Time Resolved Electron Diffraction For Chemistry Biology And Material Science Volume 184 Advances In Imaging And Electron Physics

The objective of this program was to develop an instrument for characterization of nanosecond and picosecond time evolution of surface properties. This instrument was developed based on the technique of picosecond time-resolved reflection high-energy electron diffraction (RHEED). The basic idea of this technique is the utilization of 150 ps laser pulses to generate electron pulses by the photoelectric effect. The photogenerated electrons are accelerated, focused, and collimated using electron optics. Only a very small fraction of the laser pulse energy is needed to generate the fast (10-30 kV) well-collimated electron pulses; thus, most of the laser energy is available for sample irradiation. (eg).

This book features reviews by leading experts on the methods and applications of modern forms of microscopy. The recent awards of Nobel Prizes awarded for super-resolution optical microscopy and cryo-electron microscopy have demonstrated the rich scientific opportunities for research in novel microscopies. Earlier Nobel Prizes for electron microscopy (the instrument itself and applications to biology), scanning probe microscopy and holography are a reminder of the central role of microscopy in modern science, from the study of nanostructures in materials science, physics and chemistry to structural biology. Separate chapters are devoted to confocal, fluorescent and related novel optical microscopies, coherent diffractive imaging, scanning probe microscopy, transmission electron microscopy in all its modes from aberration corrected and analytical to in-situ and time-resolved, low energy electron microscopy, photoelectron microscopy, cryo-electron microscopy in biology, and also ion microscopy. In addition to serving as an essential reference for researchers and teachers in the fields such as materials science, condensed matter physics, solid-state chemistry, structural biology and the molecular sciences generally, the Springer Handbook of Microscopy is a unified, coherent and pedagogically attractive text for advanced students who need an authoritative yet accessible guide to the science and practice of microscopy.

The Schottky electron emitter is a predominant electron-emitting source in today’s electron beam equipment. This book comprehensively covers the Schottky emitter, dealing with its theoretical as well as practical aspects. The main questions that are addressed in this book are: what is the Schottky electron emitter? How does it work? And how do its properties affect the performance of electron beam equipment? The focus is on the direct link between the operating conditions of the source and the properties of the beam at the target level. This coupling is made clear by discussing the effect of the operating conditions and the geometry of the source and gun on the emission properties of the emitting surface, the effect of Coulomb interactions on the brightness and energy spread in the first few millimeters of the beam path, and the effect of the operating conditions and the shape of the emitter on the consequences of the beam at the target. The final chapter combines all these effects to demonstrate that there is a trade-off to be made between brightness, energy spread, and shape stability.

We report the experimental demonstration of femtosecond electron diffraction using high-brightness MeV electron beams. High-quality, single-shot electron diffraction patterns for both polycrystalline aluminum and single-crystal 1T-TaS2 are obtained utilizing a 5 fC (~3 × 104 electrons) pulse of electrons at 2.8 MeV. The high quality of the electron diffraction patterns confirms that electron beam has a normalized emittance of ~50 nm rad. The transverse and longitudinal coherence length is ~11 and ~2.5 nm, respectively. The timing jitter between the pump laser and probe electron beam was found to be ~100 fs (rms). The temporal resolution is demonstrated by observing the evolution of Bragg and superlattice peaks of 1T-TaS2 following an 800 nm optical pump and was found to be 130 fs. Lastly, our results demonstrate the advantages of MeV electrons, including large elastic differential scattering cross-section and access to high-order reflections, and the feasibility of ultimately realizing below 10 fs time-resolved electron diffraction.

The transient events of the {alpha}-{beta} martensitic transformation in nanocrystalline Ti films were explored via single shot electron diffraction patterns with 1.5 ns temporal resolution. The diffraction patterns were acquired with a newly constructed dynamic transmission electron microscope (DTEM), which combines nanosecond pulsed laser systems and pump-probe techniques with a conventional TEM. With the DTEM, the transient events of fundamental material processes, that are far too fast to be studied by conventional bulk techniques, can be captured in the form of electron diffraction patterns or images with nanosecond temporal resolution. The transient phenomena of the martensitic transformations in nanocrystalline Ti is ideally suited for study in the DTEM, with their rapid nucleation, characteristic interface velocities {approx}1 km/s, and significant irreversible microstructural changes. Free-standing 40-nm-thick Ti films were laser-heated at a rate of {approx}10{sup 10} K/s to a temperature above the 1155 K transition point, then probed at various time intervals with a 1.5-ns-long, intense electron pulse. Diffraction patterns show an almost complete transition to the {beta} phase within 500 ns. Postmortem analysis (after the sample is allowed to cool) shows a reversion to the {alpha} phase coupled with substantial grain growth, lath formation, and texture modification. The cooled material also shows a complete lack of apparent dislocations, suggesting the possible importance of a "massive" short-range diffusion transformation mechanism.

An rf photocathode electron gun is used as an electron source for ultrafast time-resolved pump-probe electron diffraction. We observed single-shot diffraction patterns from a 160 nm Al foil using the 5.4 MeV electron beam from the Gun Test Facility at the Stanford Linear Accelerator. Excellent agreement with simulations suggests that single-shot diffraction experiments with a time resolution approaching 100 fs are possible.

The thesis provides the necessary experimental and analytical tools to unambiguously observe the atomically resolved chemical reactions. A great challenge of modern science has been to directly observe atomic motions during structural transitions, and while this was first achieved through a major advance in electron source brightness, the information content was still limited and new methods for image reconstruction using femtosecond electron diffraction methods were needed. One particular challenge lay in reconciling the innumerable possible nuclear configurations with the observation of chemical reaction mechanisms that reproducibly give the same kind of chemistry for large classes of molecules. The author shows that there is a simple solution that occurs during barrier crossing in which the highly anharmonic potential at that point in nuclear rearrangements couples high- and low-frequency vibrational modes to give highly localized nuclear motions, reducing hundreds of potential degrees of freedom to just a few key modes. Specific examples are given in this thesis, including two photoinduced phase transitions in an organic system, a ring closure reaction, and two direct observations of nuclear reorganization driven by spin transitions. The emerging field of structural dynamics promises to change the way we think about the physics of chemistry and this thesis provides tools to make it happen.

This book presents an Ultrafast Low-Energy Electron Diffraction (ULEED) system that reveals ultrafast structural changes on the atomic scale. The achievable temporal resolution in the low-energy regime is improved by several orders of magnitude and has enabled the melting of a highly-sensitive, molecularly thin layer of a polymer crystal to be resolved for the first time. This new experimental approach permits time-resolved structural investigations of systems that were previously partially or totally inaccessible, including surfaces, interfaces and atomically thin films. It will be of fundamental importance for understanding the properties of nanomaterials so as to tailor their properties.

In this unique illustrated book, PhD students, postdoctoral researchers, senior visiting scholars, and staff describe their personal experiences in working with the late Prof. Ahmed H. Zewail at Caltech. Their reminiscences provide snapshots of their rich interactions, reflecting the great scientific achievements, as well as the human and humorous sides of Ahmed H. Zewail.The

contributors tell us their stories covering a period of forty years, beginning from the time of Zewail's arrival at Caltech in 1976. Some of them cover the time when Zewail was starting his pioneering work on femtochemistry at the end of 80's, while others relate events long after he was awarded the Nobel Prize in Chemistry (1999) and had embarked on a new career in ultrafast electron imaging. The aims and scope of this book is to provide both scientists and non-scientists descriptions of the experiences of scientists in the early or mature stages of their careers when interacting with one of the greatest scientists of the 20th century, from developing the field of femtochemistry to pioneering ultrafast electron diffraction and imaging technology. The personal dimension of Zewail's leadership is reflected in all the contributions, and highlighted by special tributes from two of his children.The scientific and anecdotal stories recounted in the book give a rare view of experiences in shaping science. The reader will get firsthand accounts of how a Nobel Prize winner interacted daily with his co-workers to develop the laser-based science and technology for which he was internationally recognized. The recounted experiences may serve as a basis for scientists developing their own research, tutoring students, and supervising postdoctoral researchers.

The decision of Springer-Verlag to publish this book in English came as a pleasant surprise. The fact is that I started writing the first version of the book back in 1978. I wished to attract attention to potentialities inherent in selected-area electron diffraction (SAED) which, for various reasons, were not being put to use. By that time, I had at my disposal certain structural data on natural and synthetic minerals obtained using SAED and high-resolution electron microscopy (HREM), and this stimulated my writing this book. There were several aspects concerning these data that I wished to emphasize. First, it was mostly new and understudied minerals that possess the peculiar structural features studied by SAED and HREM. This could interest mineralogists, crystallo chemists, and crystallographers. Second, the results obtained indi cated that, under certain conditions, SAED could be an effective, and sometimes the only possible, method for structure analysis of minerals. This inference was of primary importance, since fine dispersion and poor crystallinity of numerous natural and synthe tic minerals makes their structure study by conventional diffrac tion methods hardly possible. Third, it was demonstrated that in many cases X-ray powder diffraction analysis of dispersed miner als ought to be combined with SAED and local energy dispersion analysis. This was important, since researchers in structural min eralogy quite often ignored, and still ignore even the simplest in formation which is readily available from geometrical analysis of SAED patterns obtained from microcrystals.

Advances in Imaging & Electron Physics merges two long-running serials—Advances in Electronics & Electron Physics and Advances in Optical & Electron Microscopy. The series features extended articles on the physics of electron devices (especially semiconductor devices), particle optics at high and low energies, microlithography, image science and digital image processing, electromagnetic wave propagation, electron microscopy, and the computing methods used in all these domains. Contributions from leading authorities Informs and updates on all the latest developments in the field

It is the goal of this research to construct an instrument for Time-Resolved Electron Diffraction (TRED) studies of the structures of short-lived, energetic molecular states and of the dynamics of primary dissociation processes found in molecules of energetic materials. The TRED experiments will be performed by focusing a pulsed ArF excimer laser onto the cold photocathode of a high voltage electron gun, where electron pulses will be generated by field-assisted photoelectric emission. These electron pulses will be scattered off the gas-phase molecules in an effusive molecular beam after some fraction of the beam population has been excited by a pulse from a XeCl excimer-pumped dye laser. Electron diffraction intensities will be recorded using the real-time gas electron diffraction (GED) technology developed at the University of Arkansas. By synchronizing the dye laser excitation pulse and the diagnostic electron pulse, intensity data can be obtained from molecules which all have the same age relative to the time of excitation. Thus, time-resolved observations of excited molecular states and of transient reaction products should be possible.

The Dynamic Transmission Electron Microscope (DTEM) project is developing an in situ electron microscope with nanometer- and nanosecond-scale resolution for the study of rapid laser-driven processes in materials. We report on the results obtained in a year-long LDRD-supported effort to develop DTEM techniques and results for phase transitions in molecular crystals, reactive multilayer foils, and melting and resolidification of bismuth. We report the first in situ TEM observation of the HMX [beta]-[delta] phase transformation in sub-[mu]m crystals, computational results suggesting the importance of voids and free surfaces in the HMX transformation kinetics, and the first electron diffraction patterns of intermediate states in fast multilayer foil reactions. This project developed techniques which are applicable to many materials systems and will continue to be employed within the larger DTEM effort.

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The aim of this project was to record time-resolved electron diffraction patterns of aligned molecules and to reconstruct the 3D molecular structure. The molecules are aligned non-adiabatically using a femtosecond laser pulse. A femtosecond electron pulse then records a diffraction pattern while the molecules are aligned. The diffraction patterns are then be processed to obtain the molecular structure.

Imaging and spectroscopy are the most important and challenging techniques for not only research on materials science, chemistry, and biology, but also medical diagnoses. In this book, we have collected information on several novel imaging and spectroscopic techniques, including time-resolved electron diffraction/microscopy for materials science, various spectroscopes for physics and chemistry, and high-resolution computed tomography for medical science. We think that the content in each chapter is impressive and we hope this book will contribute to future instrument developments and new applications.

Advances in Electronics and Electron Physics

This is an avant-garde book edited by Nobel Laureate Ahmed Zewail with contributions from eminent scientists including four Nobel prize winners. The perspectives of these world leaders in physics, chemistry, and biology define potential new frontiers at the interface of disciplines and including physical, systems, and synthetic biology. This book brings about the confluence of concepts and tools, and that of different disciplines, to address significant problems of our time: visualization; theory and computation for complexity; macromolecular function, protein folding and misfolding; and systems integration from cells to consciousness. The scope of tools is wide-ranging, spanning imaging, crystallography, microfluidics, single-molecule spectroscopy, and synthetic probe targeting. Concepts such as dynamic self-assembly, molecular recognition, non-canonical amino acids, and others are covered in various chapters as they are cornerstones in building the trilogy description of behavior-structure, dynamics, and function. The volume is uniquely structured to provide overviews with historical perspectives on the evolution of ideas and on the future of physical biology and biological complexity, from atoms to medicine. Contents: The

Preoccupations of Twenty-First-Century Biology (D Baltimore)The World as Physics, Mathematics and Nothing Else (A Varshavsky)Physical Biology: 4D Visualization of Complexity (A H Zewail)Revolutionary Developments from Atomic to Extended Structural Imaging (J M Thomas)Physical Biology at the Crossroads (C Bustamante)The Challenge of Quasi-Regular Structures in Biology (R D Kornberg)The Future of Biological X-Ray Analysis (D C Rees)Reinterpreting the Genetic Code: Implications for Macromolecular Design, Evolution and Analysis (D A Tirrell)Designing Ligands to Bind Tightly to Proteins (G M Whitesides et al.)Biology by the Numbers (R Phillips)Eppur si muove (M Parrinello)Protein Folding and Beyond: Energy Landscapes and the Organization of Living Matter in Time and Space (P G Wolynes)Protein Folding and Misfolding: From Atoms to Organisms (C M Dobson)A Systems Approach to Medicine Will Transform Healthcare (L Hood)The Neurobiology of Consciousness (C Koch & F Mormann)Computer-Aided Drug Discovery: Physics-based Simulations from the Molecular to the Cellular Level (J A McCammom)Precision Measurements in Biology (S R Quake)Potassium Channels and the Atomic Basis of Selective Ion Conduction (R MacKinnon)Symmetry Breaking, Delocalization and Dynamics in Electron Transfer Systems (N S Hush)The Initial Value Representation of Semiclassical Theory: A Practical Way for Adding Quantum Effects to Classical Molecular Dynamics Simulations of Complex Molecular Systems (W H Miller) Readership: Graduate students and researchers in life sciences (structural biology, genomics, systems biology, molecular biology, neuroscience), biochemistry, physical chemistry, chemical engineering, and biophysics. Keywords:Visualization;Complexity;Macromolecular Function;Protein Folding;Molecular Recognition;Systems Integration;Cells;Consciousness;Crystallography;Microfluidics;Spectroscopy;Synthetic Probe TargetingReviews:“Even the shorter contributions, written by masters of their fields, are penetrating.”Chemistry World “The scope of this collection of overviews of the present state and future possible developments in physical biology is very broad. The result is both informative and readable. Anyone interested in how physics, engineering and mathematics can contribute to research in biology and medicine, be it on the molecular level or on the healthcare level, should be able to find useful information and inspiration in this book.”Acta Paediatrica

Structural phase transitions, mechanical deformations, and the embryonic stages of melting and crystallization are examples of phenomena that can now be imaged in unprecedented structural detail with high spatial resolution, and ten orders of magnitude as fast as hitherto. No monograph in existence attempts to cover the revolutionary dimensions that EM in its various modes of operation nowadays makes possible. The authors of this book chart these developments, and also compare the merits of coherent electron waves with those of synchrotron radiation. They judge it prudent to recall some important basic procedural and theoretical aspects of imaging and diffraction so that the reader may better comprehend the significance of the new vistas and applications now afoot. This book is not a vade mecum - numerous other texts are available for the practitioner for that purpose.

This book brings a broad review of recent global developments in theory, instrumentation, and practical applications of electron microscopy. It was created by 13 contributions from experts in different fields of electron microscopy and technology from over 20 research institutes worldwide.

This book focuses on the use of novel electron microscopy techniques to further our understanding of the physics behind electron–light interactions. It introduces and discusses the methodologies for advancing the field of electron microscopy towards a better control of electron dynamics with significantly improved temporal resolutions, and explores the burgeoning field of nanooptics – the physics of light–matter interaction at the nanoscale – whose practical applications transcend numerous fields such as energy conversion, control of chemical reactions, optically induced phase transitions, quantum cryptography, and data processing. In addition to describing analytical and numerical techniques for exploring the theoretical basis of electron–light interactions, the book showcases a number of relevant case studies, such as optical modes in gold tapers probed by electron beams and investigations of optical excitations in the topological insulator Bi2Se3. The experiments featured provide an impetus to develop more relevant theoretical models, benchmark current approximations, and even more characterization tools based on coherent electron–light interactions.

It is the goal of this research to perform gas electron diffraction (GED) studies, time-resolved and not time resolved, of the photochemical and thermal decomposition of RDX and trinitro-azetidine. (Proposal 7MAR88 to 15AUG90; no-cost extension granted to 15AUG91. The two compounds are highly explosive. Therefore, the GED experiments are not routine and feasibility of the project had to be demonstrated first by recording the ground state data of the two systems. Two specific problems were identified and discussed in the original proposal: Can effective vapor densities of RDX or TNAZ be achieved which are sufficient for GED studies? After flash heating or laser excitation, can important intermediates of decomposition be identified in the product mixtures?

The examination of structure at the microscopic scale, between micrometers and angstrom units, has changed dramatically in recent decades. Many new types of microscopy have emerged, notably the many scanning-probe designs, some of which also allow manipulation of atoms to form wanted structures, while others now permit direct observation of moving proteins in liquids. The traditional electron microscope is being revolutionized by the arrival of aberration correctors and monochromators, which bring the resolution below the Angstrom and electron-volt level. The 'laboratory in a microscope' conce.

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The acceleration of the technical capabilities of X-ray, electron and neutron diffraction techniques in recent years has been driven by developments in synchrotron and neutron sources, detectors and computers. These allow the rapid and repeated acquisition of diffraction data as a chemical and biological structural process proceeds, following its initiation. This opens up completely new studies from timescales ranging from the sub-picosecond, at the fastest, through all the time domains up to kiloseconds, for the slower molecular processes.

This report describes the progress that has been made over the last years toward the generation of ultrashort electron pulses, and their application to time resolved electron diffraction. We have generated electron pulses of picosecond duration using an ultrafast laser system, tested a number of photocathode materials, built an apparatus to measure electron pulse durations in the femtosecond domain, and designed and built an electron diffractometer for time resolved electron diffraction. We demonstrated that the diffractometer, which uses a novel one dimensional detection scheme, is well capable of determining atomic distances in molecules to better than on hundredth of an Angstrom. A sophisticated noise suppression system maintains a signal to noise ratio sufficient for pump probe experiments. Further developments include the design of a reflectron electron gun for femtosecond electron pulses, and a two dimensional electron diffraction detector. Finally, significant progress has been made in the interfacing of adaptive optics to a learning algorithm, and the generation of spectrally tunable ultrashort laser pulses at 200 nm.

A major scientific challenge is determining the 3-D atomic structure of small nanostructures, including single molecules. Coherent diffractive imaging (CDI) is a promising approach. Recent progress has demonstrated coherent diffraction patterns can be recorded from individual nanostructures and phased to reconstruct their structure. However, overcoming the dose limit imposed by radiation damage is a major obstacle toward the full potential of CDI. One approach is to use ultrafast x-ray or electron pulses. In electron diffraction, amplitudes recorded in a diffraction pattern are unperturbed by lens aberrations, defocus, and other microscope resolution-limiting factors. Sub-A signals are available beyond the information limit of direct imaging. Significant contrast improvement is obtained compared to high-resolution electron micrographs. progress has also been made in developing time-resolved electron diffraction and imaging for the study of ultrafast dynamic processes in materials. This talk will cover these crosscutting issues and the convergence of electron and x-ray diffraction techniques toward structure determination of single molecules.

Ever since the beginning of mankind's efforts to pursue scientific inquiry into the laws of nature, visualization of the very distant and the very

small has been paramount. The examples are numerous. A century ago, the atom appeared mysterious, a “raisin or plum pie of no structure,” until it was visualized on the appropriate length and time scales. Similarly, with telescopic observations, a central dogma of the cosmos was changed and complexity yielded to simplicity of the heliocentric structure and motion in our solar system. For matter, in over a century of developments, major advances have been made to explore the inner microscopic structures and dynamics. These advances have benefited many fields of endeavor, but visualization was incomplete; it was limited either to the 3D spatial structure or to the 1D temporal evolution. However, in systems with myriads of atoms, 4D spatiotemporal visualization is essential for dissecting their complexity. The biological world is rich with examples, and many molecular diseases cannot be fully understood without such direct visualization, as, for example, in the case of Alzheimer's and Parkinson's. The same is true for phenomena in materials science, chemistry, and nanoscience. This anthology is an account of the collected works that have emerged over the past decade from Caltech. Through recent publications, the volume provides overviews of the principles, the electron-based techniques, and the applications made. Thanks to advances in imaging principles and technology, it is now possible with 4D electron microscopy to reach ten orders of magnitude improvement in time resolution while simultaneously conserving the atomic spatial resolution in visualization. This is certainly a long way from Robert Hooke's microscopy, which was recorded in his 1665 masterpiece Micrographia.

The construction and utilization of the fourth-generation ultrafast electron diffraction apparatus, UED4, is the subject of this thesis. With UED4 and its novel and universal sample delivery method based on laser desorption, we were able to vaporize thermally labile molecular samples and determine their ground-state structures and the structures of their photochemical and photophysical reaction products. Each component part of the new UED4 apparatus is described, and the experimental and computational procedures used to extract structural information from the time-resolved diffraction patterns are presented. Several molecules were studied in their ground states and photoinduced excited states or product states on the time scale of picoseconds and nanoseconds. With UED3, nitrobenzene was shown to undergo intramolecular rearrangement prior to NO loss in an ultrafast fragmentation reaction. In indole, the chromophore of the amino acid tryptophan, the involvement of a dark structure, formed on the picosecond time scale, was revealed in the nonradiative decay pathway of the initially excited state. By determining the ground state structures of the thermally labile nucleobases uracil and guanine, the first use of surface-assisted laser desorption in a pulsed electron diffraction experiment was reported using the newly developed UED4 apparatus. The determined structures of the photochemically generated species of the photochromic molecule 6-nitro-BIPS further demonstrated the capability of laser desorption electron diffraction to function as a time-resolved experiment. Finally, the fragmentation reaction of the amino acid tryptophan upon UV laser irradiation was studied with UED4. The ability to deliver increasingly large and conformationally heterogeneous molecules into the gas phase now provides new challenges and opportunities of both experimental and theoretical nature for the field of ultrafast electron diffraction.

This thesis reports on developing a femtosecond electron diffraction technique and its application in following the structural changes associated with photoexcited Si. Time resolved electron diffraction is capable in principle of capturing transient structures of photoinduced phenomena with femtosecond time resolution. In practice, however, the time resolution is severely limited by the broadening of electron pulses due to space-charge forces. Another technical difficulty lies in that diffraction in transmission geometry requires samples that are semi-transparent to electrons. These experimental challenges were dealt with using innovative engineering and software development, and novel nanofabrication methods. Subsequently, a robust highly automated experimental setup was established, and 50 nm free-standing membranes of polycrystalline Si were nanofabricated. These membranes were first excited below damage threshold with 387 nm light, at an absorbed fluence of 5.6 mJ/cm2. The observed loss of intensity and associated dynamics under these conditions are related to the Debye-Waller effect as the lattice heats up through electron-phonon processes. At high excitation levels, greater than ?6% of the valence electron density, the crystalline structure of the lattice was lost in 500 fs. This observed time scale is indicative of an electronically driven order-to-disorder phase transition due to an abrupt change in the potential-energy landscape of the lattice. In addition, it was discovered that the high sensitivity of the electron diffraction process to shear-type deformation in single crystals makes the technique ideal to the generation and detection of coherent acoustic phonons. Longitudinal and shear acoustic phonons were subsequently resolved in (001)-oriented Si crystals. This study constitutes the first direct observation of shear phonons in symmetrically cut crystals, in sharp in contrast to the all-optical methods that rely on crystals asymmetrically cut at 20° to enhance the amplitude of the shear mode in order to detect its displacements.

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