

change $\Delta\Phi$ is thus precisely $neV_H\Delta\Phi/(h/e)$, where V_H is the Hall voltage between the edges. Recalling that $I = \Delta E/\Delta\Phi$, one has immediately the desired result that the Hall resistance $R_H \equiv V_H/I = h/ne^2$.

How precise is this result expected to be? Laughlin points out that although the virtually lossless Hall current is in some sense a "supercurrent," it differs from the true superconducting case by permitting a minute electric field component in the direction of the current. This residual nonvanishing σ_{xx} results from the "hopping conductivity" of carriers that persists at any finite temperature, even when the Fermi level is in the mobility gap. This hopping conductivity, which depends on the mean free path in the material in question and vanishes at absolute zero, should affect the Hall resistivity to much less than one part in 10^8 at the temperatures of the quantized Hall experiments, he estimates.

Using von Klitzing's technique, but with a more sensitive voltage measuring system, Taylor, Marvin Cage and

their colleagues at NBS, collaborating with the Bell Labs group and Robert Wagner and Charles Levine at the Naval Research Lab, hope to have measured the fine-structure constant to within a few parts in 10^7 by the end of this year. At present, α is known to one part in 10^7 from two sources: The determination of α from the anomalous magnetic moment of the electron (the "g-2" experiments) depends on the correctness of quantum electrodynamics. The measurement of the proton's gyromagnetic moment, on the other hand, yields a QED-independent determination of α . —BMS

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Catalysis of carbon gasification

The conversion of carbon to its oxide gases or methane, by interaction with oxygen, steam, hydrogen or carbon dioxide, are reactions of great practical importance. The oxidation of carbon to CO_2 and CO provides much of man-

kind's heat and power, and the formation of "syngas" (a mixture of carbon monoxide and hydrogen) by steam gasification of carbon is an important step in the conversion of coal to synthetic petroleum (PHYSICS TODAY, August

1978, page 32). These reactions, together with the hydrogenation of carbon to form methane, and its interaction with CO_2 to form CO, are the principal gasification reactions of carbon.

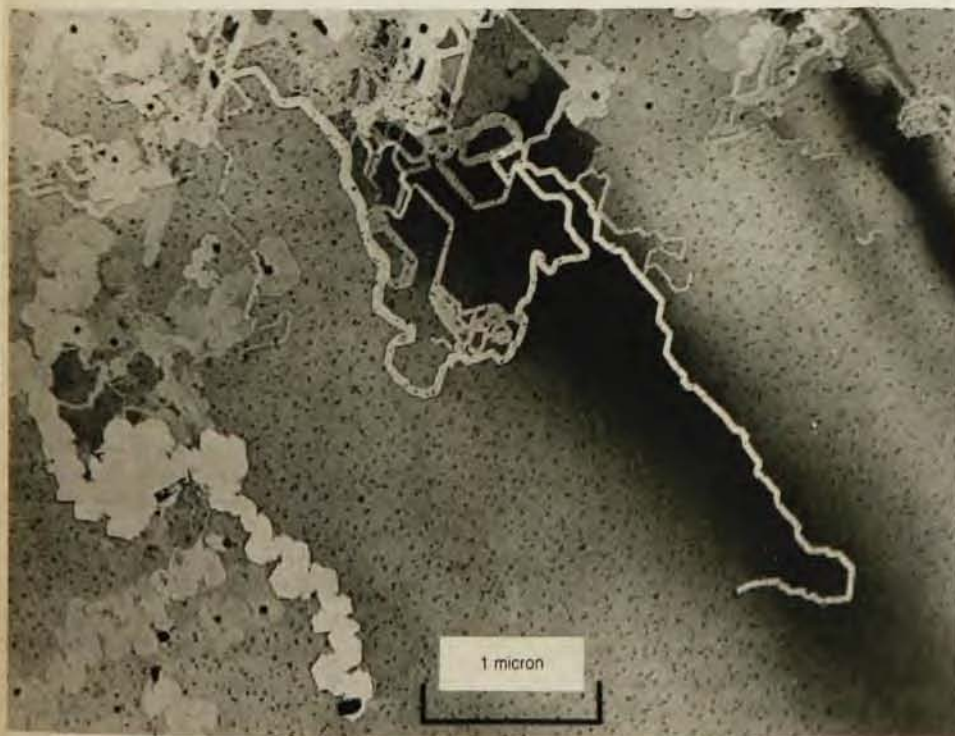
In the absence of impurities, these reactions are all surprisingly sluggish. Although it has long been known that small amounts of naturally occurring or deliberately added impurities can alter the rates of these reactions dramatically, the precise catalytic mechanisms by which these additives act have for the most part remained a mystery.

A peculiar phenomenon that appears to play a central role in these catalytic gasification reactions is the cutting of channels and pits by catalytic microparticles as they spontaneously burrow along and into the crystalline surfaces of graphite. Over the past decade, Terry Baker, first of Harwell (England) and more recently at Exxon, has developed an electron-microscopy technique for observing this channeling action in progress. With a controlled-atmosphere transmission electron microscope (CAEM) he is now able to film catalytic particles as they cut channels along the various crystal axes of the graphite basal plane in the presence of the reactant gases at temperatures up to 1350°C , with a resolution of $25\text{ \AA}$. At the March APS Meeting in Phoenix, Baker reported recent results of his CAEM studies of catalytic graphite gasification.

Although these catalytic particles and the channels they cut are frequently smaller than 100 angstroms, far below the resolution of optical microscopes, they were first observed in optical studies by John Thomas, at the University of Bangor in Wales in the 1960s. In the early 1970s Douglas McKee at General Electric extended these techniques by the use of "hot-stage" optical microscopes, which could observe the catalytic gasification processes in progress. McKee and Philip Walker's group at Penn State have also performed extensive analytical and thermodynamic investigations to elucidate the chemical reactions involved.

Hot-stage optical microscopes can observe the gasification reactions at high temperatures and high reactant-gas pressures. But the details of the unique catalytic channeling in graphite eludes their limited resolution (about one micron). The particle beams of conventional electron microscopes, on the other hand, would be badly degraded while passing through a reactant gas atmosphere on the way to the surface under inspection.

The controlled-atmosphere electron microscope addresses this problem by holding the sample in a differentially pumped gas reaction cell. A



Catalytic channeling by microparticles of platinum catalyst, burrowing along the surface of a highly purified flake of crystalline graphite in a 5-mm atmosphere of oxygen at 750°C . This unique catalytic action in the oxidation of graphite and other carbon-gasification processes is observed in progress by the controlled-atmosphere transmission electron microscope developed by Terry Baker at Exxon. In an oxidizing atmosphere, the catalytic particles dig preferentially along the $\langle 10\bar{1}0 \rangle$ axis of the graphite basal plane.

slow flow of reactant gas is passed over the sample at a precisely controlled pressure of up to 40 mm Hg. The differential pumping, first used in electron microscopy twenty years ago by H. Hashimoto in Japan, maintains this pressure for less than a millimeter over the sample surface. Except for this last millimeter, the electron beam is passing essentially through vacuum. Even this small traversal through gas, however, together with the requirement of an unusually wide-bore objective pole piece to accommodate the gas cell, degrades the resolution of Baker's CAEM by about a factor of five. A conventional transmission electron microscope would offer a resolution of roughly 2.5 Å.

A further factor of two in resolution is lost in the CAEM by the finite line widths of the microscope's videotape recording system. Still photographs can be made with a resolution of 12 Å, but the essence of the CAEM technique is its ability to record the sample surface continually while the catalytic gasification reaction is in progress, under realistic conditions of temperature and pressure. The gas pressure is however still limited to a little above half an atmosphere by the requirements of electron microscopy. Studies at high pressure can be done only with optical microscopes.

Prior to the development of the CAEM, attempts had been made to study the catalytic gasification by inspecting graphite surfaces in electron microscopes before and after the gasification reaction. But such investigations, Baker told us, "leave one to infer what has taken place in the interim." One cannot observe the channeling mechanism as it unfolds, and one is looking at a sample that may well have been seriously altered by post-reaction cooling.

In Baker's CAEM, a direct current is passed through the graphite sample to heat it resistively to temperatures as high as 1350° C. One assumes that the reaction is affected neither by this current nor by the electron beam. Any effects due to ionization of the reactant gas by the 100-keV electron beam can be monitored by comparing the end results with and without the microscope beam.

The graphite samples used by Baker are highly purified, naturally occurring, single crystals, cleaved along their basal planes to a thickness of 50 to 100 Å—thin enough to let the electron beam pass through. In this crystalline form of carbon, the atoms are arranged in basal planes about 3 Å thick, with very weak bonding between planes. Thus the planes slide over one another rather easily—like a deck of cards, as Baker describes it—making powdered

graphite an excellent lubricant. Anthracite coal is essentially disordered graphite, containing less than one percent of impurities.

The catalysts of primary interest for the carbon gasification reactions are the noble metals platinum and silver, the transition metals iron, nickel, vanadium, cobalt, manganese, copper and their oxides, and the carbonates of barium and of the alkali metals sodium and potassium. For the CAEM studies, Baker evaporates a monolayer of a particular catalyst onto the surface of a purified flake of graphite. The sample is then held in the microscope's gas reaction cell, where it is resistively heated to the desired temperature and subjected to a pressure-controlled flow of oxygen, steam, CO₂ or hydrogen.

Baker described to us what one sees on the videotape as the catalytic process unfolds with increasing temperature. As heating begins, the catalytic monolayer breaks up into discrete particles ranging in size from less than 100 to about 1000 angstroms. As the temperature of the graphite is raised further, those particles that find themselves at the edge of the flake, or at "step" sites between basal planes, begin to move across the surface plane, digging out a channel as they go. They move along the preferred crystallographic directions of the plane, but they execute abrupt, random 120° turns as they change from one crystal axis to another. At these temperatures, catalytic particles sitting on top of perfect portions of the basal plane have not yet gone into action. Particles at planar imperfection sites will begin to dig into the plane, forming "pits;" but this pitting action is much slower than the channeling motion across the basal plane.

This channeling and pitting is the beginning of the carbon gasification. The carbon that disappeared from the channels and pits has been converted to CO, CO₂ or methane, depending on the composition, temperature and pressure of the reactant gas. The ultimate purpose of these experiments is to determine the physical and chemical details of the interactions between the carbon, the catalyst and the gas. In addition to contributing to the fundamental study of catalysis, a knowledge of how these reaction rates depend on the composition, size and temperature of the catalyst particles is needed for the optimization of the gasification reactions in practical applications.

It is plausible that the catalyst particles should attack the graphite first at planar edges, steps and defects, where carbon valence bonds are dangling free. In the absence of crystal defects, the valence bonds of graphite are saturated in the basal plane. The solid-state reactions between catalyst and

graphite require that the surface of the catalyst particle surfaces be quasi-liquidified. The fact that these reactions begin at less than one half the melting temperature of the catalyst convinces Baker that the catalyst particles at edges, steps and dislocations have been locally heated well above the bulk temperature by exothermic interaction with the graphite.

In 1923, Gustav Tammann in Germany found that a number of phenomena begin abruptly at a characteristic temperature close to one half the absolute bulk melting temperature—the so-called Tammann temperature. Surface particles suddenly become highly mobile, as do surface dislocations and ions. It is at this Tammann temperature that Baker finds an abrupt increase in the number of catalyst particles participating in the gasification process.

At the Tammann temperature, the catalytic particles that had been sitting idly on the basal plane start to move. Thus they find and stick to the edge and step regions of high interaction energy at an increasing rate. Once there, they begin to channel along the planar surface. In the process they collide frequently with other catalytic particles, often coalescing into larger particles. As one raises the temperature still higher, to around 750° C, uncatalyzed gasification begins, initiating "edge recession" at the edges and steps, and the widening of previously dug channels.

Findings. Baker's CAEM work on graphite has over the past several years yielded a number of interesting results. Among his published findings is the fact that the catalyst particles move quite differently in oxidizing¹ and reducing² (hydrogen) atmospheres. In the presence of oxygen, the smaller catalyst particles cut channels at a faster rate, moving primarily along the $\langle 10\bar{1}0 \rangle$ crystal direction; in hydrogen, the larger particles move fastest, preferring the $\langle 11\bar{2}0 \rangle$ direction. (The hexagonal crystal pattern of graphite has three axes in the basal plane, with a fourth axis normal to the plane.) By transferring these data to movie film, Baker is able to measure these different channeling rates, their angles of catalytic attack and "apparent" activation energies quite accurately.

Microscopic examination alone cannot unambiguously identify intermediate and final products of the chemical reactions involved in the catalytic process. Baker is now working on the development of a hybrid CAEM system that would provide chemical and visual information simultaneously. In the meantime, McKee has been using thermal and chemical microanalysis in an attempt to pin down some of these catalytic reaction sequences. Because this work does not involve microscopy,

he is not limited to ultrathin graphite samples; he examines catalytic gasification of diamond, glassy carbon and coal.

In the steam gasification of coal to form syngas, for example, McKee has identified a cyclic series of reactions³ involving alkali carbonate catalysts such as K_2CO_3 . Dispersed catalyst particles channeling through the coal dig out three-dimensional honeycombs. In their travels, the surfaces of the particles experience a repeated oxidation-reduction cycle of interactions with the carbon and the steam—alternately forming free alkali metal and reforming the carbonate. This catalytic process is particularly attractive, McKee told us, because the alkali carbonates are cheap and abundant.

In these carbon gasification reactions one is not always interested in the gaseous reaction products. One concern is to find catalysts that will opti-

mize the energy obtained from carbon combustion. There is also interest in finding the most efficient way of getting rid of carbon where it is simply a nuisance—as in soot suppression, and the deterioration of catalytic plates in petroleum cracking plants when they become coated with carbon. One wishes also to learn better ways of suppressing the unwanted gasification reactions that damage the graphite control rods of gas-cooled nuclear reactors. —BMS

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New synchrotron lab in Hamburg

The electron synchrotron and the e^+e^- storage ring DORIS at the German Electron-Synchrotron laboratory DESY, in Hamburg, have been providing experimenters with synchrotron radiation beams for more than a decade. With the increased demand for synchrotron radiation sources in recent years, and the transfer of high-energy-physics concentration at DESY from DORIS to the new, more energetic e^+e^- storage ring PETRA, it was decided at the end of 1977 to expand considerably the synchrotron radiation capabilities of DORIS. The recent ceremonial dedication of the new Hasylab marked the realization of this decision.

Hasylab (for Hamburg Synchrotron Radiation Laboratory) will coordinate all synchrotron radiation experimentation at DESY. These will be concentrated primarily in the recently completed, large Hasylab experimental hall. Before construction began, three years ago, a single synchrotron radiation beam line emerged from the segment of DORIS now adjoining the Hasylab hall. The new experimental hall will eventually incorporate six primary beam lines, one for each of the six bending magnets in the full quadrant of the DORIS ring covered by the Hasylab hall. Three of these synchrotron-radiation beams, including the rebuilt original beam line, are now ready for experiments. Each of the primary beam lines will branch into three or four secondary lines, leading ultimately to as many as two dozen experimental stations.

The director of the new Laboratory is Christof Kunz.

Electrons and positrons can circulate in DORIS at energies up to 5 GeV. Although both produce synchrotron radi-

ation as they experience bending in the ring, the radiation beams in the Hasylab hall will come only from circulating electrons, generally with no countercirculating positrons; e^+e^- collisions tend to degrade the intensity of the synchrotron radiation. Because e^+e^- interactions are the *raison d'être* of DORIS as far as high-energy physics is concerned, synchrotron radiation experiments will be carried out primarily during dedicated runs. In the past, such experiments were generally run simultaneously with high-energy experiments. It is estimated that 30 to 40% of DORIS running time will now be dedicated to synchrotron radiation work.

The preexisting European Molecular Biology Laboratory facility, located at the far end of the racetrack-shaped DORIS ring, works with synchrotron radiation from circulating positrons. This facility, run as an outstation of the central EMBL laboratory in Heidelberg, is also building three new experimental stations in the Hasylab hall.

A continuous, intense spectrum of photons from the infrared to hard x rays (up to 100 keV) will be available to experimenters at Hasylab. Until now, the Stanford Synchrotron Radiation Laboratory, using the e^+e^- storage ring SPEAR, has had something of a monopoly on hard x-ray synchrotron beams. Hasylab expects now to enter into serious competition with SSRL in x-ray scattering and EXAFS experimentation (PHYSICS TODAY, March, page 19). This emphasis on the x-ray portion of the spectrum takes into account the fact that a lower-energy synchrotron radiation source, BESSY, will soon be available in Berlin. BESSY, which will cover the vacuum ultraviolet spec-

trum, will be comparable to the University of Wisconsin's Aladdin and the smaller of the two sources under construction at Brookhaven's National Synchrotron Light Source.

Hasylab will not, however, neglect the vacuum ultraviolet region. Four experimental stations with normal-incidence crystal monochromators are planned for beam lines with 5- to 50-eV photons. Two of these are already in operation, doing high-resolution and fluorescence spectroscopy. The other two, now under construction, will do photoemission spectroscopy and angle-resolved photoemission studies.

For the ultraviolet region from 20 eV to 1 keV, the Hasylab hall will house seven experimental stations with glancing-incidence crystal monochromators, one of which is already in operation. These experimental facilities are intended for photoemission and reflection measurements, Fresnel zone-plate microscopy and x-ray fluorescence.

For the x-ray regime above 1 keV, twelve stations are planned; three of these will be run by EMBL. A Danish collaboration has built a three-axis diffractometer, and a large, four-circle diffractometer is under construction. Several focused x-ray beams will be devoted to EXAFS studies, and there will be facilities for x-ray interferometry, x-ray topography and Mössbauer studies.

The instruments at the experimental stations are for the most part built in collaboration with universities and other outside institutions. Many of these devices are intended ultimately to become "general user facilities." Hasylab will welcome outside user groups. Non-physics users, especially, will be offered technical assistance by the Hasylab staff in performing their experiments.

Because DORIS can run with up to 480 electron bunches circulating at once, it can achieve somewhat higher currents, and hence greater synchrotron radiation intensities from the bending magnets than Stanford's SPEAR. (On the other hand, the wiggler magnets at SPEAR provide higher intensity than DORIS at the high-energy end of the x-ray spectrum.) At a circulating energy of 2 GeV, the DORIS electron current can go up to 400 milliamps. The circulating electron bunches deliver pulsed synchrotron radiation to the experimental stations with pulse widths of about 150 picoseconds, at intervals as long as a microsecond between pulses. The synchrotron beam cross section is less than 2 mm² for 2-GeV electrons, growing to 11 mm² at the maximum DORIS energy. At this highest electron energy (5 GeV), the critical synchrotron wavelength of DORIS is about half an angstrom, putting the peak in the spectral distribution near 30 keV. —BMS□