

RESEARCH ACTIVITIES VI

Department of Vacuum UV Photoscience

VI-A Electronic Structure and Decay Mechanism of Inner-Shell Excited Molecules

In this project, we have two major subjects: (a) resonant photoelectron spectroscopy and (b) resonant inelastic soft X-ray emission spectroscopy following inner-shell excitations of simple molecules and clusters. We have succeeded in development of a next-generation soft X-ray emission spectrometer. In the spectral assignments, angle(symmetry)-resolved photoion yield techniques and R-matrix/MQDT theoretical approaches are essential.

VI-A-1 Development of a Transmission-Grating Spectrometer for Soft-X-Ray Emission Studies

HATSUI, Takaki; HORIGOME, Toshio; KOSUGI, Nobuhiro

Last year we reported our new design of a transmission-grating spectrometer (TGS) for high resolution soft X-ray emission studies. This spectrometer will realize a resolution $E/\Delta E$ up to 5000 in the energy region of 50–600 eV. Here we report the first results using a prototype setup. The spectrometer has a Wolter type I mirror, a free-standing transmission grating, and a back-illuminated charge coupled device (CCD). A high collection angle up to 1.5×10^{-3} sr is achieved by introducing the Wolter mirror as a prefocusing system. The developed Wolter mirror¹⁾ shows a slope error less than 0.4 arcsec. A SiC transmission grating with a groove density of 6250 l/mm is also developed.²⁾ The CCD is mounted at 1.5 m downstream of the grating on a Rowland torus mount with a radius of 0.75 m. Diffracted x-rays are detected in the normal incidence geometry, resulting in high detection efficiency. Ray-trace simulations indicate that aberrations do not practically degrade the energy resolution as high as 5000. It is limited by the figure errors of the optical elements and the spatial resolution of the detector. The energy resolution of TGS was examined by measuring the diffuse scattering of a tungsten wire with a diameter of 3 μm illuminated. Figure 1 illustrates a line profile along dispersion direction of the CCD detector image. The results indicate that the energy resolving power is above 3100 at 110 eV. Further evaluation of the performance by introducing the entrance slit is now under progress.

References

- 1) In collaboration with A. Ohba *et al.* (Hamamatsu Photonics K.K.).
- 2) Manufactured by NTT-ATN.

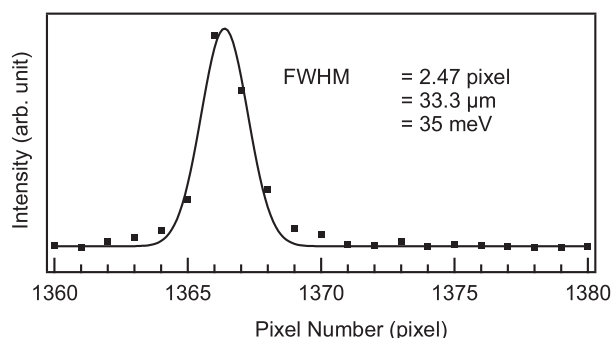


Figure 1. Line profile along dispersion direction of the CCD detector image. Full width at half maximum of 33.3 μm , which corresponds to energy resolution of 35 meV ($E/\Delta E = 3100$), has been obtained.

VI-A-2 Application of R Matrix/MQDT Method to both Valence and Core Excitations in NO

HIYAMA, Miyabi; KOSUGI, Nobuhiro

Both valence and core excitations of NO molecule are studied using the R matrix/Multi-channel Quantum Defect Theory¹⁾ program code, GSCF4R.²⁾ In the case of valence excitation, the quantum defects and the coupling constant between $\pi\pi$ -Rydberg state and $^2\Pi$ state of NO were calculated. The theoretical quantum defects for $^2\Sigma$, $^2\Pi$, and $^2\Delta$ are in good agreement with the experimental values. The theoretical coupling constants not only for B($2^2\Pi$) but also for L($3^2\Pi$) are also in good agreement with the experimental values. In the case of core excitation, the potential curves above the N 1s ionization threshold of NO were calculated. There may be six potential energy curves corresponding to the resonances around 412 eV. Finally, the twin local maxima near 412 eV in the absorption spectra of NO may be assigned to the resonances whose characters are σ^* excitation and the mixture of σ^{*-} and doubly excitations, respectively.

References

- 1) M. Hiyama and M. S. Child, *J. Phys. B* **35**, 1337–1351 (2002); **36**, 4547–4559 (2003).
- 2) M. Hiyama and N. Kosugi, *J. Theor. Comput. Chem.* **4**, 35–47 (2005).

VI-B Soft X-Ray Photoelectron-Photoabsorption Spectroscopy and Electronic Structure of Molecular Solids and Clusters

This project has been carried out in collaboration with Wuerzburg University. We have two subprojects: (a) molecules and radicals in condensed phase and in rare gas matrix, and (b) ionic fragmentations of atomic and molecular clusters following the inner-shell resonance excitation. In (a), we have measured excitation spectra in some matrix phases at the bending-magnet beamline BL4B of the UVSOR facility. In (b), we have developed a new cluster source for photoelectron measurements on a newly constructed undulator beamline BL3U.

VI-B-1 Photoionization of Small Krypton Clusters in the Kr 3d Regime: Evidence for Site-Specific Photoemission

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[*J. Chem. Phys.* in press]

Kr 3d ionization energies of small, variable size krypton clusters are investigated by photoelectron spectroscopy, where the size regime of clusters with an average size $\langle N \rangle \leq 30$ is studied. Characteristic shifts in Kr 3d ionization energies to lower binding energies are found compared to the bare atom. These are also different from those of large krypton clusters. Moreover, we find evidence for photoionization of the krypton dimer. Its 3d ionization energy is barely shifted relative to the atomic value. Results from model calculations considering different isomers and cluster sizes as well as defect sites give evidence that the experimental results can be related to photoionization from different surface sites in variable size krypton clusters. This can be related to site-specific photoemission in small Kr clusters. The results are compared to size effects in Kr 3d near-edge features of variable size Kr clusters as well as recent results on Kr 3d photoionization of large Kr clusters.

ment carry information about the molecular orientation relative to the electromagnetic field vector at the moment of excitation. The data together with analytical functions for the electron-peak profiles give clear evidence that the preferred molecular orientation for excitation only depends on the orientation of one bond, not on the total molecular orientation. The localization of the valence orbital “7a₁” is caused by mixing of the valence orbital “5b₂” through vibronic coupling of anti-symmetric stretching mode with b₂-symmetry. To the best of our knowledge, it is the first discussion of the localization of a core excitation of O₃. This result explains the success of the widely used assumption of localized core excitation in adsorbates and large molecules.

VI-B-2 Core Excitation in O₃ Localized to One of Two Symmetry-Equivalent Chemical Bonds —Molecular Alignment through Vibronic Coupling

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[*J. Chem. Phys.* **122**, 154303 (2005)]

Core excitation from terminal oxygen O_T in O₃ is shown to be an excitation from a localized core orbital to a localized valence orbital. The valence orbital is localized to one of the two equivalent chemical bonds. We experimentally demonstrate this with the Auger Doppler effect which is observable when O₃ is core-excited to the highly dissociative O_T 1s⁻¹7a₁⁺¹ state. Auger electrons emitted from the atomic oxygen frag-

VI-C Ultrafast Dynamics of Molecules in Intense Laser Fields

The behavior of molecules in an intense laser field (10^{12} – 10^{18} W/cm²) has been an attractive target of research for a deeper understanding of the light-matter interaction. In the present study, the characteristic nuclear dynamics occurring in intense laser fields, such as structural deformation and multiple breaking of chemical bonds, has been studied by a newly developed experimental method, called *coincidence momentum imaging* (CMI), which allows us to determine the momentum vectors of all the fragment ions ejected from a single parent molecule. By using the CMI technique combined with the pump-and-probe scheme, the real-time probing of the nuclear wavepacket evolution in the three-dimensional internal coordinate space is demonstrated. Based on this novel technique, it has been found that CS₂²⁺ formed in intense laser fields ($\sim 1.3 \times 10^{14}$ W/cm²) undergoes ultrafast dissociation along the symmetric stretching coordinate leading to simultaneous breaking of the two C–S bonds. A high temporal resolution pump-probe CMI measurement is under progress with sub-10 fs intense laser pulses.

VI-C-1 Probing the Ultrafast Nuclear Motion in CS₂²⁺ in Intense Laser Fields

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[*J. Chem. Phys.* **122**, 151104 (4 pages) (2005)]

The nuclear dynamics of CS₂ exposed to an intense laser field (60 fs, 1.3×10^{14} W/cm²) is studied by the pump-probe coincidence momentum imaging of the Coulomb explosion process, CS₂³⁺ → S⁺ + C⁺ + S⁺. From the dependence of momentum correlations among the fragment ions on the time delay between pump and probe pulses, the existence of the dissociation pathway along the symmetric stretching coordinate leading to concerted breaking of the two C–S bonds is identified in addition to the dissociation along the anti-symmetric stretching leading to S⁺ + CS⁺. It is also shown that the S–C–S bending motion is largely excited when both of the two C–S bonds stretch by the coupling of two different light-dressed states.

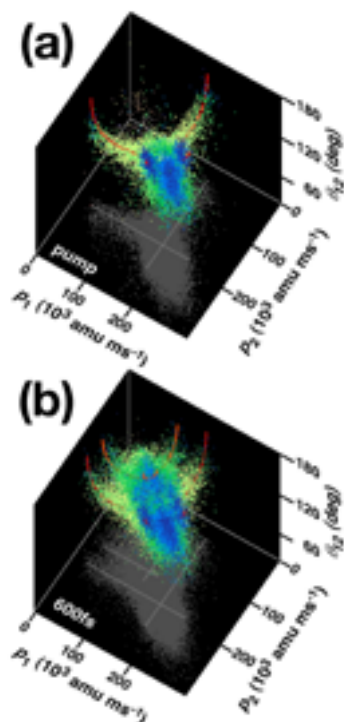


Figure 1. (a) Three-dimensional momentum correlation map for the Coulomb explosion process, CS₂³⁺ → S⁺ + C⁺ + S⁺, obtained only with a pump pulse (1.3×10^{14} W/cm², 60 fs, 800 nm), where θ_{12} represents the angle between the two momentum vectors $\mathbf{p}_1(\text{S}^+)$ and $\mathbf{p}_2(\text{S}^+)$ of the resultant S⁺ ions, and $p_1 = |\mathbf{p}_1(\text{S}^+)|$ and $p_2 = |\mathbf{p}_2(\text{S}^+)|$. Solid lines represent the result of the simulation based on the classical free-rotor model. (b) Momentum correlation map at a time delay of $\Delta t = 600$ fs, exhibiting new features with a pair of wings in the low momentum region ($p_1, p_2 < 150 \times 10^3$ amu m/s).

VI-C-2 Concerted and Sequential Coulomb Explosion Processes of N₂O in Intense Laser Fields by Coincidence Momentum Imaging

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[*J. Chem. Phys.* **123**, 154305 (8 pages) (2005)]

The Coulomb explosion dynamics of N₂O in intense laser fields (800 nm, 60 fs, $\sim 1.6 \times 10^{14}$ W/cm²) is studied by the coincidence momentum imaging method. From the momentum correlation maps obtained for the three-body fragmentation pathway, N₂O³⁺ → N⁺ + N⁺ + O⁺, the ultrafast structural deformation dynamics of N₂O prior to the Coulomb explosion is extracted. It is revealed that the internuclear N–N and N–O distances stretch simultaneously as the bond angle $\angle \text{N–N–O}$ decreases. In addition, two curved thin distributions are identified in the momentum correlation maps, and are interpreted well as those originating from the sequential dissociation pathway, N₂O³⁺ → N⁺ + NO²⁺ → N⁺ + N⁺ + O⁺.

VI-C-3 Development of an Intense Sub-10fs Laser Source with a Hollow Fiber/Chirped Mirror Compressor

TAKAHASHI, Eiji J.; HISHIKAWA, Akiyoshi

An intense ultrashort laser source was designed and developed to study the Coulomb explosion dynamics of molecules in a sub-10fs intense laser field. The output from a Ti:Sapphire laser system (800 nm, <40 fs, 1 kHz) was introduced to a hollow fiber placed on a V-shape block in a cell, filled with Ar at a pressure of ~ 0.1 MPa. During the propagation of the laser through the fiber, the

spectral bandwidth was increased up to by the self-phase modulation effect (Figure 1(a)). The output from the hollow fiber was then collimated by a concave mirror and compressed by a pair of chirped mirrors. The pulse duration after the compression was measured to be 9.0 fs from the interferometric autocorrelation trace (Figure 1(b)). The output energy from the pulse compression system exceeds 0.4 mJ/pulse, which is sufficient to generate a field intensity of $\sim 10^{16}$ W/cm² with an F/5 focusing optics.

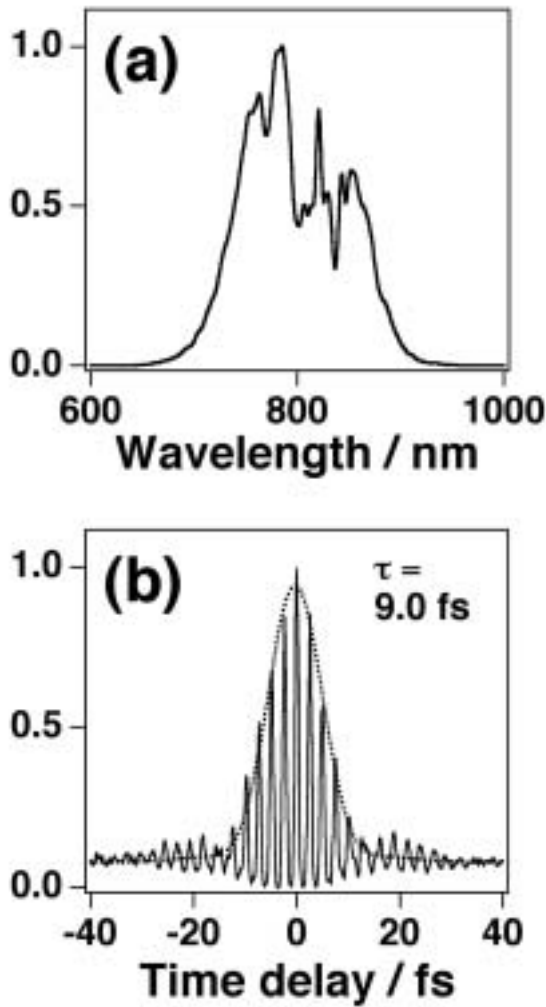


Figure 1. (a) The laser spectrum after the propagation through the hollow fiber. (b) The autocorrelation trace of the laser pulse after the compression by the chirped mirrors. The autocorrelation intensity profile obtained by Fourier transform of the spectrum (a) is plotted with a dotted curve. The pulse duration is determined to be 9.0 fs.

VI-D Synchrotron Radiation Stimulated Surface Reaction and Nanoscience

Synchrotron radiation (SR) stimulated process (etching, CVD) has excellent characteristics of unique material selectivity, low damage, low contamination, high spatial resolution and high precision, *etc.* In this project, nanolevel controlled structures are created using the synchrotron radiation stimulated process, and the reaction mechanisms are investigated by scanning tunneling microscopy and atomic force microscopy. Concerning the SR etching, we are considering to apply this technique to the microfabrication of integrated protein transistor circuits. Electric property measurement of a supported lipid bilayer membrane was achieved using a $\text{SiO}_2/\text{AgCl}/\text{Si}$ device which was fabricated by synchrotron irradiation etching.

VI-D-1 Synchrotron Radiation Induced Si-H Dissociation on H-Si(111)-1x1 Surfaces Studied by In-Situ Monitoring in the Undulator-STM System

NONOGAKI, Youichi; URISU, Tsuneo

[*J. Vac. Sci. Technol.*, A 23, 1364–1366 (2005)]

Irradiation effects of the synchrotron radiation (SR) have been investigated on the hydrogen terminated- (H-) Si (111) surfaces by using the undulator beam and the in-situ scanning tunneling microscope (STM). The small protrusions (SPs) generated by the undulator beam irradiation were assigned to the rest-atoms with missing H (Figure 1). From the observed relation among the SP density, photon energy of the undulator beam and the total photon flux, it has been concluded that the main mechanism of the Si-H bond dissociation by the undulator beam irradiation is valence electron excitations of the Si-H bond by incident photons.

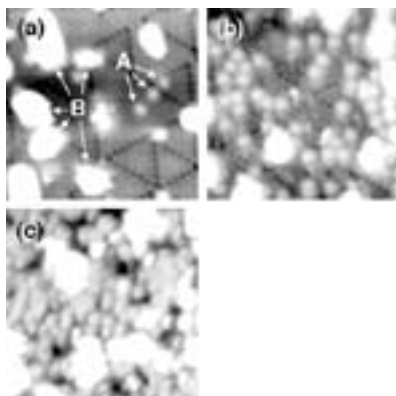


Figure 1. 10 nm × 10 nm STM images of H-Si(111) surfaces after the undulator beam irradiations with the exposure of (a) 0 mAs, (b) 5,000 mAs and (c) 10,000 mAs. The undulator gap height was fixed at 20 mm. In (a), small protrusions (SPs) and adatom islands (AIs) are indicated by A and B, respectively. It is observed that the SP density significantly increases with increasing irradiation dose. The appeared SPs are assigned to the rest-atoms with missing H.

VI-D-2 Giant Vesicle Fusion on the Microelectrodes Fabricated by Femto-Second Laser Ablation Followed by Synchrotron Radiation Etching

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[*Jpn. J. Appl. Phys.* in press]

We have developed a new technique to fabricate the hole (well) with about 1 μm diameter for the microelectrode on the surface of $\text{SiO}_2(600\text{ nm})/\text{CoSi}_2(10\text{ nm})/\text{Si}$ using synchrotron radiation (SR) stimulated etching. The Co photomask on the substrate was patterned by a femto-second-laser, and the SiO_2 layer was SR-etched. This process enabled the fabrication of the electrode holes with keeping the original nano-level flatness ($R_a \sim 0.8\text{ nm}$) of the SiO_2 surface. The lipid bilayer was formed by the giant vesicle fusion on these microelectrodes. Fluorescence microscopy, *in situ* AFM and electrical characteristics measurements showed that a single lipid bilayer with sufficiently high resistance (giga ohm seal) was successfully fabricated (Figure 1).

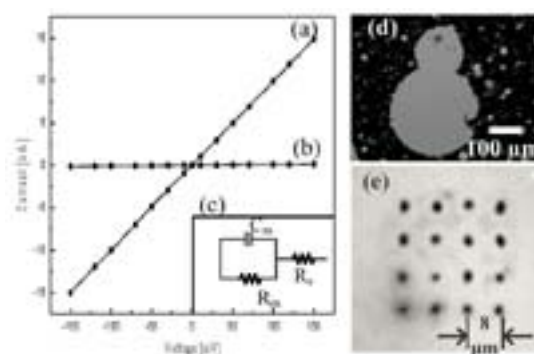


Figure 1. Current-voltage characteristics of the substrate measured under the KCl solution (10 mM), (a) before (■) and (b) after (●) the SPLB formation, and (c) the equivalent circuit of the system. The fluorescence microscopy image of the lipid bilayer formed by the rupture of the giant vesicle on (d) $\text{SiO}_2/\text{CoSi}_2/\text{Si}$ surface and (e) the electrode area.

VI-E Noble Semiconductor Surface Vibration Spectroscopy

As a new high sensitive and high resolution surface vibration spectroscopy technique, we are developing an infrared reflection absorption spectroscopy using buried metal layer substrate (BML-IRRAS), which have unique characteristics of high resolution and high sensitivity at finger print regions. Several Si surface chemical reactions are investigated using this BML-IRRAS. As a new fabrication technique of BML substrate, we have almost succeeded in developing the wafer bonding technique. It is considered that BML-IRRAS is also extremely useful in the research of bio-material integration on Si substrates.

VI-E-1 Orientation of Avidin Molecules Immobilized on the COOH-Modified SiO₂/Si(100) Surface

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Avidin molecules were immobilized on COOH-modified SiO₂/Si(100) surfaces with subnano-level flatness ($R_a < 0.1$ nm) forming covalent bonds between COOH groups on the substrate surface and NH₂ groups of the avidin molecules. The avidin-immobilized surfaces were characterized by atomic force microscopy (AFM), infrared reflection absorption spectroscopy using buried metal layer substrate (BML-IRRAS), transmission infrared absorption spectroscopy (TIRAS), and ellipsometry. BML-IRRAS and TIRAS have the sensitivities to the perpendicular and the parallel (to the substrate surface) components of the dynamic dipole moment, respectively. In the β -sheet, the amide I transition dipole moment is oriented parallel to the C=O bond, which is oriented perpendicular to the axis of the β -strand which forms a β -barrel in avidin. Therefore, considering the molecular orientation dependence of the extended β -strand (1637 cm⁻¹) peak intensity in avidin, it will be a minimum in BML-IRRAS and a maximum in TIRAS. It is concluded from these data that the avidin molecules are immobilized with the symmetry axis of the tetramer almost perpendicular to the substrate surface.

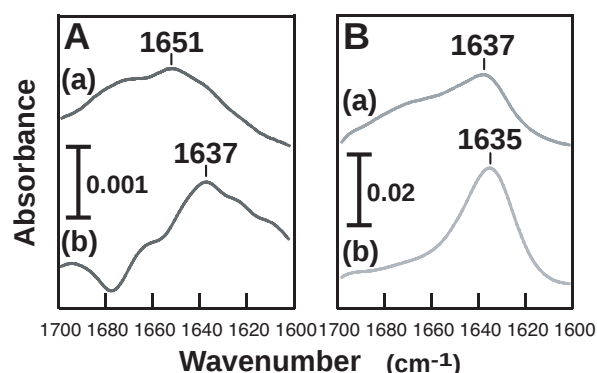


Figure 1. IR spectra of avidin molecules (A) covalently immobilized and (B) physisorbed on SiO₂/Si surfaces. The spectra (a) and (b) are measured by BML-IRRAS and TIRAS, respectively.

VI-E-2 Hydrogen-Atom-Induced Oxidation Reaction on Water-Terminated Si Surface, 2H+H₂O/Si(100)-(2x1): A Theoretical Study

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(¹Kyushu Univ.; ²Nagoya Univ.; ³AIST)

[Chem. Phys. Lett. **412**, 347–352 (2005)]

The reported oxidation reaction observed by BML-IRRAS spectra on the silicon surface system, 2H + H₂O/Si(100) (Figure 1), has been studied by an *ab initio* molecular orbital method. The highest transition state is found at $\approx +25$ kJ/mol from the reactant energy level, and the oxidation occurs easily under the experimental condition (Figure 2). The present study also accounts for the reactivity deduced from the absorption bands in the IR spectra. It is noted that the quenching of the reaction by thermal relaxation is impossible because the surface is not trapped into the metastable states located much lower in energy than the reactant.

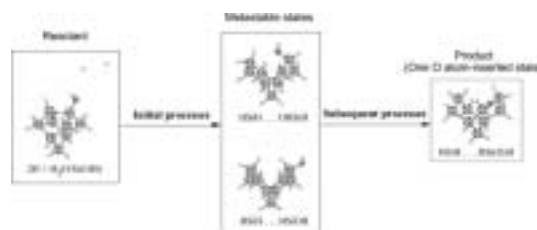


Figure 1. Reaction scheme of the H atom-induced oxidation on the H₂O-terminated Si surface system 2H + H₂O/Si(100)-(2x1), expressed by the actual conformation of the cluster model of the present work.

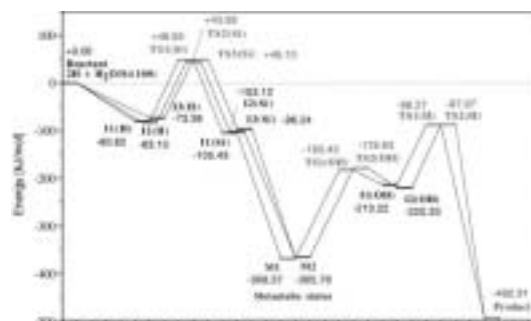


Figure 2. Energy diagram of the H atom-induced oxidation on the H₂O-terminated Si surface system. The energies (in kJ/mol) are calculated with the HF/6-31+G* method corrected for the ZPV.

VI-F Integration and Characterization of Bio-Functional Materials on Silicon Surfaces

Integration of bio-functional materials on solid surfaces is an attractive research theme and important to the development of new biosensors and screening methods in which biological reactions are directly detected on electronic circuits. We have investigated the covalent immobilization of protein, lipid bilayer membrane deposition and have characterized the characteristics of these bio-functional materials using infrared absorption spectroscopy and atomic force microscopy. We also have developed a new compact fluorescence recovery after photobleaching apparatus using a semiconductor laser, which wavelength is tuned to the absorption maximum of the fluorescence dye molecules.

VI-F-1 Fabrication of Avidin Single Molecular Layer on Silicon Oxide Surfaces and Formation of Tethered Lipid Bilayer Membranes

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(¹IMS and Kyushu Univ.)

[*e-J. Surf. Sci. Nanotech.* **3**, 237–243 (2005)]

Single molecular layer of avidin is fabricated on an atomically flat SiO₂ surface and characterized by atomic force microscopy (AFM) and infrared reflection absorption spectroscopy. Immobilization of avidin is performed as follows; i) ester-modification of the surface by silane-coupling agent, ii) carboxylation by hydrolysis in HCl and iii) amide bonding between the surface -COOH and -NH₂ in avidin molecules. Large dome structures (~60 nm height) are formed after the ester-modification, but an atomically flat surface is obtained after the hydrolysis reaction. AFM topographs and function-recognizing images (Figure 1) show that the each of avidin molecules adsorbs as a single molecule and retains the biotin-binding activity. Formation of a tethered bilayer membrane of a biotinylated phospholipid on the avidin layer is also described.

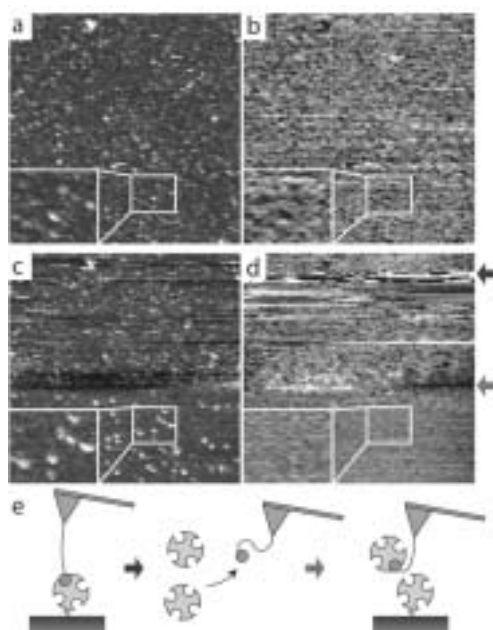


Figure 1. (a) AFM topograph ($1.5 \times 1.5 \mu\text{m}^2$) and (b) function-recognizing image of the avidin on the SiO₂ surface obtained by biotinylated cantilever. (c, d) AFM topograph and function-recognizing image continuously observed after those in (a) and (b). Avidin solution was injected to the liquid phase (white arrow) and reached to the cantilever (black arrow). (e) Schematic illustration of the blocking experiment performed in (c) and (d). The size of the magnified images is $0.28 \times 0.24 \mu\text{m}^2$. All images are obtained in the buffer solution.

VI-F-2 Deposition of Lipid Bilayers on OH-Density-Controlled Silicon Dioxide Surfaces

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(¹Okazaki Inst. Integrative Biosci.)

[*J. Vac. Sci. Technol., A* **23**, 751–754 (2005)]

We investigated the effect of the SiO₂ surface hydrophilicity on the formation of lipid bilayer membranes using the vesicle fusion method with atomic force microscopy, and applied the results to constructing membrane arrays. We obtained SiO₂ surfaces with different hydrophilicity by annealing chemically oxidized SiO₂ surfaces at various temperatures under an N₂ flow. The membrane formation rate is faster on less hydrophilic surfaces after depositing a 100-nm-filtered vesicle of dimyristoylphosphatidylcholine. Desorption of the surface hydroxyl groups causes a higher affinity between the lipid membrane and the substrate. We also describe a new method to fabricate membrane arrays using “chemical patterning.” When the surface hydroxyl groups are locally removed using a focused ion beam, a bilayer membrane selectively forms on the FIB-patterned region (Figure 1).

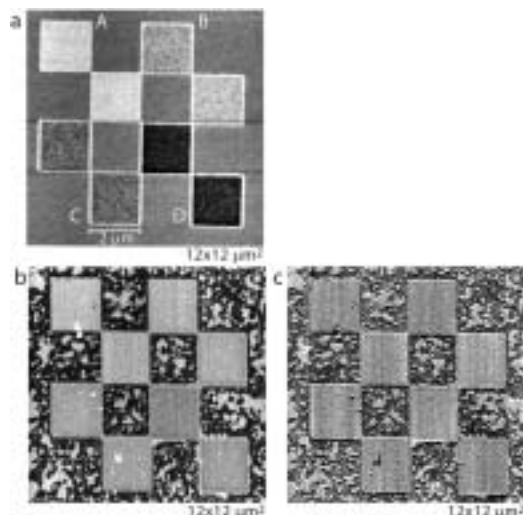


Figure 1. (a) AFM image ($12 \times 12 \mu\text{m}^2$) and line profiles of $2 \mu\text{m}$ square patterns made by FIB on the chemically oxidized SiO_2 surface, observed in air. The Ga^+ ion dosages in areas A–D were 0.5, 1.5, 3 and $6 (\times 10^{15} \text{ atoms per cm}^2)$, respectively. (b) AFM image ($12 \times 12 \mu\text{m}^2$) and line profiles of the FIB-pattern (a) after deposition of DMPC bilayers, observed in a buffer solution. (c) Phase image ($12 \times 12 \mu\text{m}^2$) obtained simultaneously with (b).

VI-F-3 Supported Lipid Bilayer Formation by the Vesicle Fusion Induced by the Vesicle-Surface Electrostatic Attractive Interaction

KIM, Yong-Hoon; RAHMAN, Md. Mashuur; ZHANG, Zhen-Long; TERO, Ryugo; URISU, Tsuneo

The effect of the electrostatic attractive force between vesicles and the substrate surface on Ca^{2+} free supported lipid bilayer formation has been investigated by using atomic force microscopy and fluorescence microscopy. When negative-charged giant vesicles were incubated without Ca^{2+} , surface coverage of lipid bilayer was extremely low on the SiO_2 surface (Figure 1a). It is well-known that the addition of Ca^{2+} induces the transformation from vesicle to the planar bilayer membrane on the solid surfaces (Figure 1b). On the other hand, in case of the positive-charged surface modified by aminopropyltrimethoxysilane, the high coverage of the lipid bilayer was obtained without adding Ca^{2+} (Figure 1c). The attractive force between the negative-charged giant vesicles and the positive-charged surface is essentially useful to induce the vesicle fusion without addition of Ca^{2+} .

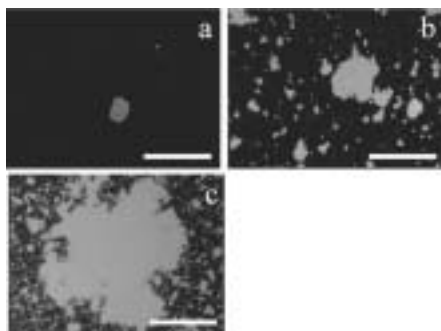


Figure 1. Fluorescence images of the lipid bilayer domain obtained under buffer solution: a) no addition of Ca^{2+} on the SiO_2 surface, b) addition of Ca^{2+} on the SiO_2 surface, and c) no addition of Ca^{2+} on the positive-charged surface. Scale bar is $50 \mu\text{m}$.

VI-F-4 The Current Noise Characteristic of a Single Ion Channel

UNO, Hidetaka; ZHANG, Zhen-Long; NAKAI, Naohito; TERO, Ryugo; NONOGAKI, Youichi; URISU, Tsuneo

We are constructing an ion channel current measurement system using a patch clamp amplifier to investigate the lipid membrane formation, the protein reconstruction and the electrical property of membranes and proteins. The analysis of a membrane protein interaction is an important subject in the post-genome, and the development of highly efficient biosensors is demanded. We have designed a Si-based micro pore chip for ion channel current measurement (Figure 1). Teflon sheets have been generally used to make the pore, but the Si-based chip will achieve a high-speed and highly integrated biosensor.

The pore size is one of the important factors of the ion channel current measurement. The dependence of the current noise on the pore size was measured using pores with different sizes on Teflon films. The noise level single ion channel of gramicidin A in a diphytanoylphosphatidylcholine black membrane was measured in different bandwidths (Figure 2). The current noise increased with the pore size. This result shows that micronization of the pore size effectively reduce the noise level. Fabrication of Si-based chips to achieve the micron-order pore using micro-fabrication techniques is now under way.

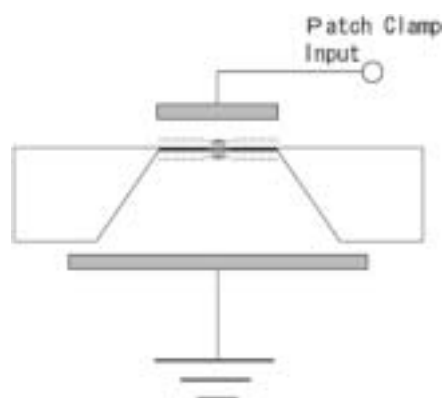


Figure 1. Schematic drawing of the Si-based micro pore chip for ion channel current measurement.

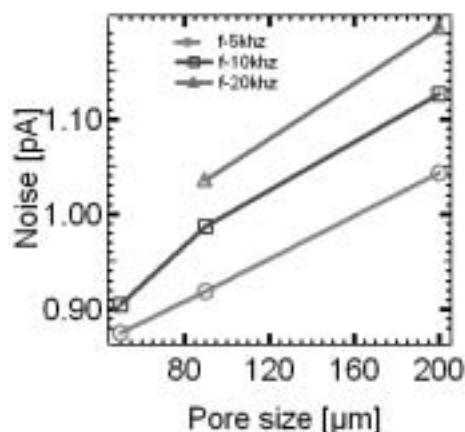


Figure 2. Channel current noise dependence on the pore size.

VI-F-5 A New Type of Fluorescence Recovery After Photobleaching Apparatus Using both Illumination Arrangements of UV Lamp and 560 nm Laser

ZHANG, Zhen-Long; MO, Yu-Jun¹; TERO, Ryugo; AOYAMA, Masaki; YOSHIDA, Hisashi; URISU, Tsuneo
(¹IMS and Henan Univ.)

A new fluorescence recovery after photobleaching (FRAP) setup was fabricated by which the FRAP curve could be directly obtained. Figure 1 shows the FRAP setup. There are two excitation light sources in the apparatus. One is an UV lamp. The other light source is a solid state laser with a wavelength of 560 nm for photobleaching and recording the fluorescence recovery process by a photomultiplier tube (PMT). In order to avoid photobleaching during the fluorescence recovery process and improve the ratio of signal to noise, a pulse-voltage generator was designed to control the laser. The pulse induced by the generator consists of two parts. Part I is continuous for photobleaching. And part II is periodic, in which the pulse width and period are 0.01 s and 1 s, respectively. The height and duration time of the two parts could be adjusted respectively according to varied samples. Two recording devices are equipped in the setup. One is a charge coupled device (CCD) connected to a computer, by which the fluorescence of the sample could be observed and the position of the photobleached spot could be confined. The other one is the photomultiplier tube (PMT) joined with an oscilloscope by which the FRAP curve could be obtained. A detection pinhole is employed to the confocal microscope before the PMT to reject the background of the fluorescence attained to PMT. Figure 2 shows the FRAP curve recorded directly by the PMT.

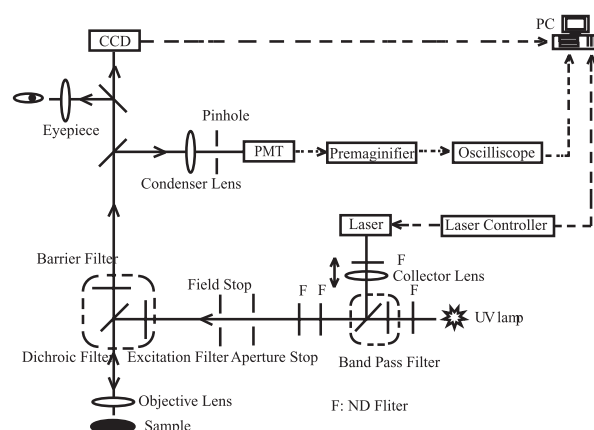


Figure 1. Schematic drawing of the new FRAP setup.

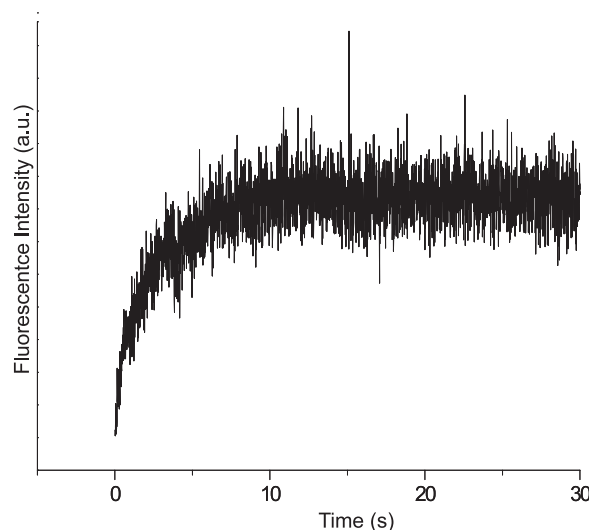


Figure 2. FRAP curve of the supported lipid bilayer (DPPC:POPS = 9:1, w/w) obtained by PMT.

VI-G Photoionization and Photodissociation Dynamics Studied by Electron and Fluorescence Spectroscopy

Molecular photoionization is a major phenomenon in vacuum UV excitation and provides a large amount of information on fundamental electron-core interactions in molecules. Especially, neutral resonance states become of main interest, since they often dominate photoabsorption cross sections and lead to various vibronic states which are inaccessible in direct ionization. We have developed a versatile machine for two-dimensional photoelectron spectroscopy in order to elucidate dynamical aspects of superexcited states such as autoionization, resonance Auger decay, predissociation, vibronic couplings, and internal conversion. In a two-dimensional photoelectron spectrum (2D-PES), the photoelectron yield is measured as a function of both photon energy $E_{h\nu}$ and electron kinetic energy E_k (binding energy). The spectrum, usually represented as a contour plot, contains rich information on photoionization dynamics.

Photofragmentation into ionic and/or neutral species is also one of the most important phenomena in the vacuum UV excitation. In some cases, the fragments possess sufficient internal energy to de-excite radiatively by emitting UV or visible fluorescence. It is widely accepted that fluorescence spectroscopy is an important tool to determine the fragments and to clarify the mechanisms governing the dissociation processes of diatomic and polyatomic molecules. For several years we have concentrated our energies on fluorescence spectroscopy of H_2O in the photon energy region of 15–55 eV.

VI-G-1 Photofragmentation Mechanisms of H_2O Studied by Ultraviolet Dispersed Spectroscopy

MITSUKE, Koichiro

[*J. Electron Spectrosc. Relat. Phenom.* **144-147**, 131–133 (2005)]

Fragmentation of H_2O has been studied by dispersed fluorescence spectroscopy at excitation photon energies above 20 eV.¹⁾ In dispersed spectra several vibrational bands begin to emerge below 320 nm with an appearance energy of $h\nu \sim 30$ eV. We have assigned these peaks to $OH(A^2\Sigma^+ \rightarrow X^2\Pi)$ transition, judging from characteristic peaks due to the R_1 band heads for the $\Delta v = v' - v'' = 0$ sequence at 306.4, 312.6, and 318.8 nm. The counter fragment of $OH(A^2\Sigma^+)$ must be $H^*(n)$ in the Rydberg state ($n \geq 2$). The above appearance energy is much higher than the dissociation limits for the $OH(A^2\Sigma^+) + H^*(n \geq 2)$ channels, but in good agreement with the vertical transition energies²⁾ for the associated Rydberg states of H_2O . There exist salient hydrogen atomic lines at $h\nu \geq 24.5$ eV constituting the Balmer series with $n = 3-9$, as indicated in Figure 1. The dispersed spectra reveal that there is no appreciable fluorescence signal from $OH(A^2\Sigma^+)$ below 30 eV. Hence, the dissociation channel of $OH(A^2\Sigma^+) + H^*(n)$ is not responsible for the Balmer emission at $h\nu = 24.5-30$ eV. The most plausible process leading to this Balmer emis-

sion is the three-body dissociation to $H^*(n \geq 3) + H(n = 1) + O(^3P_g)$, which has the thermochemical threshold at 21.7 eV with $n = 3$.

References

- 1) K. Mitsuke, *J. Chem. Phys.* **117**, 8334–8340 (2002).
- 2) J. Appell and J. Durup, *Int. J. Mass Spectrom. Ion Phys.* **10**, 247–265 (1972/73).

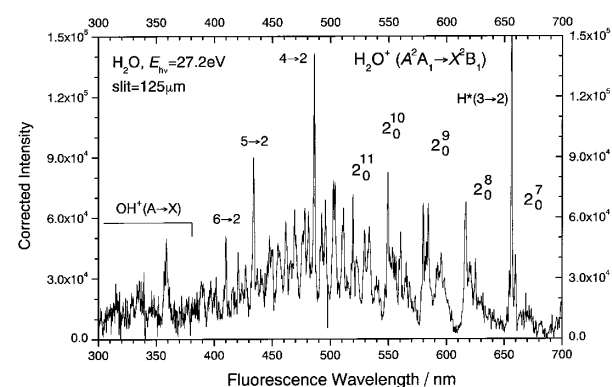


Figure 1. Dispersed fluorescence spectra of H_2O measured at $h\nu = 27.2$ eV. The $2_0^{v_2'}$ symbols designate the vibrational progression in the bending mode v_2 of the $H_2O^+[\tilde{A}^2A_1(0, v_2', 0) \rightarrow \tilde{X}^2B_1(0, 0, 0)]$ transition. The hydrogen Balmer lines $H^*[n^2L'J' \rightarrow 2^2L''J'']$ ($n = 3-9$) are indicated by the $(n \rightarrow 2)$ marks.

VI-H Extreme UV Photoionization Studies of Fullerenes by Using a Grazing-Incidence Monochromator and High-Temperature Mass Spectrometer

On the beam line BL2B in UVSOR a grazing incidence monochromator has been constructed which supplies photons in the energy region from 20 to 200 eV [M. Ono, H. Yoshida, H. Hattori and K. Mitsuke, *Nucl. Instrum. Methods Phys. Res., Sect. A* **467-468**, 577–580 (2001)]. This monochromator has bridged the energy gap between

the beam lines BL3B and BL4B, thus providing for an accelerating demand for the high-resolution and high-flux photon beam from the research fields of photoexcitation of inner-valence electrons, *L*-shell electrons in the third-row atom, and 4*d* electrons of the lanthanides.

Since 2001 we have tackled issues on photoabsorption and photofragmentation of fullerenes in the extreme UV. Geometrical structures and electronic properties of fullerenes have attracted widespread attention because of their novel structures, novel reactivity, and novel catalytic behaviors as typical nanometer-size materials. Moreover, it has been emphasized that the potential for the development of fullerenes to superconductors ($T_c \sim 50$ K) and strong ferromagnetic substances is extremely high. In spite of such important species spectroscopic information is very limited in the extreme UV region, which has been probably due to difficulties in obtaining enough amount of sample. The situation has been rapidly changed in these few years, since the techniques of syntheses, isolation, and purification have been advanced so rapidly that appreciable amount of fullerenes is obtainable from several distributors in Japan.

VI-H-1 Absolute Photoabsorption Cross Section of C_{60} in the Extreme Ultraviolet

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(¹Okayama Univ.)

[*J. Electron Spectrosc. Relat. Phenom.* **144-147**, 243–246 (2005)]

The absolute photoabsorption cross section curve of C_{60} has been determined by means of mass spectrometry with the photon source of monochromatized synchrotron radiation of $h\nu = 24.5$ –150 eV. Description has been made on a high-temperature source of gaseous fullerenes and an efficient time-of-flight mass spectrometer. The absolute cross section curve is shown in Figure 1. The obtained cross sections were 762, 241 and 195 Mb at $h\nu = 24.5$, 90, and 110 eV, respectively with about 10% errors. The cross section curve was then normalized at $h\nu = 25$ eV to the absolute photoabsorption cross section reported by Jaensch and Kamke,¹⁾ the most reliable data so far available in the valence excitation region of C_{60} . Accordingly, the present cross section data were altered to 407, 144 and 114 Mb at $h\nu = 25$, 90, and 110 eV, respectively.

Reference

1) R. Jaensch and W. Kamke, *Mol. Materials* **13**, 143 (2000).

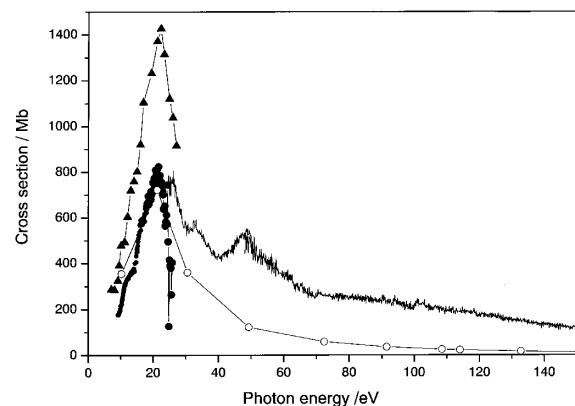


Figure 1. Absolute absorption cross section of C_{60} at $h\nu = 24.5$ –150 eV (solid line). The closed circles and triangles designate the previous data measured by Jaensch and Kamke and those compiled by Berkowitz, respectively. The open circles indicate the cross section of sixty carbon atoms.

VI-H-2 Photofragmentation of C_{60} in Valence Ionization

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[*J. Electron Spectrosc. Relat. Phenom.* **144-147**, 247–250 (2005)]

The yield curves for C_{60-2n}^+ ($n = 1$ –3) produced by photoionization of C_{60} are measured in the $h\nu$ range of 25–150 eV. The appearance energies increase with increasing n , as evidenced from Figure 1. Evaluation is made on the upper limits of the internal energies of the primary C_{60}^+ above which $C_{60-2n+2}^+$ fragments ($n \geq 1$) cannot escape from further dissociating into $C_{60-2n}^+ + C_2$. These limits agree well with the theoretical internal energies of C_{60}^+ corresponding to the threshold for the formation of C_{60-2n}^+ , on the assumption that the binding energies of $C_{60-2n+2}^+$ are equal to those proposed by Foltin *et al.*¹⁾

Reference

1) M. Foltin, M. Lezius, P. Scheier and T. D. Märk, *J. Chem. Phys.* **98**, 9624 (1993).

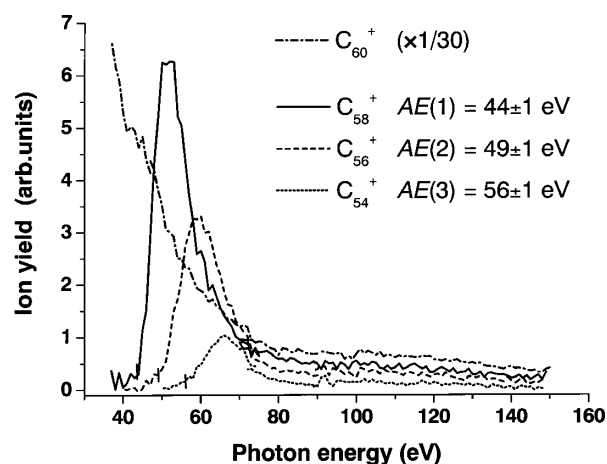


Figure 1. Ion yield curves of C_{60-2n}^+ ($n = 1$ –3) and C_{60}^+ ions. Every tick mark indicates the appearance energy $AE(n)$ determined by, after subtracting an appropriate background, reading the photon energy at which the ion yield reaches 3% of the peak height of the curve.

VI-H-3 Photofragmentation of C_{60} in the Extreme Ultraviolet: Statistical Analysis on the Appearance Energies of C_{60-2n}^{z+} ($n \geq 1$, $z = 1-3$)

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[Phys. Chem. Chem. Phys. 7, 119–123 (2005)]

The ion yield curves for C_{60-2n}^{z+} ($n = 1-5$, $z = 1-3$) produced by photoionization of C_{60} are measured in the photon energy ($h\nu$) range of 25–150 eV (see Figure 1). The appearance $h\nu$ values are higher by 30–33 eV than the thermochemical thresholds for dissociative ionization of C_{60} leading to C_{60-2n}^{z+} . Evaluation is made on the upper limits of the internal energies of the primary C_{60}^{z+} above which $C_{60-2n+2}^{z+}$ fragments ($n \geq 1$) cannot escape from further dissociating into $C_{60-2n}^{z+} + C_2$. These upper limits agree well with the theoretical internal energies of C_{60}^{z+} corresponding to the threshold for the formation of C_{60-2n}^{z+} (see Figure 2). The photofragmentation of C_{60}^{z+} is considered to be governed by the mechanism of internal conversion of the electronically excited states of C_{60}^{z+} , statistical redistribution of the excess energy among a number of vibrational modes, and sequential ejection of the C_2 units.

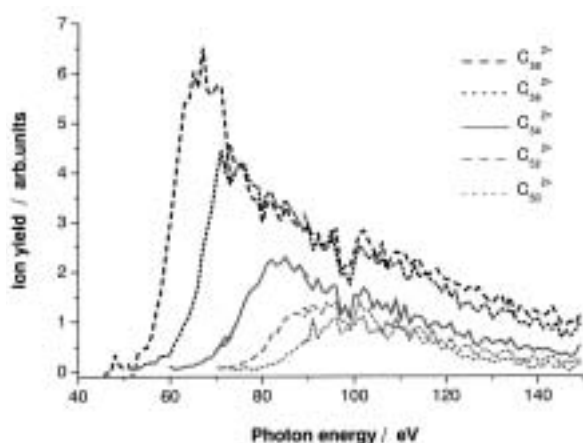


Figure 1. Ion yield curves of C_{60-2n}^{z+} ions ($n = 1-5$) obtained from time-of-flight mass spectra.

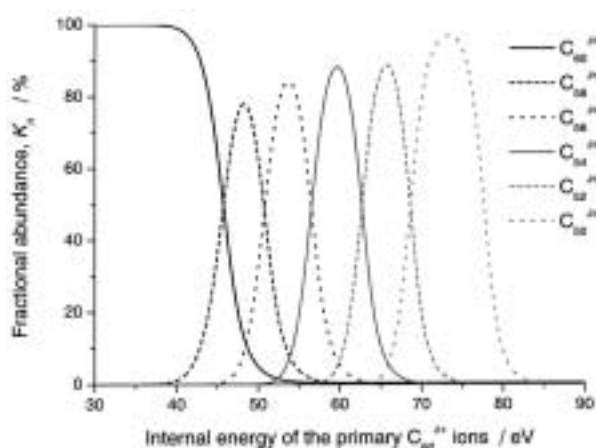


Figure 2. Fractional abundance curves of C_{60}^{z+} and C_{60-2n}^{z+} ions ($n = 1-5$) at 25 μ s after photoionization of C_{60} obtained by using the RRKM theory to calculate the rate constant for reaction $C_{60-2n+2}^{z+}$ ($n \geq 1$) $\rightarrow$ $C_{60-2n}^{z+} + C_2$.

VI-H-4 Fragmentation Mechanism of Highly Excited C_{70} Cations in the Extreme Ultraviolet

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[Am. Inst. Phys. Conf. Proc. in press]

The ion yield curves for C_{70-2n}^{z+} ($n = 1-8$, $z = 2$ and 3) produced by photoionization of C_{70} were measured in the photon energy ($h\nu$) range of 25–150 eV. The appearance $h\nu$ values were higher by ca. 34 eV than the thermochemical thresholds for dissociative ionization of C_{70} leading to C_{70-2n}^{z+} . Evaluation was made on the upper limits of the internal energies of the primary C_{70}^{z+} above which $C_{70-2n+2}^{z+}$ fragments cannot escape from further dissociating into $C_{70-2n}^{z+} + C_2$. These critical internal energies agreed well with appearance internal energies of C_{70}^{z+} theoretically obtained corresponding to the threshold for the formation of C_{70-2n}^{z+} (see Figure 1). The photofragmentation of the parent C_{70}^{z+} ions is considered to be governed by the mechanism of internal conversion of their electronically excited states, statistical redistribution of the excess energy among a number of vibrational modes, and sequential ejection of the C_2 units.

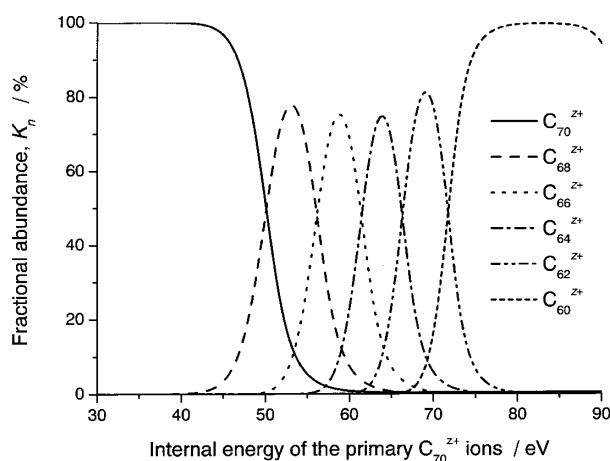


Figure 1. Fractional abundance curves of C_{70}^{z+} and C_{70-2n}^{z+} ions at 25 μ s after photoionization of C_{70} obtained by using the RRKM model to calculate the rate constants for reaction $C_{70-2n+2}^{z+}$ ($n \geq 1$) $\rightarrow$ $C_{70-2n}^{z+} + C_2$.

VI-H-5 $4d \rightarrow 4f$ Dipole Resonance of the Metal Atom Encapsulated in a Fullerene Cage: Ce@C₈₂

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[*J. Chem. Phys.* **122**, 064304 (5 pages) (2005)]

The yield curves for photoions from Ce@C_{82} are measured by using synchrotron radiation in the photon energy range from 90 to 160 eV. Parent Ce@C_{82}^{z+} and fragment ions C_{60}^{z+} and C_{70}^{z+} are observed in a mass spectrum ($z = 1$ and 2) as shown in Figure 1. The yield curves for doubly-charged ionic species exhibit broad resonance in the photon energy region of from 120 to 140 eV which is ascribed to the $4d \rightarrow 4f$ giant dipole resonance of the encapsulated Ce atom. The total photoabsorption cross section of Ce@C_{82} was determined from partial photoionization cross sections for formation of the parent and fragment ions to be $5.3^{+1.8}_{-1.1}$ and $19.6^{+6.5}_{-3.9}$ Mb at photon energies of 110 and 130 eV, respectively.

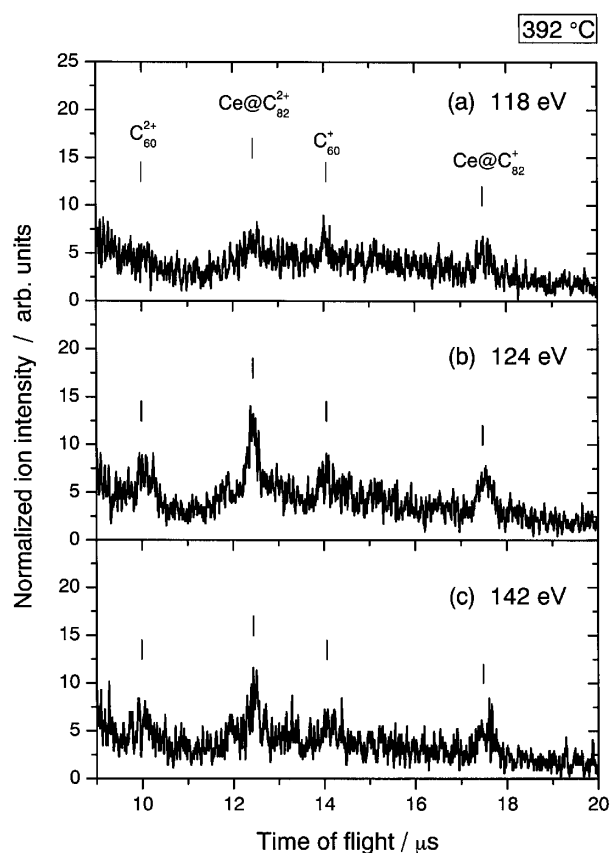


Figure 1. Time-of-flight mass spectra of the Ce@C_{82}^{z+} ($z = 1$ and 2) produced by photoionization of Ce@C_{82} at $h\nu = 118$, 124, and 142 eV. The average temperature of the sample holder was set to 392 °C. Most of C_{60}^{z+} ions arose from photoionization of C_{60} neutrals which were desorbed from the surface of mass spectrometer.

VI-H-6 Photoion Yield Curves of Dy@C_{82} in the Vacuum UV Region

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[*Int. J. Mass Spectrom.* **243**, 121–125 (2005)]

The photoion yield curves for Dy@C_{82}^{z+} ($z = 1$ and 2) from Dy@C_{82} are measured by using synchrotron radiation in the photon energy range from 24.5 to 39.5 eV. Correction has been made to compensate the effect of transient change of the density of Dy@C_{82} in the interaction region, with the help of the yield curve of C_{60}^{z+} produced from C_{60} remaining as a trace impurity in the sample. The yield of Dy@C_{82}^{z+} in Figure 1 exhibits a gradually descending curve with a flat region at 30–33 eV, similarly to the yield curve of C_{60}^{z+} from C_{60} . The total photoabsorption cross section of Dy@C_{82} was evaluated to be $(1.2 \pm 0.4) \times 10^2$ Mb at the photon energy of 39.5 eV.

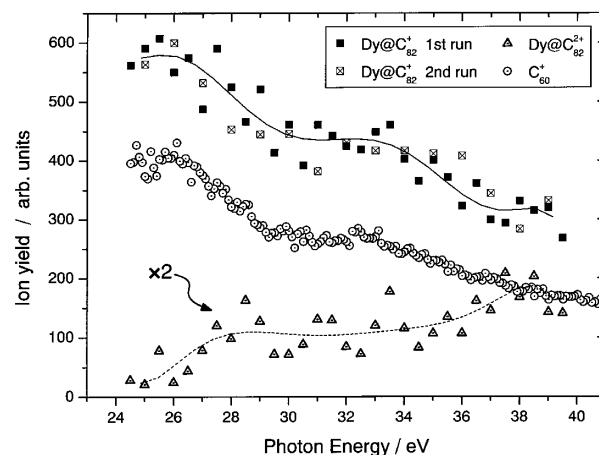


Figure 1. Yield curves of Dy@C_{82}^{z+} (solid square) and Dy@C_{82}^{2+} (open triangle) produced from Dy@C_{82} in the photon energy range of from 24.5–39.5 eV. The spectra are taken at photon energy intervals of 0.5 eV. The solid and dashed curves represent the results of the least-squares fitting to the data points of Dy@C_{82}^{z+} and Dy@C_{82}^{2+} , respectively, using seventh order polynomial functions. The open circle symbols denote the yield curve of C_{60}^{z+} from C_{60} measured by using the neat C_{60} sample.

VI-H-7 $4d$ – $4f$ Dipole Resonance of the Pr Atom in an Endohedral Metallofullerene, Pr@C_{82}

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Following our preceding paper on the photoion yield spectra of the endohedral metallofullerene, Ce@C_{82} ,¹⁾ photoion yield spectra of Pr@C_{82} were measured in the photon energy range 100–150 eV with the help of time-of-flight mass spectrometry. Parent ions, Pr@C_{82}^{z+} , Pr@C_{82}^{2+} and Pr@C_{82}^{3+} were observed in the mass spectra. Photoion yield spectra of Pr@C_{82}^{z+} and Pr@C_{82}^{2+} were obtained from the mass spectra and are shown in Figure 1. The photoion yield spectra of Pr@C_{82}^{2+} showed a broad peak at 120–140 eV. The broad peak was assigned to the $4d \rightarrow 4f$ giant dipole resonance of the encapsulated Pr atoms. Absolute photoabsorption cross sections of Pr@C_{82} were evaluated from the photoion yield spectra and found to be 23.6 ± 6.7 at 110 eV (off-resonance) and 35.0 ± 6.3 Mb at 130 eV (on-resonance).

The enhancement of photoabsorption due to the giant resonance was comparable to that in $\text{Ce}@C_{82}$. The shapes of the peaks in the $\text{Pr}@C_{82}$ spectra, which originated from interference effects induced by the fullerene cage, were, however, different from those in the $\text{Ce}@C_{82}$ spectra. This could be accounted for by that the interference effects depend on the interior metal atoms.

Reference

- 1) K. Mitsuke, T. Mori, J. Kou, Y. Haruyama and Y. Kubozono, *J. Chem. Phys.* **122**, 064304 (2005).

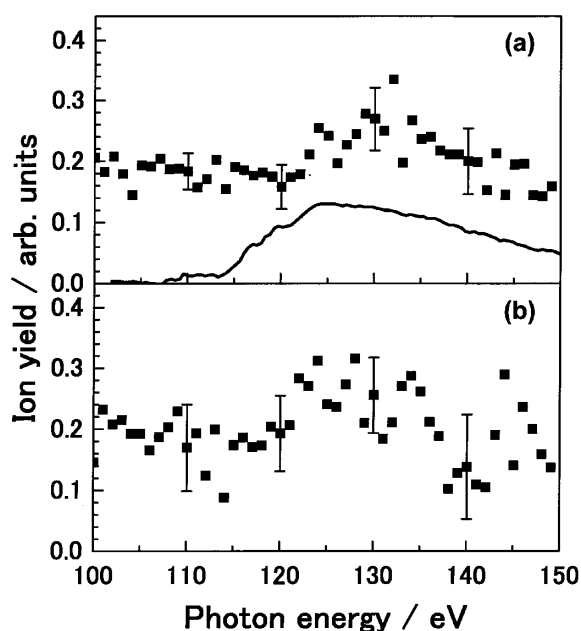


Figure 1. Ion yield curves of (a) $\text{Pr}@C_{82}^{2+}$ and (b) $\text{Pr}@C_{82}^{+}$. Error bars correspond to 1σ of five experimental runs. Solid curve in (1) are the absorption spectra of Pr atoms. For each data, the vertical scaling is arbitrary.

VI-I Kinetic Energy Analysis of the Fragment ions Produced from Fullerenes

When fullerenes gain enough amount of energy through photoionization processes, primarily formed ions are known to undergo decomposition into fragment ions with even numbered carbon atoms via sequential loss of C_2 units. We have measured the yield curves for C_{60-2n}^{z+} from C_{60} as a function of the internal energy of the parent C_{60}^{z+} ions to study the mechanisms and kinetics of the sequential unimolecular reactions. These experimental yield curves have been compared with the theoretical fractional abundance curves which have been derived by employing the RRKM theory to every process of the sequential reactions: $C_{60-2n+2}^{z+} (n \geq 1) \rightarrow C_{60-2n}^{z+} + C_2$. To a first approximation the critical activation energies of the reactants $C_{60-2n+2}^{z+}$ were assumed to be equal to their binding energies for the above reactions. Indeed the experimental and theoretical curves provide almost the same appearance energies for the formation of $C_{60-2n}^{z+} (n \geq 1)$. More reliable calculations of the rate constants of the individual reactions are needed before closer comparison between the two curves will be made. For such calculations we must know precise values of the activation energies for the reactions, together with the vibrational spectra of the transition states. This induced us to develop a new ion spectrometer for the fragment ions produced from C_{60}^{z+} and C_{70}^{z+} . We wish that the magnitude of the potential barriers of the reactions can be estimated from the average kinetic energy release measured by this spectrometer.

VI-I-1 Development of the Photofragment Imaging Apparatus to Measure Scattering Distributions of the C_{60-2n}^{z+} and C_{70-2n}^{z+} Fragments Produced by Dissociative Photoionization of C_{60} and C_{70}

KAFLE, Bhim Prasad¹; KATAYANAGI, Hideki;
MITSUKE, Koichiro
(¹SOKENDAI)

In dissociative photoionization of solitary fullerenes (C_{60} , C_{70}), we have measured the photofragment (C_{60-2n}^{z+} , C_{70-2n}^{z+}) yield curves in the photon energy

range of 45–150 eV.¹⁾ From the results we concluded that the excess energy is statistically distributed among the internal degrees of freedom of the parent ions (C_{60}^{z+} , C_{70}^{z+}) and C_2 units are ejected sequentially. Moreover, the results imply that the dissociation has no barrier and that no resonant state participates in the dissociation. To clarify these implications, we designed the photofragment imaging apparatus on the basis of the time-of-flight mass spectrometer that we had constructed. From the photofragment images, we can extract the kinetic energy and angular distributions of the photofragments. These distributions reveal clearly whether there exist a barrier and/or resonance.

We adopted the Eppink-Parker type velocity focusing electrodes²⁾ to achieve the energy resolution on the images to resolve less than 0.1 eV increase by the photofragmentation. To select a bunch of fragments having a desired mass from neighboring bunches, for example, selecting C_{66}^+ from the consecutive masses such as C_{68}^+ , C_{66}^+ and C_{64}^+ , we designed a “mass gate” which consists of a potential switch and retarding electrodes. In order to optimize the dimensions of the setup, we performed ion trajectory simulations utilizing SIMION 7.0 software. The most suitable dimensions were obtained already and are schematically illustrated in Figure 1.

The operation principle of this setup is as follows. As long as the tube of the potential switch is kept grounded, all fragments are reflected back by the retarding electrode and do not hit an imaging detector. When an entire bunch of fragments having a desired mass enters inside the tube, the pulsed voltage is applied to the tube. The potential felt by the bunch are suddenly elevated and the bunch passes through the retarding electrode and thus hits the imaging detector.

Parts of this setup are being fabricated at the machine shop in IMS. The parts will be installed in the end-station at the beam line 2B in UVSOR. We are planning to examine the performance of the setup using SF_6 as a standard sample.

References

- 1) J. Kou, T. Mori, Y. Kubozono and K. Mitsuke, *Phys. Chem. Chem. Phys.* **7**, 119 (2005).
- 2) A. T. J. B. Eppink and D. H. Parker, *Rev. Sci. Instrum.* **68**, 3477 (1997).

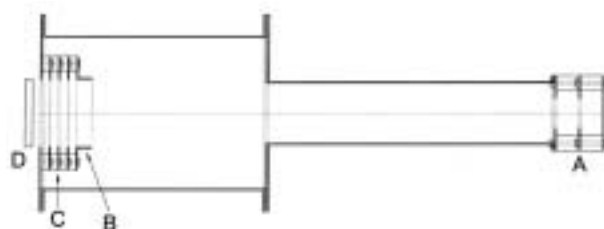


Figure 1. Schematic illustration of the photofragment imaging apparatus. A, repeller and extractor; B, potential switch; C, retarding electrodes; D, imaging detector.