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TITLE EXPERIMENTAL MOLECULAR SPECTROSCOPY IN SHOCK-COMPRESSED MATERIALS

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SUBMITTED TO THE APS TOPICAL CONFERENCE ON SHOCK WAVES IN CONDENSED MATTER,
TO BE HELD IN MONTEREY, CA, ON JULY 20-23, 1987

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EXPERIMENTAL MOLECULAR SPECTROSCOPY IN SHOCK-COMPRESSED MATERIALS*

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Shocked materials present an exciting challenge to the experimental spectroscopist. The severe constraints in time and space imposed by the shock process can also be accompanied by chemical complexity. Many standard spectroscopic techniques have recently been adapted with varying degrees of success. Those discussed here are uv/visible absorption, emission, and fluorescence, infrared absorption, and spontaneous and coherent Raman scattering. The importance of the connection to both theory and static high-pressure methods is emphasized

1. INTRODUCTION

Advances in the understanding of shock/material interactions have paralleled the advances in the experimental techniques available to probe the sample. The measurement of bulk properties such as density, temperature, mechanical deformations, and hydrodynamic effects have provided useful characterizations of some materials, but there remain difficulties in the performance and interpretation of such experiments in molecular materials, especially energetic ones. The purpose of this paper is to review the various spectroscopic techniques that have been or could be used to measure molecular-level properties of shock-compressed molecular materials. In particular, we are interested in the use of such techniques to identify the molecular physical and chemical processes that occur upon shock compression and how these microscopic processes couple to hydrodynamics and energy transport. In this context several necessary capabilities of the experimental probes can be discerned. (1) they need to be able to distinguish between chemical species; (2) they should be able to provide structural information; (3) they should have enough temporal and spatial resolution to measure the desired kinetic behavior, and (4) they should operate in real time.

There are many experimental difficulties to overcome to adapt optical techniques to the study of shock-compressed materials. For many materials, strong shock waves are believed to be less than 1 μm in thickness and have velocities of the order of 5 km/s. Therefore, if we desire to make spectroscopic measurements in the shock wave, the technique selected needs spatial and temporal resolution of better than 0.2 μm and 10 ps, respectively. However, these requirements are considerably relaxed to study the region immediately behind the shock wave. Under shock-compression many materials are opaque or become opaque. Consequently, the use of these optical techniques may be limited to phenomenological studies in a few select materials. Another limitation is the refractive index change through the shock that results in bending of optical paths. Therefore, we may be forced to use optical arrangements that are not necessarily optimum for each technique, and may not be able to use some techniques at all. We also desire that the optical probe be non-intrusive. The measurements may be distorted by probe-induced effects such as photochemistry. Finally, in inhomogeneous samples, measurements will often be averaged over the inhomogeneities,

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* Work done under the auspices of the US Department of Energy

and consequently will not reflect the details of the microstructure. This effect may also occur in samples thought to be homogeneous.[1] It should now be obvious that no one optical technique has all of the necessary spectroscopic qualities or could overcome all of the above experimental difficulties. Therefore, the spectroscopic study of these systems will necessarily be multifaceted, using a combination of several techniques in appropriately chosen select materials.

2. TECHNIQUES

2.1. Ultraviolet/Visible Methods

<u>a. UV/visible Emission Spectroscopy</u> of condensed-phase, shock-compressed materials has been reported by many authors. These measurements have been used to determine temperatures[2-4] and to identify spectral features.[2-16] Other investigations of emission during shock-compression have been reviewed by Davison and Graham.[17] The calculation of temperature by using either brightness or multi-color methods usually requires additional knowledge or assumptions about the emissivity and optical depth of the radiating source. Failure to recognize this can result in an incomplete interpretation of the observations. For example, a 6000 K black body source viewed through a 4000 K black body will not be observed since the radiation is absorbed and re-emitted at the temperature of the intervening layer. This effect has been observed during the compression of ionic crystals.[2] Also, what do temperature measurements in compressed powders mean? Does the emission originate because of friction between microcrystals at the voids or compression of gas in the voids, or is it indicative of a bulk temperature within the microcrystals?

Some spectral features differing from gray body emission have been detected. Among these are triboluminescence in shock-compressed quartz,[2,5,8] molecular spectral features in shock-compressed RDX and PETN,[3,6,7] shock-compressed benzene,[9,10,14] excimer-laser-initiated lead styphnate and mercury fulminate,[11] detonating HNS, HMX, and PETN,[10,15] and shock-compressed granular HNS and PBX 9404.[13,16] The emitting species that have been identified include C_2, CN, CH, NO_2 (tentatively),[10] and many atoms such as Na. While in some cases the emission is admittedly from gas phase products and can be understood in terms of gas phase molecular parameters, in other cases the emission arises from within the condensed sample.[9,12,14] A pressing question is how the emission features shift and broaden with pressure and chemical composition. In addition, the origin of molecular emission in granular explosives may be ambiguous as discussed above. Other serious questions are what is the role of spalled material, and what high-temperature, gas-phase reactions influence the result?

<u>b. UV/visible Absorption</u> has been used to study index of refraction changes,[2,17] and the shifts of absorption bands with pressure.[18-2[illegible]] Many improvements to the uv/visible absorption technique have been implemented to overcome experimental ambiguities. The latest refinements, one with a flashlamp in a projectile, and one with a reflecting surface removed from shock effects, implemented by Duvall, et al. are depicted in Fig. 1. The measured absorption band shifts are used to infer changes in the molecular energy levels. However, these inferences are complicated, especially in larger molecules, by the complexity of the pressure induced shifts in the molecular states. Recently, dilution studies,[illegible] and comparisons to static high pressure absorption experiments[illegible] have been used to tackle some of these ambiguities. In addition, some progress has been made in modeling the density dependence of electronic absorption bands.[illegible]

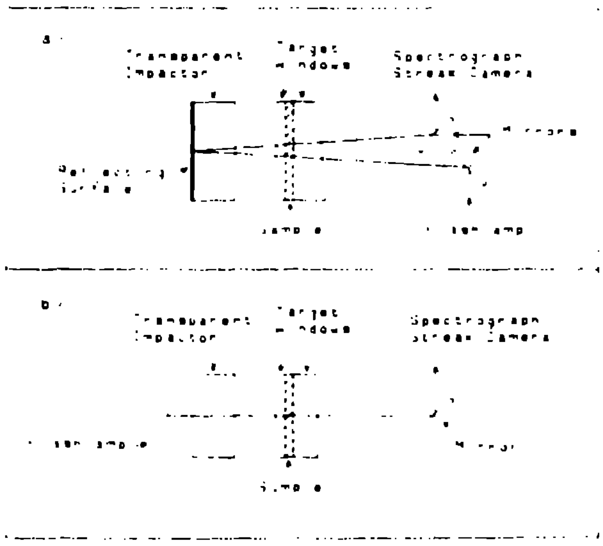

FIGURE 1
UV/visible absorption apparatus with a) reflector or b) flashlamp in the projectile.[23]

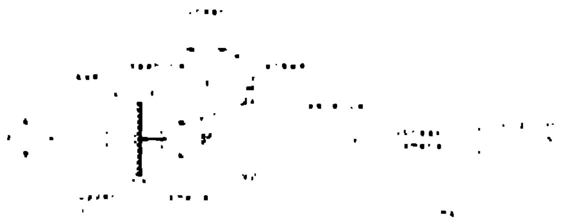

FIGURE 2
Fluorescence spectroscopy apparatus with laser shock production.[32]

c. Fluorescence is frequently used as a diagnostic for state-to-state chemical dynamics studies, measurements of intramolecular relaxation processes, and determinations of collisionally driven intermolecular relaxation rates. Current techniques use lasers as sources to selectively populate a small subset of excited states. Subsequent emission from the manifold of states populated by collisional or unimolecular processes is then monitored by a variety of fast detection methods. The character of the observed emission is determined by the energy transfer rates, the radiative lifetimes of the populated states, and the time resolution of the detection system. Recently this method (see Fig. 2) has been used to measure the viscosity and temperature of shock-loaded molecular systems,[30-32] to study the shock behavior of a simple diffusion-controlled chemical reaction,[33] and to measure wavelength shifts of R line emission in shock compressed ruby.[34,35] The problem of collisional quenching of excited states can be avoided by use of molecules well suited to such environments, such as organic dyes, and, in the region of lower shock pressures, spectral broadening can be small.[30-33] However, at higher pressures, even if pressure- and temperature-induced broadening is not a problem in specific cases, the dye molecules themselves may decompose. The key to the ruby experiments[34,35] is that the absorption is broad, but the excited Cr^{3+} ion quickly relaxes into a state whose fluorescence remains narrow to high pressures and temperatures. Other transition metal ions (or complexes) may have the same attributes.

An alternative to normal fluorescence is the use of double resonance techniques.[36] An initial pulse could be used to excite a desired electronic or vibrational transition. A second laser pulse, temporally delayed with respect to the first, could then excite the molecule to a higher energy level. The emission of the higher levels would then be monitored versus the delay time between pulses to determine the relaxation time of the intermediate level.

2.2 Infrared Methods

a. Infrared Absorption techniques were complicated until recently by the lack of fast detectors and/or fast pulsed sources. Now, however, quite fast (nanosecond) detectors are available. To be used effectively, however, a very high brightness source is required to

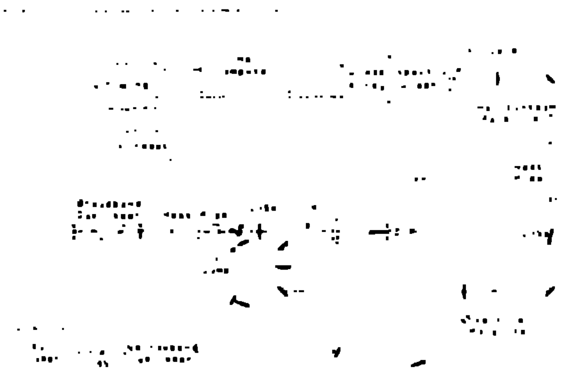

FIGURE 3
Time-resolved infrared spectral photography (TRISP) apparatus for shocked materials.[38]

overcome the brightness of the shock-compressed sample. In addition, for detection of molecular species, what is needed is an IR detector array, similar to the intensified photodiode arrays or vidicons available for work in the visible. These, unfortunately, are not yet available. A usable alternative is the time-resolved infrared spectral photography (TRISP) system devised by Sorokin and coworkers.[37] This system circumvents both of the above problems by employing stimulated electronic Raman scattering in alkali metal vapors to down-convert a broadband visible laser pulse into the infrared. This broadband infrared pulse is passed through the sample, recording the absorptions present there, and is then up-converted back to the visible, retaining the recorded absorptions, using a four-wave mixing process also in alkali metal vapors. The up-converted broadband visible pulse is then detected using a vidicon or intensified diode array.

TRISP has recently been used in shock-compressed samples and as a diagnostic to look for species in the gas-phase plume directly above detonating small pellets of high explosives.[18] The experimental apparatus for TRISP measurements in shock-compressed samples is shown in Fig. 3. A difficulty with the use of this technique as a real-time optical diagnostic in shock-compressed samples is the timing constraint placed on the method by necessarily very thin samples (timing jitter must be much less than 1 ns for use of the 1 ns IR pulse to measure absorption in a 5-μm-thick sample). An alternative is to use dilution methods to avoid saturation of the absorption, but, as in the case of uv/visible absorption,[25] dilution may eliminate the concerted-type chemical reactions that we wish to observe.

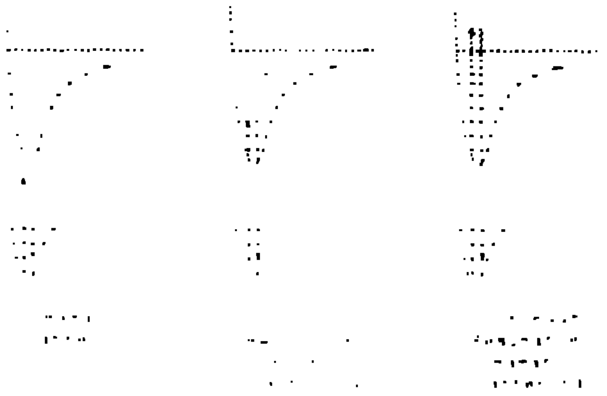

FIGURE 4
Pictoral representations of Raman and resonance Raman processes.

2.3. Raman Methods

a. Spontaneous Raman Spectroscopy in shocked materials was first performed in detonating crystalline RDX.[6] Subsequently, the technique has been used to measure the temperature of detonating[39] and shock-compressed[40] nitromethane and to observe shock modification of vibrational bands in detonating PETN,[41-44] HMX,[43] and RDX,[41,42] shock-compressed water,[45] and shock-compressed TATB.[46]

Raman spectroscopy is made possible by the inelastic scattering of a photon by molecules in the probed sample (see Fig. 4). The difference in frequency between the incident and scattered photons is equal to the frequency of a Raman active molecular transition. Raman scattering cross-sections are many orders of magnitude smaller than those for emission and absorption; hence, difficulties could be encountered in materials shock-compressed to very high temperatures. The cross-sections can be increased by using UV pump lasers rather than ones in the visible (the cross-section increases as ν^4); however, care must be taken to avoid interferences from fluorescing or photochemically produced species.

b. Resonance Raman. When the exciting laser frequency is in resonance with an actual molecular transition, the scattering cross-section for that species can dramatically increase.[47] In an isolated molecule, the resonance Raman effect can be due to (1) interaction of the excitation with a discrete transition in the molecule, or (2) interaction of the excitation with a dissociative continuum in the molecule (see Fig. 4).

As the excitation energy approaches a molecular resonance, the distinction between Raman scattering and absorption and reemission becomes blurred. What is important to the distinction between these two processes is the degree of loss of phase information in the experiment. In the condensed phase, especially when shocked to high pressures and temperatures, it is likely that most molecules will undergo phase interruption due to collisions within their normal excited state lifetime implying that discrete resonance Raman would be like fluorescence and can be said to have the same advantages and disadvantages.

When dissociative transitions are probed, the gain in cross-section due to the resonance condition may be more than offset by the small fraction of excited molecules that re-emit before dissociating.[47] Although, in gas-phase studies, resonance Raman techniques and analogs (dissociative emission) have recently undergone dramatic growth because they provide unique information about the dissociative potential energy surface,[48,49] in the study of shock compressed, condensed-phase systems their use will likely be problematic.

Large increases in Raman scattering cross-sections can also be produced when the exciting laser is just near resonance with an electronic transition in the molecule. This near-resonance enhancement in the Raman cross-section may be much larger than the normal Raman ν^4 frequency dependence, and may enable detection of some species that have the correct spectroscopic attributes.

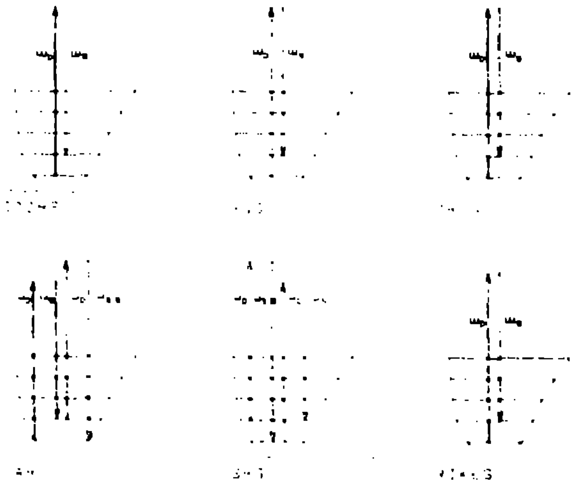

FIGURE 5
Pictoral representations of several coherent Raman processes. Heavy arrows denote coherent beams.

c. Coherent Raman Spectroscopy. Several coherent Raman techniques have been developed in recent years (Fig. 5). These techniques use large optical frequency electric fields to non-linearly drive the inelastic scattering process, leading to large scattered signals that are beam-like in nature. The consequent demands on the signal collection and detection systems are therefore significantly reduced from those required by spontaneous Raman methods.

Stimulated Raman was the first coherent Raman technique to be used in a shock compressed sample.[50] Stimulated Raman scattering[51] occurs when the incident laser intensity in a Raman-active material exceeds a threshold level and generates a strong, directional (forwards and backwards), beam at the Stokes frequency. The backwards directed beam was conveniently used in shock experiments to minimize the experimental complexity.[50] This technique does have some disadvantages: (1) the incident laser powers are large; (2) not all molecules have the required large Raman cross-section and narrow Raman linewidth; (3) only the lowest threshold transition in the condensed sample produces a signal; and (4) spatial resolution is limited to the confocal parameter of the focusing lens.

Inverse Raman or stimulated Raman loss spectroscopy (SRLS), and its analog stimulated Raman gain spectroscopy (SRGS),[52] have been suggested[53,54] as diagnostic techniques for shock-compressed materials. Signals can be obtained at power levels considerably below those required for stimulated Raman scattering because the process is driven by the presence of both the incident and Stokes laser fields (see Fig. 5). In the process, some fraction of pump photons is converted into Stokes photons. The difference in the two techniques lies in which beam is detected. In the case of SRLS, the loss of photons at the pump frequency is detected. In SRGS, the gain of photons at the Stokes frequency is detected. The lack of accurate information on vibrational frequencies at elevated pressures and temperatures, and the desire to detect more than one species, dictates the use of a broadband laser for one of the two incident pulses. The limiting detectivity is determined by the spectral noise in the broadband laser unless signal averaging or double beam techniques are used.

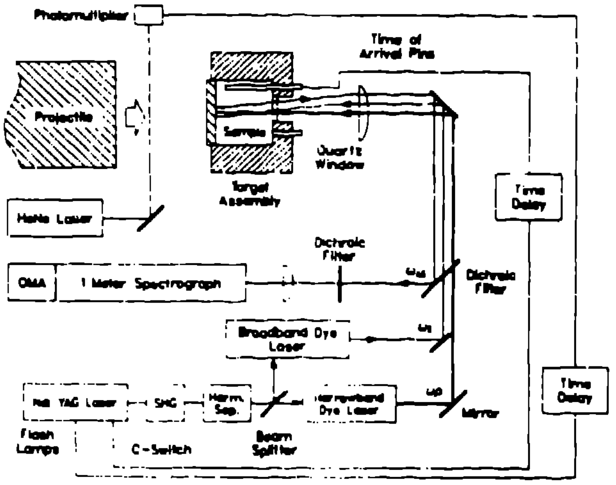

FIGURE 6
Coherent anti-Stokes Raman (CARS) apparatus for shocked materials.[55]

Coherent anti-Stokes and coherent Stokes Raman scattering (CARS and CSRS, respectively)[52] (see Fig. 5) occur as four-wave parametric processes in which three waves, two at a pump frequency and one at either the Stokes (CARS) or anti-Stokes (CSRS) frequency are mixed in a sample to produce a coherent beam at the anti-Stokes (CARS) or Stokes (CSRS) frequency, respectively. This mixing is greatly enhanced if the frequency difference between the pump and the Stokes (or anti-Stokes) lasers coincides with a Raman active mode of the sample. Like SRGS and SRLS, CARS and CSRS can be produced at incident power levels considerably below those required for stimulated Raman scattering. However, in CARS and CSRS, phase-matching is required, and possible geometric arrangements of the beams are limited. Figure 6 is a diagram of an experiment used to measure CARS spectra in shock-compressed liquids.[55-58] The noise in the broadband, Stokes, dye laser spectral profile also causes problems with the CARS measurements, but does not prevent detection of the spectrum, as it might in SRGS or SRLS techniques. To interpret the data, the spectral profile of the broadband dye-laser pulse is recorded at the same time as the CARS spectrum using another spectrometer/photodiode array.[57] Measurements that have been made to date using the CARS technique include the pressure and temperature shifts of the ring-stretching mode of benzene to 14.5 GPa,[55,59] the CN stretching mode of nitromethane to 9.0 GPa,[56,59] the fluid N_2 vibration fundamental and hot-bands to 19 GPa single shock and 41 GPa double shock,[57,58] and the fluid O_2 vibration to 9 GPa.[58] These results have shown that CARS measurements can provide very accurate molecular level information at pressures and temperatures of interest to both the shock-wave and equation-of-state communities. For example, the data have been used to test theoretical estimates of vibrational frequency shifts and bond lengths in these dense molecular fluids.[60] Through such theoretical modeling, it should be possible to characterize molecular potential functions at very high densities and temperatures.

In CARS experiments, the temporal resolution is determined by the laser pulse duration. However, since the beam crossing angle is determined (at from 1-3 degrees) by phase-matching requirements, spatial resolution is quite poor, being of the order of the confocal parameter of the focusing optics. One advantage of CARS and CSRS experiments is the large signals produced, allowing discrimination against background fluorescence and emission. However, CARS and CSRS produce signals even when there is no vibrational resonance, which is a problem when attempting to detect small concentrations of species. The resonant and non-resonant parts interfere to produce asymmetric line shapes, and ultimately, the resonant contribution will be lost in the noise (at the ppt level). However, the asymmetric lineshape has so far been used to advantage when fitting spectra to determine peak positions and linewidths.[59] Still unexplored is the possibility of using nearby electronic transitions to resonantly or near-resonantly enhance the CARS intensities (as was discussed above for spontaneous Raman).[61] However, considerable theoretical effort into the effects of broadening electronic and vibrational transitions on CARS lineshapes and intensities needs to be undertaken before resonantly enhanced CARS can become a truly useful experimental tool.

Raman-induced, Kerr-effect spectroscopy (RIKES)[52] has been suggested[53] as a diagnostic technique for performing measurements in shock-compressed systems. RIKES uses a circularly polarized pump beam and a highly linearly polarized broadband Stokes beam, and requires no phase-matching. At conditions of Raman resonance, the interaction of the two beams results in slight elliptical polarization of the Stokes beam at the Raman resonant frequencies. This elliptical component then passes through a crossed polarizer, and is detected by a spectrometer/intensified diode array. This arrangement ideally would give large signals against a small background, making it a good choice for the detection of species with small concentrations. In shock applications, however, difficulties due to birefringence in the strained shock-compressed sample, or polarization rotation on reflection from a shocked mirror could result in detection of many incident probe frequencies irrespective of a Raman resonance, and would give a large unwanted background.[62]

3. SUMMARY

We believe that the use of optical probes in shock-compressed materials will continue to spread and increase. The techniques most revealing of molecular properties, such as Raman scattering, will give specific information regarding shock-induced structure changes, chemical reactions, etc. This information, when used in conjunction with the very important recent[60,63,64] and future theoretical efforts, as well as established bulk property experiments, will provide insight into the detailed microscopic processes through and immediately behind the shock front. Complementary high pressure/high temperature static measurements will be used to test diagnostic tools, determine structure and energy transfer rates at shock-compression conditions, and, hopefully, help to distinguish between shock and static environments.

ACKNOWLEDGEMENT

The authors are indebted to most of the authors listed in the references for many useful discussions. We are particularly grateful to J. W. Shaner for his ongoing support of our research efforts.

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