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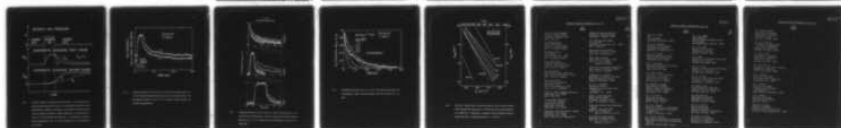
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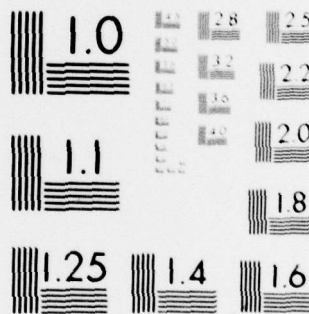
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by

G. W. Rubloff

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**PHOTOEMISSION STUDIES OF TIME-RESOLVED SURFACE REACTIONS:
ISOTHERMAL DESORPTION OF CO FROM Ni(111)*†**

G.W. Rubloff

**IBM Thomas J. Watson Research Center
Yorktown Heights, NY 10598**

ABSTRACT

A new dynamic approach to spectroscopic studies of surface reactions is described. A fast-acting UHV gas dosing system is employed to prepare the adsorbed species in an initial chemisorption state under reaction conditions of elevated temperature and/or pressure (to ~ 1 torr). Individual reaction steps are then resolved in time using photoemission spectroscopy to probe the surface electronic structure, yielding information about the kinetics of individual reaction steps, the nature of chemisorbed species, and the surface reaction path. As a first application of this approach, the kinetics of isothermal desorption of CO from Ni(111) is studied for temperatures up to ~ 230 °C. The coverage-dependence of the rate constants is revealed directly in the shape of each desorption curve. Preexponential (frequency) factors as well as desorption activation energies show significant variation with coverage.

* Supported in part by the Office of Naval Research.

† Presented at the Second European Conference on Surface Science. Cambridge, England, March 26- 29, 1979.

I. Introduction

Although they have provided much valuable information, spectroscopic studies of surface reactions usually do not directly address two important questions. First, what is the nature of chemisorbed species and reaction pathways under conditions of elevated temperature and/or reactant gas pressure? Some information on this question is gained by the use of annealing cycles to drive the reaction through various steps.¹ Second, what are the detailed kinetics of individual surface reaction steps? Deconvolution of lineshapes obtained from thermal conversion spectroscopy^{2,3} (or thermal desorption spectroscopy for reaction steps involving desorbing products) has been the most widely used (but not the only) technique to obtain kinetic parameters.

A new dynamic approach to spectroscopic studies of surface reactions is described here. By time-resolving individual reaction steps in an isothermal measurement, it presents the possibility of addressing these two questions more directly and effectively. Preliminary results are given for a first application of this approach --- to the kinetics of isothermal desorption of CO from Ni(111).

II. Time-Resolved Approach

This time-resolved approach is illustrated schematically in the example depicted in Fig. 1. With the sample held at elevated (reaction) temperature, the clean surface is exposed briefly to the reactant gas using a fast-acting UHV gas dosing system. At the end of the gas dose, an initial chemisorbed phase is present on the surface, at some coverage (e.g. saturation) and possibly having been formed at elevated pressures. Spectroscopies such as ultraviolet photoemission (UPS) are then used to monitor the time-evolution of surface species, which proceed through various reaction steps with conversion rates determined by temperature, coverage, pressure, etc. The UPS measurements provide two potential sources of information to answer two different kinds of questions:

1. Spectral dependence. By measuring the UPS spectrum of the surface species within some time window, one can determine the nature of chemisorbed species formed under these reaction conditions after a particular reaction time. The chronological sequence of species observed reveals the surface reaction path.

2. Time dependence. By monitoring the time-evolution of parts or all of the UPS spectrum for a given surface species, the kinetics (rate constants) of the preceding or following reaction step are measured. Activation energies Δ and pre-exponential (frequency) factors K_0 for these rate constants are then deduced from the temperature dependence of these rate constants $K(T) = K_0 \exp(-\Delta/kT)$. Clearly it is desirable to measure the kinetics over as wide as possible a range of sample temperatures (or equivalently, reaction times).

In this approach, time-resolution of surface reaction steps is achieved by use of the fast-acting gas dosing valve. It is a UHV bakeable device fully compatible with the vacuum quality of the surface spectroscopy system. The valve is differentially pumped and consists of multiple stages of blades which either cover or leave open a series of small apertures between the gas load chamber and the sample. It is switched on (open) or off (closed) by a fast-acting solenoid outside the vacuum, which drives a bellows and thereby the internal moving parts. The switching time of the valve is 2-3 msec, so that exposure times as short as a few msec are readily achieved, with adjustable duty cycle. The dynamic range of measurable reaction times thus extends to potentially 10^6 , from $\sim 10^{-2}$ sec to $\sim 10^4$ sec, the time for significant contamination in the UHV system. The rapid switching time of the gas dosing system thus provides access to a much broader dynamic range of reaction times than temperature-step isothermal measurements usually do.

The conductance of the valve is ~ 3 ml/sec on and $\sim 3 \times 10^{-3}$ ml/sec off. Since the local pressure P_{samp} at the sample surface is determined by gas flow through the valve in either position, $P_{\text{samp}}^{\text{on}}/P_{\text{samp}}^{\text{off}} \approx 10^3$. Furthermore, the actual valve portion of the dosing system is

located close to the sample, with a short nozzle after the valve to direct the gas onto the surface at the focal point of the electron spectrometer; this produces an enhancement of P_{samp} compared to the residual gas pressure P_{sys} in the UHV chamber so that $P_{\text{samp}}/P_{\text{sys}} \approx 10^2$, independent of valve position. An auxiliary solenoid-operated valve makes it possible to pump out the gas load chamber quickly (< 0.5 sec) when measurements are to be done for reaction times much longer than the dosing time. As a result, the gas exposure during the on time can be kept much greater ($\sim 100\times$) than the accumulated exposure during the off time.

The enhancement $P_{\text{samp}}/P_{\text{sys}} \approx 10^2$ and the large system volume (~ 150 liters) make it possible to inject a brief dose of gas at relatively high pressure (~ 1 torr) while maintaining $P_{\text{sys}} \leq 5 \times 10^{-5}$ torr; under such conditions the electron multiplier used to measure photoelectron intensity can be left on without damage. In this way, electron spectroscopy measurements can be made immediately following the dose to study adsorbed phases formed at reaction pressures much closer to those of practical interest.

III. CO/Ni(111) Desorption Kinetics

A number of methods have been employed to measure desorption kinetics,^{3,4} including temperature-programmed desorption,^{5,6} equilibrium adsorption isosteres,^{3,7} and in a few instances isothermal desorption.⁸ In most cases the coverage-dependence of the desorption activation energy Δ is obtained while "normal" coverage-independent preexponential factors $K_0 \sim 10^{13} \text{ sec}^{-1}$ are assumed. Deconvolution techniques to extract both Δ and K_0 are known.³⁻⁶ In a recent study³ of CO desorption from Ru(001) which combined all three approaches, Pfnür et. al. demonstrated that both Δ and K_0 have significant coverage-dependence.

As an initial application for this time-resolved reaction study approach, the kinetics of desorption of molecularly chemisorbed CO from Ni(111) have been measured using the time-dependence of the UPS intensity; no attempt has been made here to study the spectral

dependence to search for different chemisorbed species or reaction pathways characteristic of elevated temperature and/or pressure. The isosteric heat of adsorption for CO/Ni(111) has previously been determined from equilibrium adsorption isosteres ($\log p_{\text{CO}}$ vs. T^{-1}) at various coverages (measured by work function change $\Delta\phi$) by Christmann, Schober, and Ertl (CSE).⁷ These measurements were carried out over the temperature range $23 < T < 164^\circ\text{C}$; the accumulation of surface carbon produced by the disproportionation reaction $2\text{CO} \rightarrow \text{C}_{\text{ads}} + \text{CO}_2\uparrow$ prevented such measurements above 164°C . Their results show a coverage-dependent activation energy for desorption which varies from ~ 30.0 kcal/mole (~ 1.30 eV) at low coverage (θ) to ~ 23.5 kcal/mole (~ 1.02 eV) near saturation coverage. A normal preexponential factor $K_0 = 1. \times 10^{13} \text{ sec}^{-1}$ was assumed.

The intensity of the UPS signal (using He I radiation, 21.2 eV) at the $5\sigma/1\pi$ adsorbate orbital (~ 8 eV below the Fermi energy) was taken as proportional to the coverage of chemisorbed CO. This assumption is supported by the correspondence between the exposure-dependence of the UPS signal and the detailed adsorption results for this system obtained by CSE⁷.

The time-dependence of the UPS signal for sample temperature $T = 175^\circ\text{C}$ is shown in Fig. 3 for a 200 msec dose after a 100 msec delay (curve a) and then for a 300 msec dose after a similar delay (curve b).⁹ The CO coverage (essentially proportional to the UPS signal) saturates in ~ 50 msec, as seen clearly by comparing the two curves. Fairly rapid desorption (in ~ 250 msec) is then observed following the dose because the sample temperature is near that of the flash desorption peak. Both curves in Fig. 2 represent averages of 13 successive runs. The amplitude of the UPS intensity change (coverage change) is somewhat smaller in the second set because the disproportionation reaction has poisoned (filled) some of the surface sites for CO chemisorption at this temperature. Because a complete desorption curve can be obtained even for a single dosing cycle, this time-resolved approach is viable even when competing reactions (like disproportionation above $\sim 165^\circ\text{C}$ in this case) occur with compara-

ble rates. This represents a significant advantage for studies of the desorption reaction compared to the adsorption isostere approach, in which a single measurement involves many adsorption-desorption cycles.

Similar data showing the temperature-dependence of CO desorption rates from 122°C to 227°C is seen in Fig. 3. A change in desorption rate is clearly observable for only ~ 10°C change in sample temperature.

The coverage-dependence of the desorption rate constants can be seen directly from the shape of the desorption curve at a single fixed temperature, as illustrated in Fig. 4 for $T = 162$ °C. Here the experimental data is shown together with calculated desorption curves

$$\theta(t) = \theta_0 e^{-K_{DES}t} = \theta_0 e^{-t/\tau_{DES}} \quad (1)$$

assuming first-order desorption (desorption rate constant K_{DES} coverage-independent).

Clearly the measured curve is not simply a single exponential, but instead K_{DES} changes with coverage as well as temperature.

The temperature-dependent desorption rate constant is given by

$$K_{DES}(T) = K_0 e^{-\Delta/kT} \quad (2)$$

where Δ is the activation energy for desorption ($\approx$ heat of chemisorption) and K_0 is the pre-exponential (frequency) factor. To determine the coverage-dependence of Δ and K_0 , the coverage-dependent desorption rate constants have been calculated from the desorption curves as

$$K_{DES}(T, \theta) = - \frac{1}{\theta} \frac{d\theta}{dt} \quad (3)$$

Typical errors in these K_{DES} values are ± 10 -20%. In the simplest analysis, the resulting

Arrhenius plots (K_{DES} vs. $1/T$) can be fit by a straight line in order to determine Δ and K_0 for each coverage value.

Such straight line fits are used to represent the actual data in the Arrhenius plots shown for various coverages in Fig. 5. These results should be regarded as preliminary, and a more complete analysis and discussion will be published elsewhere. For comparison, the dashed lines show the predictions from the equilibrium adsorption-isostere results of CSE⁷, assuming a constant $K_0 = 1. \times 10^{13} \text{ sec}^{-1}$. At this stage, several features seem clear:

1. Δ decreases with θ , in agreement with the results of CSE⁷; however, somewhat smaller Δ values (by $\sim 20\%$) are found here.
2. K_0 as well as Δ changes significantly with θ , as found for CO/Ru(001)³.
3. K_0 values are significantly smaller (by a factor $\sim 10^2 - 10^3$) than "normal" values.
4. The θ -dependence of K_{DES} becomes weaker at higher temperature.
5. The variations of Δ and K_0 with coverage tend to cancel each other, resulting in desorption rate constants K_{DES} which are much less sensitive to coverage than the variation in either K_0 or $\exp(-\Delta/kT)$ alone.

IV. Conclusions

Similar to the case³ of CO/Ru(001), the present results show that both the desorption activation energy Δ and the preexponential (frequency) factor K_0 change markedly with coverage, although the resulting desorption rate constants K_{DES} are less sensitive to coverage. A reasonably direct and accurate determination of both parameters and their coverage-dependence appears necessary for a useful understanding of the kinetics of individual surface reaction steps, especially in more complicated reactions in which these details may in fact alter the predominant reaction pathway in different temperature regimes.

As illustrated in the present example of CO desorption from Ni(111), the time-resolved reaction study approach described here provides a way to directly measure the kinetics of individual surface reaction steps. This can be done even if the step involves no desorption products. It can be employed even in the presence of competing reactions. Furthermore, it gives a way to investigate the surface reaction at elevated temperature and/or pressure (closer to reaction conditions) and in the future to reveal the nature of chemisorbed species and reaction pathways under such conditions.

V. Acknowledgement.

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1. See, e.g., G.W. Rubloff and J.E. Demuth, J. Vac. Sci. Technol. 14, 419 (1977).
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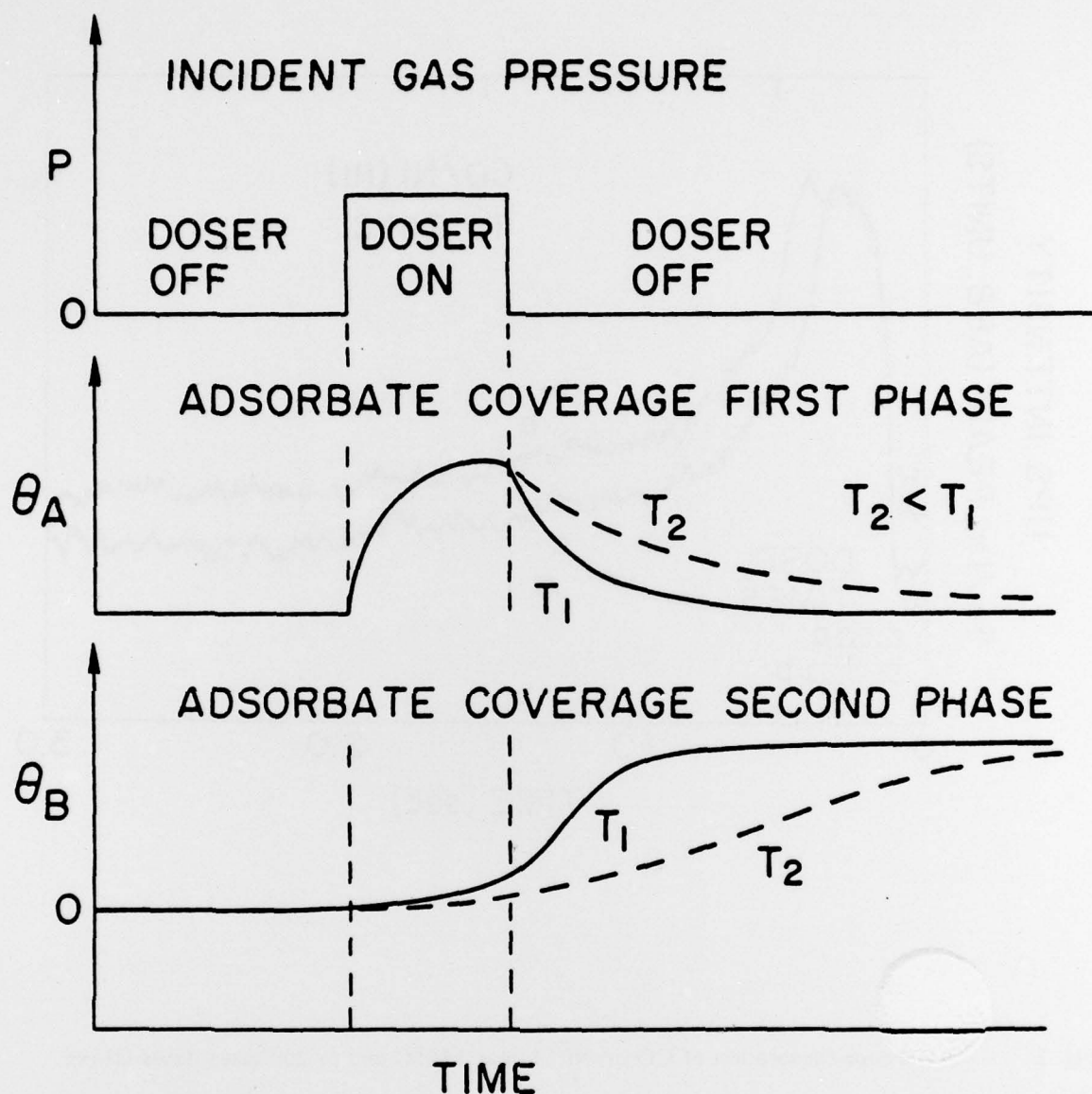


Fig. 1 Schematic example of time-resolved surface reaction. (a) Gas dosing system produces exposure during doser on-time. (b) This produces coverage θ_A of initial adsorbate phase (possibly up to saturation). θ_A then decays in time after the dose with rate determined primarily by sample temperature T_1 as surface species A converts to second adsorbate phase species B. (c) Coverage θ_B of species B grows correspondingly in time. At a lower temperature $T_2 < T_1$, this conversion time is longer.

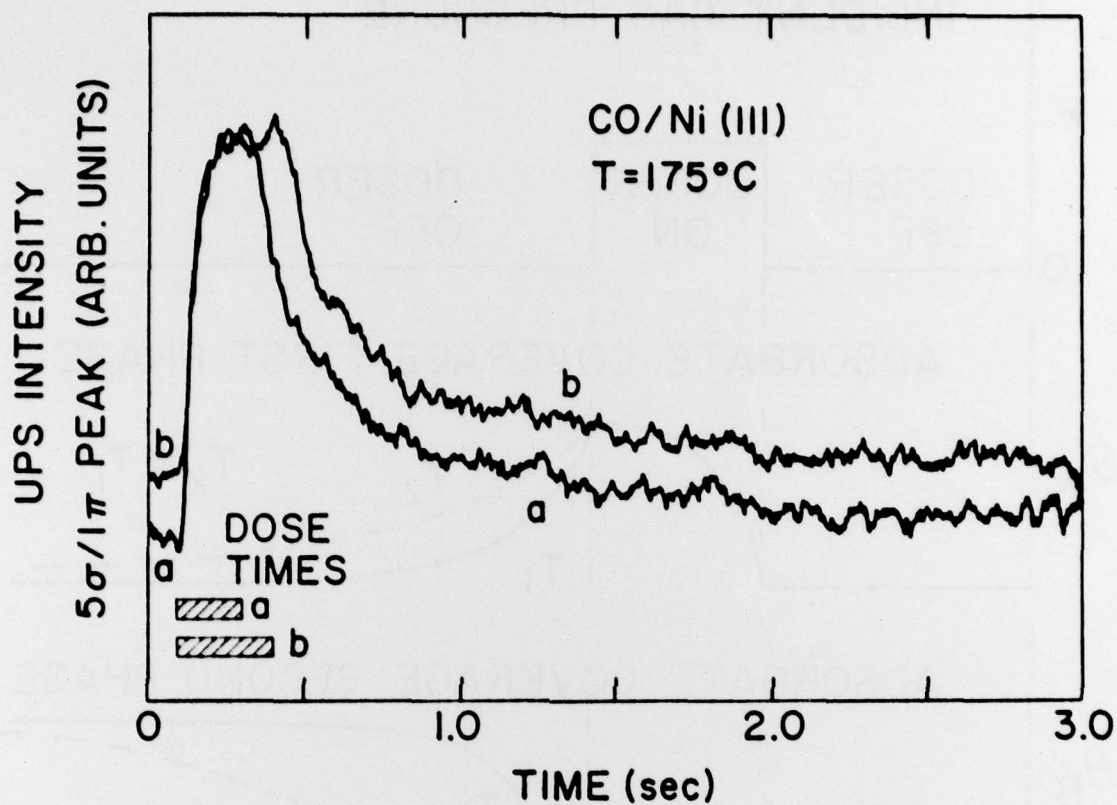


Fig. 2 Adsorption/desorption of CO on Ni(111) at 175°C due to 200 msec doses (curve a) after 100 msec delays and 300 msec doses (curve b) after similar delays. The photoemission intensity of the CO 5σ/1π molecular orbitals represents the coverage of chemisorbed CO.

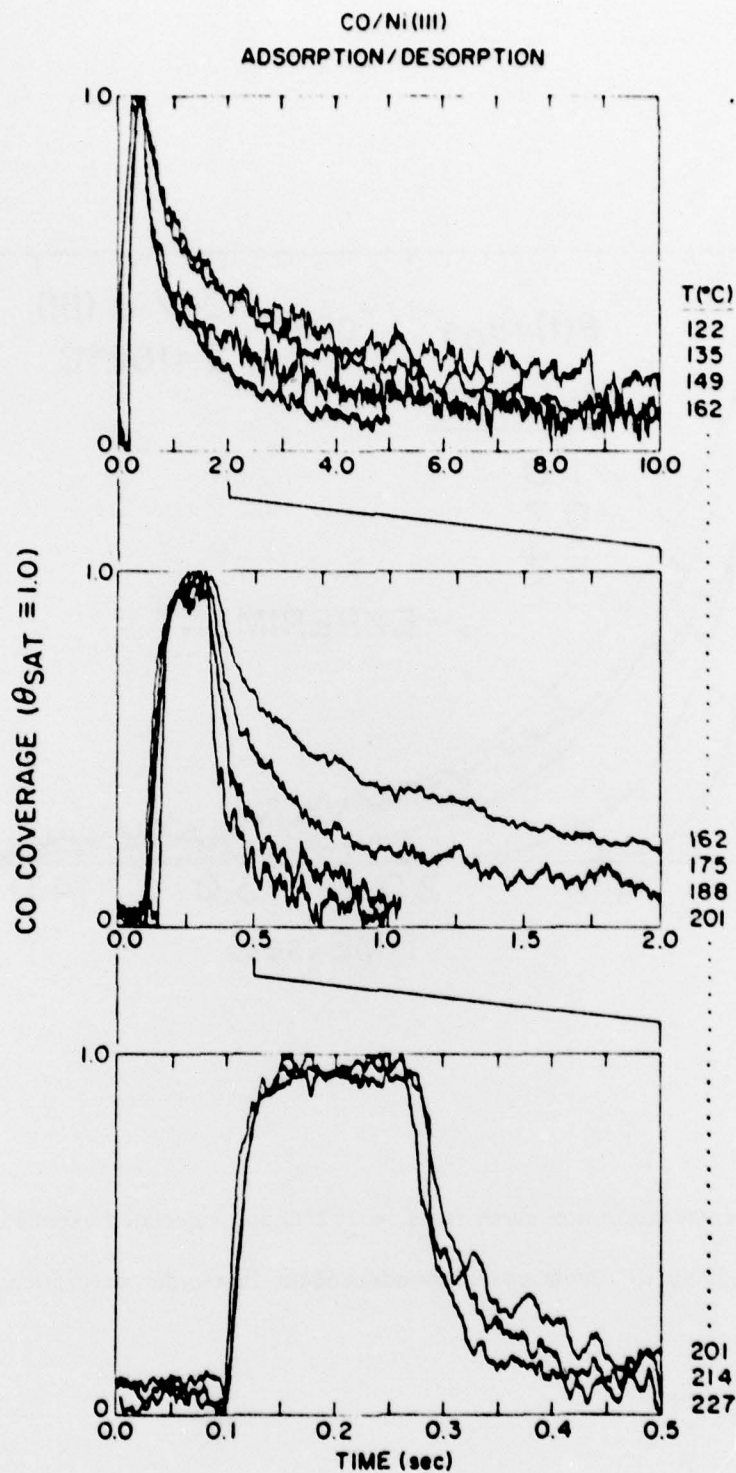


Fig. 3 Adsorption/desorption of CO on Ni(111) at various temperatures due to 150 msec dose after 100 msec delay. The CO coverage (with saturation coverage defined as $\theta_{\text{SAT}} = 1.0$) is obtained from the UPS intensity at the CO $5\sigma/1\pi$ orbital peak.

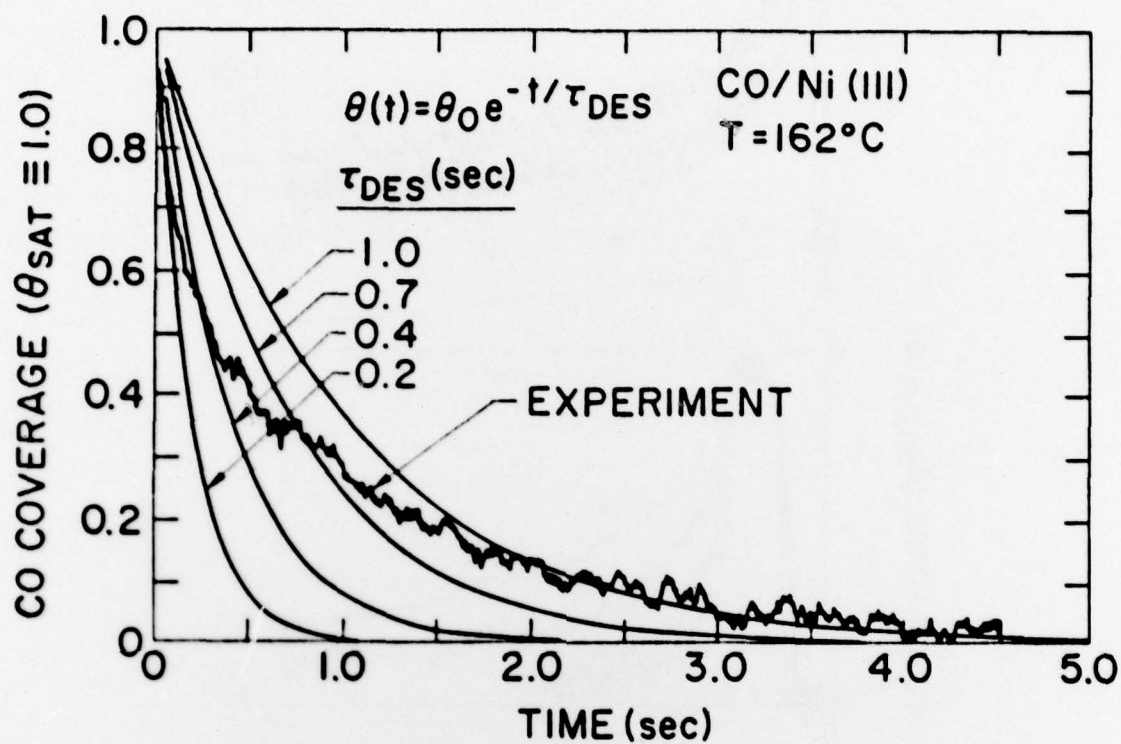


Fig. 4 Experimental desorption curve for $T = 162^\circ\text{C}$ and calculated exponential curves corresponding to simple coverage-independent first-order desorption rate constants.

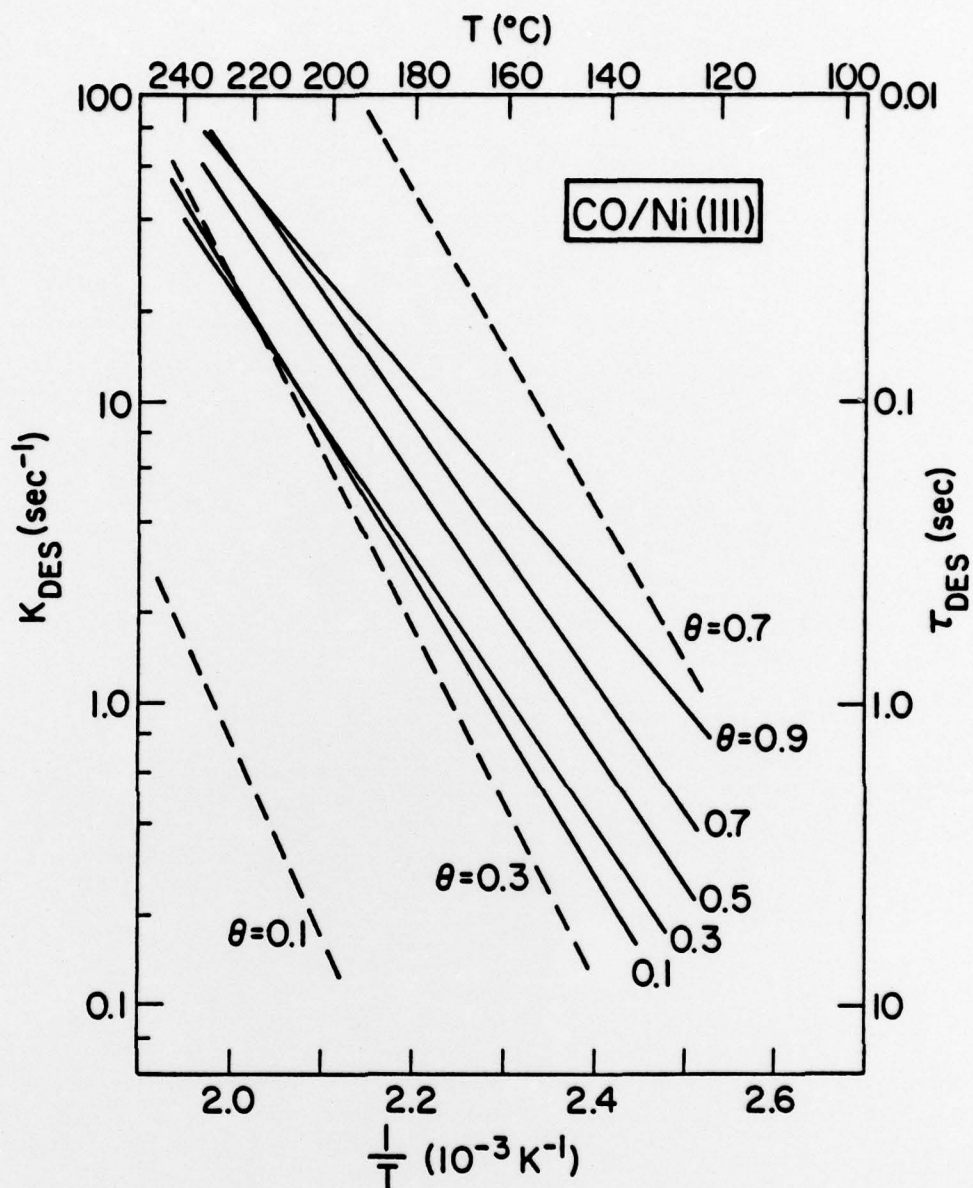


Fig. 5 Solid lines: Arrhenius plots of measured desorption rates at various coverages, with the detailed data (K_{DES} errors $\sim \pm 10\text{-}20\%$) for each coverage represented as a straight line fit. Dashed lines: predictions from the equilibrium adsorption isostere data of Ref. 7, assuming constant $K_0 = 1. \times 10^{13} \text{ sec}^{-1}$.

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