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Dynamics and Interactions in Room Temperature Ionic Liquids, Surfaces and Interfaces

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14. ABSTRACT
The main experimental techniques used in the research include ultrafast two dimensional infrared (2D IR) vibrational echo experiments, IR polarization selective pump-probe (PP) and heterodyne detected transient grating (HDTG) experiments, and optical heterodyne detected optical Kerr effect (OHD-OKE) experiments. The first 2D IR experiments on functionalized SiO₂ planar surface monolayers of alkyl chains with a vibrational probe head group were performed. Room temperature ionic liquids were also investigated. Room temperature ionic liquids (RTIL) are intrinsically interesting because they simultaneously have properties that are similar to organic liquids and liquid salts. In addition, RTILs are increasingly being considered for and used in technological applications. Metal-organic frame works were also investigated. A unique aspect of metal-organic frameworks (MOFs) is their structural "flexibility" coexisting with a degree of regularity. We showed that two-dimensional infrared (2D IR) spectroscopy with pulse-shaping techniques can probe the ultrafast structural fluctuations of MOFs in spite of seeming insurmountable experimental problems and obtained the first measurements of framework dynamics.

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The following is a brief description of the research performed during the grant “Dynamics and Interactions in Room Temperature Ionic Liquids, Surfaces and Interfaces” (FA9550-12-1-0050). The experimental investigations employed a combination of ultrafast nonlinear optical experiments to directly investigate the relationships between dynamics and structure in complex materials. The main experimental techniques included ultrafast two dimensional infrared (2D IR) vibrational echo experiments, IR polarization selective pump-probe (PP) and heterodyne detected transient grating (HDTG) experiments, and optical heterodyne detected optical Kerr effect (OHD-OKE) experiments.

I. Functionalized Surface Monolayers

Studying surface functionalized monolayers with 2D IR spectroscopy required development of the methods to measure very weak signals that are produced by the small number of molecules in the samples. We achieved the goal of observing such systems. Here very brief descriptions of some of the results and theoretical developments are given.

The ultrafast dynamics of functionalized alkylsilane monolayers with two different alkyl chain lengths, C_{11} and C_3 , were studied by 2D IR vibrational echo spectroscopy. Terminal sites of the monolayers were functionalized with an IR probe, tricarbonyl (1,10-phenanthroline) rhenium chloride ($\text{RePhen}(\text{CO})_3\text{Cl}$), to report on the structural dynamics of the monolayers in air and in solvents. $\text{RePhen}(\text{CO})_3\text{Cl}$ and related compounds are photocatalysts that take CO_2 to CO and other catalyzed reactions. Frequency-frequency correlation functions (FFCF) of symmetric CO stretching mode were extracted from 2D IR spectra. The FFCF provides information on the time evolution of surface structures that contribute to the inhomogeneously broadened IR absorption spectrum of the CO stretch by quantifying spectral diffusion. To elucidate the detailed structural dynamics with accurate time constants, FFCF decays were monitored to 60 ps, a major improvement over prior experiments.^{1,3,4} Without the presence of solvents, C_3 monolayers have significantly slower spectral diffusion (66 ps) than C_{11} monolayer (38 ps). This difference demonstrates that spectral diffusion associated with the molecular monolayer in air involves the structural dynamics of the tethering alkyl chains. The $\text{RePhen}(\text{CO})_3\text{Cl}$ itself is too rigid to contribute to spectral diffusion. With the C_{11} and C_3 monolayers immersed in dimethylformamide (DMF), in which $\text{RePhen}(\text{CO})_3\text{Cl}$ is soluble, the FFCFs of both samples display biexponential decays with the time constants of 5.6 ps and 43 ps for C_{11} and 5.9 ps and 63 ps for C_3 . The slower time constants are in good agreement with the spectral diffusion time constants observed in the absence of solvent, indicating that these slower components still reflect the monolayers' dynamics, which are not greatly affected by the presence of the solvent. Because the faster time constants were independent of chain length and on the same time scale as found for the $\text{RePhen}(\text{CO})_3\text{Cl}$ head group in bulk DMF solution, they were attributed to the dynamics of the interfacial DMF molecules. The observed ~ 6 ps solvent dynamics yields information on the effect of the solvent-surface layer interface on solvent dynamics. When monolayers were immersed in hexadecane, in which $\text{RePhen}(\text{CO})_3\text{Cl}$ is not soluble, slower dynamics were again observed for the C_3 monolayer than the C_{11} monolayer.

Prior to the experiments just described, earlier experiments could not rule out the possibility that vibrational excitation transfer was involved in the observed 2D IR measured dynamics. Surface coverage experiments were conducted to determine if vibrational excitation transport influenced the measurements. The study showed the vibrational excitation transfer did not affect the results. A new theory of surface vibrational excitation transfer including the role of spectral diffusion was developed. Excitation transfer induced spectral diffusion is the time-dependent change in vibrational frequency induced by an excitation on an initially excited molecule jumping to other molecules that have different vibrational frequencies within the inhomogeneously broadened vibrational absorption line. The excitation transfer process was modeled as Förster resonant transfer, which depends on the overlap of the homogeneous spectra of the donating and accepting vibrational chromophores. Because the absorption line is inhomogeneously broadened, two molecules in close physical proximity can have overlaps of their homogeneous lines that range from substantial to very little. In the absence of structural dynamics, the overlap of the homogeneous lines of the donating and accepting vibrational chromophores would be fixed. However, dynamics of the medium that contains the vibrational chromophores, e.g., surface alkyl chain motions, produce spectral diffusion. Spectral diffusion causes the position of a molecule's homogeneous line within the inhomogeneous spectrum to change with time. Therefore, the overlap of donating and accepting molecules' homogeneous lines is time dependent, which must be taken into account in the excitation transfer theory. The method allowed the simultaneous treatment of both excitation transfer induced spectral diffusion and structural fluctuation induced spectral diffusion.

In a liquid or other isotropic medium, it is straightforward to measure orientational relaxation using techniques such as polarization selective pump-probe anisotropy experiments, time dependent fluorescence depolarization anisotropy measurements, or polarized transient grating experiments. However, the standard method

for measuring the time dependent anisotropy is based on the assumption that the sample's transition dipoles are randomly oriented. This is not the situation for molecules bound to a surface or at an interface, and the standard approach is not applicable. We developed a model-independent theory to extract orientational correlation functions unique to interfacial molecules and other uniaxial systems based on polarization-resolved resonant third-order spectroscopies, such as pump-probe spectroscopy, HDTG spectroscopy, and fluorescence depolarization experiment.⁶ It was shown that five measurements are necessary to completely characterize the monolayer's motions: $I_{\parallel}(t)$ and $I_{\perp}(t)$ with the incident beams in a plane that contains the normal to the surface, $I_{\parallel}(t)$ and $I_{\perp}(t)$ with a non-zero incident angle, and a time averaged linear dichroism measurement. If the sample plane is vertical and the incoming beams are in the horizontal plane for the first measurements of $I_{\parallel}(t)$ and $I_{\perp}(t)$, then the sample is rotated away from vertical, e.g. by 50°, and the second set of measurements is made. This is in contrast to an anisotropy measurement in a liquid where only one determination of $I_{\parallel}(t)$ and $I_{\perp}(t)$ is necessary. Once the five measurements are performed, two orientational correlation functions corresponding to in-plane and out-of-plane motions can be obtained. The procedure is applicable not only for monolayers on flat surfaces, but any sample with uniaxial symmetry such as uniaxial liquid crystals and aligned planar bilayers. The theory is valid regardless of the nature of the actual molecular motions on interface. The general results were then applied to the wobbling-in-a-cone model, in which molecular motions are restricted to a limited range of angles. Within the context of the model, the cone angle, the tilt of the cone relative to the surface normal, and the orientational diffusion constants can be determined.

II. Room Temperature Ionic Liquids and Other Liquids⁸⁻¹²

Room temperature ionic liquids (RTIL) are intrinsically interesting because they simultaneously have properties that are similar to organic liquids and liquid salts. In addition, RTILs are increasingly being considered for and used in technological applications. RTILs are usually composed of an organic cation and an inorganic anion. The organic cation, such as imidazolium, has alkyl chains of various lengths. The disorder in the liquid produced by the presence of the alkyl groups lowers the temperature for crystallization below room temperature and can also result in supercooling and glass formation rather than crystallization. The presence of the alkyl moieties also results in a segregation of the liquid into ionic and organic regions. We conducted Optical heterodyne detected optical Kerr effect (OHD-OKE) experiments that addressed the relationship between RTIL dynamics and structure. OHD-OKE experiments were used to examine the orientational relaxation dynamics of RTILs over time scales of a hundred femtoseconds to hundreds of nanoseconds, that is, the experiments spanned more than 6 decades of time and 6 decades in amplitude. For dry samples, the results showed that RTILs have bulk liquid orientational relaxation dynamics that are indistinguishable in their nature from common nonpolar organic liquids that supercool. This behavior of the RTILs occurs in spite of the segregation into ionic and organic regions. However, when increasing amounts of water were added to RTILs at room temperature, novel dynamics were observed for the RTILs with long alkyl chains that have not been observed in OHD-OKE experiments on organic liquids. The results were interpreted as water induced structure in the ionic regions that causes the long alkyl chains to organize and "lock up." The dynamical measurements indicated that this lock up is involved in the formation of RTIL gels that occur over a narrow range of water concentrations.

In addition to the RTIL experiments, OHD-OKE measurements and NMR pulsed field gradient spin echo experiments were used to measure the rotational and translational dynamics of polyether oligomers, and a collaborative theoretical study was performed to understand prior experimental results on the migration of phenol between hydrogen bonding sites on phenylacetylene.

III. Metal Organic Frameworks¹³

A unique aspect of metal-organic frameworks (MOFs) is their structural "flexibility" coexisting with a degree of regularity. Adsorbed guest molecules can cause MOF pore shapes to deform. Pore shape changes may be related to the MOFs' high capacity and selectivity for gas adsorption and other processes. MOF flexibility and other properties are influenced by fast dynamics of the framework. Direct measurements to characterize fast motions of the MOFs had not been performed previously. We showed that two-dimensional infrared (2D IR) spectroscopy with pulse-shaping techniques can probe the ultrafast structural fluctuations of MOFs in spite of seeming insurmountable experimental problems. 2D IR data, obtained from a vibrational probe attached to the linkers of UiO-66 MOF in low concentration, revealed that the structural fluctuations have time constants of 7 ps and 670 ps with no solvent. Filling the MOF pores with dimethylformamide (DMF) slowed the structural fluctuations by reducing the ability of the MOF to undergo deformations, and the dynamics of the DMF molecules were also greatly restricted.

Methodology advances were required to remove the severe light scattering caused by the macroscopic sized MOF particles, to eliminate interfering oscillatory components from the 2D IR data, and to address Förster vibrational excitation transfer.

IV. Methodology

Multidimensional visible spectroscopy using pulse shaping to produce pulses with stable controllable phases and delays has emerged as an elegant tool to acquire electronic spectra faster and with greatly reduced instrumental and data processing errors. Recent migration of this approach using acousto-optic modulator (AOM) pulse shaping to the mid-infrared region has proved useful for acquiring two dimensional infrared (2D IR) vibrational echo spectra. The measurement of spectral diffusion in 2D IR experiments hinges on obtaining accurate 2D line shapes. Prior to our work, pulse shaping 2D IR had not been used to study the time-dependent 2D IR line shapes (spectral diffusion) of a vibrational chromophore. We compared the spectral diffusion data obtained from a standard non-collinear 2D IR spectrometer using delay lines to the data obtained from an AOM pulse shaper based 2D IR spectrometer. The pulse shaping experiments were performed in stationary, partially rotating, and fully rotating reference frames and were the first in the infrared to produce 2D spectra collected in a fully rotating frame using a phase controlled 6 pulse sequence. Rotating frame experiments provide a dramatic reduction in the number of time points that must be measured to obtain a 2D IR spectrum, with the fully rotating frame giving the greatest reduction. Experiments were conducted on the transition metal carbonyl complex tricarbonylchloro(1,10-phenanthroline)rhenium(I) in chloroform. The time dependent data obtained from the different techniques and with different reference frames were shown to be in excellent agreement. This work set the stage for our subsequent use of plus shaping methods in the study of MOFs and other samples.

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Abstract

The research performed during the grant "Dynamics and Interactions in Room Temperature Ionic Liquids, Surfaces and Interfaces" (FA9550-12-1-0050) employed a combination of ultrafast nonlinear optical experiments to directly investigate the relationships between dynamics and structure in complex materials. The main experimental techniques included ultrafast two dimensional infrared (2D IR) vibrational echo experiments, IR polarization selective pump-probe (PP) and heterodyne detected transient grating (HDTG) experiments, and optical heterodyne detected optical Kerr effect (OHD-OKE) experiments. During the grant, we performed the first 2D IR experiments on functionalized SiO₂ planar surface monolayers of alkyl chains with a vibrational probe head group. Functionalized surfaces are important in a variety of chemical and physical applications. Studying surface functionalized monolayers with 2D IR spectroscopy required development of the methods to measure very weak signals that are produced by the small number of molecules in the samples. We achieved the goal of observing such

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systems. These experiments were the first 2D IR experiments performed on a monolayer of any kind. The experiments open up a new approach to the study of dynamics and structure of interfacial monolayer systems.

Room temperature ionic liquids were also investigated. Room temperature ionic liquids (RTIL) are intrinsically interesting because they simultaneously have properties that are similar to organic liquids and liquid salts. In addition, RTILs are increasingly being considered for and used in technological applications. RTILs are usually composed of an organic cation and an inorganic anion. The organic cation, such as imidazolium, has alkyl chains of various lengths. The disorder in the liquid produced by the presence of the alkyl groups lowers the temperature for crystallization below room temperature and can also result in supercooling and glass formation rather than crystallization. The presence of the alkyl moieties also results in a segregation of the liquid into ionic and organic regions. We conducted Optical heterodyne detected optical Kerr effect (OHD-OKE) experiments that addressed the relationship between RTIL dynamics and structure.

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2. "Excitation Transfer Induced Spectral Diffusion and the Influence of Structural Spectral Diffusion," Daniel E. Rosenfeld and Michael D. Fayer J. Chem. Phys. 137, 064109 (2012).
3. "Dynamics of Functionalized Surface Molecular Monolayers Studied with Ultrafast Infrared Spectroscopy," Daniel E. Rosenfeld, Jun Nishida, Chang Yan, Zsolt Gengeliczki, Brian J. Smith, and Michael D. Fayer J. Chem. Phys. C 116, 23428-23440 (2012).
4. "Structural Dynamics at Monolayer-Liquid Interfaces Probed by 2D IR Spectroscopy," Daniel E. Rosenfeld, Jun Nishida, Chang Yan, S. K. Karthick Kumar, Amr Tamimi, and Michael D. Fayer J. Phys. Chem. C 117, 1409-1420 (2013).
5. "Dynamics of Molecular Monolayers with Different Chain Lengths in Air and Solvents Probed by Ultrafast 2D IR Spectroscopy," Jun Nishida, Chang Yan, and Michael D. Fayer J. Phys. Chem. C 118, 523-532 (2014).
6. "Theory of Third-order Spectroscopic Methods to Extract Detailed Molecular Orientational Dynamics for Planar Surfaces and Other Uniaxial Systems," Jun Nishida and Michael D. Fayer J. Chem. Phys. 140, 144702 (2014).
7. "Theory of Interfacial Orientational Relaxation Spectroscopic Observables," Zsolt Gengeliczki, Daniel E. Rosenfeld, and M. D. Fayer J. Chem. Phys. 132, 244703 (2010)
8. "Orientational Dynamics of Room Temperature Ionic Liquid/Water Mixtures: Evidence for Water-Induced Structure and

Anisotropic Cation Solvation," Adam L. Sturlaugson, Kendall S. Fruchey, and M. D. Fayer J. Phys. Chem. B 116, 1777-1787 (2012).

9. "Orientational Dynamics in a Lyotropic Room Temperature Ionic Liquid," Adam L. Sturlaugson, Aaron A. Arima, Heather E. Bailey, and Michael D. Fayer J. Phys. Chem. B 117, 14775-14784 (2013).

10. "Dynamics and structure of room temperature ionic liquids," Michael D. Fayer Chem. Phys. Lett. 616-617, 259-274 (2014).

11. "Temperature and Hydration-Dependent Rotational and Translational Dynamics of a Polyether Oligomer," Adam L. Sturlaugson and M. D. Fayer J. Phys. Chem. B 115, 945-950 (2011).

12. "Theoretical Examination of Picosecond Phenol Migration Dynamics in Phenylacetylene Solution," Lucas Kocia, Steve M. Young, Yana A. Kholod, Mark S. Gordon, Michael D. Fayer, and Andrew M. Rappe Chem. Phys. 422, 175-183 (2013).

13. "Structural Dynamics inside a Functionalized Metal-Organic Framework Probed by Ultrafast 2D IR Spectroscopy," Jun Nishida, Honghan Fei, Amr Tamimi, Sonja Pullen, Sascha Ott, Seth M. Cohen, and Michael D. Fayer PNAS 111, 18442-18447 (2014).

14. "Comparisons of 2D IR Measured Spectral Diffusion in Rotating Frames Using Pulse Shaping and in the Stationary Frame Using the Standard Method," S. K. Karthick Kumar, A. Tamimi, and M. D. Fayer J. Chem. Phys. 137, 184201 (2012).

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