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Preliminary Experiments in High Temperature Vapours of Organic Fluids in the Asymmetric Shock Tube for Experiments on Rarefaction Waves (ASTER)

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Abstract. This paper describes the design, functioning, and preliminary experiments in the Asymmetric Shock Tube for Experiments on Rarefaction Waves (ASTER), a Ludwieg-type shock tube designed and realised to measure waves propagating in dense vapour flows of organic fluids. The setup is designed to operate at pressures and temperatures of up to 15 bar and 400 °C. The high and low-pressure sections of the tube are separated by a glass-disk barrier to ensure quasi-instantaneous opening. When the glass disk is broken, a rarefaction wave propagates into the tube. The wave speed is measured using a time-of-flight method with the help of four pressure transducers placed at known distances from each other. Leakage rates of 2.2×10^{-4} mbar·l·s^{-1} at vacuum and 5×10^{-4} mbar·l·s^{-1} at superatmospheric pressures were measured, which is considered sufficient for the conceived experiments. Preliminary rarefaction experiments in the dense vapours of dodecamethylcyclohexasiloxane, D_6, were successfully performed at various thermodynamic conditions. Also, a method for the estimation of sound speeds from the pressure sensor recordings is proposed. Results are found to be within 2.5% of the values predicted by the state-of-the-art thermodynamic model for D_6.

Keywords: ASTER · Ludwieg tube · Rarefaction wave · Time-of-flight · Speed of sound · Organic vapours

1 Introduction

Non-ideal Compressible Fluid Dynamic (NICFD) effects, and nonclassical gasdynamic effects within it, characterize the dense-vapour flows of high-molecular weight organic compounds, collectively known as Bethe-Zel'dovich-Thompson (BZT) fluids, in the critical point region and the vicinity of the vapour-liquid saturation curve [1]. The local fundamental derivative of gasdynamics Γ can exhibit values lower than 1 and can sometimes even become negative in this thermodynamic region giving rise to unconventional

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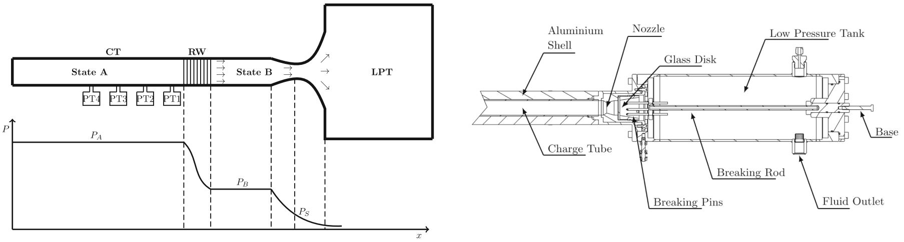
phenomena such as the admissibility of rarefaction shock waves (RSW), double sonic shocks, etc [2,3].

Though flows evolving in this region have been extensively studied theoretically, few experimental analyses have been carried out, and that too produced inconclusive results. Previous attempts to provide experimental evidence for the existence of RSWs in the vapour phase have been unsuccessful either due to the incorrect choice of working fluid or due to the presence of dissolved impurities such as air and moisture that are known catalysts for thermochemical decomposition [4,5]. The difficulty is further exacerbated by the fact that the $\Gamma < 0$ thermodynamic region spans a narrow range of pressures and temperatures. Though minor gradients in axial temperature have been shown to enhance nonlinear wave propagation in BZT fluids, large temperature fluctuations can cause mixed classical/nonclassical flows that are detrimental to the formation of RSWs, especially in a setup of limited length [6].

A series of shock tube experiments focussed on investigating NICFD flows in siloxane D_6 were pursued at TU Delft in the flexible asymmetric shock tube (FAST) facility [7]. The researchers performed preliminary speed of sound and wavespeed measurements in the non-ideal thermodynamic region of D_6 at temperatures up to 300°C and reported values within 8% and 1.6% respectively of the predictions by the state-of-the-art thermodynamic model. The authors also performed several rarefaction experiments at different initial conditions close to and in the predicted $\Gamma < 0$ region. However, they found no proof of either a RSW or a nonclassical isentropic expansion though they claimed that the measured value of Γ was close to zero [8].



(a) *(b)*

Fig. 1. (a) Schematic overview of a rarefaction wave experiment in the ASTER. The chart below shows the theoretical pressure profile in the charge tube after the breaking of the glass disk. The fluid is accelerated from rest at State A to State B and flows into the low-pressure tank through the nozzle, where sonic condition S is attained. The propagating rarefaction wave in the CT is captured using pressure transducers PT1 to PT4. (b) Cross-sectional drawing of the LPT showing the breaking pin mechanism and the glass disk

Recent measurements of the internal wall temperature of the FAST by the authors of this article showed large gradients in the axial direction. Since Γ is highly sensitive to the variation in the temperature, these measurements indicated that earlier experiments in the FAST might have suffered from mixed classical/nonclassical flow behaviour. Despite tight control and preventive measures that were already in place to control such gradients, the design of the FAST with multiple flanges and its large size made it unfeasible to maintain a constant temperature in the setup.

In this work, the design, development and commissioning of a new shock tube setup named the Asymmetric Shock Tube for Experiments on Rarefaction Waves (ASTER) are presented, along with preliminary measurements of rarefaction waves and local speeds of sound.

2 The ASTER Experimental Setup

The ASTER is designed based on the same principles of the FAST (see Fig. 1a) but is more compact and simple to operate. The working fluid is filled into the charge tube (CT), where it is heated to the desired temperature and pressure. A glass disk that separates the CT and the Low-Pressure Tank (LPT) (initially maintained at vacuum) is broken to initiate the experiment. Consequently, a rarefaction wave travels into the CT.

The CT is a 3 m long tube made of stainless steel (316Ti) with an outer radius of R = 17.5 mm and wall thickness of t = 2.5 mm. One end of the CT houses the inlet port through which the working fluid is injected with the help of a manual pump. The other end of the CT is connected to the LPT via a nozzle section. The nozzle creates a throat area to choke the flow, thereby preventing flow disturbances from travelling upstream into the CT. A 3 mm thick glass disk is placed at the exit of the nozzle. The nozzle opens into the LPT, a 1.1-litre cylindrical stainless steel tank of length 345 mm with an outer radius of 69.5 mm and a wall thickness of 4 mm.

Four dynamic high-temperature pressure transmitters PT1-PT4 (Kulite XTEH-10L-190SM-300PSI-A), each measuring pressure within 0.5% of the full range of 21 bar and with a sampling frequency of 250 kHz, are flush mounted along the CT at distances of 1.2, 1.4, 1.6 and 1.8 m from the nozzle throat respectively (at ambient temperature), for wavespeed measurements. These sensors are calibrated using a more accurate measurement from the Druck UNIK 5000 static pressure transmitter that is placed close to the inlet port of the CT.

The breaking of the glass disk is achieved using the mechanism shown in Fig. 1b. The breaking pins consist of six 8.5 mm long drill bits attached to a 320 mm long stainless steel rod that is mounted on a base of length 140 mm that protrudes outside of the LPT. Two Kalrez ® O-rings on the base ensure that the vacuum in the LPT is maintained and that the working fluid doesn't leak during experiments. To perform an experiment, the end of the breaking pin is hit with a hammer and the glass shatters in the least possible time. After every experiment, the LPT is opened to replace the glass disk and to evacuate the fluid from the setup.

The thermal energy required for heating the charge tube is supplied through two glass-silk heating jackets, each rated for 1250 W W, and having a 25 mm layer of insulation. The blankets are mounted on 17.5 mm thick aluminium shells to ensure sufficient temperature homogeneity along the CT. The temperature along the CT is measured and controlled using eight equidistantly placed K-type thermocouples that are bored into the aluminium shells to measure as close to the stainless steel wall as possible. In addition, a PT100 probe is placed into the CT via a thermowell to directly measure the fluid temperature. Both the thermocouples and the PT100 sensors are calibrated with the Fluke

9100 dry-well calibrator which has a rated accuracy of ±0.5 K. The LPT is heated using a 300 W heating jacket, the sole purpose of heating being to avoid temperature gradients at the nozzle section.

2.1 Choice of Working Fluid

Siloxane D_6 was chosen as the working fluid for experiments in the ASTER due to its high thermal stability, low levels of flammability and non-toxicity, and the large size of the non-classical thermodynamic region predicted by relatively accurate thermodynamic models [9]. The fluid was produced by Dow Corning and is certified to be 97.4% pure. The primary properties of the fluid are summarized in Table 1 [10]. Any moisture dissolved in the fluid must be removed before heating since its presence can adversely affect the thermal stability of the fluid. Dissolved water is removed by immersing 3Å molecular sieve desiccants in the fluid samples over several days.

Table 1. Relevant thermophysical properties of the working fluid D_6. MW is the molecular weight, P_c, T_c, and ν_c are the critical pressure, temperature and specific volume respectively, and ω is the accentric factor.

Fluid name	MW (kg/kmol)	P_c (bar)	T_c(oC)	ν_c (m^3/kg)	ω
D_6	444.92	9.61	372.6	0.0036	0.736

2.2 Facility Characterization

A series of leak tests were performed to assess the leak rate of the facility. The tightness of the setup is characterized using the average leak rate, defined as LR = $V\Delta P\ \Delta t^{-1}$, where V is the volume of the setup ($\sim$ 0.0032 m^3), and Δp is the rise/drop in pressure measured after a time interval of Δt. At vacuum conditions, a LR of 2.2×10^{-4} mbar·l·s^{-1} was measured (air leaking into the system). Leakage tests at superatmospheric pressures conducted at different temperatures by filling the setup with air were consistently below 5×10^{-4} mbar·l·s^{-1} (air escaping the setup). These rates were deemed sufficient for the conceived tests.

The measuring principle of the ASTER is based on the time of flight method, which is a common technique employed to measure the speed of sound in gases. The propagating rarefaction wave in the CT is measured by the pressure sensors PT1-PT4 that are placed 20 cm apart from each other. The wave speed is then calculated as,

$$w = \frac{\Delta l}{\Delta t} \tag{1}$$

where Δl is the distance between a sensor pair, and Δt is the time difference of arrival of the wave at any pair of sensors. The distance Δl is calibrated with air, since it is a well-studied fluid for which accurate speed of sound data is available in literature [11]. The change in Δl due to temperature is accounted for using,

$$\Delta l_{p,T} = \Delta l_{p_0,T_0}(1 + \alpha(T - T_0)) \tag{2}$$

where $\Delta l_{p_0,T_0}$ is the length calibrated at p_0 = 8.74 bar and T_0 = 43 °C and α is the thermal expansion coefficient.

3 Results

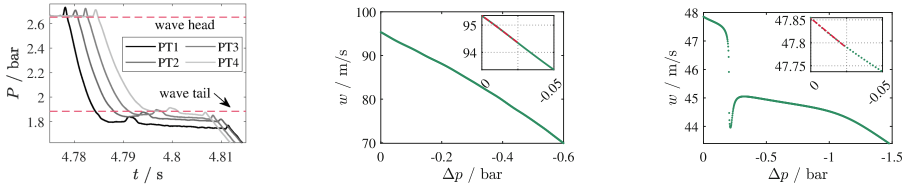
3.1 Rarefaction Tests in D_6

A series of rarefaction tests were performed with D_6 at thermodynamic conditions in both the ideal-gas region and close to the saturation curve where NICFD effects can be expected. A summary of the measured values of the temperature and pressure and the corresponding thermodynamic properties are shown in Table 2.

Table 2. Thermodynamic conditions of the initial states of the rarefaction experiments. Γ and c for Tests 1 & 2 are estimated using the iPRSV EoS [12] while 3 & 4 are computed using the Helmholtz based EoS [13]

Test No	P_0 (bar)	T_0 (°C)	Γ_{model} (-)	Z (-)	c_{model}(m/s)	c_{exp}(m/s)	Δ c(%)
1	2.66	349.8	0.86	0.87	94.23	95.55	1.44
2	5.85	357.6	0.58	0.69	73.35	74.67	1.79
3	8.87	370.9	0.096	0.49	48.18	47.94	–0.48
4	8.96	369.6	–0.052	0.46	43.08	46.62	8.21

The speed of propagation, w, of the wave is estimated using a time-of-flight (ToF) method. Before its implementation, the signals from the pressure transducers are filtered using a zero-phase digital filter to limit the impact of noise. The ToF method is then implemented as follows: first, the head of the wave is identified as the point at which the pressure differs from the initial value P_0 by more than 10 mbar (see Fig. 2a). This threshold was found sufficient to eliminate the noise generated by the strike of the hammer. The tail of the wave is arbitrarily chosen such that only the smooth pressure drop across the wave is analysed and the wave reflections arising from the end-wall of the CT are neglected. This span is divided into several intervals and the ToF is applied to the corresponding subintervals of the different sensors to compute the local wave propagation speed.



(a) (b) (c)

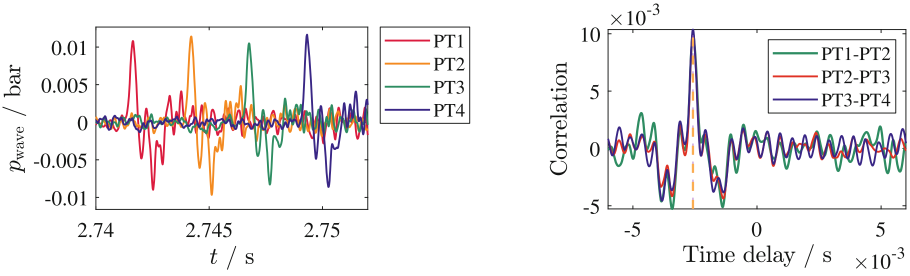
Fig. 2. (a) Recordings from sensors PT1 to PT4 for experiment 1. Red dotted lines show the limits of the relevant span of signals for ToF method; Wavespeed vs. Δp in D_6 for (b) Experiment 1 and (c) Experiment 3 (see Table 2). Figures in the inset show linear fit in dashed red line for soundspeed estimation. (Color figure online)

Figures 2b and 2c show the wavespeeds estimated using the ToF method for experiments 1 & 3 (see Table 2) respectively. For conditions in the ideal-gas region, a linear decrease in sound speed with pressure drop is expected. This is also observed in the wavespeed plot for experiment 1. The variation of the wavespeed with Δp for experiment 4, however, is not strictly linear as in the ideal case. This is an indication of the presence of NICFD effects since the wavespeed is no longer dependent only on the soundspeed and density, but also on Γ, whose value is lesser than 1 and variable in this thermodynamic region. In both cases, wavespeed monotonically decreases with Δp, indicating that Γ is always positive in these examples.

A linear fitting curve close to the head of the wave can give an estimate of the speed of sound in the undisturbed state since the wave propagation speed tends to the sound speed as $\Delta P \rightarrow 0$. The average error and the standard deviation with respect to the sound speed predicted by the thermodynamic model are $1.44\% \pm 1.63\%$ in the case of Test 1. This is very close to the model predictions, given that the model is valid only for pure D_6 while the one used in the experiments is only 97.4% pure. The predicted and the computed speeds of sound for the performed experiments are shown in Table 2. It is seen that as the experimental conditions get closer to and into the $\Gamma < 0$ region, the deviation between the model and the experimentally calculated sound speeds increases. This is expected since the thermodynamic model for D_6 is known to be inaccurate by as much as 30% in this region [7].

3.2 Alternate Speed of Sound Measurements

Though the speed of sound in D_6 can be extracted from rarefaction experiments, such runs don't allow for quick and repeated measurements at different conditions since the glass disk needs to be replaced between successive tests. This can be done only when the setup has cooled down, which increases the downtime between the experiments. To facilitate the rapid measurement of sound speeds at various thermodynamic states, an alternate approach to measuring the speed of sound is proposed here. Instead of using a glass disk, a 3 mm thick metallic disk capable of withstanding repeated strikes from the hammer is used as a barrier element. The metallic disk is actuated enough to



(a) *(b)*

Fig. 3. (a) Over-pressure recording of a small-amplitude wave by sensors PT1 to PT4 (b) Output of cross-correlation technique showing a peak at time-delay between sensors

generate a small-amplitude wave in the CT. A cross-correlation (CC) technique for the ToF method is applied to the signals to estimate the sound speed. Cross-correlation is considered a more optimal ToF implementation since it is less sensitive to noise and uses all the information contained in the signals to produce a maximum value at the time delay between the two signals [14].

Figure 3a shows the wave amplitude recordings from the pressure sensors from a sound speed test in D_6 at 6.08 bar and 368.9 °C. The results from the CC of the different sensor pairs and the estimated time delay between the sensors are shown in Fig. 3b. Using Eq. 1, the sound speed in D_6 at these conditions is calculated to be 78.22 m/s, which is 2.8% higher than the model estimated value of 76.01 m/s. Figure 4 shows the measured sound speed values in D_6 at different temperatures and the corresponding values predicted by the thermodynamic model. The comparison shows a very good agreement in terms of the trend in c vs T. However, the model is seen to consistently underpredict the speed of sound by approximately 2.5%.

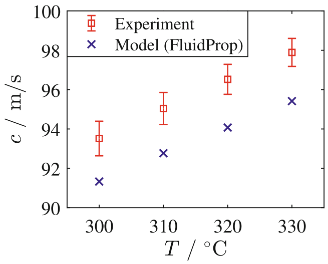


Fig. 4. Measurements of speeds of sound in D_6 at different temperatures and corresponding model predictions

4 Conclusion and Future Work

A new shock-tube facility for measurements on waves propagating in NICFD flows of organic vapours has been designed, developed and successfully tested in the Propulsion & Power Group at TU Delft. The facility is designed in particular to generate a nonclassical rarefaction shock wave in siloxane D_6 and is capable of withstanding up to 15 bar and 400 °C. Preliminary experiments on rarefaction waves in the non-ideal thermodynamic region of siloxane D_6 at temperatures and pressures up to 370 °C and 9 bar have been successfully performed. The speed of sound estimated from wavespeed measurements agrees within 2% of the state-of-the-are thermodynamic model predictions in the ideal-gas region but deviates at high P and T. A stand-alone approach to measure soundspeeds is also presented and preliminary measurements at temperatures up to 330 °C is reported, providing soundspeed values within 2.5% of the model predictions. These predicted values are however affected by high uncertainties of up to 30%. Further measurements are planned at thermodynamic conditions where nonclassical gasdynamic effects are expected. Sound speed measurements in this region can contribute towards improving the thermodynamic modelling of the fluids of interest.

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