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Catalysis and Surface Science

Modern surface physics is transforming the black art of catalysis, revealing a fascinating choreography followed by reacting atoms and molecules.

Gerhard Ertl and Hans-Joachim Freund

In 1835 the Swedish chemist Jöns Jakob Berzelius coined the term "catalysis" to describe chemical reactions in which progress is affected by a substance that is not consumed in the reaction and hence is apparently not involved in the reaction. Both the term and the phenomenon were heavily debated throughout the rest of the 19th century until the German chemist Wilhelm Ostwald proposed a now generally accepted definition: "A catalyst is a substance that accelerates the rate of a chemical reaction without being part of its final products." The catalyst acts by forming intermediate compounds with the molecules involved in the reaction, offering them an alternate, more rapid path to the final products.

Catalysis is of vital importance. In biological systems, enzymes play a catalytic role. In the chemical and petroleum industries, key processes are based on catalysis. And in environmental chemistry, catalysts are essential to breaking down pollutants such as automobile and industrial exhausts.

If the catalyst and the reacting species are in the same phase (for example, liquid), then the process is known as homogeneous catalysis. More relevant in technical processes is heterogeneous catalysis, where the catalyst is a solid and the reacting molecules interact with its surface from the gaseous or liquid phases.[1] The economic significance of heterogeneous catalysis is reflected in the fact that the worldwide market for solid catalysts in the automotive, petroleum and other industries is on the order of $100 billion per year and growing rapidly.[2]

Typically, the chemical transformation occurs in a flow reactor through which the reacting species pass. Atoms in the surface of the catalyst may form chemical bonds with atoms in impinging molecules, a phenomenon known as chemisorption. If existing bonds in the molecule break, the process is called dissociative chemisorption. The chemisorbed species are mobile on the surface and may bond to other particles, thus leading to new molecules, which eventually leave the surface (desorb) as the desired reaction products.

Detailed identification and characterization of these elementary processes is hampered, however, by fundamental problems. The reacting systems exist merely as two-dimensional phases for which most of the usual methods of investigation are not well suited, and so researchers have had to develop novel surface-sensitive tools. (See the box on page 35.) Furthermore, the surfaces of "real" catalysts are typically rather inhomogeneous. Because their efficiency increases with their total surface area (as long as no diffusion or other limiting transport process is required), finely divided particles are usually applied to a more-or-less inert support material. (See figure 1.) Catalytic activity is often further enhanced by the addition of compounds called promoters.

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Making ammonia

The synthesis of ammonia (NH_3) from the elements nitrogen (N_2) and hydrogen (H_2) represents the first—and still one of the most important—large-scale industrial processes based on heterogeneous catalysis.[3] This reaction was first realized in 1909 by Fritz Haber, on a laboratory scale. Only four years later, due mainly to work performed by Carl Bosch and Alwin Mittasch, the first industrial plant of Badische Anilin und Soda-Fabrik (BASF), one of today's big chemical companies, started operations. Currently, 150 million tons of ammonia are produced per year worldwide, most of which is converted into fertilizer.

The catalyst developed by Mittasch was essentially iron with small amounts of potassium, aluminum and calcium added as promoters. With only minor modification, it is still in use in most ammonia-producing plants. It is only in recent years that catalysts based on supported ruthenium particles with alkali metal promoters have emerged as possible alternatives; they were first proposed by Japanese researchers.

Despite its great complexity, the mechanism of this important reaction can now be regarded as known. The reaction rate can be successfully modeled on the basis of the kinetics of the elementary steps involved, as figure 2 illustrates.[4]

The necessary information was obtained largely by surface science modeling. An actual catalyst is complex, consisting of small solid particles supported on oxide powders exposing various crystal planes, usually with poorly defined composition and morphology. Consequently, model systems must be developed. By "model," we mean real but simple systems. The simplest model system would be a well-defined single crystal surface whose structure may be varied by choosing different surface orientations. Furthermore, by introducing defects and by modifying the crystal's chemical composition, the morphology of the surface may be changed to bridge the material gap between the models and the actual catalyst.

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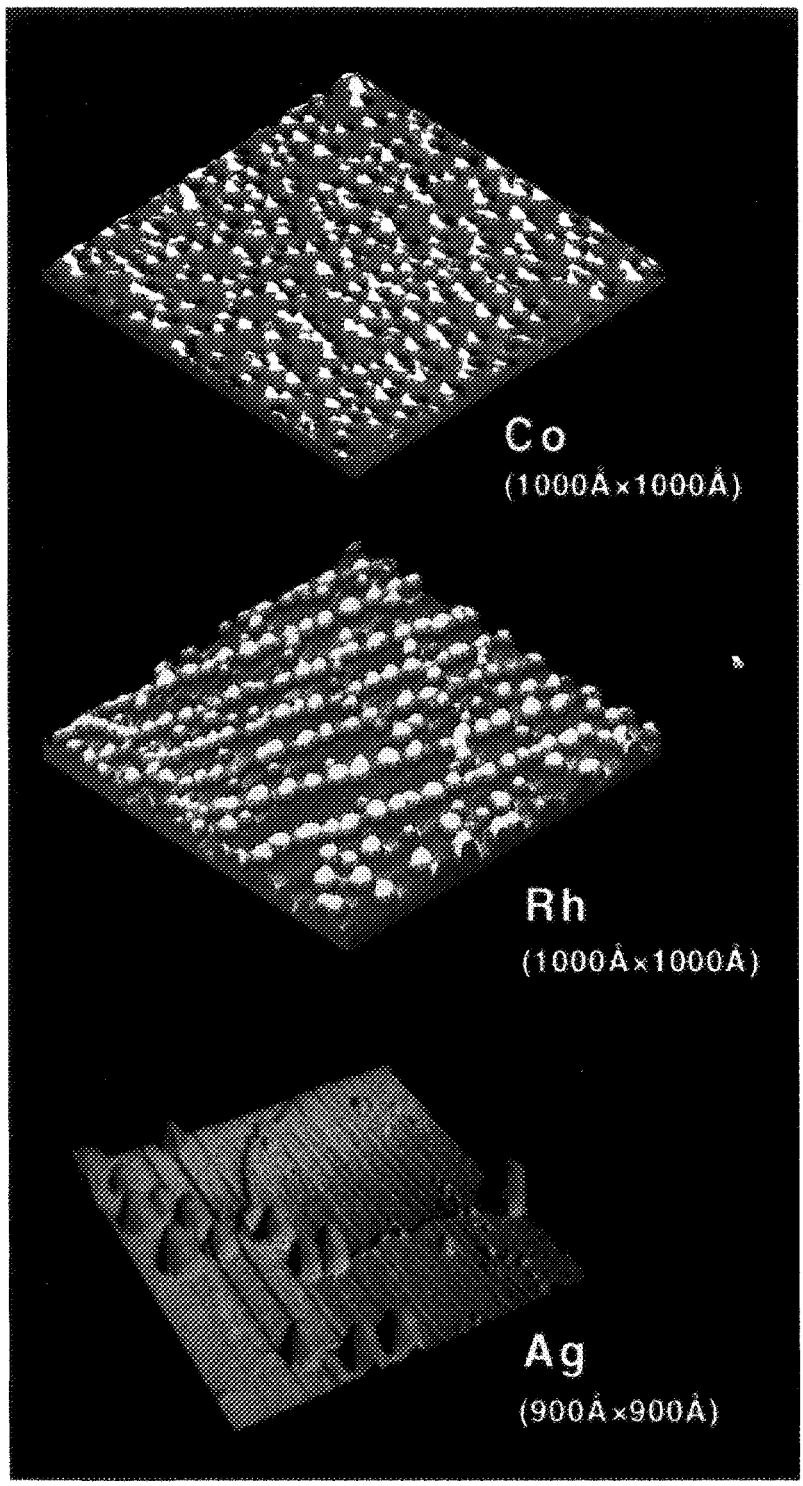

FIGURE 1. CATALYTIC SURFACES imaged by scanning tunneling microscopy. These simple catalysts are single-crystal surfaces of thin oxide films supporting evaporated metal aggregates. Such model systems allow one to use surface science methods to perform detailed studies of the elementary steps involved in catalytic reactions. The three surfaces imaged here contain similar amounts of different metals. The metal–oxide interaction strength decreases from top to bottom, leading to considerably different growth modes.

the model systems, as well as their interaction with gaseous molecules, may be studied in great detail by using the arsenal of modern surface physics.[5] (See the box on page 35.) Unfortunately, most of these techniques can be applied only at fairly low gas pressures ($\leq 10^{-4}$ mbar), whereas actual catalysis typically proceeds at atmospheric or even much higher pressures. The surface science approach, therefore, creates a "pressure gap" in addition to the "materials gap" introduced by the reduction to single crystal surfaces. There exist various ways to overcome these problems, which always have to be taken seriously.[6]

Oxidation of carbon monoxide

The catalytic oxidation of carbon monoxide illustrates the present state of knowledge. This important process in car exhaust control involves the use of finely divided platinum–rhodium particles. This reaction consists of the chemisorption of CO and the dissociative chemisorption of O_2:

$$CO + * \rightarrow CO_{ad}$$

and

$$O_2 + 2* \rightarrow 2O_{ad} ,$$

where the asterisk denotes a free adsorption site on the surface. Such a site may be formed by various configurations of surface atoms on a given surface. The two surface species then combine to form the product

$$CO_{ad} + O_{ad} \rightarrow CO_2 + 2*.$$

Interactions between the adsorbed particles usually cause the formation of ordered phases at certain concentrations, or surface coverages. The structures of these phases can be analyzed by using suitable diffraction techniques such as low-energy electron diffraction. Figure 3a shows the structure formed by CO molecules on a rhodium (111) surface at a coverage θ of 0.33, whereby the adsorbates are located in "on top" positions. The chemisorbed oxygen atoms, on the other hand, prefer threefold coordinated sites as depicted in figure 3b for the 2×2 phase $= 0.25$. The dissociative chemisorption of O_2 requires an ensemble of empty neighboring surface sites, which is why adsorption of oxygen is inhibited beyond a certain CO coverage; the adsorbed CO molecules themselves act as a poison. Therefore, the temperature has to be high enough (at least 150 °C) so that some CO is continuously desorbed and a sufficiently large fraction of the surface may be occupied by oxygen under steady-state conditions. The chemisorbed oxygen atoms, on the other hand, form a relatively open structure into which CO molecules may be additionally adsorbed so that a mixed

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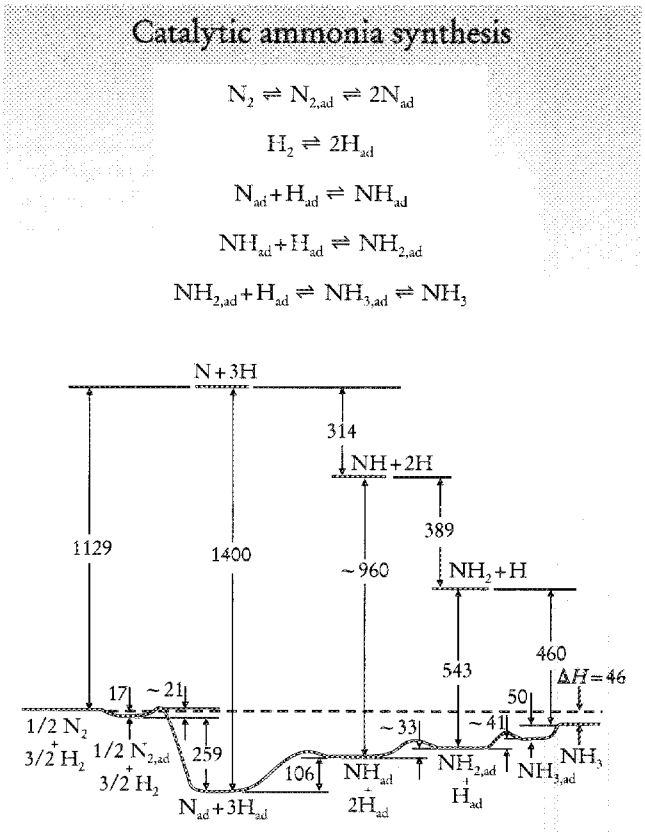

FIGURE 2. MECHANISM OF THE CATALYTIC SYNTHESIS of ammonia from the elements, $N_2 + 3H_2 \rightarrow 2NH_3$, and the associated potential energy diagram. The subscript "ad" indicates atoms and molecules that are adsorbed onto the surface of the catalyst. The numbers shown indicate energies in kilojoules per mole. ΔH is the reaction enthalpy. (After ref. 4.)

phase may form,[7] as shown in figure 3c.

In the open-structure configuration, the two surface species are in intimate contact and may react to form the product CO_2. Due to the interactions between adsorbed species, however, this process is somewhat more favorable for CO molecules outside an O_{ad} island. Hence, the reaction proceeds preferentially at the boundaries of domains formed by the two adsorbate phases, as becomes evident from inspection of the series of images in figure 4 recorded by scanning tunneling microscopy.[8] Here, the surface was first largely covered by O_{ad} and then exposed to a CO atmosphere. The progress of the reaction is reflected by the continuous growth of CO_{ad} islands at the expense of shrinking O_{ad} domains, and quantitative analysis of these data provides information about the kinetics on the atomic scale. As mentioned above, model systems of this type are still quite different from the conditions of actual catalysis.

More complex models

Let us now consider more complex model systems in which some of the restrictions prevailing for smooth single-crystal surfaces have been removed. One approach to overcoming the materials gap is to model the structurally and chemically more complex catalyst support by using an oxide single crystal or a well-ordered epitaxial oxide film grown on top of an appropriate metallic substrate. Using thin films is advantageous when the support is an electrical insulator, because it is thereby possible to avoid many experimental complications that arise when surface science techniques are applied. Metal aggregates are deposited on the oxide film supports.[9] Deposition is achieved either by selecting metal particles according to their mass in the gas phase and then depositing them on the surface with "soft landing" techniques, or by using nucleation and growth techniques in which the oxide

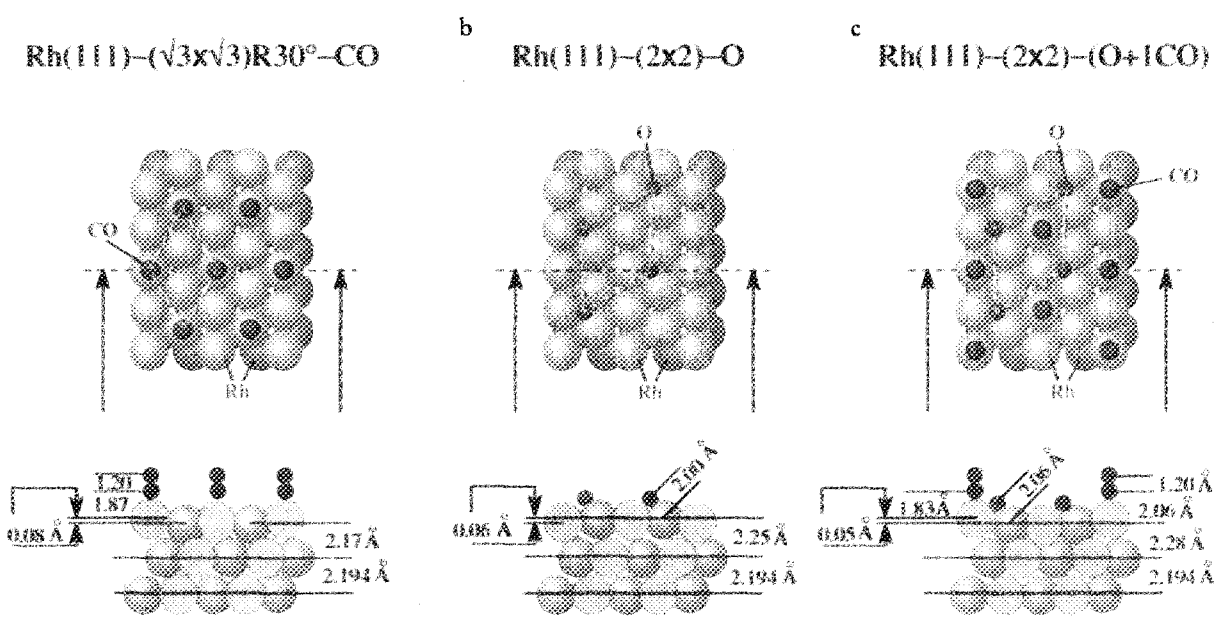

FIGURE 3. ORDERED STRUCTURES formed by atoms and molecules adsorbed by the (111) surface of the catalyst rhodium. Both top and side views are depicted. The small gray circles represent carbon atoms; the small blue circles, oxygen. **a:** Adsorbed carbon monoxide molecules. **b:** Adsorbed oxygen atoms. **c:** Adsorbed carbon monxide and oxygen. The structures were determined by low-energy electron diffraction. (After ref. 7.)

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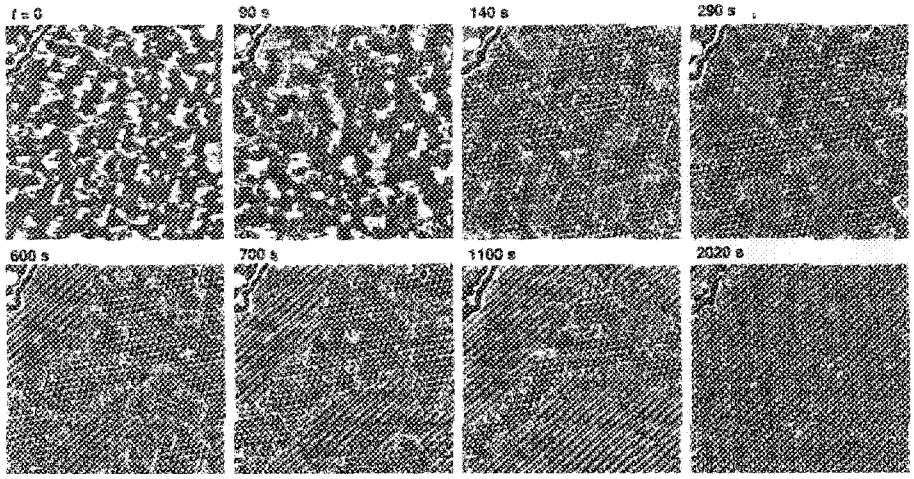

FIGURE 4. TIME SERIES showing the buildup of a monolayer of carbon monoxide on a platinum (111) surface that was initially covered by oxygen atoms (at $t = 0$). The oxygenated catalytic surface was exposed at 247 K to a continuous flow of 5×10^{-8} mbar CO. At this pressure, a monolayer of CO begins to form within the O layer after about 100 seconds. The reaction $O_{ad} + CO_{ad} \rightarrow CO_2$ proceeds at the boundaries between the growing CO_{ad} patches and the shrinking O_{ad} islands. These atomic resolution scanning tunneling microscope images follow the process. (From ref. 8.)

Methods

For the investigation of surfaces, researchers have developed a series of spectroscopic techniques, which are reviewed very briefly below.

Surface spectroscopies depend to a large extent on the strong interaction of electrons with condensed matter.[16] For electrons with energies around 100 eV, the escape depth is confined to the very first couple of atomic layers at the surfaces.

Electrons may be liberated by photons. Knowing the photon's energy and the electron's kinetic energy, we may simply use energy conservation to calculate the binding energy of the electron. This energy can then be used to help identify the chemical element. Also, the so-called chemical shift and the valence electron distribution can be used to determine the electronic structure of the sample. The method is called photoelectron spectroscopy and has been developed over the last 40 years. In combination with synchrotron radiation, it is a very versatile tool. One of the most commonly used methods for characterization of surface composition is Auger spectroscopy. Auger electrons are created through the decay of a core ionized state of the sample.

When a material is exposed to photons, the photons may induce fluorescence—that is, secondary photons. Extended absorption fine structure (EXAFS) and near-edge absorption fine structure (NEXAFS) measurements are based on these processes and may be used to elucidate both the electronic and geometric structure of matter. Although EXAFS and NEXAFS operate at high photon energies, other techniques are available that employ very low photon energies. One such very useful method is infrared reflection–absorption spectroscopy, in which infrared photons are reflected at grazing angles from a surface to increase the surface sensitivity of the method and excite vibrations of substrate surfaces or adsorbate vibrations.[17] Although IRAS is fully applicable under ultrahigh-vacuum conditions, the presence of a gas phase will absorb most of the light even at moderate pressures. An alternative method yielding comparable information is sum frequency generation. In this method, a laser beam with an energy fixed in the visible part of the spectrum is crossed at the surface with a second, scannable infrared laser beam.[17]

Instead of using photons primarily to stimulate elementary excitations at surfaces, electrons can also be used as exciting projectiles. If low-energy electrons are used, the surface structure can be determined by measuring the spatial distribution of elastically reflected electrons. This method, called low-energy electron diffraction (LEED), can be used for a full structure determination of surfaces and adsorbed layers. Inelastically reflected electrons can be measured with a kinetic energy detector so that the quanta they lost in the scattering process can be determined. Surface vibrations and adsorbate vibrations can be identified by high-resolution electron energy loss spectroscopy (HREELS). By tuning the electron energy to higher values, electronic excitations and Auger transitions can be excited, thereby providing the basis for further surface sensitive spectroscopies.

Electrons can also lead to photon emission through processes based on the creation of bremsstrahlung. If a fixed photon energy is detected while the electron energy is being scanned, the method is called inverse photoemission; it allows one to detect the material's valence levels that are unoccupied in the ground state. If the full energy-resolved bremsstrahlung spectrum is recorded, the method is called cathode luminescence.

Last but not least, electrons of very high energy (200 keV) can pass through a sufficiently thin sample and be used to image the structure of the sample. This method, called transmission electron microscopy (TEM),[18] has been used successfully for over half a century, but its surface sensitivity, of course, is limited. Many of the above-mentioned surface spectroscopies have been used to establish microscopic detection modes to combine spatial information with morphological or structural information.

A very versatile microscopic tool became available in the early 1980s with the invention of scanning tunneling microscopy (STM).[18] An atomically sharp metal tip is brought in atomic proximity to a flat surface so that electrons can tunnel between the two systems. Recording the atomic modulation of the electronic structure while scanning the tip across the surface allows one to image adsorbed species and surface morphologies. A method complementary to STM is atomic force microscopy (AFM), in which a tip is fixed to a cantilever whose position is monitored while the tip scans the surface. The force between the tip and the surface determines the position of the cantilever. When recorded in atomic resolution, the image represents a map of atomic forces at the surface. The advantage here is that the probed surface does not have to be electrically conducting.

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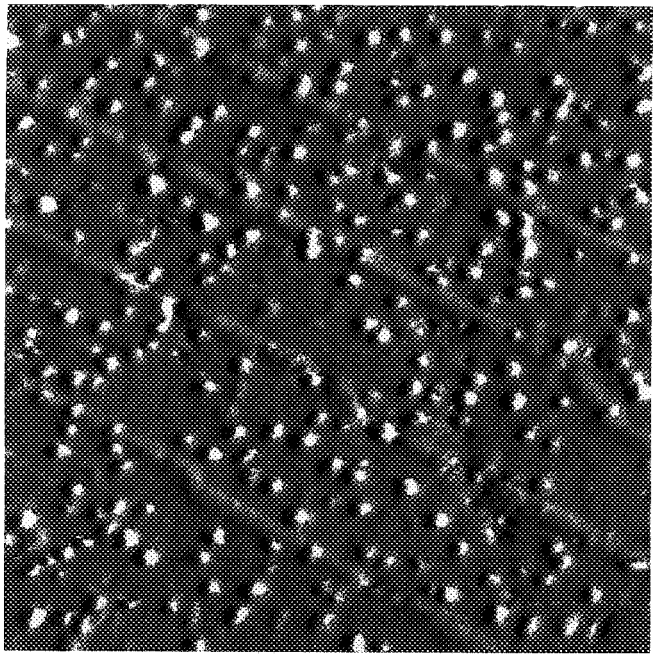

FIGURE 5. ONE-TWENTIETH OF A MONOLAYER of rhodium metal deposited at 90 K on an alumina substrate. This scanning tunneling microscope image covers an area 800 Å on a side. The light protrusions are the deposited metal aggregates. The finer structure reflects the structure of the oxidic support; the broad linear features are due to a domain defect structure at the surface. Unlike figure 1 (center), the defects do not influence the aggregates. The imaging parameters were 5.1 V tunneling voltage and 2 nA tunneling current.

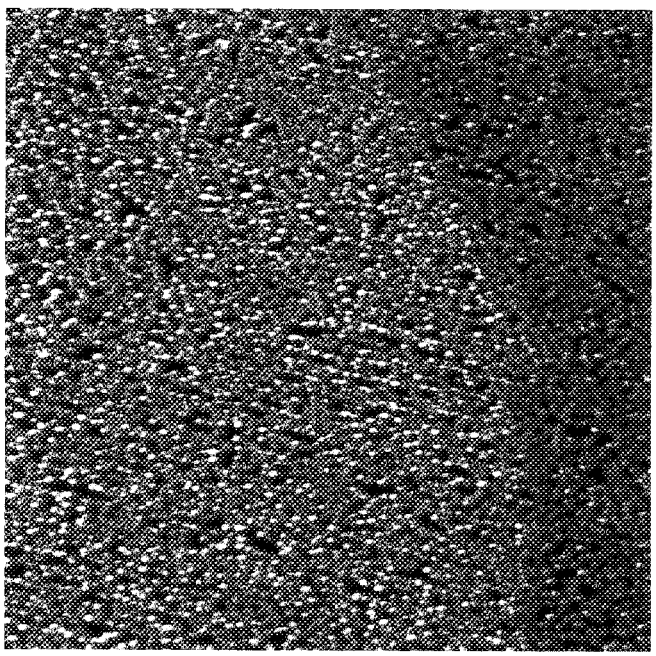

FIGURE 6. ONE-HALF OF A MONOLAYER of rhodium metal deposited at room temperature on a prehydroxylated alumina substrate. This scanning tunneling microscope image covers an area 300 Å on a side. The distribution of rhodium aggregates is considerably more uniform than in figure 1 (center) or figure 5, where the surface was not hydroxylated. The imaging parameters were 1.2 V tunneling voltage and 0.37 nA tunneling current.

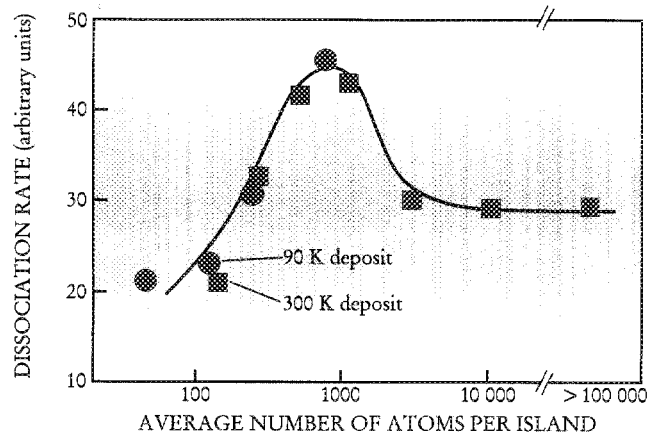

FIGURE 7. EFFECT OF SIZE of catalytic particles. Rhodium aggregates of various sizes (measured by the average number of rhodium atoms per aggregate) were deposited on an alumina film. The rhodium was then given saturation carbon monoxide coverage. The material was then heated from 90 K to 500 K and the amount of atomic carbon formed on the rhodium was plotted.

surface is exposed to a metal vapor and the surface temperature and metal vapor flux are accurately controlled.[10] Figure 1 shows several examples resulting from metal deposition on a thin alumina film. Remember that for catalysts to function, the morphology and structure of the metal deposits are very important, because they determine the activity and selectivity in the chemical process.

Different metals exhibit very different growth modes, which depend to a large extent on the strength of interaction between the metal and the oxide substrate. This interaction is strongly influenced by the presence of defects, such as steps or domain boundaries, or point defects on the substrate. Silver, for example, interacts only weakly with alumina and is therefore very mobile at room temperature. It nucleates at steps and forms relatively large but few aggregates (see the bottom of figure 1). Platinum, cobalt and nickel, on the other hand, interact more strongly with the oxide substrate, and thus are less mobile and form small particles upon deposition at room temperature (see the top of figure 1). Such investigations provide a direct indication of how the substrate has to be conditioned for a given metal so as to prepare a desired dispersion of metal particles. For example, if one wants to produce highly dispersed small silver particles, one has to ensure that the substrate exposes a sufficient number of defects of the right kind. For platinum, on the other hand, this is not such an important preparation prerequisite. Of course, not only structural defects may be used to influence the metal dispersion; chemical modification of the support surface may also be employed.

The middle panel of figure 1 shows rhodium, an active component of the automotive exhaust catalyst, deposited at room temperature on a clean alumina substrate. The defect structure of the alumina substrate is characterized by domain boundaries induced by the growth of the thin alumina film on the metallic substrate. The mobility of rhodium at this temperature is such that the aggregates nucleate at these defects without coalescence due to the particular epitaxial growth conditions, forming strings of deposited aggregates. To grow aggregates in a random distribution across the surface, one can expose the surface at lower temperature (figure 5) or hydroxylate the surface before exposure to the metal vapor.[11] If the hydroxylation

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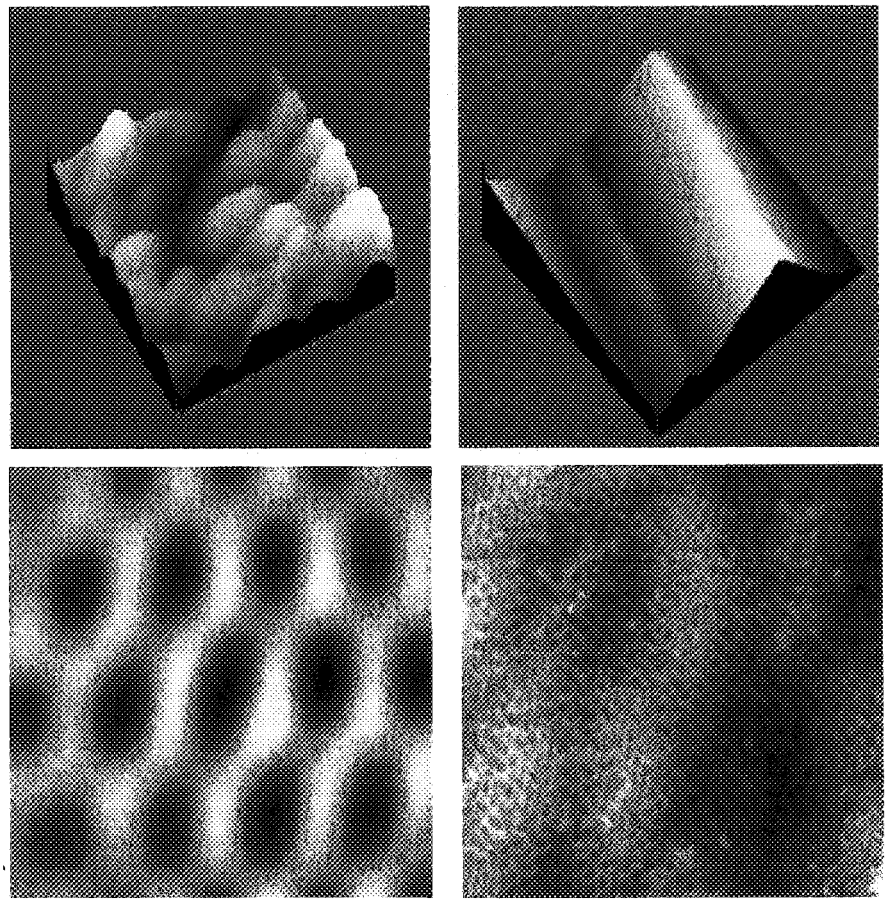

FIGURE 8. THE CATALYST SURFACE topology changes through contact with reactants. Here, a particle of silver becomes rougher after catalyzing the oxidation of methanol. The oxygen-covered catalyst shows well-structured and ordered facets, as seen in the scanning tunneling microscope image (190 nm on a side) at the upper right. On these facets, it is even possible to attain atomic resolution, as shown in the image at the lower right (17 nm on a side) and in an enlarged section of that image, at the lower left (2.2 nm on a side). The dark areas in these two lower panels are the oxygen sites. After the silver particle catalyzes methanol according to $CH_3OH + O_{ad} \rightarrow CH_2O + H_2O$, the topology changes as shown in the STM image (150 nm on a side) at the upper left.

process is used, the impinging rhodium atoms chemically bind to the hydroxyl groups according to the reaction scheme

$$\{Al^{3+}\ OH^-\} + Rh^0 \rightarrow \{Al^{3+}\ O^{2-}\}\ Rh^+ + H_{ad}.$$

This binding stabilizes smaller aggregate sizes at random distribution even at room temperature, as shown in figure 6.

Once the aggregates have been formed and have reached a given size, they are stable over a considerable range of temperatures. For example, the distribution of aggregates created by deposition at low temperatures (90 K), shown in figure 5, is morphologically stable up to 700 K. The rhodium aggregates prepared on the hydroxylated surface (figure 6) are even more stable. We may use the region of thermal stability of the aggregates to investigate chemical reactivities of such an ensemble of aggregates without changing morphologies. This puts us in a position to study a particular chemical reaction for a given particle size under very well defined conditions and to proceed a step toward bridging the above-mentioned materials gap.[12]

Effect of particle size

Furthermore, if we are able to control the size of the deposited aggregates, then we should be in a position to explore the size dependence of chemical reactions in such systems. Particle size selectivity of reactions has been used to steer catalytic reactions to certain products. CO dissociation has to be considered one reaction channel in connection with automotive exhaust control, because it can lead to the deposition of carbon on the catalyst, which in turn may poison its activity. We have chosen to investigate this very simple reaction—that is, CO dissociation on rhodium aggregates. It is known from the study of the interaction of CO with rhodium aggregates deposited on alumina powders that CO shows a varying tendency to dissociate into carbon and oxygen atoms apparently depending on the size of the deposited rhodium aggregates. By varying particle sizes through the methods of nucleation and growth of metal particles on the alumina substrate, we are now in a position to study the particle size dependence of this simple reaction in detail. Figure 7 shows the result of such a study.

The main finding is the existence of a maximum in the dissociation rate. It occurs for aggregates containing about 1000 atoms, while for smaller as well as larger aggregates the dissociation rate is lower. It is well known for flat metal single-crystal surfaces that CO does not dissociate upon adsorption but rather binds to the surface reversibly as a molecule. The introduction of steps on the surface induces dissociation, indicating that there are specific sites necessary for the dissociation reaction. A detailed analysis of the structure and morphology of the deposited aggregates as a function of size indicates that it is the creation and finally the diminution of steps that steer the reactivity.

Following the dissociation rate curve in figure 7 from the left, we can explain the peak. It has been shown that the smallest particles grow as two-dimensional layers. Before the aggregates contain 100 atoms, the second layer starts to grow. Eventually, three-dimensional particles grow, but their height never exceeds their width. When the particles contain fewer than about 1000 atoms, they coalesce and the island density decreases considerably. That is when the dissociation rate decreases and the maximum has been surpassed. Thus, it is quite easy to understand how the size-dependent reactivity comes about in this case. It turns out that the size of particles near 5 nm where we find the highest dissociation rates indeed coincides with those sizes where high catalytic activities have been observed in various—and also much more complex—chemical reactions. Obviously, for a given metal/substrate combination, certain particle sizes stabilize a maximum of active sites and thus maximize the reaction rate. Particle size, however, is only one ingredient in optimizing a catalytic reaction. Selectivity is another issue, which research has yet to address.

A perhaps even more important aspect of understanding the details of a catalytic reaction is the restruc-

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turing of the catalyst under working conditions.[13] Surface physics research has determined the relocation of surface atoms from their bulk-material positions by relaxation and reconstruction. Adsorption again alters the position of surface atoms, and the surface adjusts to changes in the chemical environment as well, leading to a full restructuring on the local and long-range scales.[14] Such massive reconstructions require surface diffusion over distances of many atomic bonds. Figure 8 shows an example—the oxidation of methanol to form formaldehyde and water on a metallic silver catalyst.[15] The catalyst is first oxygen loaded and shows well-ordered facets that may be atomically resolved. After reaction, the topology is completely changed and the surface becomes considerably rougher.

In this example, the chemical reactivity of the oxide support has not been taken into account. However, oxides in general—and transition metal oxides in particular—are known to be very good catalysts for a variety of chemical reactions. The investigation of transition metal oxide surfaces is a very active field today and has implications that also reach into other fields such as sensors and high-temperature superconductors. Our ability to study catalytic reactions by preparing model systems and understanding them in detail depends upon our increasing knowledge of the physics of compound surfaces. There are lots of opportunities for fruitful interactions between physicists and chemists in a field that has never been more lively.

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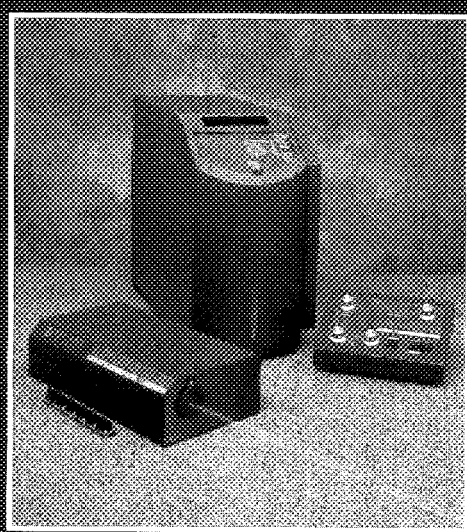

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