Nonlinear evanescent wave spectroscopies: A close look at the gas-solid interface

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Abstract. The dynamics of atom/molecule scattering at surfaces plays a crucial role in such diverse applications as heterogeneous catalysis, chemical vapor deposition and aerodynamics. This process traditionally has been investigated with the use of atomic/molecular beams under ultra-high-vacuum conditions, i.e. far from the regime required in applications that has been formulated as a ‘pressure gap’ problem. The advantages of laser spectroscopy, however, suggest a principally new approach to this dilemma. In this paper we present recent results, both experimentally and theoretically, illustrating the potential of evanescent wave spectroscopy for surface science. We demonstrate how such fundamental features of gas-surface scattering as the scattering kernel, sticking probability and velocity distribution functions can be extracted from a careful analysis of nonlinear fluorescence spectra excited by both evanescent and non-evanescent waves. We also review the prospective developments of this technique which are based on a four-wave mixing process and Raman scattering in atoms/molecules near surfaces.

1. Introduction
Gas-solid interface plays a crucial role in diverse applications and therefore it has attracted a long-term attention of researches from different fields of science. Such dissimilar at first glance phenomena as heterogeneous catalysis, film growth, heat and mass transfer between a gas and a solid or energy and momentum exchange between a gas flux and a streamlined surface have in their background common elementary processes of sticking, desorption, molecule-surface scattering or surface chemical reactions. Although it sounds paradoxically, the gas itself prevents from the extracting information about what is going on at the gas-solid interface. A surface buried under a gas atmosphere is inaccessible for tools traditionally used in surface science such as electron, ion or atom scattering as well as photoelectron spectroscopy. As regards gas-surface scattering, ”in the presence of an equilibrium gas phase, there is no means whatsoever to distinguish between desorbing, reflected, diffracted or inelastically scattered molecules” [1]. A severe demand is therefore to pump off the gas and to investigate atom or molecule scattering at surfaces under ultra-high-vacuum conditions with the use of collimated particle beams. This has been the line of attack on this problem since the origin of surface science. It is well known, however, that a surface contacting a gas is not identical with the same surface in vacuum. Adsorbed atoms or molecules which present at a gas-solid interface can cause surface reconstruction, change of work function or surface phase transitions [2]. They also can essentially...
modify the interaction potential between gas particles and a surface and hence the process of
gas-surface scattering. In other words, the validity of using the data obtained under ultra-high-
vacuum conditions for various applications is questionable.

An alternative approach to this problem can be developed basing on the advantages suggested
by laser spectroscopy. In general, the interaction of a gas with a solid surface modifies its optical
properties. The influence of the surface, however, is essential only in the gas boundary layer and
becomes negligible as the distance from the surface is increased. One can distinguish between
three length scales which characterize the optical response of the gas boundary layer. One of
them is of the order of the mean free path, $l$. It determines the so-called Knudsen layer where
interatomic collisions are very seldom. The other two can be called the population memory
length, $l_1$, and the polarization memory length, $l_2$. They determine the mean distances from
the surface at which the level populations of the atoms scattered by the surface or their induced
dipole moments, respectively, do not change remarkably. At distances from the surface much
larger than these three lengths the information on gas-surface scattering is completely destroyed
due to interatomic collisions. All three length scales therefore decrease when the gas pressure
increases. An obvious conclusion which follows from the above consideration is that the optical
properties of the gas boundary layer can be probed by an electromagnetic field strongly localized
near a surface. Well-known examples of such fields of evanescent character are electromagnetic
waves excited in total internal reflection (evanescent waves, EWs) and surface polaritons which
are coupled modes of incident light and substrate excitations. When their penetration depth into
the gas, $\delta$, is less than $l$, $l_1$ or $l_2$, one can expect that the gas optical response will be sensitive to
the change in the velocity distribution function, quenching of excitation or coherence relaxation,
respectively, in the course of gas-surface scattering. In the visible spectral region $\delta$ is in the
submicron range that ensures the sensitivity of EW optical spectra to gas-surface interface
properties within a wide interval of gas pressures.

2. Gas optical response in the linear regime
To understand how the optical response of a gas near a surface is formed it is instructive to
consider first the linear regime of interaction with an electromagnetic field. The main features of
the atom-field interaction can be reproduced in the framework of a classical model considering
an atom excited by an electromagnetic wave as an oscillating dipole with the eigen frequency $\omega_0$
driven by a periodic external force of frequency $\omega$. The general solution of this problem can
be written as

$$X(t) = A \exp(-\gamma t - i\omega_0 t) + B \exp(-i\omega t),$$

(1)

where $\gamma$ is the damping constant and $A$ and $B$ are the constants to be determined from
the initial conditions. Here the first term describes transient behavior whereas the second term
gives a steady-state solution at large times, $t \gg \gamma^{-1}$. An oscillating dipole emits radiation at
the frequency of its oscillations and therefore one expects two components of frequencies $\omega_0$ and
$\omega$ in the emission spectrum. A very similar situation occurs when one resonantly excites gas
atoms colliding with a bordering surface. Some of them which move to the surface are in the
external field for a long time and hence experience a steady-state excitation. They contribute
only to the emission line at the frequency $\omega$. The others which have been just desorbed from
the surface are out of resonance with the external field due to the atom-surface frequency shift (Figure 1). They pass the range of the surface potential in time much shorter than the relaxation
times and therefore get almost instantaneously excited at the moment of desorption. Such atoms
contribute to both emission lines, $\omega_0$ and $\omega$. The same conclusions can be obtained on the basis
of a rigorous density matrix approach.

The intensity of the transient contribution is essential, however, at distances $z \leq l_2 \sim v_T/\gamma$
from the surface with $v_T$ the most probable thermal velocity of gas atoms. On the other hand,
the intensity of the steady-state component is proportional to the gas volume illuminated by an electromagnetic wave. Thus taking the EW penetration depth so that $\delta \ll l_2$ one ensures that the transient contribution will be dominant in the spectrum. Such a situation was realized experimentally for fluorescence spectra of sodium atoms excited by an EW nearly resonant to the $3S_{1/2} \rightarrow 3P_{3/2}$ transition [3]. The spectrally resolved emission spectrum shown in Figure 2 contains two lines. The narrow line is due to the EW scattering at surface roughness, whereas the broader line represents fluorescence of Na atoms. The width of the latter line correlates with the prism temperature thus confirming that it originates from the desorbed atoms.

Figure 1. Schematic representation of the atom-surface interaction potential in the ground $|g\rangle$ and excited $|e\rangle$ atomic states. The electromagnetic field of frequency $\omega$ is in resonance with the atomic transition far from the surface and out of resonance with the transition of an adsorbed atom.

Figure 2. Spectrally resolved fluorescence of Na atoms excited in the evanescent wave at a glass prism surface. Reprinted from [3], Copyright 1997, with permission from Elsevier.

3. Excitation by counterpropagating evanescent waves

Two-photon fluorescence spectroscopy provides a much better signal-to-noise ratio as compared to the one-photon excitation scheme. Due to the high quality of the signal, this kind of spectroscopy allows one to study the fluorescence line shapes in detail. The two-photon excitation has an additional advantage when being applied in the geometry of counterpropagating waves. In this case, the overall Doppler broadening is determined by the quantity $v_T|\omega_1 - \omega_2|/c$. If the wave frequencies, $\omega_1$ and $\omega_2$, coincide or are close to each other the residual Doppler broadening can be negligible. This has allowed to resolve the hyperfine structure of the $D$-lines in the reflection spectrum from sodium vapor excited by EWs at a glass prism surface [4]. In such a case, at low gas pressures where the homogeneous linewidth is small the broadening of fluorescence lines is dictated mainly by the inverse times of flight across the EW fields, $v_z/\delta_1$ and $v_z/\delta_2$. As a result, one can expect the sensitivity of the fluorescence line shape to the velocity distribution function over $v_z$ provided that the quantities $\delta_1$ and $\delta_2$ are small enough.

This conclusion was demonstrated in the experiment on two-photon fluorescence of sodium atoms excited by counterpropagating EWs at a glass prism surface [5]. The prism was mounted inside a vacuum chamber. Sodium atoms from the dispenser were directed in the plane of incidence
of the laser beam toward the prism surface (Figure 3). One of the lasers exciting EWs was set in resonance with the $3P_{3/2} \rightarrow 5S_{1/2}$ transition whereas the second laser was scanned across the adjacent $3S_{1/2} \rightarrow 3P_{3/2}$ transition. The resulting fluorescence light from the $4P_{1/2,3/2} \rightarrow 3S_{1/2}$ transitions was observed as a function of detuning of the second laser. Due to the Doppler shift, the exciting EWs were out of resonance with the atomic flux arriving at the surface and thus its contribution to the fluorescence spectrum was negligible. The same is true for the atoms specularly scattered by the surface. The observed spectrum, therefore, originated mainly from sodium atoms desorbed from the surface.

The intensity of fluorescence originated from the desorbing flux is determined by the quantity $N_e/\tau$ with $N_e$ the total number of desorbed atoms in the excited state and $\tau$ their radiative decay lifetime. In a steady-state excitation regime the number of excited atoms can be found as

$$N_e = J \int_{v_z>0} \int_0^\infty n_e(v,t) f(v) dv dt,$$

where $J$ and $f(v)$ are the total desorbing flux and its velocity distribution function, respectively, and $n_e$ is the population of the excited state. Here, the time $t$ in the integrand is counted beginning with the moment of desorption. According to Knudsen’s cosine law $f(v) \sim v_z = v \cos \theta$ with $\theta$ the angle between the velocity vector and the surface normal. Figure 4 represents the two-evanescent-wave fluorescence spectrum obtained by tuning the frequency of the second laser across the $3S_{1/2}(F=1) \rightarrow 3P_{3/2}(F')$ transitions. Spectra calculated with different velocity distribution functions are also shown. The best fit is obtained assuming the cosine law for desorption ($f_K$), whereas the curve calculated with the Maxwellian distribution function, $f_M$, implying isotropic desorption, clearly does not describe the observed line shape.

![Figure 3](image1)

**Figure 3.** Schematic drawing of the experiment for the study of two-photon evanescent-wave fluorescence from sodium atoms.

![Figure 4](image2)

**Figure 4.** Pair of the hyperfine components (denoted by arrows on top) in the two-evanescent-wave fluorescence spectrum observed from Na atoms desorbing from a glass surface. The lines represent the theoretical spectra calculated with the velocity distribution functions $f_K$ (thick solid line) and $f_M$ (thin solid line). Reprinted with permission from [5]. Copyright 1999 by the American Physical Society.
4. Temperature dependence of fluorescence spectra

Gas spectra excited by EWs can also be influenced by atoms which are adsorbed on the surface. The adsorbate overlayer determines the boundary conditions for Maxwell’s equations at the interface and, therefore, also the EW amplitude in the gas. As a result, the gas optical spectra which depend on the EW intensity, contain indirectly information about the adsorbed layer. In particular, the atoms adsorbed on the surface are polarized by the exciting field, \( E_{i0} e^{-i\omega t} \), and thus create a two-dimensional displacement current in the surface plane

\[ j(t) = \frac{\partial P_s}{\partial t}. \]  

(3)

For small surface coverages, when the effect of the local field can be neglected, the adsorbate polarization, \( P_s \), is expressed in terms of the adatom dynamic polarizability, \( \alpha_s(\omega) \), as

\[ P_s(t) = N_s \alpha_s(\omega) E_{i0} e^{-i\omega t}. \]  

(4)

Here, \( E_{i0} \parallel \) is the tangential component of the electric field amplitude at the interface and \( N_s \) is the surface number density of adsorbed atoms. It is also assumed that the dielectric response of the adsorbed atoms is isotropic in the surface plane. Accounting for the surface current (3) in the boundary condition for the tangential components of the magnetic field results, in the case of \( s \)-polarization, in the expression for the EW amplitude

\[ E_{i0} = \frac{2\sqrt{\epsilon_1} \cos \theta_i}{\sqrt{\epsilon_1 \cos \theta_i + \sqrt{1 - \epsilon_1 \sin^2 \theta_i + 4\pi i(\omega/c)^2 N_s \alpha_s}}} E_{i0}, \]  

(5)

where it is assumed that \( \theta_i \) exceeds the critical angle. It relates the EW amplitude to the surface coverage, \( \theta \), defined in terms of \( N_s \). In the framework of the Langmuir model of adsorption the quantity \( \theta \) can be related to the surface temperature. Therefore, measuring the EW fluorescence spectra from the gas as a function of surface temperature allows one to obtain information on the adsorbate.

Figure 5. Two-evanescent-wave fluorescence spectra of sodium atoms excited near the critical angles. Each line exhibits Autler-Townes splitting. Reprinted from [6].

Figure 6. a) Dependence of the measured Autler-Townes splitting on the prism temperature for fixed EW intensity. b) Fit to the experimental data, resulting in the EW amplitude vs. surface temperature. Reprinted from [6].
An experimental realization of this idea was demonstrated for adsorption of sodium atoms at a glass prism surface in the setup described in the preceding section (see Figure 3) [6]. Both EWs were excited near the critical angle in order to increase the EW penetration depths and thus to decrease the time-of-flight broadening. In this regime, the lines in the two-photon fluorescence spectrum split into two if one of the EWs saturates the lower transition (Autler-Townes splitting, Figure 5). The value of the line splitting is proportional to the amplitude of this EW. Figure 6 demonstrates that the splitting increases at fixed EW intensity as one increases the temperature of the prism surface while continuously evaporating Na atoms from the dispenser. At relatively high surface temperatures \( T > 240 \) K the splitting reaches a steady-state value indicating that the equilibrium surface coverage tends to zero. Comparison of the theoretical curves with the measured fluorescence spectra allows one to deduce the EW amplitude corresponding to a given surface temperature. Then, using Eq. (5), one can calculate the EW amplitude normalized to its value at high surface temperatures where \( \theta \ll 1 \). A plot of this quantity versus \( 1/T \) provides the binding energy of Na atoms on a glass surface, \( E_b = 0.80 \) eV. Further fitting of the dependence shown in Figure 6 results also in a value of the polarizability of sodium adatoms, namely \( \alpha_s = 6.6 \cdot 10^{-22} \) cm\(^3\).

5. Excitation by crossed waves

An extremely small EW penetration depth into the gas permits to excite gas atoms only within the Knudsen layer with thickness of the order of the mean free path. If, in addition to the EW wave, one excites the boundary gas layer by an electromagnetic wave propagating normally to the surface one can distinguish between the contributions of atoms moving to the surface and away from it by tuning the laser frequency either below the atomic transition frequency or above it. Therefore simultaneous excitation of gas atoms by two crossed waves, one of which being evanescent, enables one to separate spectrally the contributions of the atoms immediately before the collision with the surface from those immediately after the surface scattering. The Doppler-broadened two-quantum fluorescence lineshapes are then determined by the velocity distribution functions of the atoms approaching the surface and departing from it and thus contain information about the gas-surface scattering dynamics.

![Figure 7](image-url)

**Figure 7.** (a) "Normal configuration" for studying scattering of sodium atoms at a glass surface. The pump laser excites a wave propagating normally to the prism surface which is resonant to the lower transition in the three-level cascade scheme. The probe laser excites an evanescent wave which scans across the upper transition. (b) "Inverse configuration". Here, the pump laser excites an evanescent wave, whereas the probe laser excites a wave propagating into the gas interior.

The experimental setup for studying gas-surface scattering under those conditions implements a truncated prism to allow normal incidence of one of the beams onto the interface as well as excitation of an EW with the other beam. In the experimental realization [7] continuous wave lasers excited two adjacent transitions in Na atoms. In one excitation geometry (the "normal configuration"), the pump laser beam resonant to the \( 3S_{1/2} \rightarrow 3P_{3/2} \) transition excited a wave normally incident onto the interface, whereas the probe (signal) laser beam excited an EW...
scanned across the $3P_{3/2} \rightarrow 5S_{1/2}$ transition [Figure 7(a)]. In the other excitation geometry ("inverse configuration") the character of the waves was exchanged [Figure 7(b)]. The atomic flux which was directed from the sodium source to the prism surface determined the velocity distribution function of the atoms arriving at the surface.

Typical two-photon fluorescence spectra obtained in the normal configuration are shown in Figure 8. For negative detunings of the pump beam, $\Delta_p$, a narrow peak is observed besides the Doppler-broadened line. This peak arises from the atomic flux emanating directly from the sodium source. The broader line originates from atoms arriving at the surface, too, but these atoms had undergone mutual collisions in front of the surface. For $\Delta_p > 0$ only atoms departing from the surface contribute to the spectrum. In the inverse configuration (Figure 9), the left and right wings of the spectrum arise from atoms arriving at the surface and departing from it, respectively.

Qualitatively the difference of the spectra obtained in the inverse as compared to the normal configuration can be explained as follows. The fluorescence intensity at a given probe beam detuning, $\Delta_s$, is proportional to the number of atoms for which the probe beam is resonant, i.e. $\Delta_s - k_s \cdot v = 0$ with $k_s$ the wave vector of the probe wave. In the normal configuration ($k_s$ parallel to the surface) one scans the distribution function of atomic velocities along the surface by varying $\Delta_s$. In contrast, in the inverse configuration ($k_s$ perpendicular to the surface) the
Figure 9. Two-quantum fluorescence spectra of sodium atoms observed in the "inverse configuration". \( \Delta_p = 200 \text{ MHz} \). The notations are the same as in Figure 8. Reprinted with permission from [7]. Copyright 2001 by the American Physical Society.

Figure 10. The net contribution to the fluorescence spectrum from the sodium atoms scattered by the surface (dots). The calculated spectrum of the desorbed atoms and the best fit obtained for \( \eta = 0.05 \) are shown by the thin gray and black lines, respectively. \( \Delta_p = 800 \text{ MHz} \). Reprinted with permission from [8]. Copyright 2001 by the American Physical Society.

A careful analysis of the fluorescence spectra obtained in the normal configuration permits extraction of additional important details of the gas-surface scattering process. We have seen that the contribution of the atoms departing from the surface can be separated for positive pump beam detunings at the lower atomic transition \( 3S_{1/2}(F = 2) \rightarrow 3P_{3/2}(F') \). However, for the close transition from the other hyperfine sublevel \( 3S_{1/2}(F = 1) \rightarrow 3P_{3/2}(F') \) \( \Delta_p \) is negative. That leads to a small contribution to the spectrum from the atoms approaching the surface. Subtracting this contribution gives the spectrum represented in Figure 10. Here, the calculated fluorescence line shape, calculated assuming that all atoms moving from the surface have been desorbed from it (thin gray line), is also shown. A clear discrepancy between measurement and fit is observed, which can be attributed to the atoms which were scattered without being trapped at the surface, i.e., via a direct scattering channel. The velocity distribution of such atoms can be expressed in terms of the scattering kernel \( R \), the fundamental quantity summarizing the basic information on the gas-surface interaction. It relates the velocity distribution function of atoms arriving at the surface, \( f^-(\mathbf{v}) \), with that of atoms scattered by the surface, \( f^+(\mathbf{v}') \), by means of the integral equation
\[ v'_z f^+(v') = \int R(v \rightarrow v')|v_z|f^-(v)dv. \tag{6} \]

Fitting the corresponding signal using an appropriate model for the scattering kernel allows one to obtain the mean energy transfer to surface phonons as well as the mean probability of direct scattering, \( \eta \) [8].

6. Some prospects

The idea of application of evanescent waves for the investigation of gas-solid interface can be also fruitful being exploited in the other nonlinear spectroscopic techniques. We shall consider here what additional information can be extracted from four-wave-mixing (FWM) and Raman spectra excited by electromagnetic fields localized at the interface.

Assume that the ground electronic state of a gas molecule has sublevels \(|g\rangle\) and \(|g'\rangle\), and the excited electronic state has sublevels \(|e\rangle\) and \(|e'\rangle\) [Figure 11(a)]. Consider a set up with a truncated prism as above. Let the gas molecules be excited by two laser beams of frequencies \(\omega_1\) and \(\omega_3\) travelling perpendicularly to the prism surface and by an EW of frequency \(\omega_2\) propagating along it [Figure 11(b)]. The laser frequencies are assumed to be in resonance with the molecular transitions as it is shown in Figure 11(a). Then the process of wave mixing will lead to generation of a wave of a new frequency \(\omega_m = \omega_1 - \omega_2 + \omega_3\) which will be observed in the direction of phase matching determined by the angle \(\theta_0\) which satisfies the equation

\[ \sin \theta_0 = -\frac{\omega_2}{\omega_m} \sin \theta_i \sin \theta_c \tag{7} \]

with \(\theta_i\) the incidence angle of the laser beam exciting EW and \(\theta_c\) the critical angle.

Let us assume now that both the frequencies \(\omega_1\) and \(\omega_3\) are red detuned from the transitions \(|g\rangle \rightarrow |e\rangle\) and \(|g'\rangle \rightarrow |e'\rangle\), respectively. Then to the four-mixing process will contribute only those molecules which have been excited at the flight towards the surface and kept their nonlinear polarization after the molecule-surface scattering. It is reasonable to assume that the molecules which have been adsorbed on the surface for a long time are desorbed with completely destroyed polarization. Thus only directly scattered molecules will contribute to the FWM signal. It can be shown that in such a case the FWM spectrum measured by scanning the frequency \(\omega_1\) can be expressed in terms of the scattering kernel [9]. This allows one to calculate also the velocity-dependent sticking probability.

The difference in the optical response of molecules approaching the surface and desorbing from it can be used also for studying surface chemical reactions. Under steady-state conditions the decrease of the molecular flux desorbing from the surface as compared to that arriving at it is directly related to the surface reaction rate [10]. Let us assume that the reactant molecules
approaching the metal surface are excited by a powerful surface plasmon (SP) which saturates a vibrational transition in the molecules. Then the molecules arrive at the surface with almost equally populated ground and excited vibrational levels. After getting adsorbed the molecules have a certain probability to either enter the surface reaction or to thermally desorb being in the ground vibrational state. Suppose now that one probes the vibrational state populations by exciting a SP in the visible spectral range and by monitoring the corresponding Raman scattering intensity. Due to extremely small penetration depth of the visible SP into the gas ($\leq 1 \mu m$) it probes the vibrational state populations of the molecules in the immediate vicinity of the surface. As it follows from the above argumentation, the molecules approaching the surface will almost equally contribute to the intensities of the Stokes and anti-Stokes components. In contrast, the molecules desorbing from the surface will only contribute to the Stokes component and the magnitude of their contribution will be determined by the surface reaction probability. Thus by measuring the ratio of the Raman components one can extract this parameter which in turn is related to the reaction rate.

7. Conclusion
We have reviewed nonlinear optical spectroscopic techniques exploiting electromagnetic fields strongly localized at the gas-solid interface. A rapid decrease of the field amplitude with distance from the surface allows one to excite only gas atoms or molecules in the immediate vicinity of the surface. Due to essentially transient optical response of gas particles departing from the surface, their spectral contribution is distinct from the contribution of particles arriving at the surface. Another opportunity to distinguish between gas atoms moving to the surface and departing from it is provided by the Doppler effect in the field of the wave travelling along the surface normal. In both cases the comparison of spectra arising from atomic fluxes to and away from the surface allows to extract information on the dynamics of gas-surface scattering.

The considered examples do not exhaust the possibilities of evanescent wave spectroscopies. They can be extended to gas interfaces to metals and ionic crystals by means of excitation of surface plasmon polaritons and surface phonon polaritons, respectively. A very small penetration depth of EWs allows, in principle, to apply these techniques right up to atmospheric gas pressure. The atoms excited in the close vicinity of a surface can serve as a spectroscopic tool for studying local surface fields as well as heterogeneous relaxation at a gas-solid interface. Finally, nonlinear evanescent wave spectroscopies can be used to monitor atomic transport near surfaces.

Acknowledgments
One of us (VGB) is grateful to W. Allison for the helpful and stimulating discussion.

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