing. The post sample to be cleaved also can be mounted on the similar sample holder after a minor modification. The post is inserted into the center-hole of the holder and the 3 mm bolts coming from the side push and hold the sample.

In Fig. 1(b) the schematic design of the sample holder engaged with the sample stage is viewed from the side. The flexible OFHC copper braid is connected to the cold head of the He-refrigerator. The length and the diameter of the braid are about 30 cm and 6 mm, respectively. Therefore the manipulator can have the freedom of translational and rotational motions. The tilt and X-Y-Z translational motions of the manipulator allow the precise positioning of the screw head to the vacuum screw driver. Actually the vacuum screw driver is the screw driver head fixed to the rotatable feed-through and the feed-through is mounted on the flex-coupling which can be tilted. The vacuum screw driver is used to rotate the current feeder, the 8 mm OFHC copper bolt from the bottom-center of the holder and 3 mm stainless steel screws at the bottom of holder. Also the high-current power feed-through is mounted to the same kind of the flex-coupling in order to contact with the Ta-clip. Ceramic insulator and spacers separate the cold part from the part where the current flows.

In Fig. 1(c) is shown the schematic top-view of the sample holder stage engaged with the sample holder. The large hole near the current feeder secures the space from the sample stage to prevent the accidental contact between them. The thermocouple to measure the low temperature is attached to the sample holder stage. It has been confirmed by the simultaneous temperature measurement that the temperature at the bottom of the sample differs from that at the position of the thermocouple less than 5 K when the 8 mm-OFHC copper bolt without the sapphire plate is attached to the sample.

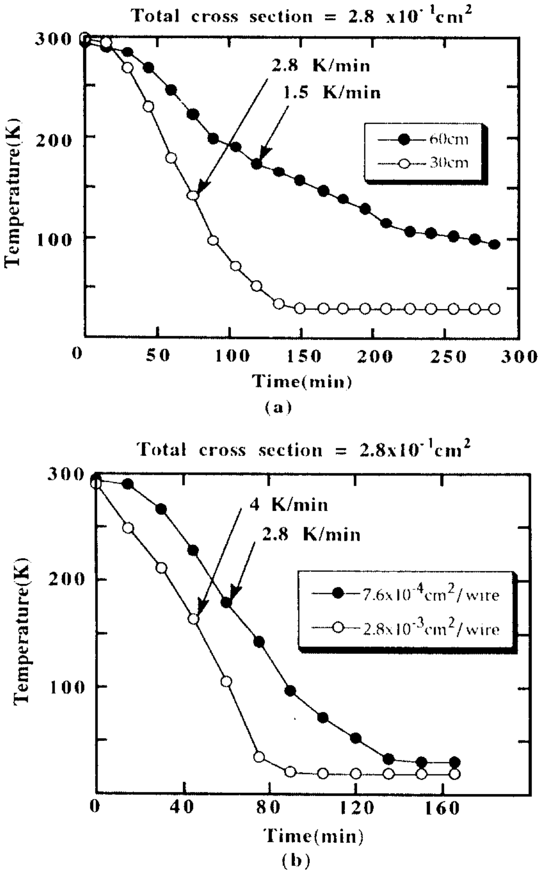

Fig. 2. Cooling rates for various braid shapes. Temperature is measured by Chromel-Alumel thermocouple attached to the sample holder stage and the time indicates the total lapse since the refrigerator is turned on. (a) Comparisons for the length of the braid. Both of braids have the common total-cross-section of 2.8×10^{-1} cm^2 (i.e., D=6 mm) and that of the consisting individual copper wire is 7.6×10^{-4} cm^2 (i.e., D=0.3 mm). The slope represents the cooling rate and it is inversely proportional to the length. The saturating temperature of the longer braid is about four times higher than that of the shorter one. (b) Comparisons for the same shape of braids except individual wire cross-section. The cooling rate of the thicker individual wire (i.e., 2.8×10^{-3} cm^2, D=0.6 mm) is about 1.4 times faster than the thinner wire (i.e., 7.6×10^{-4} cm^2, D=0.3 mm). Both of braids have the similar saturating temperature.

3. Flexible Motion and Better Thermal-Conductance

The total cross-sectional diameter of the OFHC copper braid used was 6 mm and it was inserted into the hole and bolted down by 5 mm-screws in order to make better contact with the sample stage. In Fig. 2, the comparative results of cooling rate for the different shapes of OFHC copper braid. It must be emphasized that the total period for cooling the sample stage of 300 K to the minimum temperature is about 2~3 hours depending upen the choice of OFHC copper braid. From Fig. 2(a), when the length of the braid with the same total cross-section (6 mm diameter) doubles, the cooling rate is slowed down by two times and the final saturation temperature is about 4 times higher than the shorter one. On the other hand, when the cross-section of the individual copper-wire is varied and the total length and cross-section of the braid are kept the same, the cooling rate of the thicker individual wire is about 1.4 times faster than that of the thinner one and the final equilibrium temperature is lower by 7 K. This result indicates that the heat flow in the individual copper wire of the OFHC copper braid is not viscous-like flow[2]. On the other hand the heat can be transmitted through the contacting wires, and the total cross-section and the total length are critical factors in the braid design. As a result, the shorter and thicker braid gives better thermal conductance at the sacrifice of the flexible motion.

4. Examples of Spectra from Si(111)-7×7

Finally in Fig. 3, the UPS spectra obtained from clean Si(111)-7×7 at 300 K and Si(111)-7×7 exposed by 5L (1L = 10^{-6} torr·sec) of Xe with −22 V bias at 40 K. The bottom density of states (DOS) of clean Si(111)-7×7 shows distinct surface states of well-reconstructed Si(111) surface as previously reported by UPS and Scanning tunneling microscopy (STM)[3, 4]. The wafer surface is not specially treated before inserting into UHV and the native oxide is simply decapped by resistive-annealing up to 1600 K while the He-refrigerator is turned on. But the 8 mm copper-bolt is separated from the sample during the annealing procedure. The subsequent cooling through the contact with the cold 8 mm copper bolt followed by Xe exposure to the surface results in multi-layers of Xe adsorption. The spin-orbit splitting of Xe 5p appears between 30 and 35 eV. Therefore the local work-function where Xe is adsorbed (so called, Photoemission of Adsorbed Xenon, PAX) can be probed using the characteristics of substrate vacuum-level alignment of physisorbed Xe[5]. Also the average work-function of the surface is the difference between the photon energy, 21.2 eV, and the width of DOS.

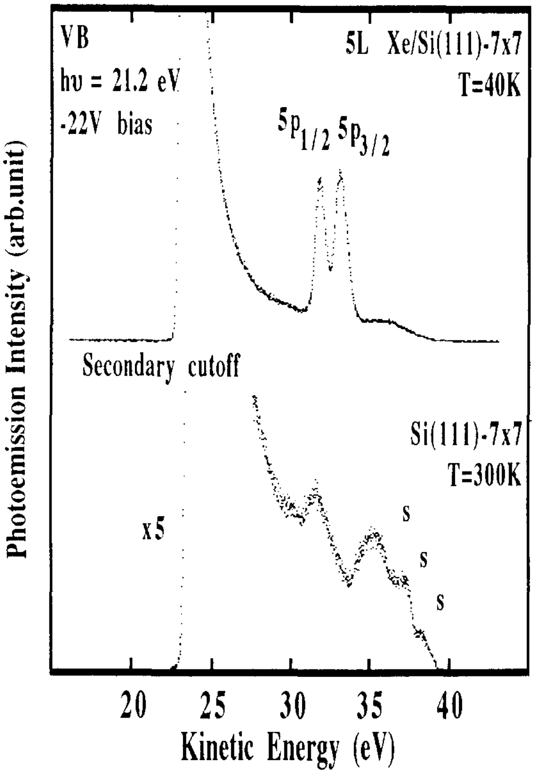

Fig. 3. Density of states for the valence bands obtained from Si(111)-7×7 mounted on the sample holder using ultraviolet photoelectron spectroscopy. Both of samples are biased with −22 V, hence the same amount of kinectic energy gain should be considered. The bottom one is obtained just after decapping the native oxide layer. Distinct surface states designated by S implies the surface is very well reconstructed without recontamination. The top one is obtained just after exposing 5L Xe to the cold and clean 7×7 surface. The position of Xe 5p levels has information on the local work-function at the site where Xe is adsorbed.

5. Concluding Remarks

Although the transfer-mechanism and the proper contact of OFHC Cu-braid with the sample holder stage are fundamental concepts of the design, the simple and movable 8 mm copper bolt circumvents the problems of resistive annealing and conductive cooling for the studies of wafer samples at the low temperature. The design will help to investigate the phase transition of adsorbed gases on the reconstructed surface and the non-thermal activation of reactive gas on the surface.

Acknowledgement

This work was supported by Korea Science and Engineering Foundation (KOSEF) through the Semiconductor Physics Research Center (SPRC) and we would like to thank Lucky metal corporation of R. O. Korea for supplying OFHC copper block.

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