

Molecular Spectroscopy at Corrugated Metal Surfaces

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Spectroscopic properties for molecules in the vicinity of metal surfaces are usually determined by the energy transfer mechanisms between the admolecules and the substrate, which can involve various collective excitations of substrate electrons such as surface plasmons and excitons. A brief review of this subject, including such phenomena as fluorescence and photoabsorption line shapes, is presented. The review includes recent work by the authors, with emphasis on the effects of surface corrugations. Examples that illustrate possible applications of such studies in the areas of surface photochemistry and heterogeneous catalysis are also discussed.

Since the work of Purcell (1), which dates back to the 1940s, it has been known that the spectroscopy of a gaseous system can be dramatically influenced by its environment. In particular, molecular spectroscopy for systems located in the vicinity of a surface has received considerable attention during the past 20 years. Photoabsorption cross sections, line shapes, and fluorescent characteristics such as line broadenings and level shifts in the presence of a substrate surface are typically the spectroscopically observable quantities of most interest. Motivations for such intensive studies are varied. From a purely academic perspective, it is of interest to understand the various interactions between the adsorbates and the substrate surface, which presents a complicated problem because it is a many-body system that does not possess bulk symmetries. From a practical perspective, it is expected that such studies will lead to applications in areas ranging from surface photochemistry to characterization of material structures and properties of the substrate surface. In the area of surface photochemistry, for example, if one assumes that the properties of the substrate

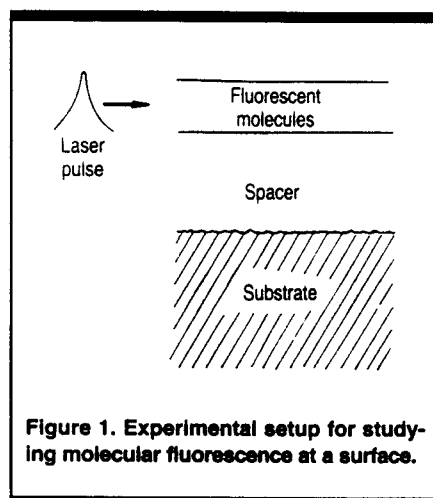


Figure 1. Experimental setup for studying molecular fluorescence at a surface.

surface are well understood (through other analyses such as low-energy electron diffraction), one can then study the photoabsorption cross section of admolecules to find useful information in regard to the ultimate goal of laser-assisted heterogeneous catalysis (2). Conversely, if the spectroscopic properties of admolecules are well understood, one can then use that knowledge to probe the electronic (and lattice) structure and properties of the substrate surface.

In this article, we will discuss spectroscopic properties of molecules at corrugated metal surfaces. The role of surface roughness has been recognized to be prominent in various surface processes since the discovery of the surface-enhanced Raman effect in 1974 (3). Because there is already a comprehensive review on surface spectroscopy covering work before 1984 (4), we will review progress made in just the past several years, with emphasis on our own work. We will pay particular attention to the fluorescence of the admolecules, which has already been discussed extensively in relation to perfectly flat surfaces (5,6). We will also review briefly some phenomena concerning photoabsorption and line shape. Finally, we will mention some possible applications in photochemistry at surfaces.

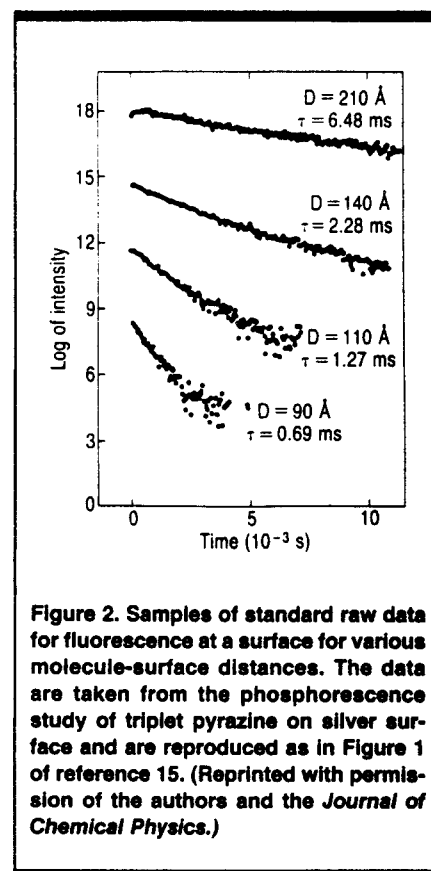


Figure 2. Samples of standard raw data for fluorescence at a surface for various molecule-surface distances. The data are taken from the phosphorescence study of triplet pyrazine on silver surface and are reproduced as in Figure 1 of reference 15. (Reprinted with permission of the authors and the *Journal of Chemical Physics*.)

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This kind of study is usually carried out using a "sandwich-type" experimental setup, as shown in Figure 1. A layer of fluorescent molecules is separated from the substrate surface by a spacer that controls the molecule-surface distance (d). An incident pulse laser of a width shorter than the lifetimes of the admolecules excites the molecules, and the deexcitation emission intensities are analyzed by various time-resolved techniques (standard raw data are reproduced in Figure 2). For a fixed fluorescence/phosphorescence emission frequency (ω), the decay rate (γ) for the

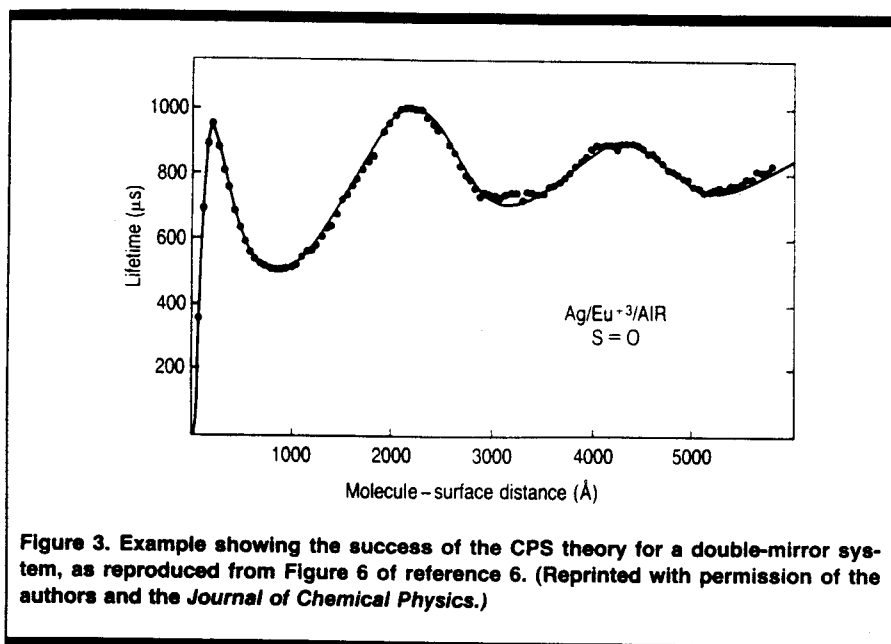


Figure 3. Example showing the success of the CPS theory for a double-mirror system, as reproduced from Figure 6 of reference 6. (Reprinted with permission of the authors and the *Journal of Chemical Physics*.)

admolecules can then be studied as a function of d ; or likewise, one can fix d and study γ as a function of ω , which will be more involved in the excitation process in the experiment. At distances very close to the surface where γ is very large, phosphorescent molecules will be preferred; otherwise, one would require a laser pulse of extremely narrow width and very fine time-resolved equipment.

The simplest theoretical description for this kind of experiment is the classical phenomenological (CP) approach in which the admolecule is modeled as a point dipole satisfying the equation of motion for a damped harmonic oscillator driven by the surface field (\vec{E}_s). This approach leads to the level shift and the induced decay rate of the following form:

$$\Delta\omega = \frac{\gamma^2}{8\omega_0} + \frac{\alpha}{2\omega_0} \text{Re}G, \quad [1]$$

$$\gamma = \gamma_0 + \frac{\alpha}{\omega_0} \text{Im}G,$$

where ω_0 and γ_0 are the free-molecule level spacing and line width respectively, α is an electromagnetic coupling constant, and the dyadic function G ($\equiv E_s/\mu$) is defined as the surface field acted on the molecule per unit dipole moment (μ). Because $\gamma_0 \ll \omega_0$ in general, it is not difficult to see why $\Delta\omega$ is given less attention than the surface effect on γ . The remaining task is then to calculate the function G .

There are two common ways of calculating E_s within the CP approach. The more exact treatment involves solving for the exact electrostatics of the dipole-surface system (where E_s is given by the reflected field at the dipole site); this is known as the energy

transfer theory (ET) because the result for the total rate γ is separated clearly in terms of the contribution from the radiative and nonradiative transfer. In another, cruder calculation method, one considers the "static limit" of the problem and replaces the effects from the surface by the image dipole, whereby E_s is simply given by the image field. For flat surfaces, this image theory (IT) simply leads to the "inverse cube rule" for the variation of γ versus d , and the oscillation behavior at far distances is well explained by the exact, energy transfer theory established by Chance, Prock, and Silbey (CPS)(5,6) through the application of Sommerfeld's theory concerning a radiating antenna above the surface of the earth (7,8). This CPS theory successfully explains the experimental data of Drexhage and co-workers (9) as illustrated by the example depicted in Figure 3 where the fluorescence of the Eu^{3+} complex has been studied using fatty acids as the spacer.

For rough surfaces, however, only the image theory approach has been taken to study the fluorescence problem because of the mathematical complications of the dynamics of the problem (10). It has been widely believed that as long as $d \ll \lambda$, where λ is the emission wavelength (e.g., $d \sim 10^2 \text{ \AA}$ in the region for the most pronounced surface morphological effects and $\lambda \sim 10^3 \text{ \AA}$ for fluorescence in the visible range), the image theory should be accurate enough to describe the phenomenon (10). In a recent analysis (11), however, it was found that the condition $d \ll \lambda$ is not sufficient and that the image theory can break down appreciably for a highly conducting substrate such as a noble metal. The physical reason behind this problem is that the dynamic Helmholtz wave equation in a metal does not reduce to the static Poisson equation in the long-wavelength limit if the conductivity of the metal becomes very large. We conclude,

therefore, that it is always desirable to rely on ET for any dipole-surface problem, for both flat and rough surfaces.

The motivation for studying decay rates at rough surfaces comes from some recent experiments. Figure 3 illustrates how well the CPS theory fits the experiment in the large distance region. However, this simple approach is expected to fail for very close distances because the surface cannot be regarded as an abruptly discontinuous boundary because of the quantum spreads of the surface electrons. Nevertheless, it has been established that as long as $d \geq 10 \text{ \AA}$ such microscopic effects can be ignored and the CP approach should be valid (12). But discrepancies were observed between the CPS theory and the data in a recent experiment carried out by Harris and co-workers (13) in which the phosphorescence of biacetyl ($^3n\pi^*$) was studied above a silver surface using liquid ammonia as the spacer for distances in the range between 20 and 100 \AA . The theory was found to lie either above or below the best fit to the data. Harris and colleagues tried to explain the discrepancies by resorting to a surface-damping model proposed by Persson and Lang (14) based on the electron-hole pair excitation mechanism. Although that is a plausible explanation, we would like to consider other alternative possibilities such as the effect of surface roughness at such close distances. As a matter of fact, researchers at Bell Laboratories had earlier carried out a similar experiment involving triplet pyrazine on silver with liquid argon as a spacer, where discrepancies between the CPS theory and the data were observed with the data lying above the theory (15). Although these researchers blamed the inaccuracy on the dielectric constants they used to compute results based on the CPS theory, we suspect that roughness may have again played a role in their experiment. In fact, in an even earlier experiment, the same researchers had reported observing, with the help of a scanning electron microscope, an almost periodic structure caused by polishing the surface finish of the underlying sapphire (16). All of these results led us recently to carry out theoretical studies on the effects of surface roughness on admolecular decay rates.

For cases in which the roughness is shallow, we have established a dynamic theory (ET) for the admolecular decay rate, adopting a perturbative approach (17). Essentially, we have solved the Sommerfeld problem (7,8) for a "rough earth." In particular, we have considered the simplest kind of surface morphology, namely, a shallow sinusoidal grating, where we have combined the CPS theory (5,6) with the theory published by Maradudin and Mills (18) for light-scattering phenomena, which applies the Green's function technique. Figure 4 reproduces some of our results. We plotted the ratio $\mathcal{R} \equiv \gamma^R/\gamma^F$ versus d with γ^R being the induced decay rate for

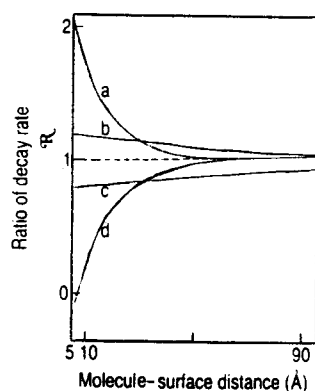


Figure 4. Ratio (R) of the decay rate for a silver grating surface to that for a flat surface as a function of the molecule-surface distance for various emission frequencies (ω) and grating wave number (Q). Curve a: $\omega = 2 \times 10^4 \text{ cm}^{-1}$, $Q = 0.08 \text{ \AA}^{-1}$; curve b: $\omega = 2 \times 10^4 \text{ cm}^{-1}$, $Q = 0.01 \text{ \AA}^{-1}$; curve c: $\omega = 3 \times 10^4 \text{ cm}^{-1}$, $Q = 0.01 \text{ \AA}^{-1}$; and curve d: $\omega = 3 \times 10^4 \text{ cm}^{-1}$, $Q = 0.08 \text{ \AA}^{-1}$. The grating amplitude ζ_0 is fixed at 0.7 \AA .

the case of a silver grating surface and γ^F being the flat surface value from the CPS theory. The curves a, b, c, and d correspond to different emission frequencies and different grating parameters. We made the following three observations:

- As one goes farther from the surface, the effect of the surface roughness disappears ($R \rightarrow 1$), as expected.
- The CP approach breaks down explicitly at very close distances [note that the curve 4 (d) dips below zero], so our numerical results are trustworthy only for d beyond, say, approximately 10 \AA .
- Most importantly, roughness can enhance or diminish the flat surface value depending on the various parameters, a finding that is in qualitative agreement with the earlier experimental observations.

In cases of large roughness, it becomes impractical to carry perturbation theory to higher orders. Here one can introduce the so-called "island-surface" model as depicted in Figure 5. Instead of looking at the actual profile of the surface, we model it as a collection of islands, or bumps, of unlimited sizes. The simplest kind of bump is a sphere. The decay rate problem of a dipole near a collection of spheres has been worked out by Liver, Nitzan, and Freed using the image theory approach, and the problem for an isolated

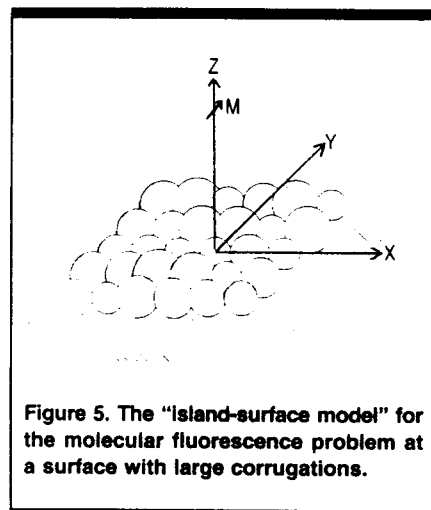


Figure 5. The "island-surface model" for the molecular fluorescence problem at a surface with large corrugations.

sphere (19) — or a single island — has been worked out using the dynamic ET approach (20–22). We are currently in the process of constructing an ET approach for a realistic island surface by generalizing our previous work to include the effects of neighboring spheres. Until this is done, we believe that a fully dynamic description of the decay problem for very rough surfaces is still unavailable for comparison with experimental results.

So far, we have been focusing on the molecule-surface distance (d) behavior of the induced decay rates for molecules at a rough surface. We have also looked into the decay rate spectrum as a function of the emission frequency at a fixed d . We have observed, in addition to the ordinary surface-plasmon (SP) peak, an extra resonance structure induced by the presence of roughness that is morphology-dependent. This extra peak is predicted only from the ET approach — not from the image theory. Figure 6 compares the two theories at a very close distance (50 \AA) for a silver grating surface (23), where the image theory is expected to be very accurate. We observe that for cases in which the grating wave number (Q) is close to the emission wave numbers (k), such as in curves b and c, ET predicts a resonance at an emission frequency with $k \approx Q$. The position of this new peak moves as Q varies, and this peak grows with the increasing amplitude of the grating. One can imagine that this morphology-dependent peak may move and grow to overshadow the ordinary SP peak, so that in actual experiments one observes a kind of peak shift in the spectrum. Previously, there has been a controversy as to whether surface roughness causes peak shifts in the energy-loss spectrum obtained from an electron scattering experiment with a Mo surface (24,25).

Our results obtained for the decay-rate spectrum shed some light on that issue. This morphology-induced resonance peak should be a very general phenomenon and should show up for any kind of rough surface. Recently, we have repeated such a decay-rate spectrum study for a spherical surface in an attempt to

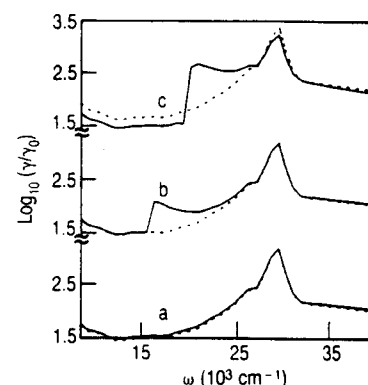


Figure 6. Decay-rate spectrum for a molecule located at $d = 50 \text{ \AA}$ above a silver grating surface of varying roughness parameters (ζ_0, Q). Curve a: $\zeta_0 = 1 \text{ \AA}$, $Q = 1 \times 10^{-2} \text{ \AA}^{-1}$; curve b: $\zeta_0 = 1 \text{ \AA}$, $Q = 1 \times 10^{-3} \text{ \AA}^{-1}$; curve c: $\zeta_0 = 10 \text{ \AA}$, $Q = 1.25 \times 10^{-3} \text{ \AA}^{-1}$. The solid line represents results from ET and the dotted line represents results from the image theory.

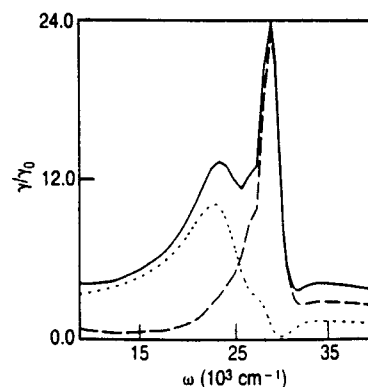


Figure 7. Decay-rate spectrum for a molecule located at $d = 700 \text{ \AA}$ from the center of a silver sphere of radius $a = 500 \text{ \AA}$ according to ET. The solid, dotted, and dashed curves denote the total, radiative, and nonradiative rates, respectively.

understand the physical origin of this new peak (26). Figure 7 shows one of our calculations in which we split the total decay rate into radiative and nonradiative components, respectively. It is obvious that the roughness-induced peak has its origin in the radiative transfer from the molecule in the presence of

the surface. The same origin should be responsible for the peak that appears for the grating surface. Moreover, nothing happens in the case of a flat surface because the surface plasmon modes can couple to radiative modes only in the presence of surface roughness.

To conclude our discussion on fluorescence phenomena, let us consider a corrugated thin film, another substrate system of great interest. When there are two rough surfaces that can each support an SP mode, an interesting phenomenon known as "cross coupling" can occur if the two SPs are excited resonantly at the same time. This phenomenon in turn can lead to dramatic effects such as SP-induced transparency in a metal film (27,28), which can have technological applications in device designs because one can then "plate" a dielectric substance (e.g., a polymer) so that it becomes an electrical conductor without becoming opaque. Although it has been controversial as to whether such a cross-coupling phenomenon has indeed been observed in various light-scattering experiments (29,30), a group at the University of Rochester recently claimed an unambiguous observation of this phenomenon through molecular fluorescence studies on a thin-grating silver film (31). We are currently trying to generalize our previous theory for a single grating surface (17) to include such a system in order to better understand the experiment.

PHOTOABSORPTION LINE SHAPE

Investigations into the photoabsorption rates and spectral line shapes can also yield useful information concerning admolecules and the substrate surface. Experimentally, one would illuminate the adlayer by a CW laser and analyze the absorption spectrum instead of using a laser pulse to excite the admolecules. This subject has been reviewed elsewhere (32), and here we will give it only a brief sketch. The two main concerns are the enhancement rate (r) in the photoabsorption/dissociation process, which is defined as the ratio between the absorption cross sections with and without the presence of the surface, and the distortions in the line shape $I(\omega)$ of the spectrum because of surface effects. There is extensive literature on this subject covering such molecular spectra as vibrational (33), rovibrational (34), and electronic (35,36) spectra for both physisorbed (33-36) and chemisorbed (37,38) molecules. Furthermore, different kinds of surface morphologies have been considered, ranging from the shallow grating model to a sphere-modeled "island surface" (35,36). Because of the surface damping effect discussed above, which will now compete with the surface-enhanced field, one can introduce the concepts of a critical molecule-surface distance (below which $r < 1$) and

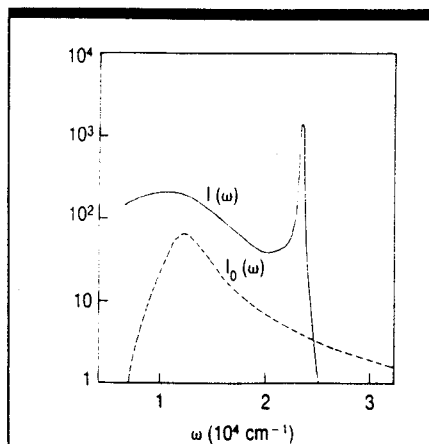


Figure 8. Distortion of the Fano profile $I_0(\omega)$ that simulates certain molecular predissociation processes at a silver grating surface. The y-axis is in arbitrary units.

a critical optical distance (at which r is maximum) to describe the photoabsorption/dissociation phenomenon (36). In addition, by looking at the distortions of $I(\omega)$, one can learn about the response of the substrate surface to a particular molecular process. For example, the double-peak feature and the sharp-edge window that appear in the distortions of a free molecular absorption (Lorentzian) profile in the presence of a spherical or shallow-grating surface signify the effects of the surface plasmon on the photoabsorption process (35,36). Langreth (39) noted that even for a flat surface the original (free) molecular line shape cannot remain Lorentzian and is distorted towards a Fano-type profile if electron-hole pairs are created in the substrate because of energy transfer from the excited admolecule. Moreover, most previous studies have assumed that the free-molecule profile $I_0(\omega)$ is symmetric (often Lorentzian) and, therefore, that asymmetric distortions are brought about by the presence of the surface.

Recently, we have initiated a phenomenological study of the case in which $I_0(\omega)$ is already asymmetric in nature (40). Some preliminary results for a profile in the presence of a shallow-grating surface have been obtained, where $I_0(\omega)$ has been taken in the form of a Fano profile, in which the double-peak feature again appears with the broadening of the original profile window at the steep edge near the low-frequency end. The creation of a new window at the high-frequency end because of the surface-plasmon resonance is also observed, as shown in Figure 8. This study should shed some light on processes such as autoionization and predissociation for molecular systems in the vicinity of a surface. We hope that more rigorous studies will be carried out in the future.

CONCLUSIONS

We have discussed various issues concerning molecular spectroscopy at corrugated metal surfaces, paying particular attention to our own work during the past two years. We believe that the most interesting issue that still awaits experimental resolution is whether roughness always enhances the flat-surface values for the induced decay rate or, as we have shown, both enhancement and diminution can occur. This issue may have practical relevance in laser-assisted heterogeneous catalysis where one wants to maximize the photoabsorption rate of the reagents through the enhanced surface fields so that the molecules can be dissociated efficiently to undergo various reactions. It is well known that the surface-induced line broadening effect will tend to suppress the absorption rate, thus lowering the photochemical yield. According to our findings, however, one can make use of the surface roughness to suppress such line broadenings by carefully controlling the factors involved in the process. Hence, an experimental clarification of this issue is of prime importance. Another interesting feature we have found that still awaits experimental verification is the roughness-induced peak in the decay-rate spectrum, although similar effects may have already been observed in electron scattering experiments (24). Finally, we want to emphasize that fluorescence and other spectroscopic studies for admolecular systems can lead to a deeper understanding of the substrate surface and of possible surface-induced chemical processes that can take place in the vicinity of the surface.

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