Solidified gas samples for shock wave experimentation

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Although shock wave experiments on a large class of ordinary solids and liquids have been obtained in the last 25 years, relatively few shock data have been reported for solidified gases initially at cryogenic temperatures. Most previous work on both liquid and solid gases has been limited to experiments using explosive lens techniques to generate shocks in the material under study. Recently data for liquid $\text{H}_2$ and $\text{D}_2$ to 0.9 $\text{Mbar}$ using pit-locator techniques have been obtained. These experiments use a container, with a chamber wall made of a material for which $\beta_{\text{H}}$ is known, in which samples are grown. Shunting pins are placed within or in contact with the sample chamber at accurately measured positions, and samples are grown around them. Measurement of the time interval between closing of the various shunting pins then gives a measure of the shock velocity. In explosive experiments the lens/plate assembly is generally shielded from the sample container by some type of thermal insulation until just before detonation (generally, a few seconds). It is assumed that appreciable warming of the sample does not occur in this time.

The experimental technique described uses a high performance propellant gun to launch 40 mm diameter projectiles in excess of 2.5 km $\text{s}^{-1}$ into an evacuated expansion and impact chamber. The solidified gas container is placed within an impact chamber and aligned with the muzzle of the gun. The optical system permits slight adjustment of the assembly after the solidified sample is produced in the chamber. Measurements of the projectile and shock velocities are carried out optically by monitoring the destruction of reflecting mirrors contained within the sample crystal. This design minimizes the effect of mechanical distortions and changes in the electrical characteristics of contactor pins as the assembly is cooled.

Technique

Samples of solid gas polycrystals are grown in situ in a target container which itself is contained within an expendable hard vacuum chamber. Target containers consist of an open-faced disc approximately 1 cm deep by 3 cm in diameter machined from type 304 stainless steel. Wall thicknesses are nominally 1.5 mm. Containers are mounted on a stainless steel driver plate. The impacted surfaces of the sample container are machined flat and parallel to 0.001 in. A copper flange is silver soldered to the open top of the target. A 3.75 mm in diameter by 1.5 mm thick fused silica window is glued to this flange with silicone adhesive (GE type 10545). Initially the flange was constructed with this window, but this often resulted in destruction of the window at 77 K. At 5 K, targets with a stainless steel flange always destroyed the windows. Since the thermal conductivity of the copper is much greater than either the fused silica window or the stainless steel chamber, it uniformly distributes the thermal stresses experienced by the window during cooling. The bond between the silica window and the copper flange was able to withstand over-pressures of two atmospheres at liquid helium temperatures with no measurable leakage at the interface. Deflection of hydrogen through the bond was negligible.

A vacuum-tight spherical bowl flange was mounted on the impact chamber. A 1 in thick stainless steel plate containing appropriate vacuum ports and Swagelock fittings was mounted on this flange. A 6 in diameter by 1 in wall stainless steel tube was fixture welded to the bottom of the plate with its axis coincident with the axis of the plate. This tube acted as a receiver for an expendable 2042 aluminum high vacuum chamber which encompassed the sample target. A projectile fired at the target entered the high vacuum chamber by penetrating a 0.001 in stainless steel foil which was bonded to an open flange on the chamber. A $\text{H}_2$ in kastite viewing port was attached to the throat by means of silicone rubber adhesive. An adjustable from surface mirror was mounted on the interior base of the dome. The mirror allowed the target to be illuminated and viewed via an optical path oriented at 65° with respect to the normal to the target.

Initial pumpdown of the high vacuum chamber was accomplished in conjunction with the pumpdown of the impact chamber of the gun. At pressures of 500 $\mu$N $\text{cm}^2$, the high vacuum chamber was closed off from the impact chamber...
vacuum and the shroud vacuum system was used to evacuate the shroud to pressures between 10^-4 and 10^-5 torr.

Sample preparation was accomplished with the aid of an external manifold. A Wallace and Tiernan pressure gauge was used to monitor sample feed pressure. The sample target chamber was first evacuated and then flushed with the appropriate sample gas. Pressure in the target was then brought up to slightly above atmospheric pressure (usually 32 in Hg) after which target cooling commenced.

Cooling of the target was accomplished by continuously passing the appropriate cryogenic liquid nitrogen in the liquid nitrogen in hydrogen experiments through a 5/16 in diameter copper coil. This coil attached to the lower 2/3 of the circumference of the stainless steel sample chamber. Two thin wall (0.020 in) stainless steel tubes were attached to the copper coil. The other end of these tubes was concentrically silver soldered to a 3/4 in diameter by 5 ft in long, 0.035 in thick stainless steel tube. These 3/4 in diameter tubes were connected to a stainless steel flange at the top of the vacuum chamber by Swaglok fittings. These inlet lines act as stand-off tubes, and minimize heat transport from the warm exterior environment to the target. A 1/4 in diameter type 304 stainless steel tube (which provided sample gas to the target chamber) was beveled to the top centre of the chamber. In the hydrogen experiments this tube was connected to the exterior gas supply by means of a stainless steel stand-off tube. For the hydrogen experiments it was found that the entrance of warm room temperature hydrogen gas into the liquid helium cooled target resulted in the formation of radial tension cracks in the fused silica window. To minimize the thermal stresses which induced this window failure, a liquid nitrogen heat exchanger was placed between the 1/4 in diameter stainless steel tube and the stand-off tube. The heat exchanger consisted of approximately 6 ft of 1/4 in copper tubing wound in a helix which was mounted in a 2½ in diameter by 9 in long brass dewar. This dewar was connected to the mounting flange by means of a stand-off tube and a copper vent tube held fast by Swaglok fittings.

To prepare argon samples, liquid nitrogen was gradually passed through the copper cooling ring until the sample gas pressure began to drop, indicating the onset of liquefaction. When the required pressure was reached, the dewar was removed and the shroud vacuum circuit re-downstreamed. After the required cooling period (usually required 45 minutes to 1 hour), the gas pressure was readjusted to the desired pressure, the shroud was evacuated, and the sample gas vented to waste.

When using liquid helium coolant, temperatures within the target varying little from 5 K were measured by means of
liquid helium vapour pressure, and 5 K was therefore adopted for the initial temperature of the solid hydrogen target. The heat input to the argon targets, as evidenced by the boil-off rate of liquid nitrogen, was very small (5 ml/s²). Thus the boiling point of liquid nitrogen (77 K) was adopted for the temperature of the solid argon targets.

Thermal cycling of the apparatus did not noticeably degrade the silicone rubber bands.

Fig. I is a schematic diagram of the essential components of the cryogenic vacuum system. The lead vacuum system was surrounded in its entirety above the spherical bowl flange.

The sample preparation manifold and the nitrogen coolant reservoir were located in a room adjacent to the gun, while the helium coolant was placed very close to the spherical bowl flange to minimize boil-off losses. Liquid helium was fed through a vacuum insulated transfer line using external helium gas pressure.

All samples which were shocked were visually free of cracks, voids, and inclusions when viewed with a 10 times magnification. The condition of the sample was visually monitored until 1 minute before impact (see Fig. 2). Sample gons were obtained from the Linde Corporation and had stated purities of 99.99% for argon and 99.999% for hydrogen.

A singular advantage of the present technique is that the sample may be held in a hard vacuum environment until projectile impact (and hence shock compression) occurs. In previous experiments, 2,3,4,5 it has not been possible to impact the condition of the solid gas sample prior to shock arrival. It is assumed that the specimen is pure and crack free. We have found that freeze-out of the sample gas from the freezing over of a partially liquid region can easily lead to flawed samples and thus the assumption of a 100% dense solid is not always justified.

Results

Argon Hugoniot data produced with this equipment are in near agreement with previous experimental data and are in excellent agreement with recent theoretical calculatations of argon Hugoniot points. 8 The data for hydrogen comprise the first such data published for the solid phase.

Data analysis and some implications of the shock wave data are presented elsewhere. 9

The technique of growing solid gas crystals in situ and performing dynamic compression experiments on them has been demonstrated to be a viable and reasonably straightforward technique.

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References