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MEASUREMENTS OF THE RUBY LUMINESCENCE SPECTRUM TO 125 KBAR AT ROOM TEMPERATURE

Paul D. Horn

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Shock Dynamics Laboratory

Department of Physics

Washington State University

Pullman, WA 99164

I. INTRODUCTION

The purpose of this report is to describe the measurements of the ruby luminescence spectrum at room temperature and under longitudinal stresses to 125 kbar. In this range, the ruby behaved elastically. The method and apparatus will be described first, followed by the experimental procedure and data analysis.

II. METHOD AND APPARATUS

The luminescence is excited by light from a cw Argon laser via an optical fiber. The laser light is shuttered on shortly before the impact to minimize laser-induced heating of the ruby sample. The same optical fiber which carries laser light to the sample also collects the luminescence and delivers it to a double spectrometer for wavelength dispersion. The spectrum is then dispersed over time by an image converter streak camera, and the streak image is recorded on film or digitized by an optical multichannel analyzer (OMA). The first experiments were performed to demonstrate the feasibility of the technique, and are described by Horn and Gupta.[1]

A. Apparatus

A schematic drawing of the apparatus is shown in Figure 1. The laser employed was a Coherent Innova 90-6, providing 3-4 Watts at 5145 Å. The optics for alignment and focusing of the laser beam into the fiber, and the luminescence into the spectrograph were mounted on a platform attached to the spectrograph. Two dielectric laser mirrors positioned and aligned the laser beam, which passed through the dichroic beamsplitter (CVI SWP [45°] 5145T/6943R) and was focused into the optical fiber (Diaguide SMY400SY-UV) by a lens of 80 mm focal length. This fiber was coupled to a second length of fiber at the target chamber, to allow for a vacuum seal, using standard SMA fiber connectors and a fiberoptic splice bushing. The two fibers were optically coupled with Cargille index matching liquid. The fiber inside the chamber was about 1 m long, and delivered approximately 2 Watts of laser light to the target, which will be described in detail below. Luminescence was collected by this same fiber and returned to the recording system, where it was collimated by the 80 mm lens used to focus the laser beam into the fiber. The collimated luminescence was then reflected by the dichroic beamsplitter and focused into the spectrograph by a second lens of 80 mm focal length. Both lenses were plano-convex and were anti-reflection coated for the ruby luminescence.

A Spex 1680 double spectrograph was employed, with 1200 groove/mm gratings, providing a 18 Å/mm dispersion. The mirrors were silver coated to enhance throughput at the red end of the spectrum. This spectrograph displayed a good deal of astigmatism at the exit plane, possibly because of the large acceptance angle of the optical fiber and collection optics (i.e. the spectrograph optics were overfilled). A cylindrical lens was placed at the exit window to correct this, although subsequent tests indicate that good results can be obtained by restricting the light cone with an aperture if the loss of intensity is acceptable.

A Cordin model 160 streak camera was employed for temporal dispersion. This camera had a

[1] P.D. Horn and Y.M. Gupta,"Wavelength shift of the ruby luminescence R lines under shock compression", Appl. Phys. Lett. *49* (14), 856-858 (1986).

25 mm wide photocathode with 6-8 line/mm dynamic resolution, providing 150-200 "resolvable lines". The output phospor on the Cordin system is not deposited on a fiberoptic plate, so that lens coupling was necessary. This did not provide sufficient signal (a lens is only capable of about 1/25 the throughput of a fiberoptic system), and the addition of a gated microchannel plate image intensifier (ITTF4113 P-11 phosphor) was required. This yielded noisy but usable signals when operated at a "medium" gain setting.

A system optical magnification of 1.0 was chosen because it provided sufficient resolution of the R-lines (separated by 14 Å, i.e. 0.78 mm at the spectrograph) when considered in conjunction with the spectral range. Because the streak was recorded by an OMA with a 12.5 mm detector area (described below), the 19 mm (wavelength axis) x 50 mm (time axis) streak camera output was reduced in a tapered fiberoptic bundle (Galileo Electro-Optic). In this way, direct coupling was maintained between the intensifier and the detector (no lenses) and the highest throughput was achieved. A 3:1 taper was chosen as a good compromise between spectral window and time window.

The streak image was recorded by an OMA (EG&G Model 1460) employing an intensified Vidicon detector (EG&G Model 1254 Silicon Intensified Target). As mentioned above, the detector area of the Vidicon is 12.5 mm x 12.5 mm (square). The recording procedure breaks the image into "tracks" (in our case spectra at chosen time intervals) and channels (wavelength). In these experiments, 50 spectra were sequentially integrated over 30 ns time intervals. The equipment settings were:

camera sweep window — 2 μs
OMA no. channels — 500
OMA ΔY — 10
OMA no. tracks — 50
OMA channel time — 40 μs

The OMA was operated in Data Acquisition Mode 3 with five prep frames; such that after the start of acquisition, the system performed five 5 scans to remove noise from the screen, then would record one scan following an external trigger signal. In these experiments, no correction was made for track-to-track distortion in the Vidicon detector and camera because test streaks of a series of holes drilled in a plate showed very little curvature in the area of the detector where the data occurred. A detailed discussion of the operational parameters of this system is presented in the WSU/SDL internal report 86-05.

B. Target configuration

Most of the experiments involved targets consisting of a thin, z-cut ruby disk (0.5% Cr_2O_3 by weight, 0.75 inch diameter, 0.005-0.010 inch thickness, crystal c-axis parallel to the impact direction) pressed between two z-cut sapphire disks (0.125 inch thickness), and followed at the back by a fused silica disk (0.125 inch thickness). The purpose of the fused silica disk was to provide a partial stress relief, thus yielding two data points per experiment. The thickness and sound speed were measured in each of the sapphire disks and the fused silica disk by a ultrasonic reverberation technique. The values in sapphire were consistently too high (greater than 11.2 mm/μs). I believe this to be due to a combination of the difficulty in measuring sound speed on thin (with respect to

transit time) samples, and to dispersion of the acoustic pulse, which makes overlap of consecutive pulses difficult to judge. An aluminum mirror was vapor deposited onto the impact side of the ruby to enhance the generation and collection of luminescence. The contact faces of the target components were coated with indium by vapor deposition to obtain good mechanical coupling. A small clear area was maintained for passage of laser light and luminescence. The components were assembled and potted into an aluminum target ring containing tilt pins and holes for trigger pins. This ring was designed to be mounted on a fin assembly for alignment with the standard target ring. The impactor also consisted of a z-cut sapphire disk. The optical fiber was simply pressed against the back of the silica disk.

III. EXPERIMENTAL PROCEDURE

A. Optical setup

The order of alignment of elements in the optical system had noticeable effects on the quality of data obtained, and the following technique proved to be a good one:

1. With the photocathode of the camera blocked or shuttered, pass light from a Helium-Neon laser through the fiber from the target chamber to the spectrometer,

2. Align the fiber to obtain the proper transit of the beam through the spectrograph, then

3. Remove the He-Ne laser and align the Argon laser beam to enter the fiber using the two dielectric mirrors.

At all times during focusing of the streak camera, the light intensity was held to a minimum to prevent permanent damage to the photocathode of the camera or Vidicon detector. A low intensity spectral calibration source (a neon lamp with neutral density filters was good for this) was placed in the target chamber to illuminate the fiber weakly, and the streak camera was set in the focus mode. The streak camera objective lens was then adjusted until the best focus was obtained. Wavelength calibrations were then recorded as described below. A spectrograph entrance slit of 250 μm worked well during the shock experiment.

B. Wavelength calibration

The spectral range observed was roughly from 6840 Å to 7040 Å No calibration standard was identified which would provide a sufficient number of spectral lines in this range. The method chosen was to employ the ruby lines themselves in conjunction with spectral lines from a Neon calibration lamp. Spectra of the R-lines were taken at different settings of the spectrograph wavelength selector to cover the detector window. The corresponding wavelengths at the spectrograph setting used in the experiment were then calculated assuming a linear dispersion. Finally, a linear least squares fit of the wavelength vs. channel number was obtained. The position of the 6929.5 Å Neon line was found to be in agreement with the fit obtained in this manner.

C. Electrical setup

The time window recorded in these experiments was 1.5 μs. Because we wanted to record several hundred nanoseconds each of shock and release data, timing of the trigger to the streak camera was crucially important. In determining the standoff trigger time, the parameters considered were of two varieties:

1. electrical delays between shorting of the trigger pin and arrival of the camera sweep at the center of the Vidicon detector area — these include delays in cables and trigger generator as well as the time between trigger at the camera and arrival of the sweep at center, and

2. mechanical and optical delays — including the transit time of the shock from the impact face to the ruby and the transit time of the light through the optical fiber.

The pretrigger time was generally chosen to place the center of the shock/release waveform at the detector center. To avoid multiple triggering, a one-shot pulse generator was employed between the trigger pin and the delay generator.

D. Recording the Reference Spectrum

Shortly before firing, a reference spectrum was obtained. All streak spectra should be taken in a shuttered mode to protect the photocathode from overexposure. In these experiments this was done manually by blocking or passing the laser light. Since the acquisition of the new Imacon camera, an electromechanical shutter (Uniblitz 26L0A2Z5) has been installed. This shutter is triggered by first motion of the projectile, and opens in approximately 0.5 ms, leaving about 10 ms for the luminescence to build up before impact (with projectile velocity 0.5 mm/μs). A manual trigger was added to the Uniblitz controller to produce a 10 ms pulse for recording the reference spectrum. In recording reference spectra, the camera is triggered by a contact on the Uniblitz shutter.

E. The Shot Sequence

The sequence of events during the shock experiment was as follows:

1. Turn off vacuum pumps

2. Check readiness of triggers for camera, shutter, and oscilloscopes (dark slides out also)

3. Start OMA acquisition

4. Check that the streak camera shutter is open (if installed)

5. Insure that OMA prep frames are finished

6. Remove any manual beam stops from the laser path

7. Fire shot, and

8. Immediately store data on data disk and backup disk

IV. DATA ANALYSIS

Typical tracks from a 100 kbar experiment are shown in Figure 2. Figure 3 is a plot of the R-line spectrum at 100 kbar overlayed on the ambient spectrum. Two methods were employed to determine the peak positions. The first was to use the WSU-SDL program ipl to visually locate the centroids of the peaks for each line. The second involved a nonlinear least squares fit performed on the WSU mainframe computer and employing the sum of two Lorentzian functions as a model. There did not appear to be any significant systematic difference in the results of these approaches, so the simpler first approach was adopted. The estimated error in peak position is $\pm$ 1 Å. The wavelength shifts were determined by subtracting the peak positions in the reference record from those in the shot record.

The strain was calculated from impact velocity by assuming ruby to follow the sapphire Hugoniot behavior.[15] The stress was determined from the nonlinear elastic analysis described in Appendix A. Shock induced heating is a small effect in sapphire at these stresses because of its stiffness, amounting to an approximately 9 K increase at 125 kbar. This corresponds to a correction of the wavelength shift on the order of 0.6 Å at 125 kbar (at a rate of 0.07 Å/K). Even though these corrections are less than the experimental error, they have been included for completeness, and the calculations of temperature increase are described in Appendix B.

A plot of wavelength shift vs. density compression is presented in Figure 4 for longitudinal stresses to 125 kbar. The solid lines in Figure 4 are quadratic least squares fits, providing the values $\Delta\lambda_{R1} = (6.50 \times 10^2 \text{ Å})\mu + (9.97 \times 10^3 \text{ Å})\mu^2$ and $\Delta\lambda_{R2} = (8.46 \times 10^2 \text{ Å})\mu + (6.89 \times 10^3 \text{ Å})\mu^2$, where μ is the density compression. The dashed line in Figure 4 is the equivalent hydrostatic result based on density compression. This was calculated assuming a bulk modulus of 2.57 mbar, a first derivative of the bulk modulus with stress of 4.00, and a 0.0365 Å/kbar rate of wavelength shift. The shock results are in reasonable agreement with the hydrostatic calculation, with the exceptions of a change in R_1-R_2 splitting, as seen in Figure 5, and some nonlinearity in the shock data. Table I is a compilation of these results, and the details of each experiment are given in Appendix C.

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APPENDIX A

Determination of a Nonlinear Elastic Relation for Uniaxial Strain Loading

In many shock wave problems, it is useful to know the elastic Hugoniot (locus of thermodynamic end states obtainable through the shock process). The method described here for calculating the nonlinear elastic stress-strain relation was formulated by Thurston [16,17]. It is applicable to finite strains and assumes pure mode propagation. We will present the case of uniaxial strain along the c-axis in sapphire, and will make no distinction between the Hugoniot and isentrope.

Consider two configurations: the initial configuration denoted by a_i, and the final or current configuration denoted by x_i. These configurations are related through the expressions

$$x_i = x_i(a_1, a_2, a_3, t) \quad ,$$

and

$$\alpha_i = x_i - a_i \quad ,$$

where α_i is the displacement. The particle velocity (u_i) is defined as

$$u_i = \left(\frac{\partial x_i}{\partial t} \right)_{\underline{a}}$$

Note that Thurston denoted the displacement by u_i and the particle velocity by v_i. A measure of finite strain is the Green strain, defined as

$$\eta_{ij} = \frac{1}{2} \left(\frac{\partial x_m}{\partial a_i} \frac{\partial x_m}{\partial a_j} - \delta_{ij} \right) \quad .$$

For uniaxial strain along the c-axis only α_{33} and, and thus, η_{33} are nonzero. Then, since $x_3 = a_3 + \alpha_3$

$$\eta_{33} = \frac{\partial \alpha_3}{\partial a_3} + \frac{1}{2} \left(\frac{\partial \alpha_3}{\partial a_3} \right)^2 \quad .$$

An engineering strain frequently used in shock wave studies is defined by

$$e = \frac{u}{D} = 1 - \frac{\rho_0}{\rho} \quad ,$$

where D is the shock velocity, the relation involving density is obtained from the Rankine-Hugoniot jump conditions [14]. The ratio of initial density to final density is equal to the Jacobian for the transformation from final to initial coordinates (the inverse of the ratio of initial to final volumes), which because of the uniaxial strain condition contains only the single nonzero term

$$J = \frac{\partial x_3}{\partial a_3} = \frac{\rho_0}{\rho} \quad .$$

This leads to the result

$$\frac{\partial \alpha_3}{\partial a_3} = \frac{\partial x_3}{\partial a_3} - 1 = -e \quad ,$$

thus,

$$\eta_{33} = -e + \frac{1}{2}e^2 \quad .$$

The negative sign here indicates the usual convention in mechanics of designating η_{ij} as positive in tension, whereas we have defined e to be positive in compression.

In order to obtain the stress-strain relation, we consider a series expansion of the internal energy at constant entropy

$$\rho_0 U(\eta_{ij},S) = \rho_0 U(0,S) + \frac{1}{2} C^S{}_{ijkl}\eta_{ij}\eta_{kl} + \frac{1}{6} C^S{}_{ijklmn}\eta_{ij}\eta_{kl}\eta_{mn} \quad .$$

where U is the internal energy per unit mass and S is the entropy per unit mass. Note that this is a generalization to solids of the complete equation of state E(S,V) used for fluids. The isentropic elastic constants are defined by

$$C^S{}_{ijkl} = \rho_0 \left(\frac{\partial^2 U}{\partial \eta_{ij} \partial \eta_{kl}} \right)_S \quad ,$$

and correspondingly for $C^S{}_{ijklmn}$. The thermodynamic stresses are given by Thurston as

$$t_{ij} = \rho_0 \left(\frac{\partial U}{\partial \eta_{ij}} \right)_S \quad .$$

Applying this to the series expansion of internal energy, and taking great care to properly account for the summations implied by repeated indices, we obtain the relation between the thermodynamic stresses and the strain as

$$t_{ij} = C^S{}_{ijkl}\eta_{kl} + \frac{1}{2} C^S{}_{ijklmn}\eta_{kl}\eta_{mn} \quad .$$

Accounting for the uniaxial strain then gives

$$t_{ij} = C^S{}_{ij33}\eta_{33} + \frac{1}{2} C^S{}_{ij3333}\eta^2{}_{33} \quad .$$

The stresses σ_{km} are related to the t_{ij} by

$$\sigma_{km} = \frac{1}{J} \frac{\partial x_k}{\partial a_j} \frac{\partial x_m}{\partial a_i} t_{ij} \quad .$$

The longitudinal (σ_{33}) and lateral (σ_{11} , σ_{22}) stresses can now be written as

$$\sigma_{11} = \frac{\rho}{\rho_0}\left[C^S{}_{1133}\eta_{33} + \frac{1}{2}C^S{}_{113333}\eta^2{}_{33}\right] ,$$

$$\sigma_{22} = \frac{\rho}{\rho_0}\left[C^S{}_{2233}\eta_{33} + \frac{1}{2}C^S{}_{223333}\eta^2{}_{33}\right] ,$$

$$\sigma_{33} = \frac{\rho_0}{\rho}\left[C^S{}_{3333}\eta_{33} + \frac{1}{2}C^S{}_{333333}\eta^2{}_{33}\right] ,$$

The value of $C^S{}_{33}$ can be calculated from the density ($\rho_0 = 3.985\frac{g}{cm^3}$) and longitudinal sound speed ($c_l = 11.19\frac{mm}{\mu s}$ [15]) as

$$C^S{}_{33} = \rho_0 c^2{}_l = 4990\ kbar\ .$$

The remaining constants are given by Hankley and Schuele [18] as

$$C^S{}_{1133} = C^S{}_{2233} = 1109\ kbar\ ,$$

$$C^S{}_{333333} = -33{,}400\ kbar\ ,$$

and

$$C^S{}_{113333} = C^S{}_{223333} = -9220\ kbar\ .$$

APPENDIX B

Heating Due to Shock Compression

Because sapphire is a very stiff material, the compressions produced in these experiments were small ($\mu < 0.025$). Accordingly, the entropy increase (which is proportional to the cube of the volume change) is also small and the shock process can be treated as isentropic. In this case, the temperature increase can be calculated from the expression

$$Tds = C_v dT + T\left(\frac{\partial P}{\partial T}\right)_v dv.$$

Since ds = 0,

$$\frac{dT}{T} = -\left(\frac{\alpha}{C_v \kappa_T}\right) dv.$$

Thus, with $\frac{\alpha}{C_v \kappa_T} = \frac{\gamma}{v}$,

$$T = T_0 \left(e^{\frac{\gamma}{v}(v_0 - v)}\right).$$

The values employed in the study were $v_o = 0.253$ cm^3/g and $\gamma = 1.27$,[13] with the assumption that $\frac{\gamma}{v}$ is constant.

APPENDIX C

Details of the Ruby Luminescence Experiments

Ruby Luminescence Shot Specifics

Shot #86-510

Component	Thickness (inches)	Sound Speed (mm/μ/s)
front sapphire	0.1254	11.15
ruby	0.0053	-----
rear sapphire	0.1256	11.36
fused silica	0.1252	5.91

Velocity (mm/μs): 0.443

Comments: The structure of this wave profile (Fig. 6.) shows some downward slope after the shock step for the R_1 line. None of the other data in the 125 kbar range show this behavior, and we currently have no explanation for it. For this reason, only the initial jump is included in the data compilation and no release data is quoted.

Ruby Luminescence Shot Specifics

Shot #86-521

Component	Thickness (inches)	Sound Speed (mm/μ/s)
front sapphire	0.1254	11.59
ruby	0.0053	-----
rear sapphire	0.1255	11.73
fused silica	0.1259	5.99

Velocity (mm/μs): 0.543

Comments: The low gain setting of the MCP image intensifier was used.

Ruby Luminescence Shot Specifics

Shot #86-536

Component	Thickness (inches)	Sound Speed (mm/μ/s)
front sapphire	0.1256	11.36
ruby	0.0104	-----
rear sapphire	0.1257	11.33
fused silica	0.1256	6.02

Velocity (mm/μs): 0.332

Comments: This experiment was performed by an undergraduate assistant, Jeannette Lawler. The ruby employed was 0.01 inches thick rather than the previously used 0.005 inch samples to obtain more signal.

Ruby Luminescence Shot Specifics

Shot #86-547

Component	Thickness (inches)	Sound Speed (mm/μ/s)
front sapphire	0.1255	11.3
ruby	0.0103	-----
rear sapphire	0.1254	11.3
fused silica	0.1257	6.0

Velocity (mm/μs): (0.454) cos 15 ° = 0.439

Comments: This was a compression/shear shot; with an inclination of 15 °. The purpose was to look for spectral changes with shear stress. If the shear were perfectly coupled into the ruby, the magnitude of shear stress would have been 14.5 kbar. No indium layers were deposited on these components; however, an aluminum mirror was deposited on the impact face of the ruby. For a shear wave velocity of 6.5 mm/μs, the shear should have arrived at the ruby 220 ns after the arrival of the longitudinal wave. As can be seen in Figures 12 and 13, any effect that occurred is smaller than the noise fluctuations. In retrospect, we may note that if the effect of shear were comparable to that of compression, 15 kbar stress would yield a shift of only 0.5 Å, so that this test is inconclusive. In order to avoid any shear effects in the release data, we include in this compilation only the data from the initial step, before the shear wave arrival.

Ruby Luminescence Shot Specifics

Shot #86-552

Component	Thickness (inches)	Sound Speed (mm/μ/s)
front sapphire	0.1256	11.29
ruby	0.0049	-----
rear sapphire	0.0623	-----
fused silica	0.1257	6.25

Velocity (mm/μs): 0.222

Comments: This was a low stress experiment to compare with earlier unloading results. In addition, a thin rear sapphire disk was employed to provide more release data before the arrival of edge effects. Figure 4 shows that the shock and release data are in good agreement.