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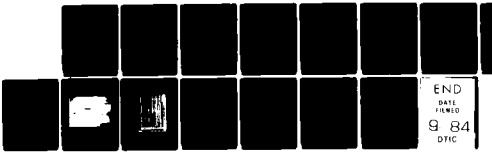

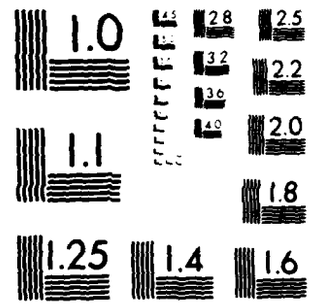

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Laser Control of Chemical Reactions

by

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CHEMICAL VAPOR DEPOSITION
HETEROGENEOUS CATALYSIS
MICROELECTRONICS

20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A review of experimental results for laser control of chemical reactions is presented, with mention of corresponding theoretical progress. Two general classes of reactions are discussed: gas-phase reactions and reactions occurring at a gas-solid interface. For the first class the following topics are reviewed: state-selected reactions, new reaction pathways and transition-state spectroscopy. For the second class the following topics are reviewed: desorption, chemical vapor deposition, heterogeneous catalysis and microelectronics.

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LASER CONTROL OF CHEMICAL REACTIONS

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A review of experimental results for laser control of chemical reactions is presented, with mention of corresponding theoretical progress. Two general classes of reactions are discussed: gas-phase reactions and reactions occurring at a gas-solid interface. For the first class the following topics are reviewed: state-selected reactions, new reaction pathways and transition-state spectroscopy. For the second class the following topics are reviewed: desorption, chemical vapor deposition, heterogeneous catalysis and microelectronics.

A1

Introduction

The phenomenon of "light amplification by stimulated emission of radiation" (the laser), discovered 25 years ago, has had a significant impact on the research programs of the physical, engineering and biological communities. Although the major effort has been in laser development, a variety of laser applications have been explored, such as the use of lasers as scalpels in surgery, as means of communication by fiber optics, and as components in programs of nuclear energy development. The application of lasers in chemistry is now established as an important interdisciplinary effort among various fields such as chemical kinetics, spectroscopy, surface chmistry and optics, and whereas this has generally been confined to basic research projects, it is beginning to see practical advantages in government and industry.

A fundamental and exciting problem in laser-induced chemistry is how lasers can be used to control chemical reaction dynamics. This article addresses this problem on two fronts. The first focuses on gas-phase chemical reactions, which are discussed in the next three sections on: state-selected reactions, new reaction pathways and transition-state spectroscopy. The second front focuses on chemical processes occurring at a gas-solid interface, where a section is devoted to desorption and chemical vapor deposition. The last two sections deal with applications of laser-induced gas-surface processes to heterogeneous catalysis and microelectronics.

State-selected reactions

The electromagnetic field associated with a laser beam has four controllable parameters: the frequency, polarization, intensity and propagation vector. The

ability of the experimentalist to vary these parameters, together with the coherence of the radiation, produces a flexible stimulator and probe of chemical dynamics. The frequency has been extensively exploited, where due to its high degree of monochromaticity laser light can be used to excite a reagent to a specific internal state. Under low pressure conditions such as in a molecular beam apparatus, one can then carry out state-selected chemical reactions and compare the reactivities of different states. Such an experiment was carried out in 1971 by Philip R. Brooks and co-workers at Rice University, who studied the reaction K + HCl(v=1) $\longrightarrow$ KCl + H with crossed K and HCl beams. A pulsed HCl laser was used to vibrationally excited HCl molecules to v=1 in the beam, and HCl(v=1) was found to react 100 times faster than HCl(v=0). The reaction rate was found to increase only 5 times for an equivalent increase (35 kJ/mol) in translational energy.

More recently, Charles T. Rettner and Ricahrd N. Zare at Stanford University utilized the polarization properties of laser light to study the reaction $Ca(^1P_1)$ + HCl $\longrightarrow$ CaCl + H in a beam-gas scattering geometry. A linearly polarized dye laser was used to prepare the beam of $Ca(^1P_1)$ atoms with their p orbital aligned along the electric vector of the laser beam. Rotation of this vector allowed selection of a p-orbital alignment in the laboratory. It was seen that when the p orbital is aligned parallel to the average relative velocity vector of the reagents, the formation of $CaCl(B^2\Sigma^+)$ is favored, whereas the perpendicular alignment favors $CaCl(A^2\Pi)$.

The laser has also been used as a key tool in product state analysis through the technique of laser-induced fluorescence, which was first realized in 1972 by Richard N. Zare and co-workers. Light from a tunable laser is directed at

the products, and as the laser frequency is varied, product molecules are excited when the laser frequency coincides with an absorption line. The excited molecules then re-emit radiation, i.e., they fluoresce, where the "excitation spectrum" obtained by recording the fluorescence intensity as a function of wavelength reflects the relative populations of the different states in which the products were formed by the chemical reaction.

New reaction pathways

In 1972 L. I. Gudzenko and S. I. Yakovlenko in Moscow, USSR, suggested that radiation might be used to directly probe and stimulate the dynamics of a molecular collision process. This can be understood in terms of case (c) in Figure 1, where cases (a) and (b) refer to state selection of reactions and resonance fluorescence of products, respectively, as discussed above. In case (c) the radiation need not be resonant with energy levels of the reactants or products, but rather can come into resonance between multidimensional potential energy surfaces of the chemical system during the course of reaction. Such a concept offers considerable flexibility in the use of lasers to control the dynamics of a reaction process. For example, since radiative transitions occur between levels of different symmetry, a laser can be used to destroy symmetry correlations in chemical reactions and hence enhance the rate of a symmetry-disallowed reaction. There is a price to pay for this attractive scheme, which is the laser intensity. As Thomas F. George and co-workers at the University of Rochester and others elsewhere have demonstrated theoretically, since the duration of a chemical reaction is on the order of a picosecond, the required laser intensity for case (c) in Figure 1 is generally greater than a

kW/cm^2. As the collision time decreases, the threshold laser intensity increases. Whereas earlier we indicated the importance of laser frequency and polarization, we now have a situation where the intensity is important.

Experimental demonstrations of case (c) in Figure 1 have been provided, beginning in 1978, for the reactions Xe + XY $\longrightarrow$ XeX(B,C) + Y, where XY represents the halogen molecules F_2, Cl_2, I_2 and ICl. The experimentalists involved in such studies include the following people and their co-workers: L. I. Gudzenko, L. V. Gurvich, V. S. Dubov, S. I. Yakovlenko, N. K. Bibinov, I. P. Vinogradov and L. D. Mikkeev in Moscow; Bruce E. Wilcomb and Robert Burnham at the U. S. Naval Reserach Laboratory; Karl L. Kompa at the Max Planck-Institut für Quantenoptik in Garching, W. Germany; and Donald W. Setser at Kansas State University. In the work of Wilcomb and Burnham, for example, a mixture of Xe and Cl_2 was irradiated at 193 nm with a pulsed ArF excimer laser, where the collision-assisted laser-induced absorption resulted in emission of light from the product XeCl(B) at 308 nm and XeCl(C) at 340 nm. It is important to note that the chemical reaction between Xe and Cl_2 under thermal conditions to form XeCl in any state does not occur in the absence of the external radiation due to a high potential barrier on the ground potential energy surface. The laser radiation causes a transition of the non-reacting Xe-Cl_2 complex to an excited potential energy surface which correlates to the B and C states of $XeCl_2$. It is also interesting to note that the radiation from the ArF laser is not resonant with any levels of Xe, Cl_2, XeCl or Cl.

While the above experiments on Xe + XY have been performed in absorption cells, case (c) in Figure 1 was also demonstrated in 1983 in a molecular beam experiment by T. C. Maguire, Philip R. Brooks and Robert F. Curl, Jr. at Rice University. Sodium D-line emission was observed from the intersection of crossed beams of K and NaCl irradiated by a cw dye laser (Figure 2), where this three

beam signal is attributed to Na(3p) resulting from photoexcitation of a fleeting KNaCl transition state. In contrast to the Xe + Cl_2 reaction, the K + NaCl reaction does occur in the absence of the laser, although ground-state sodium atoms, Na(3s), are formed instead of Na(3p).

Transition-state spectroscopy

In the words of John C. Polanyi at the University of Toronto, case (c) of Figure 1 raises the question as to "whether we could now be standing at the threshold of what one might characterize as the last frontier of reaction dynamics, in which we move from the spectroscopy of reactants and products to the spectroscopy of the collocation of particles that really interests us, *the reaction intermediate*." This concept of *transition-state spectroscopy* has been explored theoretically by Michael Hutchinson and Thomas F. George at the University of Rochester and by Polanyi and co-workers, and such a phenomenon indeed promises to play an exciting and fruitful role in the field of chemical kinetics. As a first step toward realizing this, in 1980 Polanyi and co-workers carried out measurements of the chemiluminescence (fluorescence) from the reaction F + Na_2 $\longrightarrow$ NaF + Na(3p) occurring in a crossed molecular beam experiment. In particular, they looked at emission in the wings of the atomic D-line, extending over several hundred angstroms to either side of line center. Such emission gives information on the dynamics associated with the three-body F-Na-Na interactions. The next step in this study is to use a laser which is not resonant with the D-line to probe the interaction region, where the combination of photon absorption from the laser and subsequent fluorescence offers a more flexible way of examining the transition state than the fluorescence alone. This idea was, in fact, applied this year to the reaction Hg + Cl_2 $\longrightarrow$ HgCl($B^2\Sigma^+$) + Cl by C. Jouvet and B. Soep at the University of Paris (Orsay), France, where the reactants were brought together to form a van der Waals complex which was then promoted by laser excitation to a reactive state, and the overall process was monitored by fluorescence from the product HgCl($B^2\Sigma^+$).

Gas-Solid Interface: Desorption and Chemical Vapor Deposition

So far we have been discussing gas-phase reactions. However, a greater percentage of chemistry in the real world occurs at interfaces between two different media, and the use of laser radiation to both probe and stimulate molecular rate processes at a gas-solid interface is being explored by researchers in surface science and chemical kinetics. The first process to receive serious attention in this regard was laser-induced desorption, where M. S. Djidjoev and co-workers at the University of Moscow, USSR, reported in 1976 that a low-power cw CO_2 laser (10 W/cm^2) could induce desorption of hydroxyl groups from a silica surface at room temperature far more efficiently than oven heating. This was confirmed this year by C. Bradley Moore and co-workers at the University of California in Berkeley. Detailed studies of CO_2-laser-induced desorption on others systems have also been reported, such as C_5H_5N and C_5D_5N from KCl and Ag crystals in 1982 by Tung J. Chuang and H. Seki at IBM Research Laboratory in San Jose and CH_3F from a NaCl crystal in 1982 by J. Heidberg, H. Stein and E. Riehl at the University of Hannover, W. Germany, and studies of ArF-laser-induced desorption of CO from a Cu crystal have been reported in 1982 by Eric Weitz and Peter C. Stair and co-workers at Northwestern University (the laser intensities in these experiments were several orders of magnitude higher than in the Soviet and Berkeley experiments). From the results of these and other experiments and from theoretical work by Thomas F. George and co-workers at the University of Rochester (including Franco Battaglia, Avinash C. Beri, Xi-Yi Huang, Michael Hutchinson, Ki-Tung Lee, Kung C. Liu and William C. Murphy), Jui-teng Lin at the Naval Research Laboratory, Horia Metiu and co-workers at the University of California in Santa Barbara, Karl F. Freed at the University of Chicago, Czeslaw Jedrzejek at Jagellonian University, Poland, Shlomo Efrima at Ben Gurion University of the Negev, Israel, Z. W. Gortel and H. Jürgen Kreuzer at Dalhousie University, Canada, and P. Piercy and R. Teshima at the University of Alberta in Alberta, Canada, it is clear that the study of laser-induced bond breaking at surfaces will continue grow as an important area of laser-induced chemistry.

Laser radiation can also be used to promote adsorption or the sticking of gaseous molecules to a solid surface, i.e., chemical vapor deposition. In 1979, Thomas F. Deutsch, Daniel J. Ehrlich and Richard M. Osgood, Jr. at Lincoln Laboratory of the Massachusetts Institute of Technology (Osgood is now at Columbia University) used a frequency-doubled argon-ion laser (UV light) to simultaneously photodissociate gaseous $Cd(CH_3)_2$ and induce absorption of Cd atoms onto a SiO_2 substrate. An especially exciting result of this type of experiment was reported in 1982 by S. R. J. Brueck and Daniel J. Ehrlich at Lincoln Laboratory, where they observed the growth of a periodic pattern of ripples of a Cd film on SiO_2. The dominant ripple structure was aligned perpendicular to the laser polarization direction, and the growth rate was 0.1 - 1 nm/s with a spacing of around 150 nm between adjacent ripples. This periodic growth pattern due to laser-induced chemical vapor deposition was achieved with a laser power of 10 W/cm^2; in order to induce such a pattern directly on the surface without deposition, laser powers which are orders of magnitude higher are required.

Heterogeneous Catalysis

A potential application of laser-induced gas-solid processes is in the area of heterogeneous catalysis, where laser radiation and a surface may be used synergistically to stimulate a particular reaction process. As a step in this direction, in 1980 Ming C. Lin and co-workers at the Naval Research Laboratory irradiated gaseous NO_2 with an argon-ion laser (1 W/cm^2) during its reaction with C_2H_4 over a Pt catalyst at 250°C. They observed a fourfold increase in the CO_2 product yield over that obtained without the laser. This enhancement of the CO_2 yield is believed to result from the reaction of vibrationally-excited NO_2 with adsorbed C_2H_4.

Microelectronics

While laser annealing has been shown to have advantages over oven heating for the processing of semiconductor chips, it appears that laser-induced chemistry at gas-solid interfaces will also find a place in the microelectronics industry. During the past several years, Daniel J. Ehrlich and co-workers at Lincoln Laboratory of the Massachusetts Institute of Technology have adapted laser direct-write aluminum etching and polysilicon deposition to the mask-free alteration of simple gate-array test circuits. Simple test structures on commerical CMOS chips have been reconfigured with no degradation in device or circuit performance. These new methods may be useful for rapid evaluation and optimization of integrated-circuit prototypes.

Specifically, the starting circuit for the work at Lincoln Laboratory is a 500-gate commerical CMOS gate array (1-μm-thick aluminum-silicon alloy metallization combined with polysilicon gates) manufactured using 5-μm design rules (see Figure 3). The etching process for aluminum involves laser heating of an aluminum film bathed in a capillary liquid etchant layer containing a mixture of phosphoric acid, nitric acid and potassium dichromate. The deposition process is carried out by localized laser heating of a surface in a 200-torr ambient of diborance-doped silane vapor, which induces the chemical vapor deposition of heavily B-doped polysilicon, suitable for forming ohmic contacts to aluminum. Further experiments will explore the uses of these techniques for wafer-scale restructurable VLSI, and for isolation and testing of circuits sub-structures within VLSI design prototypes.

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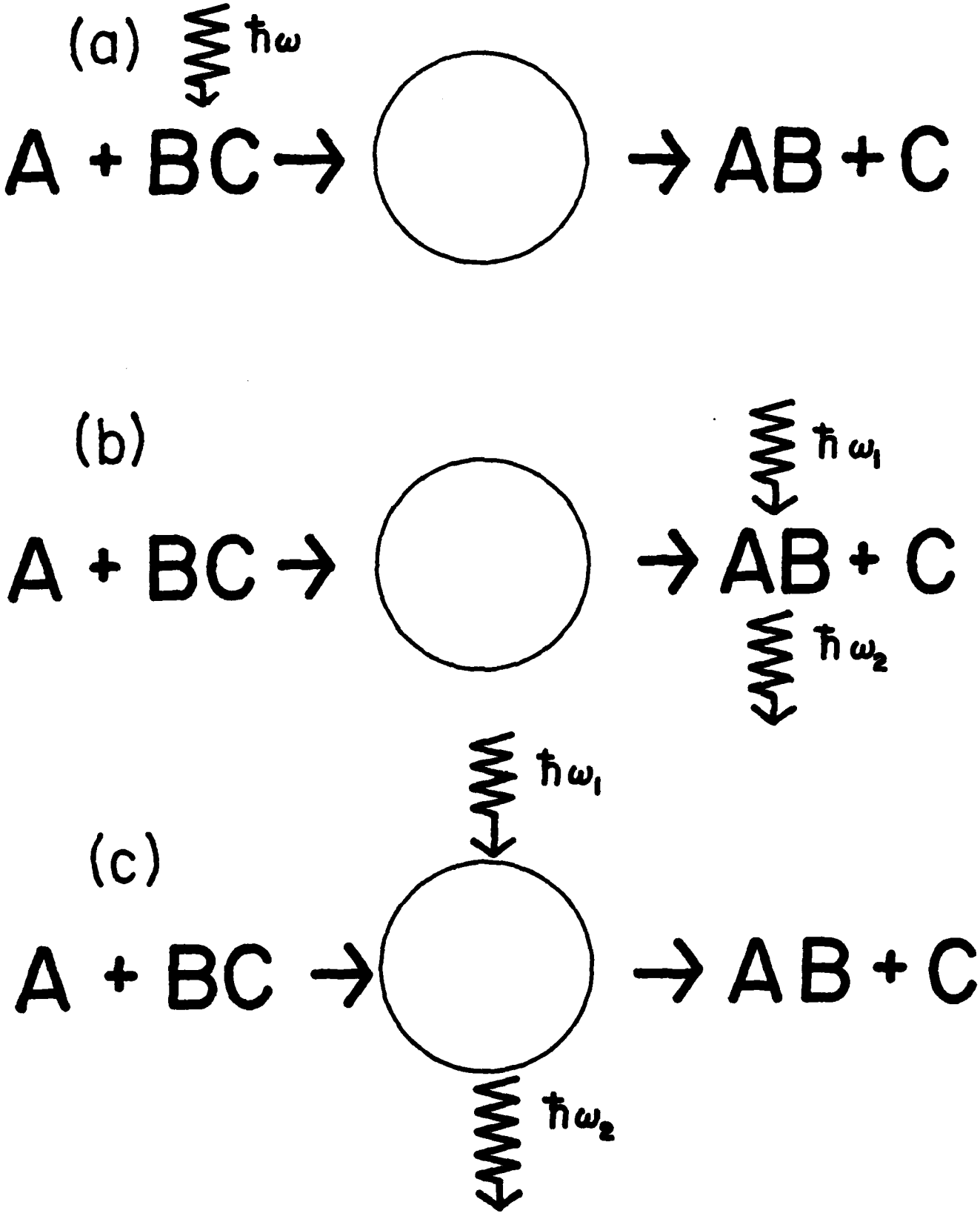
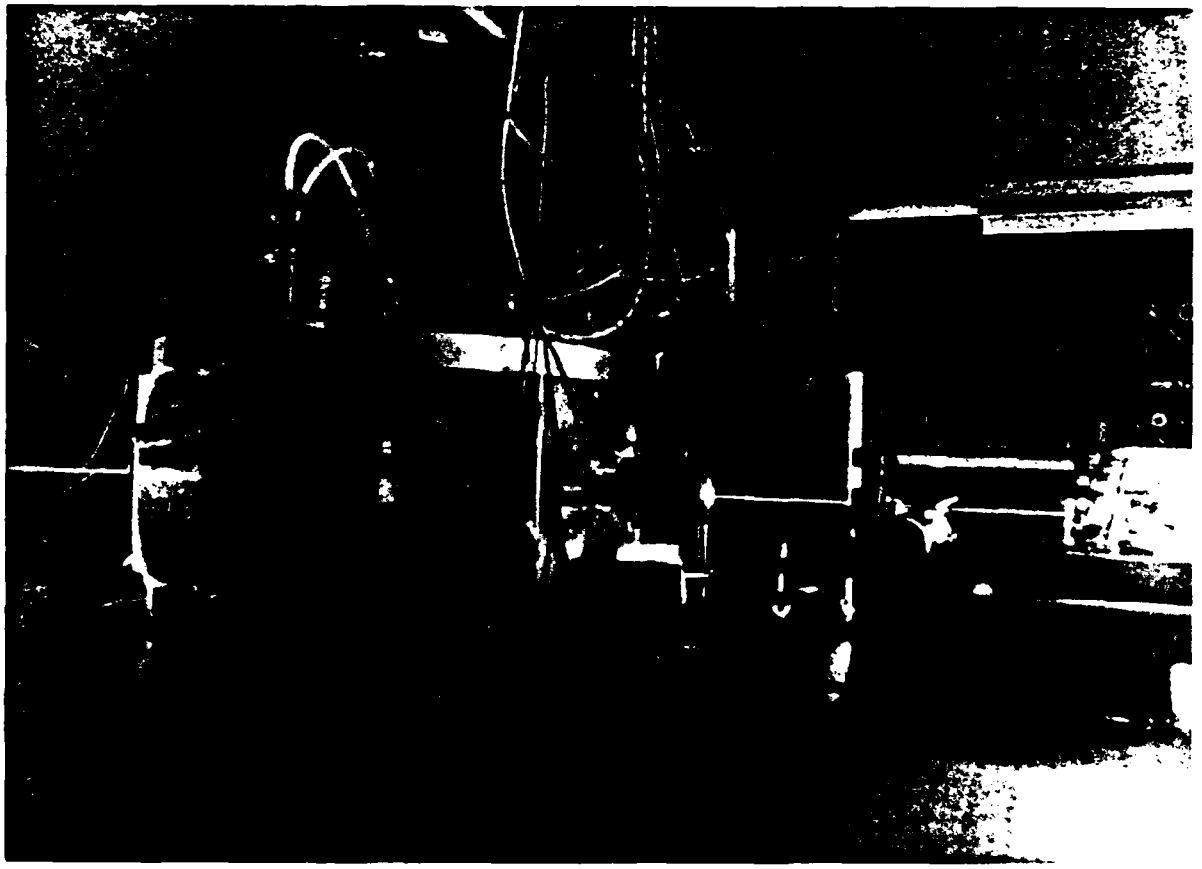
Figure Captions

1. Representative illustration of the application of lasers to studies of rearrangement reactions, where the circle denotes the interaction region. The first two cases (a) and (b) correspond to laser radiation ($\hbar\omega$ and $\hbar\omega_1$, where ω and ω_1 are frequencies) of moderate or weak intensity; (a) is laser selection of an initial energy level of the reactant BC, and (b) is the spectroscopic analysis of the product AB ($\hbar\omega_2$ corresponds to emission at a different frequency). For high enough intensity ($> 1\,kW/cm^2$), the radiation can interact directly with the reaction dynamics, which is the last case (c).

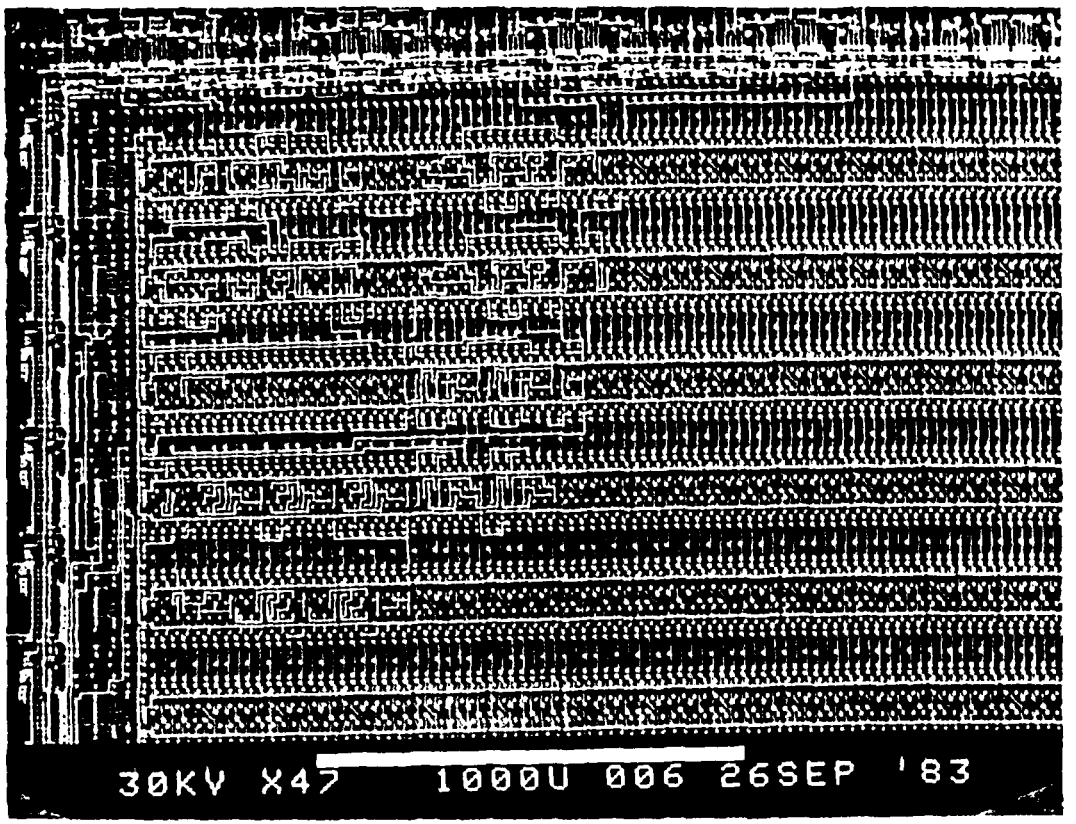
2. Laser-molecular-beam apparatus at Rice University.

3. Scanning electron micrograph of a 1-mm x 1.5-mm section of the CMOS array chip, with discretionary aluminum alloy interconnect metallization patterned to form test structures, at Lincoln Laboratory.


(a)
ħω
A + BC →
→ AB + C
(b)
ħω₁
A + BC →
→ AB + C
ħω₂
(c)
ħω₁
A + BC →
→ AB + C
ħω₂



OVERVIEW OF PORTION OF GATE ARRAY WITH DISCRETIONARY METAL



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