

EQUIPMENT
DEVELOPMENT

CONSTRUCTION OF AN APPARATUS FOR PHOTOCHEMICAL
SURFACE REACTION STUDIES

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Photo-assisted surface reactions on solid semiconductor materials have attracted considerable attention from the viewpoint of their applications to semiconductor processes. Much progress has been made using visible and UV light. Natural extension of these studies is to use vacuum UV light. However nothing is known about what would happen on solid surface when VUV light is irradiated upon it in a reactive gas atmosphere. We have thus constructed an apparatus to study photochemical surface reactions using synchrotron radiation as a light source.

Figure 1 is a schematic of the apparatus installed on beam line BL8A. It consists of two chambers, a reaction chamber(RC) and a differential pumping chamber (DPC), which are connected to each other via a channel (6mm diameter X 80mm long) (C). The RC is pumped by a 330 l/sec turbomolecular pump (TP) and the DPC by a 600 l/sec TP and a liq. N₂ cryopump (CP) as well. A thin Ti foil (T) fitted between the DPC and the beam line chamber serves both as a filter to select the wavelength range of the irradiated light and as a window to prevent reactive gas from going into the beam line.

Silicon samples (kept at ~ 27°C) (S) are held on a sample manipulator (SM) in the reaction chamber, the base pressure of which is 1.0×10^{-9} Torr. After the gate valve GV-1 is closed, Cl₂ gas is constantly flowed upon the solid surface into the reaction chamber through a multichannel capillary array. The Cl₂ pressure in the reaction chamber ranged from 0.05 to 0.5 Torr and was controlled by a needle valve in the gas inlet line plus a choke valve between the RC and chlorine trap (T), which consists of a liq. N₂ cryopump followed by a chemical vacuum pump.

The UVSOR synchrotron is operated at 750 MeV beam energy. A Ti filter 500 Å thick which is transparent mostly in the extreme vacuum region 1-20 nm was used. When the filtered SR is admitted

spectrum of the glow was recorded in the visible and UV region as shown in Fig. 2. The strong lines are identified as Cl^+ transitions. Since the formation of Cl^+ ions was identified, a rectangularly netted Ni mesh (M) with 64% transmittance was positioned 0.6 cm upstream from the Si sample. The Ni mesh was biased up to ± 75 volts to observe the effect of an applied electric field on the Si etch rate.

From the absorption spectra of metallic Ti and the SR operated at 750 MeV, we have estimated the total number of photons irradiated upon the heavily phosphorous doped polycrystalline silicon (n^+ poly-silicon) surface to be 5.2×10^{13} photons $\text{s}^{-1} \text{cm}^{-2}$ per mA stored current and the average photon energy to be 322 eV. Etching experiments were normally done at a Cl_2 pressure of 0.3 Torr. The quantum yield for the removal of Si atoms was found to be 0.5 % when the Ni mesh was kept at ground, and that to be 1.0 % when the Ni mesh was positively biased at 75 volts, providing with a $900 \pm 100 \text{ \AA}$ etch depth for 230 mA hr irradiation, as illustrated in the abstract of 28th Okazaki Conference of the present issue.

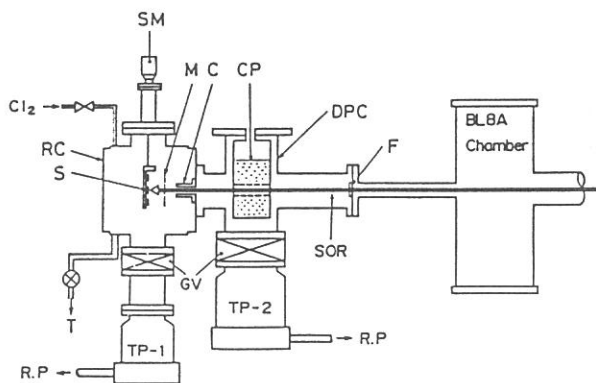


Fig. 1. Apparatus used for photochemical surface reaction studies. See the text for explanations except for R.P.: rotary pump; C: channel.

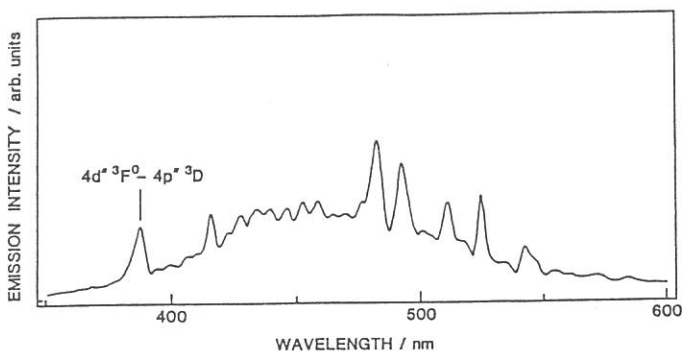


Fig. 2. Emission spectrum in the wavelength region 350-600 nm of the fluorescence from the light path of the SR (monochromator resolution: 3 nm).

ANGLE-RESOLVED UPS SYSTEM AT BL8B2

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An angle-resolving UPS system was constructed at BL8B2. The setup is shown in Fig. 1. Synchrotron radiation from UVSOR is monochromatized by the previously reported plane-grating monochromator¹⁾ which supplies radiation in the energy range of 2 - 150 eV. The photoelectron spectrometer consists of a preparation chamber, a measurement chamber, and a transfer system. Each chamber is evacuated by a combination of an sputter ion pump, a turbomolecular pump, and a Ti getter pump, with a final pressure of 10^{-10} Torr range. A hemispherical electron-energy analyzer of 25 mm mean radius can be rotated around vertical and horizontal axes. The sample mounted on a

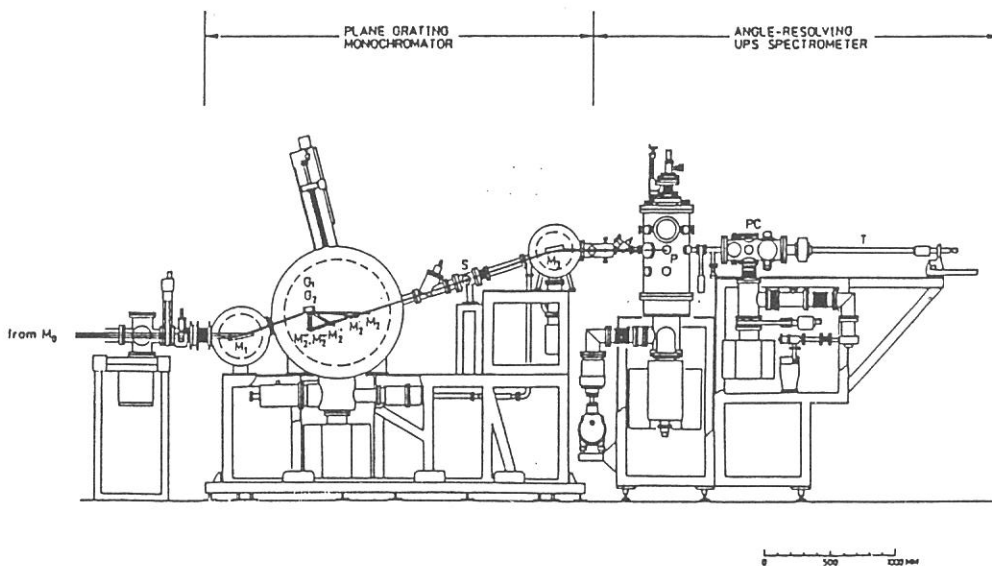


Fig. 1. Angle-resolving UPS system.

manipulator can be also rotated around two axes. The system is designed primarily for measuring various organic solids such as molecular crystals, polymers, and graphite intercalation compounds. The samples are prepared on a small disk of 12 mm in diameter by methods such as vacuum evaporation and cleavage. The total resolution was 0.2 eV, as determined by measuring the Fermi edge of gold. In Fig. 2, we show a test spectrum of RbCl taken at various photon energies. Now the electronic structures of $n\text{-CH}_3(\text{CH}_2)_{34}\text{CH}_3$ and poly(phenylene sulfide) is being measured, the former being reported in another article of this report.

Reference

1) K. Seki, H. Nakagawa, K. Fukui, E. Ishiguro, R. Kato, T. Mori, K. Sakai, and M. Watanabe, UVSOR Activity Report 1984/85 p.35; Nucl. Instrum. Methods Phys. Res., A246, 264 (1986).

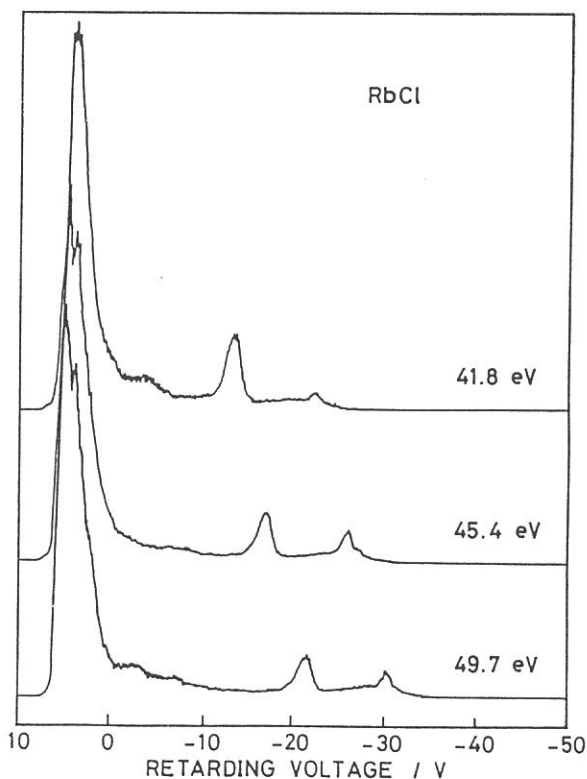


Fig. 2. Normal emission spectra of RbCl in retarding potential scale. The kinetic energy of photoelectrons increase from left to the right.