High Pressure Laser-Generated Shocks and Application to EOS of Carbon

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Abstract. This paper describes the generation of very-high pressures shocks by means of laser beams, and their use for Equation-of-State experiments on materials in the Megabar pressure ranges. In particular, we present some applications to the study of EOS of carbon. Finally, we discuss non-equilibrium processes in laser-driven shocks.

1. Introduction
The possibility of creating very high pressures (Megabar range) is very interesting for many fields of physics. Materials compressed to such high pressures undergo interesting phase changes, therefore allowing exploring the phase diagram up to extreme conditions and studying matter characteristics in such regimes.

This is interesting for Inertial Confinement Fusion (ICF) where a DT target must be compressed by laser beams to very high density and temperatures in order to create the conditions for thermonuclear fusion reactions.

Also high pressures in physics are naturally found inside stars and planets. This allows studying conditions relevant to the interior of the earth (geophysics), as well as conditions found in giant planets (planetology) and in interesting objects as brown dwarfs (astrophysics).

Pressures higher than a few Megabars can only be created for very short times by using dynamic compressions, i.e. creating a shock wave and allowing it to propagate in the material to be studied. Gas guns and chemical explosions are currently among the tools used for creating shock waves, although
even nuclear explosions have been used in the past. Most recently however high-power laser-beams have more and more been used as a tool for high-pressure shock generation.

In this paper, we will first describe the generation of shock by laser beams, then, as an example, we will describe the applications of laser driven shocks to the measurement of Equation of State (EOS) points for a physically interesting material (carbon). Finally, mainly from an experimental point of view, we will address the point of non-equilibrium conditions in laser-driven shocks.

2. Generation of “high quality” shocks, diagnostics and EOS experiments

The generation of high pressures shock wave travelling in laser-irradiated materials is a consequence of laser ablation and plasma expansion in vacuum. Due to momentum conservation the material is pushed inside (Newton’s third law). The problem has been largely discussed in literature and many scaling laws have been formulated relating the generated shock pressure to the parameters of the laser and the material. One of such laws is for instance [1]:

$$P (\text{Mbar}) = 11.6 \left( \frac{I}{10^{14}} \right)^{3/4} \lambda^{-1/4} \left( \frac{A}{2Z} \right)^{7/16} \left( \frac{Z^* t}{3.5} \right)^{-1/8} \quad (1)$$

Here I is the laser intensity on target in W/cm$^2$, $\lambda$ is the laser wavelength in µm, and A, Z, and $Z^*$ respectively are the mass number, the atomic number, and the effective ionization degree of the target, and the time t is in ns.

It is clear that irradiating a system with intensities of the order of $10^{14}$ W/cm$^2$, which can be obtained quite easily, allows getting very high pressures, of the order of 10 Mbar. However, looking again in the literature, there is a 20 years delay between the early discover of high-pressure generation [2-4] and the first reliable measurements of EOS performed with laser-shocks [5-7].

Such delay is the consequence of the fact that the intensity profile of high-power laser beams is often characterized by the presence of hot spots and in any case is far from being ideal. Different laser intensities in the spot bring to locally different pressures and to a material sample, which is non-uniformly compressed and therefore cannot be used for significant experiments. Uniform “high quality” shocks were produced using different techniques in the ‘90s [8-10]. We used an approach based on the introduction of optical smoothing techniques (Phase Zone Plates, PZP [10]), which allows eliminating the laser hot spots while getting an almost flat-top laser irradiation profile.

Fig. 1, shows an image obtained by looking at the rear side of a laser irradiated target with a streak camera. This produces a 1D image (along the horizontal direction), which is resolved in time (vertical direction, time is flowing up to bottom). The streak camera looks along a diameter of the laser focal spot and records the emissivity of the target. At the beginning the image is dark because the material is cold and it does not emit any light. But when the shock breaks out the target rear side, matter is almost instantaneously heated to a few eV temperatures and therefore become highly emissive. The image clearly shows that the breakout takes place almost at the same time in a large region (about 200 µm). This means that the shock is travelling at the same velocity in all points, and therefore the shock pressure is also the same everywhere (no 2D effects). Of course this is not true at the edges of the focal spots where the laser intensity falls down and therefore the shock pressure also falls down, implying a slower shock velocity. Indeed the image of the shock breakout is curved at the edges, because the slower shock breaks out later.
Fig. 1: Streak camera image of rear side shock breakthrough for a 13.8 µm Al target. Optical smoothing is realized with PZPs giving a measured focal spot diameter FWHM = 400 µm. On the left there is a time fiducial realized by sending a part on the incident beam on the slot of the streak camera with an optical fibre.

Once planar shocks have been produced, they can be used to uniformly compress a sample of material. The dynamics of the shock is governed by 3 equations called Hugoniot-Rankine relations:

\[
\begin{align*}
\rho_0 D &= \rho (D - U) \\
P &= P_0 + \rho_0 DU \\
E &= E_0 + U^2/2
\end{align*}
\]

The quantities \( \rho, D, U, P \) and \( E \) are respectively the density of the material, the shock velocity, the fluid velocity (velocity of the material behind the shock front), the internal energy per mass unit. The subscript \( \text{“}0\text{”} \) refers to the material before compression (unperturbed material), while the quantities without subscript refers to the material, which has already been compressed and heated by the shock passage. The Rankine-Hugoniot relations simply represent the conservation laws across the shock front: the conservation of mass, momentum, and energy respectively.

By looking at the Rankine-Hugoniot system, it is possible to see how this is a system of 3 variables containing 5 unknown quantities (i.e. the velocities \( D \) and \( U \) plus the 3 thermodynamical variables of the compressed material \( \rho, P \) and \( E \)). Therefore experimentally measuring 2 of such quantities implies closing the system, thereby obtaining the remaining 3. Therefore an EOS point of the compressed material is obtained. Experimentally the quantities, which can more easily be measured, are the velocities \( D \) and \( U \). This brings to an “absolute” determination of an EOS point and has been first done in an experiment at Livermore concerning the EOS of deuterium [7].

We have instead followed a different approach [5], which has the advantage that it can be performed even with relatively small laser systems (energy per pulse of the order of 100 J). In our approach, we use “2 steps - 2 materials” targets and we simultaneously measure the same quantity (the shock velocity \( D \)) in the two materials in the same laser shot. Now one of the two materials acts a reference, i.e. we assume its EOS to be well known. Therefore in this case we have 4 equations since the equation of state of the material, \( f(\rho, P, E) = 0 \), adds to the 3 Rankine-Hugoniot relations. Hence here the experimental measurement of the shock velocity allows closing the system and getting all the other quantities, including the shock pressure \( P \). But this is the same pressure, which drives the shock in the second “unknown” material. Therefore the system can be closed even for the second material and one EOS point can be obtained. This is a “relative” method because the EOS point of the
unknown material is used as a reference for comparison [actually the shock pressure is not the same in the two materials, but is related through so-called impedance mismatch relations which are known once the EOS is known].

Fig. 2 shows a streak camera image obtained with a stepped target of one material only, to illustrate the principle of measurement of shock velocity.

![Fig. 2: Rear side shock breakthrough for an Al target with a 7.5 µm basis and a 2.8 µm step. Optical smoothing realized with PZPs.](image)

In this way the shock velocity is simply obtained by $D = \frac{d}{\Delta t}$, where $d$ is the known target step thickness and $\Delta t$ is the shock transit time in the step, directly measurable from the image in Fig. 2. It is clear that in order to apply the method, the shock must have a constant velocity in the step. Indeed shock steadiness is another requirement of the method, which is obtained by balancing target thickness and laser pulse duration. Finally, there is another problem connected to the use of Rankine-Hugoniot relations to measure EOS points, i.e. that of preheating. Indeed the method requires the status of the material prior to shock passage to be perfectly known. However the laser irradiated plasma on the target front side is very hot (the temperature may easily be of the order of 1 keV) and can be an intense source of radiation up to the XUV and soft X-ray spectral regions. Such radiation may be a problem because it can penetrate matter ahead of the shock and heat it, thereby changing the parameters $\rho$, $P$, $E$. If this is the case it is clear that the Rankine-Hugoniot relations can no longer be successfully applied.

Preheating is a very difficult experimental problem and can be solved only by a proper combination of laser irradiation parameters (so to minimize heating of the front plasma and X-ray emission), target thickness (so to minimize the quantity of X-rays arriving in the region under study) and by putting a proper ablator on the target front side, i.e. by inserting a layer of low Z between the laser and the real target, again in order to minimize radiation generation [11].

Before closing this section, let’s just observe that the diagnostic which we have described until now (i.e. using a visible streak camera) is a “passive” diagnostics, i.e. completely relies on recording target self-emission. On the other side we also have “active diagnostics” based on the use of auxiliary beams to probe the target under shock compression. One of such diagnostics, which has proven to be very useful in laser-driven shock experiment, is the VISAR [12-13], which is based on reflecting a laser probe beam on the target rear side. This allows measuring target reflectivity (which is related to the electrical conductivity of the shock-compressed material) and target rear side velocity (through the Doppler shift of the reflected light) at the same time.
3. Application to the study of carbon

The EOS of Carbon in the Megabar pressure range is very interesting for physics and material science and it has also several applications (e.g. planetology). Its phase diagram indeed predicts carbon metallization at high pressures (also, it includes several liquid metallic and semi-metallic states). The consequences in planetology are clear. Indeed carbon (through methane, CH₄) is one of the main constituents, together with water and ammonia (H₂O, NH₃) of the mantle of planets of the size of Uranus and Neptune, the so-called «hot ices» layers. Under the huge pressures present in the mantle of such planets, pyrolysis of methane and separation of carbon are predicted and could form a separate metallic liquid carbon or diamond layer.

The existence of a fluid, conducting region (based on water and/or carbon) could indeed justify the presence of the high magnetic fields measured by Voyager 2. Also the fact that the source of such field is in the mantle (instead of the core, as in the case of earth) could indeed justify the strong asymmetry of such magnetic field.

In order to obtain new EOS points for carbon in the Megabar pressure range, we performed an experiment using the in-principle arrangement shown in Fig. 3. Some laser shots were done at LULI where three laser beams at λ= 0.53 µm were focused at intensities of ≈ 5 × 10¹³ W/cm². The pulse was Gaussian in time with a full width at half maximum (FWHM) of 600 ps. In order to increase laser energy (and shock pressure), other shots were done with the PALS iodine laser [14], with typical energy of 250 J per pulse at a wavelength of 0.44 µm, focused up to 2 ×10¹⁴ W/cm². The pulse was Gaussian with a FWHM of 450 ps. In both cases, large focal spots and Phase Zone Plates (PZP) were used to get uniform laser illumination and avoid 2D effects in the propagation of the shock.

Fig. 3. Scheme of the experimental set-up. The CH layer may be (or may be not) present in order to reduce X-ray emission from laser irradiated side. The probe laser, used at LULI only, was a Nd:YAG converted to 2ω with pulse duration of 8 ns.

Some carbon depositions were done at the University of Milan using the Supersonic Cluster Beam Deposition (SCBD) technique with appropriate masks [15, 16], which allows quite uniform layers and steep steps to be deposited. The particular deposition system allows carbon to stick on Al avoiding usual de-lamination problems. More important, it is possible to deposit carbon layers with densities variable between 1 and 2 g/cm³. In our experiment, carbon layers with initial density ρₒ = 1.45 ± 0.10 g/cm³ were used. The deposition technique allowed the realization of targets with an acceptable surface roughness (less than 0.5 micron, i.e. ≈3% of step thickness which was of the order of 15 µm). These give an error comparable to the typical ≤ 5% due to streak camera resolution. The Al step thickness was 5 µm.

Other carbon targets were fabricated at General Atomics [17] using a completely different technique based on the use of colloidal carbon. In this case, carbon with initial density ρₒ = 1.6 ± 0.10 g/cm³ was produced. Stepped targets were made of lathe machining of bulk aluminium. The Al base
was ≈ 8 µm, and the step thickness was ≈ 8.5 µm. The carbon layer was then produced and the target was machined again to produce the C step (with thickness ≈ 10 µm). The use of two different type of targets allow a comparison of measurements and a better confidence in our results.

Fig. 3. Shock breakout streak image of the target rear side in emission. Shot energy was 25.3 J. Arrows indicate the shock break-out from the Al step (right) and from the C step (left). The size of the image is 600 µm × 1.7 ns.

Fig. 3 shows a typical results obtained from the emissivity diagnostics. In total we obtained 5 good experimental points at LULI (2 for ρ₀ = 1.45 g/cm³ and 3 for ρ₀ = 1.6 g/cm³) and 4 good points at PALS (all for ρ₀ = 1.6 g/cm³). These are shown in Fig. 4 and are compared to the shock polar curve derived from the Sesame tables [18], the model QEOS [19] and the recent theory developed by Wang et al. [20].

The errors on pressure and fluid velocity are ≈ 15 % and ≈ 20 % respectively; these error bars have been estimated by calculating the propagation of experimental errors on shock velocity (5%) on the quantities determined by the mismatch method. The error on shock velocity is instead determined from the experimentally measured uncertainties on step thickness and by streak-camera temporal resolution.

All our data, for both initial densities, are below the shock polar curve derived from the models (see Fig. 4). Despite our quite large error bars (which make most points compatible with the theoretical curve), such results show a systematic deviation, and indicate a compressibility of carbon, at these pressures, much higher than what predicted by most models (the density ρ of the compressed sample is obtained from the Hugoniot Rankine relations for shocks, namely from ρ = (D-U)/ρ₀ D). However such behaviour could be also due to the presence of systematic errors in our experiment.

One possible cause often cited for explaining errors in laser-shock EOS experiments is preheating induced by X-rays. Experimentally, preheating can be evaluated: a) using the self-emission diagnostics, from the early emission from target rear before shock breakout; or by using the VISAR diagnostics b) from an early drop in reflectivity; and c) from fringe motion before shock breakout. In our case, preheating was small for the points at LULI due to the rather low laser intensity and the presence of a CH layer on laser irradiated side, which reduces X-ray generation. On the contrary, for the shots at PALS preheating was measured by calibrating the emissivity diagnostics and, for the two shots at higher energy (pressure), it was as high as a couple of eV [21]. Despite this, the LULI points are as far from Sesame as the PALS points. Therefore preheating is probably not the cause of deviation from theoretical curves (or at least not of the whole deviation). Another possible systematic
effect could be due to the high porosity of the targets, even if porous and foam targets are routinely used in EOS experiments.

Fig. 4: Our experimental results compared to the Sesame tables [18], the QEOS model [19] and the new theory developed in [20]. Here, in agreement with the choice done in [20], the ordinate represents the quantity $\rho_o/\rho$ where $\rho_o$ is the initial cold density of diamond (3.51 g/cm$^3$) and $\rho$ the density of our compressed sample.

In conclusion, concerning the carbon EOS, our experimental results show that carbon seems to be more compressible than what predicted by theoretical models. The relation between shock velocity $D$ and fluid velocity $U$ for carbon in the Megabar range is linear ($D = C + SU$, where $C$ is the sound velocity in the material in that pressure range). For $\rho_o = 1.6$ g/cm$^3$ Sesame or QEOS give $C \approx 5$ km/s and $S \approx 1.27$. A linear interpolation of our points yields $S \approx 1.08 - 1.14$. This gives a Hugoniot curve above the thermodynamic limit $P = \rho_o U^2$ corresponding to infinite compressibility (all our experimental points are above such limit). However, it seems too close to the shock polar for a perfect gas, which again could indicate an influence from systematic effects.

Despite such interpretation problems, it is worth to point our that in this work, we have presented the first Hugoniot data for carbon obtained with laser-driven shocks and we got the first experimental points at pressures higher than 8 Mbar. Moreover, we substantially increased the number of EOS data for carbon at pressures $> 1$ Mbar (here we showed 8 new EOS points against a total of about 20 points which, to our knowledge, were available in literature [22-26]).
4. Non equilibrium

Of course shock propagation in material, and the changes in the characteristics of the material it induces, is a non-equilibrium and non reversible phenomenon. However the final state of matter after shock compression will approach thermodynamic equilibrium conditions in the new state. Therefore the shock transition appears to connect two equilibrium states in the phase diagram of the material, although the shock transition in itself is irreversible and therefore does not belong to the equation of state surface of the material.

If we try to evidence non-equilibrium phenomena in these processes, we must face the problem that, due to the very high density (above solid matter) and relatively low temperatures, equilibrium is reached very rapidly, on time scales which can be of the order of a few ps, or even smaller, depending on the particular material and the particular transition under study. The problem of what are the actual time scales is very interesting and important from a theoretical point of view. On the other side, from the experimentalist’s approach, it requires the development of new techniques and new diagnostics methods. Indeed, unfortunately, the typical time resolution of the diagnostics which have been previously described (streak cameras measuring rear side emissivity, or streak cameras coupled to VISARs) is typically of the order of a few tens ps. In order to see the non-equilibrium we need diagnostics with much better time and space resolution.

A method to obtain a good precision on the shock or fluid velocity and at the same time to reach a very high temporal resolution, is the chirped pulse reflectometry (CPR). Such a method is based on using a chirped optical laser probe to determine the shock breakout time. The frequency chirp in the probe pulse gives a univocal relation between the instantaneous frequency and time. The time at which shock breakout takes place may hence be characterized by a sharp change in reflectivity at a given wavelength (again depending on the phase transition under study). Therefore the CPR technique allows to measure shock breakout time. With respect to the usual streak camera diagnostic, it also offers the possibility of higher temporal resolution, potentially 1 ps. The temporal accuracy depends on the ratio of the spectral bandwidth and the pulse length of the beam, i.e. \( \Delta \lambda / \Delta t \).

In this context, we have performed an experiment [27] based on the simultaneous use of the FDI (frequency domain interferometry) and the CPR techniques. The experimental set up is shown in Fig. 4a. This method allows, on the same laser shot:

i) The very precise determination of the shock breakout time thanks to the high temporal and spatial resolution typical of the CPR technique.

ii) The precise measurement of the fluid velocity \( U \) and/or the shock velocity \( D \) (depending on the experimental conditions) as a function of time.

iii) To check the quality of the shock front and to observe spatial and temporal microstructures not observable with the usual streak camera time resolution.

In this experiment, the target was made of a 4 \( \mu \)m layer of CH (laser side), and 10 \( \mu \)m of Al coated on 2 mm fused silica (rear side of the target). Since in this case we were not particularly interested in obtaining a flat-top irradiation profile, we didn’t use PZPs, but rather a random phase plate (RPP). This produce a focal spot characterized by an overall Gaussian shape and by small scale speckles. From the measurement of \( \Delta \lambda \) and the pulse duration \( \Delta t \) (FWHM = 75 ps) we obtained the relation \( \lambda(t) = \lambda_0 + \alpha t \), where \( \alpha \) is the chirp of the beam (see Fig. 4b). Before irradiating the target at 45\(^\circ\), the probe beam was passing through a Michelson interferometer to produce a pair of collinear probe pulses separated by \( \Delta t = 10 \) ps. The main diagnostic was based on imaging the probe light reflected from the interface quartz-quartz interface onto the entrance slit of a high resolution visible spectrograph by using a f/4 objective. A 12 bit CCD was placed on the output plane of the spectrometer as a detector. The magnification of the imaging system was 10, resulting in a spatial resolution of 4 \( \mu \)m in the target plane. Before the shock breakout the two probe beams are reflected on the unperturbed Al-SiO\(_2\) interface. In this case the spectrograph gives the fringe pattern caused by the interference in the frequency domain of the two temporally separated beams. After the shock breakout, the fringe pattern moves because of the phase shift between the two probe beams.
Fig. 5: a) experimental set-up used to implement the CPR and FDI diagnostics, b) experimental measurement of the chirp of the probe laser beam in our experimental configuration

Therefore, this diagnostic allowed us to measure both the amplitude variations and the phase shift of the reflected light induced by the modification of the optical properties of the perturbed material and by the motion of the reflecting surface. Moreover, the chirp of the two beams makes it possible to use the spectral axis of the spectrograph as a time scale and hence get the phase shift as function of time. Before each shot, a reference shot was performed by using only the probe beam. A typical image obtained with the spectrograph is shown in Fig. 5a. The horizontal cut represents the Fourier transform in the frequency domain of the sum of two chirped pulses (more precisely the squared modulus of the Fourier transform). We show an image with a shock breakout obtained with the spectrograph in Fig. 5b. The results emphasize some non-uniformities in the front shock on very small scales, connected to the small size speckles produced by the RPP. This is possible thanks to the very high temporal and spatial resolution achieved with the CPR technique (corresponding streak images show quite uniform shock breakouts). From the image of Fig. 5b, one can measure the phase shift associated with the motion of the reflecting surface as function of time and deduce its velocity. If we consider the interface moving with a velocity $U$, the Doppler phase shift is $d\Phi/dt = 4\pi Un \cos \Theta \ u/\lambda_0$, where $n$ is the refractive index of the quartz and $\Theta$ is the probe beam incident angle.
Fig. 5: Typical result obtained with the CPR /FDI diagnostics: a) reference shot (only probe beam reflecting on the Al / fused silica interface), b) real shot with a shocked target.

Fig. 6 shows the time behaviour of the velocity obtained from the experimental result of fig. 5b. When the shock arrives at the interface, a shock with approximately the same velocity is transmitted into quartz. In Fig. 6, we can distinguish three different phases:

i) For $t \leq 50$ ps the probe beam is reflected on the unperturbed quartz-quartz interface. The observed noise on the velocity corresponds to one pixel on the CCD camera.

ii) For $50 \text{ ps} < t < 80$ ps we found $U \approx 12.5 \text{ km/s}$. This corresponds to the interface velocity between aluminium and quartz, and due to the continuity of the Rankine-Hugoniot relations, to fluid velocity in both materials. Therefore, according to the SESAME tables, the shock pressure in aluminium is $P \approx 7 \text{ Mbar}$ as expected for the laser intensity used in the experiment. Here, the quartz is partially transparent and the probe beam is reflected at the interface, after going through the compressed quartz. The variation of the refraction index of compressed silica should be, in principle, considered in deducing the above fluid velocity. However, in our case very small thickness of the shocked silica, lower than one laser wavelength, allows us to neglect this effect. We finally considered the phase shift, due to the reflection at the aluminium surface under shock conditions.

iii) About 35 ps after the shock breakout, a higher velocity is observed. We think that this corresponds now to the reflection from the shock front in fused silica. We measured $D \approx 19 \text{ km/s}$, which is consistent with the fluid velocity $U \approx 12.5 \text{ km/s}$ obtained. Such velocity is not observed in the first phase because the thickness of the compressed silica increases with time as $(D-U)t$ and its absorption grows exponentially with such thickness, thereby quickly screening the Al/SiO$_2$ interface. At the same time, there is an augmentation of the electronic conduction, confirmed by an increase of the measured reflectivity. The strong shock compression induces either a plasma phase or metal transition.
Fig. 6: Behaviour of the velocity of the reflected interface obtained from the image of Fig. 5b.

In conclusion, we have shown the applicability of the simultaneous use of the FDI and the CPR techniques to measure shock parameters. A high temporal resolution of the order of 1 ps has been achieved. If step targets are used, this method could be applied to measure the shock breakout time and then the shock velocity, with a temporal precision much better than with a streak camera. Also fluid velocity can be simultaneously measured provided the back transparent layer does not metalize. Another important advantage of this technique is that it allows to evidence the presence of hot spots in the laser beam and their effects on the local shock breakout and preheating.

5. Conclusions
In this paper, we have shown how today high-power laser beams can be used to generate “high quality shocks” at multimegabar pressure and use them for EOS measurements (as well as for more general experiments aimed at measuring some other physical characteristics of compressed matter) and in order to evidence phase transitions (e.g. metallization).

This can be done even with “small” lasers (E ≥ 100 J) and allows obtaining physical data in completely new regimes. Error bars are usually still quite large (errors ≈ 10%) but this is not so relevant when one works in completely new and completely unexplored regimes. Moreover, even such error bars, sometimes already allow discrimination between different theoretical models (especially if one takes into account that low precision on the single shot can somewhat be compensated by the high output rate).

The time for reaching statistical equilibrium in matter at such high densities are very short. Therefore the diagnostics normally used in today’s shock experiments do not allow resolving the initial phase during which equilibrium is approached. However we have given an example of one experiment where such non-equilibrium phenomena are evidenced.
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