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Ultrafast Laser Diagnostics to Investigate Initiation Fundamentals in Energetic Materials

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Abstract

We present the results of a two year early career LDRD project, which has focused on the development of ultrafast diagnostics to measure temperature, pressure and chemical change during the shock initiation of energetic materials. We compare two single-shot versions of femtosecond rotational CARS to measure nitrogen temperature: chirped-probe-pulse and ps/fs hybrid CARS thermometry. The applicability of measurements to the combustion of energetic materials will be discussed. We have also demonstrated laser shock and particle velocity measurements in thin film explosives using stretched femtosecond laser pulses. We will discuss preliminary results from Al and PETN thin films. Agreement between our results and previous work will be discussed.

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NOMENCLATURE

CARS	coherent anti-Stokes Raman Scattering
DOE	Department of Energy
SNL	Sandia National Laboratories
fs	femtosecond
ps	picosecond

1. INTRODUCTION

Little is known about the chemistry of shock initiation in explosives leading up to detonation. Rapid increases in pressure and temperature drive violent and fast reaction of species behind a shockwave on sub-ns timescales not accessible using standard analytical techniques like mass spectroscopy or spontaneous Raman. The reaction of energetic materials has been measured at lower temperatures and under constant pressure in an effort to characterize possible reaction pathways. Unfortunately, this approach has failed to produce predictive models for the performance of explosive components. While a given set of reaction pathways are assumed, reaction rates must be adjusted to reproduce the results of component testing. It is thought that the inability of models to reflect the complex interplay between chemical and mechanical change behind the shockwave is a major factor in their failure to predict performance. Shock waves induce changes on picosecond (ps) timescales (rapid jumps in temperature, pressure, strain, void collapse) impossible to mimic when reactions are observed at constant high pressure or reduced temperature. Rapid compression and heating may mix electronic states or place large amounts of energy into reactive coordinates during void collapse or shock induced strain at material defects. Shock compression likely opens reaction pathways not accessible in static measurements of reactivity. The resulting uncertainties in reaction rate and the role of microstructure in reactivity lead to corresponding uncertainties in reaction temperature also critical understanding the build-up to detonation. Therefore it is impossible to build reliable models for component performance without direct measurement of chemical kinetics and temperature in shocked energetic materials 10s of picoseconds after a shock-front has passed.

Measurements within shocked material near the shock front are extremely challenging due to the rapid timescale of reaction, spatial heterogeneity, and the high pressures and temperatures present within the measurement volume. Experiments require timing jitter to shock-front arrival on ps timescales. Recent work has addressed this problem by using a stretched pulse from the femtosecond laser to generate a shock in aluminum. Explosive powders also scatter probe light making optical interrogation of shocked material within pressed powder impossible in the visible and near IR. Therefore, recent work has focused on liquid explosives or thin film explosives in which shock material is optically accessible adjacent to a metal thin film to launch a laser generated shock into the material. Ultrafast laser spectroscopy measures chemical change in the explosive while shock properties are measured using an ultrafast analog of VISAR to measure shock and particle velocity (Ultrafast Shock Interrogation (USI)). We have adopted this approach and will discuss preliminary USI measurements on aluminum thin films and PETN/Al films. Finally, many time-resolved experiments require measuring signal at a series of delays between pulses. Shot-to-noise from material heterogeneities can significantly reduce signal to noise and complicate interpretation of data if the distribution of spatial features changes within the measurement volume from shot to shot. Therefore, it is advantageous to measure the property of interest in a single shot if the signal-to-noise is sufficient. We have demonstrated two, single-shot, coherent anti-Stokes Raman scattering (CARS) measurements in room temperature gas. Results will be used to propose similar experiments in thin film explosives to follow chemical change near the shock front.

2. LASER SHOCK /ULTRAFAST SHOCK INTEROGATION IN THIN FILM EXPLOSIVES

The direct measurement of physical and chemical change within shock materials using ultrafast spectroscopy is extremely challenging. Shocked material in liquid and thin film explosives is optically accessible. However, any approach must reliably time shock generation to spectroscopic interrogation with ps time-resolution while fully characterizing the shock that initiated the observed chemistry. Traditional methods for shock generation (flier, e-gun) have timing jitter >1 ns. If the same laser used for interrogation is also used to generate shock in the material, the problem of jitter is eliminated. A short pulse of laser light incident on an Al thin film will generate a shock that will travel into an adjacent film or liquid. However, the femtosecond lasers sources required for non-linear time-resolved spectroscopy have pulse durations < 100 fs and generate shocks lasting only a few ps; too short to initiate shock induced chemistry (10-100's of ps after initial compression). McGrane et al.¹(LANL) addressed this problem by chirping the pulse (delaying frequency component linearly with respect to one another in a grating stretcher) to lengthen it in time and then clipping the leading edge of the pulse (while spectrally dispersed) to generate shocks with a ~ 3 -5 ps rise and 300 ps duration in Al thin films. Bolme and McGrane (LANL) have also demonstrated a single shot measurement of shock and particle velocity using the same stretched pulse.² We have implemented their laser shock method and a variation of the particle velocity measurement based on work by Armstrong et al (LLNL).³ Together, these approaches provide a platform for spectroscopic interrogation behind the shockwave.

2.1. LASER induced shock apparatus.

The LASER shock apparatus is shown in Figure 1. Our Ti: Sapphire amplifier (KMLabs Wyvern System -W100701) typically generates 4 mJ, 42-fs duration pulses at 1-kHz repetition rate. The amplifier output spectrum was centered near 790 nm, with a bandwidth of 25 nm FWHM. We placed a waveplate and cube polarizer within the system to remove light before compression. Stretched pulses with positive dispersion (longer wavelength components arrive first) are sent to a pulse shaper that adds negative dispersion until the pulse has a duration > 300 ps and shorter wavelengths arrive first. The system is a larger version of the grating compressor found within the Wyvern (Figure 2). Negative group velocity dispersion (GVD) is added by increasing the separation between the horizontal retro and the first bounce off the grating. A fast rise is added to the pulse by clipping the dispersed at the vertical retro. A cross correlation of the stretched pulse with a fully compressed pulse is shown in Figure 3. Because there is no focal plane within a grating compressor the pulse rise as a function of wavelength is

less rapid than reported from other groups using a stretcher design. When focused into an Al film with a spot size 30 μm and an energy of 1.2 mJ. The shock rise time is 20 ps with sustained intensity for 400 ps.

Figure 1. Schematic of pulse shaper. Pulse is removed from Wyvern before compressor (~120 ps FWHM, red leading) sent to 1200 g/mm grating, horizontal retro (chirp added), vertical retro (beam clipped within retro to add 20 ps rise to blue edge) and returned below input pulse to pick off mirror (blue leading, ~ 300ps FWHM).

2.2. Ultrafast Shock Interrogation (USI)

Our apparatus is based on a previous design reported by Armstrong et al.³ It is a frequency domain analog to VISAR for the measurement of particle velocity and shock velocity. The stretched pulse can be treated a collection of narrow band sources spanning the pulse bandwidth. The chirp added in the pulse shaper delays the sources linearly in time with respect to radial frequency. This pulse (taken from the laser shock pulse) is divided into a pulse pair in a Michelson interferometer with a 10 ps delay (Figure 3). If the pulses are combined within a spectrometer in the absence of shock, the only phase difference observed in the spectral interferogram (SI) at any given frequency will reflect the delay between them since they travel an identical path to the detection camera. However, if shock arrives at the probe surface (Al film) in the time window of the probe pair, motion of the ablator will introduce an additional phase shift between each frequency pair. The SI is then filtered in the Fourier domain and referenced to interferograms in the absence of shock. The recovered phase shift reflects motion of the ablator at the thin film/ Al interface. The derivative of the phase therefore provides a direct measure of particle velocity (u_p) of the ablator and shock velocity (D) if the ablator is confined by a liquid or thin film. (add full explanation of USI with equations)

A PBS polarizer sends the S polarized pulse pair through a $\lambda/4$ waveplate and imaging lens to the film surface. The P polarized return passes through the PBS cube to the spectrometer. The imaging lens is optimized to image the spectrometer at the spectrometer slit with ~ 100x magnification. When the probe overlaps the shock arrival, the shock creates a measurable phase shift between the frequency components of the pulse pair. When the phase shift is referenced to unshocked material and reference spectra taken pre-shock, and then divided by the time delay between the pulse pair, the result is proportional to the surface velocity of the Aluminum ablator

for that time step. Integration of the accumulated phase over consecutive time steps can then be used to calculate the surface displacement as a function of time.

Figure 2. Schematic of USI experiment. Chirped pulse split into pulse pair in Michelson interferometer and sent into shocked sample with a 10ps delay. Linear polarization rotated by 90° by $\lambda/4$ waveplate while traveling to and from sample sending return pulse to spectrometer. Spectral interferogram of pulse pair recorded on spectrometer with μm spatial resolution in one dimension. Chirp on probe pulses encodes time onto spectral access (~ 11 ps time resolution).

Figure 3. Schematic of USI/laser shock experiment with x-frog.

The phase of the intensity profile is recovered as described in Kim et al.⁴ The phase shift difference introduced by shock injection is recovered from shocked and unshocked regions of the sample as described by Armstrong et al.³

2.3. Results

We have successfully observed the shock rise in an Al thin film under laser induced shock. Raw interferograms and are shown below (Fig.4). Surface velocity within the shock region and surface displacement were then calculated as described above.

Figure 4. Interferogram from shocked Al film (left) and the resulting phase map of image referenced to unshocked regions and reference shots (right).

From this data surface velocity was recovered as a function of time for various locations within the image (Figure 5).

Figure 5. Surface velocity at different locations (displacements from the shock center) as a function of shock arrival (left) and the surface displacement as a function of time at one location plotted for as series of time points (right).

3. TWO METHODS FOR SINGLE-SHOT CARS THERMOMETRY (CHIRPED AND PS/FS HYBRID CARS) IN HOT GAS.

Measuring the temperature of explosive during shock is critical modeling the kinetics of initiation and determining the equation of state of unreacted explosive under uniaxial shock. However, shocked materials are harsh environments where material heterogeneities and large fluxuations in temperature ($> 2000\text{K}$) and pressure ($>5\text{ kPa}$) over short length scales ($< \mu\text{M}$). Approaches that require a number of shots over a series of time step will have a low apparent signal to noise, because each shot samples a different distribution of material heterogeneity. Therefore, a single-shot measure of pressure or material temperature would be advantageous for characterizing the distribution of heterogeneities with in the material and improving S/N for each shot. Therefore, we explored two single-shot methods to measure femtosecond Coherent Anti-Stokes Raman spectra (CARS) in heated gas: 1.) chirped probe CARS⁵ and 2.) hybrid fs/ps CARS.⁶ Measurements of this type have already been demonstrated at flame temperatures to measure temperature and species concentration.^{5, 6} While the rotational and vibrational structure may not exist in compressed explosive material at high pressure, the gas phase is a good place to contrast and compare different approaches for the identification of chemical species.

CARS is a coherent Raman spectroscopy that depends on three field interactions with femtosecond laser pulse(s) to generate light blue shifted by the energy of Raman transitions excited within the target molecule(s). The first two pulses (pump and Stokes) generate a coherently oscillating ensemble of rotational and vibrational coherences. A third probe pulse scatters off this coherence grating in the direction $k_{\text{signal}} = k_{\text{pump}} - k_{\text{Stokes}} + k_{\text{probe}}$ to generate the CARS signal. Broadband ns sources were first used to measure gas temperature in flames, but the interference of non-resonant background with Raman transitions, low repetition rate (10 Hz) and stochastic noise of ns sources have driven to the use of femtosecond pump and Stokes pulses combined with a picosecond probe at later times. The commercial systems used in these measurements are more stable, are less sensitive to pressure broadening and run at kHz repetition rates enabling high signal to noise thermometry and gas concentration measurements with higher sampling rates.

We have demonstrated two of a number of possible approaches for single-shot CARS measurements (Fig. 6). In chirped-probe-pulse (CPP) CARS, the probe is linearly chirped in a glass rod, so that different frequencies within the probe interact with the Raman coherence at different times. If the complex field of the probe pulse is known, the time-domain profile is contained within the spectrum of the CARS signal; which is the sum of interfering spectrum generated by each spectral component within the probe field. The second involves improving the frequency resolution of the CARS signal by narrowing the spectrum of the probe to generate a picosecond pulse (fs/ps hybrid CARS). We successfully demonstrated both methods in heated gas, using rotational transitions in N_2 to determine temperature.

Figure 6. Spectrum and time order of preparation pulses plotted with the resulting spectrum of CARS/CSRS signal for CPP-CARS (left) and ps/fs hybrid CARS (right).

3.1. Experimental set-up

A “single-box” Ti: Sapphire amplifier (Spectra Physics Solstice) is utilized to provide pump, Stokes and probe beams to the CARS/CSRS process centered at 800nm. In the CPP CARS experiment, a 30-cm-long SF-10 rod, identical to the rod used by Roy et al.⁵ in their CPP vibrational CARS experiments, is inserted into the probe beam line. The rod stretches the probe beam from ~100 fs to 1.7 ps. In the ps/fs hybrid CARS experiment, based work by Miller et al.⁶ half of a 4-f pulse shaper is used to spectrally narrow the probe to ~. The 100-fs pump and Stokes beams remain temporally coincident (± 10 fs) to prepare both Stokes and anti-Stokes rotational Raman coherences at frequencies accessed within their bandwidth. The resulting CARS/CSRS signal is nearly frequency degenerate with the applied laser fields. Carefully aligned apertures and a tight 50- μ m focus at the entrance slit of the 0.27-m imaging spectrometer provide efficient spatial separation of the strong, room temperature signal CARS/CSRS beam from the applied laser fields. An electron multiplying CCD camera (Andor Newton) was directly mated to the spectrometer to provide shot-averaged and single-shot detection at rates up to 250 Hz in our demonstration experiments. Diagrams of each experiment are shown in Figure 7.

Figure 7. Diagram of CARS experiments shown for CPP-CARS (left) and ps/fs hybrid CARS (right).

3.2. Theory

Modeling of the CARS spectra is was carried out as described in Kearney (AIAA, 2012)⁷ and Kearney (Optics Letters, 2013).⁸

3.3. Results

1.1.1. CPP-CARS

The following is a synopsis of reference 7 for N₂ rotational CPP-CARS spectra. A chirped probe pulse was obtained by passing a 100-fs duration 800-nm beam through the SF-10 rod. The probe delay was scanned from -5 to 25 ps in room-temperature N₂ plotted in Fig. 8. Here, the CARS/CSRS intensity is plotted on an arbitrary false color scale, which has been adjusted to reveal the relative intensity of the features. The structure of the CPP CARS/CSRS spectrum alternates between major- and minor-recurrence peaks. The spectra for a major recurrence peak at a probe delay of $\tau_{\text{probe}} = 12.7$ ps and for a minor recurrence peak at $\tau_{\text{probe}} = 14.7$ ps are shown in Fig. 9. The 100-shot-averaged measurements are compared to a model calculation with the probe-beam parameters used to construct the spectrograms shown in Fig. 8. Single-laser shot data were readily obtainable with our setup and 20 representative single-shots in room-temperature N₂ for a major recurrence peak at 12.7 ps are shown in Figure 10. The single-shot results are shown as thin colored lines, with the model calculation shown as a thick blue line for comparison. Data and calculations show good agreement, signal to noise detection limits and measurable temperature sensitivity in both major and minor recurrence peaks (Fig.11). However, small amounts of curvature in spectrogram features (Fig.8) and the relative amplitude and spectral location of feature of the minor recurrence peak (Fig.9) are not reproduced by the model. We believe that this disagreement is the result of imperfect characterization of the probe-

beam electric field which is not strictly Gaussian and linearly chirped as in the model calculations.

Figure 8. CPP rotational CARS/CSRS spectrograms at $T = 304$ K. The linearly chirped probe spectral bandwidth is 180 cm^{-1} and the temporal probe width is 1.7 ps. Measured spectrograms (including the nonresonant spike near zero probe delay) are on the left. Calculated spectrograms are on the right.

Figure 9. Room-temperature N_2 CPP rotational CARS/CSRS spectra for N_2 at probe time delays centered on major (left) and major (right) recurrence peaks in the time-domain molecular response.

Figure 10. Single-laser-shot N₂ CPP rotational CARS/CSRS spectra at room temperature. The probe time delay is centered on a major recurrence at 12.7 ps. The theoretical calculation is shown as a thick blue line.

Figure 11. Temperature dependence of calculated CPP rotational CARS/CSRS spectra for N₂ at probe time delays centered on minor and major recurrence peaks in the time-domain molecular response.

1.1.1. Hybrid fs/ps CARS

In the hybrid fs/ps CARS experiment we spectrally narrowed the probe pulse to $\sim 5\text{cm}^{-1}$ FWHM ($\sim 300\text{ps}$). Again the probe pulse arrives at a later time to elimination non-resonant background but is timed to a recurrence peak in the rotational N₂ time-domain spectra. Measured room temperature spectra show the more familiar distribution of rotational lines where the resolution of the lines is determined by the linewidth of the probe pulse. Unlike CPP-CARS, hybrid CARS spectra do not require accurate measurements of spectral phase over a large bandwidth and are therefore more easily fit for temperature with similar accuracy.(ref-Miller) However, the pulse shaper removes a large amount of energy from the probe pulse ($< 9\text{ uJ}$), significantly reducing signal. Despite this disadvantage, the observed signal to noise and low shot-to-shot variations in

signal intensity where exceptional, motivating hybrid fs/ps CARS studies using a high energy picosecond source (second harmonic bandwidth compressed probe) for gas thermometry in flames.⁸

Figure 12. Single-laser-shot N₂ fs/ps hybrid rotational CARS spectra at room temperature. The probe time delay is centered at 20 ps. 1000 consecutive shots overlaid.

4. CONCLUSIONS

We have successfully demonstrated the tools required to study chemical change on fast timescales behind a shock wave in thin film explosives. We have successfully demonstrated laser induced shock with durations of a few hundred femtoseconds with interferometric characterization (USI) of particle velocity on picosecond timescales. We have also demonstrated two single-shot CARS spectroscopies that could be used to follow chemical change behind the shock-wave. Preliminary measurements at room temperature suggest hybrid fs/ps CARS is the best method for this application because hybrid CARS spectrum are not strongly dependent on variations in the spectral phase of the probe field over a wide bandwidth.

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