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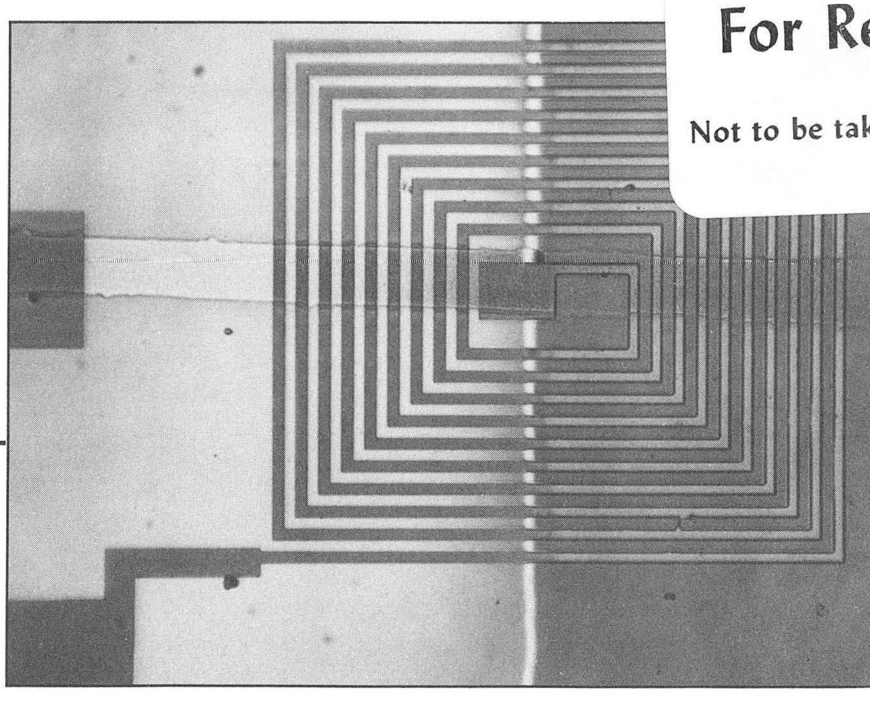

Materials Sciences Division
Lawrence Berkeley Laboratory • University of California
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Center for Advanced Materials

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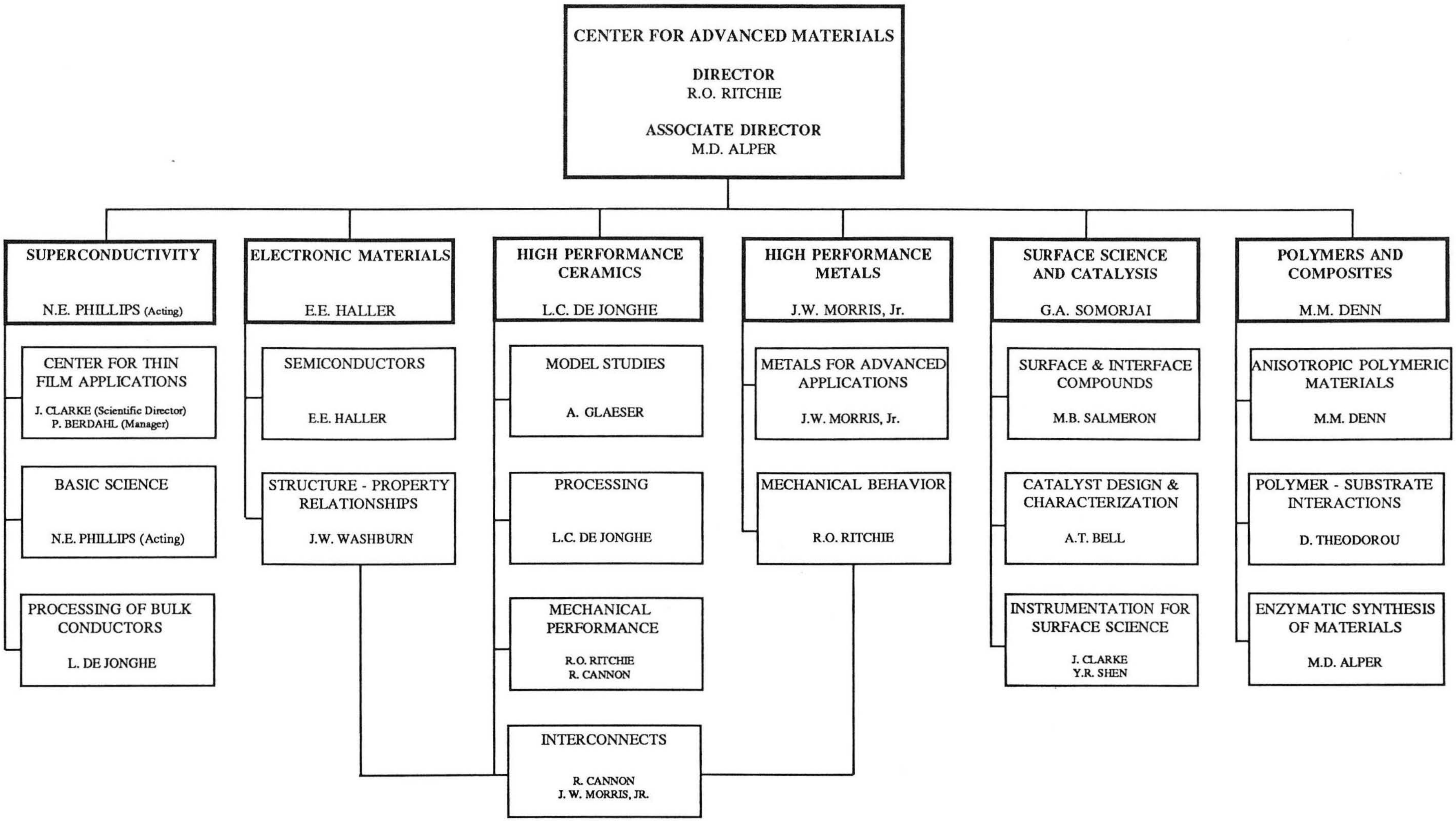
Cover: Photomicrograph of superconducting crossover. (CBB 902-874)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Center for Advanced Materials

CAM RESEARCH NOTE

INDUSTRY PARTICIPATION

"COMPETETIVE TECHNOLOGY" GRANT AWARDED FOR DEVELOPMENT OF NOVEL MATERIALS FOR OPTOELECTRONIC DETECTION SYSTEMS

The CAM project on Enzymatic Synthesis of Materials, with support from the US Department of Energy, has joined with The State of California and Biocircuits Corporation in Burlingame, California, to apply the results of its fundamental research on the design and synthesis of novel, multifunctional, self-assembling materials to the development of commercial detection systems.

The U.S. Department of Energy, Division of Materials Sciences will continue its support of CAM scientist Mark Bednarski's research on novel self-assembling materials. In addition, the State of California, as part of its new Competitive Technology Program, will contribute $k224 to CAM over the next 18 months to support the transfer of that technology to Biocircuits Corporation. The California Competitive Technology Program is designed specifically to increase direct interaction between industry and laboratory scientific groups. This is the second grant to the Center for Advanced Materials made by the program which is now in its second year (see *Research Notes* 3/2). To facilitate the transfer, Biocircuits Corporation will assign seven staff scientists to work on the project. The goal is to utilize molecular self assembling materials in the fabrication of a new generation of detection devices. The first application will be the direct detection of diseases through the specific binding of the microorganism to the device and the transduction of that binding signal directly to a display. A wide range of other applications is expected.

The novel materials are generated by the spontaneous alignment of molecules in three-dimensional space. When placed in contact with a compatible surface, the molecules bind to that surface and form well-ordered materials with molecular dimensions. Adhesion, recognition, wetting, electrochemical, and non-linear optical properties of the surface of the material can be controlled through appropriate design of the constituent molecules.

CAM Industry Participation Office: Mark Alper, (415) 486-6581

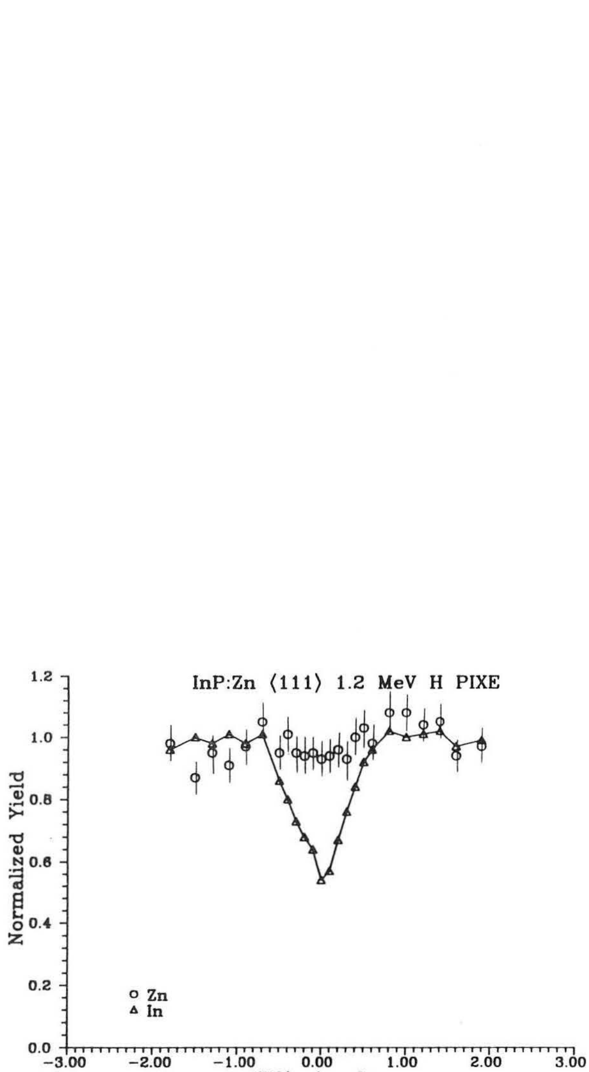

Figure 1
Angular scan of particle induced X-ray emission across the <111> channel for Zn and In atoms in Zn-diffused InP. The yield of In atoms shows a distinct minimum for bombarding particles traveling along the <111> channel, indicating that most of these atoms are on the substitutional sites. Lack of any structure in Zn yield indicates random distribution of Zn atoms in the InP crystal lattice. XBL 906-2069

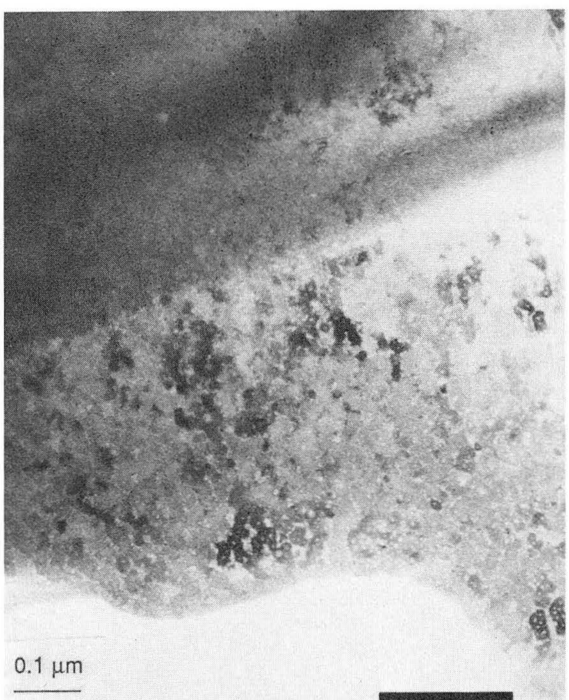

Figure 2
A TEM plan-view micrograph of Zn-diffused InP showing the presence of a large concentration of precipitates of different phases. Two phases of Zn_3P_2 and Zn microcrystals were identified by the Bragg reflection technique. XBB 903-2668

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CAM *RESEARCH NOTE*

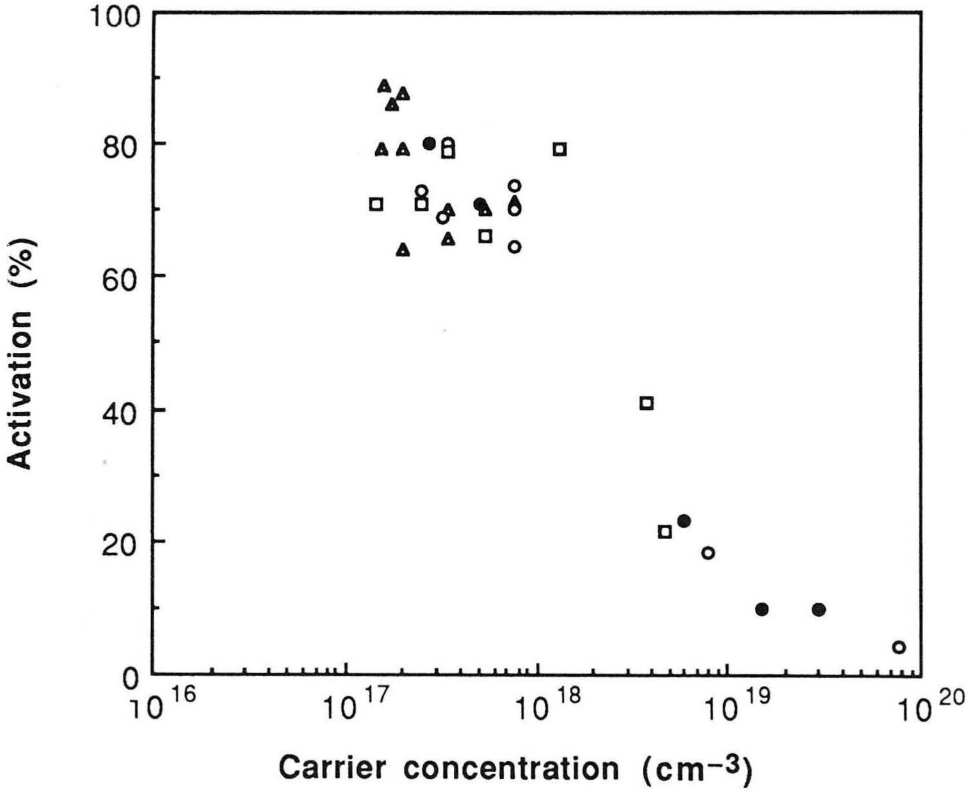
ELECTRONIC MATERIALS

SATURATION OF FREE HOLE CONCENTRATION IN Zn-DIFFUSED GaAs AND InP SINGLE CRYSTALS

The inability to increase the free carrier concentration in semiconductors above certain critical levels (saturation of free carrier concentration) can have adverse consequences for device performance. Such a saturation can limit, for example, the efficiency of emitters in bipolar junction transistors, the conductivity of ohmic contacts, as well as the number of free carriers inside the channel of field effect transistors.

CAM researchers Kin Man Yu and Lydia Chan have undertaken studies aimed at understanding the mechanism of this phenomenon in both GaAs and InP. Using a variety of electrical and structural characterization techniques, the behavior of Zn atoms diffused into these materials has been investigated. Surprisingly, the role of Zn differs in these two examples. Hall effect and capacitance-voltage measurements of Zn-diffused GaAs have shown that 95% of all the $\sim 2\times10^{20}$cm Zn atoms are acting as electrically active acceptors. This is in agreement with particle induced X-ray emission (PIXE) channeling experiments which indicate that almost all the diffused Zn atoms are substituting in Ga sites. In contrast to this result, the concentration of free holes in Zn-diffused InP (Zn concentration $\sim 2\times10^{19}$ cm^{-3}) is severely limited to only about 3×10^{18} cm^{-3}. This indicates that most of Zn atoms here are electrically inactive. Again, PIXE channeling data is illuminating, (Figure 1) here showing that Zn atoms are randomly distributed in the InP crystal lattice, not occupying In sites on which they would act as shallow acceptors, but rather forming a variety of precipitates. The presence of these Zn precipitates, identified as the Zn_3P_2 and Zn phases, was confirmed by transmission electron measurements (TEM) (Figure 2). Thus the phenomenon of free hole saturation can be attributed to preferential formation of new solid phases by acceptor impurities at the expense of their remaining dispersed in the lattice sites.

Semiconductors Project: Eugene E. Haller, Project Leader, (415) 486-5294; Wladek Walukiewicz, (415) 486-5329; Kin Man Yu, (415) 486-6656; Lydia Chan, (415) 486-5647.



Activation of silicon implants on wafer from crystals grown with dry B_2O_3. Our data (•) are compared with activation obtained by others on semi-insulating LEC grown crystals. (△) Kanber et al., J. Applied Physics, 57, 4732, (1985), and Appl. Phys. Lett., 120, 47, (1985); (○) Pearton et al., J. Electrochem. Soc., 132, 2743, (1985); and (□) Bindal et al., J. Applied Physics, 65, 1246, 1989. XBL 907-2359

Center for Advanced Materials

CAM RESEARCH NOTE

ELECTRONIC MATERIALS

HIGH PURITY SEMI-INSULATING GaAs CRYSTALS GROWN THROUGH FULLY ENCAPSULATED VERTICAL GRADIENT FREEZE

CAM researchers recently reported the reproducible growth of very low dislocation density gallium arsenide crystals through the use of an advanced vertical gradient freeze technique (see Research Notes 2/1). Crystals grown in this manner were shown to have a defect density as much as two orders of magnitude lower than those produced using the Czochralski technique. In the vertical gradient freeze technique, however, the crystal grows in direct contact with the crucible wall. As a result, the crucible must be non-reactive and must not be wetted by the GaAs melt. Clean pyrolytic boron nitride (PBN) is widely used as crucible material in the Czochralski technique, but it is partially wetted by GaAs preventing reproducible crystal growth.

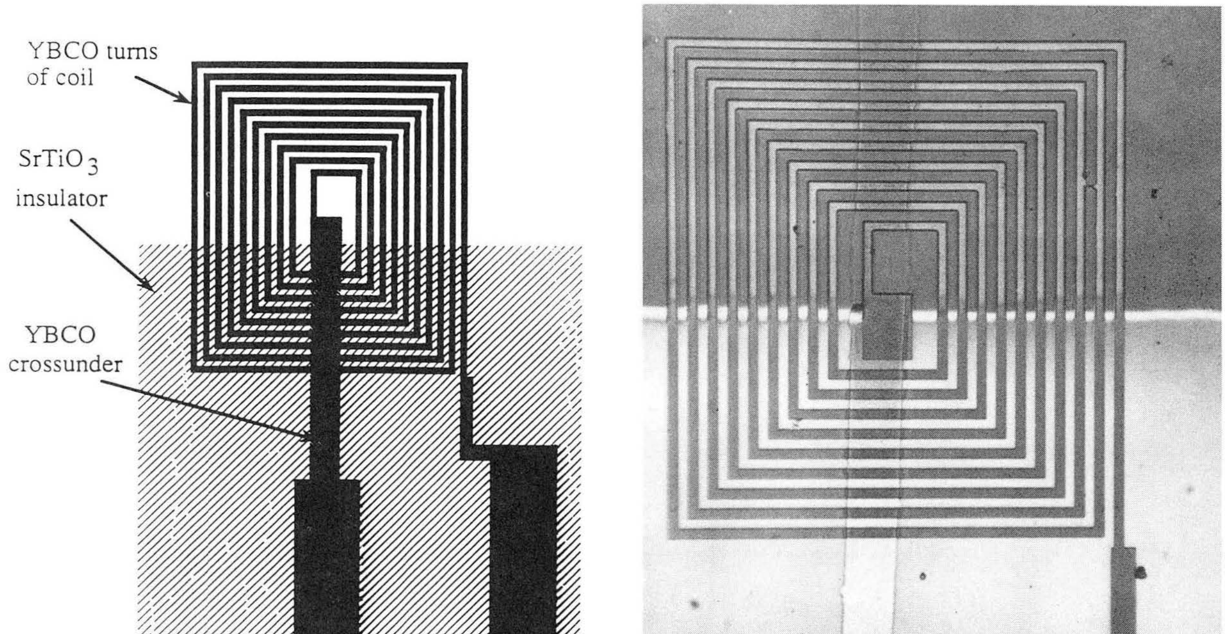
The Crystal Growth group of the CAM Electronic Materials Program has now developed a total liquid encapsulation technique for use in the reproducible growth of 50 mm diameter GaAs single crystals in PBN crucibles. The technique not only prevents unwanted nucleation due to GaAs wetting of the PBN, but also prevents silicon contamination that can arise when a quartz ampoule is used as the growth chamber.

In this technique, a "cap" of liquid B_2O_3 covers the molten GaAs and an approximately 50 μm layer of the B_2O_3 is maintained between the GaAs and the crucible. Thus, wetting is prevented and vaporized Si cannot diffuse into the growing crystal. Dry B_2O_3([OH] > 600 ppm) is fully effective in preventing contamination by silicon, although the absence of free OH also prevents the gettering of C and B from the melt. The net effect, however is the production of crystals of purity comparable to crystals grown by Czochralski but with greatly reduced defect densities. Crystals grown in this manner have been ion-implanted with silicon. Resulting activation efficiencies for implants are similar to those of implants in LEC crystals (Figure 1).

These results demonstrate that the vertical gradient freeze technique, with total liquid encapsulation is a viable technique for producing device quality semi-insulating GaAs substrates even in presence of a quartz chamber.

Bourret, E.D., and E.C. Merk, "Effects of Total Liquid Encapsulation on the Characteristics of GaAs Single Crystals Grown by the Vertical Gradient Freeze Technique," *J. Crystal Growth*, (submitted).

Semiconductors Project: Eugene E. Haller, Project Leader, (415) 486-5294; Edith Bourret, (415) 486-5553.



Schematic (a) and photomicrograph (b) of superconducting crossover. $SrTiO_3$ film insulates YBCO "crossunder" lead to center of coil from other turns of the coil. All three films are epitaxially grown on single crystal MgO. XBL 906-2138, CBB 902-874

Center for Advanced Materials

CAM RESEARCH NOTE

HIGH-T_C SUPERCONDUCTIVITY

INSULATING CROSSOVERS AND MULTITURN COILS FROM THIN FILMS OF $YBa_2Cu_3O_{7-x}$

A new process for insulating crossovers of high-temperature superconductors has been devised by CAM scientists Frederick Wellstood, John Kingston, Andrew Miklich, and John Clarke. It has been used successfully in the fabrication of planar, multiturn coils of $YBa_2Cu_3O_{7-x}$ (YBCO) designed as the input coils of thin film SQUIDs (Superconducting QUantum Interference Devices). The insulating crossover is fabricated through the successive deposition of three thin films by pulses from an excimer laser incident on a rotating target. The first film is deposited through a shadow mask as a 100-µm-wide "crossunder" of YBCO on a single crystal of MgO maintained at 735°C in 200 mTorr of oxygen. A $SrTiO_3$ film is then deposited over the lower portion of the YBCO strip with a substrate temperature of 670°C. The third film (YBCO) is then deposited at 735°C and subsequently patterned using photolithography and Ar-ion etching to produce a spiral coil, the innermost end making a superconducting contact to the crossunder. No post annealing is required in this process. Transmission electron microscope studies at LBL's National Center for Electron Microscopy confirm that epitaxy extends from the substrate through all three layers. Both 10 and 19 turn coils have been successfully fabricated; the best coil had a transition temperature of 82K. The degree of electrical isolation between the turns and the crossunder is typically $10^7\ \Omega$, several orders of magnitude greater than required. The coils are eminently suitable for operation at 77K.

Achievement of this structure demonstrates that it is now feasible to fabricate multilayer circuits from high-transition-temperature (T_c) superconductors. This is a critical advance in SQUID fabrication since spiral coils, which are used in all low-T_c planar SQUIDs to couple the input signal, require a superconducting contact to the innermost turn without producing an electrical short to the remaining turns. As far as is known, this is the first superconducting crossover technology and the first fabrication of thin film multiturn coils that are superconducting at 77K. Two patent applications have been filed. A collaboration with Conductus, Inc. of Sunnyvale, California is focused on commercialization of devices using this technology.

Wellstood, F.C., J.J. Kingston, J. Clarke, "Superconducting Thin-Film Multiturn Coils of $YBa_2Cu_3O_{7-x}$," submitted to *Appl. Phys. Lett.*

Kingston, J.J., F.C. Wellstood, P. Lerch, A. Miklich, J. Clarke, "Multilayer $YBa_2Cu_3O_x$-$SrTiO_3$-$YBa_2Cu_3O_x$ Films for Insulating Crossovers," *Appl. Phys. Lett.*, 56, 2, 1990.

High T_c Superconductivity Program: Norman E. Phillips, Program Leader, (415) 486-4896; John Clarke, (415) 642-0330; Frederick C. Wellstood, (415) 642-3069; John J. Kingston, (415) 642-3069; and Andrew Miklich, (415) 642-4376.

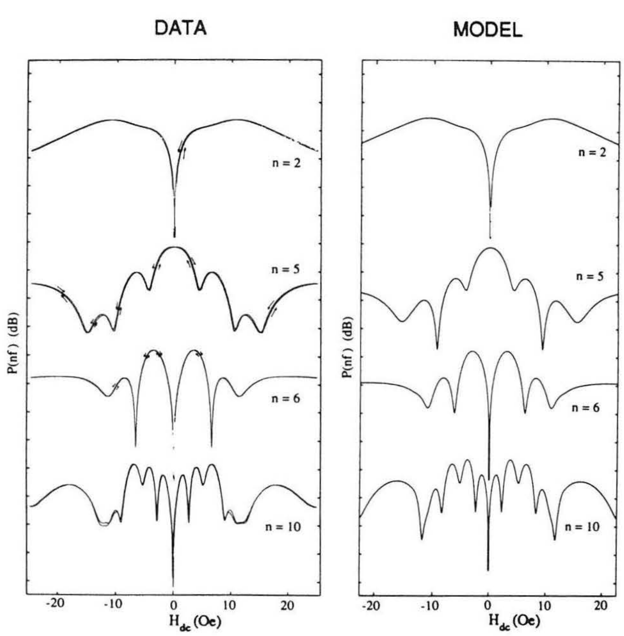


Figure 1
Data (a) and critical state model (b) prediction for harmonic generation in a YBCO rod at a temperature of 77 K driven by an ac magnetic field at 10 kHz. XBL 902-346B

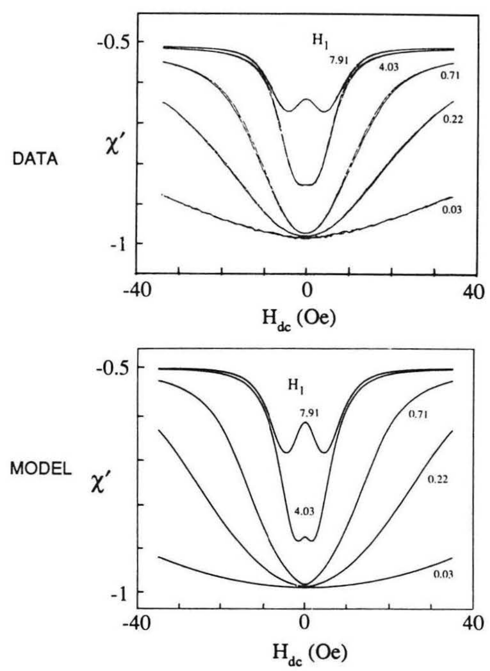


Figure 2
Ac magnetic susceptibility data and critical state model prediction for a YBCO rod at 77 K in dc magnetic field driven by various amplitudes of ac magnetic field, in Oe, at 10 kHz. At very low fields vortices penetrate the space between superconducting grains in this granular material. XBL 902-345

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CAM RESEARCH NOTE

HIGH-T_C SUPERCONDUCTIVITY

NONLINEAR ELECTRODYNAMICS IN YBCO: EXPERIMENTS AND MODELS

Shortly after their discovery it was realized by many observers that high-T_c superconductors are granular and have a complex microstructure. They may be modelled as composites of superconducting *grains* connected by *weak links*, for example, Josephson junctions. These two regions are referred to as intragranular and intergranular, respectively, and in addition to limiting the transport critical current, they give rise to rich nonlinear electrodynamic behavior: extensive harmonic generation when driven by an ac magnetic field, and anomalous dependence of the ac magnetic susceptibility on quite low magnetic fields, of the order of a few Oersteds (Oe).

Recently CAM scientists Carson Jeffries, Harry Lam, and Youngtae Kim have made a detailed experimental study of these novel effects. Using a cylindrical rod of polycrystalline $YBa_2Cu_3O_7$ at a temperature of 77K, they measured the harmonic power generated when driven by an ac field (13.5 Oe) at a frequency of 10 kHz while slowly scanning a coaxial dc magnetic field from ±20 Oe. Figure 1 shows the rather complicated dependence on the dc field for various harmonics n = 2, 5, 6, 10. Figure 2 shows ac susceptibility measurements on the same sample at the fundamental frequency (n=1), which shows a complex dependence on small ac and dc magnetic fields.

The CAM group has also developed a theoretical model that is in excellent agreement with these measurements. It is based on the idea of a critical state, developed in the 1960's by Bean, Anderson, and Kim, to explain magnetic hysteresis and loss in low-temperature Type II superconductors. The model assumes that at small applied fields, ~1 Oe, vortices penetrate the intergranular space of the cylindrical sample to a depth dependent on the maximum current density J_c. This critical current density is assumed to depend on the vortex pinning force and inversely as the square of the local magnetic field in the sample. The excellent fit between data and model is achieved with only two adjustable parameters. A similar model is applicable for fields greater than approximately 100 Oe, when vortices begin to penetrate the superconducting grains themselves.

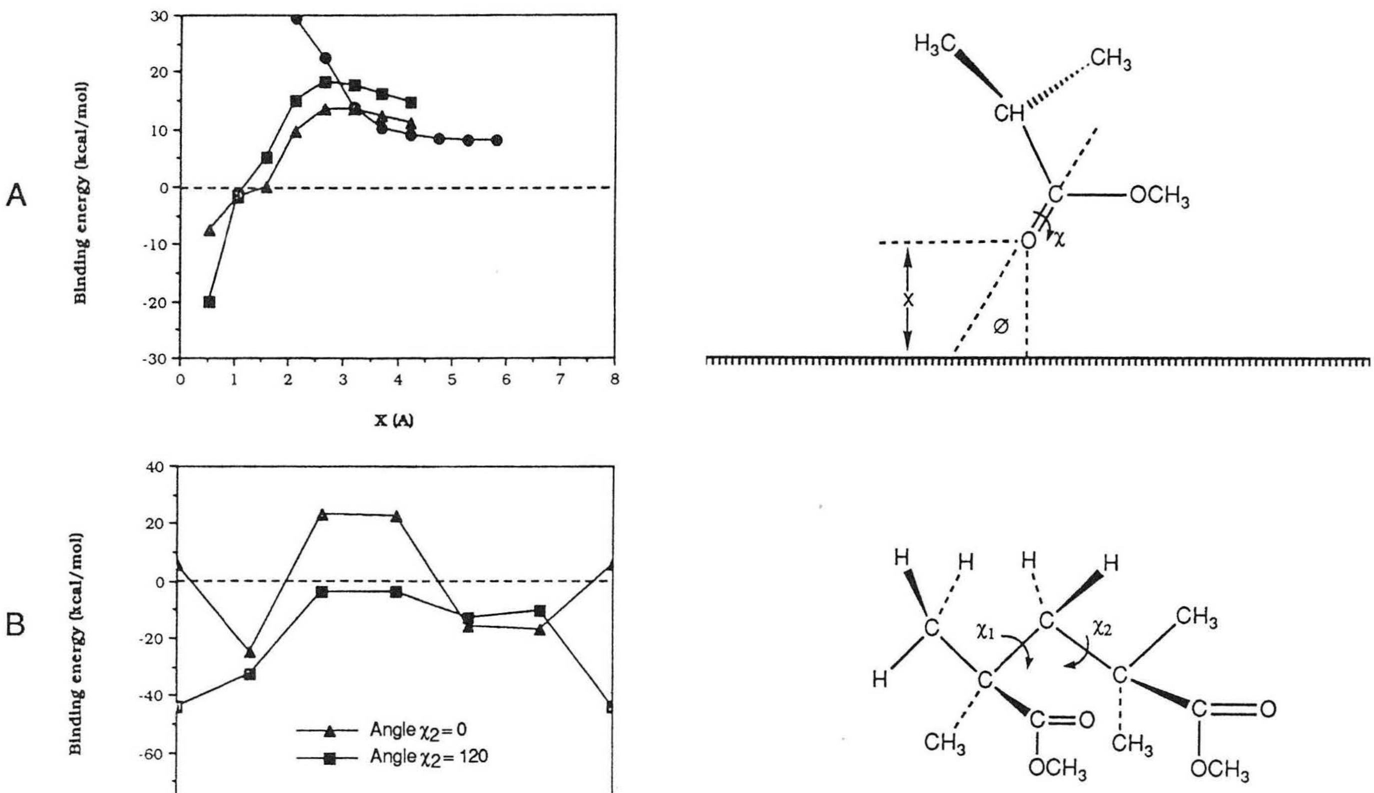
These experimental methods and models are found to be quite sensitive to pinning forces and critical currents, and are thought useful for characterization of superconductors. Continuing interest will be focused on thin films and single crystals.

Kim, Y., C.D. Jeffries, "AC Susceptibility of Granular Superconductor $YBa_2Cu_3O_7$," *Bull. Am. Phys. Soc.* 35, 338 (1990).

Lam, Q.H., C.D. Jeffries, "Harmonic Generation in Sintered $YBa_2Cu_3O_7$," *Bull. Am. Phys. Soc.* 35, 339 (1990).

Kim, Y., "Quasiperiodic Transition to Chaos in Ge; and Magnetic Susceptibility of High-T_c Superconductors," Ph.D. thesis 1990, U.C. Berkeley, Department of Physics. Advisor: C.D. Jeffries.

High T_c Superconductivity Program: Norman E. Phillips, Program Leader, (415) 486-4896; Carson D. Jeffries, (415) 486-5894; Harry Lam, (415) 642-3382; Youngtae Kim (415) 642-3017.



Representative portion of energy hypersurfaces for the monomer (A) and dimer (B) of polymethyl-methacrylate (PMMA) interacting with a jellium representation of aluminum surfaces. The different curves in (A) correspond to different values of Ø and χ. The energy hypersurfaces are highly complex and configuration dependent because of the specificity of the chemical interactions. Simple potentials (e.g., Lennard-Jones) can never represent these interactions. The energy hypersurfaces have several (fairly deep) local minima which may lead to non-equilibrium chain conformations at the surface. Molecular simulations using potentials derived from these quantum mechanical calculations will confirm the presence of the non-equilibrium structures that are suggested by the calculated energy hypersurfaces. XBL 906-2115

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CAM RESEARCH NOTE

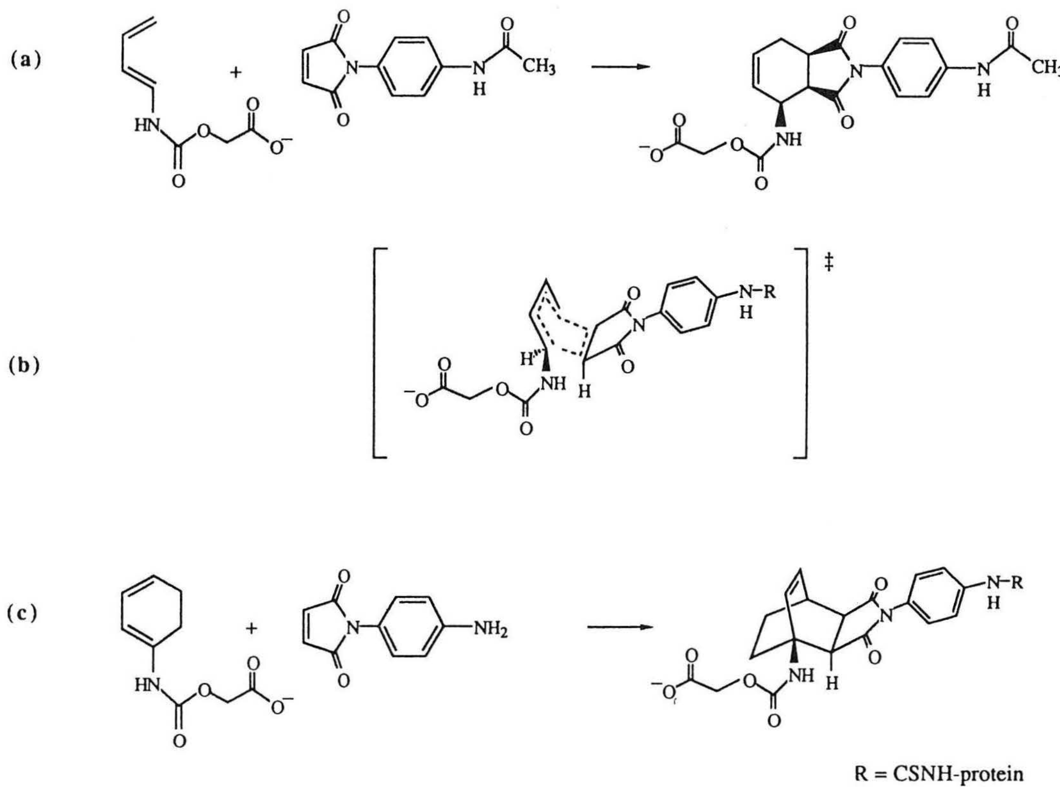
POLYMERS AND COMPOSITES

NEAR-SURFACE STRUCTURE OF POLYMER-METAL INTERFACES

Materials with interfaces between polymers and non-polymeric solid substrates are of growing importance in a variety of applications in the microelectronics, aerospace, and automotive industries. Polymer-metal interfaces are of particular interest because the functional groups of the organic polymer may interact chemically with the substrate. These interfacial chemical interactions coupled with the entropic constraints associated with confining long chains near an interface may lead to unique chain conformation in the near-surface region. Since the chain conformation in addition to the bonding interactions play a crucial role in determining the mechanical and diffusion barrier properties of the interface, a fundamental understanding of how the electronic and stereochemical structure of the polymer and the electronic structure of the substrate influence near-surface structure would greatly aid the design of new interfaces with prescribed properties.

CAM researchers Arup K. Chakraborty and James S. Shaffer, have approached these questions with large-scale computer calculations using quantum and statistical mechanical theories. Since the interfacial interactions between segments of the polymer and reactive metal surfaces cannot be represented by simple potentials, they have used quantum mechanical density functional theory to calculate the energy hypersurfaces for the interactions of oligomers with metal surfaces. Two models are used to represent the metal surface; the jellium model and the cluster model. The calculations do lead to prediction of both structure and bonding at reactive polymer interfaces and the energy hypersurfaces are found to be extremely complex and configuration dependent with several local minima. They are such that the structure of adsorbed chains is predicted to be a collection of non-equilibrium configurations. In other words, the interfacial region can be considered to be a quasi two-dimensional glass which may have rather unique properties. The modulation of the electronic properties of the surface upon adsorption is found to be spatially variant, which leads to predictions of STM images of adsorbed layers that are consistent with recently published experiments.

Polymer-Substrate Interactions Project: Doros Theodorou, Project Leader, (415) 642-0176; Arup K. Chakraborty, (415) 642-9275; J.S. Shaffer, (415) 642-7318.



R = CSNH-protein

(a) Antibody-catalyzed Diels-Alder reaction
(b) Schematic representation of the transition state
(c) Synthesis of the transition state analogue
XBL 908-2748

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CAM RESEARCH NOTE

POLYMERS AND COMPOSITES

ANTIBODY GENERATED FOR CATALYSIS OF "NON-BIOLOGICAL" REACTION

The use of naturally occurring enzymes in the synthesis of novel compounds and materials will likely be restricted to the synthesis of products commonly thought of as "biological," including unusual variants of polyamides, lipids, and polysaccharides. Important as these new materials will be, the attractive properties of enzymes for catalyzing materials synthesis—including their high rates of reaction, extraordinary specificity for starting material and product, absence of toxic by-products, and low energy requirements—make it desirable to identify enzyme activities that can catalyze reactions leading to "non-biological" substances as well.

CAM scientists Peter Schultz and Andrew Braisted have developed such an enzyme activity—in a "catalytic antibody." The reaction catalyzed is the Diels-Alder addition, one of the two most important reactions in organic synthesis. The antibodies were produced in a mouse after injection with a "transition state analog" for the reaction. This analog is a stable molecule, designed and synthesized to mimic in shape, the high energy intermediate in the actual reaction. Antibodies to that molecule bind the two substrates for the reaction and force them into the shape and relative position of the transition state, thus helping to accelerate the aqueous reaction to a rate one million times faster than the uncatalyzed reaction in a typical organic solvent such as acetonitrile.

Although there exist over 1500 known enzymes, there are no documented examples of any that catalyze the Diels-Alder reaction. In fact this reaction is not known to occur in any living organism despite its role in forming carbon-carbon bonds, the backbone of most molecules of interest, from proteins to polyethylene. Not only does this achievement therefore illustrate the power of the catalytic antibody approach for generating tailor-made enzyme-like catalysts, it also opens the door to the production of a host of interesting catalysts for the synthesis of new, complex molecules in a highly controlled manner. Continuing work involves investigating the use of such antibodies to synthesize novel polymeric materials.

Enzymatic Synthesis of Materials Project: Mark Alper, Project Leader, (415) 486-6581; Peter Schultz, (415) 642-9277; Andrew Braisted, (415) 642-6026.

This work supported by the Division of Materials Science, and also the Division of Energy Biosciences, of the U.S. Department of Energy.

Alloy	Strain Rate s^{-1}	Temp ^{o}C	Elongation %	Flow Stress MPa (ksi)	Strain-rate Sensitivity
Al-2.2Li-0.5Sc	0.0010	400	305	27 (4.0)	-
	0.0100	500	470	16 (2.3)	0.32
Al-2.0Li-2.2Mg-0.5Sc	0.0100	400	360	90 (13)	0.30

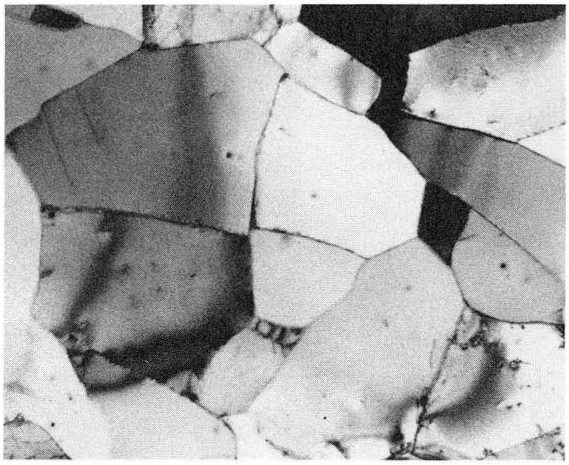

Figure 1
TEM micrograph of the deformed gage region in an Al-2.2Li-0.5Sc sample tested at 500ºC. XBB 906-4621

Center for Advanced Materials

CAM RESEARCH NOTE

STRUCTURAL MATERIALS

DEVELOPMENT OF LIGHTWEIGHT SUPERPLASTIC ALLOYS FROM THE Al-Li-Sc-Mg SYSTEM

The combination of high strength and low density in a superplastically formable alloy is a very important goal of the aerospace industry. Previous researchers have produced superplastic alloys from the Al-Sc-Mg system with high strengths using a combination of precipitation and dislocation strengthening (cold work). Because superplastic forming will destroy the dislocation strengthening, additional precipitation strengthening is required to make these alloys attractive for use in aerospace applications. Researchers at CAM have attempted to deal with this problem through the addition of lithium to the alloy in order to provide additional precipitation strengthening through the formation of ordered Al_3Li. Addition of lithium also has the advantage of lowering the density and increasing the stiffness of the alloy.

A special melting facility has been constructed to allow the first melting and casting of alloys from the Al-Li-Sc-Mg system. This furnace uses a water-cooled chill mold that solidifies the alloy at approximately 10°C/s. This rapid cooling is required to keep the scandium in solid solution so that it can be precipitated out as Al_3Sc during thermo-mechanical processing of the alloy. The furnace also uses vacuum induction melting and argon gas flushing for the removal of dissolved gases. Alloys currently being studied have the nominal compositions: Al-2.2Li-0.5Sc and Al-2.0Li-0.5Sc-2.0Mg.

While classical superplasticity involves high-angle grain boundary sliding of fine grained materials, Li, Mg alloys of Al-Sc, which the CAM group has shown are superplastic (Table 1), appear to behave in that fashion along primarily low-angle boundaries. Figure 1 shows the fine grain size of these alloys even after deformation at 500°C. Ongoing research into these alloys is focused on understanding both the mechanism of superplastic behavior and also the evolution of microstructure during thermomechanical processing and superplastic deformation.

Metals for Advanced Applications Project: J.W. Morris, Jr., Project Leader, (415) 486-6482; Roger A. Emigh, (415) 486-6035; Edwin L. Bradley, III, (415) 486-6035.

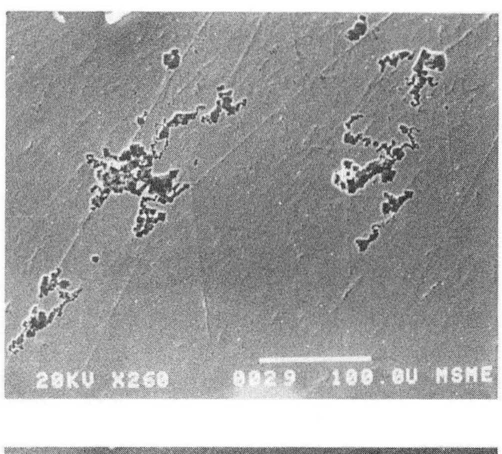

Figure 1
Low magnification (260x) scanning electron micrographs of crystallographic pit in 99.999% aluminum obtained with 25 kV electrons (a) and 4 kV electrons (b). The 25 kV electrons are sufficiently energetic to penetrate the film covering the pit. The image obtained with 4 kV electrons reveals the presence of the thin film (which is also optically transparent) that covers the pit. (XBB 903-1804)

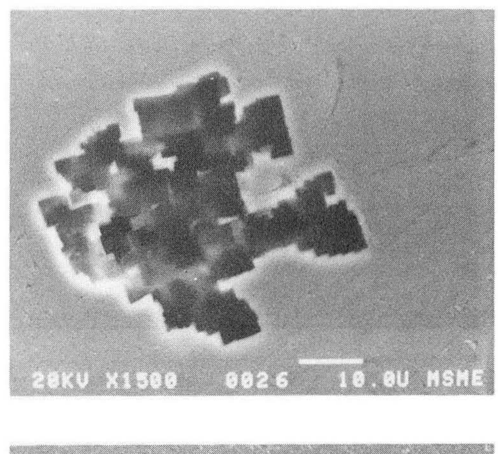

Figure 2
Higher magnification (1500x) view of high- (a) and low- (b) kV scanning electron micrographs of small crystallographic pit in 99.999% aluminum. The lower energy electrons in (b) are unable to penetrate the thin, optically transparent film that covers the pit. (XBB 903-1806)

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CAM RESEARCH NOTE

STRUCTURAL MATERIALS

ROLE OF SURFACE FILMS IN THE PROPAGATION OF LOCALIZED CORROSION IN Al AND Al ALLOYS

Paradoxically, most load-bearing components in structures that are exposed to chemically reactive environments are made of highly reactive metals such as iron, nickel, cobalt, chromium, titanium, aluminum, and their alloys. These materials are corrosion resistant as a result of the fact that they spontaneously form a thin (1-10 mm) passive film on their surfaces that isolates and protects them from the environment. These films are so protective that engineering structures made of passive alloys rarely fail by general dissolution. Instead, when corrosion-related failures do occur, they are usually the result of highly localized breakdown in the passive film. One such localized corrosion process is known as pitting corrosion.

Most pits formed in passive alloys are hemispherical in shape. In practically-occurring environments, however, the pits that form in aluminum and its alloys are distinctly crystallographic. Although this fact is well known it is not well understood. The puzzle stems from the fact that crystallographic effects are only important in corrosion reactions that occur with a low thermodynamic driving force (on the order of 0.01 volt). Yet the apparent driving force for corrosion of pits in aluminum is very large (over 1 volt).

Recently, CAM scientists Christopher Kumai and Thomas Devine have identified a possible explanation for this phenomenon. They discovered that a thin, optically transparent (probably an oxide of aluminum) film covers the crystallographic pits (Figure 1) and inhibits mixing of the bulk solution with that in the pit. As a result, large concentrations of H^+ and Cl^- develop in the pit which prevent the formation of a protective passive film on the newly corroded surface and lead to the evolution of hydrogen gas. The gas bubbles act to stretch and lift the film off of the metal surface, thereby exposing bare metal to the aggressive solution and continuing the propagation of the pit. The small cross-sectional area of the solution in the pit is further lowered by the formation of hydrogen gas bubbles. The very narrow electrolyte path that consequently exists inside the pit produces a large IR drop between the primarily anodic area at the tip of the pit and the primarily cathodic area outside of the pit. This reduces the overpotential for dissolution at the tip. Consequently, the pit propagates at a high velocity because of the reactivity of bare aluminum, but the morphology of the pit is crystallographic because the thermodynamic driving force for oxidation is low.

Kumai and Devine have also shown that a thin optically transparent film covers corroding grain boundaries in aluminum-lithium alloy 2090 and appears to play an important role in the propagation of intergrannular corrosion in that material. Current research focuses on an exploration of the function of films in the mechanism of localized corrosion of a number of important aluminum alloys.

Metals for Advanced Applications Project: J.W. Morris, Jr., Project Leader, (415) 486-6482; Thomas M. Devine, (415) 643-8118; Christopher S. Kumai, (415) 486-7404.

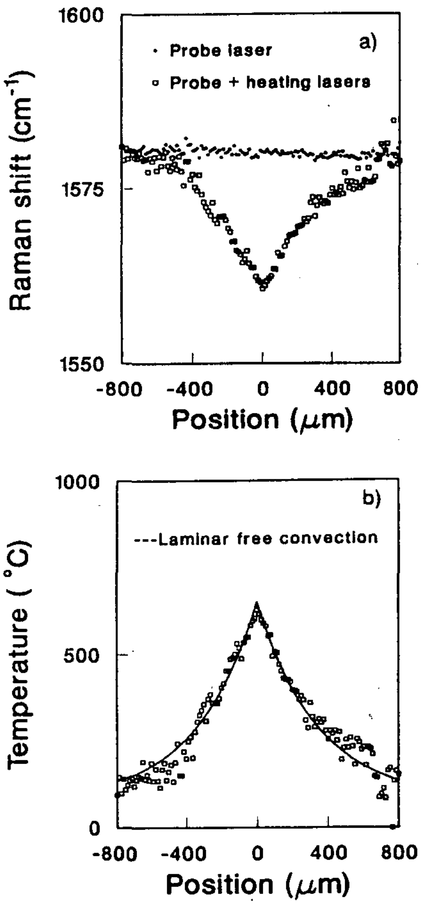

Figure 1
A profile of the change in Raman frequency along a carbon fiber shows the magnitude of laser heating. Laser heating introduces artifacts that must be corrected for to obtain the true signal from the fiber. (a) The frequency of the graphite Raman band is measured as a function of distance along the fiber illuminated by 100 mm focused heating laser. The spatially-resolved spectra are collected simultaneously. (b) Increased temperatures along the fiber are calculated from the frequency shifts. The solid line shows the temperature gradient expected for convective heat transfer. XBL 906-2067

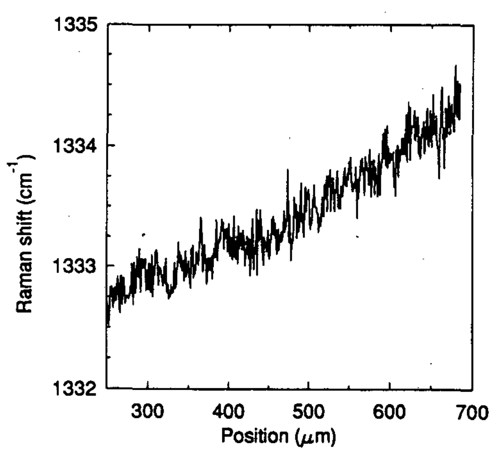

Figure 2
A profile of the Raman frequency of the 1332.5 cm^{-1} diamond peak across a plasma assisted CVD diamond film shows that the microcrystalline structure is inhomogenous. The shift to higher frequencies is attributed to increasing defect densities. XBL 906-2068

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CAM RESEARCH NOTE

SURFACE SCIENCE AND CATALYSIS

MAPPING MATERIALS PROPERTIES WITH RAMAN SPECTROSCOPY

Spectroscopic imaging of the chemical state or a physical property of a material can lead to new information on property-structure-processing relationships and thus to new insights into optimization and performance in critical applications. Imaging based upon vibrational Raman spectroscopy can provide spatially-resolved information on a number of chemical properties—chemical composition, thermodynamic phase, applied or residual stress, and temperature.

CAM researchers Gerd Rosenblatt, Kirk Veirs, Joel Ager, III, and Eric Loucks have developed an imaging Raman system based upon a low-noise, two-dimensional (1024 x 1024 pixel) detector. The system is capable of simultaneously collecting 1024 Raman spectra representing elements along a narrow laser-illuminated line. The spatial resolution is 5 μm and the spectral precision is 0.16 cm^{-1}. Automated translation of the sample across the illumination line, along with real-time data, and repeated spectral collection and analysis cycles, allows rapid generation of two-dimensional maps of chemical or physical properties.

The system has been applied to a number of materials problems through collaborations with other groups at CAM and LBL. In one case, growth of fatigue cracks in ceramics was studied. (see *Research Notes* 3/1, 1989). In another, Raman band positions, widths, and relative intensities were used to characterize high-modulus carbon fibers annealed at different temperatures. Since the laser itself heats the sample affecting the Raman spectrum (Figure 1), this effect must be understood and compensated for to determine the effects of changes in annealing temperature.

In a third application, imaging Raman spectroscopy was used to characterize carbon and diamond films grown by Ian Brown in LBL's Accelerator and Fusion Research Division. Spectra obtained along a line across a diamond film show systematic shifts to higher energies in the position of the 1332.5 cm^{-1} diamond phonon (Figure 2). The increasing shifts can be related to increasing densities of crystalline defects, thus showing that the plasma-assisted CVD process produces diamond films that are inhomogenous. Such information helps lead to improved film deposition conditions.

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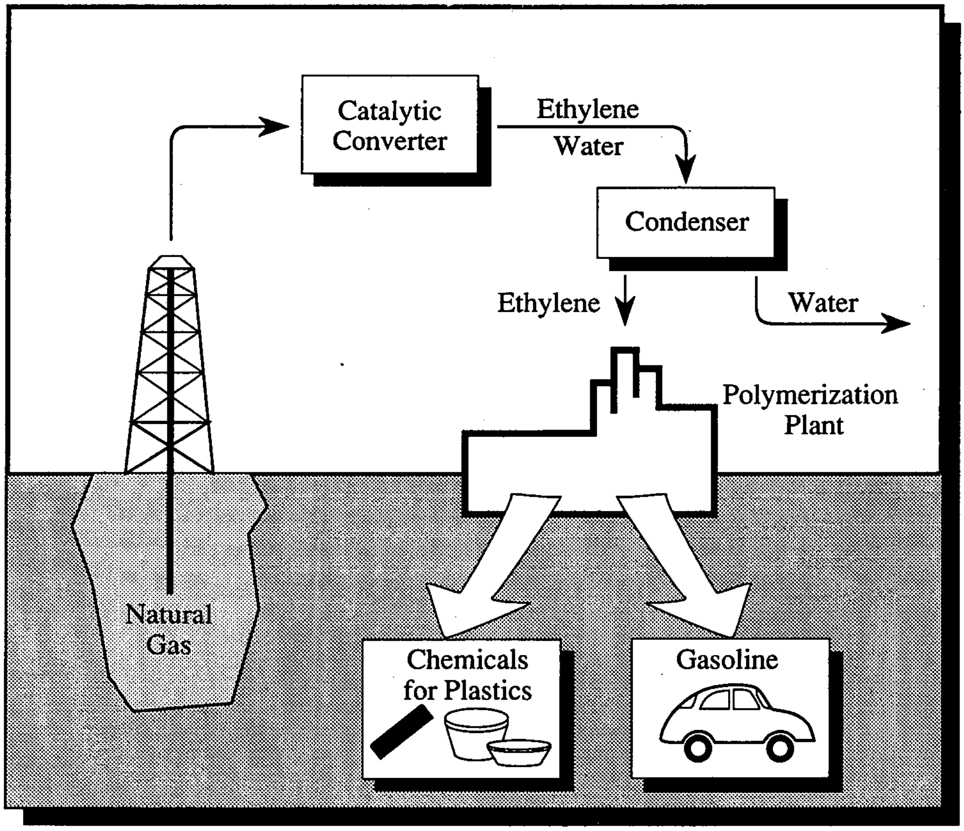
Ager III, J.W., D.K. Veirs, J. Shamir, and G.M. Rosenblatt, "Laser-Heating Effects in the Characterization of Carbon Fibers by Raman Spectroscopy," *J. Appl. Phys.*, (in press).

Instrumentation for Surface Science Project: John Clarke, Project Leader, (415) 642-0330; Gerd M. Rosenblatt, (415) 486-6606; D. Kirk Veirs, (415) 486-6715.

Supported by Exploratory Research and Development Funds.



Conversion of methane to higher hydrocarbons. (XBL 908-5735)

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CAM RESEARCH NOTE

SURFACE SCIENCE AND CATALYSIS

OXIDATIVE COUPLING OF METHANE PRODUCES OLEFINS AND PARAFFINS

The efficient conversion of methane (CH_4) to ethylene (C_2H_4), propylene (C_3H_6) and paraffins is a major, yet elusive goal in energy research. Methane is available in great abundance in the United States but is generally unreactive; ethylene and propylene are not abundant, but are easily converted into gasoline and petrochemicals.

CAM Scientists, Heinz Heinemann, Pedro Pereira, and Gabor Somorjai, have recently identified novel catalysts and operating conditions that permit these conversions. Using CH_4 and oxygen, selectivities of almost 100% are achieved with methane conversions of 10–12% per pass. Essentially no carbon oxides (CO and/or CO_2), are produced in contrast to existing published technologies which involve substantial CO_2 formation. The new process, therefore, would allow major savings in oxygen consumption and the cost of CO_2 removal from recycle streams.

The reaction is carried out at the relatively low temperature of about 600 °C in the presence of steam and below flammability limits. The catalysts which exhibit good stability and lifetime are ternary mixtures of alkali, earth alkali, and transition metal oxides. The mechanism of the reaction appears to involve surface catalysis with essentially no homogeneous gas phase reaction occurring. An induction period during early operation suggests that deposition of carbon on the catalyst may play a role in its activity.

Catalyst preparation involves careful proportioning and pretreatment. In addition to ethane and ethylene, C_3 and C_4 hydrocarbons are produced in amounts that depend on the precise method used in catalyst preparation. Similarly, the paraffin/olefin ratio of the products can be controlled by catalyst pretreatment.

Patent applications have been filed.

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Catalyst Design, Characterization and Applications Project: Alexis T. Bell, Project Leader, (415) 642-1536; Heinz Heinemann, (415) 486-5796, Pedro Pereira, (415) 486-4829; Gabor A. Somorjai, (415) 486-4831.

Supported by the Office of Fossil Energy, U.S. Department of Energy.

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October 1, 1990

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